This PDF is available from The National Academies Press at http://www.nap.edu/catalog.php?record_id=20027

FROM THE ARCHIVES

Objective Methods for Food Evaluation: Proceedings of a Symposium (1976)

Pages
310

Size
8.5 x 10

ISBN
0309025206

Committee on Food Stability; Advisory Board on Military Personnel Supplies; Commission on Sociotechnical Systems; National Research Council; U.S. Army Natick Research and Development Command

Find Similar Titles | More Information

Visit the National Academies Press online and register for...

- Instant access to free PDF downloads of titles from the
  - NATIONAL ACADEMY OF SCIENCES
  - NATIONAL ACADEMY OF ENGINEERING
  - INSTITUTE OF MEDICINE
  - NATIONAL RESEARCH COUNCIL
- 10% off print titles
- Custom notification of new releases in your field of interest
- Special offers and discounts

Distribution, posting, or copying of this PDF is strictly prohibited without written permission of the National Academies Press. Unless otherwise indicated, all materials in this PDF are copyrighted by the National Academy of Sciences.

To request permission to reprint or otherwise distribute portions of this publication contact our Customer Service Department at 800-624-6242.

Copyright © National Academy of Sciences. All rights reserved.

NATIONAL ACADEMY OF SCIENCES 1863–2013 Celebrating 150 Years of Service to the Nation

OBJECTIVE METHODS FOR FOOD EVALUATION
Proceedings of a Symposium

November 7-8, 1974

Boston Marriott
Newton, Massachusetts

Sponsored by

U.S. Army Natick Research and
Development Command
Natick, Massachusetts

Committee on Food Stability
Advisory Board on Military
Personnel Supplies
Commission on Sociotechnical
Systems
National Research Council

National Academy of Sciences
Washington, D.C.
1976

NAS-NAE
AUG 10 1976
LIBRARY

NOTICE: The project that is the subject of this report was approved by the Governing Board of the National Research Council, whose members are drawn from the Councils of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine. The members of the Committee responsible for the report were chosen for their special competences and with regard for appropriate balance.

This report has been reviewed by a group other than the authors according to procedures approved by a Report Review Committee consisting of members of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine.

International Standard Book Number 0-309-02520-6
Library of Congress Catalog Card Number 76-26723

Available from

Printing and Publishing Office
National Academy of Sciences
2101 Constitution Avenue, N.W.
Washington, D.C. 20418

Printed in the United States of America

80 79 78 77 76 10 9 8 7 6 5 4 3 2 1

FOREWORD

This Symposium was convened because of our continuing need for objective methods for assessment of food quality. This need is broadly shared among the many institutions -- military, industrial, academic, and social -- who procure and dispense a wide variety of foods according to their diverse requirements. The bibliographies accompanying the papers in these Proceedings reflect the large volume of published literature that has been generated by these needs. This literature, however, does not reflect much of the practical experience acquired by specialists who deal with the specifics of product assessment on a day-to-day basis. In organizing this Symposium, the goal of the Program Committee was to attract active participants who could reflect such experience. We believe that these Proceedings demonstrate the success of the Committee. We have here a summary of the status of objective methods in the product areas considered and a guide around the possible pitfalls in the application of these methods.

The Committee on Food Stability served also as the Program Committee. We are particularly grateful to its members. Dr. Amihud Kramer of the University of Maryland, former Chairman of the Committee on Food Stability, was very helpful in the early discussions about this Symposium during the late 1960's. Special acknowledgement is due Dr. Frank R. Fisher, Executive Director of the Advisory Board on Military Personnel Supplies and his staff for the efficient management of the administrative and operational activities which contributed to a most fruitful meeting.

Rufus E. Lester, Jr.
Colonel, Quartermaster Corps
Commanding
U.S. Army Natick Research
and Development Command

ADVISORY BOARD ON MILITARY PERSONNEL SUPPLIES

Chairman:

William O. Baker, President
Bell Telephone Laboratories, Inc.
600 Mountain Avenue
Murray Hill, New Jersey 07974

Executive Director:

Frank R. Fisher

Symposium Chairman:

Delbert M. Doty
Route 2, Box 132A
Buchanan, Tennessee 38222

Committee on Food Stability

Chairman:

Roy Teranishi
Research Leader
Food Quality Research Unit
Western Regional Research Center
U.S. Department of Agriculture, ARS
800 Buchanan Street
Albany, California 94710

Nuri N. Mohsenin
Professor, Department of
Agricultural Engineering
The Pennsylvania State University
University Park, Pennsylvania 16802

Harold S. Olcott
Professor, Marine Food Science
Institute of Marine Resources
Department of Food Science
and Technology
University of California
Davis, California 95616

Arthur J. Siedler
Head, Department of Food Science
567 Bevier Hall
University of Illinois
Urbana, Illinois 61801

CONTENTS

G E N E R A L S E S S I O N

Chairman,

Dale H. Sieling

Technical Director
U.S. Army Natick Laboratories

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

SYMPOSIUM OBJECTIVES

Delbert M. Doty

The stated purpose of this Symposium is to explore and summarize the current status of objective methods for 1) clarifying and improving specifications for procurement of foods for the military, 2) predicting storage life or determining past history of military food items, and 3) evaluating by appropriate research the applicability of existing objective methods for new food products.

Let us examine, for a moment, the unique need of the military for objective methods to evaluate foods. To assure high quality and long shelf-life of many military food items, products must be purchased on specifications. Good specification documents are therefore a "must" for the procurement process. And good specification documents ideally require good end-product tests. End-product tests that are readily understood by both the supplier and the procurement agencies must be objective rather than subjective.

It is true, of course, that objective end-point testing of foods and food ingredients is used to some extent in the day-to-day operations of the food industry. But in many cases these tests are used to establish and maintain a prescribed level of economic value rather than a minimum quality level. The same basic philosophy applies to some extent to grade standards that have been established for many food products. These standards may relate roughly to quality, but their primary use and function is to provide a basis for day-to-day commercial transactions. For this purpose, the objective tests used for the establishment and maintenance of grade standards are adequate. But the need for objective end-product test methods for military food specifications is much more critical.

Obviously, a short symposium of this nature cannot cover the whole spectrum of foods and food products procured for the military. Consequently, this Symposium will attempt to concentrate primarily on meats and meat products, including fish and poultry products, and fats and oils in prepared foods. Furthermore, the talks and Round Table discussions will not include, except incidentally, evaluation of objective methods for determining nutritional or microbiological quality of foods and food products.

However, the Round Table discussions are somewhat broader in scope and are designed to consider objective methods for evaluating texture, appearance, flavor, and fat stability from a more general basic standpoint without limiting the discussions strictly to meats and meat products.

There may be some individuals who feel that a symposium of this type is not necessary -- that all valid objective methods for evaluating food quality are available in the scientific and technical literature. I do not think that this is necessarily true. Furthermore, many objective techniques are applied in new ways and to a variety of products that may not be obvious from the original application. It is only in a meeting of this type, which includes representatives of industry, government agencies, independent laboratories, and universities, that the complete picture of objective methods and their uses can be developed. In addition, it is hoped that a comprehensive discussion of methods now used only for research purposes may lead to the development of simplified techniques suitable for use by procurement agencies.

Thus, this Symposium will attempt to summarize and update our knowledge on objective methods for the evaluation of sensory characteristics of food, especially meat and meat products and the stability of fat in prepared high-fat food products. We hope that the formal presentations and the Round Table discussions will be of interest and value to all of you -- those representing the military, as well as scientists from industry, government, and universities.

T E C H N I C A L S E S S I O N I

Chairman,

Delbert M. Doty

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

EVALUATION OF BEEF TEXTURE

Harold K. Herring

In the last decade, we have seen a proliferation in development of objective methods for food evaluation. Many of these methods have been developed by those interested in meat texture. More specifically, the majority of the work has been with beef because of the importance of texture in beef acceptance and because of the variation in beef texture, which is due to many factors.

Traditionally (including USDA Grading Standards), meat texture has been defined in terms of appearance and is a visual function of the size of bundles of fibers, or fasciculi, due to the presence of the perimysium[1]. In more modern terms, texture, or the kinesthetic (or hapaethetic) characteristics of food, relates to those attributes of quality associated with the sense of feel as experienced by the fingers, the hands, or the mouth. Texture, therefore, is capable of precise objective measurement by mechanical or instrumental means[2].

Meat texture encompasses those properties that affect how meat feels in the mouth and includes tenderness, toughness, juiciness, firmness, mealiness, stringiness, chewiness, hardness, and softness. Szczesniak and Torgeson[3] state "Since the structural properties of a food cannot be separated from its eating quality, it is preferable (following the precedent set for other foods) to use the more inclusive term 'texture' in discussing that general class of characteristics."

Meat tenderness and toughness, of all the quality attributes, are the most important in affecting consumer acceptance. It is not too surprising to find, therefore, that most studies on meat texture have dealt primarily with tenderness or toughness.

While this Symposium deals with objective methods, I'd like to point out that ever present in our minds must be the relationship a particular objective or instrumental method has to subjective or sensory panel findings. Hence, most instrumental methods when developed for texture measurement are evaluated in relation to how human subjects rate or evaluate the same product.

FACTORS AFFECTING MEAT TEXTURE

Before discussing objective methods and instruments used for evaluation of texture, a review of some of those factors that can affect it seems appropriate. More thorough reviews on factors affecting texture are available[3-6].

Structural Relationships

The beef animal consists of some 300 muscles that have structural and physiological similarities and dissimilarities. The highest-quality muscles, those that command the highest price, are located along the dorsal area between the fifth thoracie and last lumbar vertebrae. Other muscles in the neck, i.e., brisket, plate, shank, and flank are tough and sometimes ground or chopped and included in processed meats.

The toughness of individual muscles can be altered. If the animal when slaughtered is held or placed in a position approximating that in life, rather than being hung from the hind leg, some muscles along the dorsal region and in the round are improved in tenderness while others are toughened[7,8]. Thus, muscle microstructure becomes a quantitative predictor of the change in tenderness. Hanging a carcass by the aitch bone improves tenderness of the longissimus by 21 to 27 percent, the semimembranosus by up to 25 percent, the semitendinosus by 3 to 19 percent, the biceps femoris by up to 27 percent, the gluteus medius by 15 percent, and the adductor by 18 percent[7-9]. However, the psoas major is toughened by 17 percent and the rectus femoris 18 percent using this method. These treatments alter the repeating unit of muscle fibrils (sarcomere length) by exerting tension and stretching the muscles. It was once thought that the effect of sarcomere length was exemplified primarily on the muscle fibers. However, it has since been shown that stretching muscle can cause measurable structural changes in the ratios of the different types of collagen components, or α, β, and γ subunits[10,11]. Therefore, an increase in α subunits is related to an increase in tenderness.

The Role of Connective Tissue

Animal age is the primary factor determining how strongly connective tissue exerts its effect on texture of a given muscle. As the animal matures, the covalent cross-linking of collagen is increased and the solubility of collagen in various media and after heating is decreased[12-14]. Muscle-to-muscle variation in tenderness can often be attributed to differences in the amount of connective tissue[14-16]. Muscles full of connective tissue are tougher than those that are low, e.g., the latissimus dorsi and biceps femoris are high and the psoas major and serratus ventralis are low in collagen.

Cold-Shortening

Let's return to the role of the muscle fiber in toughness. It had long been known that severe toughness could occur in meat from young animals[17]. This was a variable phenomenon, however, and when a muscle was cooked at varying times after death of the animal, it may or may not have been tough. Then a paper published in 1960 by Locker[18] of the Meat Industry Research Institute of New Zealand provided a clue as to what was happening. Extreme muscle shortening or contraction of excised (cut) psoas major muscle was associated with the development of toughness. Contraction was proved by evidence of shortened sarcomere lengths to 1.8 μm or less as determined by phase microscopy. Non-excised psoas muscles were stretched and had sarcomere lengths of 3.5 μm.

Further work[19] at the New Zealand Institute indicated that pre-rigor muscle, if free, was stimulated to contract by cold air temperatures of 2 to 5°C, and the shortened muscle was tough after cooking. If muscle shortens by more than 20 percent to sarcomere lengths of 1.8 μm or less, meat toughness is usually evident. This shortening-type toughness was termed "actomyosin toughness" by Marsh and Leet[20], as opposed to "background toughness," which they had observed in unshortened muscles of old cows and bulls.

It is commercial practice in this country to chill beef carcasses in "hot boxes" with fast moving air of about 0°C. Since there is ample fat covering and carcasses are packed tightly, problems with extreme toughness due to cold shortening are minimal[21].

Aging Post-Mortem

The effect of post-mortem aging on beef texture is well-documented[3,4]. Physical and chemical alterations lead to improved texture, which reaches a maximum after aging for 10-12 days. Protein solubility increases, water-holding capacity increases, rigor mortis is resolved[22], and tenderness improves.

Recently, investigations have been carried out by Goll and coworkers at Iowa State in order to define and clarify post-mortem changes in beef in relation to tenderness improvement due to aging[23-30]. This first well-documented, post-mortem change is a modification of the actin-myosin interaction which results in a) changes in ATPase activities of myofibrils, b) changes in *in-vitro* contractile properties of actomyosin, c) lengthening of rigor-shortened sarcomeres, d) changes in dissociability of the actin-myosin complex. The underlying causes are 1) limited proteolysis by a calcium-activated enzyme (see discussion below), 2) post-mortem alteration of actomyosin sulfhydryl groups, and 3) post-mortem pH decline[22-24,29,30].

A second phenomenon that is certain to help explain texture improvement has also been seen. This phenomenon is the gradual

disintegration of the Z line during post-mortem aging[27-29]. There is a weakening of the lateral attachments that maintain myofibrils in precise register, probably at the level of the Z line[31]. There is also a weakening of structures within the myofibrils, resulting in a break at the junction of the thin filament with the Z line. Third, actual loss of some of the Z-line structure leading in a few cases to its complete dissolution has been observed, using light and electron microscopy[32-34]. This degradation and disruption is apparently due to a calcium-activated protease, which becomes activated due to the loss of calcium from the sarcoplasmic reticulum after aging commences. This proteolytic enzyme has been isolated and partially purified and characterized[24,35].

There may also be subtle, yet chemically undefined, changes in collagen during aging. Proteolysis of collagen has been described by Laakkonen[36]. The available evidence indicates this proteolysis may be due to the action of a calcium-activated enzyme as EDTA inhibits its activity.

These changes, then, are involved in the aging phenomenon in meat, which is so important to texture and consumer acceptance.

Other Factors

Some other factors have been involved in meat texture and changes in texture[3,4]. These will only be listed:

1) possible role of sarcoplasmic proteins -- (apparently include proteolytic enzymes)
2) ground substance
3) water-holding capacity
4) pH
5) cooking time and temperature
6) marbling or intramuscular fat (important in juiciness and flavor)
7) fiber size
8) fiber-bundle size
9) fiber extensibility
10) animal factors such as breed, sex, strain, diet, and ante-mortem stress

METHODS OF MEASURING BEEF TEXTURE

Development of methodology for the measurement of meat texture dates back to 1907 when Lehmann (cited in [3]) described two mechanical devices. Needless to say, there has been considerable activity since then in development of instrumental test methods. Since it has been realized that texture is extremely important in consumer acceptance, considerable attention has been paid to correlating instrumental methods with sensory methods. In many circumstances sensory methods alone are impractical, such as in

abbatoirs, or in long studies (progeny testing or genetic studies); therefore, an objective method needed to be developed. Szczesniak[37] has stated that sensory evaluation is faced with many methodological, psychological, and physiological problems. It is time-consuming and costly, and for many people the results have a connotation of opinion. Instrumental methods are more apt to be regarded as relying upon "facts"[37].

Objective methods for measuring texture can be classified as physical, or instrumental, chemical, and histological. For purposes of this discussion, however, only instrumental methods will be reviewed.

Instruments used for evaluating meat tenderness can be classified according to their principal action: shearing, biting, penetrating, stretching and breaking, and compressing[37,38]. For more thorough reviews of different methods, Pearson[39] Szczesniak[37,40], Finney[41], and Voisey[42] should be referred to.

Shearing Devices

Warner-Bratzler Shear

This apparatus employs a single blade and measures the maximum force required to shear a core sample of meat (Figure 1[43]). It is simple, low in cost, and easy to use. The blade is 1 mm thick and has a triangular hole in which the meat is placed. The blade is led through a slit between two bars by a motor-driven gear system. Force exerted on the blade is detected by a dynamometer spring and is read on a dial scale.

This instrument is the most widely used for measuring tenderness and usually correlates well with sensory measurements[3]. However, some workers have been disappointed with its correlation with sensory tenderness. Szczesniak and Torgeson[3] stated that in 41 of 51 studies good to highly significant correlations between Warner-Bratzler Shear and sensory tenderness were found. Correlations ranged from +.17 to -.90 for beef. Thus, the reliability of this instrument has sometimes been questioned. It is highly susceptible to experimental error[44,45]. Consequently, efforts have been made to improve upon the Warner-Bratzler shear. Voisey and Hansen[46] developed an instrument that records force versus time using the same principle as the Warner-Bratzler but with 3 blades. Bouton *et al.*[47] and the author[21] have used a modified Warner-Bratzler shear available from Instron, which is used with the Instron Universal Testing Machine (Figure 2). The Instron permits one to record force-distance (time) curves to quantify parameters of texture other than maximum shear force such as time, or deformation at failure, slope of the curve, and work expended in shearing.

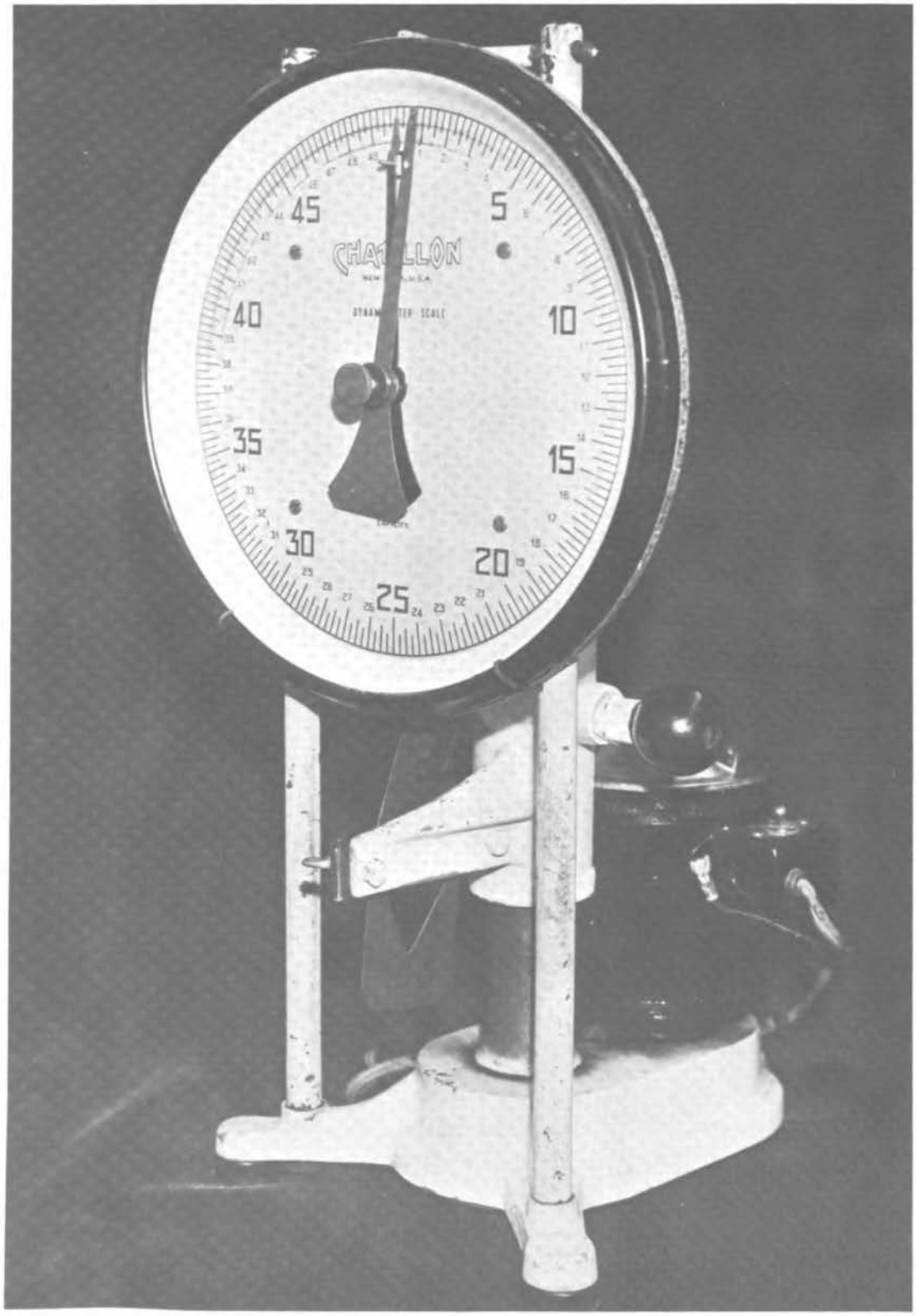


FIGURE 1 Warner-Bratzler Shear Press.

FIGURE 2 Instron Universal Testing Machine with Warner-Bratzler attachment.

Kramer Shear Press

The L. E. E. Kramer Shear Press was developed by Kramer and his associates at the University of Maryland for use on fruits and vegetables (Figure 3 [48]). It was subsequently adapted to meat. This instrument uses a stationary rectangular box with slots in the bottom to hold the sample and a moving probe composed of 10 bars 0.114 in. thick to drive through a sample. The probe with bars shearing the meat is driven by a hydraulic drive system powered by a gear pump attached to an electric motor. Resistance is measured by the compression of a proving ring and is read out usually on force-distance recorder. The Kramer Shear Press, although more expensive than the Warner-Bratzler Shear has been used quite extensively for measuring meat tenderness. Correlations of Kramer Shear Press and sensory tenderness have ranged from -.18 to -.89 by different investigators[3].

A drawback of this system is the necessity of making a standard sample size. However, this instrument, as was the case with the Warner-Bratzler Shear, correlates well with sensory tenderness. Also, shear-force measurements on the 2 instruments correlate well with each other[3,39].

Biting Devices

Voledkevich Bite Tenderometer

The Voledkevich apparatus was probably the first instrument developed to measure texture in a manner simulating biting by the teeth[49]. Two wedges with rounded points are substituted for teeth. The lower wedge is fixed on a frame while the upper one is moved vertically by the action of levers. Meat, usually a slice of a certain thickness, is placed on the lower wedge and its resistance to the squeezing force is recorded on a revolving drum, giving a force-distance curve.

Sale[50] modified this apparatus, rounding the wedges to a radius of curvature that is durable and easy to reproduce (Figure 4).

Mac Farlane and Marer[51] of the Meat Industry Research Institute of New Zealand described a modified Voledkevich apparatus that consists of a pivoted horizontal beam, counter-balanced at one end and on top of which is mounted a 4-wheeled loaded carriage. The blunt wedges are used on a 5-8 mm thick sample and a steadily increasing load is applied by the loaded carriage, which is moved by a motor-operated screw. Shearing load, which is proportional to elapsed time, and work are recorded on a revolving drum. Marsh *et al.*[52] reported sensory assessment of tenderness was either 1) inversely proportional to force required to shear the sample (r = .72 to .93), or 2) directly related to the square root of the force (r = -.68 to -.95).

FIGURE 3 Kramer Shear Press.

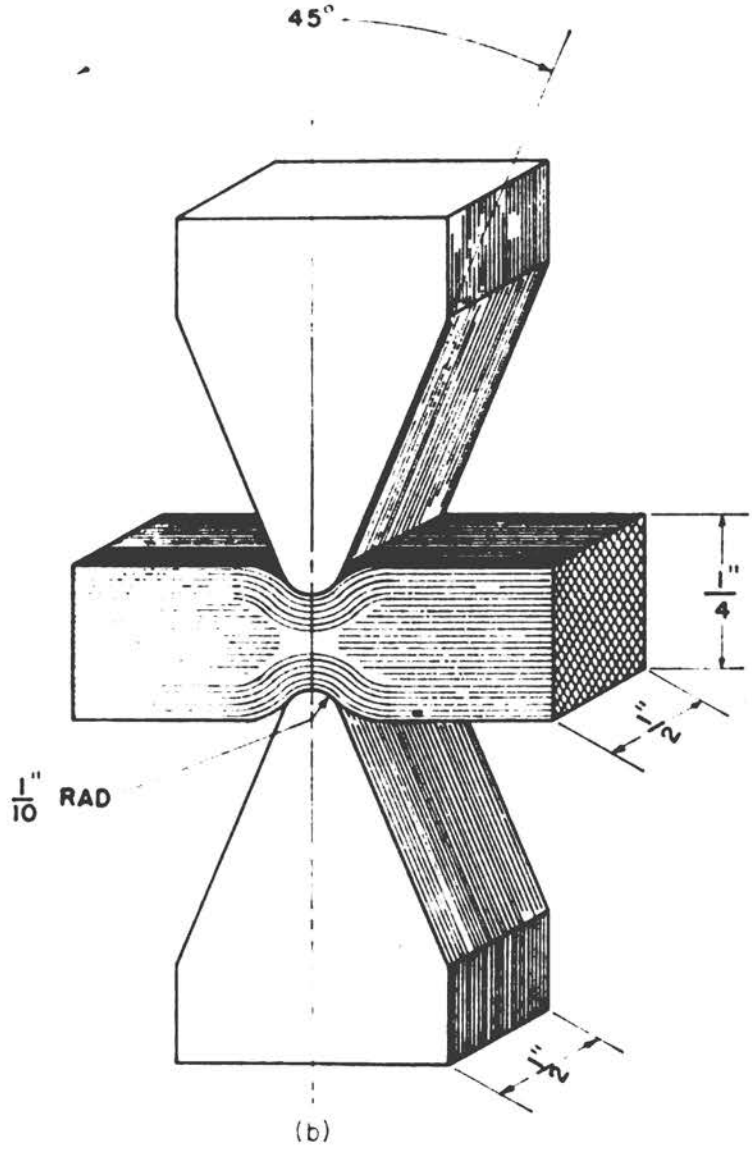


FIGURE 4 Wedges of bite Tenderometer (after Sale[50]).

More recently, Rhodes *et al.*[53] reported a modified Voledkevich apparatus adapted to the Instron (Figure 5). Samples 1 cm thick were used and a force-deformation curve was obtained (Figure 6). Data obtained from the curve included 1) three deformation or distance measurements, 2) three force measurements, 3) three work measurements, and 4) a slope measurement. Correlations of instrumental parameters with subjective assessments of texture were higher (r = .5 to .85) on samples tasted cold than those tasted warm (r = .45 to .65). Force measurements and work measurements were the factors accounting for the highest variance in subjective texture assessments.

General Foods Texturometer

This instrument was developed in the research laboratories of General Foods and was a modification of the Massachusetts Institute of Technology Denture Tenderometer[37,54]. The instrument is comprised of a mechanical masticator or Hanau articulator driven by a variable-speed motor, a power supply, a Wheatstone-bridge circuit with a balancing potentiometer, and a fast-speed recorder. The strain-gage sensing unit is positioned on a plate support arm. Several chewing speeds and plunger sizes and shapes are available. The plunger first exerts a little shearing action with its edge and then compresses the food with the entire surface. A sample is placed unconfined on the platform and then the force-distance recording of the plunging action is made through 2 "chews" (Figure 7). Qualities of meat that are measured include 1) hardness, 2) cohesiveness, 3) elasticity, and 4) chewiness. Chewiness was calculated as a product of hardness, cohesiveness, and elasticity. Szczesniak[3] reported highly significant correlations with a trained panel (hardness, r = 77; chewiness, r = .64; juiciness, r = .55). More recently, Kapsalis

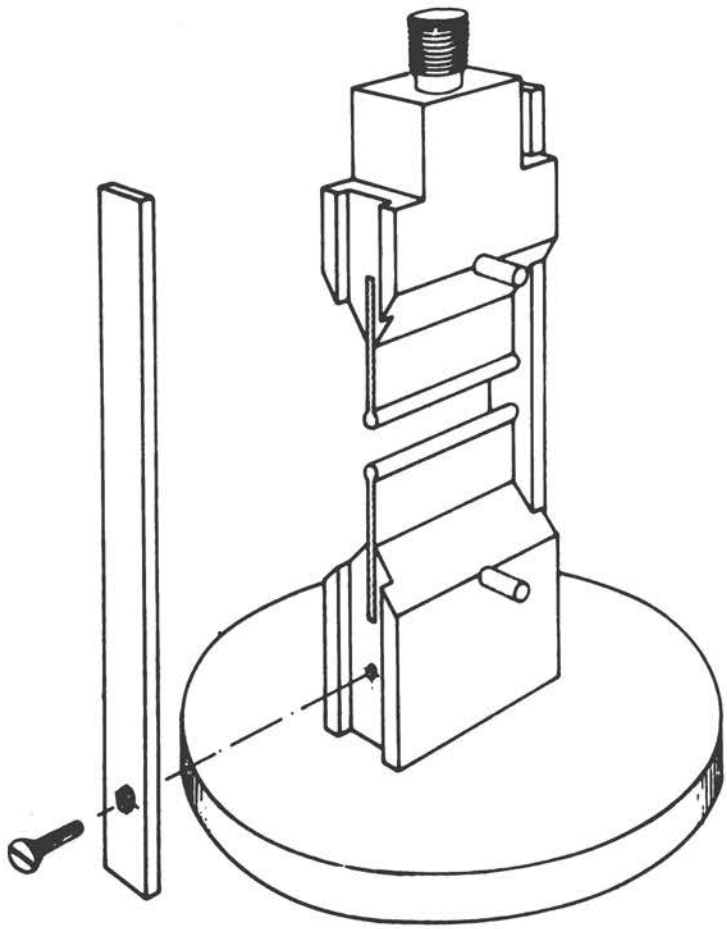


FIGURE 5 Voledkevich blunt-type jaws used for texture testing (courtesy *J. Texture Studies*[53]).

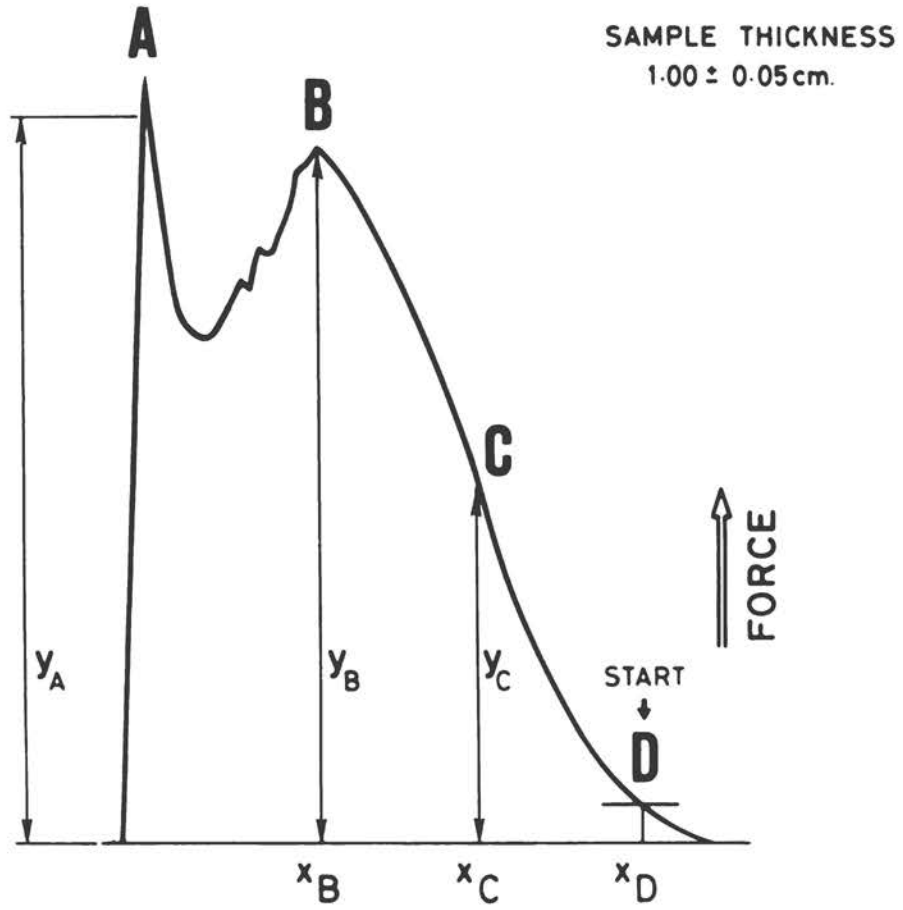


FIGURE 6 Force-deformation curve for roast beef under compression between blunt jaws[53].

et al.[55] using a modified General Foods Texturometer, or Masticometer, reported significant correlations of sensory tenderness with force (r = .42 to .77), cohesiveness (r = -.71 to -.92), and crushability index (r = .84 to .99).

Penetrating Instruments

Slice Tenderness Evaluator (STE)

This instrument was designed at the USDA and uses a combination of penetration and shear with the Instron[56]. A thin slice of meat (.250 in.) is mounted in a sample holder and held in position with a cover plate. It is first punctured with .125 in. tip and then sheared by a .372 in. circular rod by a sudden change in diameter. There is a clearance of 0.003 in.

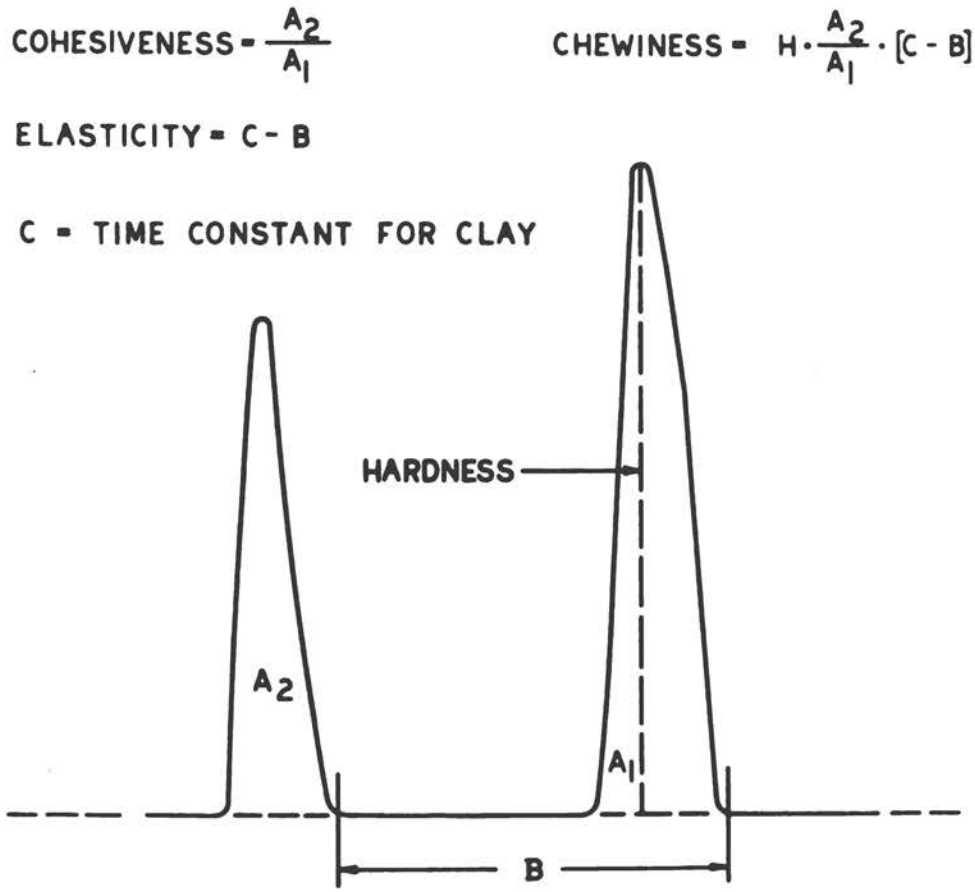


FIGURE 7 A typical Texturometer curve for meat (courtesy Academic Press[3]).

between the shearing edge and the opening in the base plate. A force-distance curve is obtained. Alsmeyer *et al.*[56] reported correlations for beef longissimus of STE shear and sensory tenderness (r = -.71) and STE puncture and tenderness (r = -.55).

U.S. Army Meat Penetrometer

The meat Penetrometer developed at the U.S. Army Natick Laboratories consists of 5 semi-blunt needles used to pierce a slice of meat with an Allo-Kramer shear press[57,58]. The semi-blunt needles, .125 in. in diameter, are pressed through a slice of meat and through annular openings in the base portion. The land diameter is .007 in. (but in the patent it is described as .059 in.[58] and the taper was .472 in./in.). The authors[57] reported correlations of Penetrometer forces and sensory scores of .63 to .86 (lower sensory score, more tender) with pork. In a later report on beef from the same laboratory, Galloway *et al.*[59]

reported a correlation of raw meat samples and cooked taste panel scores of -.78 and a correlation of -.84 for cooked meat Penetrometer readings and taste panel scores. The muscles used were biceps femoris and longissimus; data were apparently pooled for statistical analysis.

Penetrometer (CSIRO)

Bouton *et al.*[47] at the Meat Research Laboratory of CSIRO Division of the Food Preservation Laboratory of Australia, described the use of a .63 cm (.250 in.) diameter flat-ended plunger, which was driven vertically 80 percent of the way through a 1.3 cm thick sample of cooked meat with the Instron. Muscle fibers were oriented perpendicular to the direction of plunger penetration. The plunger was driven twice into the meat at a given location and the force penetration curves were recorded. "Hardness" was the force required to achieve the first penetration and "cohesiveness" the ratio of work done during the second penetration to that work performed on the first. "Chewiness" was defined as a product of "hardness" and "cohesiveness." They found Instron "hardness" was highly correlated with "initial impression" (r = .88), while "chewiness" was highly correlated with "residual impression" (r = .90).

Armour Tenderometer

Hansen[60] reported the development of the Armour Tenderometer, which utilizes a probe with ten .125 in. diameter needles (.750 in. apart) in 2 rows, 1 in. apart. The probe is attached to a strain-gage force transducer (Figure 8). The probe is pushed 2 in. into raw meat and the peak force signal is read off a dial housed in a small box. This instrument was designed to provide a non-destructive tenderness test for selection of beef carcasses. Correlations were reported for peak force with sensory tenderness in the Choice grade (r = -.77), and in the Good grade (r = -.69). Measurements were taken on the rib eye of cattle the day after slaughter, and sensory tenderness was determined after aging the beef 1 week.

Studies with the Armour Tenderometer have been reported also by some universities. In one study[61] with 99 uniform Angus steer carcasses, a partial correlation analysis between Tenderometer and shear force, holding either extract and longissimus muscle area constant, gave a value of .43 ($P < .01$). This indicates that fat content and muscle area size affect the Armour Tenderometer determinations. Carpenter *et al.*[62] reported the Armour Tenderometer was effective in grouping Choice beef carcasses into tough and tender categories as measured by both sensory tenderness and Warner-Bratzler Shear. Correlations of sensory tenderness and Armour Tenderometer values ranged from -.15 for the Good grade to -.35 for the Choice grade.

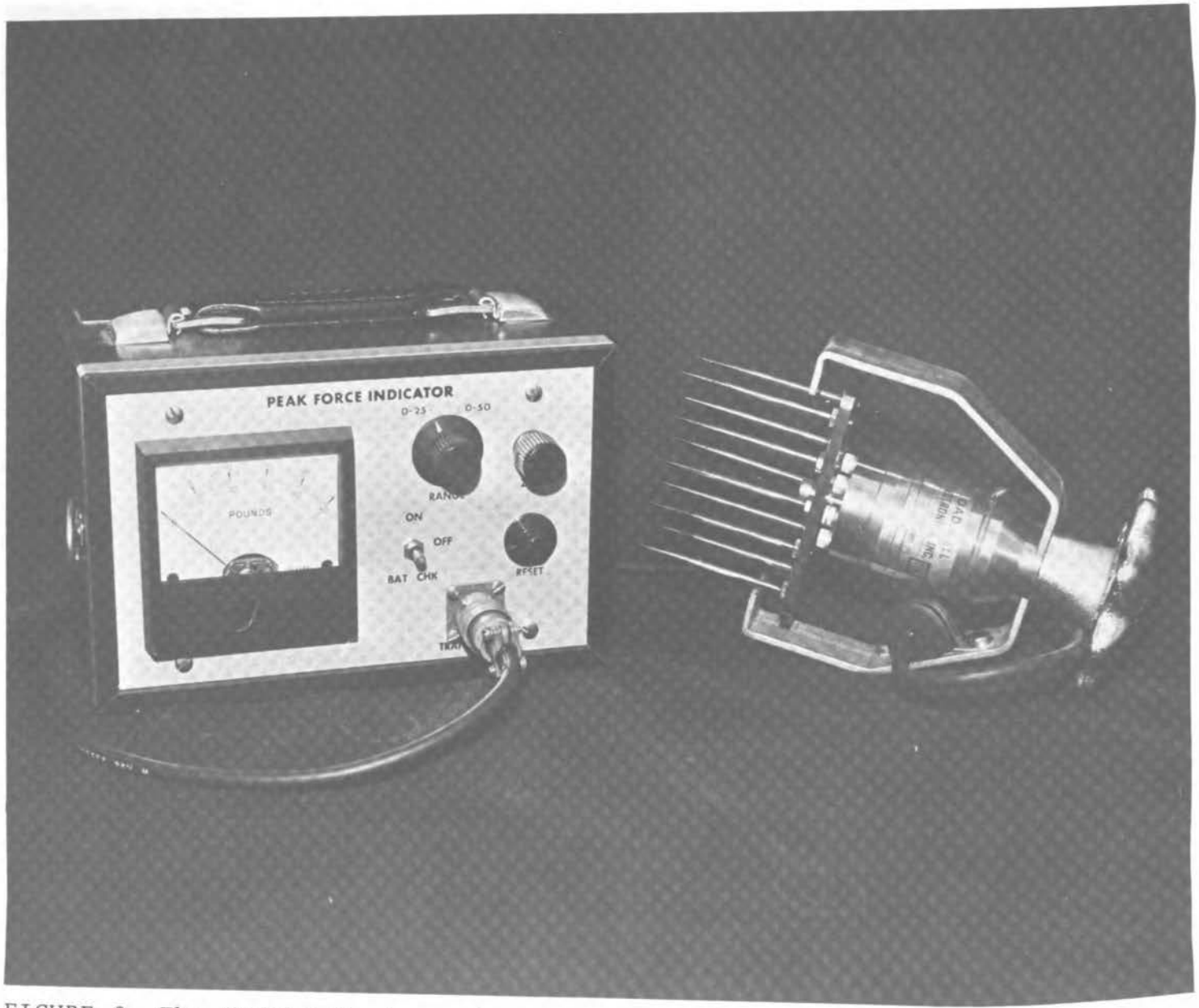


FIGURE 8 The Armour Tenderometer.

Parrish *et al.*[63] reported correlations of Armour Tenderometer values at 1 day and sensory tenderness of -.40 at 1 day, -.49 at 3 days, and -.34 at 7 days. In another experiment, sensory tenderness of rib steaks was related with Warner-Bratzler Shear values (r = -.83) and Armour Tenderometer values (r = -.50).

Huffman[64] used quadrant analysis to determine the efficiency of USDA Quality grade, marbling, and the Armour Tenderometer for placing carcasses into uniform tenderness groups[64]. Based on taste-panel scores as the standard, the Armour Tenderometer correctly classified 78 percent of the samples, marbling score correctly classified 64 percent, and Quality grade 59 percent. Using the Warner-Bratzler Shear values as the standard, the Tenderometer, marbling score, and Quality grade correctly classified 76 percent, 60 percent, and 55 percent, respectively.

MEASUREMENT OF TENSILE PROPERTIES

Tensile properties of muscle also have been measured for many years. In 1907, Lehmann (cited in [3]) devised an instrument for the determination of the breaking strength of meat tissues.

Wang *et al.*[65] at the American Meat Institute Foundation studied individual muscle fiber extensibility as it relates to tenderness. Muscle was macerated with a modified Waring Blender and extensibility was measured by gripping individual fibers with watchmaker's steel forceps, 5 mm between the points. The fiber was stretched until it was broken and extensibility was expressed in mm stretch to the breaking point. Extensibility was related to sensory tenderness (r = -.43 to -.80) and Warner-Bratzler shear (r = .36 to .45). Other workers have reported low but significant correlations of fiber extensibility and tenderness (cited in [3]). The method is limited as it deals only with fibers themselves.

Tensile properties of muscle strips have been measured by Bouton and Harris[66,67] and Stanley *et al.*[68,69] using the Instron equipped with a tension cell. Bouton and Harris[66,67] reported the force required to pull or break cooked fiber bundles (1.5 cm^2 x .67 cm long) placed transversely to the applied strain in pneumatically operated jaws. This was termed adhesion and gave a measure of the effect of connective tissue on texture. Adhesion was correlated with Instron compression or penetration force (r = .9) and Warner-Bratzler shear (r = .45)[67]. Tensile strength of cooked meat also can be measured when the fibers lie parallel to the direction of the applied strain (r = .85 with shear)[67,70].

Stanley *et al.*[69] reported evaluating tensile properties of uncooked beef muscle in relation to sensory properties. Raw samples of parallel fibers 5 cm long and 3.5 cm across (10 mm^2) were used and fibers were oriented parallel to the direction of the applied force. These tests conducted included 1) breaking strength, 2) break elongation, 3) elasticity at 15 percent stretch for 40 cycles, and 4) relaxation or loss of stress at

15 percent extension. Not all properties measured were highly related to each other (r = -.17 to +.99). Muscle differences were seen. Muscle contraction increased the work of rupture, breaking strength, and stress relaxation, but decreased elasticity. In a previous study on pork, Stanley *et al.*[68] reported breaking strength accounted for 59 to 90 percent of the variation in tenderness. It would be interesting to know whether the relationship would hold for beef.

COMPRESSION

Tenderness Press

This device was developed at the USDA. It is a mechanical method based on measuring the pressure required to force a standard-sized piece of meat through a small hole in the bottom of a cylinder[3,71]. Actually, this instrument measures both compression and shearing forces. Sperring *et al.*[71] reported the instrument showed promise for measurement on raw meat (r = .899 with tenderness at 3 days). However, Bratzler and Smith[72] found no relationship between sensory tenderness and tenderness press measurements.

CONCLUSION

All methods of measuring texture by instrumental means must continue to be based on sensory tests. It is apparent, too, that not all studies on a particular instrumental method have shown that method to be useful. Some workers have reported a high relationship of an instrumental method with sensory tenderness or another instrument, whereas others have reported little or no relationship between methods. All too often, insufficient range and heterogeneity in texture have existed in a particular study. This lack of heterogeneity results in no correlation between methods and a conclusion that the method is not useful. This is unfortunate.

From this review it appears that a number of principles can and are being used in measuring beef texture. These principles include use of shear, bite, penetration, tensile properties, and compression. The adoption of a particular texture method should be dictated by the need, usefulness, and confidence of the user in the instrument used.

REFERENCES

1. J. Hammond, *Growth and Development of Mutton Qualities in the Sheep* (Oliver and Boyd, London, 1932).

2. A. Kramer, *Texture Measurements of Foods*, A. Kramer and A. S. Szczesniak, Eds., 1 (D. Reidell, Boston, 1973).

3. A. S. Szczesniak and K. W. Torgeson, *Advan. in Food Res.*, *14*, 33 (1965).

4. E. Laakkonen, *Advances in Food Res.*, *20*, 257 (1973).

5. R. P. Newbold and P. V. Harris, *J. Food Sci.*, *37*, 337 (1972).

6. A. J. Bailey, *J. Sci. Food Agric.*, *23*, 995 (1972).

7. H. K. Herring, R. G. Cassens, and E. J. Briskey, *J. Food Sci.*, *30*, 1049 (1965).

8. R. L. Hostetler, B. A. Link, W. A. Landmann, and H. A. Fitzbugh, Jr., *J. Food Sci.*, *37*, 132, (1972).

9. P. E. Bouton, P. V. Harris, W. R. Shorthose, and R. J. Baxter, *J. Food Sci.*, *38*, 932 (1973).

10. W. G. Kruggel and R. A. Field, *J. Food Sci.*, *36*, 1114 (1970).

11. N. E. Pfeiffer, R. A. Field, T. R. Varnell, W. G. Kruggel, and I. I. Kaiser, *J. Food Sci.*, *37*, 897 (1971).

12. F. Hill, *J. Food Sci.*, *31*, 161, (1966).

13. H. K. Herring, R. G. Cassens, and E. J. Briskey, *J. Food Sci.*, *32*, 534 (1967).

14. R. A. Field, A. M. Pearson, and B. S. Schweigert, *J. Agric. Food Chem.*, *18*, 280 (1970).

15. S. Cover, S. J. Ritchey, and R. L. Hostetler, *J. Food Sci.*, *27*, 469 (1962).

16. J. M. Ramsbottom and E. J. Strandine, *Food Res.*, *13*, 315 (1948).

17. J. M. Ramsbottom and E. J. Strandine, *J. Animal Sci.*, *8*, 398 (1949).

18. R. H. Locker, *Food Res.*, *25*, 304 (1960).

19. R. H. Locker and C. J. Hagyard, *J. Sci. Food Agric.*, *14*, 787 (1963).

20. B. B. Marsh and N. G. Leet, *J. Food Sci.*, *31*, 450 (1966).

21. D. B. Watt and H. K. Herring, *J. Animal Sci.*, *38*, 928 (1974).

22. D. E. Goll, N. Arakawa, M. H. Stromer, W. A. Busch, and R. M. Robson, *Physiology and Biochemistry of Muscle as a Food*, *2*, E. J. Briskey, R. G. Cassens, and B. B. Marsh, Eds., 755 (The University of Wisconsin Press, 1970).

23. D. E. Goll, M. H. Stromer, R. M. Robson, J. Temple, B. A. Eason, and W. A. Busch, *J. Animal Sci.*, *33*, 963 (1971).

24. W. A. Busch, M. H. Stromer, D. E. Goll, and A. Suzuki, *J. Cell Biol.*, *52*, 367 (1972).

25. M. Fujimaki, N. Arakawa, A. Okitani, and O. Takagi, *Agric. Biol. Chem.*, *29*, 700 (1965).

26. H. K. Herring, Ph.D. Thesis (University of Wisconsin Library, Madison, Wisconsin, 1968).

27. C. L. Davey and K. V. Gilbert, *J. Food Technol.*, *2*, 57 (1967).

28. K. Takahashi, T. Fukazawa, and T. Yasui, *J. Food Sci.*, *32*, 409 (1967).

29. M. H. Stromer, D. E. Goll, and L. E. Roth, *J. Cell Biol.*, *34*, 431 (1967).

30. K. Strandberg, F. C. Parrish, D. E. Goll, and S. A. Josephson, *J. Food Sci.*, *38*, 69 (1973).

31. C. L. Davey and M. R. Dickson, *J. Food Sci.*, *35*, 56 (1970).

32. C. L. Davey and K. V. Gilbert, *J. Food Sci.*, *34*, 69 (1969).

33. T. Fukazawa, E. J. Briskey, F. Takahashi, and T. Yasui, *J. Food Sci.*, *34*, 606 (1969).

34. H. M. Chaudhry, F. C. Parrish, and D. E. Goll, *J. Food Sci.*, *34*, 183 (1969).

35. D. E. Goll, N. H. Stromer, D. G. Olson, W. R. Dayton, A. Suzuki, and R. M. Robson, Proceedings, Meat Industry Research Conference, AMI, 1974.

36. E. Laakkonen, Ph.D. Thesis (Cornell University, Ithaca, New York, 1969).

37. A. S. Szczesniak, *Texture Measurements of Foods*, A. Kramer and A. S. Szczesniak, Eds., 71 (D. Reidel Publ. Co. Boston, 1973).

38. N. N. Mohsenin, *J. Texture Studies*, *1*, 133 (1970).

39. A. M. Pearson, Proceedings, Meat Tenderness Symposium, Campbell Soup Company, Camden, New Jersey, 1963, 135.

40. A. S. Szczesniak, *J. Food Sci.*, *28*, 410 (1963).

41. E. E. Finney, *J. Texture Studies*, *1*, 19 (1969).

42. P. W. Voisey, *J. Texture Studies*, *2*, 129 (1971).

43. K. F. Warner, *Proceedings, Am. Soc. Animal Production*, *21*, 114 (1928).

44. M. F. Pool and A. A. Klose, *J. Food Sci.*, *34*, 524 (1969).

45. C. L. Davey and K. V. Gilbert, *J. Food Technol.*, *4*, 7 (1969).

46. P. W. Voisey and H. Hansen, *Food Technol.*, *21*, 37A (1967).

47. P. E. Bouton, P. V. Harris, and W. R. Shorthose, *J. Food Sci.*, *36*, 435 (1971).

48. A. Kramer, G. J. Buckhardt, and H. P. Rogers, *Canner*, *112* (2), 34 (1951).

49. N. N. Voledkevich, *Food Res.*, *3*, 221 (1938).

50. A. J. H. Sale, *Texture in Foods*, 103 (S.C.I. Monograph No. 7, London, 1960).

51. P. G. Mac Farlane and J. M. Marer, *Food Technol.*, *20*, 838 (1966).

52. B. B. Marsh, P. R. Woodhams, and N. G. Leet, *J. Food Sci.*, *31*, 262 (1966).

53. D. N. Rhodes, R. C. D. Jones, B. B. Crystall, and J. M. Harries, *J. Texture Studies*, *3*, 298 (1972).

54. H. H. Friedman, J. E. Whitney, and A. S. Szczesniak, *J. Food Sci.*, *28*, 390 (1963).

55. J. G. Kapsalis, B. K. Drake, and B. Johansson, *J. Texture Studies*, *1*, 285 (1970).

56. R. H. Alsmeyer, J. W. Thornton, and R. L. Hiner, *J. Animal Sci.*, *27*, 526 (1968).

57. L. C. Hinnergardt and J. M. Tuomy, *J. Food Sci.*, *35*, 312 (1970).

58. L. C. Hinnergardt and J. M. Tuomy, U.S. Patent 3,732,727 (1973).

59. D. G. Galloway, J. M. Tuomy, and L. C. Hinnergardt, *J. Agric. Food Chem.*, *21*, 880 (1973).

60. L. J. Hansen, *J. Texture Studies*, *3*, 146 (1972).

61. R. R. Henrickson, J. L. Marsden, and R. D. Morrison, *J. Animal Sci.*, *37*, 857 (1972).

62. Z. L. Carpenter, G. Smith, and O. D. Butler, *J. Food Sci.*, *37*, 126 (1972).

63. F. C. Parrish, D. G. Olson, B. E. Miner, R. B. Young, and R. L. Snell, *J. Food Sci.*, *38*, 1214 (1973).

64. D. L. Huffman, *J. Animal Sci.*, *38*, 287 (1974).

65. H. Wang, D. M. Doty, F. J. Beard, J. C. Pierce, and O. G. Hankins, *J. Animal Sci.*, *15*, 97 (1956).

66. P. E. Bouton and P. V. Harris, *J. Food Sci.*, *37*, 140 (1972).

67. P. E. Bouton and P. V. Harris, *J. Food Sci.*, *37*, 218 (1972).

68. D. W. Stanley, G. P. Pearson, and V. E. Coxworth, *J. Food Sci.*, *36*, 256 (1971).

69. D. W. Stanley, L. M. McKnight, W. G. S. Hines, W. R. Usborne, and J. M. Deman, *J. Texture Studies*, *3*, 51 (1972).

70. P. E. Bouton, F. D. Carroll, A. L. Fisher, P. V. Harris, and W. R. Shorthose, *J. Food Sci.*, *38*, 816 (1973).

71. D. D. Sperring, W. T. Platt, and R. L. Hiner, *Food Technol.*, *13*, 155 (1959).

72. L. J. Bratzler and H. D. Smith, *J. Food Sci.*, *28*, 99 (1963).

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

EVALUATION OF COLOR AND OTHER PROPERTIES OF FROZEN BEEF

Donald H. Kropf, H. J. Tuma, C. C. Allen, M. C. Hunt, M. L. Sandberg, and D. E. Schafer

INTRODUCTION

Our interest in meat-color evaluation began with acquisition of a Bausch and Lomb 600 spectrophotometer with reflectance attachment. Our first study involved spectral changes in fresh beef muscle under display conditions from immediately post cutting to the extreme discoloration shown after display for 240 hr.

This interest quickly turned to frozen meat, which in time involved study of all variables listed in Table 1. Evaluation of color redness or brightness of degree of discoloration becomes essential. This involved both subjective and objective color evaluation.

This paper covers reflectance of beef longissimus muscle in reduced myoglobin (redMb), oxymyoglobin (oxyMb), and metmyoglobin (metMb) forms, followed by development of beef-color visual standards, which have been widely used in the industry.

The next study reported involves correlation of visual color brightness and visual bleach scores to reflectance data at various times in the processing-display sequence. The final study involves additional correlations between objective and subjective measures of color in a study of uniform versus non-uniform color between muscles in beef T-bone steaks.

REFLECTANCE CHANGES IN FRESH BEEF LONGISSIMUS POST CUTTING

Experimental Procedure

Longissimus muscle from the left wholesale rib cut of 60 steer-beef carcasses (241 to 438 kg) was studied. Carcasses of unknown history were selected in commercial packing plants at 24 hr post mortem to represent both small and moderate marbling levels within each of 3 USDA physiological age groups; A- A, A+ B-, and B B+ (referred to as young, intermediate, and approaching maximum maturity, respectively). Degree of maturity was

TABLE 1 Factors studied in frozen-meat research involving beef and lamb color.

Quality level (grade)
Aging time
Storage time
Bull versus steer
Saw versus knife cutting
Bloom time
Freezing-packaging sequence
Freezing cycle (time, temp.)
Freezing system
Film O_2 permeability
Display time
Display temperature
Lighting intensity
Type of lighting
Display case management
Pure light work
Selection for muscle-color uniformity

determined by the amount of bone ossification and color of lean as defined by the Federal Grading Service (USDA, 1965). Color of lean was used only as a "sample elimination factor" if it did not conform to the color typical for a maturity group. "Dark cutting" carcasses were not included in this study. Ten carcasses were selected per experimental cell.

The wholesale ribs were transported to Kansas State University meat laboratory and cut at 10 days postmortem. A rib steak approximately 5 cm thick was removed over the 8th thoracic vertebra. The longissimus muscle was cut into two 2.5 cm thick portions immediately before the first objective color reading. Elapsed time between cutting the longissimus and beginning the first objective color reading never exceeded 30 sec.

The freshly cut (anterior) surface of the posterior half of longissimus muscle sample was used for subjective and objective color measurements. This steak was wrapped in oxygen-permeable fresh-meat cellophane with an opaque fiber board backing and sealed. The packaging film was approximately 0.028 mm thick,

including about 0.006 mm low-density polyethylene on a base sheet of cellophane. Fresh steaks were stored in approximately 860 lumens/m^2 of incandescent light at about 6°C.

Both subjective and objective color measures were made at 26 time intervals; namely, immediately after cutting; also, 5, 10, 15, and 20 min; and 1, 2, 3, 10, 24, 48, 72, 96, 128, 136, 149, 154, 158, 163, 173, 178, 182, 187, 192, 216, and 240 hr post cutting.

Subjective color values were estimated under 484.2 lumens/m^2 of cool white fluorescent light and on a white cotton background, using an 11-point scale (Figure 1).

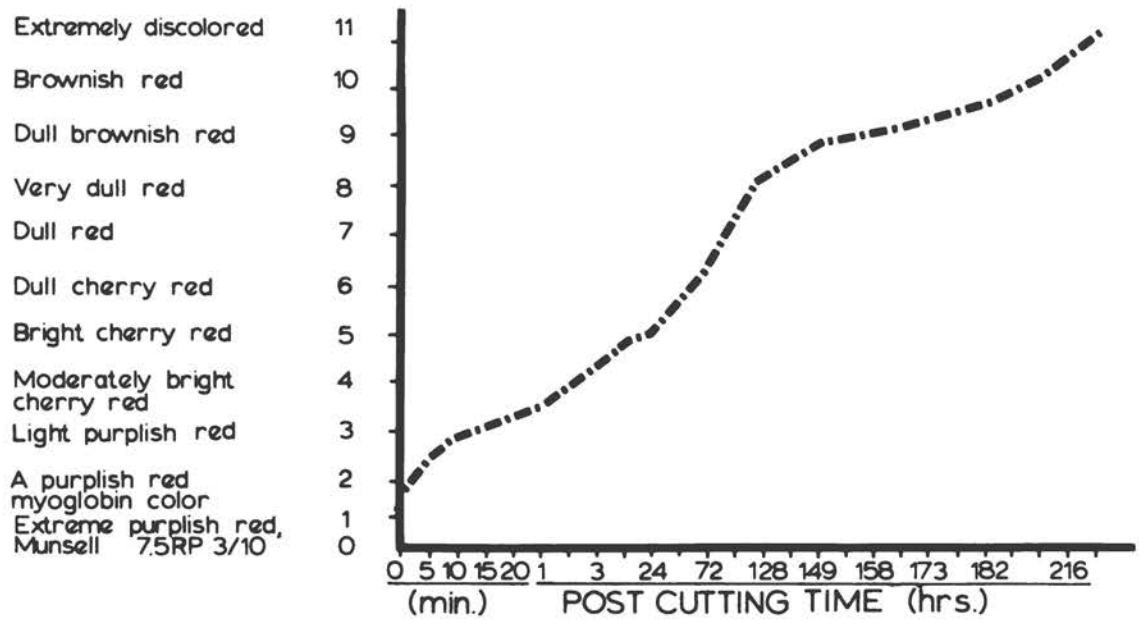


FIGURE 1 Fresh-beef longissimus visual color at various times post cutting.

Color reflectance was recorded in the visible light range of 400 to 700 nm on a Bausch and Lomb 600 reflectance spectrophotometer at a scan speed of 250 nm/min using a $MgCO_3$ block wrapped in fresh-meat cellophane for 100 percent reflectance. Care was taken to scan the same portion of sample surfaces each time and to exclude large fat or connective tissue areas from the sampled area. Duplicate reflectance scans were not recorded as color changes during the early time intervals were too rapid.

Analysis of variance and least significant difference (LSD) procedures were used to detect differences between time period means for percent reflectance at each of 8 light wavelengths

(474, 525, 538, 571, 600, 610, 620, and 630 nm), for 2 reflectance ratios (R474/R525 nm, R571/R525 nm), and for subjective visual values.

Results and Discussion

Steaks changed visual color as expected. A purplish red color was evident immediately post cutting with an overall visual mean (60 samples) of 1.75 (Figure 1). Maximum bright red color developed somewhere between 3 and 24 hr post cutting with a visual mean of 4.3 at 3 hr and 5.0 at 24 hr. Brown metMb color appeared between 96 and 163 hr under these conditions with a pattern similar to that mentioned by Pirko and Ayres[1].

Figures 2 through 6 (also Table 2) illustrate the mean percent reflectance for each reading time for all steaks at the 8 wavelengths and 2 wavelength ratios studied. Points are not spaced at distances comparable to times of color determination.

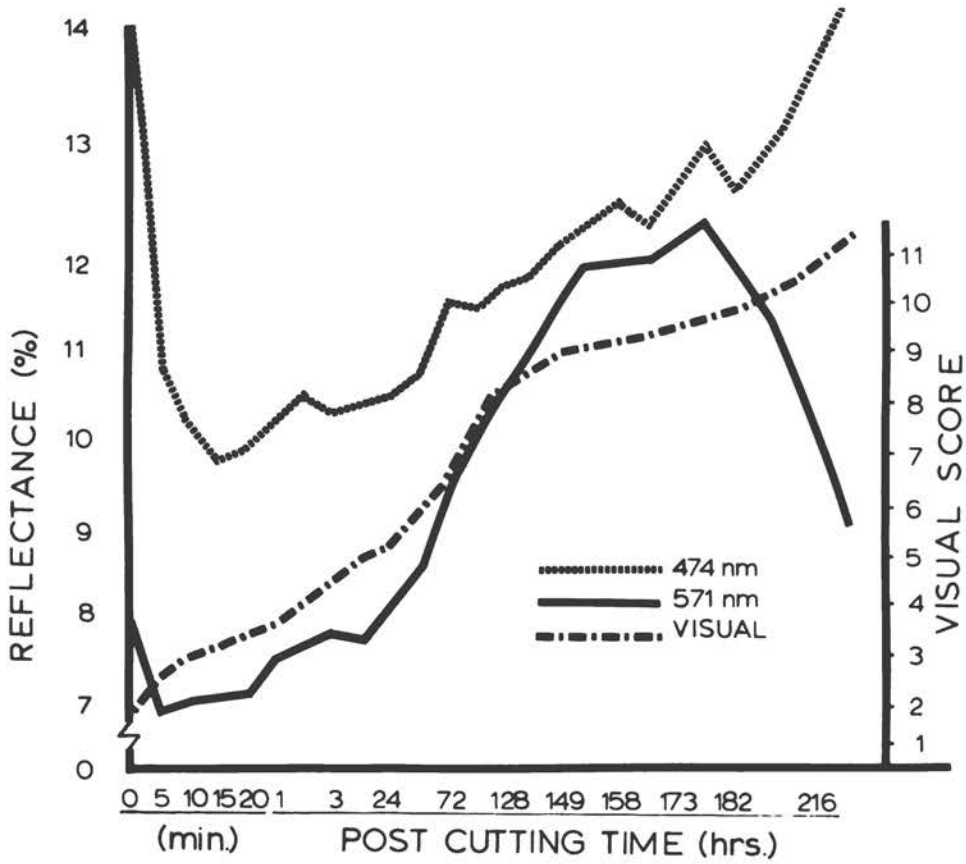


FIGURE 2 Fresh-beef longissimus reflectance (474 and 571 nm) at various times post cutting.

TABLE 2 Fresh-beef longissimus visual color and reflectance at various times post cutting.

Time	Visual score	474/525	571/525	474	525	538	571	600	610	620	630
0 min	1.75^{a}	1.332^{m}	0.727^{abc}	14.26^{k}	10.77^{cdef}	8.06^{bc}	7.87^{bc}	15.65^{abc}	21.48^{cd}	26.09^{gh}	29.01^{ij}
5 min	2.51^{b}	1.153^{fg}	0.707^{a}	11.01^{abcd}	9.60^{a}	6.41^{a}	6.82^{a}	16.28^{bcdef}	22.58^{def}	27.21^{hi}	30.14^{jkl}
10 min	2.95^{c}	1.090^{de}	0.714^{ab}	10.53^{a}	9.69^{ab}	6.38^{a}	6.92^{ab}	17.09^{efgh}	23.51^{efg}	28.08^{ij}	31.07^{klm}
15 min	3.14^{cd}	1.064^{cd}	0.716^{ab}	10.28^{a}	9.67^{ab}	6.36^{a}	6.94^{ab}	17.57^{fghi}	24.06^{efgh}	28.49^{ij}	31.57^{klm}
20 min	3.30^{d}	1.060^{bcd}	0.719^{ab}	10.32^{a}	9.77^{ab}	6.37^{a}	7.02^{ab}	17.77^{ghi}	24.27^{fghi}	28.65^{ij}	31.80^{lm}
1 hr	3.60^{e}	1.052^{abc}	0.727^{abc}	10.62^{a}	10.11^{abc}	6.75^{ab}	7.37^{abc}	18.92^{ij}	25.32^{hij}	29.00^{ij}	32.42^{mno}
2 hr	3.95^{f}	1.044^{abc}	0.735^{abc}	10.80^{abc}	10.34^{abcdef}	6.94^{ab}	7.61^{abc}	19.64^{jk}	25.88^{ij}	29.11^{j}	32.58^{mno}
3 hr	4.34^{g}	1.029^{ab}	0.744^{abc}	10.69^{a}	10.40^{bcdef}	7.04^{ab}	7.78^{abc}	20.39^{kl}	26.52^{jk}	29.43^{jk}	32.67^{mno}
10 hr	4.82^{h}	1.049^{abc}	0.745^{abc}	10.75^{ab}	10.25^{abcd}	6.86^{ab}	7.67^{abc}	22.55^{nop}	29.13^{lm}	31.66^{l}	34.38^{o}
24 hr	5.06^{h}	1.023^{a}	0.764^{bc}	10.84^{abc}	10.60^{cdef}	7.04^{ab}	8.11^{c}	23.31^{op}	29.83^{m}	31.96^{l}	33.88^{no}
48 hr	5.72^{i}	1.024^{a}	0.779^{c}	11.02^{abcd}	10.77^{cdef}	7.26^{b}	8.42^{cd}	23.46^{p}	29.49^{lm}	31.13^{kl}	32.28^{mn}
72 hr	6.24^{j}	1.042^{abc}	0.837^{d}	11.65^{cdef}	11.18^{f}	8.09^{bc}	9.43^{e}	22.86^{nop}	27.98^{kl}	29.19^{j}	29.70^{jk}
96 hr	7.40^{k}	1.053^{abc}	0.908^{e}	11.59^{bcde}	11.01^{def}	8.40^{bcd}	10.06^{efg}	21.98^{mno}	26.34^{jk}	27.13^{hi}	27.20^{hi}
128 hr	8.19^{l}	1.069^{cd}	0.946^{ef}	11.81^{defg}	11.06^{ef}	8.79^{cde}	10.58^{fgh}	21.56^{lmn}	25.23^{ghij}	25.78^{g}	25.64^{gh}
136 hr	8.51^{m}	1.086^{de}	0.997^{fg}	11.87^{defg}	10.93^{def}	9.04^{de}	10.94^{ghij}	20.59^{klm}	23.68^{efgh}	24.07^{fg}	23.83^{fg}
149 hr	8.80^{n}	1.111^{e}	1.050^{gh}	12.19^{efgh}	10.98^{def}	9.55^{efg}	11.58^{ijkl}	19.91^{jk}	22.43^{de}	22.75^{ef}	22.38^{ef}
154 hr	8.91^{no}	1.131^{ef}	1.080^{hij}	12.38^{efgh}	10.98^{def}	9.84^{fg}	11.90^{jkl}	19.34^{jk}	21.63^{cd}	21.78^{de}	21.35^{de}
158 hr	9.07^{o}	1.147^{f}	1.101^{hij}	12.51^{fghi}	10.94^{def}	10.02^{fgh}	11.97^{kl}	18.51^{hij}	20.56^{bc}	20.73^{cd}	20.28^{cd}
163 hr	9.17^{op}	1.159^{fgh}	1.129^{ij}	12.37^{efgh}	10.71^{cdef}	10.02^{fgh}	12.03^{kl}	17.74^{ghi}	19.55^{ab}	19.62^{abc}	19.17^{abc}
173 hr	9.36^{pq}	1.178^{ghi}	1.134^{j}	12.63^{ghi}	10.78^{cdef}	10.24^{gh}	12.17^{kl}	17.50^{fghi}	19.22^{ab}	19.36^{abc}	18.94^{abc}
178 hr	9.46^{q}	1.185^{hi}	1.133^{j}	13.02^{hij}	11.05^{ef}	10.66^{h}	12.50^{l}	17.41^{efgh}	19.13^{ab}	19.33^{abc}	18.94^{abc}
182 hr	9.58^{qr}	1.201^{ij}	1.117^{ij}	12.66^{ghi}	10.58^{cdef}	10.19^{fgh}	11.82^{jkl}	16.50^{cdefg}	18.13^{a}	18.42^{a}	18.14^{abc}
187 hr	9.84^{r}	1.219^{jk}	1.077^{hi}	12.93^{hi}	10.68^{cdef}	10.18^{fgh}	11.51^{hijk}	16.06^{bcde}	17.93^{a}	18.42^{a}	18.27^{ab}
192 hr	10.18^{s}	1.246^{k}	1.107^{g}	13.29^{ij}	10.71^{cdef}	10.13^{fgh}	10.83^{fghi}	15.76^{abcd}	17.94^{a}	18.75^{ab}	18.87^{abc}
216 hr	10.63^{t}	1.295^{l}	0.913^{e}	13.87^{jk}	10.78^{cdef}	9.82^{fg}	9.94^{ef}	15.06^{ab}	17.97^{a}	19.57^{abc}	20.21^{bcd}
240 hr	10.88^{t}	1.343^{m}	0.847^{d}	14.28^{k}	10.70^{cdef}	9.45^{ef}	9.10^{de}	14.56^{a}	18.10^{a}	20.40^{bcd}	21.60^{de}
LSD	0.268	0.031	0.056	0.886	0.790	0.768	0.979	1.439	1.754	1.882	1.989

[a]Means within columns with same superscript letter are not different (P<.05).

Mean reflectance at 474 nm (Figure 2) showed a marked and significant ($P < .05$) decrease from 0 time to 5 min post cutting but with no further significant difference through 48 hr of display. A gradual increase in reflectance at 474 nm occurs from 96 through 240 hr. Both patterns seem to conflict with Snyder's[2] data, reporting very little reflectance change at 474 nm during 8 days of storage. Steaks with both redMb predominating

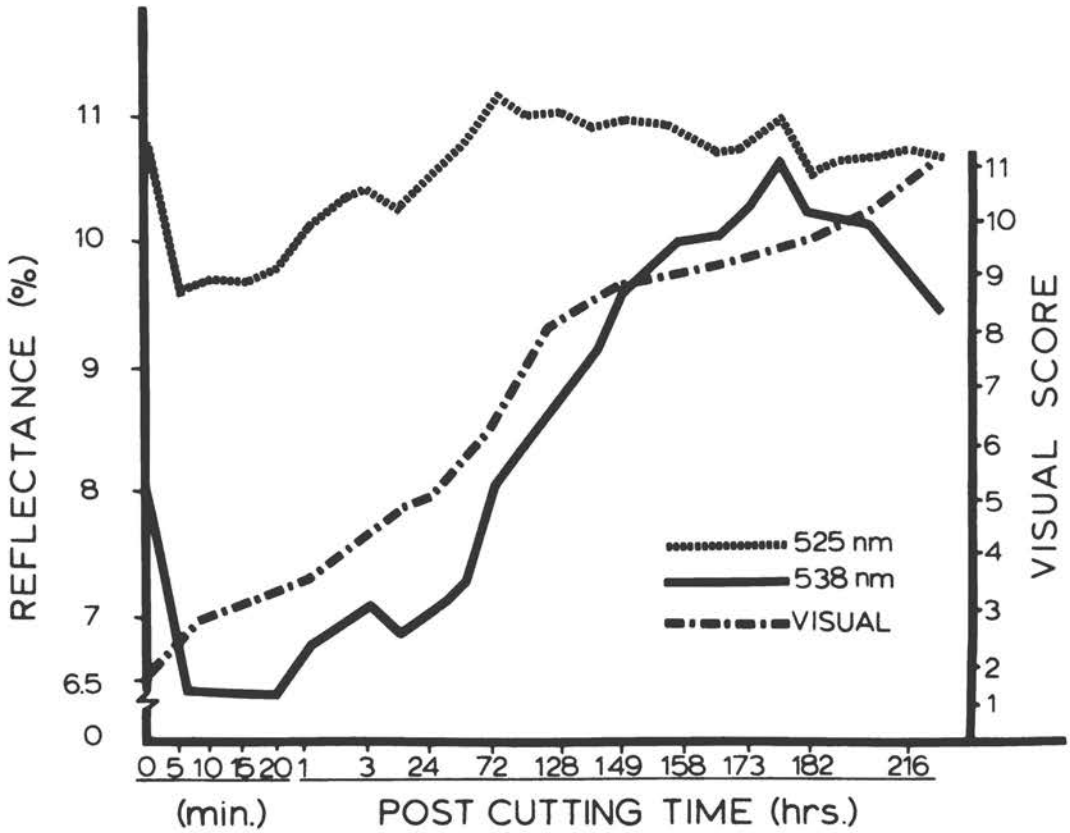


FIGURE 3 Fresh-beef longissimus reflectance (525 and 538 nm) at various times post cutting.

(0 time) or metMb (later time periods) possess greater light reflectance at 474 nm than those with primarily oxyMb (3 to 24 hr post cutting). Minimal mean reflectance of 10.28 percent occurred at 15 min post steak cutting and the reflectance values at 5, 10, and 20 min, and at 1, 2, 3, 10, 24, and 48 hr were not statistically different from this minimum.

Reflectance percentages at 525 nm (Figure 3) exhibited a significant ($P < .05$) decrease from 0 time to 5 min but the change is less than at 474 nm. The smallest range in mean percent

reflectance of all wavelengths studied was noted at 525 nm (1.59 percent total range). Between 24 and 240 hr, means are not significantly different. These data tend to agree with the isobestic nature of this wavelength for the 3 myoglobin (Mb) forms reported by Snyder[2], and Steward *et al.*[3].

The mean reflectance percent pattern at 538 nm (Figure 3) also presented a significant ($P < .05$) decrease from 0 time to 5 min post cutting, then did not increase significantly

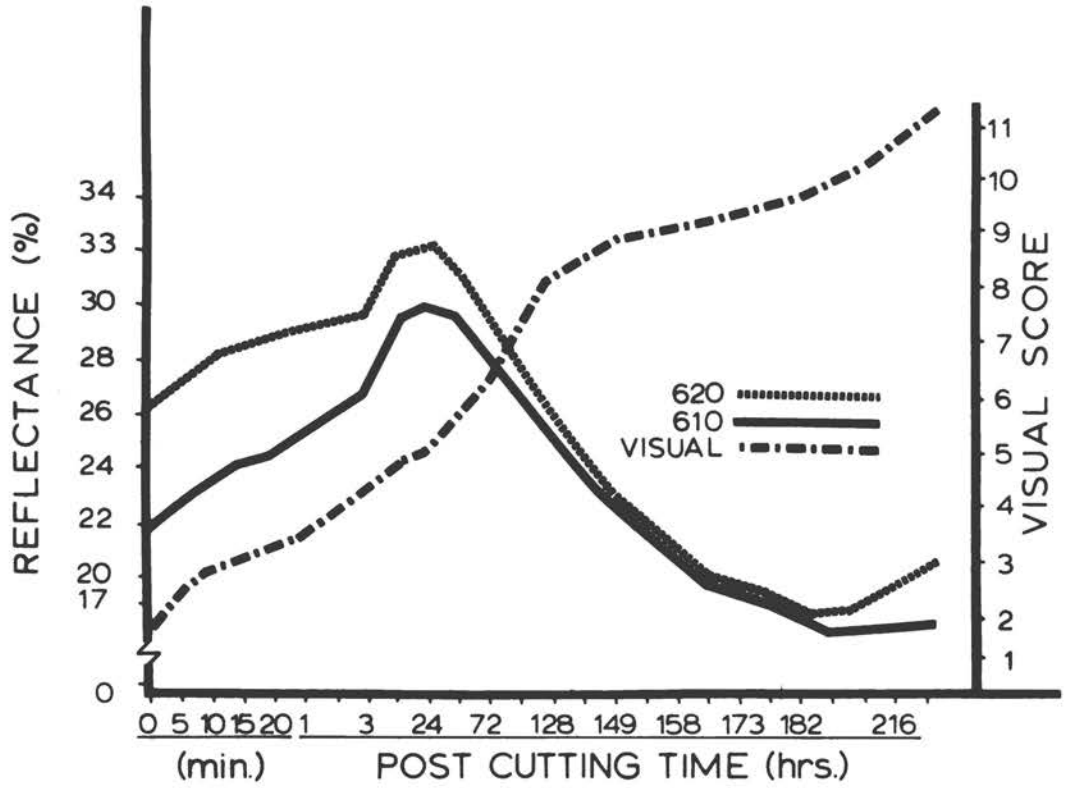


FIGURE 4 Fresh-beef longissimus reflectance (620 and 610 nm) at various times post cutting.

through 24 hr of display. Between 48- and 72-hr periods, a significant ($P < .05$) increase in reflectance occurs. A gradual increase in 538 nm reflectance percent was observed between 10 and 192 hr after exposure to oxygen. Maximum reflectance percent at 538 nm occurred at 178 hr, but percentages between 158 and 192 hr are not significantly different from that value.

The mean reflectance percent pattern at 571 nm is shown in Figure 2. Again a signficiant ($P < .05$) decrease from 0 time to 5 min is noted. From 48 hr to 72 hr a significant ($P < .05$) increase from 8.4 to 9.4 was noted. This latter increase

occurred when mean visual score changed from maximum bloom to initiation of flat brown visual score. Otherwise, reflectance percentages at this wavelength appear to be insensitive in relation to visual color changes. The data presented for 571 nm seem to contradict Snyder's[2] observations at this wavelength made on fresh beef stored for 8 days.

Reflectance percentages at the longer wavelengths (600, 610, 620, and 630 nm) did not show significant decreases between 0 and 5 min as did other shorter wavelengths, although a trend

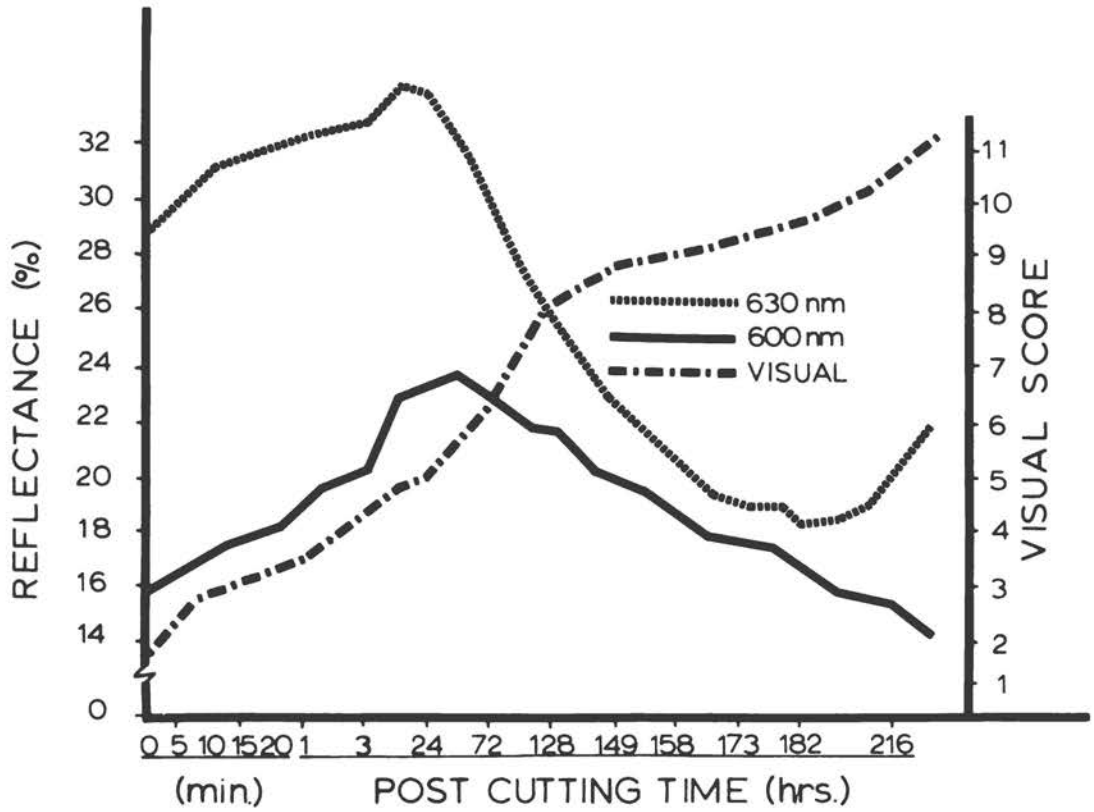


FIGURE 5 Fresh-beef longissimus reflectance (630 and 600 nm) at various times post cutting.

toward increased reflectance was shown. At 600 nm (Figure 5) the greatest mean reflectance occurred at 48 hr just after visual values indicated maximum appearance of bloom. This reflectance value of 23.46 percent was not significantly different from that at 10, 24, or 72 hr. At 610 and 620 nm, shown in Figure 4, maximum mean reflectances of 29.83 percent and 31.96 percent respectively occurred at 24 hr; the same period a mean visual value of 5.0 (bright red) occurred. Maximum reflectance at 630 nm was 34.38 percent occurring at 10 hr post cutting; however, reflectance percentages at 1, 2, 3, and 24 hr were not

statistically different. Muscle in a predominatly metMb state had lower light reflectance at these wavelengths than those with redMb or oxyMb predominating.

Reflectance ratio at R474/R525 nm (Figure 6) displayed significant ($P < .05$) decreases in value between 0 time and 10 min. A gradual increase in value between 24 and 240 hr time periods corresponded to visual color deterioration during

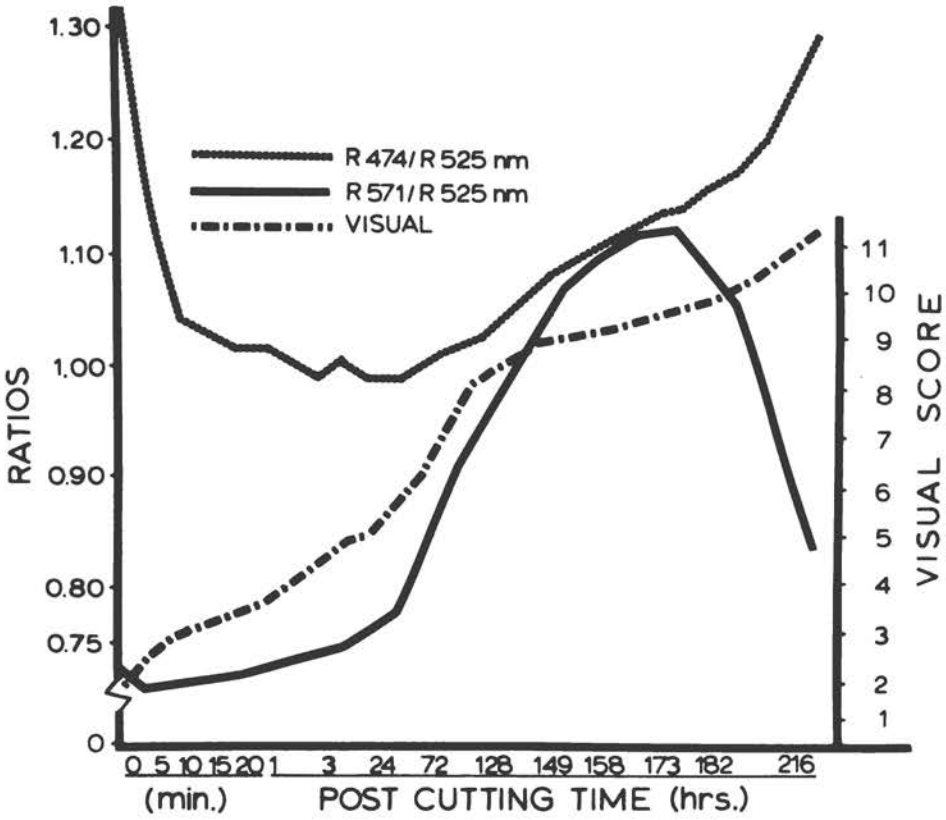


FIGURE 6 Fresh-beef longissimus reflectance ratios (R474/R525 nm and R571/R525 nm) at various times post cutting.

the same period. Ratio R474/R525 nm appeared to be more useful in following fresh-beef color brightening as compared to ratio of R571/R525 nm also shown in Figure 6.

Dean and Ball[4], using wavelengths of 473, 507, 573, and 597 nm, alluded to some conversion of redMb to oxyMb immediately after a fresh-meat surface is exposed. Data at 474, 525, 538, 571 nm and ratio R474/R525 nm of this research conclusively demonstrated a definite reflectance change occurring at the sample surface between immediately post cutting and 5 min post cutting.

The post-cutting time pattern for percent reflectance at 474, 600, 610, and ratio R474/R525 nm showed possibly 2 slopes indicating a similarity between steaks in predominantly redMb or metMb form. Therefore, perhaps these objective measurements did not distinguish predominantly between these states of beef longissimus, although some appear to distinguish between oxyMb and the other 2 forms.

UNFROZEN BEEF REFLECTANCE

Table 3 summarizes various wavelengths and methods of calculation that have been used for measuring color or Mb pigment state in unfrozen beef muscle. Numerous parameters appear to distinguish between redMb, oxyMb, and metMb and some were highly correlated to visual scores that seemed to involve lightness and darkness of muscle and theoretically could have been affected by both pigment level and chemical state.

TABLE 3 Reflectance for measuring color or chemical state of Mb (unfrozen beef).

Authors	Methods and findings
Pirko and Ayres[1]	555, 580, 635 nm minus reflectance of meat with pigment extracted.
Dean and Ball[4]	$\frac{K/S \text{ at } 507 \text{ nm}}{K/S \text{ at } 573 \text{ nm}}$ metMb
	$\frac{K/S \text{ at } 473 \text{ nm}}{K/S \text{ at } 597 \text{ nm}}$ for redMb
	OxyMb calculated by difference
Snyder[2]	Measured R_d, a and b on Gardner automatic color difference meter. Pigments differed in a, b, and a/b.
Snyder[5]	Reflectance as R_A, adjusted to $R_A = 1.0$ at 525 nm, value at 474 nm distinguishes redMb, value at 571 nm distinguishes metMb.
	Varying proportions of oxyMb and metMb in sample holder gave S shaped curve.

TABLE 3 Reflectance for measuring color or chemical state of Mb (unfrozen beef) (continued).

Authors	Methods and findings
Franke and Solberg[6]	Varying proportions of metMb and oxyMb in sample holder. Used R_A at 632 nm. Constructed a linear response curve.
	Light beam may not have been uniform as authors had hoped.
Satterlee and Hansmeyer[7]	K/S at 580 nm used to calculate percent oxyMb.
	Calculated Mb auto-oxidation rate constants to determine effects of different lighting systems.
Ockerman and Cahill[8]	Used visual scale (7 = very light, 6 = light, 5 = moderately light, 4 = cherry red, 3 = moderately dark, 2 = dark, 1 = very dark). Percent R at 485, 505, 565 or 685 nm; Tristimulus value X, Y, Z or combined XYZ and Munsell value had correlations of .695 or higher to visual score.
Jeremiah *et al.*[9]	Used visual scale (1 = very pale pink, 2 = pale pink, 3 = pink, 4 = slightly pale red, 5 = cherry red, 6 = slightly dark red, 7 = moderately dark red, 8 = dark red, 9 = very dark red).
	Correlated numerous color measurements to visual score and total heme pigments, including parameters from Munsell, Gardner, Photovolt, Spectronic 20 reflectance at many wavelengths, Macbeth-Munsell systems and calculated tristimulus X, Y, Z values from these. Highly significant correlations for 31 or 35 parameters used.

TABLE 3 Reflectance for measuring color or chemical state of Mb (unfrozen beef) (continued).

Authors	Methods and findings
Zimmerman and Snyder[10]	Used $\frac{K/S\ 474\ nm}{K/S\ 525\ nm}$ and $\frac{K/S\ 571\ nm}{K/S\ 525\ nm}$ to calculate pigment form. Assumed linear relationships.
Ledward[11]	Adjusted R_A to 1.0 at 525 nm, then calculated $\frac{K/S\ 572\ nm}{K/S\ 525\ nm}$. Stated metMb could be calculated within 6 or 7 percent.
Setser *et al.*[12]	Calculated $\frac{K/S\ 474\ nm}{K/S\ 525\ nm}$ and $\frac{K/S\ 571\ nm}{K/S\ 525\ nm}$. Also reported percent uncorrected reflectance at 474, 525, 571, 600, 630, 650 and 685 nm. Did not calculate percent of various pigment forms.
Eagerman *et al.*[13]	Reported difference in percent reflectance 632 nm minus 614 nm. Greatest for redMb, less for oxyMb and least or minus values for metMb.
Van den Oord and Wesdorp (1971)	Determined relative proportions of oxyMb and metMb by R_A 630 nm minus R_A 580 nm. Established linear relationship.
Strange *et al.* (1974)	Consumer acceptability under 80-foot candles of Cool White had correlation of 0.91 to Gardner Color Difference "a" value, -.86 to percent reflectance at 630 nm minus that at 580 nm and -.86 to percent R 580 nm/percent R 630 nm. Used a rating scale 0 to 50 with 50 extremely acceptable and beef semitendinosus samples were rated each day for 5 to 8 days at 4°C.

Table 4 summarizes some work on relation of unfrozen beef-muscle reflectance to heme pigment concentration. Stewart *et al.*[3] corrected for reflectance characteristics of non-pigment part of muscle by using pigment-free muscle as a base line and reported pigment to be linearly related to K/S at 525 nm, an isobestic wavelength for redMb, oxyMb, and metMb. Jeremiah *et al.*[9] reported numerous correlations, but the highest relationship to pigment level was Z calculated from Photovole measurements (r = -.54).

TABLE 4 Reflectance for measuring pigment concentration.

Authors	Methods and findings
Stewart *et al.*[3]	K/S at 525 nm linearly related to pigment concentration. Used pigment free (peroxide treated) muscle at base line.
Jeremiah *et al.*[9]	Correlated 35 color parameters to pigment level, with following correlations of P < 1 percent; Gardner R_d -.38, Y and Z calculated from Gardner -.38 and -.46, Photovolt blue -.53, Photovolt green -.41, Y and Z calculated from Photovolt -.41 and -.54; Spectronic 20 reflectance at 415 nm -.40, at 445 nm -.41, at 535 nm -.39, at 565 nm -.41, at 595 nm -.40, and Spectronic 20 calculated Y -.38; Macbeth-Munsell Yellow -.39, White -.50, Macbeth-Munsell calculated XYZ (r = -.32, -.37 and -.41).

COMPARISON OF REFLECTANCE OF redMb, oxyMb, AND metMb IN FRESH AND FROZEN BOVINE LONGISSIMUS

Procedure

Paired beef wholesale cut loins were taken from a heifer carcass of B maturity and Standard plus grade to assure a low intra-muscular fat content and a relatively high total heme pigment concentration of muscle. Twenty longissimus muscle steaks, representing either medial or lateral half, were knife cut 2.5 cm thick and randomly assigned to 1 of 3 treatments:

1. Packaged in oxygen-impermeable Saran for at least 4 hr to obtain redMb.
2. Allow muscle to bloom at least 30 min to obtain oxyMb and package in oxygen-permeable Iolon film (3 mil).
3. Treat muscle surface with 1 percent acqueous solution of $FeK_3(CN)_6$ intermittently every 30 min for 3 hr. Blot excess solution off muscle surface prior to packaging in Iolon film (3 mil).

Muscle samples were scanned for total reflectance by a Bausch and Lomb 600 reflectance spectrophotometer from 400 to 700 nm at a scan speed of 250 nm/min using a $MgCO_3$ block with appropriate film as 100 percent reflectance standard. Percent reflectance to the nearest 0.1 percent was taken at 17 different wavelengths in the range of 400 to 685 nm.

Scanning was done as soon as the desired pigment form was achieved (fresh, unfrozen) and immediately after freezing. Freezing of packaged muscles was for about 18 hr in a blast freezer at -26°C. Frozen steaks were stored at -29.4°C in the dark until they were scanned.

Results and Discussion

Reflectance spectra of fresh and frozen bovine longissimus in various pigment forms are given in Figures 7 through 10. Mean percent reflectance and standard error of the mean for each wavelength are given in Table 5.

Figure 7 and Table 5 show the spectra of fresh and frozen muscle in the redMb state. They are not different in percent reflectance at 400, 450 or 474 nm, but the frozen samples have greater reflectance values at the longer wavelengths and larger difference in reflectance at 632 minus than at 614 nm.

Spectra of fresh and frozen bovine longissimus in the oxyMb form are presented in Figure 8. Again, little difference in reflectance is noted at the 3 shortest wavelengths, but at all other wavelengths the frozen sample is more reflective. However, the 632-614 nm difference was markedly smaller for oxyMb in frozen than in unfrozen muscles.

Figure 9 shows the spectral reflectance of unfrozen and frozen muscle in metMb form. Spectra are quite similar, with slightly higher reflectance for unfrozen muscles at 400, 450, 474, 500 and 650 nm and for frozen at 685 nm. The 632-614 difference is less for the frozen form.

Figure 10 shows the spectral reflectance of frozen muscle in redMb, oxyMb, and metMb forms.

Reflectance for redMb is greater than for the other 2 forms at wavelengths in the range of 400 to 632 nm and for the difference between 632 and 614 nm reflectance. The results of 474 nm and for 632-614 appear to support earlier findings, but the difference at 525 was not expected as this wavelength was reported to be isobestic for all 3 Mb forms by Snyder[2]. The higher value at 632 nm is of doubtful significance.

TABLE 5 Percent reflectance at various wavelengths of fresh- and frozen-beef longissimus in Mb, oxyMb, and metMb states (mean ± S.E.).

Wavelength, nm	Fresh			Frozen, Day 1		
	Mb	oxyMb	metMb	Mb	oxyMb	metMb
400	6.02 ± 0.18	3.80 ± 0.13	2.98 ± 0.31	5.82 ± 0.71	3.48 ± 0.28	2.03 ± 0.19
450	8.44 ± 0.29	6.48 ± 0.26	10.00 ± 0.31	7.94 ± 0.15	6.96 ± 0.31	8.23 ± 0.38
474	14.60 ± 0.48	10.46 ± 0.31	11.68 ± 0.42	14.92 ± 0.33	10.32 ± 0.35	9.30 ± 0.23
500	12.82 ± 0.54	10.32 ± 0.26	10.22 ± 0.34	14.70 ± 0.27	11.72 ± 0.21	9.26 ± 0.49
507	11.32 ± 0.42	9.74 ± 0.29	9.96 ± 0.32	13.54 ± 0.25	11.82 ± 0.19	9.18 ± 0.46
525	9.72 ± 0.29	8.26 ± 0.22	10.14 ± 0.32	12.56 ± 0.37	11.24 ± 0.36	9.52 ± 0.42
555	7.66 ± 0.26	6.38 ± 0.18	10.86 ± 0.40	10.08 ± 0.46	9.36 ± 0.38	11.18 ± 0.63
572	7.62 ± 0.26	6.86 ± 0.32	11.78 ± 0.48	10.18 ± 0.42	10.26 ± 0.49	12.72 ± 0.67
582	8.18 ± 0.49	6.34 ± 0.19	12.10 ± 0.51	10.54 ± 0.47	8.96 ± 0.49	13.08 ± 0.66
597	9.44 ± 0.43	9.12 ± 0.14	13.60 ± 0.53	14.18 ± 0.18	16.36 ± 0.30	14.66 ± 0.62
600	10.38 ± 0.37	10.40 ± 0.22	13.94 ± 0.55	15.22 ± 0.37	18.46 ± 0.22	15.00 ± 0.65
630	20.08 ± 0.58	20.74 ± 0.62	17.10 ± 0.44	26.16 ± 0.62	26.54 ± 0.42	16.92 ± 0.68
635	21.36 ± 0.52	22.32 ± 0.67	17.14 ± 0.43	27.78 ± 0.77	27.44 ± 0.45	16.92 ± 0.68
650	23.66 ± 0.65	24.66 ± 0.85	19.78 ± 0.60	30.28 ± 0.75	30.28 ± 0.49	18.44 ± 0.65
685	28.62 ± 0.38	30.18 ± 0.80	33.86 ± 0.91	37.32 ± 1.06	39.86 ± 0.58	36.26 ± 0.96
614	16.34 ± 0.57	17.80 ± 0.58	16.54 ± 0.47	22.52 ± 0.67	20.74 ± 2.40	16.62 ± 0.57
632	21.04 ± 0.61	22.34 ± 0.74	17.14 ± 0.41	27.40 ± 0.76	23.12 ± 2.55	16.66 ± 0.56
632-614	4.70 ± 0.05	4.54 ± 0.22	0.60 ± 0.16	4.88 ± 0.10	2.38 ± 0.51	0.04 ± 0.06

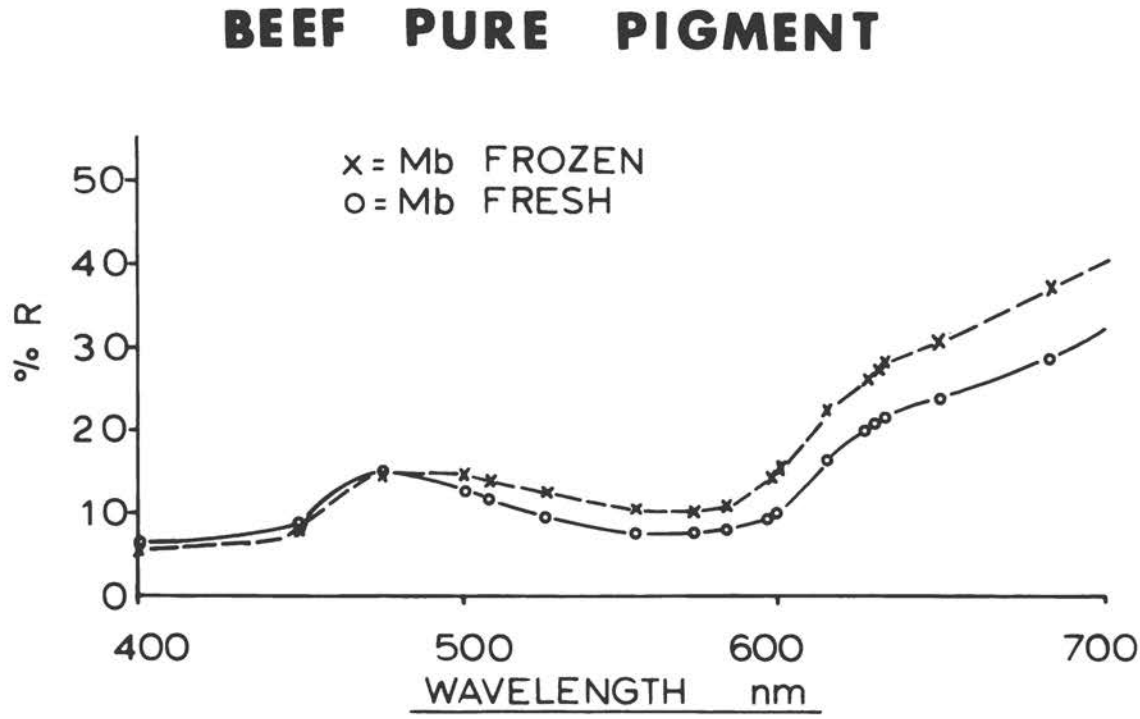


FIGURE 7 Reflectance spectra of fresh (unfrozen)- and frozen-beef longissimus in redMb state.

OxyMb showed greater reflectance than the other 2 forms at wavelengths of 597 and 600. MetMb had greater reflectance at 572 and 582 nm as expected from earlier reports for unfrozen samples.

MetMb showed a lower reflectance than both other forms in the range of 400 to 650 nm which suggests these are useful wavelengths for quantitative estimation of metMb. The difference between reflectance at 632 and 614 nm also seems potentially useful.

Table 6 shows reflectance values for frozen bovine longissimus in the redMb, oxyMb, and metMb when corrected to 10 percent reflectance at 525 nm for all 3. The reflectance for redMb was higher at 474 nm as reported in earlier studies with unfrozen samples, as were the values for metMb at 572 and 582 nm.

Finally, Table 7 presents some reflectance ratios for frozen bovine muscle in the 3 chemical states. The ratios involving reflectance of 474 nm/525 nm and 474 nm/597 nm seem able to distinguish redMb. The 474/525 ratio was suggested for measuring redMb by Snyder[2]. A ratio involving absorbance at 473 nm/597 nm was suggested by Broumand *et al.*[16] Later, Dean and Ball[4] using K/S values involving these wavelengths found them useful in measuring redMb.

Ratio of reflectance involving 572 nm/525 nm seems to distinguish metMb from the similar values for redMb and oxyMb.

TABLE 6 Reflectance percent, 525 corrected, for frozen-beef longissimus in redMb, oxyMb, and metMb.

λnm	Mb	oxyMb	metMb
474	12.36	9.08	9.78
507	10.98	10.58	9.66
572	7.62	9.02	13.20
582	7.98	7.72	13.56
600	12.66	17.22	15.48
630	23.60	25.30	17.40
650	27.72	29.04	18.92

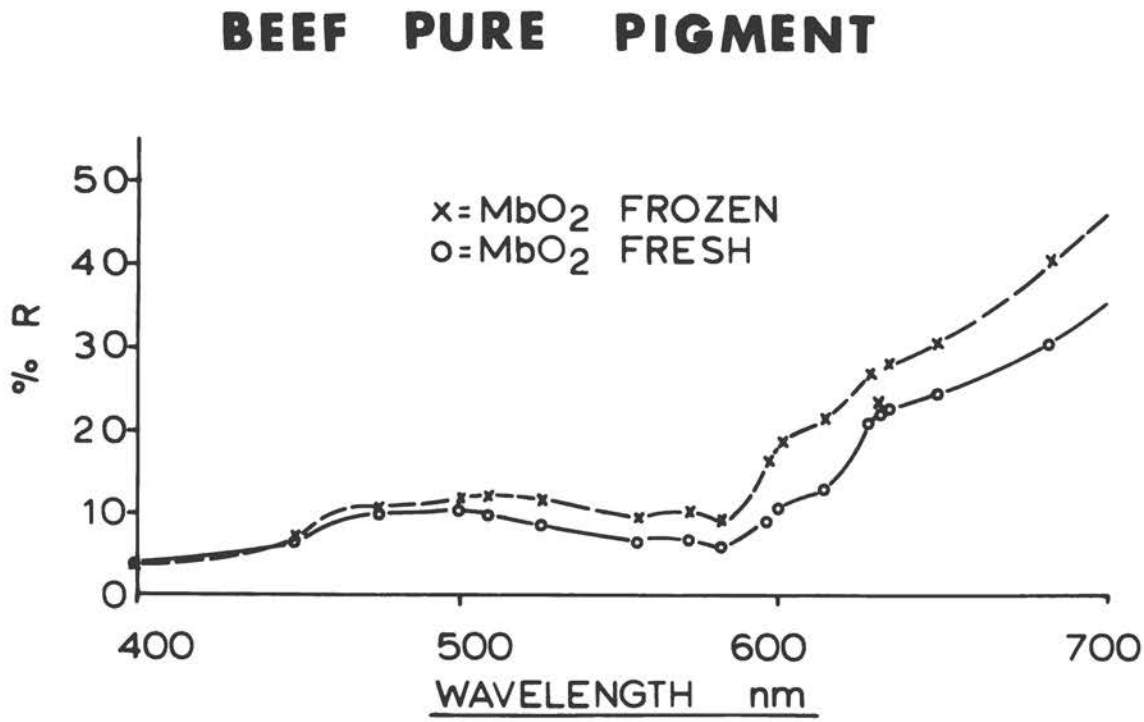


FIGURE 8 Reflectance spectra of fresh (unfrozen)- and frozen-beef longissimus in oxyMb state.

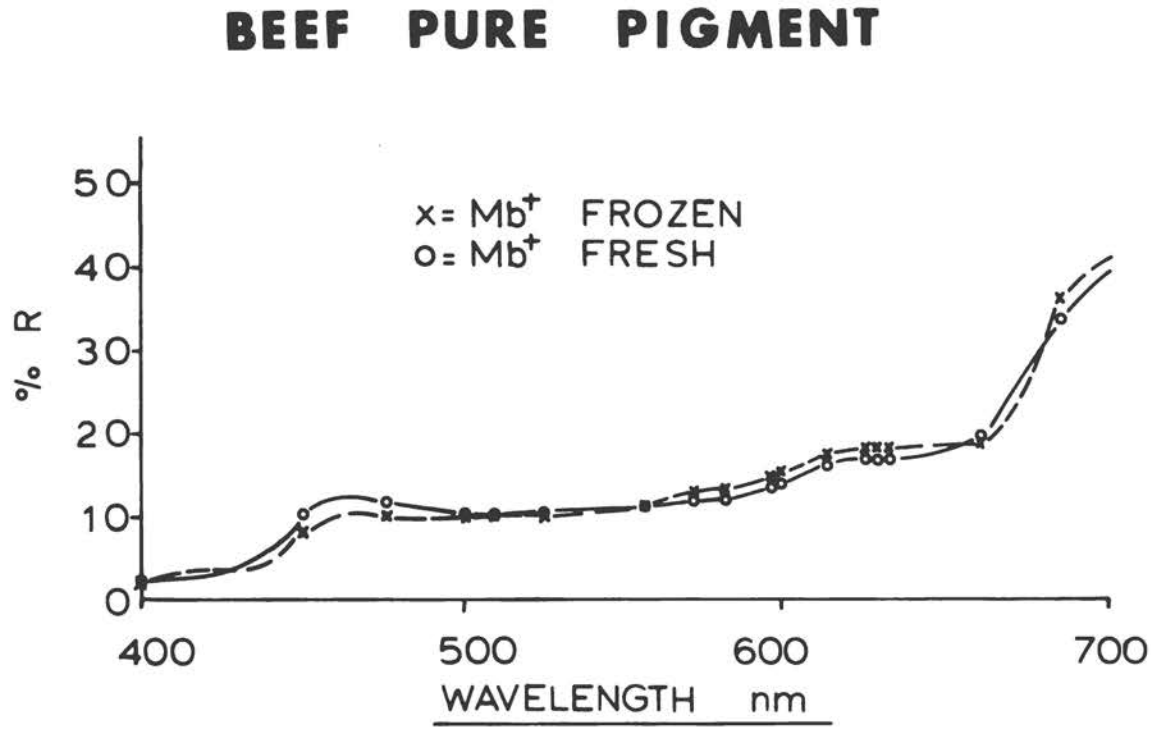


FIGURE 9 Reflectance spectra of fresh (unfrozen)- and frozen-beef longissimus in metMb state.

TABLE 7 Reflectance ratios for frozen-beef longissimus in redMb, oxyMb, and metMb forms.

	Mb	oxyMb	metMb
474/525	1.19	0.92	0.98
572/525	0.81	0.91	1.34
507/572	1.33	1.15	0.72
474/597	0.98	0.63	0.63
630/525	2.08	2.36	1.78

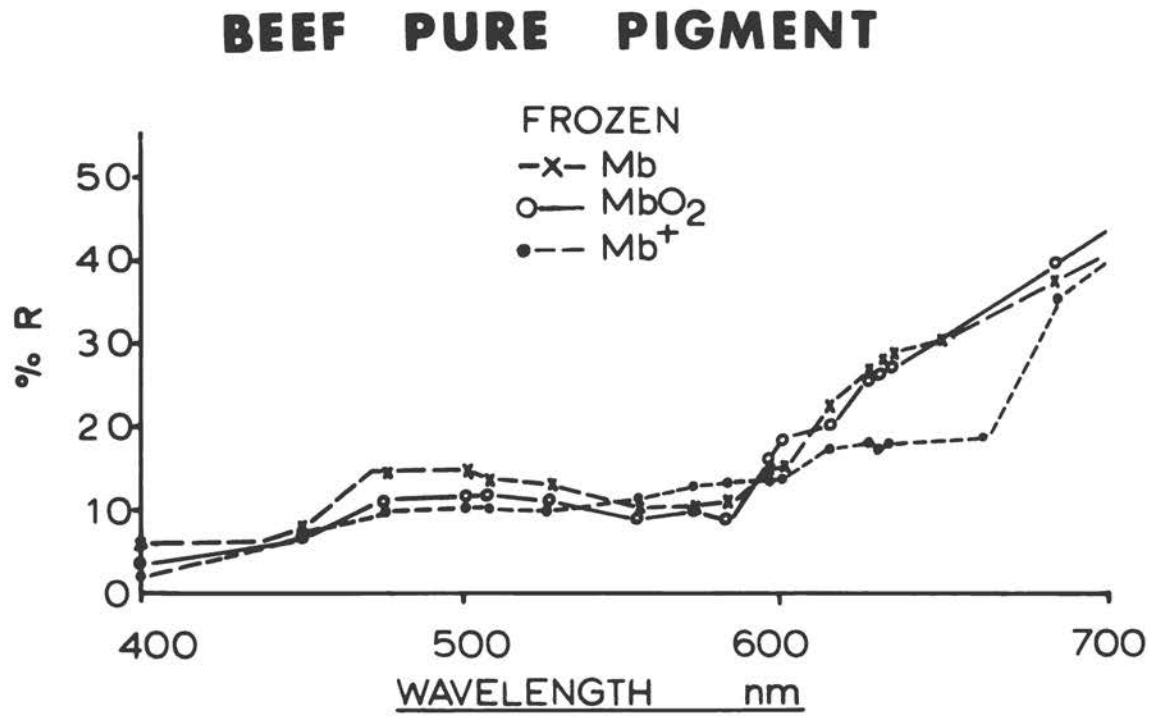


FIGURE 10 Reflectance spectra of frozen-beef longissimus in redMb, oxyMb, and metMb states.

This was expected based on Snyder's report that reflectance at 572 nm was isobestic for redMb and oxyMb and that at 525 nm was isobestic for all 3 forms.

The ratio involving reflectance at 507 nm/572 nm appeared able to distinguish metMb from the other 2 forms, but differing ratios were also noted for redMb and oxyMb forms, while Dean and Ball suggested such ratios (using K/S) would be the same.

The ratio of reflectance 630 nm/525 nm was expected to distinguish the metMb contribution to reflectance from that of the other 2 forms. However, similar values were not calculated for redMb and oxyMb forms and this ratio seems limited in usefulness in estimating metMb.

Table 8 shows K/S values (Judd and Wyszecki[17]) for frozen-beef longissimus in the redMb, oxyMb, and metMb forms. RedMb muscle had a different K/S at 474 nm than the other 2 forms, while oxyMb differed in K/S at 600 nm. MetMb showed a different value particularly at 572, 630 and 650 nm, but similar values were calculated for redMb and oxyMb forms at these wavelengths.

Various K/S ratios (Table 9) give similar trends to the ratios in Table 7 with regard to their ability to distinguish frozen-beef muscle in various pigment states.

TABLE 8 K/S for frozen-beef longissimus in redMb, oxyMb, and metMb states.

Wavelength, nm	Mb	oxyMb	metMb
474	2.426	3.897	4.423
507	2.760	3.289	4.489
525	3.044	3.504	4.300
572	3.962	3.921	2.995
582	3.796	4.625	2.905
600	2.361	1.8009	2.408
630	1.0421	1.0166	2.040
650	0.8027	0.8027	1.8038

TABLE 9 K/S ratios for frozen-beef longissimus in redMb, oxyMb, and metMb states.

	Mb	oxyMb	metMb
475/525	0.797	1.112	1.029
572/525	1.302	1.119	0.697
507/572	0.697	0.839	1.499
474/597	0.929	1.819	1.782
630/525	0.342	0.290	0.474

VISUAL BEEF-COLOR STANDARDS

Our research team has had considerable contact with industry efforts to freeze, distribute and merchandize frozen beef cuts in transparent film. Difficulty was encountered in attempting to describe product appearance via telephone conversations, which led to the conclusion that visual beef-color standards were needed.

Kansas State University researchers developed the circular, *Beef Color*[18], which has been very helpful in describing color problems and presenting useful suggestions regarding freezing, packaging, storage and distribution, and retail display. Pictorial standards are given for frozen beef of acceptable color and various degrees of brown, dark red, or bleached (whitened) discoloration.

Table 10 gives various tristimulus parameters for pictures from two different copies of *Beef Color* which had apparent visual

TABLE 10 Tristimulus parameters for different copies of *Beef Color* circular[a].

Sample	Background	x	y	Brightness	Dominant wavelength	Percent purity	Visual
3 Bleach I	White	.398	.334	27.05	598	30	Redder
II	White	.405	.345	26.38	594	33	
II	Black	.419	.349	24.45	595	36	
3 Brown I	White	.416	.339	14.93	600	32	Brighter,
Red II	White	.392	.357	16.22	587	33	less brown
I	Black	.429	.341	16.20	599	37	
3 Dark red I	White	.430	.328	16.93	608	36	Lighter
II	White	.418	.313	13.82	616	28	
II	Black	.400	.322	15.10	608	25	

[a] *Beef Color*[18].

differences, both using the $MgCO_3$ block behind the circular when the standard was scanned on the reflectance spectrophotometer. One picture was scanned with a white and then a black background. As this was a class exercise, data accuracy may be questioned, but considerable difference is noted between the two copies of the circular. In spite of the difference, this circular has been a widely used tool of meat-product evaluation.

CORRELATIONS OF VISUAL-COLOR AND VISUAL-BLEACH SCORES TO REFLECTANCE MEASUREMENTS

Experimental Procedure

Six wholesale beef loins from animals slaughtered the same day were used for each of 3 replications. The loins weighed between 25 and 27 kg, had small to modest amounts of marbling at the 13th rib and less than 1.3 cm of fat cover. For each replication, six 2.5 cm steaks were cut from each loin from immediately posterior to the point at which the psoas major muscle was large enough to cover the aperture of the spectrophotometer used for color measurement. In addition, extra steaks were cut for purposes of temperature recording during freezing and storage. The 36 steaks thus obtained were then randomly assigned to 36 treatment combinations. Different tables of random numbers were used for each replication. An experimental design including all the variable combinations used in this study is presented in Table 11. Factors studied include 3 film permeabilities, high (ethylene vinyl acetate, 465.00 ml $O_2/m^2/24$ hr/atm); medium (iolon, 209.30 ml $9_2/m^2/24$ hr/atm); and low nylon-polyethylene, 4.65 ml $O_2/m^2/24$ hr/atm); 2 freezing systems (liquid nitrogen vapor and freon immersion); 2 packaging temperatures (before and after freezing) and 3 display-case temperatures (-29, -21, and -12°C). T-bone and porterhouse steaks were used since they contain both a light-colored muscle (longissimus) with a relatively low oxygen requirement and a dark-colored muscle (psoas major) with a high oxygen requirement.

Liquid-nitrogen-treated steaks were frozen in an NCG-Ultra-Freeze Simulator Freezer using a freezing cycle of -18°C for 1/2 min, -46°C for 1/2 min, -73°C for 1 min, -101°C for 1 min, -129°C for 1 min and tempering for 1 min. Freon-treated steaks were frozen in a Dupont Laboratory model freezer, at a constant -31°C until the center of the steaks reached -5°C (about 6 min).

After a 30-min bloom period, 1/6 of the steaks in each replication were packaged in each of the 3 previously mentioned films. Packages were subjected to 13.6 kg of vacuum and heat-sealed, so that a skin-tight package was obtained. Remaining steaks were packaged in the same manner, after freezing.

Quickold freezer cases (no defrost cycles) were used to display steaks held at -12°C and Hussman cases were used for those

TABLE 11 Experimental design.

Film permeability to oxygen	Freezing system	Packaging time	Display-case temperature
Low	Nitrogen	Before freezing	-20°F (-28.9°C) - 5°F (-20.6°C) +10°F (-12.2°C)
		After freezing	
	Freon	Before freezing	(same for each as specified above)
		After freezing	
Medium	Nitrogen	Before freezing	
		After freezing	
	Freon	Before freezing	
		After freezing	
High	Nitrogen	Before freezing	
		After freezing	
	Freon	Before freezing	
		After freezing	

steaks stored at -21°C and -29°C. Case covers were placed over the steaks at 7 p.m. each night and were removed at 7 a.m. each morning, to simulate a 12-hr store operation. General Electric Delux Cool White Fluorescent lights were used at an intensity of 1076 lumens/m^2 at steak surfaces. Room lights remained off to remove effects of background lighting. Care was taken to display and evaluate the same surface during the entire study.

Both longissimus and psoas major muscles were subjectively scored for color and bleach development immediately post freezing, and after 1, 7, 21, and 42 days display. The muscles were scanned from 400 to 700 nm with a Bausch and Lomb Spectronic 600 with $MgCO_3$ blocks used for 100 percent reflectance standard at a scan speed of 250 nm/min. Data used included percent reflectance at 474, 525, 572, 610 and 685 nm. In addition, the areas under the reflectance curves from 400 to 700 nm (total reference), 650 to 700 nm (red reflectance), and 440 to 474 nm (blue reflectance) were obtained and 2 reflectance ratios were calculated (R474/R525 nm and R572/R525 nm).

Correlation coefficients of objective color measurements with subjective scores were calculated on a within-time-period basis.

Results and Discussion

Simple correlation coefficients between visual redness scores and objective variables (Tables 12 and 13) were generally low for both muscles. Significant correlations were obtained at various time periods between visual redness and percent reflectance at 610, 650, 685 nm, reflectance scan areas and the 2 ratios. The highest correlations for each muscle occurred between visual redness and 685 nm reflectance and 572/525 nm reflectance ratios. Ockerman *et al.*[8] studied fresh-beef longissimus color and reported correlations as high as 0.88 between visual color scores and percent reflectance at 685 nm. Freezing might have affected spectrophotometric properties of the meat surfaces in some way, causing lower correlations to be found in the present study.

Correlations between visual bleach scores and objective variables are presented in Tables 14 and 15. Correlations were significant between reflectance percentages and areas and bleach

TABLE 12 Simple correlation coefficients between visual redness scores[a] and objective variables (longissimus).

Day variable	0	1	7	21	42
474 nm	0.19	-.07	0.02	-.09	0.13
525 nm	0.18	-.01	-.01	-.12	0.10
572 nm	0.19	-.05	0.07	-.06	0.17
610 nm	0.09	0.02	-.24*	-.25*	-.03
650 nm	0.06	-.37*	-.29*	-.28*	-.06
685 nm	0.09	-.47*	-.22*	-.25*	-.12
A-1[b]	0.18	-.39*	-.14	-.16	0.04
A-2[b]	0.09	-.22*	-.21*	-.30*	-.16
A-3[b]	0.24*	-.41*	-.08	-.10	0.13
R-1[c]	0.17	0.02	0.31*	0.42*	0.48*
R-2[c]	0.11	0.38*	0.48*	0.42*	0.42*

[a]Visual redness scores: 1 = very bright red, 2 = bright red, 3 = slightly dark red or brown, 4 = dark red or brown, and 5 = extremely dark red or brown.
[b]A-1 = Area 400-700 nm (under scan curve).
A-2 = Area 650-700 nm.
A-3 = Area 440-474 nm.
[c]R-1 = R474/R525.
R-2 = R572/R525.
*($P < .05$).

TABLE 13 Simple correlation coefficients between visual redness scores[a] and objective variables (psoas major).

Day variable	0	1	7	21	42
474 nm	-.08	-.20*	0.01	-.07	0.04
525 nm	-.13	-.22*	-.01	-.08	0.01
572 nm	-.12	-.19	0.04	-.02	0.07
610 nm	-.25*	-.37*	-.20*	-.21*	-.12
650 nm	-.24*	-.43*	-.28*	-.27*	-.19
685 nm	-.16	-.40*	-.17	-.18	-.12
A-1[b]	-.10	-.31*	-.11	-.12	-.06
A-2[b]	-.14	-.36*	-.19	-.18	-.16
A-3[b]	-.11	-.20*	0.03	-.01	0.05
R-1[c]	0.24*	0.18	0.34*	0.13	0.17
R-2[c]	0.07	0.21*	0.55*	0.48*	0.45*

[a]Visual redness scores: 1 = very bright red, 2 = bright red, 3 = slightly dark red or brown, 4 = dark red or brown, and 5 = extremely dark red or brown.
[b]A-1 = Area 400-700 nm (under scan curve).
A-2 = Area 650-700 nm.
A-3 = Area 440-474 nm.
[c]R-1 = R474/R525.
R-2 = R572/R525.
*($P < .05$).

scores but were nonsignificant between bleach scores and the calculated ratios. These results were expected because severely bleached steaks had reflectance percentages higher ($P < .05$) than those obtained from non-bleached steaks. Bleach did not affect the ratios because the division of two high-reflectance percentages canceled out.

CORRELATIONS BETWEEN SUBJECTIVE COLOR SCORE AND OBJECTIVE COLOR MEASURES (UNIFORM, NON-UNIFORM STEAK STUDY)

Experimental Procedure

T-bone steaks from 61 steer carcasses each representing 1 of 14 breed groups originated from the USDA Meat Animal Research Center beef germ plasm study at Clay Center, Nebraska, and were treated similarly for breed group comparisons.

The fresh muscles of all steaks (longissimus and psoas major) had been visually scored for color redness using the Kansas State

5-point scale. Steaks were bloomed for 30 min and frozen with liquid-nitrogen vapor in an NCG Ultra-Freeze Simulator Freezer and packaged. A step-wise freezing cycle from -18°C to 129°C required 8 to 9 min to achieve half-radius freezing (1/4 penetration from each side at thinnest dimension). The crust frozen muscles were then surface-warmed just enough to dissipate frost and immediately vacuum-packaged in 3-mil Iolon transparent film (O_2 permeability 209 ml $O_2/m^2/24$ hr/atm) using a Dupont MSP-1 packaging machine.

Longissimus and psoas major were also scanned from 400 to 700 nm by a Bausch and Lomb Model 600 reflectance spectrophotometer. Color observations were made immediately after freezing and packaging (day 0) and after frozen display for 1, 3, 7, 14, and 42 days at -25°C and under 1076 lumens/meter^2 of Delux Cool White Fluorescent lighting for 24 hr per day.

Simple correlation coefficients were calculated to determine the relationship between percent reflectance at each wavelength

TABLE 14 Simple correlation coefficients between visual bleach scores[a] and objective variables (longissimus).

Day variable	0	1	7	21	42
474 nm	0.57*	0.55*	0.64*	0.74*	0.76*
525 nm	0.58*	0.58*	0.64*	0.74*	0.77*
572 nm	0.57*	0.56*	0.60*	0.74*	0.74*
610 nm	0.46*	0.54*	0.62*	0.72*	0.78*
650 nm	0.39*	0.44*	0.58*	0.71*	0.77*
685 nm	0.38*	0.39*	0.41*	0.64*	0.67*
A-1[b]	0.18	0.50*	0.48*	0.67*	0.73*
A-2[b]	0.36*	0.03	0.33*	0.55*	0.36*
A-3[b]	0.63*	0.33*	0.39*	0.74*	0.28*
R-1[c]	0.20*	0.01	0.03	-.06	-.02
R-2[c]	0.05	0.14	-.14	-.13	-.20

[a]Visual bleach scores: 1 = no bleach, 2 = slight bleach, 3 = moderate bleach, 4 = very bleached, and 5 = extremely bleached.
[b]A-1 = Area 400-700 nm (under scan curve).
A-2 = Area 650-700 nm.
A-3 = Area 400-474 nm.
[c]R-1 = R474/R525.
R-2 = R572/R525.
*($P < .05$).

TABLE 15 Simple correlation coefficients between visual bleach scores[a] and objective variables (psoas major).

Day variable	0	1	7	21	42
474 nm	0.71*	0.42*	0.66*	0.60*	0.62*
525 nm	0.72*	0.45*	0.68*	0.70*	0.64*
572 nm	0.69*	0.42*	0.66*	0.69*	0.64*
610 nm	0.66*	0.46*	0.68*	0.71*	0.60*
650 nm	0.60*	0.43*	0.64*	0.69*	0.57*
685 nm	0.54*	0.30*	0.55*	0.68*	0.52*
A-1[b]	0.67*	0.38*	0.59*	0.66*	0.63*
A-2[b]	0.50*	0.18*	0.37*	0.60*	0.38*
A-3[b]	0.67*	0.20*	0.58*	0.65*	0.56*
R-1[c]	0.07	-.08	-.04	0.01	0.08
R-2[c]	0.20*	0.10	-.08	-.05	-.01

[a]Visual bleach scores: 1 = no bleach, 2 = slight bleach, 3 = moderate bleached, 4 = very bleached, and 5 = extremely bleached.
[b]A-1 = Area 400-700 nm (under scan curve).
A-2 = Area 650-700 nm.
A-3 = Area 440-474 nm.
[c]R-1 = R474/R525.
R-2 = R572/R525.
*($P < .05$).

(474, 500, 507, 525, 555, 572, 582, 597, 600, 630, 635, 650, and 685 nm) and subjective color-brightness score for 61 frozen T-bone steak muscles (longissimus and psoas major) at 6 different time periods of Delux Cool White Fluorescent lighted display (Tables 16 and 17). Also, strength of relationship was determined between 7 ratios of the percent reflectance at selected wavelengths (507/572, 474/597, 474/525, 572/525, 582/525, 630/525, 635/525) and subjective color scores for each muscle.

Results and Discussion

The simple correlations in Tables 16 (longissimus) and 17 (psoas major) do not show a wide dissimilarity among all reflectance percentages at the various wavelengths within day as related to subjective color score. Generally, the reflectance percentages are the best predictors of subjective color on day 1 or day 3 for

TABLE 16 Correlations of subjective color scores[a] to reflectance percent and ratios for frozen[b]-beef longissimus under Delux Cool White display lighting[c].

	Days					
(n = 61)	0	1	3	7	14	42
Percent R at:						
474	-.30*	-.43**	-.30*	-.21	-.11	0.04
500	-.34**	-.46**	-.34**	-.22	-.15	0.05
507	-.34**	-.47**	-.35**	-.22	-.16	0.05
525	-.33**	-.47**	-.35**	-.13	-.04	0.05
555	-.27*	-.44**	-.30*	-.17	-.08	0.08
572	-.30	-.46**	-.30*	-.12	-.01	0.08
582	-.27*	-.42**	-.25	-.12	-.12	0.10
597	-.34**	-.46**	-.34**	-.17	-.15	0.03
600	-.36**	-.47**	-.34**	-.20	-.25*	-.02
630	-.44**	-.48**	-.40**	-.30*	0.26*	-.08
635	-.45**	-.48**	-.40**	-.29*	-.25*	-.08
650	-.45**	-.48**	-.42**	-.27*	-.21	-.08
685	-.44**	-.43**	-.40**	-.29*	-.35**	-.06
Ratios of percent R/ percent R':						
507/572	-.15	-.08	-.25	-.42**	-.47**	-.18
474/597	-.20	0.13	0.13	-.07	0.08	0.04
474/525	-.01	0.08	0.18	0.02	0.15	-.01
572/525	0.05	0.06	0.28*	0.40**	0.36**	0.18
582/525	0.45**	0.23	0.36**	0.44**	0.31*	0.27
630/525	0.16	0.35**	0.00	-.17	-.15	-.28
635/525	0.17	0.35**	0.03	-.15	-.15	-.27

[a]Color scores: 1 (very bright red), 2 (bright red), 3 (slightly dark or brown), 4 (moderately dark or brown), 5 (extremely dark or brown).
[b]-25°C, 2 defrost cycles/day.
[c]1076 lm/m^2 fluorescent, 24 hr/day.
*Significant at $P < .05$.
**Significant at $P < .01$.

TABLE 17 Correlations of subjective color scores[a] to reflectance percent and ratios for frozen[b]-beef psoas major under Delux Cool White display lighting[c].

	Days					
(n = 61)	0	1	3	7	14	42
Percent R at:						
474	-.23	-.56**	-.53**	-.13	-.34**	-.15
500	-.22	-.62**	-.58**	-.17	-.41**	-.18
507	-.28*	-.62**	-.59**	-.17	-.42**	-.18
525	-.29*	-.62**	-.59**	-.18	-.43**	-.18
555	-.24	-.53**	-.47**	-.11	-.35**	-.16
572	-.26*	-.56**	-.49**	-.15	-.37**	-.14
582	-.22	-.55**	-.45**	-.12	-.34**	-.12
597	-.19	-.49**	-.56**	-.10	-.39**	-.20
600	-.21	-.51**	-.58**	-.15	-.44**	-.24
630	-.27*	-.63**	-.71**	-.33**	-.61**	-.39**
635	-.27*	-.64**	-.72**	-.35**	-.63**	-.39**
650	-.29*	-.64**	-.72**	-.36**	-.63**	-.37**
685	-.26*	-.53**	-.58**	-.32*	-.53**	-.29*
Ratios of percent R/ percent R':						
507/572	-.08	-.41**	-.59**	-.31*	-.27*	-.18
474/597	-.11	-.04	0.25*	0.10	0.27*	0.13
474/525	0.12	0.37**	0.32*	0.37**	0.31*	0.02
572/525	0.09	0.37**	0.56**	0.35**	0.30*	0.17
582/525	0.21	0.48**	0.69**	0.47**	0.48**	0.29*
630/525	0.11	0.05	-.42**	-.48**	-.55**	-.35**
635/525	0.12	0.03	-.42**	-.50**	-.58**	-.37**

[a]Color scores: 1 (very bright red), 2 (bright red), 3 (slightly dark), 4 (moderately dark), 5 (extremely dark).
[b]-25°C, 2 defrost cycles/day.
[c]1076 lm/m² fluorescent, 24 hr/day.
*Significant at P < .05.
**Significant at P < .01.

both muscles. At this time, under conditions of limited oxygen availability within the package, discoloration due to increased amounts of redMb would be expected to predominate and more color variability between steaks and muscles is due to variations in redMb, even though low partial oxygen (O_2) pressure would also favor rapid metMb formation. Day 0 reflectance for longissimus and day 14 reflectance for psoas major also were good predictors of subjective color score. At each day of lighted display, percent reflectance at 630, 635, and 650 nm had the highest correlations with subjective color scores for both muscles. This could be interpreted in reverse since subjective color score is a good indicator of muscle redness (reflectance above 600 nm). Significant correlations at the lower wavelengths (below 600 nm) no longer appeared by day 7 or after for both muscles (except day 14 for psoas major); this might be interpreted as metMb becoming the principal color determinant or variant shortly after 3 days' display under lights. Since low reflectance above 600 nm, especially at 630 or 635 nm, is a measure of metMb formation, other contributing sources to color at the lower wavelengths apparently become minor and metMb formation may predominate. Low correlations at day 42 likely also indicate a high degree of color deterioration in all muscles, i.e., very little color variability.

The best predictors of subjective color score were reflectance at 630, 635 or 650 nm on day 1. Redness should be expected to be most intense and easily detected visually early in the display period when effect of other competing factors for O_2 have had the least time to be expressed, but higher correlations could also result from greater color and pigment chemical form heterogeneity at this time.

The low correlation coefficients for both muscles on day 7 may indicate color deterioration due to redMb in some muscles and to metMb in others. The Kansas State color redness scale essentially does not distinguish between color deterioration contributed to these 2 pigment forms, although some reflectance measurements can.

Day 42 percent reflectance correlations for 630, 635, and 650 nm for psoas major were significantly related ($P < .01$) to subjective color brightness score. These results could be indicative of sufficiently greater pigment content variation which allowed metMb differences (Franke and Solberg,[6]) to be detected or to greater metMb reducing activity (Stewart *et al.*[3]) in psoas major resulting in larger, more detectible color differences.

The correlations for 7 ratios of percent reflectance to subjective color scores are found on the lower half of Tables 16 and 17. Marked differences are found for muscle and days of lighted display. Ratios may be useful in minimizing effects of non-pigment factors upon color measurements.

Ratios of 474/597 and 474/525 exhibited the least value for predicting subjective color score in either muscle at any time.

Dean and Ball[4] reported 473/597 ratio was a measure of relative proportion of Mb to oxyMb plus metMb while 507/573 was a

measure of metMb compared to oxyMb plus redMb. Snyder[5] used ratio K/S 572 nm: K/S 525 nm to measure metMb accumulation. Correlations for 582/525, 507/572, and 572/525 ratios with subjective color scores were approximately equal in predictive value at all times after 3 days' display, but the 582/525 ratio was a better predictor for most time periods. Marked differences between muscles were not found, but the psoas major correlations for 582/525 with subjective color were generally a little higher. That ratio should be indicative of the metMb portion of total pigment. The 507/572 ratio demonstrated time differences with muscle; correlation coefficients were the highest at days 7 and 14 for the longissimus while they were highest at days 1 and 3 for the psoas major. This may result from the more rapid discoloration in some psoas major samples causing more color variability on days 1 and 3.

The 630/525 and 635/525 correlation coefficients tended to closely parallel each other within muscle; however, between muscles an early correlation (day 1) for longissimus is highest while subsequent time values for psoas major (days 7 and 14) are highest. Perhaps metMb reducing activity differs in the 2 muscles, indicating that early darkening in longissimus may be due to metMb accumulation while in the psoas major it may be due to redMb.

Again, the reflectance data should be adjusted to K/S and re-subjected to correlation with visual scores, but time has not allowed this yet.

PROBLEMS IN COLOR APPRAISAL

A problem that hinders higher correlations between visual color and reflectance data appears to be the color variation encountered in a frozen muscle, especially in advanced stages of color deterioration. In visual appraisal, an average evaluation of the entire muscle surface seems logical, but the light beam of a reflectance spectrophotometer strikes a very small portion of muscle surface that is not "average" for the entire surface. A larger light beam would be useful and instruments with such a beam may show a closer correlation with visual appraisal.

Sample surface may show a slight concave or convex effect. Steaks packaged before freezing showed little of this problem, but those frozen before packaging had considerable surface irregularity.

Discoloration can result from darkening due to increased redMb, from darkening or brownish discoloration of metMb or to further oxidation of Mb. Even under frozen conditions, activities such as use of oxygen by muscle enzyme systems and Mb reduction and oxidation continue.

GENERAL SUMMARY

Freezing does not appreciably alter the reflectance spectrum in a redMb chemical state. Spectra for oxyMb and metMb are altered by freezing, but considerable difference between frozen-beef longissimus in predominantly redMb, oxyMb, and metMb form is found at wavelengths that are also useful for measuring proportion of various chemical states of Mb in unfrozen-beef muscle.

Ratios of reflectance or K/S of 474 nm/525 nm or 474 nm/597 nm appear to distinguish the reduced from the other 2 forms, as does K/S at 474 nm.

K/S at 600 nm appears able to distinguish oxyMb. K/S at 572, 630 or 650 nm appears capable of distinguishing metMb as does a reflectance or K/S ratio involving 572 nm/525 nm.

Samples with varying degrees of bleach show low correlations of visual redness to reflectance, but reflectance ratios are more closely related to visual redness. Degree of bleaching (whitening) of frozen-beef longissimus and psoas major was strongly correlated to reflectance at 474, 525, 572, 610 or 650 nm but not to reflectance ratios.

Visual beef-color standards with variations in brightness to darkness or brown discoloration and variations in degree of bleach have been a useful tool in industry. Most reflectance spectrophotometers or colorimeters are too unwieldly to use at various points in frozen-beef color evaluation. Hopefully, portable fiber optic scanners can be developed from information gained from above objective reflectance measurements.

REFERENCES

1. P. C. Pirko and J. C. Ayres, "Pigment Changes in Packaged Beef During Storage," *Food Technol.*, 11:461 (1957).
2. H. E. Snyder, "Analysis of Pigments at the Surface of Fresh Beef with Reflectance Spectrophotometry," *J. Food Sci.*, 30:457 (1965).
3. M. R. Stewart, M. W. Zipser, and B. M. Watts, "The Use of Reflectance Spectrophotometry for the Assay of Raw Meat Pigments," *J. Food Sci.*, 30:464 (1965).
4. R. W. Dean and C. O. Ball, "Analysis of the Myoglobin Fractions on the Surface of Beef Cuts," *Food Technol.*, 14:271 (1960).
5. H. E. Snyder, "Analysis of Pigments at the Surface of Fresh Beef with Reflectance Spectrophotometry," *J. Food Sci.*, 30:457 (1965).
6. W. C. Franke and M. Solberg, "Quantitative Determination of Metmyoglobin and Total Pigment in an Intact Meat Sample using Reflectance Spectrophotometry," *J. Food Sci.*, 36:515 (1971).

7. L. D. Satterlee and W. Hansmeyer, "The Role of Light and Surface Bacteria in the Color Stability of Prepackaged Beef," *J. Food Sci.*, 39:305 (1974).

8. H. W. Ockerman and V. R. Cahill, "Reflectance as a Measure of Pork and Beef Muscle Tissue Color," *J. Animal Sci.*, 28:750 (1969).

9. L. E. Jeremiah, Z. L. Carpenter, and G. C. Smith, "Beef Color as Related to Consumer Acceptance and Palatability," *J. Food Sci.*, 37:476 (1972).

10. G. L. Zimmerman and H. E. Snyder, "Meat Pigment Changes in Intact Beef Samples," *J. Food Sci.*, 34:258 (1969).

11. D. A. Ledward, "Metmyoglobin Formation in Beef Stored in Carbon Dioxide Enriched and Oxygen Depleted Atmospheres," *J. Food Sci.*, 35:33 (1970).

12. C. S. Setser, D. L. Harrison, D. H. Kropf, and A. D. Dayton, "Radiant Energy Induced Changes in Bovine Muscle Pigment," *J. Food Sci.*, 38:412 (1973).

13. B. A. Eagerman, F. M. Clydesdale, and F. J. Francis, "Determination of a Method for the Measurement of Fresh Meat Color," *Inst. Food Technol.*, abs. #250 (1974).

14. A. H. A. Van den Oord and J. J. Wesdorp, "Analysis of Pigments in Intact Beef Samples," *J. Food Technol.*, 6:1 (1971).

15. E. D. Strange, R. C. Benedict, R. E. Gugger, V. G. Metzger, and C. E. Swift, "Simplified Methodology for Measuring Meat Color," *J. Food Sci.*, 39:988 (1974).

16. H. Broumand, C. O. Ball, and E. F. Stier, "Factors Affecting the Quality of Prepackaged Meat," II.E., "Determining the Proportions of Heme Derivatives in Fresh Meat," *Food Technol.*, 12:65 (1958).

17. D. B. Judd and G. Wyszecki, "Color in Business, Science and Industry," (John Wiley and Sons, Inc., 1963).

18. Kansas State University, *Beef Color*, Circ. No. 398, Department of Animal Science and Industry.

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

QUALITY EVALUATION OF FROZEN ENTREE PRODUCTS

Justin M. Tuomy

When Dr. Giffee and I first discussed the idea of having this Symposium, I was all for it. If anyone at all really knows the need for more and better "Objective Methods for Food Evaluation," it is those of us who work with food specifications in combination with the government procurement system where the low bidder takes everything. Therefore, I am very happy to participate in the Symposium and hope for nothing less than a resounding success.

Before I get on with the real subject of my talk -- evaluation problems with frozen entrees -- I would like to propose a general concept for your consideration and possible use during the Symposium. Most of you will agree that research and production people just don't speak the same language, nor do they usually know or care about each other's problems. Because of this, I suspect that many answers to production food evaluation problems are buried in research notebooks or publications with just a little work needed to make them effective. What we really have is a bunch of answers looking for problems. Just maybe the greatest successes resulting from our work here will come in helping those answers find their problems.

The trend in the military is more and more toward the use of convenience food including frozen entree items. The Army is implementing at selected bases the concept of central food preparation where frozen entrees along with other products are prepared centrally for later distribution and use in satellite dining halls. The Air Force has a single serving frozen system at Warren Air Force Base to supply isolated missile sites, and there are other situations in which frozen entrees are used or contemplated for use. In addition to in-house preparation, there is considerable pressure to buy frozen entrees from industry. We thus have two problem areas -- quality assurance if we buy the entree item, and quality control plus quality assurance if we make it. From our standpoint, the most difficult problem with a purchased item is quality assurance. Solutions to this problem will, for the most part, also solve quality control.

As this audience well knows, the basic parameters of food quality are subjective if we ignore health and safety

considerations. Quality is really what the consumer thinks it is and he bases his opinion on subjective factors. Even some of the things we ordinarily think of as objective quality criteria are based on subjective factors. For example, what is so magic about a maximum of 20 percent fat in ground beef? If we slip up to 21 percent or 22 percent, will the consumer know it? Of course, such tests are valuable and necessary for specification limit purposes, but to think of them as exact measures of consumer acceptance can lead to trouble. Recognizing, then, the subjective nature of food quality, let us look at the system we have to live with and the tools with which we work.

MILITARY PROCUREMENT SYSTEM

Military procurements are based upon specifications, either federal or military, prepared by Natick Laboratories. Ideally, a specification should contain only end-item criteria, with the government accepting or rejecting on tests and examinations of a representative sample of the product as delivered by the vendor. As a practical matter, almost all food specifications have to contain in-process requirements of one kind or another. Contracts are awarded to the lowest bidder. In addition, more than 60 percent of food procurements are set aside for small business. The combination of low bidder, small business set-aside, and the subjective nature of food quality make military food procurements a monumental headache.

Our current instructions for writing specifications are to use as little in-process inspection as possible, the ideal being complete acceptance of the delivered product on end-item, quality-assurance inspection and tests. Actually, our specifications are a hodgepodge of in-process requirements and end-item tests. Why? Simply because we do not have the tests to define a product clearly and adequately for a bid system. We don't really have very satisfactory in-process requirements either, but, currently, we can do a better job of defining a product with them than we can with end-item tests.

What our specification requirements really amount to is a collection of a fairly large number of rather insignificant tests, inspections, and product-history requirements that, taken collectively, we hope will assure us the product we want and need. The validity of practically any one of these requirements can be and often is questioned. For example, with a beef stew, what is so sacred about a particular meat grade? Or particular meat cut? Or the percentage of carrots in the formula expressed to the first decimal? It is very easy for a contractor to fudge on one or more of these requirements without making a provable change in product quality.

Three general methods by which the requirements can be circumvented are substitution of ingredients, dilution of ingredients, and shorting of process resources. Substitution of

ingredients could range from complete substitution such as one beef grade for another or partial substitution such as soy protein for 10 percent of the meat. Many of these substitutions can be very difficult or impossible to prove in the final product. One of the reviewers of this paper -- Mr. Bustead whom I am sure many of you know -- commented "if you can't tell, why do you care?" That opened up a rather sensitive, controversial area as he well-knew it would. In many cases we probably shouldn't care, but we really don't know for sure. Too many times we have seen a change made because it did not seem to matter, only to find acceptance of the product gradually decreasing for no known reason except the change. Dilution of ingredients such as adding more water to the formula or decreasing the amount of the expensive ingredient in the formula is also difficult to prove. Addition of more water should be detectable by a moisture test, but even here I have seen some arguments based upon differing compositions of raw materials from different lots. Shortening of process resources involves such things as speeding up the cooking process or avoiding some hand labor involved in making a good lasagna.

The procurement system the manufacturers of frozen entrees would like us to use is just to hand them (not their competitors, of course) a purchase order for their standard product or, at the very least, let them bid their standard product with no restrictions. Many of them claim, and with some justification, that their product is just as good or better than the specification item. The problems, however, are obvious when you consider that we buy on low bid, we set aside more than half of our food procurements for small business, and every manufacturer is not as honest as the day is long. But what do we really want when we buy frozen entrees?

PROCUREMENT CONCERNS

Basically, there are only four areas of concern to the government in buying frozen entrees. These are storage life remaining in the product when received, economics, customer satisfaction, and health and safety of the consumer. The using Services set down their minimum criteria for the product in these areas and we develop the specification to give them what they say they have to have. For the purposes of this discussion we can leave out health and safety of the consumer since they are two whole areas by themselves, both of which are better defined than the other three concerns. By economics I do not mean the overall cost to the government or the cost per serving. Rather, I mean value to the government and whether or not the government gets what it thinks it is paying for. The other two concerns are self-explanatory.

Storage Life Remaining When Received

Whether we like it or not, in the present military supply system much longer storage times are required than are customary in industry. With central procurement, the normal time from receipt to consumption is usually considered to be 4 months. This is with normal feeding at stateside bases. With overseas facilities, isolated bases, and other special uses the storage time after receipt may be as long as 1 year. What means do we have to assure ourselves that the products we receive will store satisfactorily for whatever time is necessary?

From a practical standpoint, we have nothing that can be used for a frozen entree item that will tell it is good for X more months in storage. Tentative efforts have been made to use TBA tests and the like, with little success. About the only thing we can do is establish in-process specification requirements that we know will give us the storage life we have to have.

Economics

If we are to ask for bids on a frozen entree item, we must know and be able to communicate to prospective bidders the product we expect to get. Furthermore, ambiguity has no place in the bid system; we must all be talking about the same thing. Then, when the successful bidder starts to deliver, our quality-assurance tests must show whether he is, in fact, delivering the product that we and all the other bidders thought we had specified. If not, then our rejection of the product on the basis of the failed test must be upheld by experts in the field and even by a court of law. Such tests are not included in our present specifications.

Consumer Satisfaction

While it is recognized that the present Army food service system is anything but consumer oriented, we still have to develop and buy products with consumer appeal. So, to introduce a product into the system we develop or adapt a product the consumer will accept. We then write a specification around it, using quality-assurance provisions and factors including in-process requirements we expect will result in procuring a satisfactory product. We would much prefer to have a test that would tell us that the product received is what its name says, and that the troops will like it.

At this point many of you in the audience are probably ready to shout "Taste Panel." Unhappily, we haven't as yet been able to establish taste panels as standard end-item tests in

specifications except in a few rather special cases. As it stands now, taste panels have problems that preclude their general use with Armed Service procurements, such as:

-- Taste panels are very awkward and expensive to use in day-to-day procurements. This is especially true for large-volume items, for which simultaneous procurements are going on hundreds of miles apart.

-- It is difficult to establish and maintain panels that truly represent consumers.

-- Panels are still subjective and it is impossible at the moment to set reject numbers that will really stand up under pressure.

Food-Evaluation Needs

What are we really looking for in food evaluation? Of course, what we would like is the magic black box that promptly flashes a green "accept" or red "reject" light when the product is inserted in it. What we will accept is any little improvement you can come up with that makes food qualities easier to define. And that is the name of the game: Define the product so that there is no argument on the storage life left in the product, the value of the product, and consumer acceptance of the product.

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

MODERN ANALYTICAL TECHNIQUES FOR INSPECTION PROBLEMS

F. M. Bordeaux and William L. Brown

INTRODUCTION

The assigned topic covers a wide field. It has often been said that practical application lags knowledge from basic research by about 20 years, and it is not possible to cover 20 years of methodology in a short paper.

We will attempt in this paper to discuss some methods that are available for measurement of food quality that could conceivably be used to evaluate attributes of interest in frozen entree products. Some of the knowledge and methodology discussed could be easily reduced to practical inspection techniques, some merely offer potential.

The reason for the lag between basic research in these areas and practical application in inspection and approval procedures is probably two-fold. One reason is that the necessary follow-through and gathering of data is expensive; the other is that more training and education are necessary for the more sophisticated techniques. With regard to the latter, however, note that some theoretically sophisticated principles have been reduced to the level of "insert sample, press button, and read dial" by enterprising companies. Examples are the rapid fat and moisture testers employed in quality control by meat and dairy industries. Some of these are based on such exotic (by 1950 standards) principles as X ray, microwave, infrared, and other electromagnetic absorption properties. This also points up the facts that if the money is available, the lag is not too long, and that what is "impractical" can change very rapidly under proper conditions.

Pertinent attributes of the frozen entree products are those connected with: (1) consumer satisfaction, (2) consumer safety, (3) economics, and (4) storage life (which is intimately related to the first three). The discussion will be concerned with available objective means of determining some of the attributes.

CONSUMER SATISFACTION

Consumer satisfaction is a major point to be considered. It is probably the most difficult to handle from a practical standpoint. Note that the taste test is the number one means of determining food acceptability. Flavor, odor, and texture are difficult to measure objectively. Ultimately, laboratory instruments may replace the taste panel, but most of the objective tests used are (and will be) based upon subjective judgments. For example, the familiar TBA test for rancidity is still based on subjective evaluation and correlation with numbers generated in the laboratory.

The importance of volatile content in product odor and flavor has been discussed by many authors. Modern techniques may be used to recover, concentrate, identify, and measure volatile components of foods. This information may be initially related to taste-panel results, and thus a major step may be taken in the objective assessment of flavor and acceptance.

The instrument most used for these studies is the gas chromatograph. Major variations in technique are concerned with methods for removing the volatiles from the sample and getting them into the chromatograph.

The gas chromatograph is a device for separating complex mixtures of compounds into their component parts and measuring them separately. It does this based on differences between compounds in their boiling points, molecular weights and sizes, and polarities. The sample is introduced into the chromatograph at the entrance of a long (often coiled) tube, or "column." The column is usually packed with a porous material or material with large surface area, which is often coated with an organic polymer. The column is continually swept with an inert "carrier" gas, and maintained at a temperature at which the compounds of interest are volatile. The inlet of the column is generally kept at a higher temperature, so that the mixture is instantly vaporized upon injection. As the vapor is swept by the carrier gas down the column, components of the mixture are differentially retained in the column, due to interactions with the column packing based on the previously mentioned differences in the compounds. As the compounds leave the column they enter the detector, where their presence in the carrier is converted into an electrical signal. The various types of detectors will not be discussed here. The electrical signal is fed to a chart recorder where the results are graphically displayed as a series of peaks. The time between injection of the mixture and the appearance of a given peak is a means of identifying a given compound, and the size of the peak is proportional to the amount of that compound in the mixture. Often, the compound is trapped (by condensation or absorption) as it leaves the detector, and identified by the mass spectrometer or infrared or ultraviolet spectrophotometer. Trapped materials may also be subjected to organoleptic tests, especially in odor work.

Sometimes in flavor characterization, the peaks are not identified, but used merely as "fingerprints." The pattern of peaks becomes the flavor profile for the product under study, and the presence or absence of peaks is correlated with panel results. Often, the number and complexity of patterns obtained by gas chromatography requires computer storage and manipulation. There are already on the market gas chromatographs interfaced with mass spectrometers and/or computers. Interfacing equipment is available for converting output of the gas chromatograph to digital form in order to process the information by any of a number of small laboratory-size computers.

Some really elegant studies of this type have been done by Persson and von Sydow[1,2]. In their work on the aroma of canned beef, they identified 95 compounds in the headspace gas of canned beef. Using a computer, they developed regression models relating sensory data to the chemical data measured. They were able to show, using their approach and the models used, that sensory properties may be predicted using gas chromatographic techniques.

Sterken and Kempton[3] reported on a quality-control program for the dairy industry using gas chromatographic techniques. They measured the fatty-acid composition of ripening cheeses, using techniques that could be applied to quality assessment of other foods. They were also able to detect incipient growth of yeast on a packaged cheese by the presence of ethanol, which is not a normal constituent of the product. This suggests a possible use of the technique in storage-life assessment.

Dupuy *et al.*[4] reported on direct gas-chromatographic examination of volatiles in salad oils and shortenings. They examined numerous samples and reported that the better-quality oils had only a small amount of volatiles.

Reymond[5] reviewed several analytical techniques used in quality assessment of foods, including gas chromatography and ultraviolet absorption of volatile constituents. He pointed out that sometimes the compounds correlated with disagreeable qualities in a product may not be responsible for the qualities, but may vary in concentration to the same extent as the less easily detectable "guilty" compounds.

Objective tests for color are now easily made. Two devices for such measurements now on the market are the Hunter Color Difference Meter and the Gardner Color Difference Meter. In addition, one may obtain reflectance attachments for many commercial spectrophotometers. Strange *et al.*[6] showed that such devices could be used to predict consumer acceptance of meat color. It is likely that such measurements could be of value in other products not yet tested.

Texture is an important component in acceptance of foods. Tenderness is correlated with shear strength of the product. Texture qualities may be measured by a variety of devices, many of them custom made. These devices measure and plot low-intensity force-time curves. Such a "mechanical mouth" is described by Desrosier[7]. The expressable fluid of a cooked product is related

to its tenderness, juiciness, and overall acceptance. It is possible that correlations between this value and acceptability could be made for given products and used in objective testing.

CONSUMER SAFETY

Discussion of consumer safety could fill many volumes. We will just mention a few areas and confine detailed discussion to microbial aspects, which tie in with product storage life.

In measurement of pesticide residues, progress is being made in shortening the presently lengthy sample clean-up procedures by using automated liquid chromatography on gel columns to separate residues from fat, prior to gas-chromatographic measurement of residues.

Heavy metals are now capable of detection in the parts-per-billion range in a number of foods, with very little sample preparation, by means of the flameless atomic absorption spectrophotometer, which vaporizes the sample in various types of electrically heated furnace attachments.

Work is being done on rapid measurement of aflatoxins, using the new techniques of high-pressure liquid chromatography in conjunction with fluorescence detectors.

Modern rapid methods for the detection, quantification, and identification of bacterial contaminants have proliferated in the past few years. Many of the methods would require some modification before they could be applied to foodstuffs, but in principle, they have great potential in this area.

Crum[8] reported a rapid method of detection of bacterial cells by measuring the uptake of radioactive phosphorous in the growth medium. One company offers an automated system* to determine bacteria in blood cultures, based on the release of radioactive carbon dioxide from C^{14} labeled substrates. There are also on the market several devices which quantify living bacterial cells by their ATP content and which can be calibrated to read out directly in cell numbers instantaneously. This type of system, however, is subject to many interferences at this time, and has found application mostly in monitoring very simple systems. Immunofluorescence methods for detection of Salmonella are promising, but problems still exist with this technique, as pointed out by Insalata *et al.*[9].

In the area of organism identification, there are also several modern techniques which may find application in protection of the consumer. Two of these use the gas chromatograph. One approach is to measure specific metabolites when the organism in question is grown on selected media. Another is to inject a suspension of cells into a small furnace at the entrance to the

---

*Johnston Labs "Bactec" systems.

chromatographic column and use the peak profile of the pyrolysis products as a kind of "fingerprint" to help in identification of the organism.

In our laboratory, we use the types of organisms present and their growth characteristics to determine the history of product handling throughout the processing and storage of the food item. Different products will have different microbe loads and different species depending on handling characteristics.

ECONOMICS

On attributes connected with economics, I will just mention methods to detect deliberate or inadvertent adulteration of products with lower grade or substitute materials. Some modern methods for measurement of moisture and fat content of foods were described in the introduction. With the growth in use of soy protein products and the increase in types of soy products on the market, methods will be necessary to measure these as potential deliberate additives. Simple methods exist for the less refined products, but once the protein is isolated from other components of the soya material, it is only detectable if it has been properly "tagged" with titanium. It is possible that available electrophoretic techniques may be simplified and standard methods developed for the detection and quantitation of foreign proteins in meat products. It should also be noted that rapid automated methods (Technicon Co.) now exist for total protein determination.

STORAGE LIFE

As mentioned before, storage life is interrelated with the other factors of consumer satisfaction or acceptance, safety, and economics. Actually, "product history" might be a more accurate term, since handling or mishandling of the product (and ingredients) prior to and during formulation, cooking, and freezing directly determines the storage life of the finished product.

Methods for determining whether foods have been frozen and subsequently thawed are important, but outside of the more obvious visible results in some foods, objective methods are lacking. The Association of Analytical Chemists[10] has recently added to its "Methods" a test for the detection of frozen and thawed shucked oysters. This test utilizes gel electrophoresis and ultraviolet spectrophotometry to separate out and measure enzyme activity that is latent in the whole tissue but is released by the freezing and thawing process. Pherhaps similar methods are possible for other foodstuffs. Advantage may also be taken of other histological changes in the tissues, such as fiber diameter and void space. Use could be made here of the now relatively common scanning electron microscope.

The maximum internal temperature that a precooked item has attained is a very important parameter. Current methods are either crude (coagulation test) or time-consuming (phosphatase activity). A rather sophisticated test was reported by Y. B. Lee *et al.*[11]. They used SDS-acrylamide gel electrophoresis to determine the maximum temperature to which bovine muscles had been cooked, and reported that characteristic electrophoretic profiles were obtained for each temperature examined (at 5° intervals from 60°C to 90°C), which were quite reproducible. This suggests that it may be possible to use high-pressure liquid chromatography with molecular exclusion gels to obtain a more rapid test. The coagulation test, where the temperature at which an aqueous extract of the sample forms a "cloud" may also provide an indication of previous maximum temperature. Perhaps a device could be used similar to that used for measuring the thermal denaturation or "melting point" of bacterial DNA. It consists of a temperature-programmable cuvette holder with an electronic thermometer installed, mounted in a recording spectrophotometer. The thermometer output is fed to one axis of an X-Y recorder and the absorbance output of the spectrophotometer is fed to the other. In use, the temperature of the sample in the cuvette is raised gradually and the readout is a graph showing an inflection in absorbance at the point where the DNA helix "unwinds." A simpler version of such a device could be used to automate the coagulation test.

A real need exists for some method or methods of recording the time/temperature history of the finished product. If one knew the quality of the ingredients in a product, such a record would eliminate the need for many of the other tests discussed. K. H. Hu, at U.S. Army Natick Laboratories, reported on a time-temperature recording system in 1972[12]. This system is based on the principle that the permeation of oxygen through plastic films is a function of time and temperature. A suitable oxygen-reactive chemical system is enclosed in a thin plastic pouch; the extent of reaction that occurs in the system is a function of the amount of oxygen that permeates the pouch. In a practical system, sodium anthroquinone sulfonate and zinc dust were dissolved in aqueous alkali, yielding an opaque red solution. Upon exposure to oxygen, the solution becomes transparent. Thin pouches filled with the red solution were used to cover the lettering in an expiration message on the product container. Thus, the time at which the message became visible was then dependent on the temperature at which the product was held. There are many possibilities for use of this clever approach or modifications of it.

There are also possibilities in the electronics field for recording time/temperature histories in a simple manner. For example, perhaps a chemical system could be devised, the conductivity of which changes permanently with time and temperature, and which could be measured periodically with a conductance bridge to determine status of the product.

Desrosier[7] said that an enormous body of information is needed on which to establish the effective storage life of foods,

including data on changes in color, odor, flavor, texture, nutrient content, rancidity, and changes in product acceptability. We have discussed the objective measurement of several of these. Since several of the papers given at this Symposium deal directly with the problems of fat and oil stability, I will report just a little on the subject of rancidity in this paper.

Methods for detecting incipient rancidity or predicting future rancidity problems with a product are needed. If we put aside the measurements of peroxide or TBA values, which are of limited use in finished products, we are left with a fairly small number of current approaches in this area. Again, the gas chromatograph has been used by several investigators in rancidity studies. Fore *et al.*[13] use a very simple method for studying the shelf-life of peanut butter. They merely twist a glass rod in the sample and place it in a liner directly in the entrance to the column and measure the volatiles evolved. The ratio of methylbutanal to hexanal in stored peanut butter was found to be correlated with flavor scores. Correlation coefficients were significant to 0.5 percent. Warner *et al.*[14] measured pentane in the headspace gas of vegetable oils and potato chips. Pentane formation in the initial stages of autoxidation is indicative of rancidity in these samples. They found significant linear correlations between the amount of pentane developed and rancidity descriptions given by an 18-member panel.

Actual measurements of changes in lipid composition have been used by some investigators. Moerck and Ball[15] studied lipid autoxidation in mechanically deboned chicken meat. They measured oxidation of polyunsaturated fatty acids in the phospholipids by gas chromatography of their methyl esters, following the decrease in the ratio of amounts of unsaturated fatty acids to palmitic acid (unsaturation ratio). They also measured TBA values and found a high correlation between phospholipid fatty acid oxidation and TBA values.

St. Angelo *et al.*[16] used peroxide values and a spectrophotometric method to study the effects of various additives on shelf-life of peanut butter. They found that the increase in conjugated diene hydroperoxide found by measurements of absorption at 234 nm correlated well with peroxide values as rancidity developed. During the oxidation of unconjugated fatty acids, double bonds are rearranged to absorb strongly in the ultraviolet region.

The gas chromatographic profiles of volatile constituents of food products previously mentioned are useful in shelf-life measurements. By establishing the profile for freshly made product, the basic foundation exists to measure changes due to aging. Chromatographic profiles of aging product samples are made while the keeping qualities of these samples are investigated by a taste panel. Next, flavor changes can be correlated to instrumental data. As correlations develop and specific peaks or ratios are identified with flavor defects, it should become possible to study the effects of aging or even estimate the age of the product.

Nutrient levels in a product may also be used as indicators of mishandling or age. For example, Vitamin C and thiamin both decrease faster in storage than either color or palatability[7].

The quality of the finished product will undoubtedly improve as measurement techniques for control of agricultural products improve raw materials. A recent publication by E. E. Finney[17] deals with the subject of objective techniques for quality control of agricultural products. This publication discusses such approaches as transmittance and reflectance measurement, x-radiation, sonic and ultra-sonic energy for product evaluation.

In summary, we need to apply existing technology to establish meaningful chemical and microbiological procedures for food product specifications.

REFERENCES

1. T. Persson and E. von Sydow, *J. Food Sci.*, *38*, 377-385 (1973).
2. T. Persson and E. von Sydow, *J. Food Sci.*, *39*, 537-541 (1974).
3. Sterken and A. G. Kempton, *Dev. Ind. Microbiol.*, *15*, 24.
4. H. P. Dupuy, S. P. Fore, and L. A. Goldblatt, *J. Am. Oil Chem. Soc.*, *50*(9), 340-342 (1973).
5. Dominique Reymond, *Food Technol.*, *25*, 11, 78-82 (1971).
6. E. D. Strange, R. C. Benedict, R. E. Gugger, V. G. Metzger, and C. E. Swift, *J. Food Sci.*, *39*, 988-992 (1974).
7. N. W. Desrosier, *The Technology of Food Preservation*, 3rd edition, (13), 416-435 (1970).
8. M. G. Crum, *Dev. Ind. Microbiol.*, *12*, 191-196 (1970).
9. N. F. Insalata, C. Mahnke, F. C. A. Sunga, and G. H. Schell, *Dev. Ind. Microbiol.*, *13*, 353-364 (1971).
10. "Detection of Frozen and Thawed Shucked Oysters" Method 18.C05, 3rd supplement to 11th edition (1972), *Official Methods of Analysis*, Association of Official Analytical Chemists.
11. Y. B. Lee, D. A. Rickansrud, E. C. Hagberg, and E. J. Briskey, *J. Food Sci.*, *39*, 428-429 (1974).
12. K. H. Hu, *Food Technol.*, August, 56-62 (1972).
13. S. P. Fore, H. P. Dupuy, J. I. Wadsworth, and L. A. Goldblatt, *J. Am. Peanut Res. Educ. Assoc.*, *5*(1) 59-65 (1973).
14. K. Warner, C. D. Evans, G. R. List, B. K. Boundy, and W. F. Kwolck, *J. Food Sci.*, *39*, 761-766 (1974).
15. Kurt E. Moerck and H. R. Ball, Jr., *J. Food Sci.*, *39*, 876-879 (1974).

16. A. St. Angelo, R. L. Ory, and L. E. Brown, *J. Am. Peanut Res. and Educ. Assoc.*, *4*(1), 186-197, (1972).

17. E. E. Finney, Jr., *Measurement Techniques for Quality Control of Agricultural Products*, ARS-USDA (Am. Soc. of Agric. Engs., 2950 Niles Road, St. Joseph, Michigan 49085).

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

QUALITY EVALUATION OF EGGS AND POULTRY MEATS

Lee-Shin Tsai

INTRODUCTION

Shell eggs, egg products, and poultry meats are the end products of the poultry and egg industry. They constitute a broad spectrum of food products with distinguishing physical characteristics, such as the unique structure of shell eggs, the tremendous range of viscosity of egg products, and the dark and white meats of poultry. They are perishable but can be preserved in refrigerated, frozen, and dehydrated forms. Because of these variations and the various ways in which they are consumed, the quality criteria for them are different. Consequently, the methods of evaluation of these qualities are numerous, and some are general and some specific. I would like to discuss with you those which I think would best fit the theme of the Symposium during the next half hour.

The methods of evaluating poultry meat qualities are similar to those applied to other carcass meats. I shall not cover the evaluation of meat tenderness and color, which has been discussed by Drs. Herring and Kropf.

SHELL EGGS

The rigid but fragile shell of shell eggs is the only part exposed to customers. Traditionally, its shape, color, smoothness, and porosity have played an important role in determining its quality, although it has long been recognized that these factors bear no direct relationship to the quality of the edible portion. The quality of the edible portion of egg is a reflection of the freshness of egg. Its grade is considered to be highest when laid, and degenerates with storage. The change in observed physical conditions, such as the enlargement of air cell, the weakening of vitelline membrane, and the thinning of albumen are the criteria of quality evaluation. In the United States, shell eggs are classified arbitrarily into AA, A, B, and C grades according to these physical conditions[1]. After these grades are established, the egg quality is correlated with other

factors such as genetic strain and age of the hen, nutrition, laying conditions, etc. These factors are known to directly or indirectly affect the quality of eggs[2-4].

External Quality -- The Soundness of the Shell

It is my opinion that we should not use the shape, color, smoothness, and porosity of the shell as quality criteria, especially in institutional food service where the majority of the eggs are presented to the consumers without their shells. For this reason, I shall not elaborate on the objective evaluation methods that have been developed for these qualities.

However, the soundness or the strength of the shell, which is related to the percentage of shell breakage, is of great economical significance, especially for eggs that are stored and transported for long distances. The methods for evaluating this property are reviewed here.

R. G. Wells[5], in his comprehensive review of the subject, has given details of the methods developed for the assessment of the soundness of egg shell. Similar information in bibliographic form can be found in the *Recommended Methods for the Analysis of Eggs and Poultry Meat*, published by the North Central Regional Technical Committee[6].

The methods can be divided into two categories. One is to measure the resistance to breakage or deformation of egg shell by an applied force. A test using a steel ball falling on the shell was suggested to measure the resistance of shell to impact force[7,8]. Other methods subject the egg shell to continuous compression at a point or a surface and result in puncturing or crushing[7,9-12]. The deformation of the shell by compression was studied by Schoore and Boersma with a specifically designed piece of equipment[13] and by a modified Kramer press[14]. One advantage of the method is that the shell remains intact after testing.

In the other category of methods for the evaluation of shell soundness, the thickness of egg shell is used as an indicator. Tyler has reviewed the shell properties such as pore number, shell protein content, membrane thickness, shell curvature, shell color, and egg size, and found that shell thickness was the only factor related to its soundness[15].

The direct measurement of shell thickness requires not only the careful removal of adhering membrane but also the measurement of a significant number of pieces to make the value reliable. The method is laborious and impractical. Many methods were developed to avoid using the direct measurement of shell thickness. They include the determination of shell weight per unit area[16-18], and the weight of shell as a percentage of total egg weight[19]. A routine method used widely in the egg industry is the determination of specific gravity of the whole egg. The method, suggested by Olsson[20], is based on the fact that the specific gravity of the shell is about 2.3, while that of the whole egg is

approximately 1.085. Thus, variations in specific gravity of whole eggs are due largely to differences in the amount of shell. The flotation method, using graded series of brine solutions of various specific gravities is simple and rapid but less accurate and is widely used for routine purposes in the production phase of the industry. However, since the increase in size of the air cell during storage changes the specific gravity, this method is not suitable for testing stored eggs.

James and Retzer[21] measured egg shell strength by electronically counting the number of emitted beta particles that are reflected by a given area of shell in a specific length of time. The authors claim that the amount of backscattering depends on both thickness and density of the shell and is an indicator of shell strength.

The ultimate purpose of the assessment of shell soundness is to predict the probability that eggs will not be cracked in the normal course of transit. However, there is little evidence in the literature of the reliability of these techniques in terms of field results. This perhaps is to be expected from the complicated field conditions and the numerous variabilities of packing materials available. The simplified laboratory tests in most cases can only be correlated to a single type of motion. For example, the falling steel ball test can be correlated with the ability of the shell to withstand a sudden impact force, and the shell crushing test with its ability to withstand constant pressure.

The beta backscatter test is based on the basic structure of shell rather than on applied forces. It also is not likely to be affected by the size of the air cell and could be applied to both fresh and stored eggs. A comparison of this laboratory test to tests under field conditions should be carried out to establish its validity in the egg industry.

Internal Quality

When the shell egg is intact, the internal quality is judged by candlers who observe the egg against a soft light source. Candlers are trained to detect defects such as enlarged air cell, blood and meat spots, broken vitelline membrane, rots, cracks, dirt, and deformations of the shell. There is no effective objective method available.

USDA has established series of photographic standards of broken-out eggs on a leveled smooth surface to show upper and lower limits of each grade[1,22]. The broken-out quality along with the shell quality determine the total quality of the shell egg. The broken-out quality is based on the firmness of the yolk membrane and the viscosity of the albumen. The objective methods developed to measure the variation of these physical conditions have been reviewed by Brant *et al.*[22], Wesley and Stadelman[23], and R. C. Wells[5]. They can be summarized into two categories: one measures the physical condition of the yolk; the other, that of the albumen.

Yolk index which is the yolk height on a flat surface divided by its average diameter was recommended by Sharp and Powell[24]. This method is too laborious for practical use. Wolk *et al.* recommended the use of yolk height only as an indicator of egg quality[25], the variation of yolk height with egg weight limits its application[2]. Fromm and Matrone[26] have devised a technique that involves placing a 2 mm capillary tube against the vitelline membrane, applying a given vacuum in the capillary tube, and measuring the time required to rupture the membrane.

The numerous methods of measuring the physical state of albumen have been reviewed by R. G. Wells[5], Brant and coworkers[22], and others[27,28]. They include the determination of the percentage of thick albumen in total albumen[29], the height of the thick albumen[30], the albumen index, which is the quotient of the height of thick albumen and its mean width[31], the albumen area index, which is the ratio of the area of the thick albumen to its weight[27], the Haugh unit, which is the logarithm of the height of the thick albumen corrected for egg weight differences[32], and the viscosity of the egg contents[33].

Brant *et al.*[22] concluded that the Haugh unit was the most satisfactory measurement of albumen condition because of its reliability, simplicity, and speed. The Haugh unit is based on the observation by Haugh in 1937 that the logarithm of the height of the thick albumen is an indicator of its internal quality. It implies that a change in height from 10 to 9 mm (or 1 mm) represents an unimportant difference in the quality of an egg, but a change in height from 2 to 1 mm, again 1 mm, represents a very significant difference. The logarithm of the thick albumen height of a 2-oz egg was chosen as an index. Eggs of different weight are corrected to a 2-oz egg by the following formula.

$$\text{Haugh Unit} = 100 \log [H - 1.7\, W^{0.37} + 7.6]$$

Where H: height of thick albumen in mm

W: weight of egg in grams

The widespread acceptance of the Haugh unit was aided by the introduction of a simple albumen-height measuring gauge[34] and the internal quality calculator for eggs by Brant *et al.*[22]. The development of an electronic digital albumen-height measurement gauge by Buckley and Reid[35] has simplified the measuring procedure and increased the reproducibility. Brant *et al.*[22] also correlated the Haugh unit with visual scoring by the USDA chart. They have shown that eggs above 79 Haugh unit are AA quality, below 31 are C quality, and that the difference between grades is approximately 16 Haugh units.

In reviewing the literature, I could not help wondering about the adequacy of this grading system, which arbitrarily classifies shell eggs into AA, A, B, and C grades according to their physical appearance. The implication of such a grading

system is that the quality of the eggs degenerates in that sequence. However, there has not been any convincing evidence to indicate that consumer preference parallels the grading system. The United States Procurement Grades is the specification that guides governmental agencies in their shell-egg purchasing[36]. It specifies that at their destination, eggs shall consist of 80 percent and 60 percent or more of A quality or better for Grade I and Grade II, respectively. The remaining 20 percent in Grade I may be below A quality, but not more than 5 percent may be below B quality. The remaining 40 percent in Grade II may be below A quality, but not more than 10 percent may be below B quality. The emphasis of A and B grades in their specification is an indication of their appreciation of A and B qualities. Unfortunately, the use of A and B grades implies the recognition of shell appearance as a factor in egg quality.

EGG PRODUCTS

The egg products are eggs removed from their shells. They may be in a liquid, frozen, or dehydrated state, and may consist of albumen, yolk, or any combination of the two. Certain additives such as sodium chloride, sucrose, and corn syrup are used in egg products. However, the egg product designation does not include those that have been heat-treated or cooked to cause extensive coagulation and loss of original characteristics. This precludes many precooked egg products from the definition even though they contain a high proportion of eggs[37,38].

Egg products are widely used as ingredients by bakeries, noodle factories, confectioneries, mayonnaise, salad dressing, and baby-food manufacturers, and to a lesser extent are used by commissaries and food-service institutions. However, the usage by the food-service industry has increased in recent years. This is partially due to the improvement in convenient packages such as the substitution of 5-, 8-, or 10-pound containers for 30-pound metal cans. It is also due to the improvement of egg-product quality and, specifically, the keeping quality.

Chemical Properties

The commonly determined quality for all egg products is their solids content (or moisture). The egg-solids content of commercial whole egg is 24.7 percent; and of yolk, 43 percent[38]. Commercial liquid albumen normally contains at least 11.5 percent solids. The specification on egg-solids content defines the ratio of yolk and albumen in the liquid and frozen egg products. The moisture content of the dehydrated products is normally less than 5 percent for yolk products and less than 8 percent for albumen products[39].

The drying-oven method established by the Association of Official Analytical Chemists (AOAC) for the determination of total solids is widely used[40]. Despite its wide acceptance, the time requirement, at least 4 hr, has prohibited its usage in places where immediate quantitative results are necessary. For speed, a refractometer is used to measure the solids content of egg magma. Refractive indices for whole eggs and yolks are difficult to determine due to the obscure dividing line in the refractometer. But it can be sharpened by adding 5 percent saturated salt solution to the sample[41]. Due to the variation in refractive index of individual eggs, the method has been limited to situations in which only rapid, but less accurate, determination of egg solids is needed.

Whole egg contains about 12 percent lipids contributed entirely by the yolk, which contains about 33 percent lipids[42,43]. Fresh egg albumen contains negligible amounts of lipids[44]. The total lipid content of commercial yolk is approximately 27 percent because of dilution by approximately 16 percent albumen. The yolk-containing products usually specify a minimum lipid content to limit the amount of albumen contamination. Similarly, the maximum allowable lipid content of the albumen product is specified to limit the yolk contamination, which is detrimental to the foaming property of albumen. The most commonly used method of lipid determination in egg products is the "Acid Hydrolysis" method[40]. The egg magma is first boiled with concentrated hydrochloric acid for 30 min, and then it is extracted with diethyl and petroleum ethers. The method is a determination of the ether extracts of the acid hydrolysates of the egg lipids.

To determine the minute yolk contamination in albumen products, both the "Acid Hydrolysis" method and the "Monomolecular Film Method" described by Bergquist and Wells[45] are used. The "Monomolecular Film Method" consists of extracting lipids with ethyl alcohol, diethyl, and petroleum ether; the ether extracts are then spread on the surface of an acetic acid solution. The area of the film is measured and converted to the lipid content of the albumen sample. The reproducibility has been good, but the technique, to many users, is quite tedious and laborious.

In the AOAC method for the determination of total lipids in egg products, the egg product is mixed with equal volumes of chloroform and ethanol. The clear liquid phase is then dried and redissolved in chloroform. The lipid content in the filtered chloroform solution is determined gravimetrically[40].

Undoubtedly, a uniform, simple, and reliable method for the determination of lipids in egg products is needed. There are several quantitative lipid-extraction methods for biological systems that may be readily adapted for egg products[46,47]. In the well-known method of Bligh and Dyer[46], for example, tissue is homogenized in a single-phase solution of chloroform, methanol, and water in the ratio of 1:2:0.8; then more chloroform and water are added to a final ratio of 2:2:1.8. The homogenate separates into chloroform and aqueous phases. The quantity of lipids in

the chloroform phase is then determined gravimetrically. The efficiency of a single extraction of fish tissue was reported to be 96 percent.

Even for the determination of minute contamination of yolk in albumen products, this method may be easily adapted by using 90 g of liquid albumen, which contains approximately 80 g of water, as testing material. For example, if the sample contains 0.01 percent lipids, the extractable lipids will be 8.6 mg. This is within the range of the sensitivity of regular laboratory instruments. The technique is simple, requires no heating, and can be carried out with the least amount of oxidation. It should be examined for the determination of total lipids in egg products.

Egg color is the other important egg-product quality. It is especially important to the noodle, mayonnaise, and frozen food manufacturers since eggs are responsible for the color of their finished products. Although the demands of the yellow intensity in these products have been gradually decreasing in recent years, the egg color of food items such as scrambled eggs, omelets, etc. in food service and the precooked frozen food industries remains an important quality characteristic.

Two objective methods are used in the industry for color determination. They are both based on the light absorption of the pigments extracted by acetone. The National Egg Products Association (NEPA) method compares the color of acetone extracts to the color of a series of aqueous potassium dichromate solutions using a colorimeter with blue filter[48]. The other method uses synthetic beta-carotene acetone solutions as standards and measures the absorption at 450 mμ with a spectrophotometer[49], and expresses the results in terms of equivalent micrograms of beta carotene per gram of egg. The latter has higher precision and reproducibility than the NEPA method[50]. It has been adopted by AOAC[40].

Unfortunately, neither of the chemical determinations of color correlates well with the visual response[51]. Bornstein and Bartov[51] compared the beta-carotene equivalent of yolk to the visual scores of dichromate solutions and found that the visual scoring is linearly proportional to the logarithm of beta-carotene in acetone extract. Therefore, once a certain visual color is reached, the further increase in visual effect will require excessive increase of beta-carotene equivalent, where the chemical determination of color could be misleading.

De Groote[52] reported that the percent reflectance of yolk at 520 mμ measured with a Beckman Model B Spectrophotometer with reflectance attachment was linear to the visual score of the Roche color fan. He attributed the linear relationship between the two methods to their identical basic vision principle. The reflectance method, I feel, should be further studied to extend its uses to egg-containing products such as noodles, mayonnaise, frozen scrambled eggs, omelets, etc. Then the color indices of egg, egg products, and egg-containing products would be measured objectively by one method which is linearly proportional to the visual scoring.

Functional Properties

In addition to the common quality requirements such as the microbiological, chemical, and organoleptical properties, each egg product's user often requires specific functional property tests to guarantee the performance of the egg product in his final manufactured products. These tests are designed by individual users to meet their specific needs, and consequently, should only be applied to the set of conditions for which the test is designed.

Albumen, both liquid and dehydrated, is largely consumed in the form of meringue-like products. The foaming ability and foam stability of albumen are among its most important qualities. The test commonly used for these properties is to whip liquid or the rehydrated dried albumen in a mixer at fixed speeds for certain periods of time. The height of the foam or specific volume of the foam is a measure of its foaming ability. Then the foam is left undisturbed for 30 min or longer, and the liquid draining from the foam is recorded as an indication of the foam stability[53]. Although the method is widely cited, its relation to the performance of albumen in different finished manufactured products is inconsistent. One reason for this discrepancy is the use of a fixed whipping time in the method. Albumen from different lots often requires different whipping times to reach maximum volume. The use of a fixed whipping time actually ignores the foam condition, whether it is under-beaten, optimum, or over-beaten. To compare the stability of foams whipped to stages other than their optimum is basically unsound.

In the baking industry, the foaming quality of the albumen product is more closely tested with the angel food cake method[54]. Several similar formulas and baking conditions were reported[55,56]. All formulas used a high percent albumen to increase the sensitivity of the test. The angel food cake test depends, in addition to foaming ability, on the foam stability at elevated temperature, the coagulating temperature of the albumen, and the interaction of added ingredients. These factors are not accounted for in the simple whipping test.

The role of yolk in the finished manufactured products is more complicated than that of albumen. Yolk acts as an emulsifier, leavening agent, and/or coagulating agent, depending on the product formula. The emulsifying ability of yolk and whole eggs has been evaluated with a mayonnaise test[57]. The yolk content of the test mayonnaise formula was reduced to a minimum from the commercial mayonnaise to decrease its stability, and to increase its sensitivity to the changes of egg products. A true sponge cake formula was used by Hanson *et al.*[58] to test the functional property of the yolk in baked goods. The formula includes 47 percent whole eggs without leavening agents, and has been demonstrated to be sensitive in detecting the possible effect of heat or mechanical damage to whole eggs[59].

Many more formulas and procedures for the functional tests could be cited, but these tests, like the few examples mentioned above, are not truly objective evaluation methods, because the quality of the baked product depends partially on the experts' subjective evaluation, and baking, in itself, is still an art.

The development of methods for scientific evaluation of the functional properties is hindered greatly by insufficient knowledge of the roles of the individual components of egg in these finished manufactured products. The basic physical structure of yolk elucidated by Cook and coworkers[60], and knowledge about the egg albumen proteins[61] have initiated studies to correlate the functional properties to individual fractions of egg[62-65]. However, the results are not yet conclusive.

POULTRY MEATS

The methods of characterizing the qualities, such as tenderness, juiciness, flavor, and color in poultry meat are broadly similar to those of other meats. Many objective evaluation methods have been developed to apply to all carcass meats. The methods for measuring beef tenderness reviewed by Dr. Herring and for beef color by Dr. Kropf should, with minor modifications, be adaptable to poultry meats.

The problem of the oxidative deterioration of refrigerated and frozen poultry is related to its lipid content and the content of naturally occurring antioxidants. The investigation of oxidative deterioration of chicken and turkey meats has been based on the induced changes of their lipid moiety. The testing methods include estimation of polyunsaturation of lipids by gas chromatographic analysis or by iodine value[66,67], determination of peroxide value[66,68], carbonyl formation[66,69] or the 2-thiobarbituric acid value[70-72], and observation of the induction periods of the extracted lipids[66]. All these methods were originated and used in the investigation of lipid stability, which is discussed by Drs. Erickson, Thomas, Henning, and Jacobson. Therefore, I shall omit the subject from my discussion.

The current federal specifications for ready-to-cook chicken and turkey (chilled and frozen)[73,74] define the requirements of each type, class, style, and grade. They include 5 types of chicken and 4 types of turkey. The differences are based on whether the products are fresh-chilled, frozen not more than 60 days, frozen more than 60 days, specially frozen, or individually frozen. The emphasis on chilled and frozen products has raised the question of whether the frozen-and-thawed products can be distinguished from the chilled products by objective methods.

Dr. Kenneth E. Beery of our laboratory has worked on a project under a Natick contract to find an objective method to differentiate between fresh and frozen-and-thawed meats. The results show that the amount of mitochondrial isozyme of glutamic

oxalacetic transaminase in the muscle press juice of an unknown sample can be used as a sensitive index for this determination. Dr. Beery found that the amount of mitochondrial isozyme present significantly increases upon freezing, whereas the amount of sarcoplasmic isozyme remains nearly constant[75]. Based on these findings, Dr. Beery has proposed a test in which the original sample is divided into 2 parts. One of them is held chilled as a control and the other is frozen to -18.7°C for at least 4 hours. Then the frozen portion is thawed, pressed, and the 2 isozymes of glutamic oxalacetic transaminase separated electrophoretically. Following separation, the isozymes are stained and their concentration is determined with a densitometer. The original sample is judged to be fresh if the mitochondrial isozyme activity of the laboratory frozen sample is at least 50 percent higher than the control. If the 2 values are equal, the original sample has previously been frozen and thawed, as refreezing does not cause a further significant increase. The test can be completed within 1 day and has been successfully demonstrated for beef, pork, lamb, chicken, and turkey.

CONCLUSION

In conclusion, I would like to emphasize that it is the user's subjective assessment of the quality of food products that finally determines whether they are acceptable. Therefore, despite the considerable research that has been and will continuously be undertaken on the objective methods for quality assessment, their ultimate effectiveness must be judged by their correlation with the subjective assessment, carried out either by a single expert or by a trained panel, or by the statistical evaluation of consumer preference. The grading standards established for the objective evaluation methods should conform to the scientific subjective evaluation. It is unreasonable for industry and scientists to attempt to force their preconceived opinions of quality on consumers.

ACKNOWLEDGMENT

The author is grateful to Dr. Helen H. Palmer and Mr. Kosuke Ijichi for their advice and assistance in preparing this paper and to Dr. John A. Garibaldi and Mr. Eldon L. Pippen for reviewing it.

REFERENCES

1. *Egg Grading Manual*, Agriculture Handbook No. 75, U.S. Department of Agriculture, Agricultural Marketing Service, Washington, D.C., 14-23.

2. S. Bornstein and B. Lipstein, *British Poult. Sci.*, *3*, 127 (1962).

3. R. H. Harms and C. R. Douglas, *Poult. Sci.*, *39*, 75 (1961).

4. R. H. Harms, W. B. Lester, and P. W. Waldroup, *Poult. Sci.*, *41*, 578 (1962).

5. R. G. Wells, *Egg Quality*, T. C. Carter, Ed., 207 (Oliver and Boyd, Edinburgh, 1968).

6. *Recommended Methods for the Analysis of Eggs and Poultry Meat*, An annotated bibliography, R. Dam, G. W. Froning, and J. H. Skala, Eds., North Central Regional Research Publication No. 205 (1970).

7. F. R. Frank, M. H. Swanson, and R. E. Burger, *Poult. Sci.*, *43*, 1228 (1964).

8. C. Tyler and F. H. Geake, *British Poult. Sci.*, *4*, 49 (1963).

9. J. F. Richard and M. H. Swanson, *Poult. Sci.*, *44*, 1555 (1969).

10. C. Tyler and F. H. Geake, *British Poult. Sci.*, *5*, 37 (1964).

11. S. J. Sluka, E. L. Besch, and A. H. Smith, *Poult. Sci.*, *44*, 1494 (1965).

12. R. G. Wells, *British Poult. Sci.*, *8*, 131 (1967).

13. P. Schoorl and H. Y. Boersma, *12th World's Poult. Congress Proceedings*, 432, Sydney (1962).

14. H. S. Paul, D. C. Snetsinger, M. H. Swanson, L. H. Neagle, and L. G. Blaylock, *Poult. Sci.*, *45*, 1114 (1966).

15. C. Tyler, *British Poult. Sci.*, *2*, 3 (1961).

16. C. Tyler and F. H. Geake, *J. Sci. Food Agric.*, *4*, 587 (1953).

17. C. Tyler and F. H. Geake, *J. Sci. Food Agric.*, *9*, 473 (1958).

18. C. Tyler and F. H. Geake, *J. Sci. Food Agric.*, *12*, 281 (1961).

19. V. S. Asmundson and G. A. Baker, *Poult. Sci.*, *19*, 227 (1940).

20. N. Olsson, *Studies on Specific Gravities of Hens' Eggs. A New Method of Determining the Percentage of Shell in Hens' Eggs* (Leipzig, Otto Harrassowitz).

21. P. E. James and H. J. Retzer, *Poult. Sci.*, *46*, 1200 (1967).

22. A. W. Brant, A. W. Otte, and K. H. Norris, *Food Technol.*, *5*, 356 (1951).

23. R. L. Wesley and W. J. Stadelman, *Poult. Sci.*, *38*, 474 (1959).

24. P. F. Sharp and C. K. Powell, *Ind. Eng. Chem.*, *22*, 909 (1930).

25. J. Wolk, E. H. McNally, and A. W. Brant, *Poult. Sci.*, *31*, 586 (1952).

26. D. Fromm and G. Matrone, *Poult. Sci.*, *41*, 1516 (1962).

27. C. H. Parsons and L. D. Mink, *U.S. Egg Poult. Mag.*, *43*, 484-491 and 509-512 (1937).

28. L. A. Wilhelm, *U.S. Egg Poult. Mag.*, *45*, 565-573, 588-594, 619-624, 657-679, and 587-696 (1939).

29. F. W. Lorenz and H. J. Almquist, *U.S. Egg Poult. Mag.*, *40*, 30 (1934).

30. H. S. Wilgus and A. Van Wagenen, *Poult. Sci.*, *15*, 319 (1936).

31. V. Heiman and J. S. Carver, *Poult. Sci.*, *15*, 141 (1936).

32. R. R. Haugh, *U.S. Egg Poult. Mag.*, *43*, 552-555 and 572-573 (1937).

33. J. V. Atanasoff and H. L. Wilcke, *J. Agric. Res.*, *54*, 701 (1937).

34. A. W. Brant, *Food Technol.*, *5*, 384 (1951).

35. D. J. Buckley and W. S. Reid, *Poult. Sci.*, *50*, 1326 (1971).

36. *Intern Federal Specification*, Egg, Shell C-E-00271H (Army-GL) (U.S. Government Printing Office, 1972).

37. *Federal Register*, Vol. 36, No. 104, Pt. II, Department of Agriculture Consumer and Marketing Service, Eggs and Egg Products Inspection. (U.S. Government Printing Office, Washington, D.C. 20420, 1971).

38. *Eggs and Egg Products, Definitions and Standards Under the Federal Food, Drug, and Cosmetic Act*, FDC Regs., Pt. 42 (U.S. Department of Health, Education, and Welfare, FDA, 1966).

39. R. H. Forsythe, *Egg Quality*, T. C. Carter, Ed., 262 (Oliver and Boyd, Edinburgh, 1968).

40. *Official Methods of Analysis of the Association of Official Analytical Chemists*, 11th edition, William Horwitz Ed., 283-293 (Association of Official Analytical Chemists, Washington, D.C. 20044, 1970).

41. F. J. Cahn and A. K. Epstein, Method of Analysis, U.S. Patent 2,065,114 (1936).

42. R. A. Chung and W. J. Stadelman, *British Poult. Sci.*, *6*, 277 (1965).

43. W. P. Jaffe, *British Poult. Sci.*, *5*, 295 (1964).

44. A. L. Romanoff and A. J. Romanoff, *The Avian Egg*, 318 (John Wiley and Sons, Inc., New York, 1949).

45. D. H. Bergquist and F. Wells, *Food Technol.*, *10*, 48 (1956).

46. E. G. Bligh and W. J. Dyer, *Canadian J. Biochem. Physiol.*, *37*, 911 (1959).

47. J. Folch, M. Lees, and G. H. Sloane Stanley, *J. Biol. Chem.*, *226*, 497 (1957).

48. O. J. Kahlenberg, *Food Ind.*, *21*, 467 (1949).

49. R. H. Forsythe, *J. Assoc. Off. Agric. Chem.*, *41*, 274 (1958).

50. L. Cambell, M. Lally, and R. H. Forsythe, *Poult. Proc. and Marketing*, 12 (1960).

51. S. Bornstein and I. Bartov, *Poult. Sci.*, *45*, 2 (1966).

52. G. De Groote, *World's Poult. Sci. J.*, *26*, 435 (1970).

53. *Chemical and Bacteriological Methods for the Examination of Egg Products*. Institute of American Poultry Industry, 67E Madison Street, Chicago, Illinois 60603, 1968.

54. L. R. MacDonnell, H. L. Hanson, R. B. Silva, H. Lineweaver, and R. E. Feeney, *Food Ind.*, *22*, 273 (1950).

55. L. Kline, T. F. Sugihara, M. L. Bean, and K. Ijichi, *Food Technol.*, *19*, 105 (1965).

56. *Recommended Methods for Analysis of Eggs and Poultry Meat*, R. Dam, G. W. Froning, and J. H. Skala, Eds., North Central Regional Research Publication No. 205, 65.

57. H. H. Palmer, K. Ijichi, S. L. Cimino, and H. Roff, *Food Technol.*, *23*, 148 (1969).

58. H. L. Hanson, B. Lowe, and G. F. Stewart, *Poult. Sci.*, *26*, 277 (1947).

59. T. F. Sugihara, K. Ijichi, and L. Kline, *Food Technol.*, *20*, 100 (1966).

60. W. H. Cook, *The Egg Quality*, T. C. Carter, Ed., 109 (Oliver and Boyd, Edinburgh, 1968).

61. C. M. Ann Baker, *The Egg Quality*, T. C. Carter, Ed., 67 (Oliver and Boyd, Edinburgh, 1968).

62. L. R. MacDonnell, R. E. Feeney, H. L. Hanson, A. Campbell, and T. F. Sugihara, *Food Technol.*, *9*, 49 (1955).

63. V. B. Kamat, B. Partrick, R. Yvell, and J. M. Stubbs, *J. Food Technol.*, *9*, 79 (1974).

64. J. R. Schultz and R. H. Forsythe, *Bakers Digest*, 56, (February 1967).

65. C. M. Chang, W. D. Powrie, and O. Fennema, *Can. Inst. Food Sci. Technol.*, *5*, 135 (1972).

66. E. P. Mecchi, M. F. Pool, G. A. Behman, M. Hamachi, and A. A. Klose, *Poult. Sci.*, *35*, 1238 (1956).

67. K. E. Moerck and H. R. Ball, Jr., *J. Food Sci.*, *39*, 876 (1974).

68. M. F. Pool, H. L. Hanson, and A. A. Klose, *Poult. Sci.*, *29*, 347 (1950).

69. M. F. Pool and A. A. Klose, *J. Am. Oil Chem. Soc.*, *28*, 215 (1951).

70. W. W. Marion and R. H. Forsythe, *J. Food Sci.*, *29*, 530 (1965).

71. J. E. Marion, *Poult. Sci.*, *48*, 301 (1969).

72. A. J. Farr and K. N. May, *Poult. Sci.*, *49*, 268 (1970).

73. *Federal Specification, Chicken, Chilled and Frozen (Ready-to-Cook)*, PP-C-248H, April 5, 1973 (U.S. Government Printing Office).

74. *Federal Specification, Turkeys, Chilled and Frozen (Ready-to-Cook)*, PP-T-471K, April 5, 1973 (U.S. Government Printing Office).

75. K. E. Beery, *J. Food Sci.*, (Paper presented at 1974 IFT Meeting, New Orleans, and submitted for publication).

T E C H N I C A L S E S S I O N I I

Chairman,

Harold S. Olcott

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

DETERMINATION OF SEAFOOD QUALITY

Peter A. Lerke

Before we embark upon this subject, we must define our terms. What is seafood quality? Is it the number of patches of skin left on a fillet of sole, the evenness of the cut of a salmon steak, the number of larval worms in a fillet of cod? Or does quality have a connection with public health and such things as the amount of histamine in scombroid fishes, the presence of hepatitis virus, or fecal *E. coli* in shellfish? If that is quality then we do not have a problem, because anyone can measure such defects and compare the results with a predetermined scale of values.

I shall discuss a particular aspect of seafood quality; namely, how to measure the degree to which decomposition of the flesh has occurred. I should qualify this by specifying spoilage at temperatures above freezing, for degradative processes go on even at freezing temperatures but are mostly confined then to development of oxidative rancidity in lipids, and this topic should be well covered at this Symposium. For the purpose of this talk, therefore, I define seafood quality as the stage of decomposition of the nitrogenous substances of the flesh.

In contrast to our taste for beef and most game animals, we usually prefer our seafood in as fresh a state as possible. Consequently, commercial realities coupled with the inordinate susceptibility of seafoods to spoilage have limited the distribution of non-frozen seafoods to areas fairly close to fishing grounds. Certain species of fish, such as sole, are caught close inshore and are therefore delivered to market in fairly good condition. Others, such as halibut, are fished far from port and may be stored in ice over periods of 10 to 14 days before they are brought ashore and either sold on the so-called fresh market or frozen for future distribution. In either type of fishery, at least on the West Coast, combinations of poor handling and delays in distribution frequently cause the fish to reach the consumer in a condition that is somewhat different from that of freshly caught animals. One of the functions of the institutional buyer is to make certain that what he buys is wholesome and will remain so up to the intended time of use.

How to tell whether seafood is good or bad. How to place a number on an unacceptable product so that there will not be room for argument. How to tell how much storage life is left in seemingly good fish. How to ask for seafood that meets requirements. These are questions that we have to face.

These questions are not new, for the first publication on objective evaluation of seafood quality dates back to 1891.[1] Already at that time it must have been apparent that subjective evaluation, although exquisitely sensitive at the hands of experts, has obvious drawbacks when it comes to communicating results.

I do not intend to go into a detailed discussion here of the merits and disadvantages of sensory evaluation. I think we all know that while sensory evaluation has its usefulness, and is, in some instances, irreplaceable, there is a real need for reliable and simple objective tests. Neither do I intend to list every objective test that has ever been proposed, along with its pros and cons. Several good reviews that adequately cover this subject[2,3] have been published.

Rather I shall discuss certain aspects of seafood spoilage and of its measurement, about which I have first-hand knowledge. Much of this is not new but I think it very useful to bring these things into focus from time to time and to evaluate some of the newer developments in objective methods for spoilage determination.

We can safely state that decomposition starts as soon as the organism dies: enzymatic changes begin almost immediately, followed closely by multiplication and spread of bacteria. The metabolic activities of bacteria soon become the predominant in relation to spoilage. Under certain conditions, decomposition eventually proceeds to a stage where it becomes noticeable to the senses and finally so objectionable that the food becomes unfit for consumption. Organoleptically, the most obvious characteristic that accompanies this process is the formation of foul odors.

We may visualize the development of decomposition as proceeding along a line. In the initial section of the line the fish flesh is perfectly acceptable to the senses even though there may be subtle changes in its odor components. Then, there is a short segment in which there may be some doubt about its acceptability, after which the fish begins to exhibit obvious and increasing signs of spoilage. We may call that first section a zone of freshness and the third section a zone of spoilage. In theory, a quality test takes a measure of what is going on in the flesh and gives the result in terms of a number along the line. Thus, we can distinguish between freshness tests and spoilage tests depending on what part of the line is being tested. A freshness test ideally measures the amount of hidden deterioration that has occurred within the freshness zone and should give an idea of how long it will be before the flesh reaches the point of non-acceptability under given conditions of storage.

The value of a good freshness test to an institutional buyer lies, to a large extent, in this predictive ability. Bearing in mind that we are talking about non-frozen seafoods, what can we measure that will have predictive value? We know that the initial deteriorative changes are enzymatic: substances are being broken down and others formed independently of bacterial activity. One of the earliest changes is the breakdown of adenosine triphosphate (ATP) through the diphosphate and monophosphate to inosine monophosphate (IMP), inosine, finally resulting in the accumulation of hypoxanthine and ribose. The latter two products have been used as indices of freshness in various fish.[4,5] I shall not go here into the theoretical pitfalls that may be encountered because of the number and rates of the reactions and side reactions that are involved. Suffice it to say that measurement of hypoxanthine has been shown to be a satisfactory index of freshness for a number of species provided a separate scale is established for different species and that storage conditions are kept constant.

While enzymatic changes predominate soon after death, bacteria are not idle. It is just that initial contamination is usually very low and, under conditions of commercial refrigeration, the lag phase of bacterial growth may be extended. In fact, bacteria can be said to be in lag phase throughout the freshness zone and, as they enter their logarithmic phase of growth, their greatly increased total metabolic activity produces dramatic changes that become detectable organoleptically. The product is then entering its spoilage zone. This continuous increase in bacterial numbers furnishes us another possible index. Unfortunately, the usual methods of counting bacteria take at least 24 hours and, if one wants to know what the condition of the fish will be in 48 hours, such a test would obviously not be suitable because one must decide within a few hours whether to accept or reject the product.

This was the problem facing the Hospital Purchasing Department at the University of California Medical Center in San Francisco a few years ago. Non-frozen fillets of sole or rock cod had to be purchased on Wednesday for serving on Friday, when they would often no longer be acceptable. Moreover, by that time it would be too late to obtain a fresh supply, let alone exchange the fish.

The hospital needed a method for judging the life expectancy, so to speak, of the fish as it was received. We developed such a method based on direct bacterial counts. It consisted simply in scraping the surface of fillets with a microscope slide and spreading the collected material, as a thin film, on another slide. The preparation was then stained and examined under the microscope, and the number of bacterial cells per field determined. After establishment of the necessary correlation between this number and the keeping quality at refrigerator temperature, we were able to predict, on the basis of a 5-minute test, whether the fish would become unacceptable

in 1, 2, or 3 days. This allowed the hospital to decide whether to accept the product or request an immediate exchange. In time, the suppliers came to accept our judgment and even, on occasion, would send us samples prior to shipping the entire lot to the hospital.[6]

It should be kept in mind, however, that this is one specific instance of a successful freshness test performed under a given set of conditions. Any change involving fish species or keeping conditions would require the establishment of new relationships. This would apply to hypoxanthine determinations as well.

While freshness tests attempt to measure hidden changes in an organoleptically fresh product, spoilage tests are aimed at detecting substances that are present around and beyond the grey area of acceptability. Freshness tests, as a rule, are not concerned with products of bacterial metabolism for, at that stage, these substances occur in such low concentrations as to be undetectable by present methods. As bacterial multiplication proceeds, however, some of these products of the interaction between bacterial enzymes and fish substrate increase to such an extent as to become organoleptically detectable. A spoilage test is designed to measure such products. Assuming that the substance being measured is indeed an index of decomposition, a good spoilage test will either confirm the sensory finding that the food is acceptable or give a numerical value to any organoleptically detectable spoilage. In other words, it will give an objective measure of the spoilage detectable by the senses but may also give an indication of how good the material is, provided it is more sensitive than human senses to the substance being measured.

Much of the search for a good spoilage test has been aimed at finding the one universal indicator that would be present in all cases of spoilage. Such a substance has not yet been found and good theoretical reasons for this become apparent when we stop to think that spoilage is primarily the result of the interaction between bacteria and fish flesh or, even perhaps more accurately, the low-molecular-weight nitrogenous extractives of fish flesh.[7] The exact composition of the end products of this interaction depends on the combination of a particular flora with a particular fish substrate.

What are some of the factors that may affect the composition of the bacterial flora? We know that the latter depends on geographical location, season, and species of fish. When the fish is caught and dies, some of the bacteria carried by the animal take part in spoilage but other organisms are usually introduced, first from the boat environment and then from the shore plant. At all stages leading to spoilage, the flora is further affected both qualitatively and quantitatively by temperature and whether ice or mechanical refrigeration is used for cooling. Qualitative changes in the bacterial flora are especially important for it has been shown that only a small proportion of the total bacteria

present on fish are active spoilers, in the sense of being able to produce off odors.[8] Assuming that we are dealing with a single species of fish, one can already appreciate the variety of spoilage patterns and, consequently, of end-products that may result. It is not surprising, therefore, that many a proposed test based on a single substance has not performed consistently.

If we now couple this large potential variability of the bacterial flora to the additional variable of substrate, which itself depends on such factors as species, season, physiological state, and so on, we find it easy to explain why certain tests that work fairly satisfactorily for certain species do not work at all for others: with an almost limitless number of bacterial-substrate combinations we should expect a corresponding variability in the end-products encountered. There again, as in the case of freshness tests, the chances for success are increased if the test is tailored to a specific situation; in other words, the probability of finding a universal test that would apply to all species under all circumstances is small.

Let us consider some examples and use them to illustrate a few additional points. A good, though not the only, example of a successful seafood spoilage test that measures a single chemical compound is that for trimethylamine (TMA). Although TMA in fish flesh can be formed starting from several different substrates, it appears that it results almost exclusively from the reduction of trimethylamine oxide. The latter substance occurs in white-meat, salt-water fishes such as sole and cod. In spite of all the complicating factors enumerated previously, TMA has been found to be an excellent spoilage test for that group of fish. But here too, different scales of values have to be set up for different species and the test will not work with other fish such as dark-meat pelagic species like mackerel or tuna. Aside from giving a good correlation with organoleptic judgment within its area of applicability, the TMA test has the further advantage of being simple and rapid.

Some investigators, wanting to get away from the risky single-product test, adopted the opposite approach; namely, trying to detect a whole array of end-products of decomposition at one time. In this approach, the applicability of the test should be much wider since it would presumably detect decomposition products almost regardless of their chemical nature. A good example of such a test is the measurement of Volatile Reducing Substances (VRS)[9-11] where an alkaline solution of potassium permanganate oxidizes volatile substances carried to it from a sample by a stream of air in a closed recirculating system. In this approach, the test would register any substance that is both volatile and oxidizable by alkaline permanganate. While this greatly enlarges the scope of the test, there may be products of protein decomposition that lack either volatility or oxidizability or both. Nevertheless, because of its very nature the test tends to parallel odor intensity and has shown itself to be useful for a variety of seafoods. Here again, however, an

individual scale of values must be established for each product; such as, for example, canned albacore and canned skipjack. One of the drawbacks of the VRS test, especially to the analytical chemist, stems from the fact that oxidizable volatiles are measured as a group, and one therefore does not know what is being measured.

An objective test, ideally, should be closely linked to sensory assessment since, after all, our organoleptic acceptance of a product is what counts. Objective tests simply assign numbers to our sensory impressions. Because odor is the main characteristic of spoiled seafood, tests like the VRS have been developed to try to quantify the olfactory function. The advent of gas-liquid chromatography provided and excellent opportunity to extend this approach. The technique generally involves collection of volatiles from the foodstuff, separation of the mixture on a column, and quantification of the components. This technique allows eventual identification of the various volatiles, which may lead to the recognition of individual compounds or groups of substances that may be characteristic of certain types of spoilage. This may also lead to the development of a simplified and more specific test. At the present time, volatiles analysis of seafoods is much too cumbersome a technique for routine application and, in spite of its sophistication, it too measures only a fraction of the spectrum of volatile substances present in decomposed seafood.

I have attempted to give you an idea of the complexity of the process of seafood spoilage and to point out some of the reasons for the generally poor performance of objective quality tests. While it would not be realistic to look forward to a universal spoilage test, specific methods designed to measure a particular characteristic in a given seafood could probably be developed; however, this would require rather extensive basic investigations into the bacteriology and biochemistry of the seafood under varying conditions of storage.

REFERENCES

1. Eber, W., "Ein Chemisches Merknal der Fäulnis 2. Fleish-u. *Milchhyg.*, *1*, 118-119 (1891).
2. Farber, L., "Freshness Tests," *Fish as Food*, G. Borgstrom, Ed., *4*, 65-126 (1965).
3. Fields, M. L., B. S. Richmond, and R. E. Baldwin, "Food Quality as Determined by Metabolic By-Products of Microorganisms," *Advan. Food Res.*, *16*, 161-229 (1968).
4. Shewan, J. M., and N. R. Jones, "Chemical Changes in Cod Muscle During Chill Storage and Their Possible Use as Objective Indices of Quality," *J. Sci. Food Agric.*, *8*, 491-498 (1957).

5. Spinelli, J., M. Eklund, and D. Miyauchi, "Measurement of Hypoxanthine in Fish as a Method of Assessing Freshness," *J. Food Sci.*, *29*, 710-714 (1964).

6. Lerke, P., and L. Farber, "Direct Bacterial Count as a Rapid Freshness Test for Fish Fillets," *Appl. Microbiol.*, *17*, 197-201 (1969).

7. Lerke, P., L. Farber, and R. Adams, "Bacteriology of Spoilage of Fish Muscle, IV, Role of Protein," *Appl. Microbiol.*, *15*, 770-776 (1967).

8. Adams, R., L. Farber, and P. Lerke, "Bacteriology of Spoilage of Fish Muscle, II, Incidence of Spoilers During Spoilage," *Appl. Microbiol.*, *12*, 277-279 (1964).

9. Farber, L., and M. Ferro, "Volatile Reducing Substances (VRS) and Volatile Nitrogen Compounds in Relation to Spoilage in Canned Fish," *Food Technol.*, *10*, 303-304 (1956).

10. Farber, L., and P. Lerke, "A Review of the Value of Volatile Reducing Substances for the Chemical Assessment of the Freshness of Fish and Fish Products," *Food Technol.*, *12*, 677-680 (1958).

11. Farber, L., and P. Lerke, "Colorimetric Determination of Volatile Reducing Substances," *J. Food Sci.*, *32*, 616-617 (1968).

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

FISH OIL COMPOSITION

Robert G. Ackman

INTRODUCTION

This subject can be confusing since the terminology is sometimes loosely used for the total lipid in fish or fish muscle as well as for triglyceride oils obtained in bulk by reduction of fish or fish scrap primarily for fish meal production. The distinction may be important or unimportant, depending mostly on the particular circumstances, since in fish the same important fatty acids occur in the cellular lipids and in the depot fats.

POLYUNSATURATED FATTY ACIDS

In land animals the muscle cellular fats have moderately important amounts of polyunsaturated fatty acids, mostly of the ω6* or linoleic series (Figure 1). Less important amounts of the ω3 or linolenic series (Figure 2) may also be found in the cellular lipids of land animals, and both occur in the C_{20} and C_{22} chain lengths. In the depot fats, both visible and invisible, these polyunsaturated fatty acids are poorly represented except for some 18:2ω6 and a little 18:3ω3[1-10]. Meat from wild animals may have more longer-chain (C_{20}, C_{22}) polyunsaturated fatty acids of the ω3 series than domesticated species, possibly with some important consequences for human dietary intake of fatty acids[6-8].

In marine fish oils and lipids, the dominant polyunsaturated fatty acids are those of the ω3 series found chiefly in 2 fatty acids, 20:5ω3 and 22:6ω3 (Table 1). More extensive reviews have been published on this subject[11-13]. Although sweeping generalizations are hazardous, it appears that many fish such as capelin, menhaden, and anchovetta feeding directly on or near the marine

*A shorthand notation based on chain length, number of double bonds, and position of the double bond closest to the terminal methyl group (Figure 1, Figure 2) will be used. It is assumed that all multiple double bonds are methylene-interrupted.

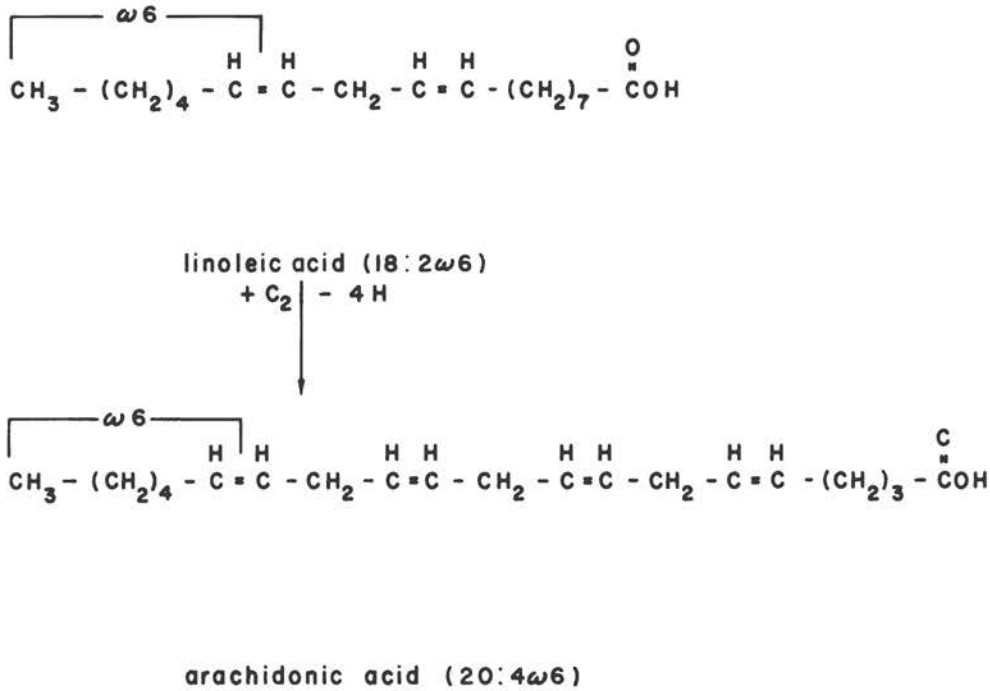


FIGURE 1 Principal fatty acids of the ω6 or linoleic acid family. Linoleic acid is a characteristic acid of terrestrial plants and, together with arachidonic acid, is considered an "essential" fatty acid in terrestrial animals.

phytoplankton food base, where 20:5ω3 is very prevalent among the fatty acids[14], accumulate 20:5ω3 in their depot fat or oil and chain-extend only a portion of this to 22:6ω3. Thus the normal composition for oils of these fish is 22:6ω3 < 20:5ω3. The larger, longer-lived species -- such as cod, mackerel, etc. -- appear to accumulate more 22:6ω3, so that in their oils the rule is 22:6ω3 > 20:5ω3. In blubber of finwhales 22:6ω3 = 2 x 20:5ω3, but in harp seals 22:6ω3 = 20:5ω3[15]. Many species have reasonably consistent oil composition, as illustrated by Table 2 with lipids of the redfish *Sebastes marinus* (or *Sebastes mantella*) and the closely related Norway haddock *Sebastes viviparus*. This is probably because this fish is a relatively long-lived, deep-water species. In this case the 22:6ω3 < 20:5ω3 aspect of the depot fat could reflect some relationship to the chief food items, krill such as *Thysanoessa inermis*[16]. In other commercial oils, such as herring, fatty acid variations due to size of fish, season, sexual maturity, and other factors have been documented[17-20].

Although the weight percent <u>total</u> for polyunsaturated fatty acids (PUFA) in a marine oil can be calculated[21] from the Wijs iodine value by the empirical formula

Percent PUFA = 10.7 + 0.337 (iodine value oil - 100),

it is important to stress that irrespective of their proportions the 2 acids 20:5ω3 and 22:6ω3 usually total over 60 percent of the polyunsaturated fatty acids in all marine oils and lipids of animal origin. The nominal amounts of some other fatty acids are: of 18:2ω6, 1 percent; of 18:3ω3, 0.5 percent; of 18:4ω3, 1 percent; of 20:4ω6, 0.5 percent; and of 22:5ω6, 0.5 percent (see Tables 1-5). It is probably because of the low proportions of ω6 acids, and the competitive effects of the dominant ω3 acids, that fish oils are usually regarded as poor in acids originally defined as "essential" by their dermal effects[22]. The unique chemical properties of the

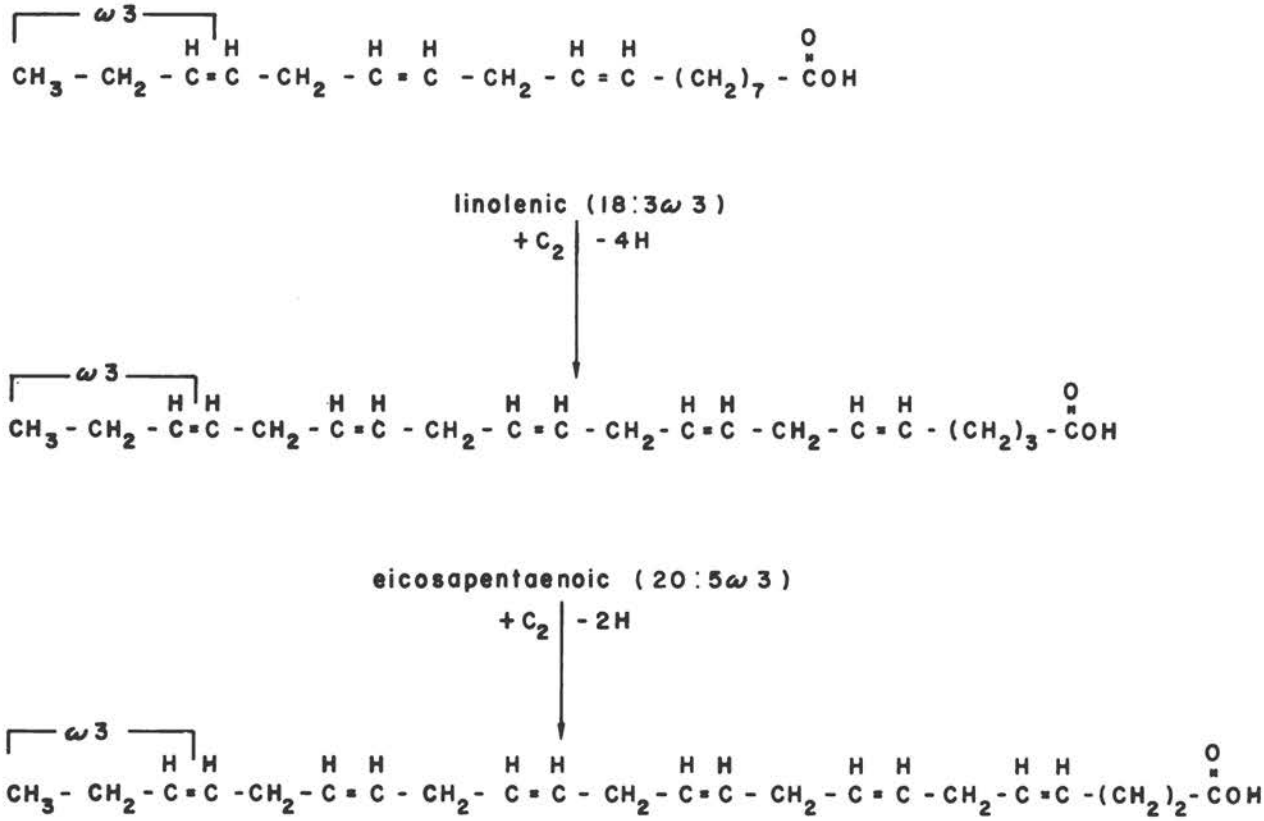


FIGURE 2 Principal fatty acids of the ω3 or linolenic acid family. Linolenic acid is a minor acid in marine animal fats but the successor acids with 5 and 6 ethylenic bonds are 2 of the fatty acids most characteristic of marine animal fats and lipids.

TABLE 1 Weight percentages of some fatty acids of special interest in some large-volume commercial fish oils.

Fatty Acid	Capelin Canada[121]	Herring Norway (?)[122]	Anchovy Peru[122]	Menhaden U.S.A.[122]
14:0	6.2	6.1	7.5	7.3
16:0	9.9	10.8	17.5	19.0
18:0	1.2	1.4	4.0	4.2
16:1	14.3	7.3	9.0	9.1
18:1	15.0	10.3	11.6	13.2
20:1	17.0	13.4	1.6	2.0
22:1	15.8	21.3	1.2	0.6
18:2ω6	0.7	1.0	1.2	1.3
18:3ω3	0.2	2.0	0.8	1.3
18:4ω3	0.7	3.2	3.0	2.8
20:4ω6	0.2	TRA	0.1	0.2
20:5ω3	6.1	7.5	17.0	11.0
22:5ω3	0.6	0.8	1.6	1.9
22:6ω3	3.7	6.8	8.8	9.1
Above Total	91.6	91.9	84.9	83.0

marine oils, and the lipid-based quality problems in fish, can however both be conveniently considered in terms of 1 or both of the 2 acids, 20:5ω3 and 22:6ω3.

The nutritional benefits from these 2 fatty acids are not well-established, although epidemiological evidence of a decreased incidence of multiple sclerosis in areas where fish consumption is high[23] has been published. The etiology of these diseases is seldom simple and latitude, state of sanitary development, and other factors have been considered as well[24]. Some of the areas with low multiple sclerosis incidence and moderate development of sanitary facilities also obviously have a high fish intake (e.g., coastal villages in Norway). Other comparisons, such as a lower incidence in New Orleans, Charleston, and San Francisco than in Winnipeg, Kingston, and Rochester, are less striking from these points of view, but fish consumption could be a factor. The association of these highly unsaturated acids with the human nervous system has led to considerable interest in their role in brain development[25-29]. A demonstration of the direct dietary effect of fish oil on rat brain fatty acid composition[29] has recently been supplemented by the

TABLE 2 Weight percentages of some fatty acids of special interest in redfish triglycerides and one phospholipid. Canadian and Polish redfish samples from *Sebastes (mantella) marinus*. German and Norwegian samples probably from the closely related Norway haddock *Sebastes viviparus*.

Fatty Acid	Nape TG[123] (63 percent fat)	Canadian Muscle TG[123] (1.5 percent fat)	Commercial Oil[124]	Polish Bodyscrap Oil[125]	German Red Muscle[126] Total (7-9 percent fat)	Norwegian Fillet TG[127]	Lipid PL[127]
14:0	5.1	5.6	4.9	6.1	4.3	6.4	2.6
16:0	13.1	11.9	10.4	13.2	13.4	14.3	24.9
18:0	2.3	1.9	2.9	3.0	0.6	1.8	2.5
16:1	13.1	13.8	11.4	10.1	8.3	7.0	3.2
18:1	18.5	16.5	16.6	20.2	22.0	21.5	11.9
20:1	11.7	13.7	15.5	15.3	20.2	10.8	2.7
22:1	15.2	19.9	17.0	14.6	10.4*	9.3	1.3
18:2ω6	0.6	0.6	0.6	1.2	1.8	1.2	1.1
18:3ω3	0.3	0.2	0.2	0.7	0.9	0.8	0.4
18:4ω3	0.8	0.9	1.1	1.6	ND	2.9	1.3
20:4ω6	0.1	0.1	0.3	0.4	--*	0.5	2.2
20:5ω3	9.6	7.4	8.0	4.6	11.2	10.2	13.8
22:5ω3	0.5	0.2	0.6	0.1	0.2	1.0	1.3
22:6ω3	4.5	2.4	4.3	2.7	1.8	6.6	25.6
Above Total	95.4	95.1	93.8	93.8	95.1	94.3	94.8

*Listed as 20:4ω6

TABLE 3 Weight percentages of some fatty acids of special interest in oils and lipids from freshwater fish muscle.

Fatty Acid	Catfish		Trout			Eel[56]	Carp[129]
	Min.[48]	Max.[48]	Commercial[128]	Experimental TG[45]	Experimental PL[45]		
14:0	1.0	2.3	2.7	3.7	2.6	5.9	3.1
16:0	15.2	22.2	20.9	23.5	28.3	14.0	16.8
18:0	3.9	9.3	8.3	3.2	6.2	1.4	4.3
16:1	2.9	5.6	3.9	9.5	9.8	12.4	17.1
18:1	29.7	49.7	18.4	31.0	21.6	27.8	28.3
20:1	0.9	2.0	ND	2.8	2.2	28.3	3.9
22:1	ND	ND	ND			TRA	ND
18:2ω6	10.0	15.7	7.3	15.7	18.3	1.0	13.2
18:3ω3	0.5	2.9	1.6	1.6	1.4	TRA	2.3
18:4ω3	0.4	1.0	3.2	ND	ND	ND	ND
20:4ω6	0.8	5.5	1.7	ND	1.8	0.5	2.5
20:5ω3	0.2	2.5	5.8	1.3	ND	0.8	3.2
22:5ω3	0.2	1.3	ND	0.4	1.1	0.7	ND
22:6ω3	0.6	6.1	7.0	1.3	5.6	0.5	ND
Above Total	--	--	80.0	94.0	98.9	93.3	94.7

TABLE 4 Weight percentages of some fatty acids of special interest in total or specific lipids of crustacea and mollusca meats.

Fatty Acid	Crab		Shrimp	Oyster		Ocean Quahaug	
	King[130]	Queen[58]	Alaska[131]	American[132]	European[132]	TG[133]	PL[133]
14:0	1.4	0.5	2.5	3.5	8.9	3.9	1.1
16:0	9.2	14.0	16.0	28.9	33.8	23.6	19.6
18:0	4.3	2.4	2.6	3.6	10.1	5.0	9.0
16:1	5.0	5.9	5.8	4.2	6.0	7.8	3.1
18:1	15.0	21.7	19.0	8.2	7.3	11.7	7.5
20:1	3.5	2.6	2.4	5.0	3.3	5.3	2.8
22:1	3.9	0.6	1.6	0.3	1.0	0.1	ND
18:2ω6	3.2	0.7	1.5	2.0	0.8	0.7	0.3
18:3ω3	3.3	0.2	1.4	3.3	3.8	0.2	2.2
18:4ω3	2.3	0.1	1.0	2.6	1.0	1.7	ND
20:4ω6	0.6	3.9	0.4	2.3	0.7	0.7	1.1
20:5ω3	21.5	30.5	22.0	11.2	3.4	20.1	13.8
22:5ω3	1.4	0.9	1.2	0.3	0.1	0.4	ND
22:6ω3	10.2	13.3	16.0	9.7	1.3	5.1	9.7
Above Total	84.8	97.3	93.4	85.1	81.5	92.6	70.2

TABLE 5 Weight percentages of some fatty acids of special interest in phospholipids of fish muscle.

Fatty Acid	Mackerel Canada[20]	Cod U.K.[134]	Herring Canada[19]	Halibut U.K.[134]	Rockfish U.S.A.[135]
14:0	0.5	0.5	1.8	0.4	0.5
16:0	20.4	20.6	21.4	17.2	20.0
18:0	7.4	4.2	3.2	6.7	5.5
16:1	1.8	1.6	4.6	1.5	2.4
18:1	9.2	10.1	13.0	7.1	8.9
20:1	1.6	1.5	2.4	ND	0.6
22:1	1.0	TRA	1.0	1.0	ND
18:2ω6	1.6	0.8	0.9	TRA	0.6
18:3ω3	0.5	TRA	0.3	ND	0.2
18:4ω3	0.2	0.2	0.2	ND	ND
20:4ω6	1.7	2.9	1.4	3.4	ND
20:5ω3	10.7	14.6	12.2	8.1	9.1
22:5ω3	1.6	1.2	0.8	TRA	ND
22:6ω3	36.3	35.4	32.7	45.2	47.0
Above Total	94.5	93.6	96.5	99.6	94.8

discovery with rats that trace amounts of 22:6ω3 in free fatty acid form fed along with large amounts of neutral fats have an extraordinarily important impact of this type[30]. Thus the formation of free fatty acids by lipid hydrolysis during iced or frozen storage of fish[31] may promote absorption of 22:6ω3 in free acid form. This acid often occurs in the 2-positions of triglycerides and phospholipids of fish lipids[32]. This means that when bound to glycerol during transfer across the intestinal wall, 22:6ω3 would tend to stay bound and could be resynthesized immediately into a relatively inactive triglyceride instead of being distributed in free fatty acid form bound to serum albumin.

As the developing rat brain receives preference in the utilization of limited amounts of precursor 18:3ω3[33], it is also possible that traces of 20:5ω3 or 22:6ω3 such as may have originally been received from maternal milk would have been selectively mobilized into the developing mammalian brain. Fish lipids may thus have been of only indirect benefit to development of a normal

human central nervous system. However, this role for 22:6ω3 in a fish as a food must be balanced against other factors such as the need for an adequate dietary intake of antioxidants (see below) as well as the loss of this type of highly unsaturated fatty acid by oxidation *in situ* in stored fish[34-40]. The amount of 22:6ω3 in many fisheries products has been tabulated elsewhere[41-43].

Superficially, the familiar marine fatty acid patterns are less applicable to cultivated fish, particulary catfish reared in large quantities in the United States, and the cultivated carp popular in Asia and Europe. These fish are usually fed diets which contain plant materials, and inexpensive vegetable oils or oilseed cake may be included. Commonly, these diets are much higher in 18:2ω6 than in 18:3ω3 and, as a result, 18:2ω6 may be as high as 10 percent of fish fatty acids and its successor acid, 20:4ω6, is also elevated compared to marine lipids (Table 3). Much depends on the composition of the diet[44-50] which may include some natural food[51-53] as well as environmental factors[54-56]. Nevertheless, reliable analyses of the flesh of freshwater fish, including trout, shows that 18:3ω3 is normally converted to 20:5ω3 and 22:6ω3[44-46]. On the other hand, 18:2ω6 may accumulate and also is in fact converted to 20:4ω6 (as well as 22:4ω6, 22:5ω6, etc., in less important amounts). The acids of the ω6 family are basically important to a balanced diet, but from a spoilage point of view the 20:5ω3 and 22:6ω3 are still likely to be the most important fatty acids, with some modifications in flavor arising from 18:2ω6 and 20:4ω6. Local flavor problems of mineral or biological origin should not be confused with those arising from fatty acids or related materials[57].

The crustacea and shellfish (Table 4) show lipid compositional fatty acid patterns of the same fatty acids listed for fish. The differences are mostly that monoethylenic acids are less important than in fish and that 20:5ω3 is usually dominant over 22:6ω3. For practical purposes, the lipid levels of edible muscle meat from shellfish and crustacea is almost universally less than 2 percent wet weight, and thus is so low that the actual original fatty acids are of little concern relative to either nutrition or spoilage.

The polyunsaturated fatty acids are relatively very important (20:5ω3 and 22:6ω3 alone usually total 50 percent or more) in the phospholipids of marine fish of commercial interest (Table 5). An even more basic relationship is involved, since total phospholipids in fresh fish and crustacean muscle are dominated by phosphatidyl ethanolamine (cephalin) and phosphatidyl choline (lecithin) in the ratio of 1:2 in crustacea[58] or 1:3 in fish[59]. These lipids are basic cellular lipids of muscle[10] and important in texture changes and related quality factors in some species (see below). Quantitatively, the phospholipids that occur universally at 0.5 percent of white muscle and about twice that level in dark muscle have only a minor role to play in diet and nutrition in man. However, the interaction of oxidized polyunsaturated fatty acids and proteins is a complex and economically important issue in fisheries technology[34-40] and possibly in human health[9].

MONOUNSATURATED FATTY ACIDS

As Tables 1-5 indicate, 4 monoethylenic fatty acids may make up half the fatty acids of marine oils, but in crustacea or shellfish, and in fish phospholipids, only 16:1 and 18:1 are significant. All 4 acids occur as mixtures of isomers, although the dominant isomers are 16:1ω7, 18:1ω9, 20:1ω9, and 22:1ω11[15,18,20,44,58]. Monoethylenic acids are relatively stable to antioxidation and are readily digested and absorbed in animals[60,61]. They are presumably readily metabolized in man, but the 22:1 acid in particular, and less obviously 20:1, have been shown to participate in cardiac lesion development in juvenile (weanling) male rats and some other experimental animals under special conditions of constant dietary intake[62-64]. As yet, this observation has not been demonstrated to have any significance in normal human nutrition. A detailed bibliography of relevant publications is given in Appendix A.

SATURATED FATTY ACIDS

In Tables 1-5 the totals for the saturated fatty acids are 20-25 percent of 100 percent. Many other saturated fatty acids exist in these oils and lipids[65]. Traces of 12:0 and 20:0, but little or no 22:0, are usually observed in addition to the 14:0, 16:0, and 18:0 reported in these tables[17-20]. In addition to those acids shown, the depot fats contain normal odd-chain acids (mostly 0.5-1.0 percent each of 15:0 and 17:0), and iso and anteiso C_{15} and C_{17} acids totaling about the same percentage as the normal isomers. All these acids are common in the fats of higher animals[66,67]. Odd-chain fatty acids appear to be harmless and possibly metabolically useful in some circumstances[68,69] and the odd-chain analogues of even-chain polyunsaturated acids, which occur in marine lipids, have partial essential fatty-acid activity depending on structure[70].

The isoprenoid methyl-branched fatty acids total 1 to 5 percent of oil fatty acids[71], usually being less in polar lipids. They are harmless except to victims of the very rare Refsum's syndrome[72]. They also occur in terrestrial animal fats, especially ruminant fats, at about the same level as in marine oils[66].

FAT DISTRIBUTION IN FISH

To understand both the nutritional potential of fishery products and the problems connected with their stability, distribution, and preparation, one needs to appreciate that fish muscle is of 2 types: the basic "white" muscle and the lateral-line, "dark", "flank", or "red" muscle ("FL" in Figures 3 and 4). The dark muscle is almost always richer in lipids, including polar lipids, than is the white muscle[20,73,74], reflecting different

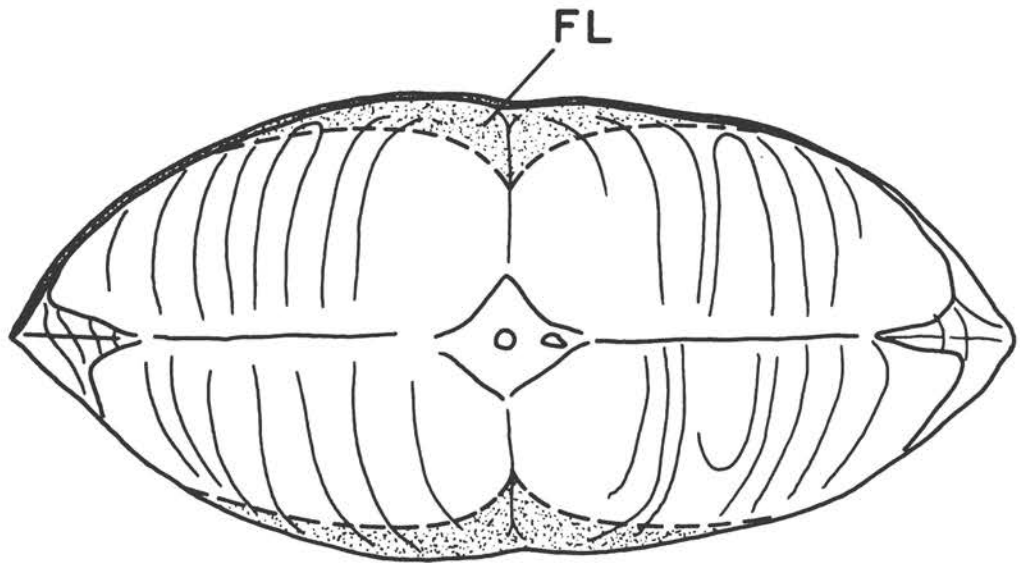


FIGURE 3 Section through back part of body of typical fish showing position of flank (FL) muscle, otherwise known as red, dark, or lateral-line muscle, outside the principal or white muscle.

biochemistries[75]. The "oil" or depot-fat composition in terms of fatty acids tends to be fairly consistant throughout the fish body[74,76], although the distribution throughout the body is not necessarily the same even in fish of superficially similar morphologies[77,78]. The belly flaps ("BF" in Figure 4) may be much fatter than other muscle, 30 percent fat not being uncommon[20]. For this reason and due to the activity of adjacent visceral enzymes, this is often a starting point for post-mortem spoilage problems. In the redfish (Table 2) a dorsal fat deposit at the back or nape of the head is avoided in filleting for similar reasons.

MINOR LIPID COMPONENTS

1. Vitamins. The significance of these types of materials has fluctuated wildly over the years. Vitamins A and D and fish oils are no longer synonymous except to nutritionists in poultry and similar industries. Vitamin E, mostly α-tocopherol in marine products[79], is, on the other hand, of very considerable interest at this time. Fresh fishery products contain reasonable amounts of α-tocopherol, nominally 200 to 300 μg/g oil[79-83]. Other antioxidant materials[84], including ubiquinones[83], are present. The slow or rapid disappearance of the α-tocopherol in the lipids of

frozen sole has been shown to be linked to seasonal storage problems in frozen sole[13,31] and it may be supposed that this is a common phenomenon in frozen fish products stored for long periods[81]. However, little attention has been paid to the actual role of α-tocopherol. The other aspect of the α-tocopherol content of fishery products is that *in vivo* in experimental animals, and presumably in man, any intake of polyunsaturated fatty acids must be accompanied by a sufficient intake of tocopherols, preferably the α-form[85-88], to prevent the appearance of toxic symptoms due to peroxide formation *in vivo*. The vegetable oils added to many fishery convenience products (Table 6) provide additional tocopherols, and North Americans are probably adequately supplied with tocopherols[89]. The range of 0.5 to 0.6 for mg α-tocopherol ÷ g PUFA is suggested as desirable[88] and many fishery products achieve or approach this figure despite a high PUFA content (Table 6).

2. Peroxides and Related Factors. It follows from the polyunsaturated acid content that there will usually be hydroperoxides and sundry degradation products such as aldehydes in

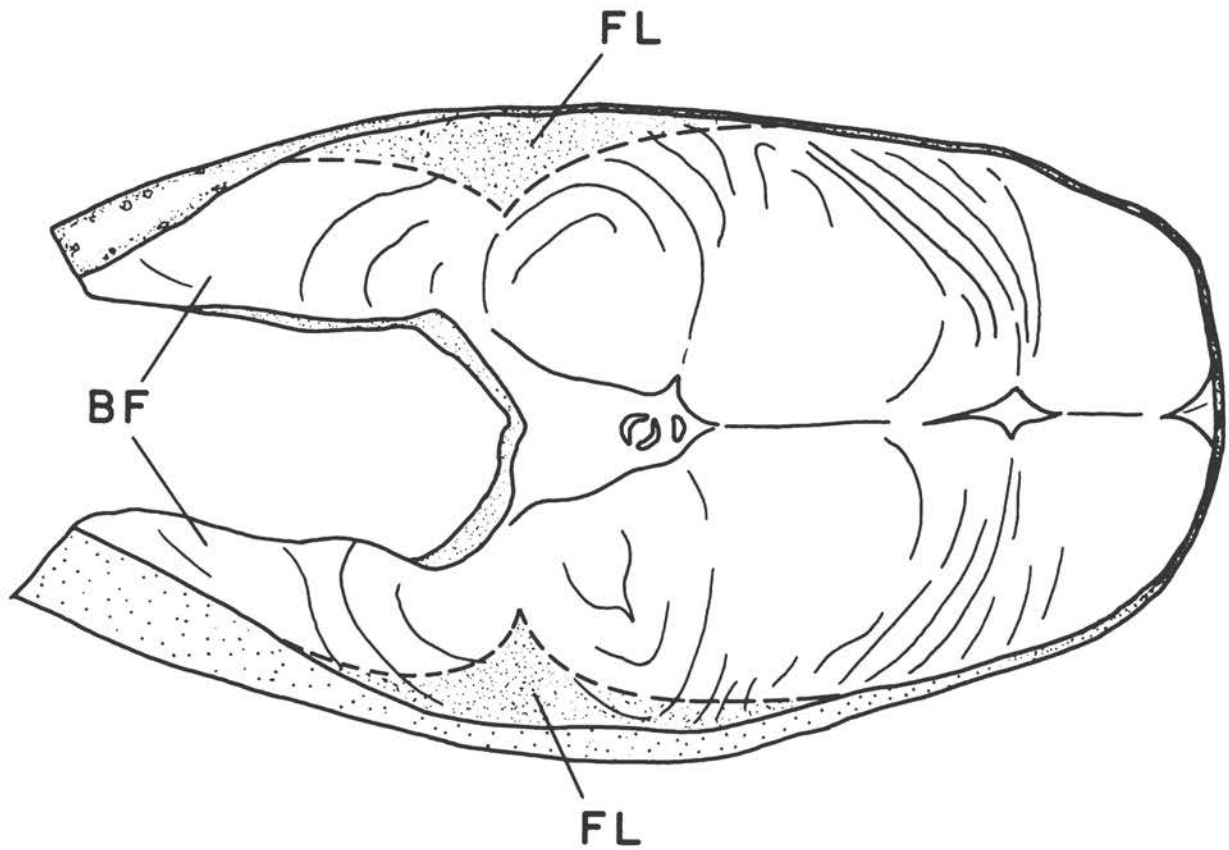


FIGURE 4 Section through front part of body of a typical fish showing positions of flank (FL) muscle and of belly flaps (BF) on each side of visceral cavity.

TABLE 6 Lipid details for some "convenience" fishery products as purchased[128].

		Data per 100 g sample			
		g Lipid (Bligh & Dyer)	g Polyunsaturated acid (by Lipoxidase)	α-tocopherol (mg)	mg α-tocopherol ÷ g polyunsaturated acid
Cod fillet		0.7	*	0.42	1.4†
Cod sticks (breaded)		8.3	0.4	2.10	5.3
Haddock Krisps (breaded)		17.6	8.8	3.40	0.4
Boston Bluefish (breaded)		7.4	1.2	3.60	3.0
Ocean Perch (breaded)		8.3	3.2	*	--
Scallops (breaded)		6.3	0.7	3.60	5.2
Shrimp (breaded)		1.1	0.3	0.45	1.5
Salmon, Pacific, canned					
Keta††	Meat	6.5	2.0	0.22	0.1
	Liquor	6.1	1.2	0.19	0.2
Pink††	Meat	6.1	1.6	0.38	0.2
	Liquor	3.0	0.6	0.16	0.3
Sockeye**	Meat	11.6	2.2	0.14	0.1
	Liquor	18.7	3.9	1.50	0.4
Coho††	Meat	7.7	1.8	0.42	0.2
	Liquor	5.9	1.2	1.40	1.2
Salmon, Atlantic, fresh		1.3	0.3	0.45	1.5
Tuna, canned					
Albacore**	Meat	8.3	*	3.90	--
	Liquor	73.3	33.9	0.75	0.02
Skipjack††	Meat	6.5	3.1	0.39	0.1
	Liquor	24.5	12.9	7.00	0.5
Yellowfin††	Meat	6.5	3.1	0.27	0.1
	Liquor	25.5	13.7	*	--
Mackerel, canned					
	Meat	7.9	1.9	0.12	0.1
	Liquor	20.2	4.4	0.27	0.1
Sardines, canned**					
	Meat	21.9	3.6	0.14	0.04
	Liquor	94.5	15.4	2.40	0.2
Kipper fillets, can contents		27.7	9.9	3.70	0.4
Lobster, canned (a)		0.8	0.2	0.16	0.8
(b)		1.1	0.2	1.10	5.5

*Not analyzed.
**Imported.
†Estimated.
††Domestic.

trace amounts in marine lipids exposed to air for even brief periods. Aesthetically, these are very objectionable[90], and consumer rejection of rancid fishery products limits consumption to low levels. Furthermore, hydroperoxides themselves are not absorbed readily, so that the early stage of autoxidation also may not be a major problem. Continual excess exposure could, however, be serious[91-96].

3. Hydrocarbons, Alcohols, Wax Esters, etc. These classes of alkyl compounds of C_{12}-C_{20} chain length seldom account for more than a fraction of 1 percent of marine oils or lipids in common edible species[97-99], although occasional higher levels are reported[100,101]. They are not usually objectionable in man at the lower levels[102,103] and, at worst, high levels of wax esters cause only mild digestive symptoms[104]. Glyceryl ethers are similarly relatively inactive in man; in those fish in which they occur they may not be in the parts commonly eaten[105].

4. Cholesterol. This is one of the more widely publicized and little understood aspects of fishery-product utilization. In the first instance, polyunsaturated fatty acids are widely thought to lower serum cholesterol in man. The prudent reader should consider a lengthy critique[106] of a review article before making any conclusions based on these few comments. However, there is broad agreement that control of dietary intake of cholesterol is beneficial[9,42,106-111] and this has led to adverse comments on the inclusion of marine foods in restricted diets.

The tabulation of sterols in Tables 7 through 10 shows that, with a few exceptions, a "serving" of fish, crustacea, or shellfish meat need not contribute more than 50 mg of cholesterol. This 50 mg is about half the figure for ordinary "lean" beef[112], and is comparable to pork and chicken cholesterol contents. This is due in part to the fact that a portion of the cholesterol is cellular in nature and can be associated with protein in the "Crude Cholesterol Protein Index" (Table 11) of Koga[113] with favorable aspects for the use of many fishery products. Thus, there is ample leeway to include fishery products in low-cholesterol diets for variety and promotion of a balanced diet.

CONVENIENCE FOODS AND THE FUTURE

In times when overall world fishery resources are being overexploited, the convenience foods commonly available at present extend the available fish protein in various ways. Table 6 gives analytical data for a few contemporary North American products. As can be seen, the inclusion of absorbed fat or canning oils from frying of breaded products completely alters the dominant fat or lipid characteristics of the basic fishery-product protein matrix. A further extension of the protein resource will take the form of comminuted or minced fish obtained mechanically from whole fish or filleted fish residues[114-116]. New lipid-oriented problems undoubtedly will arise with these products since the mechanical destruction of tissue exposes lipid to accelerated oxidation and

TABLE 7 Cholesterol contents of muscle from edible fish from European waters[136].

Fish	Percent fat*	Cholesterol†		Percent in fat-free dry substance
		Percent in fat	mg/100 g muscle	
Cod	0.30- 0.57	7.0 - 9.3	20.0-40.0	0.11-0.21
Codling	0.21- 0.35	10.0 -10.5	22.0-35.0	0.12-0.17
Haddock	0.29- 0.42	6.5 -11.1	27.2-35.0	0.15-0.21
Pollack	0.43- 0.45	7.2 - 8.0	31.0-36.0	0.15-0.18
White halibut	0.35- 0.60	6.0 - 6.9	24.0-34.0	0.11-0.16
Ling	0.34- 0.57	7.4 - 9.6	29.0-46.0	0.15-0.24
Coalfish††	0.56- 0.83	6.2 - 8.2	46.0-53.0	0.26-0.28
Whitefish	0.46- 0.53	8.7 -10.7	40.0-50.0	0.23-0.29
Sole	0.60- 2.10	2.8 - 8.4	41.5-58.5	0.21-0.29
Catfish	1.05- 4.20	0.8 - 4.6	30.2-56.0	0.18-0.29
Red perch	1.30- 7.70	0.5 - 1.9	21.3-54.4	0.19-0.29
Dogfish	2.10-16.30	0.4 - 1.9	28.4-73.0	0.18-0.45
Herring	10.60-24.20	0.2 - 0.6	53.2-66.4	0.33-0.57
Mackerel	17.90-22.50	0.2	34.0-38.8	0.21-0.24
Black halibut	8.90-17.20	0.24- 0.6	43.0-50.0	0.29-0.37

*Petroleum ether extractable after HCL digestion.
†Digitonide precipitate from this extract.
††Saithe or North American Atlantic pollack.

TABLE 8 Cholesterol contents of muscle from European edible fish or prepared foods[136].

Pack	Percent fat*	Percent in fat	Cholesterol	
			mg/100 g muscle	Percent in fat-free dry substance
Cod in bars	0.53	6.6	34.8	0.22
Fish sticks	0.94	6.1	57.0	0.29
Fish steaks	0.58	8.4	48.5	0.25
Breaded cod	0.98	3.3	32.2	0.11
Salmon steak	0.91	5.8	53.0	0.26
Cod fillet	0.41	10.0	41.2	0.22

*From meat portion only.

TABLE 9 Sterol and fat contents of some North American fishery products. Fish sterol reported in the total sterol listed is considered to be all cholesterol. The sterols of crustacea are also considered to be all cholesterol, but shellfish sterol figures refer to actual cholesterol determined by gas-liquid chromatography[112].

Fishery product	mg/100	Percent fat
Caviar	300	15
Cod, fresh	50	0.3
Cod, dried	[82]	0.7
Flounder, flesh	50	0.8
Haddock, flesh	60	0.1
Halibut, flesh	50	1.2
Herring, flesh	85	11.3
Mackerel, flesh	95	12.2
Salmon (sockeye or red), flesh	[35]	--
Sardines, canned in oil, drained	140	11.1
Trout, flesh	55	11.4
Tuna, canned in oil, drained	65	8.2
Tuna, canned in water, not drained	[63]	0.8
Clam meat	50**	1.6
Oyster meat	50**	2.0
Scallop meat	35†	0.2
Crab meat	100	1.9
Lobster meat	85	1.5
Shrimp, canned, drained	150	1.1

**40 percent of total sterol.
†30 percent of total sterol.

TABLE 10 Cholesterol contents of muscle from edible fish and crustacea and the meats of molluscs (North America).

	Percent sterol in non-saponifiables		Apparent cholesterol content (mg/100 g) calculated on an edible wet weight basis		
	Cholesterol	Total other sterols reported	a	b	c
Haddock	93.1	5.9	90	-	-
Pollock	94.1	-	75	-	-
Salmon	96.1	-	95	-	-
American lobster	99.2	0.8	170	-	-
American lobster, canned	99.5	0.5	-	-	90
Spiny lobster, frozen	100	-	-	-	70
Crayfish, freshwater, fresh	99.2	0.8	-	-	120
Shrimp	95.6	-	200	157	-
Shrimp, fresh	99.0	1.0	-	-	140
Crab	57.4	42.6	140	52-98	-
Alaska King crab, frozen	62.2[d]	37.9[d]	-	-	-
Atlantic Queen crab, frozen	93.8[d]	6.1[d]	-	-	60
Atlantic rock crab, fresh	96	4.0	-	-	90
Atlantic Jonah crab, fresh	97	2.9	-	-	70
Atlantic toad crab, fresh	87.9	12.0	-	-	90
Atlantic green crab, fresh	96.8	3.1	-	-	40
Atlantic red crab, fresh	99.9	0.1	-	-	50
Japanese "Kegani" crab, canned	98.1	1.8	-	-	50
Oyster	41.4	58.6	150	37-58	-
Clam	36.7	63.3	190	82	-
Scallop muscle	25.7	74.3	175	60	-

[a]Data from reference No. 137.
[b]Data from reference No. 138.
[c]Data from reference No. 139.
[d]Data from reference No. 140.

TABLE 11 Cholesterol crude protein index (CCPI) (mg cholesterol in 100 g crude protein) for some fishery products available in Japan[113].

Sample	Percent crude fat	mg cholesterol in 100 g fresh sample	CCPI
Bonito	1.96	5	27
Mackerel	5.23	15	74
Gurnet	1.92	25	152
Sole	0.99	51	242
Albacore (dark muscle)	3.72	53	271
Carp	5.74	72	396
Abalone	0.66	121	792
Oyster	1.71	76	1256
Blue crab	2.22	63	412
Yarika squid body	4.59	345	1937
Sea urchin ovary	3.12	498	5491

enzymatic action[117]. The ultimate solution is to use the nutritional value of fish in the stable form of fish-protein concentrate with essentially no lipid[118,119], and this product must be regarded as merely in an arrested state of development[115]. In the interim, one can still enjoy traditionally preserved fishery products in the knowledge that many lipid-based benefits remain safely preserved[120].

REFERENCES

1. T. P. Hilditch and P. N. Williams, *The Chemical Constitution of Natural Fats*, 4th edition, (Chapman and Hall, London, 1964), 664 pp.
2. R. S. Emery, *Animal Growth Nutr.*, E. S. Hafez and I. A. Dyer, Eds., 236-255 (Lea and Febiger, Philadelphia, 1969).
3. W. W. Christie, D. M. Jenkinson, and J. H. Moore, *J. Sci. Food Agric.*, *23*, 1125-1129 (1972).
4. J. D. Wood and D. Lister, *J. Sci. Food Agric.*, *24*, 1449-1456 (1973).
5. K. F. Gander, *Oléagineux*, *26*, 39-44 (1971).
6. M. A. Crawford, M. M. Gale, and M. H. Woodford, *Biochem. J.*, *115*, 25-27 (1969).
7. M. A. Crawford, M. M. Gale, M. H. Woodford, and N. M. Casped, *Internat. J. Biochem.*, *1*, 295-305 (1970).
8. M. A. Crawford and M. H. Woodford, *Internat. J. Biochem.*, *2*, 493-496 (1971).
9. R. J. Jones, *J. Am. Oil Chem. Soc.*, *51*, 251-254 (1974).
10. Z. Mahrla and J. Zachar, *Comp. Biochem. Physiol.*, *47B*, 493-502 (1974).
11. M. E. Stansby, Ed., *Fish Oils: Their Chemistry, Technology, Stability, Nutritional Properties and Uses* (AVI Publ. Co., Westport, Connecticut, 1967), 440 pp.
12. M. E. Stansby, *World Review of Nutrition and Dietetics*, 46-105 (Karger, Basel/New York, 1969).
13. R. G. Ackman, "Marine Lipids and Fatty Acids in Human Nutrition," *Proceedings, FAO Conf. on Fish Prod.*, Tokyo, 4-11/1973 [Fishing News (Books) Limited, London], in press.
14. R. G. Ackman, C. S. Tocher, and J. McLachlan, *J. Fish. Res. Board Can.*, *25*, 1603-1620 (1968).
15. R. G. Ackman, S. Epstein, and C. A. Eaton, *Comp. Biochem. Physiol.*, *40B*, 683-697 (1971).
16. R. G. Ackman, C. A. Eaton, J. C. Sipos, S. N. Hooper, and J. D. Castell, *J. Fish. Res. Board Can.*, *27*, 513-533 (1970).

17. R. G. Ackman and C. A. Eaton, *J. Fish. Res. Board Can.*, *23*, 991-1006 (1966).

18. R. G. Ackman, C. A. Eaton, and P. J. Ke, *J. Fish. Res. Board Can.*, *24*, 2563-2572 (1967).

19. R. F. Addison, R. G. Ackman, and J. Hingley, *J. Fish. Res. Board Can.*, *26*, 1577-1583 (1969).

20. R. G. Ackman and C. A. Eaton, *Can. Inst. Food Technol. J.*, *4*, 169-174 (1971).

21. R. G. Ackman, *J. Am. Oil Chem. Soc.*, *43*, 385-389 (1966).

22. R. T. Holman, *Progress in the Chemistry of Fats and Other Lipids*, Vol. *IX*, Pt. 5, 607-682 (Pergamon Press, Oxford, 1970).

23. J. Bernsohn and L. M. Stepanides, *Nature*, *215*, 821-823 (1967).

24. M. Alter, *Progress in Multiple Sclerosis, Proceedings, Internat. Symposium 1970*, V. Leibowitz, Ed., 99-131 (Academic Press, New York, 1972).

25. M. A. Crawford and A. J. Sinclair, *J. Nutr.*, *102*, 1315-1322 (1972).

26. U. M. T. Houtsmuller, *Lipids, Malnutrition and the Developing Brain*, K. Elliot and J. Knight, Eds., 213-225 (Assoc. Sci. Publ., Amsterdam, 1972).

27. A. J. Sinclair and M. A. Crawford, *FEBS Letters*, *26*, 127-129 (1972).

28. A. J. Sinclair and M. A. Crawford, *J. Neurochem.*, *19*, 1753-1758 (1972).

29. G. Galli and E. Przegalinski, *Pharmacol. Res. Comm.*, *5*, 239-248 (1973).

30. D. E. Eddy, Ph.D. Thesis, *Dietary Fat Influences on Brain and Liver Fatty Acid Compsition: Importance of Docosohexaenoic Acid (22:6ω3)* (University of Nebraska, 1973), 141 pp.

31. R. G. Ackman, *J. Food Technol. (U.K.)*, *2*, 169-181 (1967).

32. H. Brockerhoff, R. G. Ackman, and R. J. Hoyle, *Archives Biochem. Biophys.*, *100*, 9-12 (1963).

33. C. Alling, A. Bruce, I. Karlsson, O. Sapia, and L. Svennerholm, *J. Nutr.*, *102*, 773-782 (1972).

34. G. Wood and L. Hintz, *J. Assoc. Off. Anal. Chem.*, *54*, 1019-1023 (1971).

35. R. J. Braddock and L. R. Duggan, Jr., *J. Food Sci.*, *37*, 426-429 (1972).

36. T. Shono and M. Toyomizu, *Bull. Jap. Soc. Sci. Fish.*, *37*, 912-918 (1971).

37. T. Shono and M. Toyomizu, *Bull. Jap. Soc. Sci. Fish.*, *39*, 411-416 (1973).

38. T. Shono and M. Toyomizu, *Bull. Jap. Soc. Sci. Fish.*, *39*, 417-421 (1973).

39. K. Takama, K. Zama, and H. Igarishi, *Bull. Jap. Soc. Sci. Fish.*, *38*, 607-612 (1972).

40. C. -S. Wu and M. Toyomizu, *Sci. Bull. Fac. Agric., Kyushu University*, *27*, 165-173 (1973).

41. M. E. Stansby, *J. Am. Diet. Assoc.*, *63*, 625-630 (1973).

42. J. Ohashi (and numerous colleagues), *Jap. J. Clin. Nutr.*, *39*, 72-77 (1971).

43. J. C. Bonnet, V. D. Sidwell, and E. G. Zook, *Mar. Fish. Rev.*, *36*(2), 8-14 (1974).

44. J. D. Castell, D. J. Lee, and R. O. Sinnhuber, *J. Nutr.*, *102*, 93-99 (1972).

45. T. Watanabe, I. Kobyashi, O. Utsue, and C. Ogino, *Bull. Jap. Soc. Sci. Fish.*, *40*, 387-392 (1974).

46. T. C. Yu and R. O. Sinnhuber, *Lipids*, *7*, 450-454 (1972).

47. R. R. Stickney and J. W. Andrews, *J. Nutr.*, *101*, 1703-1710 (1971).

48. R. E. Worthington, T. S. Boggess, Jr., and E. K. Heaton, *J. Fish. Res. Board Can.*, *29*, 113-115 (1972).

49. O. R. Braekkan, G. Lambertsen, and J. Andresen, *Influence of Dietary Fat on the Fatty Acid Pattern of Muscle and Liver Lipids in Rainbow Trout*, *5*, No. 8 (Fiskeridirek. Skrift. Ser. Tek. Undersøk., 1971), 12 pp.

50. R. E. Worthington and R. T. Lovell, *J. Fish. Res. Board Can.*, *30*, 1604-1608 (1973).

51. M. L. Albrecht and B. Breitsprecher, *Z. Fisch. Hilfswiss.*, *17*, 143-146 (1969).

52. T. Farkas, *Ann. Biol. Tihany*, *38*, 143-152 (1971).

53. R. G. Ackman, *Comp. Biochem. Physiol.*, *22*, 907-922 (1967).

54. J. W. Andrews and R. R. Stickney, *Trans. Am. Fish. Soc.*, *101*, 94-99 (1972).

55. H. Ikekawa, M. Matsui, T. Yoshida, and T. Watanabe, *Bull. Jap. Soc. Sci. Fish.*, *38*, 1267-1274 (1972).

56. I. Reichwald and A. Meizies, *Z. Ernahrungswiss.*, *12*, 86-91 (1973).

57. T. S. Boggess, Jr., E. K. Heaton, and A. L. Shewfelt, *J. Food Sci.*, *36*, 969-973 (1971).

58. R. F. Addison, R. G. Ackman, and J. Hingley, *J. Fish. Res. Board Can.*, *29*, 407-411 (1972).

59. R. F. Addison, R. G. Ackman, and J. Hingley, *J. Fish. Res. Board Can.*, *25*, 2083-2090 (1969).

60. L. R. Njaa, O. R. Braekkan, G. Lambertsen, and F. Utne, *Nutr. Metab.*, *13*, 207-221 (1971).

61. J. Bjørnstad and P. J. Hansen, *Meldinger fra SSF*, (2), 36-47 (1973).

62. B. Teige and J. L. Beare-Rogers, *Lipids*, *8*, 584-587 (1973).

63. P. Astorg and G. Rocquelin, *C. R. Acad. Sci. Paris*, *277*, 797-800 (1973).

64. R. G. Ackman, *Lipids*, *9*, (1974) in press.

65. J. L. Iverson, *J. Assoc. Off. Anal. Chem.*, *53*, 1074-1079 (1970).

66. R. G. Ackman and S. N. Hooper, *Can. Inst. Food Sci. and Technol. J.*, *6*, 159-165 (1973).

67. J. Flanzy, *Rev. Franc. Corps Gras*, *19*, 359-364, 540 (1972).

68. I. Gontzea and Z. Barduta, *Annales de la Nutrition et l'Alimentation*, *26*, 209-220 (1972).

69. F. X. Pi-Sunyer, *Diabetes*, *20*, 200-205 (1971).

70. H. Schlenk, *Fed. Proc.*, *31*, 1430-1435 (1972).

71. R. G. Ackman and S. N. Hooper, *Comp. Biochem. Physiol.*, *24*, 549-565 (1968).

72. A. K. Lough, *Progress in the Chemistry of Fats and Other Lipids*, R. T. Holman, Ed., Vol. *XIV*, Pt. 1 (Pergamon Press, Oxford, 1973), 48 pp.

73. E. G. Bligh and M. A. Scott, *J. Fish. Res. Board Can.*, *23*, 1025-1036.

74. T. Ueda, *J. Shimonoseki University of Fisheries*, *20*, 145-161 (1972).

75. C. Wittenberger, *Mar. Biol.*, *16*, 279-280 (1972).

76. M. Kobayashi, J. Fukushima, and S. Noguchi, *J. Home Economics (Japan)*, *24*, 511-515 (1973).

77. A. Flo, N. Hagen, and V. Mohr, *Res. Project B. 01033858*, 16 (Norwegian Research Council for Technical and Natural Sciences, Trondheim, 1972).

78. V. Mohr, N. Müller, and A. Flo, *Fatty Tissues in Fish, Forsknings Prosjekt 38C, Report 1* (Inst. for Tech. Biochem., Norwegian Technical University, Trondheim, 1973), 25 pp.

79. R. G. Ackman and M. G. Cormier, *J. Fish. Res. Board Can.*, *24*, 357-373 (1967).

80. M. Mega, *Reports of the Hokkaido Inst. of Public Health*, No. 15, 93-97 (1965).

81. S. Ikeda and T. Taguchi, *Bull. Jap. Soc. Sci. Fish.*, *32*, 346-351 (1966).

82. K. Sugii and T. Kinumaki, *Bull. Jap. Soc. Sci. Fish.*, *34*, 420-428 (1968).

83. H. Higashi, K. Terada, and T. Nakahira, *Bitamin (Vitamins, Japan)*, *45*, 113-120 (1972).

84. V. I. Chernyshov, *Nauchnyye doklady vysshey shkoly*, *15*, 40-45 (1972).

85. R. B. Alfin-Slater, P. Wells, L. Aftergood, and D. Melnick, *J. Am. Oil Chem. Soc.*, *50*, 479-484 (1973).

86. L. A. Witting, *Progress in the Chemistry of Fats and Other Lipids*, R. T. Holman, Ed., Vol. *IX*, Pt. 4, 517-553 (Pergamon Press, Oxford, 1970).

87. O. S. Privett and R. Cortesi, *Lipids*, *7*, 780-787 (1972).

88. J. G. Bieri and R. P. Evarts, *J. Am. Diet. Assoc.*, *62*, 147-151 (1973).

89. J. H. Thompson, J. L. Beare-Rogers, P. Erdody, and D. C. Smith, *Am. J. Clin. Nutr.*, *26*, 1349-1354 (1973).

90. M. E. Stansby, *J. Am. Oil Chem. Soc.*, *48*, 820-823 (1971).

91. E. G. Perkins, S. M. Vachha, and F. A. Kummerow, *J. Nutr.*, *100*, 725-731 (1970).

92. H. Kaunitz and R. E. Johnson, *Lipids*, *8*, 329-336 (1973).

93. K. Fukuzumi, *Fette. Seifen. Anstrichmittel*, *72*, 853-855 (1970).

94. A. Ogawa and H. Igarishi, *Bull. Fac. Fish. Hokkaido University*, *23*, 191-203 (1973).

95. M. Yoshioka, K. Tachibana, and T. Kaneda, *Yukagaku*, *23*, 327-331 (1974).

96. H. Suzuki, *Tohoku J. Exper. Med.*, *106*, 329-342 (1972).

97. A. Karleskind, *Rev. Franc. Corps Gras*, *14*, 251-258 (1967).

98. J. P. Wolff, *Riv. Ital. Sost. Grasse*, *XLV*, 634-642 (1968).

99. R. G. Ackman, *Lipids*, *6*, 520-522 (1971).

100. G. Lambertsen, 11th World Congress Internat. Soc. Fat Res., Göteborg, Sweden, 18-22/6/72.

101. R. G. Ackman, R. F. Addison, and C. A. Eaton, *Nature*, *220*, 1033-1034 (1968).

102. H. K. Mangold, "Unsichtbare Fette und Lipoide in Lebensmitteln," *Wiss. Veroff. Deut. Ges. Ernaehr.*, *24*, 32-38 (1973).

103. H. K. Mangold, A. Meizies, and I. Reichwald, "Resorption und Exkretion Ungewohnlicher Lipide," abs., Deutschen Gesell. Fettwissenschaft, Berlin, Germany, 1-4/10/73, *Fette. Seifen. Anstrichmittel*, *75*, 633 (1973).

104. Y. Sato and Y. Tsuchiya, *Tohoku J. Agric. Res.*, *21*, 176-182 (1970).

105. R. Hardy and P. R. Mackie, *J. Sci. Food Agric.*, *22*, 382-388 (1971).

106. A. Keys, F. Grande, and J. T. Anderson, *Am. J. Clin. Nutr.*, *27*, 188-212 (1974).

107. W. E. Conner, *J. Am. Diet. Assoc.*, *52*, 202-208 (1968).

108. W. E. Conner, *Atheroscler., Proc. Internat. Symposium, 2nd, 1969*, R. Jones, Ed., 253-261 (Springer, New York, 1970).

109. R. E. Hodges, *J. Am. Diet. Assoc.*, *52*, 198-201 (1968).

110. F. H. Mattson, *Am. J. Clin. Nutr.*, *25*, 589-594 (1972).

111. E. Quintao, S. M. Grundy, and E. H. Ahrens, Jr., *J. Lipid Res.*, *12*, 233-247 (1971).

112. R. M. Feeley, P. E. Criner, and B. K. Watt, *J. Am. Diet. Assoc.*, *61*, 134-149 (1972).

113. Y. Koga, Nihon Shoka Kenkyudai, *Japanese Food Res. Assoc., Studies on Japanese Foods, Report No. 46*, *23*, 412-421 (1970).

114. J. N. Keay and R. Hardy, "Utilization of Fish Resources," *J. Hotel Cat. Manag. Assoc.*, No. 25, 9, 11, 13, 16 (1974).

115. L. Regier, *Food in Canada*, *34*, 36-37 (1974).

116. D. L. Crawford, D. K. Law, J. K. Babbit, and L. S. McGill, *J. Food Sci.*, *37*, 551-553 (1972).

117. H. Morii and R. Kanazu, *Bull. Fac. Fish. Nagaski University*, *33*, 75-80 (1972).

118. H. E. Power, *J. Fish. Res. Board Can.*, *21*, 1489-1504 (1964).

119. V. J. Smith, J. S. Linn, and H. S. Olcott, *Fish. Bull.*, *72*, 845-847 (1974).

120. A. Meizies and I. Reichwald, *Z. Ernahrungswiss.*, *12*, 248-251 (1973).

121. R. G. Ackman, C. A. Eaton, and K. D. Spencer, *J. Fish. Res. Board Can.*, in press (1974).

122. W. Schokker and H. Boerman, private communication.

123. R. G. Ackman, unpublished results.

124. R. G. Ackman and P. J. Ke, *J. Fish. Res. Board Can.*, *25*, 1061-1065 (1968).

125. B. Drozdowski and H. Niewiadomski, *Prace Morskiego Instytutu Rybackiego*, *15B*, 131-140 (1970).

126. W. V. Reimold and N. Lang, *Z. Ernahrungswiss.*, *11*, 69-79 (1972).

127. G. Lambertsen, "Lipids in Fish Fillet and Liver -- A Comparison of Fatty Acid Compositions," *5*, No. 6 (Fiskeridirek. Skrift. Ser. Tek. Undersøk., 1972), 15 pp.

128. E. Varesmaa, J. J. Laine, and F. P. Nhnivaara, *Z. Lebensm.-Unters. Forsch.*, *138*, 150-154 (1968).

129. M. Toyomizu and Y. Tomiyasu, *Bull. Jap. Soc. Sci. Fish.*, *28*, 526-533 (1962).

130. R. A. Krzeczkowski, R. D. Tenney, and C. Kelley, *J. Food Sci.*, *36*, 604-606 (1971).

131. R. A. Krzeczkowski, *J. Am. Oil Chem. Soc.*, *47*, 451-452 (1970).

132. T. Watanabe and R. G. Ackman, *J. Fish. Res. Board Can.*, *31*, 403-409 (1974).

133. R. G. Ackman, S. Epstein, and M. Kelleher, *J. Fish. Res. Board Can.*, *31*, in press (1974).

134. J. Olley and W. R. H. Duncan, *J. Sci. Food Agric.*, *16*, 99-104 (1965).

135. G. Wood, L. Hintz, and H. Salwin, *J. Assoc. Off. Anal. Chem.*, *52*, 904-910 (1969).

136. J. Wurziger and G. Hensel, *Fette. Seifen. Anstrichmittel*, *69*, 937-942 (1967).

137. D. Kritchevsky, S. A. Tepper, N. W. DiTullo, and W. L. Holmes, *J. Food Sci.*, *32*, 64-66 (1967).

138. M. H. Thompson, *Fish. Ind. Res.*, *2*(3), 11-15 (1964).

139. D. R. Idler and P. Wiseman, *Internal. J. Biochem.*, *2*(7), 91-98 (1971).

140. D. R. Idler and P. Wiseman, *Comp. Biochem. Physiol.*, *26*, 1113-1117 (1968).

APPENDIX A

A Select Bibliography of Recent Papers Reporting the Feeding of Marine Oils to Animals With Special Reference to Cardiac Alterations and the Docosenoic Acids Present in These Oils.

J. Raulin, Cl. Richir, and R. Jaquot, "Nutritional and Pathological Repercussions of Food Use of Fish Oil Deodorized by Heating," *Acta Chim. Hung.*, *23*, Annexe VI, 227-233 (1960).

A. Fricker and K. Lang, "Nutritional-Physiological Properties of Red Barsch Oil," III, "The Influence of Long-Term Feeding of Red Barsch Oil on the Blood Lipid Values of Rats," *Zeit. Ernahrungswissenschaft*, *105*, 105-111 (1964).

O. R. Braekkan, C. Lambertsen, F. Utne, and L. R. Njaa, "Hydrogenated Marine Fat, etc.," "Influence on the Fatty Acid Composition of Depot Fats and Liver Lipids in the Rat," *Nutr. Dieta*, *10*, 24-44 (1968).

E. Aaes-Jørgensen and G. Hølmer, I, "Growth and Testes Development," *Lipids*, *4*, 501-506 (1969).

G. Hølmer and E. Aaes-Jørgensen, "Essential Fatty Acid-Deficient Rats," III, "Distribution of Lipid Classes in Rat Testes After Feeding Partially Hydrogenated Oils," *Lipids*, *4*, 515-521 (1969).

D. Miller and P. Robisch, "Comparative Effects of Herring, Menhaden and Safflower Oils on Broiler Tissues Fatty Acid Composition and Flavor," *Poult. Sci.*, *48*, 2146-2157 (1969).

J. L. Beare-Rogers, E. A. Nera, and H. A. Heggtveit, "Cardiac Lipid Changes in Rats Fed Oils Containing Long-Chain Fatty Acids," *Can. Inst. Food Technol. J.*, *4*, 120-124 (1971).

D. Blitek and J. Jeske, "Comparative Studies on the Values of Oils of Norway Haddock and Cod Liver Oil," *Acta Polon. Pharmacol.*, *28*(5), 347-552 (1971).

J. Budzynska-Topolowska, *et al.*, "Incorporation of Trans Fatty Acids into Body Lipids of Rats Digesting Margarine Based on Rapeseed Oil," *Oléagineux*, *26*, 701-706 (1971).

K. Lang and W. V. Riemold, "Changes in the Lipid and Fatty Acid Composition in the Heart Muscle Tissue of the Rat and the Influence of Ocean Perch Oil and Coconut Oil in a Prolonged Feeding Trial," *Z. Ernahrungswiss.*, *10*(4), 325-333 (1971).

S. Molnar, H. Neumann, and U. ter Meulen, "Studies in the Fatty Acid Composition of Lipid Fractions of Liver, Heart and Lung in Pre- and Postnatal Phases of Pigs," *Z. Tierphys. Tiernahr. und Futtermittelkunde*, *27*, 159-177 (1971).

B. L. Walker and M. G. Mackey, "Transfer of Erucic Acid From the Maternal Diet in the Offspring," *Proc. Can. Fed. Biol. Soc.*, Quebec, 1972.

L. R. Njaa, O. R. Braekkan, G. Lambertsen, and F. Utne, "Hydrogenated Marine Fat Effect on Growth and Liver Weight in Young Rats Given a High Fat, Low Protein Ration," *Nutr. Metab.*, *13*, 207-221 (1971).

P. H. Odense and H. Brockerhoff, "Effects of Adaptive and Non-Adaptive Feeding of Hydrogenated and Raw Herring Oil on Hearts and Livers of Weanling Rats," *J. Fish. Res. Board Can.*, *28*, 1793-1795 (1971).

J. L. Beare-Rogers, E. A. Nera, and B. M. Craig, "Accumulation of Cardiac Fatty Acids in Rats Fed Synthesized Oils Containing C_{22} Fatty Acids," *Lipids*, *7*, 46-50 (1972).

J. L. Beare-Rogers, E. A. Nera, and B. M. Craig, "Cardiac Lipids in Rats and Gerbils Fed Oils Containing C_{22} Fatty Acids," *Lipids*, *7*, 548-552 (1972).

K. Flatlandsmo, "Marine Fat," "Digestibility of its Fatty Acids in Young Calves," *Acta vet. scand.*, *13*, 260-262 (1972).

B. Teige and J. L. Beare-Rogers, "Cardiac Fatty Acids in Rats Fed Marine Oils," *Proc. Can. Fed. Biol. Soc. 1972 Meeting*, abs. 233.

W. A. G. Veen, "Animal and Vegetable Fats in Milk Replacer for Veal Calves," 5, "Fatty Acid Pattern in Various Adipase Tissues," *Z. Tierphysiol. Tierernahrung u. Futtermittelkunde*, *30*, 1-19 (1972).

S. Ziemlanski, *et al.*, "Biological Evaluation of the Nutritive Value of Edible Oils."
I. *Polish Med. J.*, *XI*, 1602-1611 (1972) (also *Rocz. Panst. Zak. Hig.*, *XXIII*, No. 4 (1972).
II. *Polish Med. J.*, *XI*, 1612-1624 (1972) (also *Rocz. Panst. Zak. Hig.*, *XXIII*, No. 5 (1972).
III. *Polish Med. J.*, *XI*, 1625-1633 (1972) (also *Rocz. Panst. Zak. Hig.*, *XXIII*, No. 6 (1972).

H. B. S. Conacher, B. D. Page, and J. L. Beare-Rogers, "Monoethylenic Isomers in Cardiac Lipids of Rats Fed Partially Hydrogenated Herring Oil," *Lipids*, *8*, 256-258 (1973).

B. Teige and J. L. Beare-Rogers, "Cardiac Fatty Acids in Rats Fed Marine Oils," *Lipids*, *8*, 584-587 (1973).

P. A. Astorg and G. Rocquelin, "Short-Term Effects of Partially Hydrogenated Herring Oil on the Total Lipids of Heart and Liver of the Weanling Rat," *Comptes Rendus Acad. Sci.*, (D), *227*, 797-800 (1973).

A. M. M. Abdellatif and R. O. Vles, "Short-Term and Long-Term Pathological Effects of Glyceryl Trierucate and of Increasing Levels of Dietary Rapeseed Oil in Rats," *Nutr. Metab.*, *15*, 219-231 (1973).

J. K. G. Kramer, S. Mahadevan, J. R. Hunt, F. D. Sauer, A. H. Corner, and K. M. Charlton, "Growth Rate, Lipid Composition, Metabolism and Myocardial Lesions of Rats Fed Rapeseed Oils (*Brassica campestris* var. Arlo, Echo and Span, and *B. napus* var. Oro)," *J. Nutr.*, *103*, 1696-1708 (1973).

J. Bjørnstad and P. J. Hansen, "Digestibility of Hydrogenated Marine Fat (HMF) in Calf Milk Replacers," *Meldinger fra SSF*, (2), 36-47 (1973).

H. A. Heggtveit, E. A. Nera, and J. L. Beare-Rogers, "Cardiotoxic Effects of Rapeseed Oil: Histological and Biochemical Studies," *Recent. Adv. Stud. Cardiac Struc.*, *12*, 449-454 (1973).

G. Rocquelin, J. -P. Sergiel, P. O. Astorg, and R. Cluzan, "Effects of Rapeseed and Canbra Oils on Cardiac Lipids and Anatomy of the Rat: Short-Term Study," *Ann. Biol. Animal Biochem. Biophys.*, *13*, 587-609 (1973).

J. L. Beare-Rogers and E. A. Nera, "Effects of Dietary Docosenoic Acid Upon Rats in Cold," *Lipids*, *9*, 365-367 (1974).

B. Bulhak-Jachymczyk and E. Hübner-Woźniak, "Effect of Dietary Erucic Acid on the Level of Long-Chain Acylcarnitines in Rat Heart and Liver," *Bull. Acad. Pol. Sci., Ser. Biol. C1. II*, Vol. *XXII*, No. 1, 19-23 (1974).

M. A. Swarttouw, "The Oxidation of Erucic Acid by Rat Heart Mitochondria," *Biochem. Biophys. Acta*, *337*, 13-21 (1974).

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

OBJECTIVE DETERMINATION OF FAT STABILITY IN PREPARED FOODS

D. R. Erickson and R. H. Bowers

An extensive body of literature is available to the researcher interested in the objective determination of fat stability in foods. It is not the intent of this presentation to review this literature but rather to discuss in a general way the complexity of oxidative lipid deterioration and suggest options available to an investigator.

This presentation will be primarily concerned with oxidative deterioration of fats. Fat stability could properly include hydrolytic reactions with subsequent release of free fatty acids. Hydrolytic stability would be of concern only where a food contained fat with short chain fatty acids such as those present in the lauric acid fats or milk fats. The method used to study this objectively would be determination of the free fatty acid content.

Whatever objective method is chosen, it must be realized at the outset that the method must be correlated with some type of organoleptic evaluation of the food in question. There is only one direct method for detection of lipid deterioration in a prepared food; that is, to prepare, package, and store the prepared food under the conditions normal to that food while examining it periodically by organoleptic means.

Examination of a prepared food by organoleptic means is usually not considered to be objective since it relies upon subjective judgment by individuals. Proper training of personnel and use of appropriate statistical sampling and evaluation can result in objectivity approaching that expected from chemical analyses. It may well be that this becomes the only reliable option available to an investigator and should therefore not be automatically rejected as too expensive or unwieldy. As will become apparent during this presentation, lipid oxidation is an extremely complex process. Consequently, more time and effort could be spent on developing and correlating a chemical test than would be expended by relying solely on an organoleptic evaluation.

Rather comprehensive reviews of the mechanisms of lipid oxidation in foods are available in the literature[1,2]. A much oversimplified version of the chemical reactions involved in lipid

oxidation is shown in Figure 1. This points out the classes of possible compounds derived from lipid oxidation and simplifies consideration of the reactions involved.

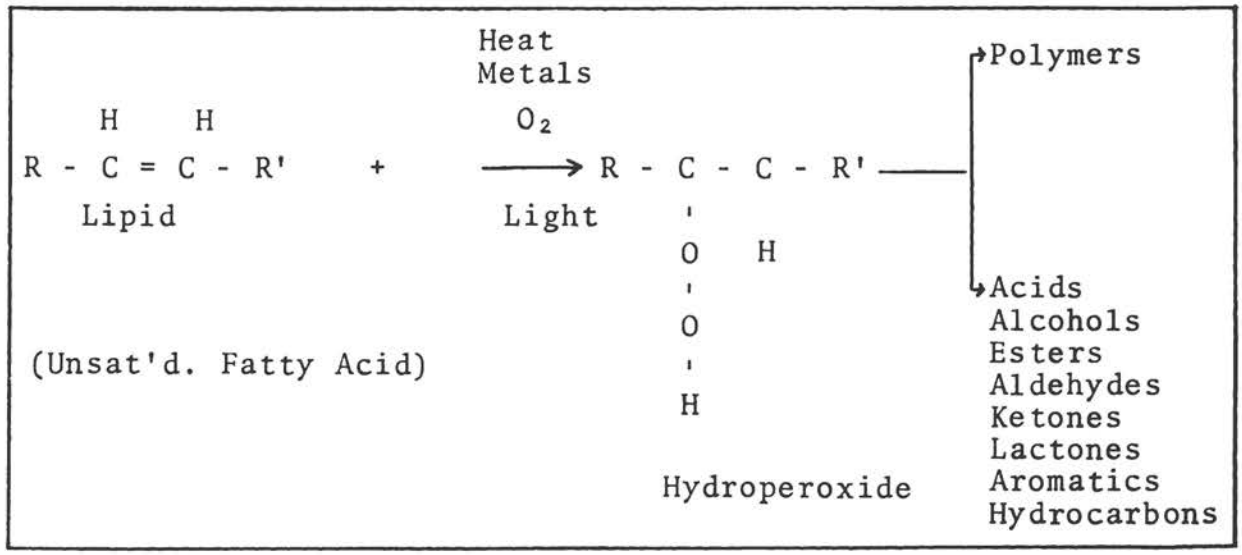


FIGURE 1 Chemical reactions involved in oxidation of lipids.

In terms of the reactants, the lipid portion can be considered with regard to its expected lability to oxidation, which may be deduced if the composition is known. The oxygen absorbed is, and can be, an indication of the progress of lipid oxidation with the reservations to be discussed later. The intermediate formation of lipid peroxides may also be an indicator of the progress of oxidation but such methodology is more appropriate to a pure fat or isolated fat system than a prepared food. Finally, the measurement of one or more of the final reaction products or classes of products resulting from peroxide decomposition may be used as an indication of the progress of oxidation.

What needs to be recognized by all investigators is the very low taste and/or odor thresholds associated with some of the products of peroxide decomposition. This is excellently shown in a review by Forss[3], who has assembled a table of threshold values for aliphatic aldehydes. Selected values from his review are shown in Table 1. Particular attention is directed to those compounds showing less than ppb threshold values. Such low values defy normal analytical technique in terms of detection. To further emphasize the extremely low levels of reactants involved, Labuza[1] has estimated that on a molar basis less than 0.00002 percent of a fat has to be oxidized to produce objectionable quantities of hexanal in oil or milk.

TABLE 1 Selected flavor thresholds (ppm) of aliphatic aldehydes in various media[3].

	Water	Milk	Vegetable oil	Paraffin oil
Propionaldehyde	0.17	0.43	0.2	1.0
Valeraldehyde	0.07	0.13	--	0.15
Hexanal	0.03	0.049	0.3	0.6
Lauraldehyde	0.0009	--	0.9	0.4
trans-2-Nonenal	0.001	0.0042	0.08	0.4
trans-2, cis-6-Nonadienal	0.0001	--	--	0.0015
trans-6-Nonenal	--	--	--	0.00035

These four factors, i.e., lipid composition, oxygen absorption, intermediate peroxide formation, and final products of reaction or peroxide decomposition products, are a summation of the options available to an investigator choosing objective methods for determining fat stability in prepared foods.

Before going into the four factors, it should be emphasized that the oxidation process in a lipid system is a dynamic or continuing series of reactions; therefore, any individual determination is subject to errors related to this dynamism.

LIPID COMPOSITION

The determination of the lipid composition of a prepared food necessitates isolation of that system by extraction. This could be considered an alternative to an objective analytical technique used on an intact food.

In making judgments as to the inherent stability of a lipid system *per se* the major consideration is the nature and proportion of the unsaturated fatty acids present. Generally speaking, the higher the proportion and degree of unsaturation of the fatty acids the more labile the system will be to oxidation.

By isolation of the lipid system, the investigator is working with a much cleaner system and, more importantly, such an isolated system can be analyzed for composition, which by itself can allow the investigator to make a judgment as to its inherent stability.

Both the attributes above are attractive. Unhappily, they may have little to do with the stability of lipid systems in prepared foods. Isolation of the lipid system quite possibly will separate it from important antioxidant or pro-oxidant factors present in the intact food. These factors may be chemical, physical, or a combination of these. For instance, isolation of a lipid system

from a dry or semi-moist food may give a false high stability because of a simple reduction of the surface available to attack by oxygen.

In addition to removal from its real environment leading to possibly false conclusions, the extraction process itself can produce artifacts through use of impure solvents, failure to exclude oxygen during extraction, selective extraction of pro-oxidants or antioxidants along with the lipid, etc.

Finally, the completeness of extraction is always in question and it could well be that the most labile lipid, such as that associated with phospholipids and/or lipoprotein complexes, may not be extracted, leading again to false conclusions.

There are thus several pitfalls in the technique of isolating a lipid system, studying its stability, and then using these results to predict stability of the food from which it was taken.

If such an approach is taken, then the usual fat stability tests can be run. These tests will be covered by Dr. Thomas in a following presentation.

MEASUREMENT OF OXYGEN UPTAKE

In Figure 1, it was shown that oxygen is a principal reactant in lipid oxidation; hence, measurement of its uptake could be considered especially attractive as an objective method.

At this point, it is necessary to reflect back upon what was said earlier about the very low threshold values of off-flavor compounds resulting from lipid oxidation. It is apparent that such compounds could well be formed without any measurable oxygen uptake by the system. Because of this, oxygen uptake is most useful when used with an accelerated method.

To understand the application of this technique, it is helpful to consider how oxygen might be absorbed by a prepared food. Figure 2 shows two typical examples of what could occur. It should be noted that the curves both exhibit an induction period followed by an accelerated linear uptake.

In Curve A, one could consider use of the induction period as an end point since there is apparently no meaningful or measurable uptake of oxygen during this time. On the other hand, Curve B shows an apparent measurable degree of oxygen absorption during the induction period and use of length of induction period becomes of more dubious value.

All other things being equal, a negligible slope of the oxygen absorption curve during the induction period directs one toward length of this period as a measurement. Any significant or measurable slope in this portion of the curve requires use of the time until an arbitrary amount of oxygen is absorbed, instead of the time during which the rate of absorption is negligible. Likewise the measurement of time until an arbitrary amount of oxygen is absorbed would be the only choice when there is no induction period.

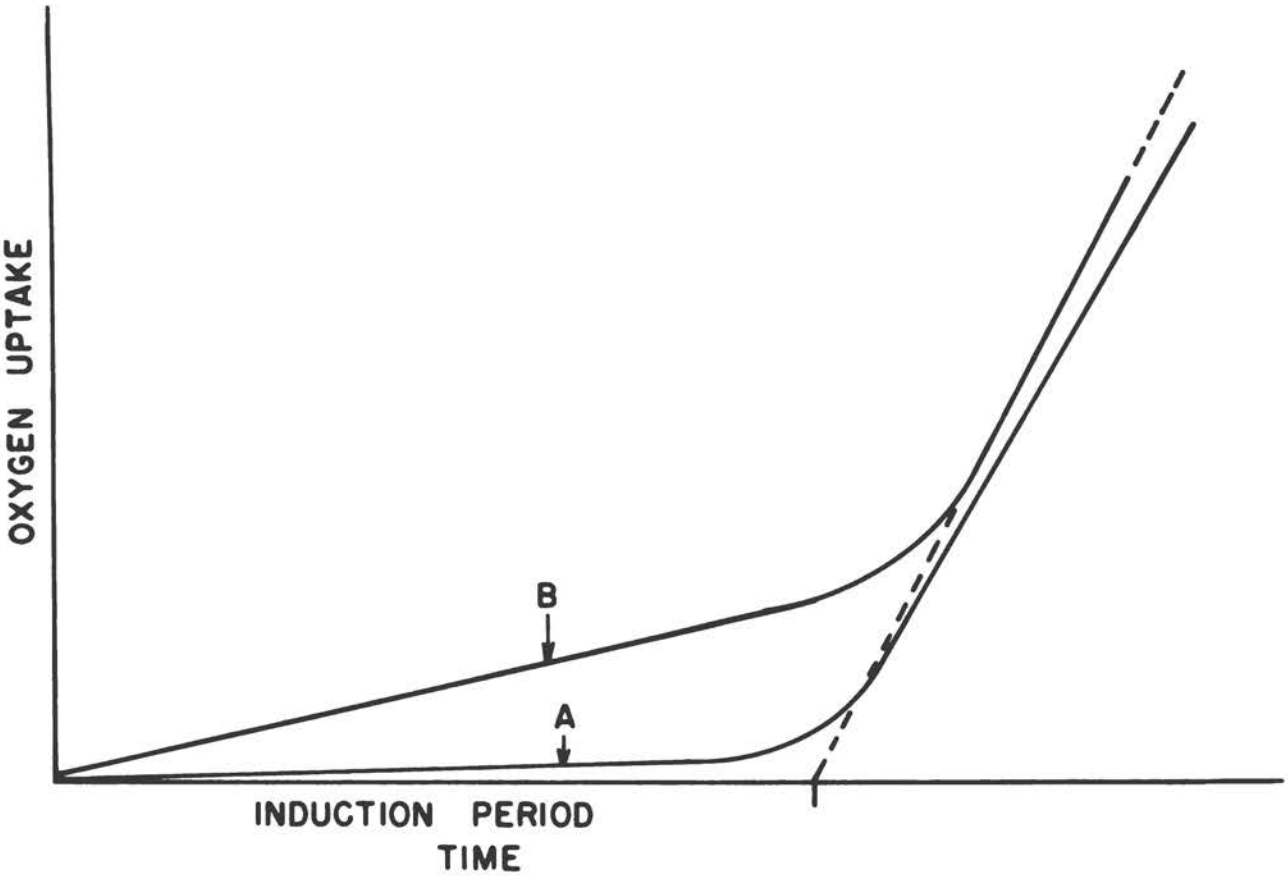


FIGURE 2 Oxygen uptake during lipid oxidation. Curve A -- no measurable oxygen uptake during induction period. Curve B -- measurable oxygen uptake during induction period.

It is thus necessary to develop an absorption curve for any product under investigation, allowing selection of a measurement which can be meaningfully applied. Even this information provides only a starting point, since any value chosen must be correlated with an appropriate organoleptic score.

One final consideration of oxygen uptake is the caution that non-lipid constituents may also absorb oxygen. This might be especially true for prepared foods with a very low lipid content.

In summary, oxygen uptake is an attractive procedure since it is measuring one of the principal reactants and can be applied to intact foods. Some food systems in which oxygen absorption has been used are meats, processed meats, peanut butter, bakery products, and edible oil products[4].

PEROXIDE FORMATION

Determination of peroxide formation is of very limited value in most foods other than a pure fat system. It can, of course, be applied to fat extracted from a food but the shortcomings of extraction have already been discussed.

Peroxide development in a lipid system usually follows the curve shown in Figure 3. Up to the point where peroxide formation falls off, it is similar to the curve shown earlier for oxygen absorption. This is not surprising since peroxide formation is a direct result of oxygen absorption.

The difference is that peroxides represent an intermediate and the amount present at any time is a function of the rate of peroxide decomposition. If the peroxides were decomposed at the same rate as formed, one could get significant development of off-flavor during the apparent induction period. Alternatively, if

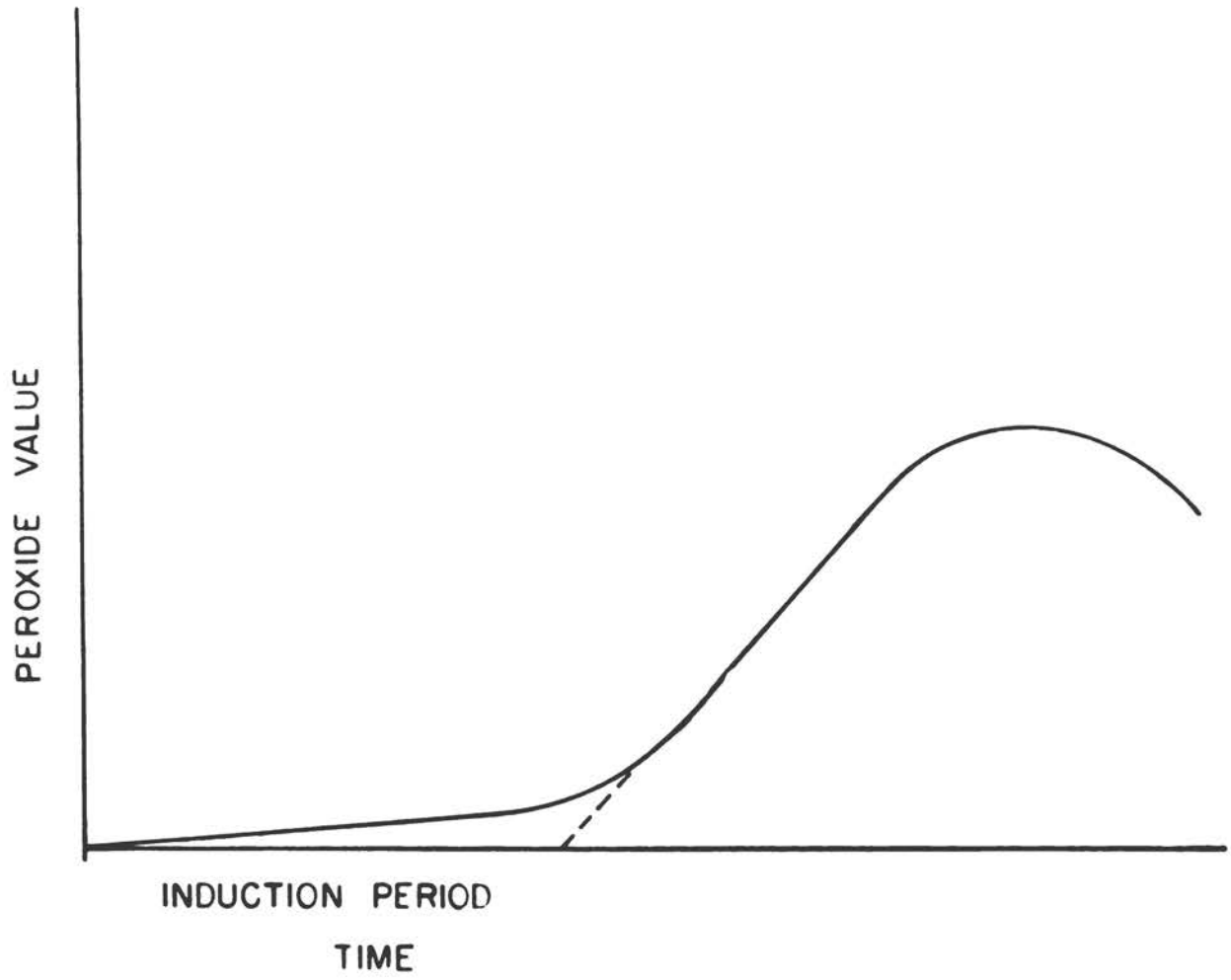


FIGURE 3 Peroxide development during lipid oxidation.

peroxide decomposition were retarded, peroxides could theoretically accumulate beyond the induction period without affecting flavor since peroxides *per se* are essentially flavorless.

PEROXIDE DECOMPOSITION PRODUCTS AND/OR FINAL REACTION PRODUCTS

To this point we have discussed measurement of all the reactants involved in lipid oxidation except those that are finally responsible for the off-flavors. It would seem that analysis of these compounds would be the ultimate in objective measurement since they are the end result of the oxidation of lipids.

Unfortunately, there is a bewildering array of these compounds formed and some of them can cause off-flavors at levels[3] analytically undetectable with existing techniques.

When analysis of end products of oxidation is undertaken it should be with the understanding that detectable levels are usually well beyond those causing off-flavors in almost all foods.

One of the more widely used tests to determine final reaction products has been the TBA test. This test relies on the development of a red pigment when malonaldehyde and 2-thiobarbituric acid react as shown in Figure 4[5]. The pigment formed absorbs at about 535 nm and can be formed in an intact, or nearly intact, food and then extracted with suitable solvents. The chemistry of the test has been explored by Tarladgis *et al.*[6,7]. In addition to the reaction with malonaldehyde, there may be other compounds formed during the reaction that may or may not involve oxidation reaction products[8].

There is a question as to whether malonaldehyde exists as such or is formed during the reaction but this is academic insofar as utility of the test is concerned. Formation of malonaldehyde has been postulated to occur by the mechanism shown in Figure 5[9].

In any event, the utility of the TBA test is in the ability to use it on a food already in, or suspended in, an aqueous medium followed by extraction of the formed pigment with

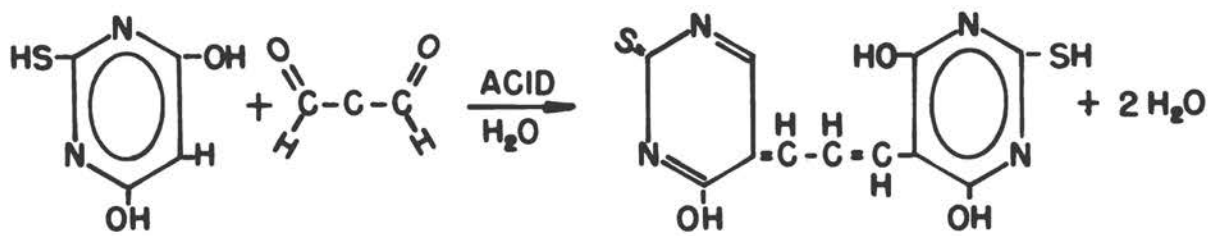


FIGURE 4 Reaction of 2-thiobarbituric acid (TBA) with malonaldehyde[5].

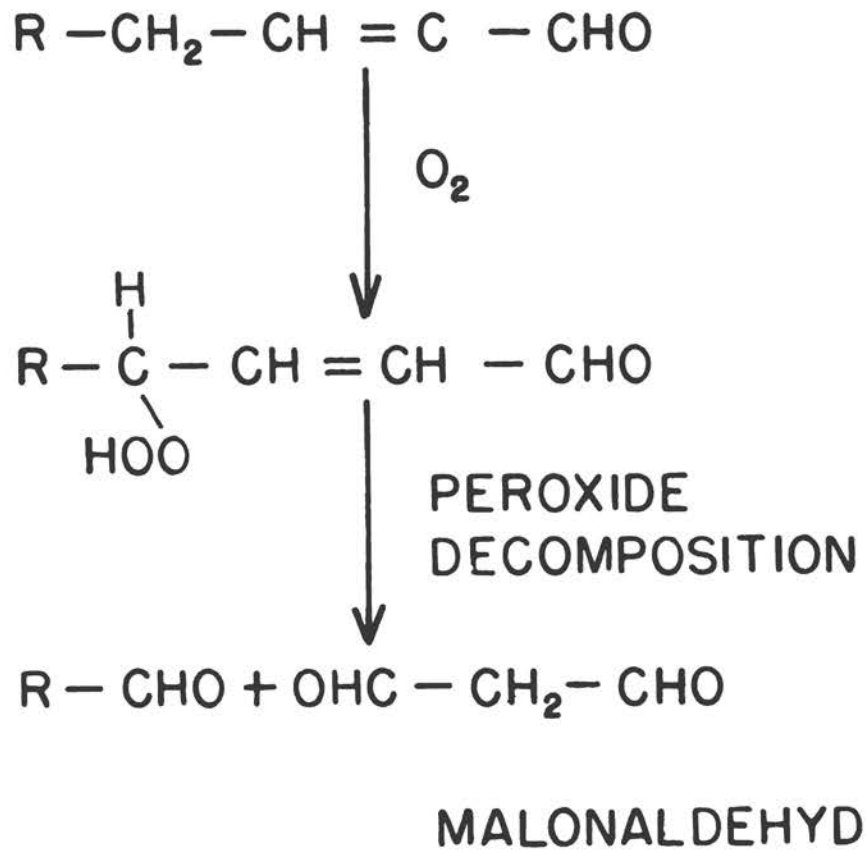


FIGURE 5 Proposed mechanism of malonaldehyde formation[9].

appropriate solvents. Some of the products to which the TBA test has been applied are milk and milk products[10,11], meats[12], edible oils[13], and fish[14].

Users of the TBA test have discussed various precautions such as the instability of the TBA reagent to acid, peroxide, and heat[6,7]. Also, the length of reaction time and the purity of the reagent itself can be important considerations.

Even when the investigator has carefully avoided the above mentioned pitfalls, one final caution has been somewhat neglected by researchers. This overlooked factor is a lack of emphasis on examination of the total visible spectrum instead of reading at a single wavelength. The TBA test almost always results in a positive response, which may or may not be due to formation of the TBA-malonaldehyde condensation product.

A good illustration of this is the spectrum obtained when the TBA test is applied to fresh fluid milk. In this case an absorption maximum at 450 nm is observed, which results in a measurable absorption at 535 nm[10]. In Figure 6 a series of curves that might be generated during the development of oxidized flavor

in fluid milk are shown. The fresh milk shows apparent initial absorption at 535 nm, with successive buildup at that wavelength as oxidation proceeds.

If the course of oxidation is being followed from zero time in a given product, then the blank value at zero time can be taken as the base value and increases from that point rather easily determined. If, however, a sample is taken "off the shelf" without prior knowledge of its zero-time value, then a total spectrum needs to be run to judge the actual increase in TBA value. This consideration becomes even more important when other red pigments such as those in red meat are present.

In any system to which the TBA test is applied, it is strongly recommended that a total spectrum be run to assure meaningful interpretation by the researcher and by those attempting to follow any TBA procedure published in the literature.

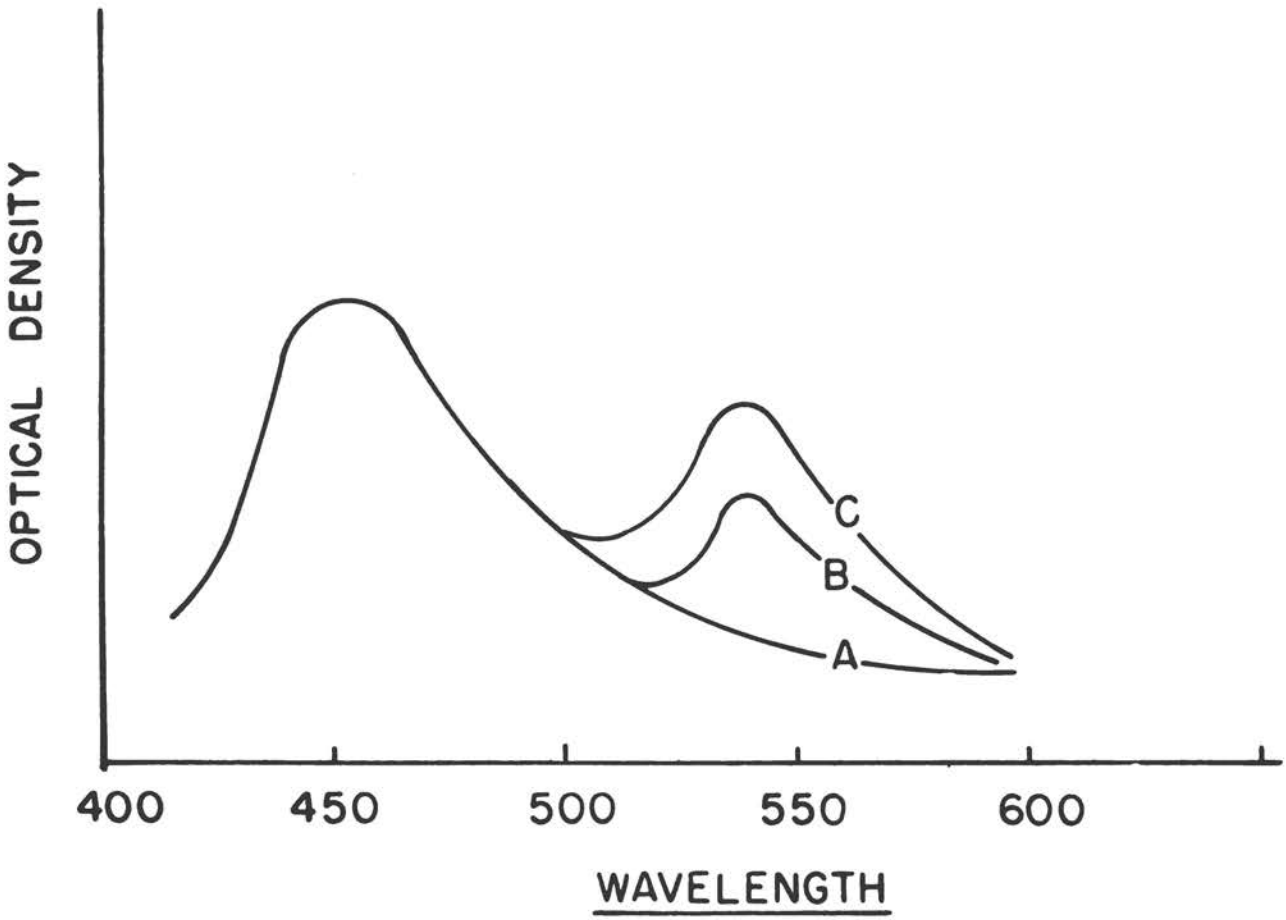


FIGURE 6 Development of TBA value in oxidized milk[10]. A, B, and C represent progression of oxidized flavor development in milk.

Other techniques to quantify final oxidation reaction products may also be applied. The reaction of carbonyls with such reagents as 2,4-dinitrophenylhydrazine, benzidine, and the Girard T reagent may be used indirectly by distillation of such products from foods. This procedure is subject to question since distillation may produce artifacts and also is not sensitive enough to be used on most foods during the early stages of objectionable flavor development.

Another technique used more recently to quantify peroxide decomposition products is gas liquid chromatography (GLC). Some applications have been made to vegetable oils[15,16], peanuts[17], and oilseed meals and flours[18].

As with all other methods, the sensitivity of this method is not yet good enough to detect components formed during the early stages of off-flavor development.

PREDICTIVE METHODS

We have thus far touched upon the major objective methods that could be used for determination of fat stability in foods. There are several choices available to an investigator, none of which is apparently as sensitive in the early stages of off-flavor development as is organoleptic evaluation. This being the case, the chemical tests are usually measuring oxidation beyond the point of off-flavor development. Extended periods of time are frequently required for the development of off-flavor in certain foods. It is therefore essential to the development of a practical test that the above mentioned chemical tests be coupled with intentionally accelerated development of off-flavor.

A tabulation of the factors influencing the rate of lipid oxidation is shown in Table 2. Any one of the accelerative factors may be applied to a food and, in combination with one of the tests previously mentioned, be used to predict oxidative stability.

TABLE 2 Factors operative in influencing lipid oxidation.

Accelerators	Inhibitors
High temperature	Freezing or refrigeration
Metals (Cu, Fe, etc.)	Metal sequesterants
Peroxides (oxidized fats)	Antioxidants
Enzymes (lipoxidase)	Blanching
Oxygen pressure	Inert gas or vacuum packaging
Light (UV, blue)	Opaque packaging
Polyunsaturation of fatty acids	Hydrogenation of unsaturated fatty acids

Prior to application of accelerative techniques, strong consideration must be given to the kinetics and/or mechanism of lipid deterioration. These must be understood to avoid a completely artificial situation; e.g., the use of UV to accelerate oxidation when the food under study would never be exposed to such attack. In this case, the actual reaction pathways as well as the kinetics are probably different. For example, there is good evidence that a different class of flavor compounds is formed in both vegetable oil[19] and milk[20] when exposed to light rather than ordinary oxidation.

Another point to consider is the relative reaction rates of oxygen with unsaturated fatty acids. In comparison to oleic acid, linoleic reacts 15 times faster and linolenic 30 times faster.

Under normal conditions, such as room temperature, linolenic acid would be expected to react first and would be the principal reactant, but if the temperature were elevated, then the other unsaturated fatty acids might also react, changing the reaction pathway.

A good example of an accelerated test widely used and often indiscriminately applied is the AOM test used in the edible oil industry[21]. This test is completely artificial in terms of fat usage. The temperature is elevated to 97.8 ± 0.2°C, the sample aerated continuously, and the hours necessary to reach some predetermined peroxide value is taken as the end point.

If the conditions of the test are understood, as in the case of AOM and one has the past experience to correlate this value to other criteria of òff-flavor development, then, and only then, does it become useful. An AOM value, in and of itself, is not indicative of the flavor stability of an edible fat.

In conclusion, whatever approach to the study of lipid oxidation is chosen by an investigator from the extensive literature available, he should be acutely aware of:

1. The dynamic character and complexity of the reactions involved in lipid oxidation.
2. The extremely low levels of peroxide decomposition products that can cause significant off-flavors.
3. The need for realistic correlation of such tests with organoleptic evaluation in all cases.
4. The importance of judicious selection and interpretation of accelerating techniques as they relate to the normal situation in foods.

REFERENCES

1. Labuza, T. P., *CRC Crit. Rev. Food Technol.*, 355-405 (October 1971).
2. Schultz, H. W., E. A. Day, and R. O. Sinnhuber, *Symposium on Foods: Lipids and Their Oxidation* (AVI Publ. Co., Westport, Connecticut, 1962).

3. Forss, D. A., *Prog. Chem. Fats Other Lipids*, 181-258 (Pergamon Press, 1973).

4. Eastman Chem. Prod. Publication ZG-195, "Oxygen Bomb Method for Comparing Oxidative Stabilities of Food, Oils, and Food Products" (Kingsport, Tennessee, 1973).

5. Sinnhuber, R. O. and T. C. Yu, *Food Res.*, 23:626 (1958).

6. Tarladgis, B. G., A. M. Pearson, and L. R. Dugan, *J. Am. Oil Chem. Soc.*, 39:34 (1962).

7. Tarladgis, B. G., A. M. Pearson, and L. R. Dugan, *J. Sci. Food Agric.*, 15:602 (1964).

8. Marcuse, R. and L. Johansson, *J. Am. Oil Chem. Soc.*, 50:387 (1973).

9. Day, E. A., *J. Dairy Sci.*, 43:1360 (1966).

10. Dunkley, W. L. and W. G. Jennings, *J. Dairy Sci.*, 34:1064, (1951).

11. Lillard, D. A. and E. A. Day, *J. Dairy Sci.*, 44:-- (1961).

12. Zipser, M. W. and B. M. Watts, *Food Technol.*, 12:(7)102 (1962).

13. Jacobson, G. A., J. A. Kirkpatrick, and H. E. Goff, *J. Am. Oil Chem. Soc.*, 41:124 (1965).

14. Sinnhuber, R. O. and T. C. Yu, *Food Technol.*, 12:(1)9 (1958).

15. Dupuy, H. P., S. P. Fore, and L. A. Goldblatt, *J. Am. Oil Chem. Soc.*, 50:340 (1973).

16. Jarvi, P. K., G. D. Lee, D. R. Erickson, and E. A. Butkus, *J. Am. Oil Chem. Soc.*, 48:121 (1971).

17. Brown, D. F., F. G. Dollear, and H. P. Dupuy, *J. Am. Oil Chem. Soc.*, 49:81 (1972).

18. Fore, S. P. and H. P. Dupuy, *J. Am. Oil Chem. Soc.*, 49:129 (1972).

19. Moser, H. A., C. D. Evans, J. C. Cowan, and W. F. Kwolek, *J. Am. Oil Chem. Soc.*, 42:30 (1965).

20. Dunkley, W. L., J. D. Franklin, and R. M. Pangborn, *Food Technol.*, 16:(9)112 (1962).

21. AOCS *Official and Tentative Methods*, Method Cd 12-57 (Champaign, Illinois).

STABILITY OF VEGETABLE OIL SYSTEMS AND OBJECTIVE METHODS OF MEASUREMENT

Alexander E. Thomas, III

INTRODUCTION

The term "stability" as applied to vegetable oil systems is often thought of as synonymous with oxidative stability. However, oxidation is only one factor, albeit an important one, affecting the overall stability of vegetable oil systems. Perhaps, more importantly, it is the one factor affecting the stability of a formulated food product produced from vegetable oil. After all, few of us consume all our fat intake in the form of visible fat. Fat is incorporated into the diet mainly as part of a more complete food item.

In the broader sense, the concept of "stability" encompasses physical, chemical, and microbiological phenomena that have the potential to produce instability in a vegetable oil. Although the extent to which such phenomena affect stability is inherent in the composition of the fat system, the rate at which such phenomena affect stability is often dependent upon the presence of other constituents or conditions that act either to retard or accelerate instability. Such constituents or conditions may be present in the fat system or may be externally introduced from ingredients or equipment used in the preparation of a more complete food item.

The vegetable oil industry utilizes such processing techniques as refining, bleaching, deodorization, hydrogenation, fractionation, "votation," and fluidization to alter the properties of crude vegetable oils, with the objective of producing functional, stabilized oil systems. The ultimate test of stability and functionality of vegetable oil systems has been, and will continue to be, based upon evaluation of the complete food under ideal and stressed processing conditions as well as under ideal and stressed storage environments. However, the extensive time requirements of this approach provide the impetus for the continuing development and utilization of supplemental objective methods of measurement, particularly where such methods provide data of a predictive nature.

The scope of this presentation will be limited to the relative stability of vegetable oil systems as it applies to

three of the most important phenomena; i.e., oxidation, hydrolysis, and polymorphism, as well as objective methods of measurement commonly used as predictive tools.

As a basis for better understanding the objective methods employed and the comparative stabilities obtained, let us first consider the composition and structure of vegetable fat and oil systems.

COMPOSITION OF VEGETABLE OIL SYSTEMS

All vegetable fat and oil systems share a simple chemical classification; i.e., a triglyceride, which is simplistically represented in Figure 1. All fat and oil systems share in this element of commonality irrespective of their source or prior processing.

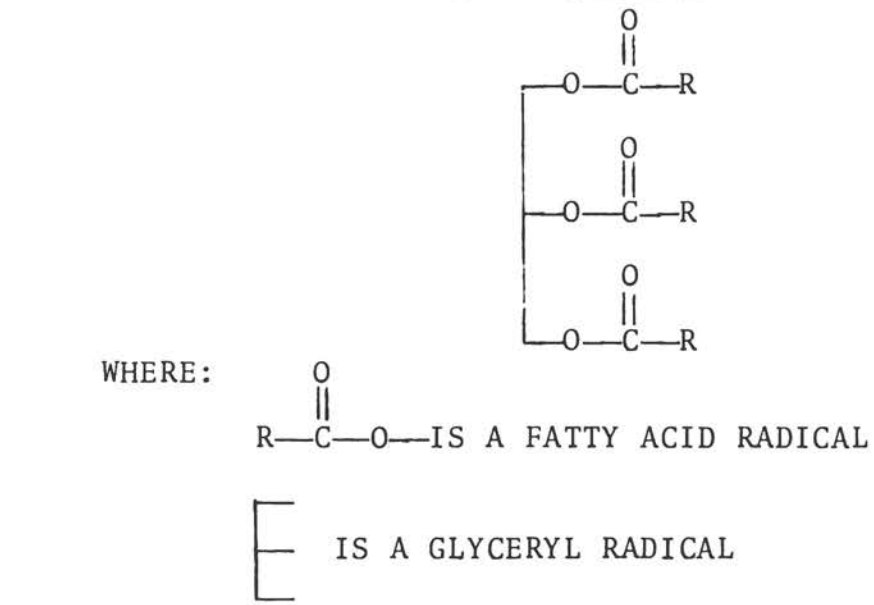


FIGURE 1 Structural commonality of fat and oil systems.

Perhaps more important, however, are the differences in chemical structure and composition that influence functionality and stability. These differences, shown in Figure 2, are fatty acid chain length, unsaturation, and position.

The fatty acids most frequently present in high percentages in vegetable fats and oils are those containing 12, 16, or 18 carbon atoms in the chain. The fatty acids of coconut, soybean, cottonseed, and palm oils shown in Table 1 are typical. Fatty acids of other chain lengths are present, but in lesser quantity.

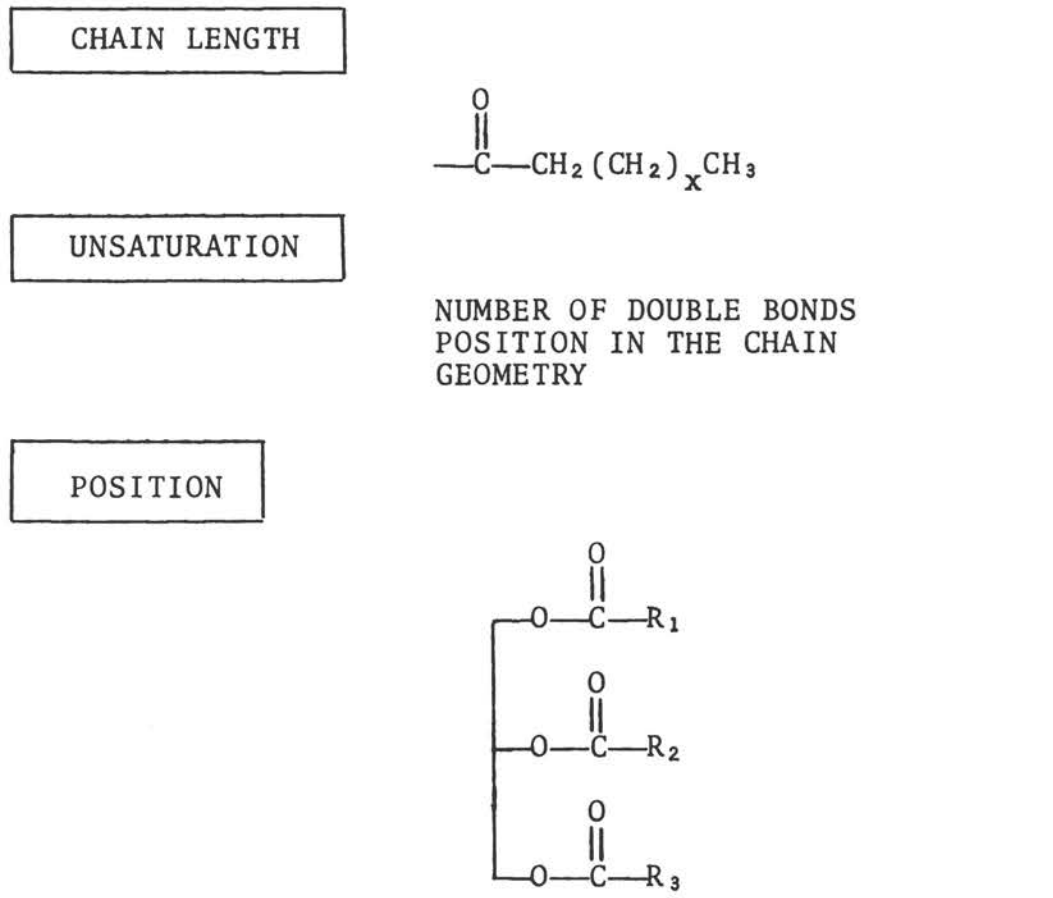


FIGURE 2 Composition and structural differences of fats and oils.

Because of the ester structure, all vegetable fat and oil systems have the potential to hydrolyze in the presence of water to form free fatty acids and other products whose composition depends upon the extent of hydrolysis. In general, the rate of hydrolysis for fats ranging in chain length from 12 to 18 carbons is similar, although chains shorter than 12 carbons tend to hydrolyze more rapidly due to their increased water solubility. Hydrolytic tendencies of vegetable fat and oil systems may vary due to other factors such as catalytic substances, water content, and temperature. Objective methods for predicting or measuring the extent of hydrolysis are most frequently based on the development and measurement of free fatty acid content.

In addition to chain length, other differences in chemical structure due to unsaturation of the fatty acid radical influence functionality and stability. Although saturated glycerides can be oxidized under extreme conditions, the real potential for oxidation of fats in food systems resides for the most part in the unsaturated structure. The rate of oxidation is dependent

TABLE 1 Fatty acid composition of selected vegetable oils.

Fatty acid	Carbon number	Coconut	Soybean	Cottonseed	Palm
Caproic	6.0	1			
Caprylic	8.0	8			
Capric	10.0	6			
Lauric	12.0	48			
Myristic	14.0	18		1	1
Palmitic	16.0	8	11	25	45
Stearic	18.0	3	4	2	5
Oleic	18.1	7	23	18	39
Linoleic	18.2	1	53	53	9
Linolenic	18.3		8		
Other	--		1	1	1

upon the number of double bonds, their position in the chain and their geometry; i.e., cis versus trans. The rate of oxidation is further influenced by the presence of pro-oxidants such as trace metals, or antioxidants either added or indigenous, and by available oxygen content, temperature, etc. Objective methods for predicting or measuring the extent of oxidation are most frequently based on the development and measurement of peroxides.

Because of the trifunctional nature of glycerol and the large number of fatty acids, fats may range in composition from those containing a few triglycerides to those containing several hundred. With a knowledge of the number of fatty acids present, and assuming a random distribution, the maximum number of theoretically possible triglyceride structures may be calculated. As seen in Table 2, a large number of triglyceride structures is possible from a relatively small number of fatty acids. Although all vegetable oil systems are not comprised of fatty acids whose distribution is completely random, processing techniques such as blending, fractionation, hydrogenation, and interesterification are employed to produce a wide range of triglyceride composition differing in functional characteristics and in polymorphic behavior.

Polymorphic form in crystalline fats is dependent upon composition and is influenced by processing and thermal history. Objective methods for predicting or measuring polymorphic stability are frequently based on heats of transformation or on molecular orientation in the crystal lattic.

TABLE 2 Theoretical number of triglycerides as a function of the number of fatty acid structures.

Number of fatty acids (n)	Number of triglycerides (N)
1	1
2	4
3	9
4	20
5	35
6	56
7	84
8	120
9	165
10	220

$$N = n + n(n-1) + n(n-1)(n-2)/6$$

OBJECTIVE METHODS OF MEASUREMENT AND TYPICAL STABILITIES

Oxidation

The Active Oxygen Method (AOM) of the American Oil Chemists' Society[1] has been used extensively to evaluate the relative oxidative stability of vegetable oil systems. Although the time required for the test has led to the development of more rapid methods such as the Oxygen Bomb Method (OBM)[2] or the Differential Scanning Calorimetry (DSC) method[3], the AOM method continues to be the one most often employed by the industry. Typical AOM values for oxidative stability of refined, bleached, deodorized vegetable oil systems are shown in Table 3. The very high AOM of coconut oil is due to the limited amount of unsaturated fatty acids present, as seen earlier in Table 1.

Partial hydrogenation of oxidatively less stable oils serves not only to alter functional properties but to enhance their stability. Table 4 shows the effect of partial hydrogenation on the oxidative stability of a typical soybean oil. The presence of pro-oxidants such as copper or iron may act to lower the oxidative stability of vegetable fats. Conversely, the use of oxygen scavengers and chelating agents will, in general, increase oxidative stability.

TABLE 3 Typical oxidative stability of refined, bleached, deodorized vegetable oil systems.

	AOM
Coconut	310
Cottonseed	13
Palm	25
Soybean	11

TABLE 4 The effect of hydrogenation on the oxidative stability of vegetable oil systems (soybean oil).

Iodine value	AOM
117	9
97	25
86	54
78	70
73	439
70	860

Hydrolysis

Unlike oxidation, analytical methodology for predicting the hydrolytic stability of a vegetable oil system is not well standardized. In our own laboratories we generally employ a modification of the procedure described by Fritsch *et al.*[4], which consists of titrating the quantity of free fatty acids formed by hydrolysis of the fat under specific and controlled conditions. Hydrolytic stability constants for selected coconut, cottonseed, and soybean oils are shown in Table 5. Considerably greater variation in hydrolytic constants are found in oils of commerce. It is speculated that this greater variation in hydrolytic constant is related to the extent of hydrolysis which has occurred between harvesting and refining.

Polymorphic Behavior

Vegetable fat systems that contain crystalline solids exhibit polymorphism; i.e., they can exist in more than one

TABLE 5 Typical hydrolytic stability constants.

Coconut	0.10
Cottonseed	0.12
Soybean	0.11

crystalline form. Using the nomenclature of Hoerr[5], the lowest melting of these, the α (alpha) form, is normally obtained by rapidly cooling from the melt. Upon heating to a temperature just below its melting point, the α form transforms to β' (beta prime), intermediate, and finally the β (beta) form, the highest melting and most stable. Each form exhibits a characteristic X-ray diffraction pattern that can be readily obtained and recognized using X-ray diffraction methodology[6]. All lipid systems transform to a stable polymorphic state, but may do so at differing rates and to different extents. Therefore, the rate and extent of transformation may be monitored by taking periodic X-ray spectra until transformation is complete.

Since processing conditions employed to solidify a vegetable-fat system, e.g., spray-chilling, flaking, "votation," affect the extent to which polymorphic stability has been achieved, the X-ray spectra technique may be used to determine the extent of polymorphic stability achieved in processing. This is an important consideration since, in many instances, further polymorphic change tends to be detrimental; e.g., in a beaded fat, the heat of transformation may cause particle clumping if it occurs in the package.

Polymorphic behavior of selected vegetable-oil systems is illustrated in Table 6. The effect of temperature on the rate of polymorphic transformation from β' to β for a 61 iodine value soybean oil is shown. For example, after 10 days at 70°F (21.1°C), only 4 percent of the sample is in the β form; at 85°F (29.4°C), approximately 56 percent has transformed; and at 100°F (37.8°C), 90 percent has transformed.

The extent of hydrogenation of a vegetable fat affects the number of glycerides; hence, it also affects its polymorphic behavior. This effect can be seen in the faster transformation of 0 iodine value soybean oil versus 62 iodine value soybean oil.

SUMMARY AND CONCLUSIONS

Three of the most important phenomena, i.e., oxidation, hydrolysis, and polymorphism, affecting the stability of vegetable-oil systems were identified. The role of glyceride structure, composition, and external factors such as environment

TABLE 6 Polymorphic behavior of vegetable oil systems.

A. 62 Iodine Value Soybean Oil			
	Storage temperature		
	70°F	85°F	100°F
Days	Form β'/β	Form β'/β	Form β'/β
0	100/0	--	--
1	100/0	92/8	74/26
3	100/0	80/20	28/72
8	100/0	54/46	14/86
10	96/4	44/56	10/90
14	94/6	36/64	8/92
21	91/9	23/77	7/93
28	87/13	21/79	4/96
36	81/19	19/81	--
71	51/49	7/93	0/100

B. 0 Iodine Value Soybean Oil		
	Storage temperature	
	70°F	122°F
Days	α/β'/β	α/β'/β
0	100/0/0	100/0/0
4	--	0/3/97
60	0/90/10	--

and trace ingredients were reviewed. A general description of the most useful analytical methodology applicable as predictive tools for these phenomena was presented. Finally, some typical stability data on selected vegetable-oil systems were provided.

REFERENCES

1. *Official and Tentative Methods of the American Oil Chemists' Society*, Vol. *I*, 3rd edition, AOCS (Champaign, Illinois, 1964).

2. Gearhart, W. M., B. N. Stuckey, and J. J. Austin, *J. Am. Oil Chemists' Soc.*, *34*, 427 (1957).

3. Cross, C. K., *J. Am. Oil Chemists' Soc.*, *47*, 229 (1970).

4. Fritsch, C. W., V. E. Weiss, and R. H. Anderson, *J. Am. Oil Chemists' Soc.*, *48*, 392 (1971).

5. Hoerr, C. W., *J. Am. Oil Chemists' Soc.*, *37*, 539 (1960).

6. Hoerr, C. W., *J. Am. Oil Chemists' Soc.*, *41*, 4 (1964).

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

EVALUATION OF FAT STABILITY IN EMULSIFIED FOODS

Gerard J. Henning

INTRODUCTION

Microbial spoilage and oxidative deterioration of fats are the most important causes for the rejection of foods for human consumption. While generally accepted methods are available for the measurement and the prediction of microbiological stability, this is not so for the evaluation of fat stability.

The time between production and consumption varies considerably for different food products. Depending on the type of processing, packaging, storage, and distribution, shelf-lives from 1 week to over 1 year may be encountered.

For assessing the taste-keepability of food products, storage for different lengths of time at various temperatures, followed by taste-paneling, is most widely used. Since these methods are rather time-consuming, and taste panels are difficult to maintain, objective methods for judging and predicting the taste-keepability of foods are badly needed.

In the following areas, the more common methods for objectively determining fat stability toward oxidation and their potential for judging this stability in emulsions are reviewed.

FAT OXIDATION IN EMULSIONS

For the sake of completeness, let us first recall briefly the mechanism by which oxidation of triglyceride oils proceeds[1,2].

It is well known that the more prone fatty acids are to oxidation, the more unsaturated they are. Roughly speaking, the sensitivity to oxidation for oleic, linoleic, and linolenic acids is in the proportion 1:15:30.

A simple scheme for the autoxidation of mono-linoleate is given in Figure 1. At stage 0 (zero), unchanged linoleate is present, which is characterized by its structure with two non-conjugated (methylene-interrupted) cis double bonds.

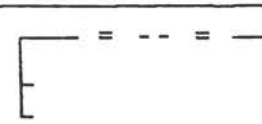
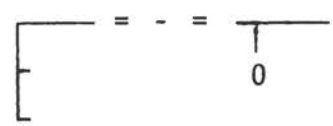
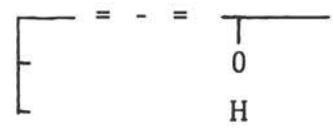
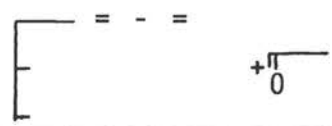
Stage	Compounds	Characteristic structure
0	Linoleate	
1	Hydroperoxides Bishydroperoxides (Alkoxy radicals)	
2	Chain conservation Hydroxyglycerides Dimeric ethers Ketoglycerides Trienes	
	Chain scission Volatiles Non-volatiles	

FIGURE 1 Autoxidation of mono-linoleate.

Through initiation of the oxidation by light, temperature, or metal ions, free radicals are formed, which by reaction with oxygen and further propagation and termination reactions give rise to a complex mixture of hydroperoxides and bishydroperoxides (stage 1).

Since peroxidation involves attack at allylic carbon atoms, the structure of the fatty-acid chain in these compounds is of the conjugated type, the cis-trans diene isomers being the predominant species. The molecular weight of the compounds formed in this stage is about equal to that of the parent linoleate, whereas they are tasteless, odorless, and relatively non-toxic.

By dismutation of hydroperoxides, hydroxy radicals and alkoxy radicals are formed. Alkoxy radicals, being reactive species, can undergo a number of reactions leading to secondary products of oxidation (stage 2). Thus, by abstraction of a hydrogen atom from linoleate, a hydroxyglyceride is formed, whereas addition to the double bond of linoleate would lead to a dimeric ether. In all these reactions, the carbon chain of the

alkoxy radical is preserved; the compounds formed have conjugated double bonds and molecular weights equal to or higher than that of the linoleate. The compounds are not likely to contribute to the taste or the odor of an oil either.

It is the chain scission of alkoxy radicals in stage 2 of the oxidation that gives rise to the formation of lower-molecular-weight compounds to which the off-flavors of oils can be attributed. Among the volatiles formed are aldehydes, alcohols, acids, ketones, and hydrocarbons, the aldehydes generally having the lowest threshold values (1 ppm or less) and therefore being the most characteristic of the oil off-flavors.

It should be noted that chain scission always leads to the formation of more than one fragment. In addition to the volatiles mentioned, non-volatile compounds are thus formed; e.g., aldehydroglycerides. Compared with the non-volatile products mentioned earlier, they have somewhat lower molecular weights and less conjugation.

Most of the work on autoxidation has been carried out with oils and fats as such, rather than emulsions. From what we know of the off-flavors formed in emulsified foods we can conclude that the autoxidation mechanism in emulsions is basically the same. We should bear in mind, however, that there are important differences between the two systems.

First, upon emulsification there is a large increase in interfacial area, which should make the fat more prone to attack by oxygen. In its generality this is not true because other factors are also known to influence the oxidation, such as pH, salt content, the presence of metals, proteins, and phospholipids, factors that can offset the effect of the increase in interface upon emulsification.

Second, in food emulsions we often are concerned with fat blends rather than with single oils. Components in such blends range from stable oils (e.g., coconut) through medium stabile oils (e.g., maize) to unstable oils (e.g., soybean). Usually, a certain amount of hydrogenated oil is also present, which upon autoxidation can give rise to the formation of "hardening" flavors[3]. The autoxidation pattern in fat blends may be different from that of either of the constituent oils.

Third, the threshold for the detection of weakly polar compounds, such as aldehydes, is generally lower in emulsions than in fat[4], which makes off-flavor formation in emulsified foods noticeable at still lower degrees of oxidation than with oils.

Autoxidation in oils and fats on the one hand and oils in emulsion on the other would seem to follow the same pathways, though differences might be observed in the rate of off-flavor formation or in the degree to which off-flavors can be perceived. In principle, therefore, the same objective tests that are used to assess the degree of oxidation of oils or to predict their future keepability can be used for emulsions also.

METHODS FOR MEASURING FAT OXIDATION

The more commonly used analytical methods[5] for measuring fat oxidation are collected in Table 1. They have been divided into static methods, which measure the degree of oxidation at a certain moment in time, and dynamic methods, in which the fat is subjected to a kind of accelerated aging process.

Static Methods

Ultraviolet Absorption Measurements[6]

One of the simplest methods of obtaining some insight into the stage of oxidation of a fat is the measurement of its UV absorption.

As we have seen, autoxidation is accompanied by conjugation of double bonds and therefore should lead to a progressive increase in light absorption in the diene region around 230 nm.

As an example, UV absorption spectra for crude and fully refined soybean oils are given in Figure 2. The curves exhibit

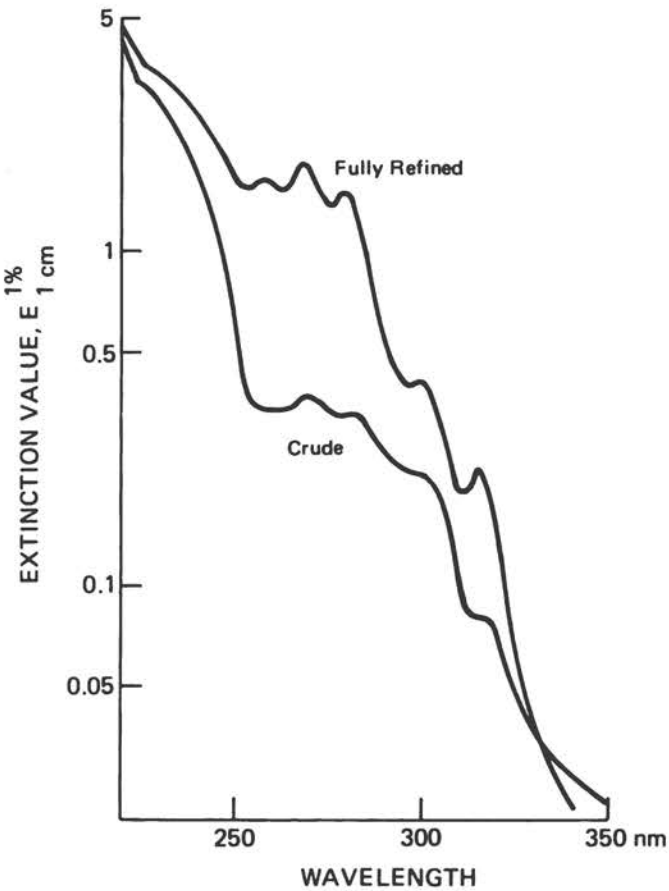


FIGURE 2 Ultraviolet absorption spectra of crude and fully refined soybean oils.

TABLE 1 Analytical methods for measuring fat oxidation.

Test	Assessment of	Applicability to emulsions*	Predictive value
Static Methods			
UV extinction	Conjugated dienes/trienes	+	low
Peroxide value	Peroxides	+	low
Oxygenated fatty acids	Oxygenated fatty acids	+	low
Carbonyl value	All carbonyl functions	±	low
Anisidine value	α,β-unsaturated carbonyls	±	medium
TBA value	Malonic dialdehyde	+	medium
GLC analysis	Volatile compounds	±	low
Dynamic Methods			
Schaal test	Peroxides	+	medium
Warburg manometric test	O_2 absorption	+	medium
Active oxygen method	Peroxides	-	medium
Bomb test	O_2 absorption	-	medium
Filter-paper test	Peroxides	-	low

* + = applicable
  ± = of limited use
  - = cannot be applied

several maxima, of which the shoulder at 232 nm is indicative of the diene content, the maximum at 268 nm being related to the triene content. Compounds with trienoic conjugation, e.g., ketodienes, true trienes, doubly and triply unsaturated aldehydes, are formed as secondary products of autoxidation. As is apparent from Figure 2, their content increases during refining, notably after bleaching.

Though perhaps not relevant to the judgment of oil quality in prepared food products, it should be borne in mind that even in carefully treated crude oils, compounds with dienoic and trienoic conjugations are present, which presumably are formed as by-products during the biosynthesis of unsaturated fatty acids.

UV extinctions for a number of oils are given in Table 2. Since E 1%, 1 cm (extinction value) for conjugated dienoic fatty acids is around 1,000 and for conjugated trienoic fatty acids around 2,000, the amount of conjugated material is only a few tenths of a percent. Most off-flavor compounds are perceptible at levels at least three orders of magnitude lower. Even when we take the differences in molecular weight into account, the contribution of dienals and similar compounds to the extinction values normally is negligible.

TABLE 2 UV extinction data for some fully refined oils[6].

Wavelengths	Extinction value (E 1%, 1 cm)			
	Soybean	Groundnut	Sunflower seed	Rapeseed
232 nm	4.2	2.5	4.0	3.0
268 nm	2.2	1.5	3.9	1.1

The great value of the UV extinction measurement is that it gives information on the oxidative damage done to a crude oil and on the efficiency of the refining process. Unless extremely high extinction values are observed (e.g., > 5 at 232 nm), its value for judging the quality of refined oils is very limited.

Peroxide Value (POV)

Many variants are described in the literature for determining the peroxide value of fats; i.e., the number of milli-equivalents of peroxide oxygen present per kg of fat. The most popular methods are iodometric procedures in which the amount of iodine liberated from potassium iodide by the oxidative action of the peroxides is measured.

Well-refined oils have peroxide values below 1, which upon exposure of the oil to air in daylight may reach values of 2 to 3 in a couple of days for bean oil or sunflower oil and a value of 10 for tallow.

The peroxide value attained depends upon the speed with which peroxides are formed, but also on the speed with which they are decomposed. The stability of the peroxides formed in such oils as bean oil and sunflower seed oil is low compared to those from tallow and consequently peroxide values are higher in the latter.

The peroxide value for 100 percent oxidation being around 6,000, a POV of 10 would mean that 0.15 percent of the fatty acids were peroxidized. Even if we would assume that only 10 percent of the peroxides suffered chain scission, the amount of volatiles generated would be a few 100 ppm, more than sufficient to generate a strong off-flavor. Indeed, on many occasions off-flavors are noticeable in systems in which measured peroxide values are far below 1. Peroxide values are therefore of limited use either for judging the oxidative history of fats or for predicting future keepability.

Oxygenated Fatty Acids (Triglycerides)

Part of the oxygen absorbed in the autoxidation process is retained in the form of triglycerides, carrying one or more oxygenated fatty acid residues.

In general, such products are tasteless, but their presence in oils may give an indication of the oxidative history of the fat mixture under consideration.

Several methods are described in the literature to determine the contents of oxidized fatty acids in oils, particularly in relation to the assessment of the quality of used frying oils. For oils with relatively high levels of oxygenated fatty acids, the insolubility of the latter in petroleum either can be used to separate them from non-oxidized material. At levels of less than 1 percent, chromatographic methods, particularly thin-layer chromatography, must be used[7].

The contents of oxygenated fatty acids in various oils is given in Table 3.

TABLE 3 Percentages of oxygenated fatty acids in some crude and fully refined oils[7].

Stage	Soybean	Groundnut	Sunflower seed	Rapeseed
Crude	1.3	3.1	--	0.8
Refined	0.8	1.6	1.3	0.5

As indicated in Table 3, the levels in crude and refined oils do not differ very much; this corresponds to our own experience.

Refined oils with contents of oxygenated fatty acids higher than 1 percent should be considered with care since their future keepability may be rather low.

A method for separating oxidized triglycerides into slightly oxidized, medium-oxidized, and strongly oxidized materials by thin-layer chromatography on alumina is described by Pokorný and Davidkowá[8].

Determination of Carbonyls

A number of methods have been described to determine the level of carbonyl compounds in autoxidized oils[9].

With most methods the total carbonyl content is determined -- the sum of the free volatile carbonyls (about 10 percent of the total); the free, less volatile carbonyls; and the carbonyls present in bound form (e.g., aldehydo-glycerides). By vacuum or steam distillation the volatile carbonyls can be separated from the other carbonyl compounds present and their contents estimated separately.

The most commonly used reagent for assessing total carbonyl contents is 2,4-dinitrophenylhydrazine, which in acid medium yields yellow- to orange-colored hydrazones. Measurement of the light absorption in alkaline medium at 440 nm gives an indication of the total carbonyl content, the carbonyl value being expressed as mmol hexanal/kg fat. Care should be taken to use methods in which no decomposition of hydroperoxides occurs[10].

A somewhat better measure for judging the oxidative history of an oil or to forecast its future keepability is the anisidine (benzidine) test[11]. This test is based on the reaction between anisidine (or benzidine) and aldehydes yielding colored Schiff's bases, the light absorption of which is measured at 350 nm. Since the molar extinction of the reaction products formed with α,β-unsaturated aldehydes is much higher than with saturated aldehydes, the anisidine value is essentially a measure of the contents of volatile and non-volatile α,β-unsaturated aldehydes.

A third test often used to estimate the degree of fat oxidation in fat-containing food products is the thiobarbituric acid test (TBA), which is based on the formation of a colored complex between 1 molecule of malonic dialdehyde (one of the secondary oxidation products from highly unsaturated fatty acids) and 2 molecules of thiobarbituric acid. Measurement of the light absorption is carried out at 530 nm, the value obtained being given as such or expressed as mg malonic dialdehyde/kg fat (TBA value).

In well-refined oils, carbonyl values are in the range of 0.5 to 2 mmol/kg, anisidine values are between 1.0 and 10.0. Since the TBA method is highly empirical, extinction values differ according to the procedure chosen.

For judging the degree of oxidative damage done to crude oils and for following the efficiency of the refining process, determination of anisidine values (and to a lesser extent carbonyl values) is useful. For following the progress of oxidation in refined oils, these methods are too crude; there, the TBA test seems to give better results.

It should be noted that values obtained with the 3 methods are not comparable for different oils. Thus, whereas for a refined bean oil anisidine values of ≥ 10 would be an indication of poor future keepability, this is certainly not the case for sunflower seed oil.

Gas Chromatography of Volatiles

Gas chromatography is a powerful tool for the detection of the amount and type of volatiles present in oils. Enrichment of volatiles prior to GLC (gas-liquid chromatography) is indicated, either by vacuum distillation, entrainment, or by using special precolumns[12].

In our laboratory the potential of gas-liquid chromatography of volatiles for judging the quality of soybean oils was investigated. The method appeared to be of limited use.

Dynamic Methods

Dynamic methods for judging the susceptibility of fats to oxidation have been in use now for over 50 years. In principle all methods are the same; i.e., oxidation of the fat is accelerated by changes in the external conditions (temperature, oxygen pressure, light intensity) so that the time-span in which oxidative changes occur is reduced from weeks or months to hours or days. The state of oxidation reached is assessed either organoleptically or by determining analytical data such as peroxide values.

Schaal or Oven Test

In the Schaal test, fat samples are stored at elevated temperatures (40 to 70°C) in beakers, dishes, etc., and samples are withdrawn at suitable intervals for organoleptic or chemical analysis. The number of days elapsing before off-flavors are perceptible or before a certain peroxide value is reached is taken as a measure of the oxidative stability of the fat.

Particularly for the more stable oils there is a fair correlation between the result of the Schaal test and the keepability of the oils at room temperature.

Oxygen Absorption Methods

Oxygen absorption by a fat is usually characterized by an induction period during which oxygen absorption is very slow, followed by a period of accelerated oxygen uptake. The length of the induction period often is taken as a measure of the oxidative keepability.

In all the methods currently used the oxidation is carried out at elevated temperatures, but the methods differ in the way oxygen is admitted into the system.

Oxygen absorption at normal pressure is used in the Warburg manometric methods.

In the Active Oxygen Method (also called Swift stability test), the time in hours for a sample of fat to acquire a peroxide value of 100 is measured when bubbling air through at 100°C.

In the ASTM (American Society for Testing and Materials) and DIN (Deutsche Norm) Bomb Tests, oxidation of the fat is done at 100°C and at an initial oxygen pressure of up to 7 atmospheres in a closed system. As soon as the reduction in oxygen pressure in time exceeds a certain value (e.g., 0.15 atm in 15 or 30 min), the breakpoint in the oxygen absorption curve (i.e., the end of the induction period) is thought to be reached.

There is much controversy about the value of these tests for predicting fat keepability[9]. They are useful for comparing the susceptibility to oxidation of different batches of the same oil (or oil blend), but not for comparisons between different oils.

Filter-Paper Tests[5]

To obtain a quick impression about the susceptibility of a certain fat to oxidation, filter-paper tests can be made. The fat sample under investigation is adsorbed onto filter paper, which then is subjected to storage under various conditions (e.g., 100°C in the dark or room temperature in diffuse daylight). The progress of oxidation can be followed by determining peroxide values or by utilizing spray reagents that are sensitive to peroxides. Since such methods are difficult to standardize, they give only a very rough estimate of the susceptibility to oxidation.

MEASUREMENT OF FAT OXIDATION IN EMULSIONS

Relatively few published data are available on the common food emulsions.

Oil-In-Water Emulsions

Mayonnaises and salad dressings are very prone to off-flavor formation, not only because they usually contain rather unstable oils like soybean oil, but also because of the presence of egg-yolk phospholipids. Their relatively low pH is a further complicating factor.

From our own experience we know that only well-refined oils of prime quality can be used in these products to meet the shelf-lives required (e.g., 3 months at room temperature).

No good objective method for predicting mayonnaise keepability is available as yet.

Oxidation of the lipids in fluid milk and milk products has been of great concern to the dairy industry. Both the phospholipids and the triglycerides present in these products are susceptible to oxidation, attack of the phospholipids predominating in emulsified systems, triglyceride oxidation in water-free systems like butter oil or dried milk products[13].

An impressive number of off-flavor compounds occurring in a variety of milk products has been identified, these being

associated with such flavor descriptions as oxidized, cardboard-like, oily, painty, fishy, grassy, metallic, and beany. Chemically, they are saturated and unsaturated aldehydes and ketones, the formation of which is explained by the autoxidation mechanism discussed earlier[14,15]. It should be noted that most of these compounds occur already at peroxide values lower than 1.

No studies have been made of the oxidation rate of liquid milk. Usually, the microbial stability is too low to give shelf-lives appreciably over 1 week, a time-span that would seem necessary for developing oxidative rancidity. Only if the metal content in the milk were to exceed by far the normal levels of 40 µg copper and 250 µg iron per liter would the development of off-flavors be faster. With the use of non-corrodible equipment, very high Cu and Fe levels are rare.

In general, heat treatment of fluid milk products, either for extending pasteurization (milk, cream) or for preparing derived products such as condensed milks, makes the products less susceptible to the development of oxidized flavors. This phenomenon is attributed to a decrease in the redox potential of the system because of the formation of free sulfhydryl groups[16]. The flavor in such systems is similar to that of caramel and cooked milk rather than to that of oxidative origin. No studies have been reported on objective tests for lipid stability in these products.

Also, in creams stored at -15 to -25°C, development of oxidized flavor is not problematic, storage times of 6 months to 1 yr not uncommon.

The effect of storage of an UHT (ultra high temperature) cream at 4, 10, and 18°C was followed by TBA tests, peroxide determinations, and taste-panel scores[17]. The interrelationship between TBA values and flavor scores is summarized in Table 4.

TABLE 4 Frequency distribution of creams stored at 4 to 18°C, according to flavor score and TBA values[17].

Flavor score	TBA values (percent)		
	≤ 0.08	0.08 to 0.16	≥ 0.16
Acceptable	90.0	44.5	7.0
Doubtful	6.5	41.0	18.0
Unacceptable	3.5	14.5	75.0
Total	100	100	100
Number of samples	201	110	264

As shown in Table 4, 90 percent of the samples with TBA ≤ 0.08 had an acceptable taste, whereas 75 percent of the samples with a TBA ≥ 0.16 were clearly unacceptable. For TBA values between these limits, the percentage of samples with a doubtful taste score is at its maximum.

Similarly, at peroxide values ≥ 2.0, about 92 percent of the samples were unacceptable, whereas at values < 2.0 some 43 percent were acceptable, but 32 percent still unacceptable.

Finally, in ice cream, no problems with oxidative deterioration are encountered for obvious reasons.

Water-In-Oil Emulsions

The two most important products here are butter and margarine, bakery emulsions and similar products being left out of consideration. Flavor defects occurring in cold-stored ripened cream butter were extensively studied by Badings[15]. Off-flavors are of oxidative origin and usually develop in the following order: metallic, fatty, oily, trainy, fishy, and tallowy. The oily and trainy flavors are considered to be the most offensive.

The main factors influencing oxidative keepability are the pH of the butter serum and the copper content. As to the latter, the copper content of the milk globule membrane seems to be decisive.

The results of Badings are in confirmation with earlier findings of McDowell[18] who, from storage trials with various butters (sweet cream, slightly ripened cream, fully ripened cream) at -10°C for up to 8 months, derived the following recommendations to ensure an acceptable flavor after storage:

unsalted butter:	pH > 5.2
	Cu < 0.12 ppm
	Fe < 0.50 ppm
salted butter (1.5 percent):	pH > 6.0
	Cu < 0.08 ppm
	Fe < 0.50 ppm

Though the number of experiments was too small, Badings' results with TBA value determinations on butters with different Cu contents, stored for up to 5 months at -10°C, would seem to suggest that TBA values parallel the intensity of the off-flavor perceived.

The possibility of predicting the stability of butter during storage at freezing temperatures by means of an accelerated storage test of the Swift type was investigated by Cherney *et al.*[19]. Samples that reached peroxide values ≤ 0.016 in this test retained their quality upon storage for 6 to 12 months. Samples with peroxide values > 0.022 deteriorated during that period.

Similarly, carbonyl-value determinations during 1 month aging at 20°C were found to be useful for predicting storage stability of butter at -10°C[20].

One of the more recent studies on the effect of storage of butter fat at temperatures ranging from -27°C to +50°C revealed that autoxidation flavors developed at all temperatures, but tended to be more intense at the lower temperatures (-27°C, -10°C)[21]. Peroxide and TBA values were found to correlate well with the flavor scores for the samples kept at higher temperatures but not for the cold-stored samples. In fact, for the latter, hardly any increases in peroxide and TBA values were detectable.

Very few studies have been done on objective determination of the oxidative keepability of margarines[22,23]. Unfortunately, in the investigations published, no simultaneous studies were done on determination of flavor intensity.

Similar to what was observed for butter, a decrease in pH or the addition of salt appeared to favor oxidation[22]. Judging from peroxide values attained, margarine was more susceptible to oxidation than the fat phase when stored alone[23].

In our own laboratory, simultaneous determinations of carbonyl values and flavor scores were carried out during storage of bean oil margarines for up to 6 weeks at various temperatures. Carbonyl values were found to be a poor substitute for taste-panel scoring[24].

CONCLUSION

Objective test methods for fat oxidation would serve two useful purposes: would substitute for taste panels for routine control of food products; would forecast future keepability of these products.

The huge amount of data on oils and fats and the relatively few data on emulsions show that there is no single test available as yet which would be a substitute for taste panels on all occasions.

It would seem to me that there is a good explanation for this. If we consider again the data on TBA values in stored creams (Table 4), we find that we are trying to relate a hedonic rating to what essentially is a measure of the concentration of certain compounds present. Since such pleasantness/unpleasantness scores are highly individual and their dependence on concentration is certainly not linear, overall flavor scoring is inappropriate.

We should ask our taste panels to give information on the intensity of the off-flavor perceived, either in a yes/no answer (i.e., a threshold determination) or as a true intensity scoring.

In the threshold situation, then, we should not expect that our objective method is more precise than an ordinary threshold determination; i.e., also our objective method will rightly or wrongly reject or accept a certain percentage of the samples.

Also, when we ask our taste panels for a true intensity rating, we should be aware of the fact that intensity is related

to the stimulus strength logarithmically, not linearly. The analytical parameter we have chosen to measure stimulus strength is also logarithmic.

Furthermore, the stimulus strength can be related to our chosen parameter in one product, but not in the other -- or can be a good measure only under certain storage conditions.

Objective test methods, therefore, are bound to give us much more limited information than the human sensory system; we should be prepared to accept this.

With these restrictions in mind, what sort of tests should we do on a particular food emulsion to judge its future keepability.

As to the static methods described, we should realize that the data obtained with different methods are fairly well correlated. We can therefore limit ourselves to one or two tests (e.g., UV extinction and anisidine value), which may be supplemented by a determination of copper content or oxygen content.

In addition to this, a dyanmic test will have to be done, although unfortunately none of the methods available is specifically designed for testing food emulsions. On this ground alone they are liable to fail. A new test method is needed in which the emulsion remains intact and the development of off-flavors is not too different from that under normal storage conditions.

For the time being, it seems best to rely upon taste-panel scores. As long as the shelf-life requirements are not unrealistic, the food manufacturer will try to meet those by careful selection of raw materials, by adapting processing conditions and formulation, by restricting the level of susceptible oils, and by proper choice of packaging and distribution.

SUMMARY

Autoxidation in oils and fats and in emulsions seems to follow the same pathways, though differences are apparent in the rate of off-flavor formation and in the degree to which off-flavors are perceived.

The applicability of UV absorption measurements; determination of peroxides, carbonyl compounds, and oxygenated fatty acids; and accelerated storage tests for assessing fat oxidation is reviewed.

Relatively few investigations have been done on objectively testing the keepability of emulsions such as milk, cream, butter, mayonnaise, and margarine.

As yet, no single test is available which can substitute the taste-panel assessment of off-flavors. Causes for this failure are discussed.

A new accelerated storage test specifically designed for emulsions is needed.

REFERENCES

1. A. M. Parsons, Proceedings, 3rd International Symposium on Metal Catalysed Lipid Oxidation, Paris (1973).
2. W. O. Lundberg, *Lipids and Their Oxidation*, H. W. Schultz, Ed., 31 (AVI Publ. Co., Westport, Connecticut, 1962).
3. J. G. Keppler, M. M. Horikx, P. W. Meyboom, and W. H. Feenstra, *J. Am. Oil Chem. Soc.*, *44*, 543 (1967).
4. T. P. Labuza, *Critical Revs. Food Technol.*, *2* (3), 355 (1971).
5. H. Pardun, *Handbuch der Lebensmittelchemie*, J. Schormüller, Vol. *IV* (Lipids) 403, 853 (Springer Verlag, Berlin, 1969).
6. H. Pardun, *Deutsche Lebensmittel-Rundschau*, *62*, 6 (1966).
7. J. Graille and M. Naudet, *Rev. Franc. Corps Gras*, *18*, 609 (1971); *id.*, *20*, 203 (1973).
8. J. H. Pokorný and E. Davidkowá, *Fette. Seifen. Anstrichmittel*, *68*, 91 (1966).
9. G. Hoffmann, *Chem. and Ind.*, 729, 1970.
10. F. Linow, M. Roloff, and K. Täufel, *Fette. Seifen. Anstrichmittel*, *68*, 866 (1966).
11. U. Holm and K. Ekbom-Olsson, 11th Congress International Society for Fat Research, Göteborg, 1972, abs. 100.
12. H. P. Dupuy, S. P. Fore, and L. A. Goldblatt, *J. Am. Oil Chem. Soc.*, *50*, 340 (1973).
13. S. Patton, *Lipids and Their Oxidation*, H. W. Schultz, Ed., 190 (AVI Publ. Co., Westport, Connecticut, 1962).
14. J. E. Kinsella, S. Patton, and P. S. Dimick, *J. Am. Oil Chem. Soc.*, *44*, 449 (1967).
15. H. T. Badings, *Neth. Milk Dairy J.*, *24*, 145 (1971).
16. O. W. Parks, *Fundamentals of Dairy Chemistry*, B. H. Webb and A. H. Johnson, Eds., 197 (AVI Publ. Co., Westport, Connecticut, 1965).
17. W. K. Downey, *J. Dairy Res.*, *35*, 429 (1968).
18. A. K. K. McDowell, *J. Dairy Res.*, *31*, 221 (1964).
19. P. Chernev, R. Rachev, and Kh. Sakhanekov, *Food Sci. Technol. Abstracts*, *5*, No. 8, 1212 (1973).
20. J. H. Labuschagne, *S. Afr. J. Dairy Technol.*, *4*, 171 (1972); *Food Sci. Technol. Abstracts*, *5*, No. 3, 395 (1973).
21. D. L. Hamm, E. G. Hammond, and D. K. Hotchkiss, *J. Dairy Sci.*, *51*, 483 (1967).

22. E. Sambuc, G. Reymond, and M. Naudet, *Rev. Franc. Corps Gras*, *18*, 17 (1971).

23. L. Forman and J. Zajiic, *Fette. Seifen. Anstrichmittel*, *71*, 493 (1969).

24. C. Poot, private communication.

THE TEXTURE OF FATTY OILS AND EMULSIFIED FOODS

Philip Sherman

INTRODUCTION

Oils and fats are utilized in a wide range of food products (Table 1). In some products they are used in their original form while in others, and these probably constitute a larger proportion of the available foods, they are used in emulsified form. The oil or fat is then distributed as microscopic-size drops (dispersed phase) in an aqueous medium (continuous phase) or, alternatively, microscopic-size water drops are distributed in a continuous oil medium. Utilization of oils or fats in emulsified form has at least two major advantages over their use in the original state. First, the oil or fat can be presented to the consumer in a form which has a more acceptable taste and flavor. Second, by emulsification one can build into a product a much wider range of textural characteristics; e.g., pourability, spreadability, spoonability.

TABLE 1 Fatty oil and emulsion-based food products.

Products utilizing oils in original form	Emulsion-based products	
Shortenings (solid, fluid, liquid, and powdered)	Salad dressings	(O/W) *
Cooking oils	Mayonnaise	(O/W)
Salad oils	Butter	(W/O)
Confectionery coatings	Margarine	(W/O)
Peanut butter	Ice cream	(O/W)
	Dairy desserts and toppings	(O/W)
	Low-calorie spreads	(W/O)
	Milk	(O/W)
	Imitation milks	(O/W)
	Cream	(O/W)
	Imitation cream	(O/W)
	Coffee whiteners	(O/W)

*O = oil
W = water

RHEOLOGICAL CHARACTERISTICS

The textural characteristics of oils, fats, and food emulsions will be more readily understood if we first discuss their rheological characteristics. There is a close relationship between the two. There is no sharp distinction between a fat and an oil; the terms merely reflect the state of the glyceride or glyceride mixture at ambient temperature. An oil behaves as a simple liquid and exhibits a shear independent (Newtonian) viscosity; i.e., the shear stress-shear rate relationship derived by viscometry is linear (Figure 1) and the gradient represents the viscosity. Obviously, the oil has the same viscosity at all shear stresses and shear rates.

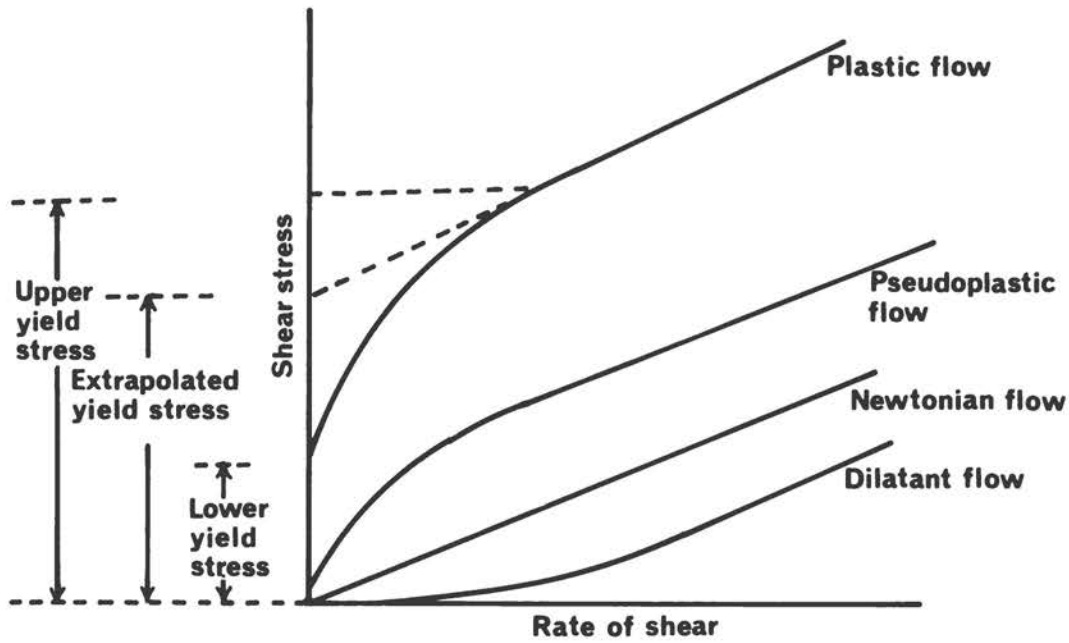


FIGURE 1 Shear stress-shear rate plots for the various types of flow.

When the glyceride(s) is semi-solid or solid, either at room temperature or as a result of chilling, the viscometric behavior is more complex. This is because the glyceride(s) now has a well-developed internal structure in which solid fat crystals are linked together in a three-dimensional network within a continuous fluid phase. Lard is a good example of this state. Intermediate between this state and the completely fluid state, we have fluid shortenings in which hard fat or solid emulsifiers are dispersed in a fluid medium, and food emulsions. Both types of product are dispersed systems in which the dispersed particles exhibit a

tendency to come together and form an interlinked network as in lard. In general, the concentration of dispersed particles is much lower than in lard so that the network structure is weaker in fluid shortenings and food emulsions.

Food emulsions such as butter, margarine, and ice cream contain crystalline material due to partial crystallization of oil (butter and margarine) or water (ice cream) during passage through the low-temperature votator in processing. The internal structure of these products depends, therefore, upon both the emulsified liquid drops and fat crystals.

When the dispersed particles are present in sufficient concentration to be able to link up together, and this can occur at quite small-volume concentrations, food emulsions no longer behave as Newtonian systems. Shear stress and shear rate do not exhibit a simple linear relationship; instead, the relationship is now curvilinear and the flow characteristics are described as non-Newtonian. The two categories of non-Newtonian flow generally encountered in food emulsions are pseudoplastic and plastic (Figure 1). In both categories the gradient of the shear stress-shear rate plot decreases as the shear rate increases, so that the viscosity decreases as the shear rate increases (Figure 2). Thus, systems exhibiting either of these types of flow cannot be defined by a single viscosity. Instead, we refer to either an "apparent viscosity" at a particular rate of shear or to the viscosity at selected shear rates.

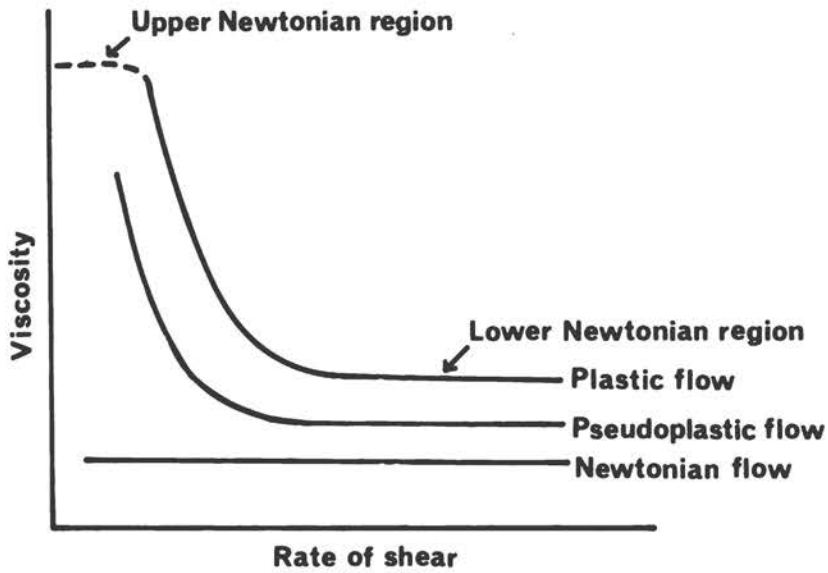


FIGURE 2 Viscosity-shear rate plots for the various types of flow.

The principal difference between plastic and pseudoplastic flow is that, in the former, a certain shear stress (yield stress) has to be applied before flow commences, whereas, in the latter, flow is initiated at very small shear stresses. Two other yield stresses are often referred to in the literature. These are the upper-yield stress, which is the value of the shear stress at which the flow curve becomes linear (Figure 1) and the extrapolated-yield stress, which is the shear stress obtained by extrapolating the linear part of the flow curve back to the shear-stress axis. When viscometric data are plotted as in Figure 2, it is difficult to differentiate between plastic and pseudoplastic flow. Ideally, plastic-flow curves show two regions in which the viscosity is independent of shear rate, i.e., upper and lower Newtonian regions, whereas pseudoplastic-flow curves show only a lower Newtonian region. Unfortunately, commercial viscometers do not operate at sufficiently low shear rates to examine the upper Newtonian region in detail, and other techniques are required to do this.

Food emulsions with intermediate concentrations of dispersed phase usually exhibit pseudoplastic flow. When the dispersed-phase concentration is high, flow becomes plastic.

Appendix A lists equations commonly used to define the relationship between shear stress and shear rate in pseudoplastic and plastic flow.

A third category of non-Newtonian flow is found in some concentrated dispersions. This is dilatant flow (Figures 1 and 2), in which the gradient of the shear stress-shear rate plot increases as the shear rate is increased. When a system of this type is sheared, the closeness of packing of the dispersed-phase particles diminishes initially, leading to a volume increase so that the particles can move over one another. The author is not aware of any food emulsion that exhibits dilatant flow.

Appendix B lists some of the commercially available viscometers that are suitable for studying non-Newtonian as well as Newtonian flow.

INTERNAL STRUCTURE

Figure 3 shows the shear-rate dependence of the viscosity of milk. Milk is an O/W (oil-water) emulsion in which the fat particles constitute a little over 4 percent of the total volume. Yet, in spite of this low-volume concentration of dispersed phase, the viscosity at 4.5°C falls drastically from around 40 poise at a very low shear rate to 1-2 poise at about 120 sec^{-1}. By the time the shear rate has reached 25 sec^{-1}, this sharp decrease in viscosity is almost complete[1]. The viscosity of milk is independent of shear rate, i.e., the flow is Newtonian, when its temperature exceeds 40°C.

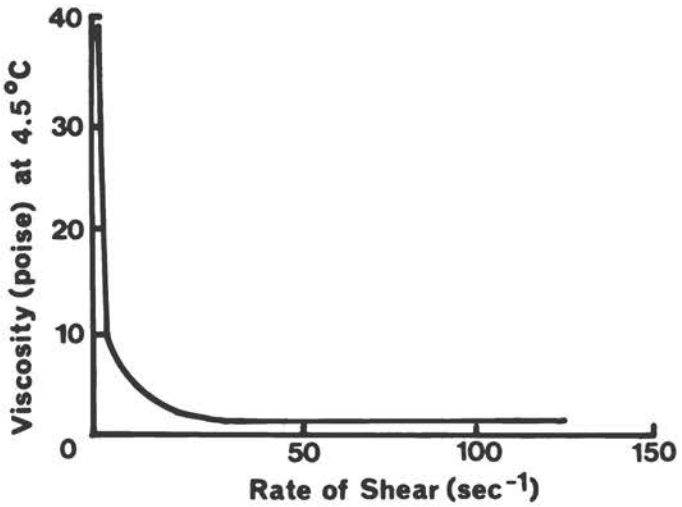


FIGURE 3 Viscosity-shear rate data for milk at 4.5°C.

Temperature also influences the rheological characteristics of fats. Table 2 provides some typical data for mixtures of GNO [ground nut (peanut) oil] with fractions of the same oil after hardening[2]. The percentage of hardened GNO increases from 0 to 50 in samples 1 to 9. Rheological data were obtained on these samples with an extrusion capillary viscometer. The extrapolated yield stress and the apparent viscosity were derived from the shear stress-shear rate data (from the linear portion of the plot).

It is apparent from the data in Figure 3 and Table 2 that under certain circumstances both food emulsions and fatty oils have pronounced internal structure. The dispersed particles, whether they are fluid drops or fat crystals, interlink to form a three-dimensional network structure in which the particles are held together by van der Waal's attraction forces[3]. When a shearing stress is applied to these systems, the network structure breaks down to an extent that depends upon the magnitudes of the applied stress and of the attraction forces between the dispersed particles. The lower Newtonian region in the viscosity-shear rate plots given in Figures 2 and 3 indicates complete rupture of internal structure. On the other hand, the upper Newtonian region in Figure 2 indicates that the distance between particles in the network structure increases slightly as the shear stress increases. However, the displacement is not sufficient to overcome the attraction forces between the particles and there is, as yet, no structure breakdown.

The three different yield stresses that can be measured in plastic flow (Figure 1) can be used as a measure of the internal structure in the sense that the higher the yield stresses, the stronger the internal network structure. This conclusion is obviously of limited value. More useful information about the structure in the stationary state can be derived by carrying out creep compliance-time studies at very low shear stresses, or dynamic studies involving transient application of stress[4] so that structural damage is minimized.

TABLE 2 Influence of temperature on the rheological properties of mixtures of unhardened and hardened GNO.

Fat mixture	Melting point (°C)	Extrapolated yield stress (dyne cm^{-2})				Apparent viscosity (cp)			
		16°C	20°C	24°C	28°C	16°C	20°C	24°C	28°C
1	--	0	0	0	0	98	81	68	57
2	32.4	0	0	0	260	1210	1130	968	273
3	38.7	3875	1950	1000	1345	2190	1790	2030	400
4	41.2	8750	2600	--	930	2750	4000	--	1400
5	43.1	16450	10800	3720	2330	7280	3430	3920	2150
6	44.3	25700	13650	9050	5250	7060	8830	3730	4960
7	45.7	42400	31200	23550	15600	9680	8830	2670	1090
8	47.7	76600	58200	48600	30600	83400	49000	19400	11100
9	49.5	145800	115800	97500	70100	177000	79000	27900	18000

TEXTURAL CHARACTERISTICS

There is still much confusion regarding the textural characteristics that contribute to an acceptable fat or food emulsion product. This is due, at least in part, to the existing problems over terminology. A recent statement[5] that typifies the present situation reads as follows: "Mayonnaise quality is difficult to assess properly . . . Viscosity becomes meaningless in attempting to relate mayonnaises of different formulation and process history to each other. Other attributes enter into the picture. One such attribute may be called 'body.' Two mayonnaise products can have identical viscosity measurements. However, one can be removed from the container a spoonful at a time without change in firmness throughout the operation. The other mayonnaise could become pourable before half the jar is emptied. The first has a firm body, the second a soft one. Texture is another attribute. The mayonnaise should be a rigid gel. On storage the . . . rigidity will show transformation to a softer and glossier texture. Such change will become apparent within 1 to 2 months. However, the . . . viscosities can remain constant throughout these obvious textural changes." Within this short narrative, 5 different textural terms have been used -- viscosity, body, firmness, gel, and texture -- and no attempt is made to define their usage or to differentiate between them. In the dairy industry the situation has become even more confusing with a product being regarded as of good textural quality because structural faults are absent[6].

Viscosity appears to be regarded by consumers as the most important textural characteristic of fatty oil and emulsion food products. In a few instances, stickiness may also be exhibited. In the case of a product that exhibits Newtonian (shear-independent) flow, there is no problem over the use of the term viscosity. However, when the product exhibits non-Newtonian flow, the situation is more complicated. The perceived viscosity now depends upon the shear stress and shear rate to which the product is subjected when in the mouth, when poured from a bottle, when stirred, when spread, etc., during evaluation. The shear rates and shear stresses associated with some of these modes of evaluation have been determined by analyzing sensory and viscometric data on a wide range of food samples and appropriately selected standards for comparison[7,8]. The results indicate not only that different shear stresses and shear rates are associated with consumer evaluation of viscosity by different methods, but also that substantial variations may be observed when evaluating viscosity by any one procedure (Table 3). For example, the shear stresses and shear rates associated with viscosity evaluation by the tilting-a-container method, or by mouthability, depend upon the rheological characteristics of the samples being examined. Viscosity evaluation by stirring depends much less on the sample's rheological characteristics.

TABLE 3 Summary of shear-rate and shear-stress ranges associated with consumer evaluation by various methods[7].

Sensory method for viscosity evaluation	Approximate range of shear stresses associated with viscosity evaluation (dyne cm^{-2})	Approximate range of shear rates associated with viscosity evaluation (sec^{-1})	General form of sensory shear stress-shear rate plot
Stirring contents of a container	$10^{1.5}$-$10^{4.0}$	$10^{1.5}$-$10^{2.0}$	approximately linear
Tilting container	$10^{2.0}$-$10^{3.0}$	$10^{-1.0}$-$10^{2.0}$	approximately linear
In the mouth	$10^{2.0}$-$10^{4.5}$	$10^{1.0}$-$10^{4.0}$	non-linear

The complexity of the evaluation mechanics when examining non-Newtonian samples can be demonstrated further by reference to Figure 4. This shows a "master curve" that delineates the limits of shear stress and shear rate associated with the sensory evaluation of viscosity in the mouth for a range of food products with different flow characteristics. Superimposed on this "master curve" are the viscometrically derived shear stress-shear rate data for some fatty oil and emulsion food products. The viscometric data for peanut butter and chocolate spread intersect on the master curve and then cross over. This means that these two products will appear to have the same viscosity in the mouth. This viscosity is given by the shear stress-shear rate ratio at the point of intersection. On the other hand, the viscometric data for condensed milk and yoghurt intersect to the right of the "master curve," so that in the mouth yoghurt appears more viscous than condensed milk.

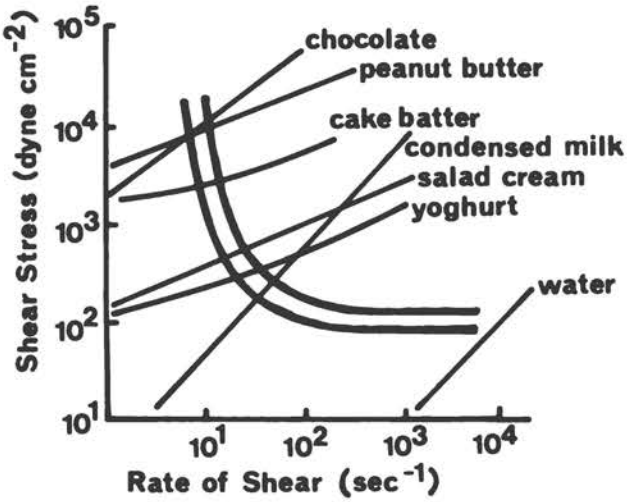


FIGURE 4 Shear stress-shear rate conditions associated with viscosity evaluation in the mouth.

Provided the sample is not too viscous, oral evaluation of viscosity depends on the ease with which it flows between the upper surface of the tongue and the roof of the mouth[7,9]. Discussions with panelists indicated that evaluation of viscosity by stirring depends on the resistance offered to the stirring motion; viscosity evaluation by tilting the container is based on the rate at which the sample first flows down the side of the container and not upon the flow rate of the bulk of the sample.

Figure 5 shows in detail the shear stresses and shear rates associated with viscosity evaluation by stirring and by tilting the container. For comparison purposes the data for oral evaluation of viscosity are also included. By superimposing the instrumental shear stress-shear rate data for a particular sample over these three plots, and noting the shear stress and shear rate at the point of intersection with each graph, one can predict

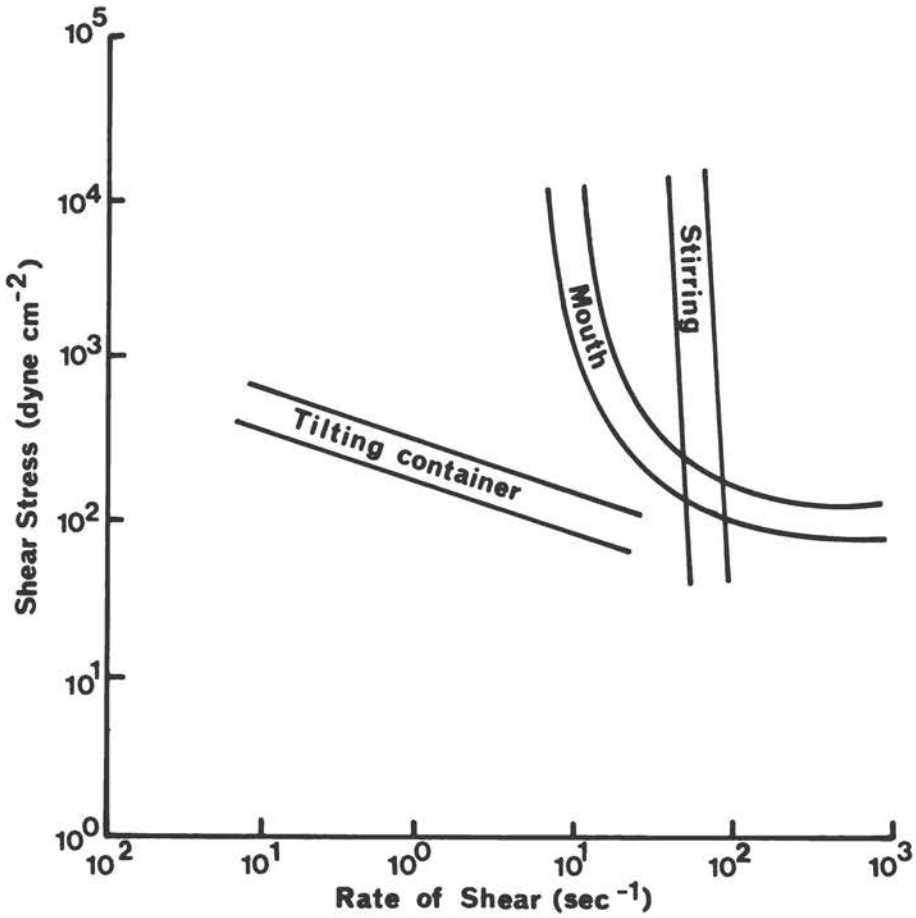


FIGURE 5 Shear stress-shear rate conditions associated with viscosity evaluation by different sensory methods.

the consumer's evaluation of the viscosity (shear stress-shear rate ratio at each intersection) by each of the three procedures. It should also be possible to use these principles to establish what the consumer regards as the most desirable flow characteristics in a product.

Highly viscous samples suffer less structural disintegration than more fluid samples during sensory evaluation and this could explain why descriptive terms other than viscosity have sometimes been used to describe their textural characteristics, as, for example, in the extract quoted previously.

The spreading characteristics of fatty oil and emulsified foods have not been examined but analogous studies on cosmetic creams spread on human skin have shown that here also the shear stress-shear rate conditions associated with the evaluation vary according to the flow characteristics for the sample[10].

Non-emulsified fatty oil products may not spread spontaneously over the internal surfaces of the mouth or other surfaces as do emulsion-based products. In such instances an additional factor, the spreading coefficient, may influence viscosity evaluation. The spreading coefficient (S) is given by

$$S = \gamma_{s/a} - \gamma_{s/1} - \gamma_{1/a} \cos \phi$$

where $\gamma_{s/a}$, $\gamma_{s/1}$, $\gamma_{1/a}$ are the respective surface tensions between the surface and air, the surface and the sample, and between the sample and air; ϕ is the contact angle.

CONCLUSIONS

The most important textural attribute of fatty oils and emulsified foods is viscosity. The criteria on which its sensory evaluation is based are closely related to the rheological (flow) properties, and in particular to the shear stress developed at the shear rate to which the sample is exposed during the evaluation procedure. Fatty oils exhibit Newtonian flow at ambient temperature, i.e., the shear stress-shear rate ratio is constant, so that the mechanics of viscosity evaluation are relatively simple. However, fatty oil products, which contain both solid fat and liquid oil, and most food emulsions exhibit non-Newtonian flow, i.e., the shear stress-shear rate ratio is no longer constant, and the mechanics of viscosity evaluation are much more complicated.

The shear stress and shear rate associated with the evaluation change not only with the method of evaluation but also with the inherent flow properties of the sample.

REFERENCES

1. H. Randhahn, *Milchwissenschaft, 28*, 620 (1971).
2. P. Soltoft, *On the Consistency of Mixtures of Hardened Fats*, 79, 103 (Bjarne-Kristensen, Copenhagen, 1947).
3. M. van der Tempel, *J. Colloid Sci., 16*, 284 (1961).
4. P. Sherman, *Ind. Rheology*, 23, 56 (Academic Press, London and New York, 1970).
5. T. J. Weiss, *Food Oils and Their Uses*, 161 (AVI Publ. Co., Westport, Connecticut, 1970).
6. J. H. Prentice, *J. Texture Studies, 3*, 415 (1972).
7. F. Shama and P. Sherman, *J. Texture Studies, 4*, 111 (1973).
8. F. Shama, C. Parkinson, and P. Sherman, *J. Texture Studies, 4*, 102 (1973).

9. F. W. Wood, *Rheology and Texture of Foodstuffs*, 40 (S.C.I. Monograph No. 27, 1968).

10. B. W. Barry and A. J. Grace, *J. Pharmacol. Sci.*, *61*, 335 (1972).

APPENDIX A

Equations used to define Newtonian and non-Newtonian flow

1. Newtonian flow

$$\dot{\gamma} = \frac{1}{\eta} (\tau)$$

2. non-Newtonian flow

a) no yield stress

1) $\dot{\gamma} = \frac{1}{\eta_p} (\tau)^n$ Ostwald-de Waele

When $n > 1$, viscosity decreases as τ increases; when $n < 1$ viscosity decreases as τ increases (dilatant flow)

2) $\eta_p = \eta_r (\dot{\gamma})^{n-1}$ Van Wazer, Lyons, Kim, and Colwell

$n = 0 - 1$ for pseudoplastic flow, and $1 - \infty$ for dilatant flow

b) with yield stress

$$\dot{\gamma} = \frac{1}{\eta_p} (\tau - \tau_0)^n$$ Herschel and Bulkley

c) linear region of shear stress-shear rate curve

$$\eta_{app} = \frac{\tau}{\dot{\gamma}}$$

Where $\dot{\gamma}$ is the shear rate, τ is shear stress, η is the viscosity, η_p is a parameter corresponding to viscosity, η_r is a reference viscosity at unit rate of shear, η_{app} is the apparent viscosity at a reference shear rate, and n is a constant.

APPENDIX B

Commercially available viscometers that are suitable for investigating Newtonian and non-Newtonian flow

a) Moderately priced instruments

Haake 'Rotovisko'
Contraves 'Epprecht'
Ferranti cone-plate
Brookfield synchro-electric (with cylindrical bob)

b) Expensive instruments

Weissenberg rheogoniometer
Rheometrics mechanical spectrometer
Instron cone-plate

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

THE STABILITY OF FATS AND OILS IN FOODS

G. A. Jacobson, D. J. Aldridge,
and J. A. Kirkpatrick

INTRODUCTION

It has been said in various ways that nothing in this world stays the same. This axiom is certainly true of the flavor of foods. Most food manufacturers are well aware that changes in flavor start during growth and development of either plant or animal ingredients of food, and continue as the food is processed. The final changes in flavor take place during the storage phase before the food is consumed. If all of a food is not consumed at a meal, then one can encounter what has been called "warmed over flavor" which is not only important in the home, but has importance for those who manufacture frozen prepared foods.

Meaningful studies of the effects of lipids on the flavor of foods can be made during any or all of the stages mentioned previously. For example, Kazeniac and Hall[1] and Stone, Hall, and Kazeniac[2] have shown by isotope studies that the enzyme systems of the tomato convert the linolenic acid of that vegetable into a number of key flavor components, such as cis-3-hexenal and cis-3-hexenol during processing. These and related aldehydes and alcohols are extremely important to the desirable flavor of tomato juice and other products containing tomatoes. Similarly, Tressl and Drawert showed that the ripening of bananas involves enzymatic changes in fatty acids to form esters, ketones, alcohols, and aldehydes. Specific aldehydes formed enzymatically from linoleic and linolenic acid as the bananas ripened included hexanal, trans-2-hexenal, trans-2-nonenal, and trans-2,cis-6-nonadienal[3]. Although the flavor role of these potent low-threshold flavor components of fruits is not always clear, there is little uncertainty about their negative role in the flavor acceptance of fats and fat-bearing foods. Another example of how early enzymatic changes are important in ingredient quality is in the harvesting and processing of the oil palm fruit. Avoidance of cell rupture and fruit damage during harvest can have a profound effect on color and free fatty acid development in the extracted palm oil. The free fatty acid content in a bruised fruit can reach a level as high as 60 percent in a short time at tropical temperatures, with proportionate reduction in the flavor and color stability of

the oil after refining. These pre-processing enzymatic changes are just a few of the examples that could be cited of factors that affect the flavor and flavor stability of finished food products. Other factors will be examined below.

Many different types of lipid-bearing foods are available in the market place today, and each one has its own flavor-stability characteristics. For example, the quality and stability of fried products are both highly dependent upon the type of fat or oil used as a frying medium, as well as its history in terms of frying equipment and time and temperature of use. An abused frying fat or oil will not only result in unpleasant fried-food flavor, but also may have questionable nutritive properties. In baked goods, the shortening or margarine used can have an appreciable effect on flavor stability as well as on the nature and rapidity of the physical changes of the staling process. Fats and oils used in canned foods must be bland in flavor before canning, since quantities in the parts-per-million range of such off-flavors as hexanal will interfere with flavoring materials (such as spice components) present at similar levels. In frozen prepared foods, the fat or oil used must not only be bland initially, but must have enough potential flavor stability to resist changes in flavor that arise from the formation of secondary oxidative decomposition products such as aldehydes and ketones. In any of the product applications mentioned, the onset of unwanted flavor changes can be retarded by antioxidant materials such as the tocopherols present in the oils, the addition of synthetic antioxidants, browning reaction products, flavonoids, or the addition of certain spices.

METHODS OF STUDYING FLAVOR AND FLAVOR STABILITY IN FOODS

In some foods, the gradual decline in the quality of flavor can be measured by following the level of a few key flavor components. For example, the TBA test, benzidine test for aldehydes, anisidine value, and carbonyl value can be useful in measuring carbonyl-type off-flavors in fats and oils or in many other foods[4-7]. Their value lies in the fact that a given lipid system will usually produce the same type or proportion of carbonyl-type off-flavors, providing the preparation and storage conditions are constant; i.e., a chicken pot pie held in the freezer will develop a specific array of carbonyl-type flavor components during aging, and roasted peanuts held at room temperature will develop another characteristic array of components. The various forms of chromatography have been invaluable for separating key volatile flavor components for identification and quantification. This instrumental approach permitted the identification of hydrocarbons, such as pentane, which was predicted by Evans to occur in the autoxidation of oils containing linoleic acid[8]. Mookherjee, Deck, and Chang studied the changes in flavor components of potato chips as the chips became stale from aging in air at room temperature by

using liquid-liquid partition chromatography to separate the 2,4 dinitrophenylhydrazine derivatives formed[9]. These workers found that the decline in flavor quality of the chips was accompanied by a reduction in the level of 2,4 decadienal and the formation of such compounds as hexanal, 2-hexenal, and alkanones. This progressive sequence appears to apply in many types of fried products. More recently, Warner *et al.* studied flavor changes in potato chips aged at 60°C in a closed jar equipped for gas-liquid chromatography (GLC) headspace analysis. After 20 days, the pentane level in the headspace was 0.03 ppm, with 69 percent of the panel describing the chips as rancid at this point[10]. While there is little doubt that the pentane level was helpful as an indicator for evaluating the flavor quality of these chips, it would be interesting to see whether the pentane level would have the same usefulness if the chips were held at room temperature instead of 60°C. Fuller and co-workers did in fact find that GLC headspace analysis did not reflect deterioration of potato chips fried in various oils and aged at room temperature in jars sealed in air and exposed to fluorescent light[11]. At least part of the difference in the findings of these 2 groups of workers might be due to the light exposure used by Fuller *et al.*

Dupuy has devised a unique GLC method for following off-flavor development in peanuts and several food oils, based again on the measure of key volatiles such as hexanal, methyl butanal, and pentane[12]. In this method, volatiles are swept by the carrier gas from the dispersed heated sample directly onto the GLC column for separation. Correlation coefficients between the GLC peaks of specific volatiles and flavor scores of some materials were significant at the 99 percent confidence level. Lin, Smouse, and Allen have developed a method of evaluating flavor volatiles of oils where the volatiles are removed *in vacuo* and condensed in a U-tube cooled by liquid nitrogen[13]. This U-tube is then connected to the gas chromatograph for vaporization of the trapped volatiles and subsequent separation. The application of this method to a series of fresh oil samples revealed a series of 15 GLC peaks. It was found, however, that 2 peaks in particular correlated well with the flavor scores of 4 of 6 oils tested. Application of the Lin, Smouse, Allen method to potato chips or the oils in which the chips were fried, yielded many peaks that appeared to affect the total flavor impression. Lin and co-workers sorted out these peaks by determining correlation coefficients, peak by peak, with flavor scores. Those peaks that correlated positively with flavor scores varied in number in potato chips fried in various oils, but there were generally many more peaks that correlated negatively with flavor scores than there were that correlated positively (good flavor notes).

I have cited several ways to use instrumental techniques to supplement flavor scores obtained on a sample at a given moment of time. I will now give some additional examples of off-flavor development in various types of lipid-bearing food products, and

then, several methods of predicting flavor stability of foods. In these experiments, the emphasis will also be on flavor evaluation.

MATERIALS AND METHODS

A hedonic-type scoring scale was used in the flavor panels (Figure 1). The following numbers were assigned to the scale for statistical evaluation: 10 = excellent, 8 = good, 6 = fair, 4 = poor, and 2 = very poor.

Conjugated Diene Values were determined by American Oil Chemists' Society (AOCS) method Ti-1a-64.

AOM Values were determined by AOCS method Cd-12-57 to a peroxide value end point of 125 me/kg.

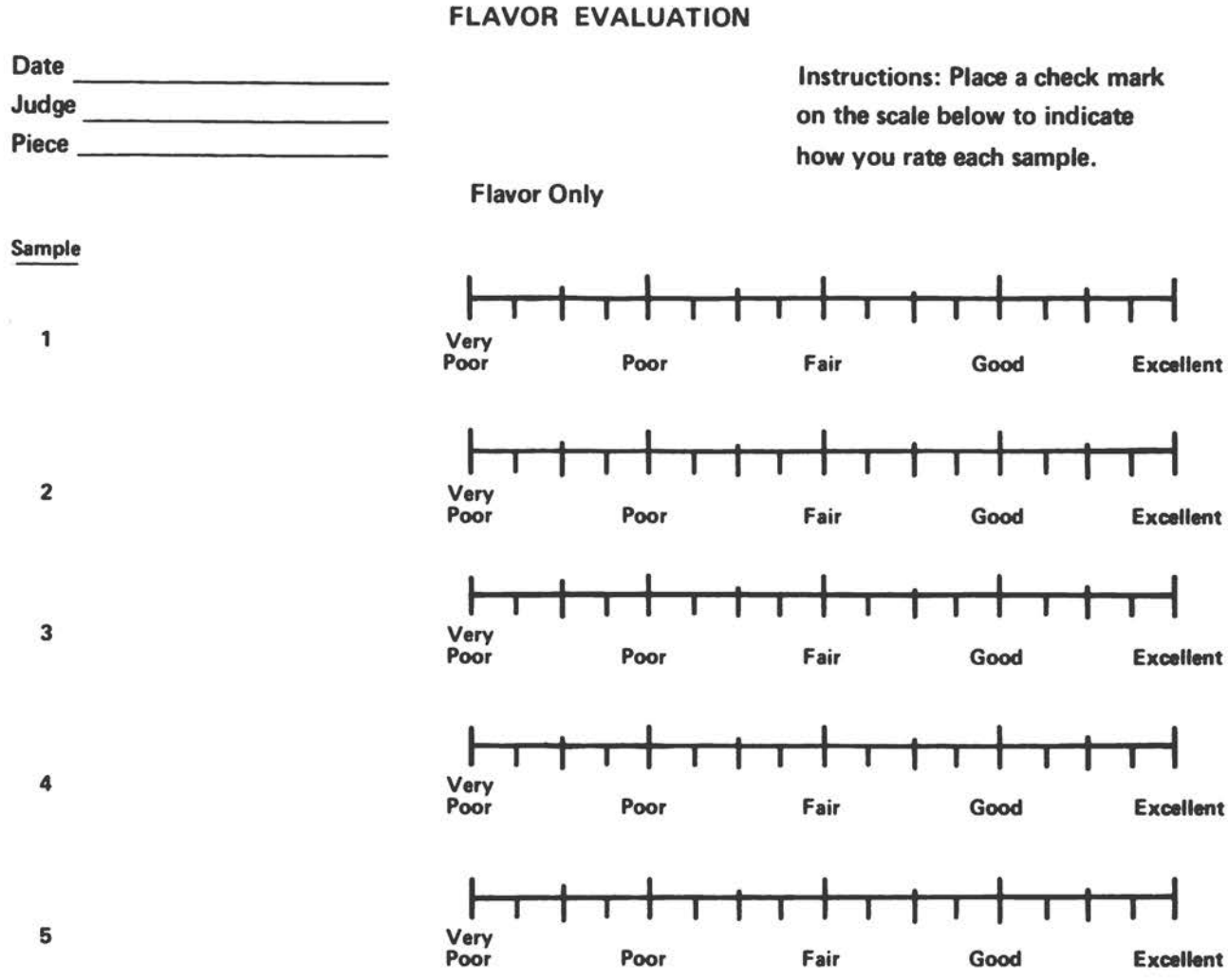


FIGURE 1 Flavor evaluation score sheet.

TBA Values on meat products were carried out by the method of Turner *et al.*[14] modified to permit the reaction to be carried out in a sealed milk-dilution bottle.

TBA Values for the shrimp-breading study were determined as described below. Duplicate 20 g samples were placed in a 1000 ml Florentine flask, to which was added 200 ml distilled water, 100 ml of 20 percent (W/V) trichloroacetic acid solution and 5 ml of 1:2 (V:V) concentrated hydrochloric acid:distilled water. A water-cooled condenser was attached to the flask and the contents were heated to boiling and stirred magnetically at a moderate speed for 20 min. The samples were removed from the heat, cooled for 5 min in an ice and water bath, and the condenser was removed. The sample solution was transferred to a stoppered Erlenmeyer flask for further cooling and then filtered through Eaton-Dikeman 617 and Whatman No. 1 filter paper successively. Ten ml of TBA reagent was then added to the clarified solution, held in a milk-dilution sample bottle. The bottle was sealed tightly and heated in a boiling water bath for 35 min. The samples were then cooled in running tap water for 10 min and the optical density was read at 530 nm against a reagent blank. The TBA reagent consisted of 0.02M 2-thiobarbituric acid in 90 percent acetic acid.

RESULTS AND DISCUSSION

Margarines are widely used in the food industry, and their potential flavor stability is highly dependent upon the potential flavor stability of the fats and oils used to prepare them. Of primary concern was the performance of the margarines in frozen products (Figure 2). Several margarines were aged for 8 mo in the freezer in a thin layer in covered aluminum trays, then evaluated for flavor. These margarines were made from hydrogenated cottonseed oil. No difference in flavor acceptability was found in the fresh margarines, but after aging, improved flavor stability was observed in the margarine prepared from oil with 20 percent or 30 percent cottonseed oil present. The difference between mean flavor scores of the aged samples containing 0 percent to 10 percent cottonseed oil and those of the other 2 aged samples was significant at the 95 percent confidence level.

The changes just noted were brought about by long-term storage, but more subtle changes can take place much sooner. Specially prepared frozen dinners were aged at 3 temperatures to determine how long it would take for a trained panel of 8 persons to detect a change in flavor at each temperature. The control dinners were held at -18°C. The components of the dinners were peas, a veal parmigiana patty, a muffin, and sliced apples. The time necessary for one or more of the components of the dinner to reach a mean flavor score of 7.0 at the 3 temperatures appeared to be fairly short but proportionately uniform over the temperature range studied (Figure 3). A breakdown by dinner components of the

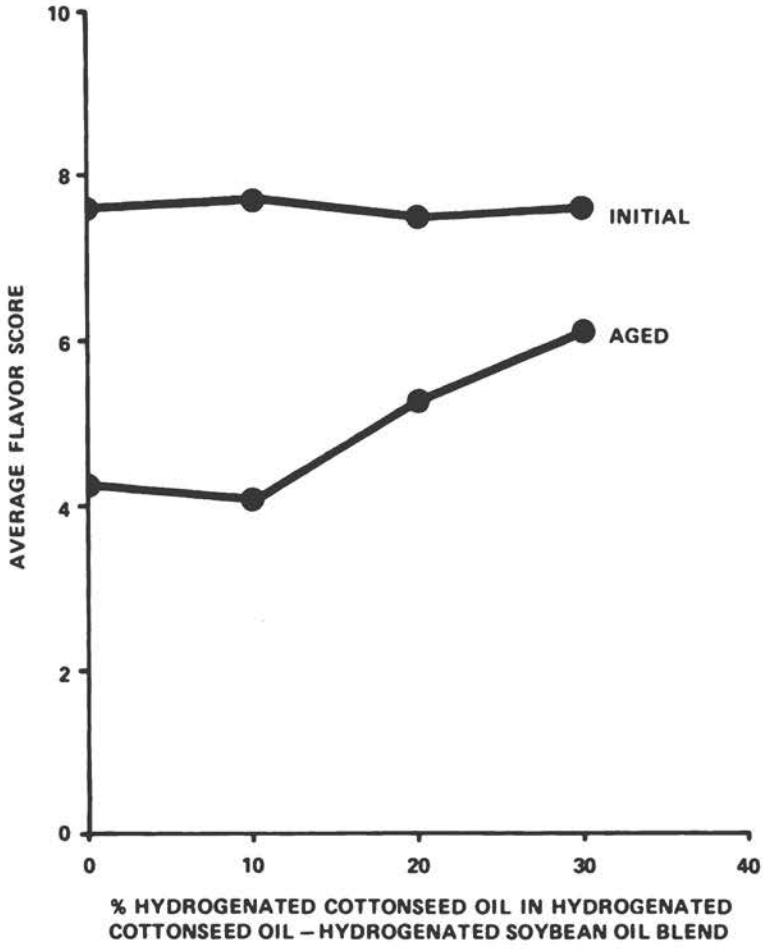


FIGURE 2 Flavor changes in margarine aged 8 months in the freezer.

length of time to reach a mean flavor score significantly different from that of the control held at -18°C showed that almost any of the components can be the weak point (Table 1). These data also illustrate that the holding temperature for frozen foods is extremely critical in maintaining good flavor during storage, whether the food processor or the retailer has the food.

Frozen fried foods pose special problems in storage stability, since some of the desirable flavor components, such as the lactones and dienals, are relatively unstable. Further, if the frying-fat flavor is too strong and unbalanced, the frozen fried product can begin storage with a marginal flavor and get rapidly worse. In a chicken-frying test it was found that the conjugated diene value of frying fat used in a commercial frying operation increased and the flavor scores decreased. Thus, at about 10 frying periods the flavor acceptance of the fat was borderline, and at 20 periods was unsatisfactory (Figure 4). An examination was also made of samples of frying fat from a fried potato processor and it was found that a plot of the conjugated diene value and AOM values for this series of samples yielded a straight line over the range studied (Figure 5). The conjugated

TABLE 1 The time to reach a significant difference in mean flavor score of components of veal parmigiana dinners aged at various temperatures.

Temperature	Aging/Time*	Component
40°F	8 days	Peas
26°F	10 days	Patty, muffin, apples
15°F	18 days	Patty

*Time in days to reach a point where the mean flavor score of the test component is below and significantly different from that of the control component held at 0°F.

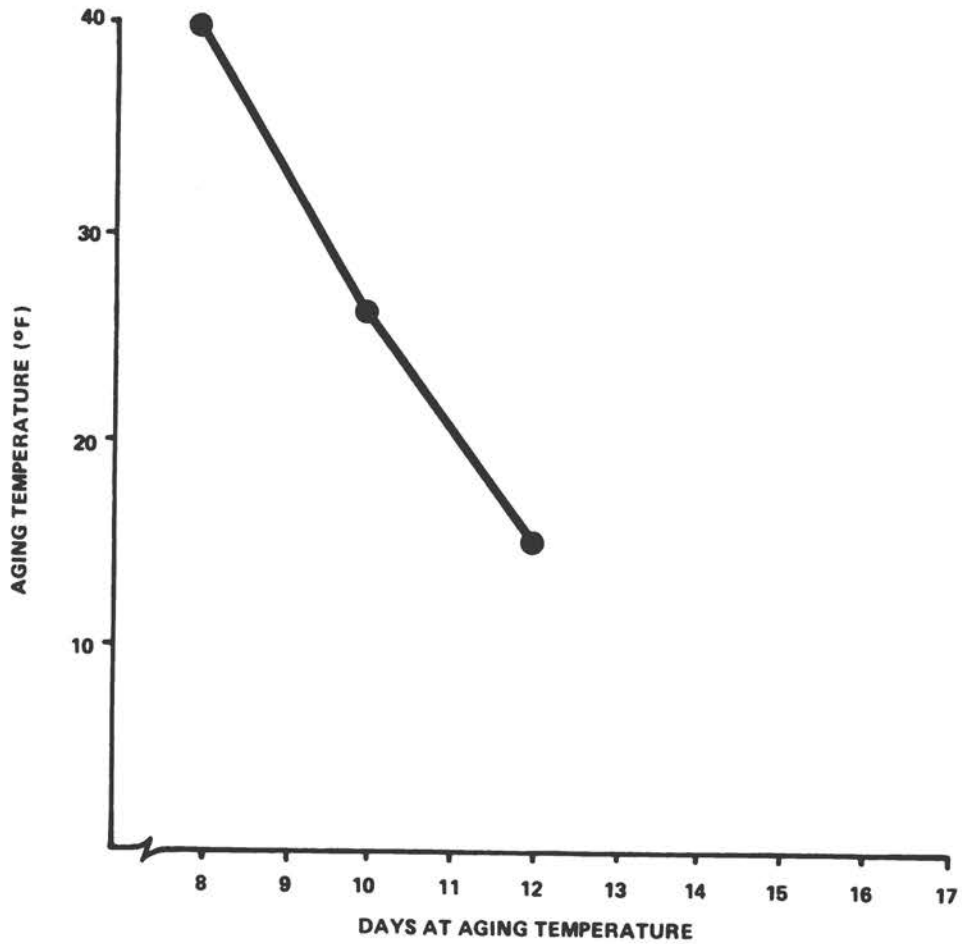


FIGURE 3 Time in days to reach a mean flavor score of 7.0 of components from frozen dinners aged at various temperatures.

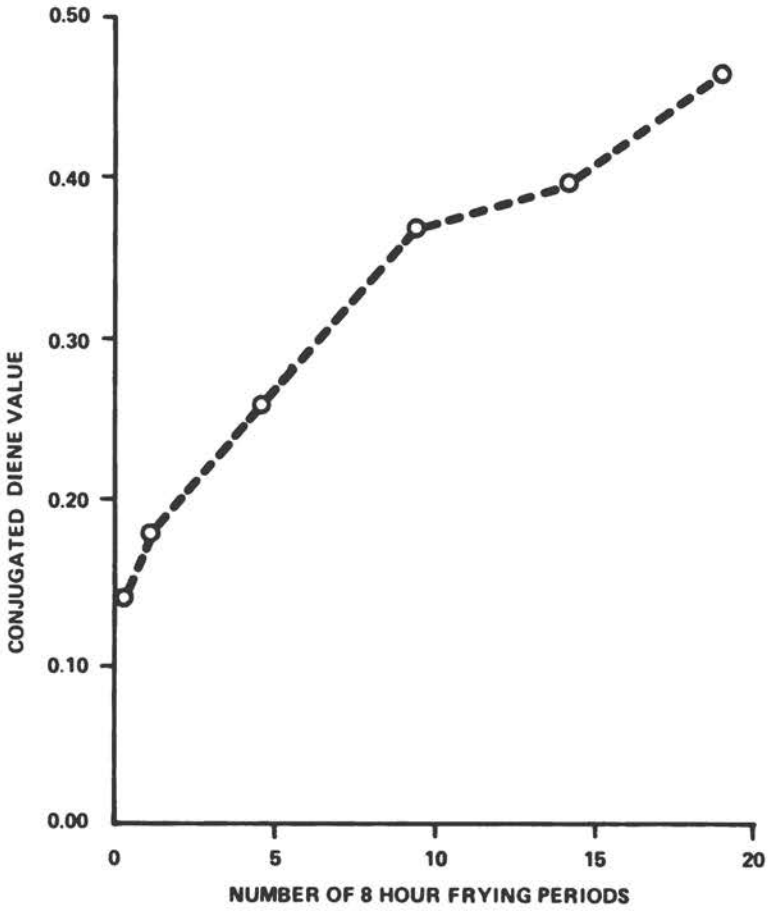


FIGURE 4 Changes in conjugated diene value of frying fat during commercial chicken-frying operations.

diene value also showed a relationship with the flavor of these frying fats that could be useful in the maintenance of good flavor in fried products (Figure 6). It should be remembered, however, that the initial conjugated diene value and the rate of increase during frying will vary from system to system because of the difference in composition of frying fats and oils, the type of food being fried, and the degree of oxidation of the fat during frying.

Dilution of a stable frying fat with a less stable fat from the material being fried in it can have an adverse effect on the heat stability of the frying fat. An example of this is shown by heat-stability profiles formed from a plot of AOM stability measurements of samples held at 190°C under simulated frying conditions for 0, 2, 4, and 6 h (Figure 7).

Canned foods are not immune from flavor problems from fat or oil ingredients, and the most critical point of concern appears to be the flavor of the lipid material just prior to canning. For example, pork fatty tissue for canned pork and beans was aged for 4 mo in the freezer and samples were taken monthly for TBA analysis (Table 2). At 3 mo of frozen storage of the tissue, the flavor of the canned products prepared from it was still satisfactory. After the pork was aged for 4 mo, however, the flavor score

TABLE 2 Changes in pork on aging, as measured by the TBA test, and flavor of canned pork and beans.

Sample number	Length of storage	TBA absorbance	Flavor score
1	1 mo, -12°F	0.11	
2	2 mo, -12°F	0.13	
3	3 mo, -12°F	0.10	7.9*
4	4 mo, -12°F	0.17	6.5*
5	1 week, 38°F	0.55	

*Mean flavor score significantly different at the 99 percent confidence level.

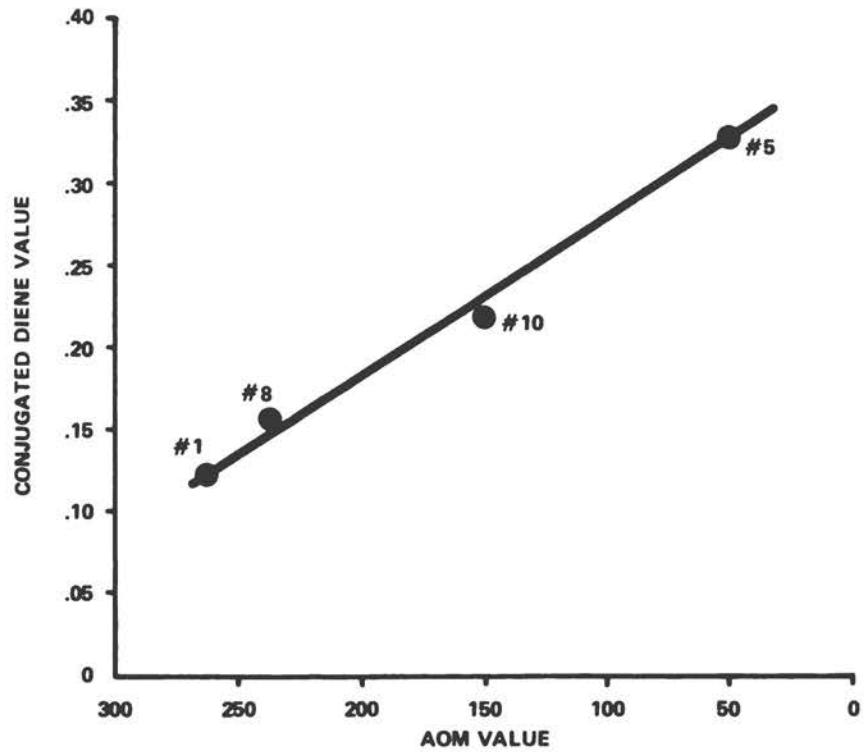


FIGURE 5 Correlation of conjugated diene value with AOM value.

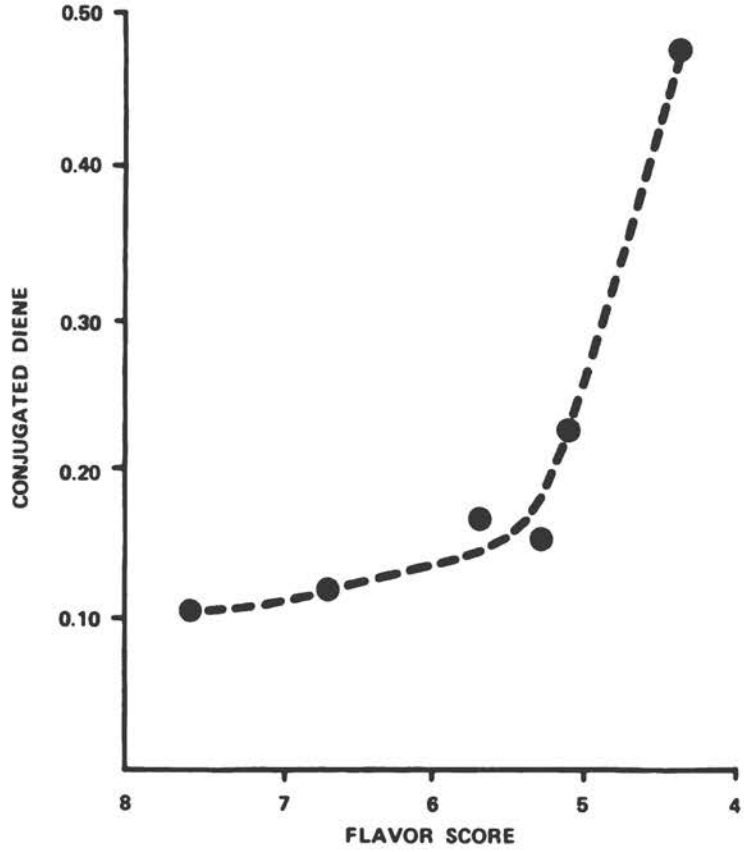


FIGURE 6 The relationship of frying fat flavor to the conjugated diene value.

of the canned product had become unacceptable. This drop in flavor score was accompanied by an appreciable increase in TBA value. Further, the advantage of freezer storage over refrigerator storage is evident when the TBA value of Sample 5, Table 2, is compared to those for tissue aged in the freezer.

Many tests are in use for the prediction of the flavor stability of lipids or lipid-bearing foods. One that was found useful for predicting the potential flavor stability of breading for fried or baked products was derived from the Active Oxygen Method (AOM). In this test, finely divided breading was held at 110°C, while 2.33 cc air/second was bubbled through it. The development of aldehydes was measured by the TBA test. A typical breading with satisfactory potential oxidative stability followed the pattern of aldehyde development shown by the unaged sample (Figure 8). As the breading was held covered in static air at 37°C, TBA values from the accelerated test started higher and developed as shown by the breading samples held in air 4 and 8 days at 37°C. When some of the breading samples were oxidized to a certain point, the TBA values actually declined during the test as shown by the samples held 1 or 2 weeks at 37°C (Figure 9). A plot of the various times under AOM conditions to reach a TBA absorbance of 0.40 for a good sample of breading as it was held

at 37°C showed a fairly uniform decline in potential stability (Figure 10). Thus, the sample held 8 days at 37°C was oxidized enough to reach 0.40 TBA absorbance in less than 1 h under AOM conditions. Two samples of breading that had unsatisfactory flavor stability in fried product had TBA profiles shown by curves A and B of Figure 11.

Another useful method of predicting the potential storage stability of some food products consists of evacuating a suitable air-tight container and back-flushing with an oxygen atmosphere. The food is then aged at the desired temperature under oxygen, and some measurement of the oxidative state is employed. We normally carry out the test in a vacuum desiccator which is evacuated prior to back-filling to atmospheric pressure with oxygen. This accelerated aging technique has been applied to baked products aged at room temperature, margarines aged in the refrigerator, various cooked meat products stored frozen, vegetables in butter sauce, and various other fried products. The most reliable index

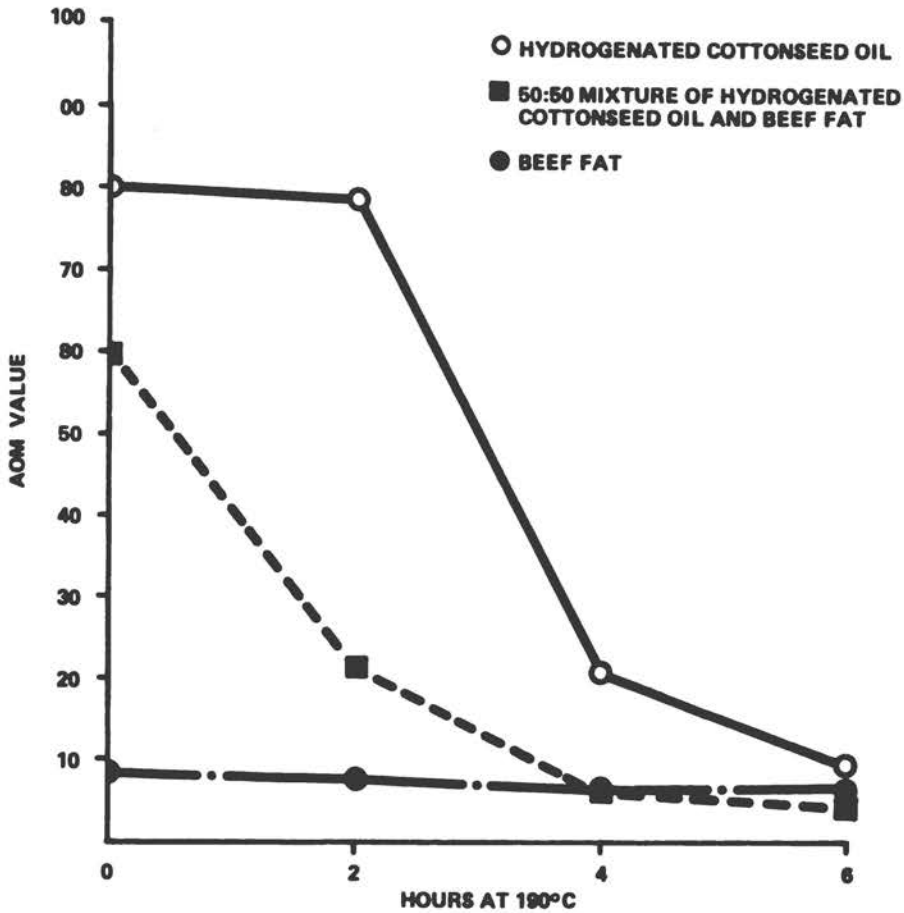


FIGURE 7 Heat stability characteristics of 3 types of fats.

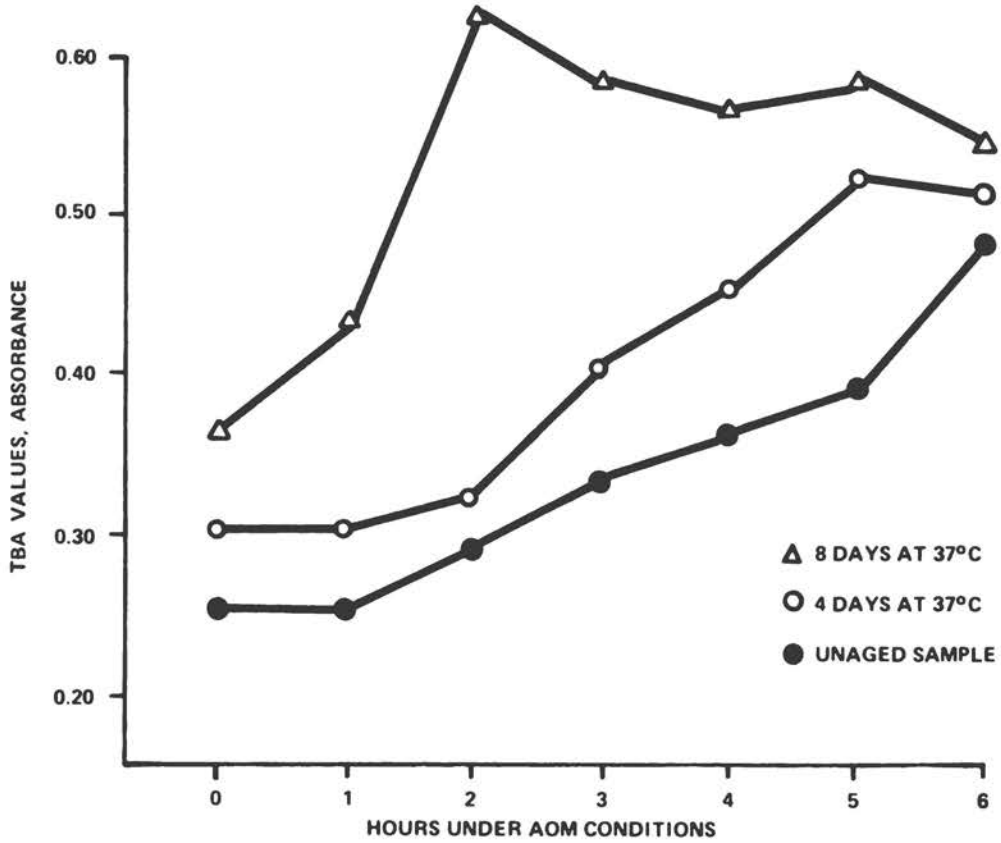


FIGURE 8 Changes in oxidative resistance of a shrimp breading aged various lengths of time, as measured by a modified AOM test.

of the state of oxidation has been flavor evaluation by a panel. The findings with some foods, such as sausage, indicate that the degree of rancidity normally encountered after 3 mo of frozen storage will occur in 1 week in the freezer when the sausage is aged under an oxygen blanket that is replenished daily. One of the applications of the accelerated aging technique was to predict the flavor stability of several crouton samples (Figure 12). The degree of acceleration for off-flavor development in the croutons was about sixfold, compared to about twelvefold for cooked sausages stored frozen. A series of experiments was then conducted which was designed to give us a better indication of what to expect with accelerated aging under oxygen at various temperatures. The model system consisted of noodles fried in various fats used for commercial chicken-frying operations. The fried noodles were aged in air or under oxygen at room temperature or in the freezer. Samples were presented periodically to a trained panel for odor and flavor evaluation. The end point was the time to reach a significant difference (95 percent confidence level)

in the mean flavor and aroma scores of the samples aged in air or under oxygen. Noodles stored at room temperature reached the flavor end point at 14 days and the frozen noodles at 27 days (Figure 13). Again the rate of acceleration of oxidation found at room temperature was about double that found in the samples stored in the freezer. Seemingly inconsistent fluctuations were encountered in the aroma data plots for the fried noodles. Curves such as these seem to be typical for this type of product and no doubt reflect the dynamic system of the flavor components; i.e., some good and some bad flavor components are being generated and lost at various times during aging (Figure 14).

Cooked ground pork patties were also aged to estimate the degree of acceleration generated by aging under oxygen. After 5 days in the refrigerator, the mean flavor scores for the cooked pork aged in air or under oxygen were significantly different at the 97.5 percent confidence level. The end point was reached for the freezer samples after 18 days, more than a threefold increase. TBA measurements were less sensitive in detecting the difference

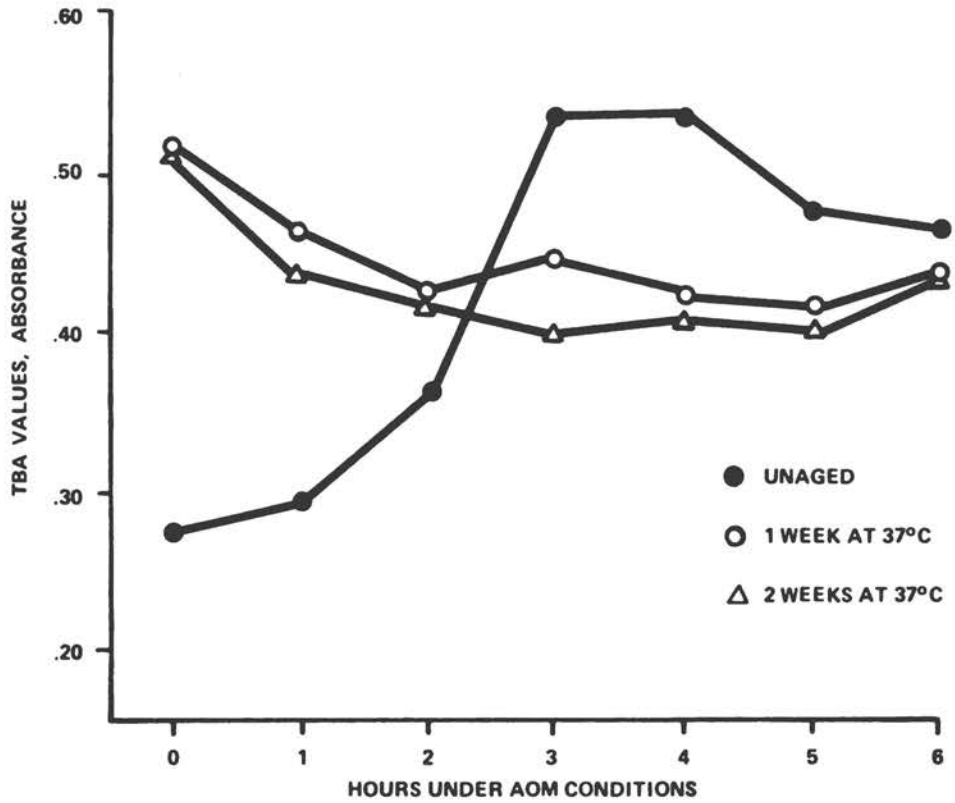


FIGURE 9 Changes in oxidative resistance of a shrimp breading aged various lengths of time, as measured by a modified AOM test.

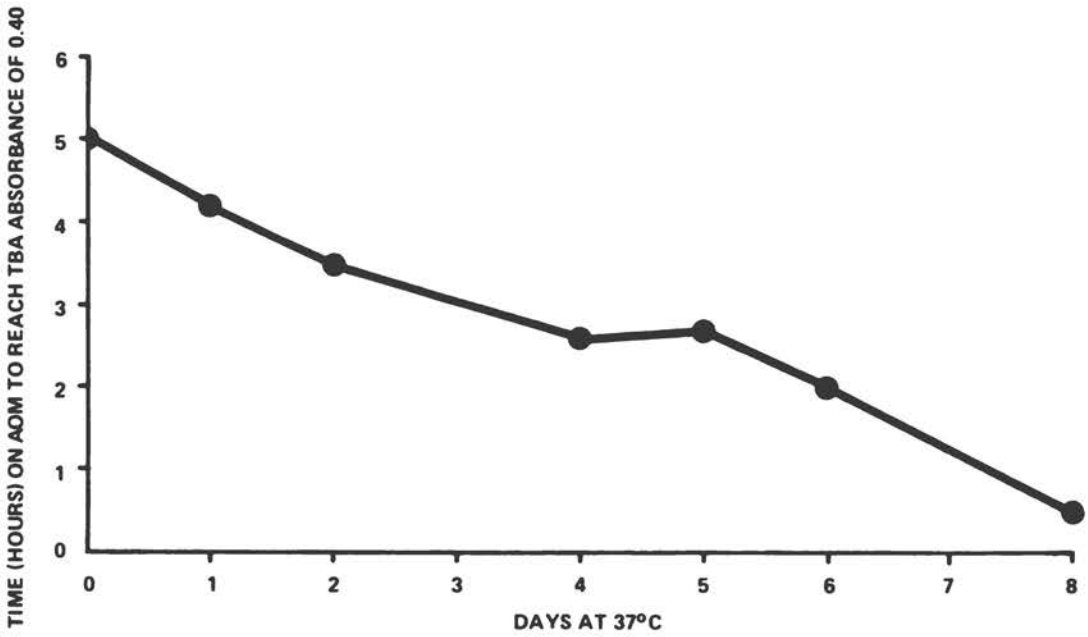


FIGURE 10 The loss of oxidative stability of a shrimp breading on aging at 37°C, as measured by a modified AOM test.

in refrigerated samples, with an end point of 12 days. The TBA end point of 18 days for the frozen samples, however, agreed very well with the flavor data.

While accelerated aging under oxygen is an empirical method, it was found to be useful in measuring the overall resistance of a food to potential off-flavor development from oxidative changes in storage. Refinements of this approach could lead to the development of mechanistic and perhaps kinetic data from the less complicated lipid-bearing food systems. For example, the combination of GLC or GLC-mass spectrometry headspace analysis and the accelerated aging technique would appear to be particularly useful. Regardless of how sophisticated the approach to these problems, however, one cannot afford to lose sight of the fact that the most important judge of flavor is still the consumer.

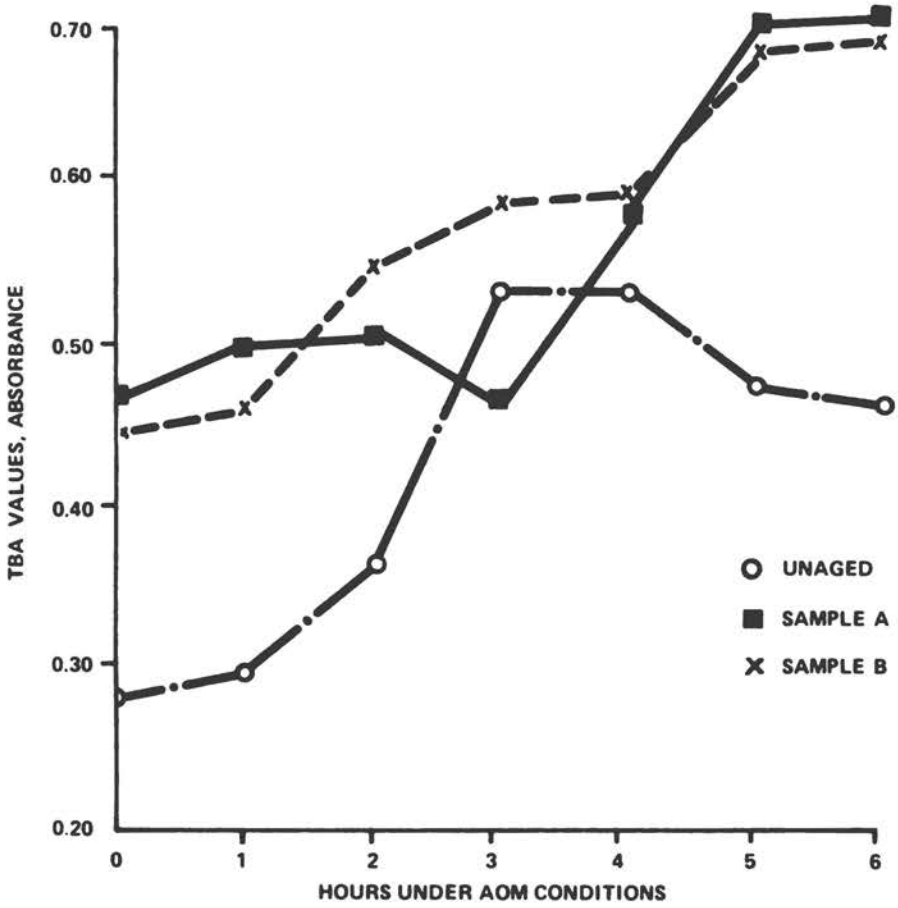


FIGURE 11 A comparison of the oxidative resistance of 2 shrimp breading samples, using a modified AOM test.

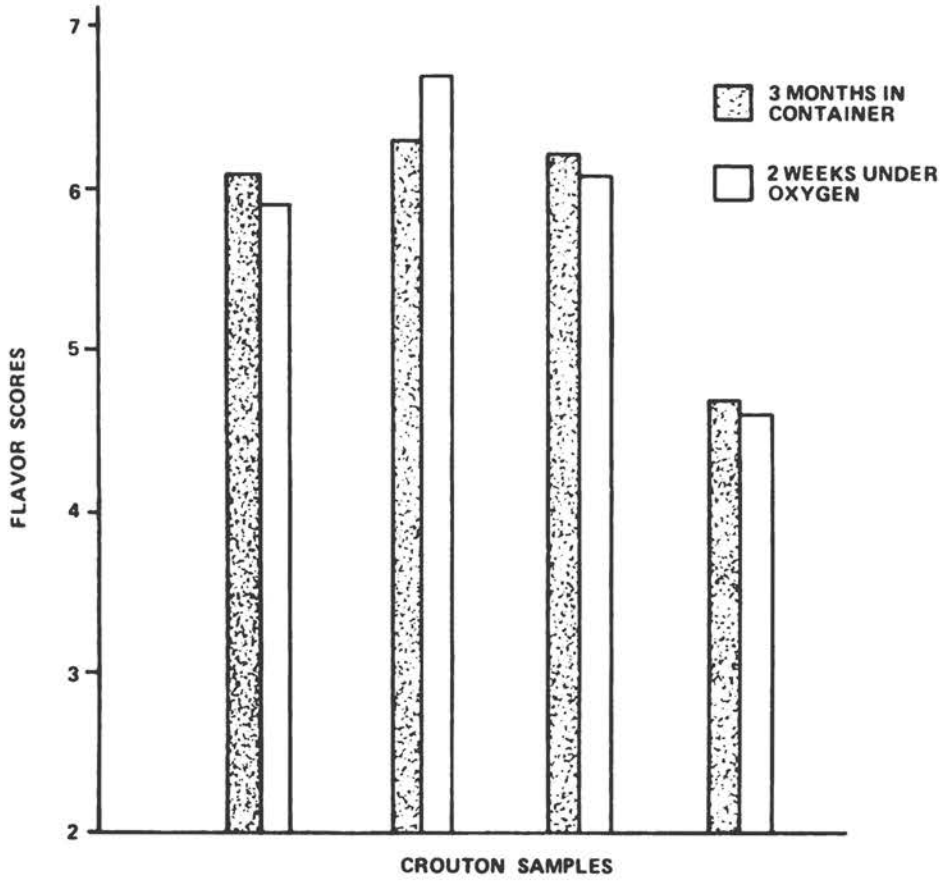


FIGURE 12 Application of an accelerated stability test to various crouton samples held at room temperature.

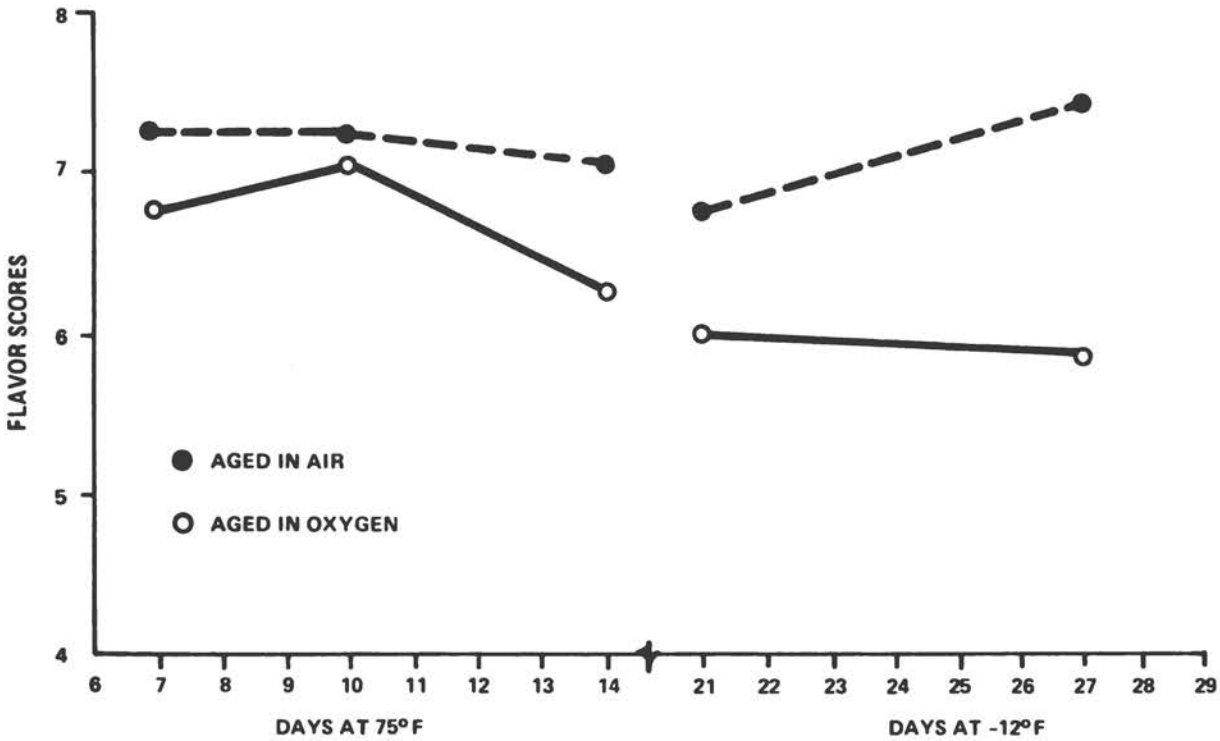


FIGURE 13 Changes in flavor of fried noodles aged in air or in oxygen at 2 temperatures.

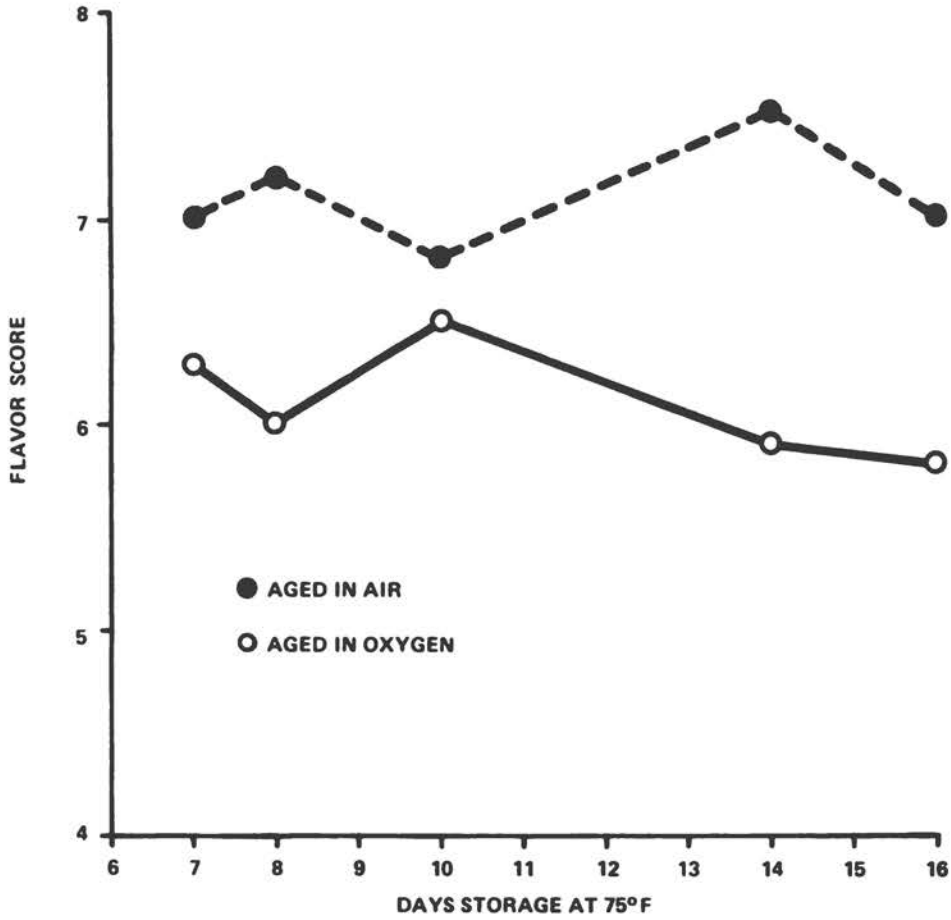


FIGURE 14 Changes in aroma of fried noodles aged in air or in oxygen at 75°F.

REFERENCES

1. S. J. Kazeniac and R. M. Hall, *J. Food Sci.*, *35*, 519 (1970).
2. E. Stone, R. M. Hall, and S. J. Kazeniac, private communication.
3. R. Tressl and F. Drawert, *J. Agric. Food Chem.*, *21*, 560 (1973).
4. W. L. Dunkley, *Food Technol.*, *5*, 342 (1951).
5. U. Holm, K. Ekbom, and G. Wode, *J. Am. Oil Chem. Soc.*, *32*, 13 (1955).
6. U. Holm, *Abstracts, International Society for Fat Research Congress*, Göteborg, Sweden, June 1972.
7. S. S. Chang, F. A. Kummerow, *J. Am. Oil Chem. Soc.*, *32*, 341 (1955).
8. C. D. Evans, Proceedings, Flavor Chemistry Symposium, Campbell Soup Company, 123 (1961).
9. B. D. Mookherjee, R. E. Deck, and S. S. Chang, *J. Agric. Food Chem.*, *13*, 131 (1965).
10. K. Warner, C. D. Evans, G. R. List, B. K. Boundy, and W. F. Kwolek, *J. Food Sci.*, *39*, 761 (1974).
11. G. Fuller, D. G. Guadagni, M. L. Weaver, G. Notter, and R. J. Horvat, *J. Food Sci.*, *36*, 43 (1971).
12. D. F. Brown, F. G. Dollear, and H. P. Dupuy, *J. Am. Oil Chem. Soc.*, *49*, 81 (1972).
13. S. S. Lin, T. H. Smouse, and R. R. Allen, Presented at the American Oil Chemists' Society Spring Meeting, Mexico City, 1974.
14. E. W. Turner, W. D. Paynter, E. J. Montie, M. W. Bessert, G. M. Struck, and F. C. Olson, *Food Technol.*, *8*, 326 (1954).

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

SESSION III:

ROUND TABLE DISCUSSIONS

Texture
Appearance
Flavor
Prediction of Fat Stability

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

TEXTURE

Nuri N. Mohsenin, Chairman

The makeup of the panel was a good blend of representatives of the food industry, the government (USDA and Natick Laboratories), and the universities, including one from England. Dr. Sherman and Dr. Szczesniak, in addition to their research interest in texture, are the Chief Editors of the *Journal of Texture Studies*, the only scientific publication devoted entirely to this subject.

The first hour of this Round Table discussion was somewhat structured, with each participant giving his brief comments in relation to the following questions which were proposed to the panel in advance.

1. Problems, contributions, and needed research in measurement of meat tenderness. Dr. Szczesniak and Dr. Herring commented on this subject.
2. Texture measurement as a means of predicting past history and storage life of foods. Dr. Sherman and Dr. Kapsalis presented their views and research examples on this subject.
3. How can the fundamental studies contribute to texture measurement? Dr. Kapsalis, Dr. Morrow, and Dr. Mohsenin gave examples of research to show the role of this phase of texture studies.
4. Is there a need for establishing recommendations for objective texture measurement? Do we know enough to develop some tentative recommendations? If so, what agency or groups should take the leadership in developing and promoting such recommendations?

The second hour was spent in answering questions and hearing comments from the audience, which consisted of about 40 persons. The following is a summary of this 2-hr Round Table discussion.

Of all the commodities and of all the textural characteristics, the tenderness of meat has received the greatest attention. Despite this high level of research activity, which can be traced back 70 years, and a long list of instrumental techniques, progress has been rather limited in correlating significantly the instrumental measurements with sensory evaluation. The reasons for this limited success have been: lack of knowledge of what to measure to simulate human judgment and of what is being

measured (as well as other factors having to do with the variability of the material), the problem of selection and use of instruments and trained panels, the application of appropriate statistical methodology, and the analysis and interpretation of data. In the case of meat, we all recognized the tremendous heterogeneity of the material and the difficulties in sampling. It is often impossible to be certain that the sensory panel and the instrument are testing the same or corresponding sample. The lack of knowledge of the mechanical behavior of the material under the stress-strain conditions operating in the mouth combined with an exceedingly high heterogeneity unsurpassed by any other food product are the main obstacles in tenderness evaluation of meat.

Despite this rather grim picture of the state of the art, there are indications over the past decade that we are beginning to understand some of the fundamental problems in psychorheological aspects of texture. Considerable work has been done in rheological and fundamental studies related to food texture as a whole. Measuring instruments are now more sophisticated; i.e., more precise and more reproducible. They also produce data faster. The advent of digital and analog computers permits development of new methods of statistical analysis of data. The experts in the field now generally support the use of well-defined physical parameters if they are applicable, understood, and measurable.

It has been emphasized[1] that certain conditions must be met to maximize the correlation between subjective and objective measurements. The first is the precision with which something is measured; e.g., measurements of tenderness by mechanical shear should be done under standardized conditions. The samples should cover entirely the commercial range of texture and not be restricted to values close to the mean value. A valid approach is to determine texture values on the sample which uniformly covers or represents the population. With just a few samples, a high or low correlation may be obtained by chance only. The samples also should cover all conditions and variations. This prevents the measurement of a particular sample from going off scale. The size of the correlation coefficient under these conditions can be used to indicate the accuracy of the objective method. A correlation of 0.9 is adequate since 81 percent of the variation can be accounted for. If the correlation is 0.7, then less than half of the variation in 1 trait can be accounted for by another.

There is a need for a functional relationship between two factors, x and y^2. The correlation coefficient is dependent upon the range of values covered, the number of values involved, and the magnitude of the variations within the block of samples. Variability within samples should be less then the variability between samples. One needs to be certain of the type of relationship between the sensory response and physical response, which in many instances has been shown to follow a logarithmic or semilogarithmic relationship.

It has been found[3] that by expressing the experimental data as deviations from their contemporary mean, extraneous sources of variation were minimized, resulting in a substantial improvement in the degree of correlation as theoretically expected. A contemporary mean is defined as an average value derived from observations collected in the same substratum, which is assumed to be homogeneous by virture of proper experimental design. The analysis and evaluation of experimental data derived from several runs or experiments without proper control of internal and external influences will likely result in erroneous conclusions.

Texture-measuring procedures can be used not only for quality control but also for predicting the past history and storage life of specific foods. Using these procedures, one should be able to establish the rate of change in selected physical parameters within a fixed period of time under standardized storage conditions. From these data, rate constants could be calculated that can be utilized to predict changes over longer periods of time until the product has undergone such change that it is no longer acceptable to the consumer. In order to establish the limits of acceptability by the consumer, detailed consumer (and texture profile) panel studies will be necessary. Panel studies will also help establish the correct instrumental test conditions. This last information is essential since the values of physical parameters derived from instrumental tests depend upon the conditions employed in the test; i.e., the values of the parameters derived in instrumental tests are not absolute values.

Whenever the rate of change of a physical or chemical property is involved, the use of analog computers has proved to be a successful means of predicting quality. An example was given of an analog-computer model for predicting chemical and physical properties of selected fruits during storage and also for animal muscle post mortem[4].

The changes of the modulus of elasticity of selected plant products in relation to the gradual loss of moisture under controlled conditions was also given as an example for assessing the past history of the food sample. Standard curves of the changes of modulus of elasticity as the sample progressively lost water and its turgor properties changed were obtained. On the basis of these curves one could estimate the past history of the sample (how old the sample was) and to predict the time necessary to reach a certain degree of deterioration (wilting). The results apply strictly to the type of sample and the dimensions used. However, construction of standard curves for other samples within certain "tolerance limits," which will take into consideration the variety and other factors, will eventually be possible[5].

On the importance of fundamental studies, it was pointed out again that one of the problems in instrumental measurement of texture of solid foods is lack of understanding of what is being measured, and whether or not a change in geometry, depth of

penetration, or other factors related to mechanics of force application influence the results. Conceivably, if the instrument is measuring a well-defined property of the material, such variations should not influence the magnitude of the property measured, allowing for experimental errors and sample variations.

To verify this theory, research was reported on the mechanical interpretation of selected texture-measuring instruments. One attempt reported in that direction was the analysis of the mechanics of a very common fruit pressure tester[6]. The other example was the work in progress to analyze the mechanics of an "Armour Tenderometer" type of probe to determine if the material's property can be assessed independently of the geometry or depth of penetration of a series of needle-like probes used in this device[7].

The results on two non-food materials have shown that it is possible to use the plasticity theory to predict yield pressure as a criterion of strength of the material being tested. The consistency between calculated yield pressures was adequate for different angles of the probes and depth of penetrations. Studies are now being conducted on actual meat samples.

Well-defined compression tests to examine the mechanical properties of cylindrical beef samples before and after cooking, carefully labeled for the position in the muscle, have shown that by matching adjacent samples between slices, zones of similar tenderness could be differentiated along the muscle[8]. The greatest variation of mechanical properties was found to be across the muscle (lateral to medial) rather than along the muscle. Correlation coefficients averaging 0.87 were obtained between raw and cooked beef by carefully following regions of similar characteristics from raw to cooked slices.

This work showed certain apparent inconsistencies in the expected relative tenderness of some muscles when the modulus of elasticity and other mechanical properties were used for measurement. This led investigators to suspect that testing by uniaxial compression may not provide the necessary information to characterize the mechanical properties of raw and cooked meat. To test this concept a mechanical model of springs and dashpots was constructed consisting of side arms and transverse elements simulating the structure of beef, which is made up of parallel fibers connected by cross links. Solution of this model using only the elastic (spring) elements, as a first approach, showed that closely similar stress-strain curves (similar magnitudes of the modulus of elasticity, area under the curve, and other parameters) could be obtained for two basically dissimilar materials if the materials were tested by uniaxial compression. Preliminary results, which also include the viscous elements, support the above hypothesis. It was suggested that part of the disagreement among different laboratories on the magnitude of the correlation coefficient, when the method of compression testing has been used, is due to this fact. We need to know the response of meat to mechanical testing not only in one direction but also in its

transverse dimension. For this purpose a device was constructed to measure the Poisson's Ratio, which is the ratio of the transverse to axial strain, together with the other properties measured by compression. Using this arrangement, correlation coefficients between mechanical measurements and panel ratings (both on cooked meat) were as high as 0.90. The results of this work will be published in a forthcoming paper now in preparation at Natick Laboratories.

On the question of understanding just what is being measured, an example from published work was given that has proved that Warner-Bratzler shear test has little to do with fiber adhesion. Well-defined tensile tests of the muscle fiber, however, were shown to be highly correlated with Warner-Bratzler, indicating that this test must be basically a tensile test and that there is no point in trying to correlate the results to compression, shear, or adhesion test of muscle fibers[9]. Similarly, punching a plunger into a meat specimen across the fibers is really a punch-shear test, highly correlated to fiber adhesion rather than resistance to compression or penetration. The question is, then, can the mechanics of the Warner-Bratzler shear test be modified such that test results of this simple apparatus can be converted to well-defined, tensile-strength values that can be obtained only by more elaborate and expensive machines.

It was generally agreed that there is a need for better-defined terms, for simplifying the mechanics of the conventional methods of force application, and for knowing the contribution and importance of each mechanical property to the overall sensory-evaluation value.

There is a need for a system involving instruments measuring a number of mechanical attributes such as resistance to shear, compression, adhesion, tensile stress, etc., separately in terms of well-defined parameters with the capability of combining these parameters according to their relative importance and contributions to the overall textural quality of the food. These results can then be expressed in some mathematical or graphical form to correlate a single textural index with sensory data. The work at the Natick Laboratories combining the well-defined instrumental testing capability with the expertise in the behavioral sciences in the area of sensory testing, where both standard rating scales and the method of magnitude estimation have been employed to food-texture problems, is a step in that direction[10,11]. The researchers foresee eventual development of an instrument calibrated in numbers that will reflect sensory responses, in order to quantify consumer attitudes to the textural characteristics of a specified food commodity.

On the other hand, the problems and the difficulties involved in the basic and fundamental approach to an understanding of texture measurement are well recognized. Such an approach requires special training and takes more time than the industry, with its immediate and pressing problems, can afford. Therefore,

while this type of approach is most essential and must be continued, the industry should perhaps continue to use whatever method has proven to be objective and reliable for any given specific application. In the absence of such methods, sometimes, instead of relying on texture-evaluation data, the quality-control problem has been solved indirectly. The use of mechanical tenderizing of meat was given as an example of this indirect approach.

On the question of establishing a recommended procedure for testing, analysis of data, and reporting of results so that work at different laboratories can be compared, there was a general agreement that development of such recommendations in certain areas is badly needed. In the case of meat tenderness, for example, it is essential to establish a standardized and readily available objective procedure for measuring tenderness. Equally important is a standardized procedure to relate units of instrumental measurements to sensory judgments of tenderness established by sufficiently large and adequately representative consumer panels.

It was pointed out that there are many instruments, procedures, and sample types that have in most cases been demonstrated to be well correlated with subjective estimates of meat tenderness. The different instrument-procedure combinations have been shown to be in many cases sufficiently correlated with one another that they are equivalent for measuring differences and changes in tenderness. Correlation coefficients between different objective methods have been as high as +0.995.

However, in general, units of measurement from one procedure cannot be related to those of another. One of the most complicating factors has been the size (as well as location and orientation) of the samples used. For example, it has been demonstrated that shear force by the Warner-Bratzler instrument is more nearly proportional to equivalent diameter than to cross-sectional area at shearing locations[9]. Moreover, the relationship between the force reading and diameter of the sample is a logarithmic relationship following a power law equation. This means that, other things equal, the force reading from one laboratory working with one sample size can be converted to the equivalent force reading in another laboratory working with a different sample size. Likewise, in evaluating the firmness of fresh fruits, recent work has shown that, by knowing the shear strength of the flesh, the readings from a 5/16-in. (0.79 cm) Magness-Taylor pressure tester used at one laboratory can be converted to equivalent readings with a 7/16-in. (1.11 cm) pressure tester at another[6].

The use of such information, already in the literature, for development of tentative standards or recommendations requires action by a group of experts with the authority to develop, test, promote, and publicize such procedures. The development and publication of the ASAE (American Society of Agricultural Engineers) R368 "Compression Test of Food Materials

of Convex Shape"[12] was given as an example of such effort by an engineering society in which an established mechanism for such activities already exists. The recommendation follows the ASTM (American Society for Testing and Materials) style in establishing standards, covering the topics of purpose, scope, definitions, apparatus, test specimens, conditioning, number of test specimens, speed of testing, testing procedure, calculations, and report. Such standardization activity can be initiated by utilizing the interest and the expertise of the industry, ASTM, the Institute of Food Technology (IFT), as well as interested government agencies.

REFERENCES

1. Kramer, A. "The Relevance of Correlating Objective and Subjective Data," *Food Technol.*, *23*, 926 (1969).
2. Szczesniak, A., "Correlation Between Objective and Sensory Texture Measurement," *Food Technol.*, *22*, 981 (1968).
3. Gacula, M. C., J. B. Reaume, K. J. Morgan, and R. L. Luckett, "Statistical Aspects of the Correlation Between Objective and Subjective Measurements of Meat Tenderness," *J. Food Sci.*, *36*, 185 (1971).
4. Jabbari, A., N. Mohsenin, and W. S. Adams, *Transactions of the Am. Soc. of Agric. Eng.*, *14*, 319 (1971).
5. Kapsalis, J. G., R. A. Segars, and J. G. Krizik, "An Instrument for Measuring Rheological Properties by Bending, Application to Food Materials of Plant Origin," *J. Texture Studies*, *3*, 31 (1972).
6. Yang, Y. M. and N. Mohsenin, "Analysis of the Mechanics of the Fruit Pressure Tester," *J. Texture Studies*, *5*, 213 (1974).
7. Morrow, C. T. and N. Mohsenin, "Mechanical Interpretation of Objective Measurement of Meat Tenderness," *Proceedings, 4th International Congress of Food Science and Technology* (Madrid, Spain, 1974).
8. Segars, R. A., H. A. Nordstrom, and J. G. Kapsalis, "Textural Characteristics of Beef Muscles," *J. Texture Studies*, in print (1974).
9. Pool, M. F. and A. A. Klose, "The Relation of Force to Sample Dimensions in Objective Measurement of Tenderness of Poultry Meat," *J. Food Sci.*, *34*, 524 (1969).
10. Moskowitz, H. R. and J. G. Kapsalis, "Toward a General Theory of Texture Psychophysics," *Proceedings, 4th International Congress of Food Science and Technology* (Madrid, Spain, 1974).

11. Moskowitz, H. R., R. A. Segars, J. G. Kapsalis, and R. A. Kluter, "Sensory Ratio Scales Relating Hardness and Crunchiness to Mechanical Properties of Space Cubes," *J. of Food Sci.*, *39*, 200 (1974).

12. American Society of Agricultural Engineers, "Compression Test of Food Materials of Convex Shape," *ASAE Yearbook*, 386-389 (ASAE, St. Joseph, Michigan, 1974).

APPEARANCE

OBJECTIVE METHODS FOR APPEARANCE EVALUATION --
Richard S. Hunter, Chairman

ABSTRACT

Of the sensory attributes of food, those related to appearance are the most susceptible to objective measurement. There are two separate types of categories of appearance:

1. Color attributes related to the wavelength distribution of light.
2. Geometric attributes related to the spacial distribution of light.

Of these two, color is by far the most important so far as foods go. The most frequent use of food color measurements is as objective indices of food quality.

Color is measured by duplicating in an instrument the known wavelength response characteristics of the color receptors in the human eye. Geometric attributes, on the other hand, are measured only by cut and try procedures selected for their correlation with visual estimates.

The potential for appearance analyses of foods can be seen by the extent to which objective analysis of appearance attributes are established within American technology. This can be done by reviewing the list of methods published for non-food materials by the American Society of Testing and Materials.

With proper attention to test method preparation, there is no reason why objective measurements of food appearance cannot be used for the following applications:

1. Quality index measurements of raw and processed foods for use in records and communication.
2. Determinations of conformity of food quality to specifications.
3. Analyses of quality deterioration as a result of food processing, food storage, and other hazards.

INTRODUCTION

The food industry is large and vitally important to society. It is concerned with perishable products in which quality is

highly fugitive and increasingly important. Although it is not the same as quality, the appearance of foodstuffs is widely associated with and used to evaluate quality. Color can be measured more easily than taste, odor, and texture. In the market place, where the potential purchaser makes his selections, he is seldom allowed to taste or eat food to test it; he can, however, look at it and judge it by appearance. Objective evaluations of the appearances of foods are made primarily for the purpose of obtaining indications of this potential food purchaser's visual judgments.

Appearance analyses of foods are used primarily in conjunction with food development and improvement, and with quality assessment:

A. Food development and improvement:
   1. Measurements relating to breeding and selection of variety.
   2. Studies of growing conditions (fertilizing, spraying, etc.).
   3. Harvesting (timing, methods, handling of products for processor).
   4. Development of new and improved processed foods.

B. Food quality assessment and maintenance:
   5. Quality evaluation of fresh fruits and vegetables as received from the farm.
   6. Maintenance of product quality throughout and at end of processing.
   7. Maintenance of quality during transportation, storage, and marketing.

Color and geometric attributes of appearance. Actually there are two separate categories of appearance attributes:

1. Color attributes related to the wavelength distribution of light.
2. Geometric attributes related to the spacial distribution of light.

Of these two, the color attributes are by far the most important. The measurable color attributes are:

1. The spectrophotometric curve that corresponds to the absorption and scattering properties of ingredients, and does not evaluate color appearance until the color responses of the human eye are combined with it.
2. CIE tristimulus attributes: X,Y,Z; Y,x,y; Y,Λ,p; a universal color identification system, but difficult to relate to color appearance.
3. L,a,b opponent-colors measurements of objects and materials, where L measures darkness-lightness; a, redness-greenness; and b, yellowness-blueness. (Hunter, Adams-Nickerson, CIE cube root).
4. Color difference, usually measured in L,a,b opponent-colors dimensions.
5. Special food color quality indices such as TC (tomato color), CR and CY (citrus redness and yellowness), YI (yellowness indices), etc.

The chief geometric attributes are:

1. Gloss (specular, image-reflection quality, contrast luster).
2. Haze and turbidity by reflection or transmission.
3. Luminous reflection and transmission factors; opacity.
4. Texture (optical methods for texture measurement have not been established).

Color is measured by duplicating in an instrument the known wavelength response characteristics of the color receptors in the human eye. Geometric attributes, on the other hand, are measured only by cut and try procedures selected for their correlation with visual estimates.

Complexity of Appearance

Total appearance analyses combining all the color and geometric attributes are too cumbersome for practical measurement or use. Instead, partial measurements are made of only those attributes that are important for each different product and problem.

Professor Fred Billmeyer of Renssalaer Polytechnic Institute showed, in his Round Table discussion (attached), that the underlying physical phenomena responsible for all appearance attributes are simply reflection, scattering, and absorption, each variable with wavelength. Within the limits of specimen variability, these three phenomena can be measured. Their relations to the appearance attributes just described can be computed, but are usually quite complex.

If we can measure either the specific attributes of appearance or the specific optical attributes of foods responsible for appearance, it is reasonable to ask which are most useful in objective specifications. Generally it is the appearance attributes recognized visually that are most useful because these are what the purchaser and consumer of food recognize.

It would be wrong, however, to totally discount optical properties such as absorption and scattering. Objective ratings of food quality are usually the ultimate target of any measurement effort. I had an experience in Florida some years ago with the grading of reconstituted frozen orange-juice concentrate. I was surprised to discover that the USDA graders, doing their work by wholly visual methods, seemed to be able to identify differences in absorption related to orange pigment content. In the visual color of the samples, this pigment content was masked by differences in scattering associated with pulp content and particle size. These differences in orange pigment content were detected in spite of rather marked differences in "milkiness" associated with fineness of pulp dispersion introduced during the homogenizing process.

Optically, there are opaque foods seen wholly by reflected light. Examples of such foods are bread, flour, tomato juice,

cheeses, and meat. Other products are listed in Table 1, which gives the optical classes of foods, examples of each, and their appearance attributes. The second optical class of foods consists of translucent products. These are seen partly by reflected and partly by transmitted light. Many fruit juices, jams, custards, and cooked foods fall into this category. Because the paths that light follows when it encounters these products are varied and complex, instrument conditions of measurement have to be rigorously controlled to obtain reproducible results. The third class of foods is transparent. Clear juices, wines, jellies, gelatins, soft drinks, etc. fall into this category.

Variety of optical types. Foods are not as uniform in their optical characteristics as is suggested by the Table 1 division into three categories. Foods are optically varied in translucency and turbidity, as well as color. There is a great variety in texture and structure since many of them are naturally grown materials that retain characteristics of the appearance and structure of the initial product. For optical measurement in practice, the most important fact about foods is the manner in which a major fraction of them depart significantly from the opaque, diffusing materials encountered in paints, papers, textiles, ceramics, and plastics. An almost infinite variety of degrees of product translucency varies all the way from cakes, cookies, and other prepared foods that are nearly as opaque as paints, to the highly transparent jellies, clear juices, and the like. Between these extremes, the vast majority of food products are partly translucent. Most of the fruit purees and fruit juices fall in this range. So far as techniques of measurement go, so do granular products such as lima beans, peas, whole strawberries, and the like.

With all color measurements, results depend importantly on the form of the specimen. Thus, in the food industry, the techniques of specimen handling are crucially important. In only a few of the examples listed are the specimens available as opaque flat surfaces, uniform throughout the area being tested. In measuring food products so that numbers characteristic of appearance are obtained, specimen non-uniformity and specimen translucency create frequent problems. Where products are textured and non-uniform in color, as with lima beans, meat, and containers with fresh strawberries and cranberries, one must measure so that the color of a large area of the specimen is averaged to give the results reported. This can be done by using either a large viewing area, or by spinning or moving the sample past the exposure window so that the various elements in the area are averaged over a time basis.

To demonstrate the translucency problem, Figure 1 is a photograph of a glass of grapefruit juice with a collar of amber cardboard around the lower half. A 3/4 in. by 1 in. window is cut in this collar. At the top of the glass, we see the juice color as it appears in everyday use. It is lighter than the cardboard. Through the 3/4 in. window, we see the juice color as measured in

TABLE 1 Examples of foods which are opaque, translucent, and transparent.

Optical type and character	Typical foods	Appearance attributes measured
Opaque: Seen by reflected light:		Reflection color L,a,b or Y,x,y, etc.
Powder	Flour, powdered egg yolk, corn meal	
Granules, flakes	Fresh strawberries, dry dog food, corn flakes	
Liquid	Tomato juice, milk	Color quality measured for a specific product on a scale designed for that product. (Example: TC -- Tomato Color Index)
Paste or slurry	Tomato catsup, mustard, prepared baby food (vegetables)	
Object	Frankfurters and sausage, baked bread, cheese	
Translucent: Seen by reflected and transmitted light:		Reflection color L,a,b, etc.
Liquid	Orange juice Pineapple juice	Translucency: measured by T/R
Paste or slurry	Strawberry jam Applesauce	
Object	Pineapple slice	
Transparent: Seen by transmitted light:		Transmission color L_{trans},a,b or Y,x,y, etc.
Liquid	Apple juice Wine	Haze or turbidity
Paste or slurry	Apple jelly	
Object	Clear gelatine	

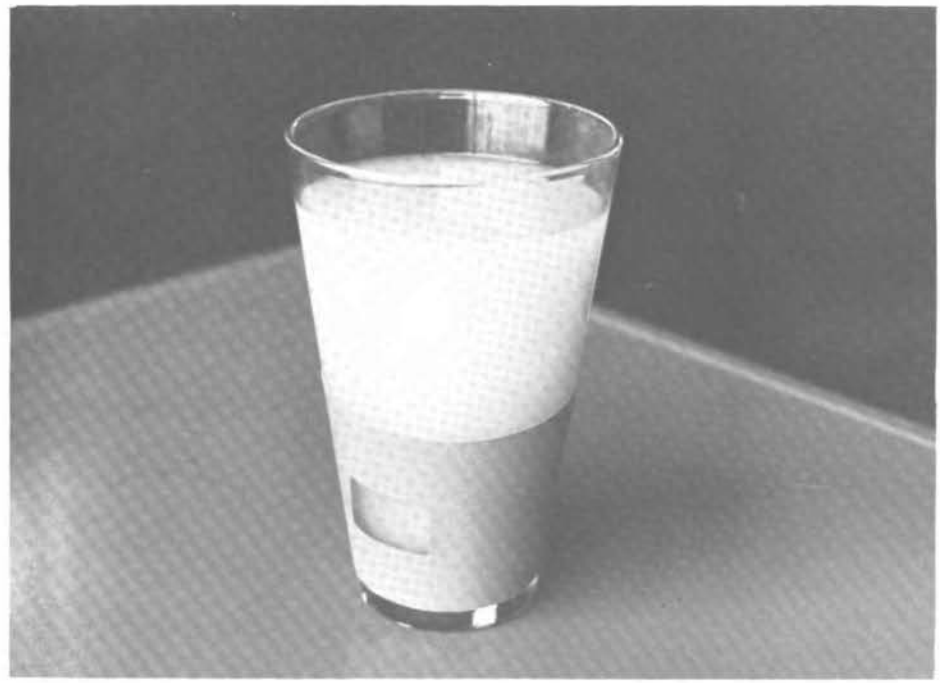


FIGURE 1 Photograph demonstrating the translucency error. Grapefruit juice looks darker seen through an aperture (as in the usual instrument) than when lighted on all sides.

an instrument where the incident beam fills the window as in Figure 2, top. The juice is darker than the cardboard because light is lost by travel within the juice around the edges of the window. The way to avoid most of this error and obtain measurements that correlate with visual observation is shown in Figure 2, bottom. Make the area of specimen viewed larger than that illuminated.

In practice, the problems of translucent specimens have been serious because they have been frequently unappreciated by the investigators reporting the results of color measurements. Many of the data on measurement of food colors now in the literature are of little value for comparisons with other measurements because of failure to control and report on effect of the translucency factor.

Standardization of methods of food color measurement is barely started. Tables 2 and 3 are taken from a recent ASTM published paper by Hunter showing the variety of standardized methods for appearance measurement available in the paint, plastic, paper, and other industries. Industry-wide standards of this scope do not yet exist in the food field. The best known written objective procedures for evaluation of food appearance are those used by the federal government for quality evaluation and standards of identity.

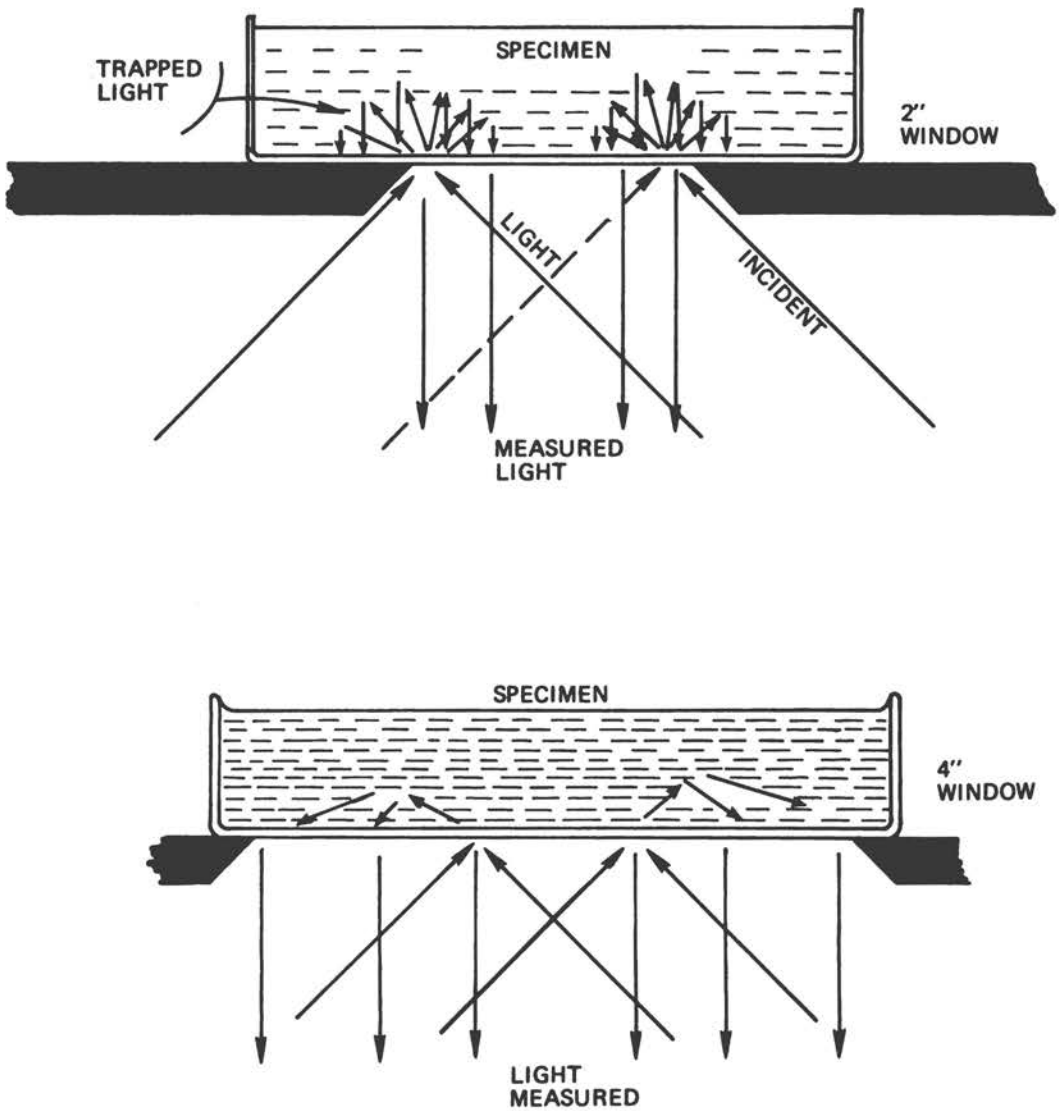


FIGURE 2 Diagrams showing trapping behind the edges of a 2 in. window of light reflected by a translucent liquid (above), and freedom from trapping with a 4 in. window (below).

TABLE 2 ASTM methods for appearance identification of diffusely reflecting materials.

Attributes	Products and ASTM committees involved				
	All products E-12	Paints D-1	Plastics D-20	Paper D-6 Soap D-12	Ceramics C-21, C-22, C-15
Chromatic attributes					
Spectrophotometric curves	E 308-66				
CIE color: X,Y,Z,x,y L,a,b, surface color	E 308-66				
Munsell color		D 1535-68			
Color difference in L,a,b		D 2244-68			C 609-67 (1971)
Color differences in other scales		D 2244-68 D 2616-67			
White colors:					
Whiteness indices	E 313-67				
Yellowness indices	E 313-67		D 1925-70		
Luminous (green) reflection factor	E 97-55			D 3050-72T	C 523-68
Blue reflection factor	E 97-55(1971)			D 985-50	
Pigment quality:					
Colored		D 3022-72			
White		D 2745-70			
Geometric attributes					
Goniophotometric curves	E 167-63(1970)				
Luminous reflection factor	E 97-55(1971)				C 347-57 (1967) C 523-68
Opacity (hiding power)		D 2805-70		D 589-65(1970)	
Retroreflectance factor		D 1011-52(1970)			
Specular gloss		D 523-67(60°,20°) D 1471-69(60°)	D 2457-70(60°, 20°,45°)	D 1223-67(75°) D 1834-65(1970) (20°)	C 346-59 (1967)(45°) C 584-71(60°)
Luster					
Distinctness of image gloss					C 540-67
Sheen		D 523-67(85°)			

TABLE 3 ASTM methods of appearance identification of metals and light transmitting materials.

Attributes	Products and ASTM committees involved: All products metals E-12	Resins, oils, liquids D-1,D-2 D-23,D-26,E-15	Plastic sheet, film D-20	Waxes polishes, D-21	Other materials
Chromatic attributes					
Spectrophotometric curves	E 308-66				
CIE color: X,Y,Z,x,y	E 308-66				
L,a,b, object color		E 450-72T			
Color difference		E 450-72T			
Yellowness indices	E 313-67	D 1500-64(1968) (Union) D 1209-69 (Platinum-cobalt) D 1544-68 (Gardner) D 1686-61(1972) (Platinum-cobalt) D 156-64(1968) (Saybolt) D 2108-71 (Platinum cobalt)	D 1925-70		
Whiteness indices				D 3210	
Geometric attributes					
Goniophotometric curves	E 166-63(1970) E 167-63(1970) (Refl.)				
Diffuse reflection factor	E 429-71				
Reflection haze	E 430-71				
Specular gloss	E 429-71 E 430-71		D 2457-70(60°, 20°, 45°)	D 3206 D 1455-64 (60°)	
Distinctness-of-image gloss	E 430-71				
Surface directionality	E 429-71 E 430-71				
Luminous transmission factor			D 1003-61(1970) D 1494-60(1969)		
Transmission haze			D 1003-61(1970)		
Transmission distinctness of image(transparency)			D 1746-70		
Transmission image displacement			D 881-48(1970) D 637-50(1970)		

In Table 1 of his contribution to the Round Table (see below) John Yeatman has listed seven of these established methods for food color identification.

Typical applications. Table 4 lists some typical applications of color measurements of fresh fruits and vegetables. One of the methods is that for tomato color index according to an equation, which is given. Measurements of color by this equation are being used extensively in the tomato canning industry to evaluate color quality of fruit brought to the cannery by farmers. The tomato color index is the numerical value that in part determines the price that the farmer receives for the load of tomatoes he submits. Fresh strawberries, cranberries, peas, corn, lima beans, and other fruit and vegetable products have also been measured for color quality, but with these other products, specific equations establishing color value of the product have not been put into commercial use.

Color index scales similar to those for fresh foods can be employed for food processing as shown in Table 5. The same tomato index color scales used for raw fruits have been used to evaluate processed tomato juice, catsup, soup, paste, and barbecue sauce. A similar citrus color score has been developed to evaluate the color of reconstituted frozen orange juice. It may be expected that further product color index scales will be developed as the usage of color measurements increases and the need for measurement of color quality in processed fruits and vegetables becomes more firmly identified. Further, increased use of color measurements to monitor quality of food products as they pass through stages of processing is anticipated. Color measurements can be used to evaluate the efficiency of processes in obtaining or maintaining desired product color. With many products the same heating that is necessary to process and preserve the product will also damage the color if care is not taken to keep time and temperatures to the minimum required for preservation.

Table 6 lists two tests typical of those used to evaluate the colors of compounded foods. Cooking or baking is generally involved in the production of such compound foods and, here, color is employed not only in evaluating the raw materials that go into this compounded food, but also in evaluating the cooking or baking process and its contribution to the final color. Bread and cakes are measured to assure that the body of the product has the desired color.

Because it occupies such an important place in the market, dog food color receives frequent attention. The importance of dog food color lies in the fact that it is the dog owner, not the dog, that chooses the product for appearance quality.

Table 7 lists three tests used in evaluating meats and meat products. Many of the measurements made in the meat industry are used to follow color changes as meat is prepared, processed, and stored. A desirable red color of fresh beef results from the presence of oxidized myoglobin. If denied access to oxygen, the myoglobin will turn to the blue-purple dark color of reduced

TABLE 4 Applications for fresh fruits and vegetables.

Purpose of Method	1. Attribute and product	Color index of fresh tomatoes (TC)	Color of fresh strawberries
	2. Industry	Canning	Jam manufacture
	3. Applications		
	a. In development	Improve plant breeds	
	b. In quality control	Grading picked tomatoes	Color evaluation of fresh strawberries
	c. In service		
	4. Benefits	Gives objective rating of color quality	Gives numerical rating of fruit maturity
Details of Method	5. Apparatus required	Tomato Colorimeter	Color Difference Meter
	6. Preparation and form of specimens	Fruits juiced, sieved, deaerated and placed in transparent cup	Whole fruits washed and dried, put in large container level to top.
	7. Exposure conditions	Through bottom of cup (diameter of area viewed 1 in. greater than area illuminated)	Top of fruit observed. Advantageous to move fruit under window
	8. Types of standards		
	a. High scale	Porcelain-enamel tomato-color standard	White diffuse reflectance standard -- strawberry red standard
	b. Intermediate		
	9. Specific quantity measured	$TC = 100 \left(\frac{21.6}{L} - \frac{7.56}{L_a} \right)$	L,a,b
	10. Results reported	TC	L,a,b of specimen
	11. Notes		
	12. References	*J. Opt. Soc. Am. 51*, 552 (1961)	

TABLE 5 Applications for processed fruits and vegetables.

	1. Attribute and product	Color of canned baby food	Citrus redness of reconstituted frozen orange juice	Reflection color of strawberry jam	Color of tomato juice, catsup, paste
	2. Industry	Canning	Citrus processing	Preserving	Canning
Purpose of Method	3. Applications a. In development b. In quality control c. In service	Quality of processed product Color stability	Quality grade of processed juice	Quality of processed product Color retention in storage	Quality of processed product
	4. Benefits	Gives numerical designations for comparisons	Places color grading on objective basis	Provides measurements of critically important retention of color in processing and storage	Measures retention of color in processing and storage
Details of Method	5. Apparatus required	Hunterlab Color Difference Meter	Hunterlab D45 Citrus Colorimeter	Hunterlab Color Difference Meter	Hunterlab Tomato Colorimeter or Color Difference Meter
	6. Preparation and form of specimens	Pureed material poured into cup with window in bottom	Reconstituted juice into 1 in. tubes -- (screw top lab glass)	Jam poured into cup with transparent window in bottom	Specimen poured into cup with transparent window in bottom
	7. Exposure conditions	Through bottom of cup (diameter area viewed is 1 in. greater than area illuminated)	Through side of glass tube	Through bottom of cup (diameter area viewed is 1 in. greater than area illuminated)	Through bottom of cup (diameter area viewed is for juice and catsup -- 1 in. greater than area illuminated)
	8. Types of standards: a. High scale	White diffuse reflectance standard	White $BaSO_4$ in tube	White diffuse reflectance standard	White diffuse reflectance standard
	b. Intermediate	Tan or brown standard	USDA OJ plastic standards	Maroon ceramic standard	Tomato ceramic standard
	9. Specific quantity measured	L,a,b	ECS = 22.51 + 0.165CR + 0.111CY	L,a,b	TC (see raw tomatoes) or L,a,b
	10. Results reported	L,a,b or color difference from standard	CR = 200(A/Y-1) CY = 100(1-0.847Z/Y)	L,a,b or color difference from standard	TC or L,a,b
	11. Notes:				
	12. References		*Food Technol.*, *20*, No. 5, 100 (June, 1967)		

TABLE 6 Applications for compounded foods.

Purpose of Method	1. Attribute and product	Color of dog food	Reflectance of roast coffee
	2. Industry	Cereal processing	Coffee roasting
	3. Applications		
	a. In development		
	b. In quality control	Quality of processed product	Degree of roasting
	c. In service		
	4. Benefits	Departure from desired color shows need to change ingredients oven temperature	Reflectance provides a numerical measurement of roasting progress
Details of Method	5. Apparatus required	Hunterlab Color Difference Meter	Reflectometer for $Y_{45°0°}$, such as Hunterlab D40
	6. Preparation and form of specimens	Granular material must pass 1/2 in. sieve but not 1/4 in. sieve	Whole roast beans are ground to specified size; poured into container and pressed level
	7. Exposure conditions	Level top of granules in cup observed	Observe reflectance of level surface of ground coffee
	8. Type of standards		
	a. High scale	White porcelain	White porcelain
	b. Intermediate	Brown ceramic standard	Gray or brown ceramic
	9. Specific quantity measured	L,a,b	45°0°
	10. Results reported	L,a,b of specimen	Y or G reflectance
	11. Notes and references		Lockhart, *Food Technol.*, *14*, No. 11, 597 (1960)

TABLE 7 Applications for meats and meat products.

Purpose of Method	1. Attribute and product	Color of bologna	Color of reconstituted freeze-dried meat	Color of plastic wrapped fresh beef
	2. Industry	Meat product	Meat processing	Meat packing
	3. Applications			
	a. In development		Evaluate method of freeze-drying and reconstituting	Selection of packaging materials
	b. In quality control	Quality of compounded product		
	c. In service			Evaluation of rate of deterioration
	4. Benefits	Off-color bologna shows need to change ingredients or processing conditions	Color measurements show numerically the approach to original color	Color measurements permit intercomparison of packaging materials and methods
Details of Method	5. Apparatus required	Hunterlab Color Difference Meter	Hunterlab Color Difference Meter	Hunterlab Color Difference Meter
	6. Preparation and form of specimens	Slice bologna with slicing machine	Use slices of lean meat after freeze-drying and reconstituting	Use uniform flat areas of cuts of lean beef
	7. Exposure conditions	Back top slice with 1/2 in. of meat from same case	Select areas to test which are lean and uniform in color	Measure through transparent plastic film
	8. Types of standards			
	a. High scale	White porcelain	White porcelain	White porcelain
	b. Intermed.	Brown-red ceramic standard	Red-brown ceramic standard	Red-brown ceramic standard
	9. Specific quantity measured	L,a,b	L,a,b	L,a,b
	10. Results reported	L,a,b of specimen	L,a,b of specimen	L,a,b of specimen
	11. Notes and references		Swedish Meat Inst., Göteborg	

myoglobin. However, a drift of the desirable red color toward brown may alternately be associated with the first stages of deterioration.

Prof. Donald H. Kropf, in his contribution (later in this presentation) to the Round Table discussion, suggests that where, as in beef muscle, the pigments contributing to color appearance are few and specific, unique wavelength measurements may be used as indicators of color quality.

CONCLUSION

Universally accepted general-purpose methods for the objective specification of food appearance are not widely available. Only in a few cases are standardized procedures available for specific food products. The potential for expanding and improving the objective specification of food appearance is very great.

REFERENCES

R. S. Hunter, "Visually Perceived Attributes of the Appearance of Materials and ASTM Progress Toward Their Measurement," Sensory Evaluation of Appearance of Materials, ASTM STP 545, American Society for Testing and Materials, 18-34, 1973.

R. S. Hunter and J. N. Yeatman, Direct-Reading Tomato Colorimeter, *J. Opt. Soc. Am.*, *51*, 552-554, 1961.

R. S. Hunter, "The Measurement of Appearance," to be published by Wiley, 1975.

FUNDAMENTAL PROCESSES LEADING TO THE APPEARANCE OF FOODS --

Fred W. Billmeyer, Jr.

INTRODUCTION

The underlying physical phenomena, resulting from the interaction of light with matter, which lead to the appearance of foods and other objects are fortunately few and relatively familiar. Aside from the possibility of fluorescence, which I will not consider, they are the absorption and scattering of light in the interior of the food, and the reflection of light at its surface. Each of these depends upon the wavelength of the light used.

With some limitations due to sample preparation and instrument design, each of these quantities can be measured. The real problem arises in relating the results of these measurements to the appearance of the food. Great care must be taken in defining and specifying the parameters derived from physical measurements that are intended to correlate with the visual attributes of appearance.

The techniques for obtaining these derived parameters and their correlation with perceived color and other aspects of appearance are described in well-known books[1,2].

DISCUSSION

The Role of Absorption. Absorption is the process by which the energy in a beam of visible light is transformed into other forms of radiation, usually heat. A substance that absorbs little light ranges from colorless to white, depending upon the amount of scattering as indicated below; one that absorbs most of the light striking it is dark in color. The selective absorption of light of various wavelengths leads to color; red meat absorbs light of all wavelengths except red.

The Role of Scattering. The scattering of light accounts for the translucency and opacity of foods and the appearance attribute called whiteness. For example, milk is white and translucent because of the scattering of light by fat globules suspended in water. The selective absorption (and to a small extent, of scattering) of light as a function of wavelength adds color. The role of scattering in determining the appearance of meat has been described by MacDougall[3-5].

The Role of Surface Reflection. A small amount of light is always reflected at the surface of a food. If this reflected light travels in the mirror or specular direction, the specimen is said to be glossy; otherwise, it is to some degree diffusing or matte. In observing appearance visually, it is usual to assess the nature of the surface reflection in terms of the gloss of the sample, and then to ignore it as much as possible in determining color.

Turbid-Medium Theory. The analysis of the interaction of light with foods, as with most other materials, begins with the adoption of a model, which may not represent the true physical situation but describes the behavior of the specimen adequately. In most cases the model proposed[6,7] by Kubelka and Munk has proved adequate. This model assumes the specimen is a sheet with plane, parallel faces, infinitely large but of finite thickness. The optical properties of the material are assumed to be described, at any single wavelength, by a scattering coefficient S and an absorption coefficient K, describing the amounts of light involved in these processes per unit length of travel of the light beam in the specimen. Surface reflection is accounted for by suitable corrections so that only the light inside the sample is considered.

Mathematical treatment of this model leads to equations relating K and S to the transmittance and reflectance, R (observable quantities), of the sample. For fully opaque specimens, the result is simple:

$$K/S = (1-R)^2/2R \qquad (1)$$

In the usual application, the turbid-medium equation is combined with a mixing law that describes how K and S, describing the material as a whole, are related to the K's and S's of scattering and absorbing components (such as dyes and pigments) in the sample and their concentrations, C:

$$K = K_1C_1 + K_2C_2 + ... \quad (2)$$

$$S = S_1C_1 + S_2C_2 + ... \quad (3)$$

In the case of foods, the scattering and absorbing species are often not well defined, and this approach may have less value.

Relation to Perceived Appearance. The fundamental physical parameters describing the appearance of foods are, in my opinion, the reflectance and transmittance of the sample or the Kubelka-Munk scattering and absorption coefficients S and K, each as a function of the wavelength. But these parameters are not simply related to perceived color and other appearance aspects. The derivation of new quantities with these properties is well understood[8] and the methodology and fundamental data required are documented[8,9]. The parameters themselves -- CIE tristimulus values and chromaticity coordinates, a variety of opponent color scales of lightness (L), redness-greenness (a), and yellowness-blueness (b), color differences, and special food color quality indices -- are well described in the literature cited and Mr. Hunter's introductory remarks[10] and will not be discussed here.

It is important to stress the ways in which the fundamental and derived parameters are appropriately used, the limited extent to which they can be interchanged, and the place of each in objective methods for food evaluation:

The fundamental parameters (reflectance, transmittance, K, and S as functions of wavelength) contain all the physical data necessary to describe the food, but they do not themselves correlate well with color or appearance. In my opinion, it is fruitless to attempt to establish such correlations, as for example between reflectance or K/S at one or two wavelengths and scales of color or desirability of a particular product. The literature documents many abortive attempts of this sort in other areas[11].

The derived parameters (color, color difference, and special scales) abridge the fundamental data in ways that are specifically designed to correlate with color or other appearance attributes. It is these quantities which should be used whenever it is desired to provide instrumentally derived equivalents (as nearly as is possible) to scales of perceived color and appearance.

However, it is not appropriate to use the derived parameters where the fundamental ones should be used because of the abridgement involved. For example, Kubelka-Munk K and S should never be computed from abridged coordinates such as tristimulus values or colorimeter readings. Except in cases of totally nonselective absorption and scattering, quite rare in foods, wrong answers will inevitably result and false conclusions can easily be drawn.

Procurement specifications can be written around either the fundamental or the derived parameters, as long as the writer is aware of the distinctions between them and has chosen the type consistent with the purposes of the specification. Thus, if it is desirable to specify the appearance of the product, appropriate derived parameters must be used. If, however, the purpose of the specification is analytical, such as to ensure adequate strength or concentration of one or more components, the use of properly selected fundamental parameters is necessary.

ACKNOWLEDGMENTS

I thank D. B. MacDougall for providing color slides based on his research, with which the oral version of this paper was illustrated. The work of The Rensselaer Color Measurement Laboratory is supported in part by the Allied Chemical Co., Ford Motor Co., Munseil Color Foundation, Plastics Institute of America, and Sherwin-Williams Co. This is Contribution No. 65 from The Rensselaer Color Measurement Laboratory.

REFERENCES

1. F. W. Billmeyer, Jr., and M. Saltzman, *Principles of Color Technol.* (Interscience-Wiley, New York, 1964).
2. G. MacKinney and A. C. Little, *Color of Foods* (AVI, Westport, Connecticut, 1962).
3. D. B. MacDougall, *J. Sci. Food Agric.*, *21*, 568 (1970).
4. D. B. MacDougall, *J. Sci. Food Agric.*, *22*, 427 (1971).
5. D. B. MacDougall, *Color 73*, R. W. G. Hunt, Ed., 432 (abs. only) (Halsted-Wiley, New York, 1973).
6. P. Kubelka, *J. Opt. Soc. Am.*, *38*, 448 (1948).
7. D. B. Judd and G. Wyszecki, *Color in Business, Science, and Industry*, 2nd edition, 387-405 (Wiley, New York, 1963).
8. Billmeyer, *Color Technology*, 31-45; MacKinney, *Color in Foods*, 28-48; Judd, *Industry*, 106-130.
9. CIE Document 15, *Colorimetry* (Bureau Central de la CIE, Paris, 1971, c/o U.S. National Committee, CIE, National Bureau of Standards, Washington, D.C.).
10. R. S. Hunter (this Symposium).
11. F. W. Billmeyer, Jr., *Materials Res. & Stds.*, *6*, 295 (1966).

POSSIBLE APPLICATIONS OF PSYCHOPHYSICAL FUNCTIONS AS SPECIFICATIONS FOR BOTH VISUAL AND CHEMICAL COMPOSITION -- Fergus M. Clydesdale

There has been a great deal of excellent work performed in colorimetry on the methodology of measurement of food materials. However, with the changing emphasis in technology and the instrumentation available, there seems to be a clear need for more research effort in two specific areas. One of these areas may be termed the "optical characterization of food materials by colorimetric techniques." This area involves the investigation of all the physical phenomena that occur when light strikes a food material and the delineation of such effects along with appropriate use of colorimetric data to predict those effects. The second major area is the need for colorimetric techniques to predict both visual and chemical changes occurring in food by means of either raw data or reduced data, which would eliminate the need for prolonged and lengthy chemical analyses along with visual approaches.

There has been a great deal of interest in the use of natural pigments to replace some of the synthetic food colorants currently used in the food industry. This trend, along with other factors in our everchanging technology, calls for methods described previously to measure such natural pigments in their existing state in food material. Methodology used to predict tinctorial strength of synthetic food colorants, such as extinction coefficients, becomes rather ineffectual in relation to natural pigments within biological systems. Such systems have degradation of pigments along with the formation of other compounds due to any number of oxidative or other chemical changes. Therefore, new measuring devices should be developed for predicting visual changes along with the chemical changes occurring in the pigments, as well as the formation of other colored compounds that might form during these reaction processes.

In order to do this, a research investigation must involve an overall look at all the possibilities of using raw data in any form feasible for predicting such changes. This reduction of data may or may not follow absolute theoretical guidelines, but the philosophy behind this approach calls for acceptance of measurements that will provide the necessary predictive capability whether or not they follow classical theoretical procedures. Certainly, there is ample precedent for using such an empirical approach at times, as evidenced by the development of such equipment as the citrus colorimeter and the tomato colorimeter.

Recently, in our laboratories, new color scales were developed for the measurement of pigment content in dark-colored beverages. These scales were produced to predict a linear relationship between pigment content and colorimetric readout. Moreover, a slightly different approach was attempted for predicting pigment degradation in the presence of the formation of new compounds while the pigments are degrading. This would

take into consideration the optical characteristics of the food material and also the development of terminology to specifically define tinctorial strength within a biological system. There would be many advantages to such a development because simple colorimetric measurements could predict chemical changes, formation of other colored compounds, and perhaps visual assessment of the material under examination.

In this work, the techniques involved using bulk lots of cranberry juice cocktail, heated in a water bath a 68°C to cause degradation of pigments and formation of brown products. Color measurements were taken every hour on the General Electric Recording Spectrophotometer, the Gardner Color Difference Meter, and the Hunter Color Difference Meter.

Also, pigment content was measured hourly using a pH differential method. This method involves taking optical density readings at 510 nm for aliquots of cranberry juice cocktail buffered at pH 4.5 and 1.0 respectively. The buffered juice was then allowed to equilibrate in the dark for 2 hr prior to transmittancy measurements being taken on the Hitachi Perkin-Elmer Spectrophotometer.

This approach produces a valid measurement of pigment degradation; following this, color parameters were used to evaluate the material. The parameters used were CIE, X, Y, Z, and also CIE functions of purity and chroma; Gardner and Hunter raw data were used as well as functions involving hue and chroma.

Obviously, such methodology would not be complete without visual assessment of the material under study. The materials were assessed by a panel with normal color vision.

Tristimulus values were correlated with both visual rankings and pigment concentration. Correlation coefficients of 0.95 or better were obtained with 6 psychophysical functions (CIE, X, Y, x, $(Y^2 + Z^2)^{1/2}$, $(X^2 + Z^2)^{1/2}$, and Hunter L) with either visual ranking or pigment concentration. The visual assessment in this case, of course, includes both the amount of pigment degraded and the amount of colored compounds formed during heat degradation.

The colorimetric scales mentioned previously were used to determine their use in pigment degradation studies, not just for the measurement of pigment concentration. There is certainly a difference in this approach because a measurement of pigment concentration does not take into consideration other off-colored compounds that are forming during heat degradation. From the results obtained it seemed that such equations could be used to predict degradation of pigments as well as pigment concentration.

It is hoped that these approaches will allow some interesting new areas of colorimetry to be studied in the future, and will make the analysis of food materials much simpler and provide more information with less laborious technology.

COLOR AND APPEARANCE IDENTIFICATION AND EVALUATION OF VEGETABLES -- Amihud Kramer

It is important to differentiate between identification and evaluation of appearance properties of all foods, and especially of fruits and vegetables. Not only species but even varieties (cultivars) are frequently identified and differentiated solely on the basis of their appearance, with properties other than color being of major importance. Thus varieties of apples can be identified on the basis of their shape, and certainly apples can be differentiated from pears, although there are some cultivars of apples (golden delicious) that approach pears in shape, and cultivars of pears that approach apples in shape (Bartlet). In fact, there are few fruit and vegetable varieties that can not be identified by shape. A good example is green beans. Some varieties approaching a circle in cross-section are known as round, others are oval, and still others are flat.

Certainly color differences are also helpful in identification. A deep red delicious can be identified and differentiated from red delicious or golden delicious solely on the basis of difference in dominant wavelength and lightness.

Identification of appearance properties *per se*, such as defining size in a size universe, or color in color space is of academic interest, and is essential for establishing specifications. For purposes of evaluation and grading, not all dimensions need to be measured. In each instance, only those dimensions should be evaluated that are applicable to the particular commodity and that have significance in determining acceptability of one unit of a commodity in relation to others. Size of pickles, for example, can usually be evaluated, entirely satisfactorily simply by measurement of cross-section diameter. For green beans, however, such a measurement is not adequate because green beans may vary in shape from flat to round. In other instances, particularly when the intrinsic shape or misshape of units is not involved, an excellent means of size measurement is simply in terms of count per volume, or count per weight. It is remarkable that so many evaluations of fruits and vegetables are based entirely on one size measurement, such as length in case of bananas, and diameter in case of asparagus.

Beyond conformance to established limits, determining shape is frequently a matter of evaluating extent of misshape. Thus, although green beans should be as straight as possible, particularly if they are to be packed as a "vertical" pack, a misshapen bean is one that curves to the extent that it may curl. Pickles, on the other hand, may show excessive curviture, or may not be uniform in diameter.

Regarding color as such, we have reported previously that, for purposes of evaluation and grading, there are certain instances (applesauce) when a complete tristimulus measurement is required, while in other instances (tomatoes) only two discrete measurements are required, and in a substantial number

of cases only one is necessary. We have since then found that satisfactory determination of color quality can be made on the raw produce using just one dimension, but the same product after processing may require the measurement of more than one, and not necessarily the same, dimensions of color.

These changes in color measurements can be explained on the basis of the pigments present in the raw produce, and changes in the pigments caused by processing. Thus in the case of applesauce, for which a complete tristimulus measurement is needed it is necessary to establish lightness of the sauce -- the lighter the better. At the same time two pigments are involved -- yellow and green. Presence of the yellow pigment is desirable, but not essential. In the case of the tomato, there are also two pigments involved -- yellow and red. Lightness, however, is of no concern, so that two measurements are sufficient with high levels of the red pigment and low level of the yellow pigment indicating the level of appearance acceptability. Appearance quality of raw cherries can be determined by measurement of lightness only. The darker the cherries the better. During heat processing, however, there is considerable browning, so that lightness is no longer valid, and instead the red-to-yellow ratio should be used as an appearance quality indicator.

When evaluating color of raw produce for processing, it is important to measure those color dimensions that will predict color quality of the finished product. Thus complete color measurements must be made of the raw product and these values related to the color quality of the finished product. It may be found that the parameters used to measure the color quality of the raw product for processing are not necessarily the best indicators of raw quality, but will predict the color quality of the finished product. Similarly, surface appearance of the raw produce is useless in determining the appearance of a comminuted product or just one that is peeled, etc. Thus measuring the redness of apples is of no value in indicating the color quality of applesauce. In some instances, even surface redness of tomatoes is of little value in determining color quality of the processed tomato product since color of the tomatoes at the surface may be redder or less red than internal tomato color, depending largely on environmental conditions.

While geometric attributes such as gloss are important in certain fruits and vegetables, in most instances color quality of fruits and vegetables can be determined satisfactorily without measuring specular reflectance. Undoubtedly the most serious limitation to adequate color measurement, particularly of fresh fruits and vegetables, is their shape and texture. As has already been pointed out, it is extemely difficult to measure color quality of the surface of a pear, when there is no flat surface to measure, and even if one approaching flatness could be obtained, how can one get a good measurement of the color of an apple when one cheek is bright red, and the opposite is green? Techniques for rotating sample or lens are satisfactory if some

average value is useful. But an average value reading between red and green certainly does not provide an indication of the appearance of the apple that is red on one cheek and green on the other.

A related problem is that fruits and vegetables, even more than animal products, are living, respiring systems. Thus they are continuously changing in appearance, particularly if they are peeled and further comminuted when color changes become extremely rapid. The only way to inactivate such enzymatic changes is by application of heat, which in turn may cause other changes in appearance.

MEAT COLOR EVALUATION -- Donald H. Kropf

The idea in objective color measurement is to have a referee system that will make decisions as to product acceptance or rejection that is indisputable and will stand up to legal objections.

A comparison of reflectance values for muscle (Table 1) with a visual color of 4 (unacceptable color), taken from various research studies at Kansas State University, shows widely different values for different studies. This is true whether considering reflectance percentage at wavelengths of 525, 572, 600 or 630 and also K/S for these same wavelengths. It would

TABLE 1 Reflectance values at visual 4.0.

	Longissimus	Psoas major
Percent R		
525	18.64, 9.79, 11.75	17.09, 8.15, 12.05
572	19.18, 11.16, 12.7	16.38, 8.09, 12.5
600	14.60, 16.70	7.65, 15.10
630	20.35	19.60
K/S		
525	1.7756, 4.156, 3.315	2.011, 5.176, 3.210
572	1.7028, 3.536, 3.001	2.135, 5.221, 3.062
600	2.498, 2.078	5.574, 2.387
630	1.5588	1.6490
Ratio		
572/525	1.029, 1.140, 1.081	0.959, 0.993, 1.037
K/S		
572/525	0.959, 0.851, 0.905	1.062, 1.009, 0.954

appear that when more bleach is found, reflectance values are higher and steaks of poor color might be accepted merely because they are bleached.

Reflectance percentage values and K/S values also differ between the longissimus and psoas major muscles; therefore one would have to specify the muscle on which color should be measured if objective measurement is to be used for product acceptance or rejection.

Reflectance ratios may offer promise and K/S ratios at reflectance also offer possibilities.

Finally, the reflectance spectrophotometer is a very bulky and unwieldy instrument to move around. Ideally, for color acceptance and rejection, a portable instrument would be most useful. Hopefully, a portable fiber optic scanner can be developed that might be used for objective color measurement and using wavelengths as determined by working with a reflectance spectrophotometer.

FOOD COLOR STANDARDS IN THE FEDERAL GOVERNMENT -- John N. Yeatman

Standards of identity and quality of food and food products have been promulgated in many government agencies, such as the Department of the Treasury, Department of Commerce, Department of Defense, Department of Agriculture, and the Food and Drug Administration. Of these, the Department of Agriculture (USDA) quality grade standards are perhaps most widely used in the food industry. Color appearance standards are mentioned in both USDA grade standards for fresh and processed products, in particular fruits and vegetables, as well as Food and Drug Administration (FDA) standards of identity for cheddar cheese and canned tuna, and standards of quality for canned tomatoes. These and other color standards have been used in military specifications. Some have techniques for objective measurement of color.

A few products for which color standards have been mentioned in the FDA standards and USDA quality grades are shown in Table 1.

Except for standards that include colorimetric or spectrophotometric measurement of a color-related attribute, none of the visual match standards have pinpointed color designations that are reproducible with any degree of precision. One exception is the USDA frozen lima bean quality grade standard, which specifies the following:

Color designation	Daylight directional reflectance	CIE chromaticity coordinates		Munsell renotation		
	Percent Y	x	y	H	V	C
USDA lima bean green color standard	40.4	.3715	.4576	4.6GY	6.9/	7.4
USDA lima bean white color standard	63.5	.3731	.4016	7.7Y	8.3/	5.0

Munsell renotations for these colors, calculated by the author, are shown.

The example set by this excellent standard should be used for all others. If a product color should change through varietal selection or a new processing technique, the standard could be amended by specifying new CIE chromaticity coordinates that redefine the color for the product.

Standards developed as visual aids without definition in terms of CIE chromaticity coordinates would be subject to the vagaries of human judgment and to the errors of manufacturing reproduction.

Color designations for canned tomatoes in the USDA grade standard and the FDA minimum standard of quality have remained unchanged for 36 years. Whenever new Munsell color papers were needed, the color pigments for the papers were presumably matched with old papers. Unfortunately, conditions such as viewing, personnel, and pigment formulation were not the same each time; consequently, the new papers were perceptibly different. An additive color mixture of the Munsell color components has resulted in the changes in disk color shown in Figure 1. Visual match of tomato samples to Munsell disks from papers in 1939 would have a significantly different color grade using disks from 1972 papers. Tomatoes with a minimum grade A color score in 1939 or as recently as 1966 would be grade B or substandard today. Likewise, with the FDA standard of quality for canned tomatoes, a product of minimum grade C according to USDA color grade in 1939 would be substandard by the current FDA regulation.

The CIE color space plots of Munsell disk colors for minimum USDA grades A, C (partial), C, and FDA standards for canned tomatoes for 1939 and 1972 are shown in Figure 1. They have similar hue lines but have significantly different lightness and saturation notations. Munsell renotations and CIE values for each of the disks for the 2 yr are shown in Table 2.

Since 1939, the papers for Munsell component colors for tomato products have been reproduced many times. The relative positions in the CIE color space of the component colors for canned tomatoes are shown in Figure 2.

TABLE 1 Representative appearance standards for products under FDA regulation or USDA inspection.

Agency	Product	Specification	Determination	Reference
FDA	Cheddar cheese	Phenol equivalents; units of blue color.	Maximum transmitted light; 610 nm aqueous solution 650 nm butyl alcohol.	21CFR §19.500 (f)iii
	Canned tuna	Munsell notation; N6.3, 33.7 percent lum.refl. N5.3, 22.6 percent lum.refl.	Visual comparison; 555 nm filter, illum.A variable.	21CFR §37.1(d) and (g)
	Canned tomatoes	Munsell notation; percent components of 5R 2.6/ 13 2.5YR 5/ 12 N1 or N4	Visual comparison; disk colorimeter; min color.	21CFR §53.41(a) 1 and (b)
USDA	Frozen lima beans	USDA plastic color stds.; Lima bean green Y = 40.4 percent, x = .3715, y = .4576 4.6GY 6.9/ 7.4 Lima bean white Y = 63.5 percent, x = .3731, y = .4016 7.7Y 8.3/ 5.0	Visual comparison; illum. C.	7CFR §52.501
	Orange juice	USDA plastic color tubes; OJ 2-6	Visual comparison; illum. C., 40 percent total score.	7CFR §52.1551 7CFR §52.1581 7CFR §52.5641
	Table maple syrup	Brice color comparator; glass filters- light amber, med. amber, and dark amber.	Visual comparison; transmitted light.	7CFR §52.5961
	Fresh tomatoes for processing	USDA Tomato Color Index; $TCI = \frac{21.6}{Y_C 1/2} - \frac{3.0}{Y_C 1/2}\left[\frac{Y_C - Z_C}{X_C - Y_C}\right]$	USDA Tomato Colorimeter; non-deaerated raw juice.	7CFR §51.3317 (a)

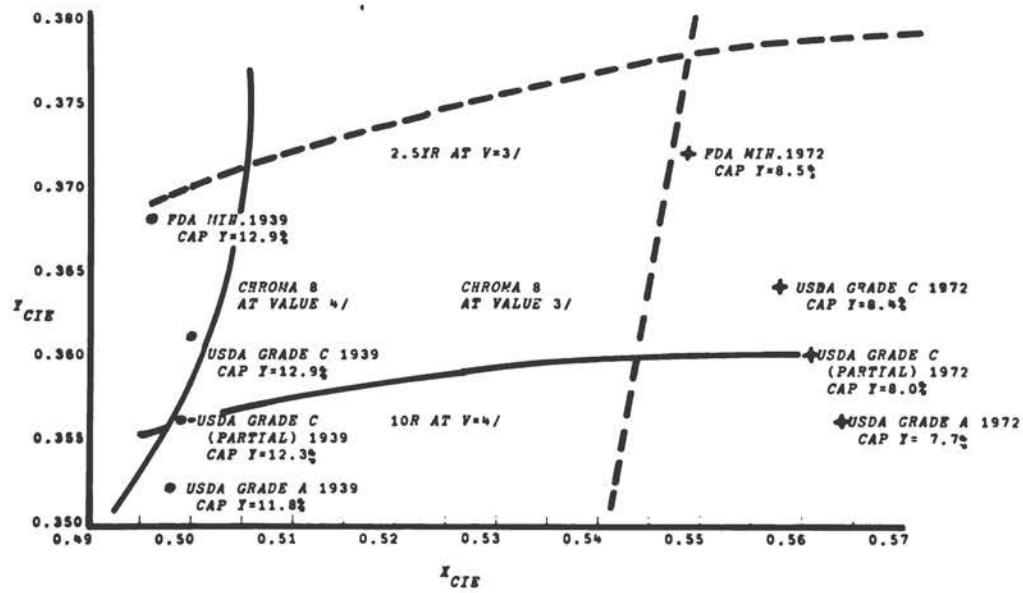


FIGURE 1 Positions in CIE color space for additive mixture of Munsell component colors for canned tomatoes, 1939 and 1972.

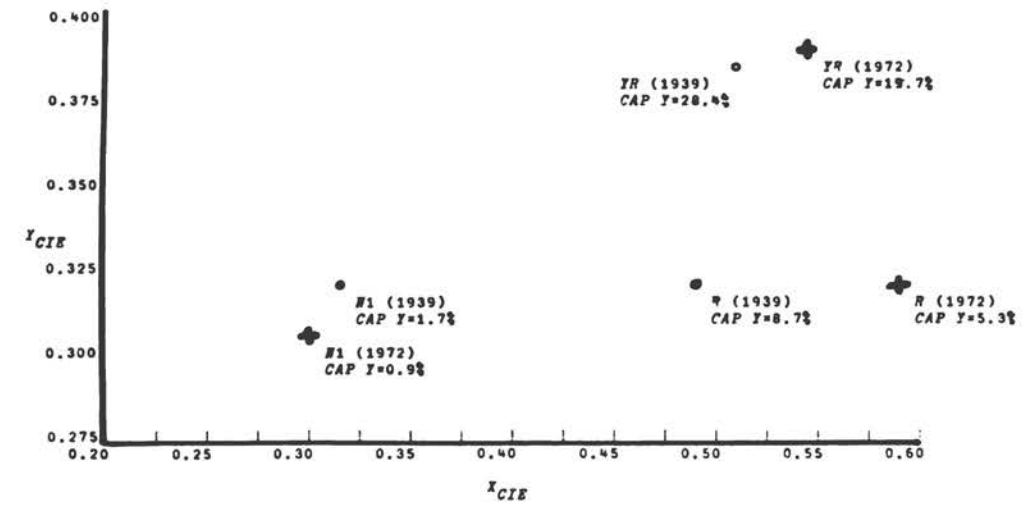


FIGURE 2 Relative position in CIE color space of the component Munsell colors for canned tomatoes, 1939 and 1972 (R=red; YR=yellow-red; and N1=black, glossy finish).

TABLE 2 CIE values and Munsell notations for tomato product grades, 1939 and 1972.

Tomato product grade	CIE values[1]						Munsell notations[2]					
	1939			1972			1939			1972		
	Percent Y	x	y	Percent Y	x	y	H	V	C	H	V	C
A	11.8	.4981	.3522	7.7	.5618	.3552	9.5R	4.0/	8.1	0.4YR	3.3/	9.6
C (partial)	12.3	.4991	.3564	8.1	.5585	.3594	0.0YR	4.1/	8.2	0.6YR	3.4/	9.5
C	12.9	.5000	.3608	8.5	.5554	.3633	0.5YR	4.2/	8.2	0.9YR	3.5/	9.5
FDA minimum	12.9	.4959	.3675	8.5	.5450	.3714	1.4YR	4.2/	7.9	1.6YR	3.5/	9.0

[1] Illuminant C, diffuse.
[2] H = hue, V = value, C = chroma.

USDA grades and FDA standards of quality should have complete notations as shown in Table 2. CIE values and Munsell renotations for a given color disk or for the product standard would be easily and precisely reproduced by a manufacturer; this would preclude the errors of the past.

REFERENCES

1. Anonymous, U.S. Standards for Grades of Processed Fruits and Vegetables, and Certain Other Products (April, 1973).

2. Code of Federal Regulations, Titles 7 and 21 (1974).

3. Munsell Color Company, Personal Communication (1957, 1960, 1964, and 1974).

4. Nickerson, Dorothy, File Correspondence (1939 and 1941).

5. Yeatman, J. N., A. P. Sidwell, and K. H. Norris, "Derivation of a New Formula for Computing Raw Tomato Juice Color From Objective Color Measurement," *Food Technol.*, *14*(1), 16-20 (1960).

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

FLAVOR

Roy Teranishi, Chairman

SEAFOOD FLAVORS -- Peter A. Lerke

Our interest in flavors is related to our work on seafood spoilage determination. We are not concerned with flavor duplication and only indirectly concerned with flavor analysis. Our primary aim is to relate decomposition odor to the presence of a specific volatile compound or compounds that need not necessarily be odoriferous.

Since the most obvious indication of decomposition of seafoods is the formation of odors, it follows that one logical way to go about measuring spoilage is to measure odor. There are two ways in which this might be attempted. One would be through sensory evaluation (a measure of true odor, especially in the qualitative sense) and the other by means of some objective qualitative or quantitative measurement. We have been working on this approach for some time.

Our first attempt involved a quantitative technique; that is, we tried to measure total odor intensity. Fresh seafood has a very mild odor and any marked increase in total odor intensity is always connected with the appearance of spoilage odors. Therefore, measuring total odor made sense. We used the Votatile Reducing Substances (VRS) Method[1,2] which consists of stripping volatiles from a liquid sample by means of an air stream and subsequently oxidizing these volatiles with alkaline potassium permanganate. This method resembles olfaction in that it is carried out at room temperature and gives results in terms of an integrated value that is the sum total of the specific "oxidizabilities" of the individual volatiles. While the VRS has proved to be of great value in seafood spoilage determination, it suffers from lack of discrimination with consequent poor precision and sensitivity.

Recently we began to investigate the usefulness of gas-liquid chromatography in tuna spoilage determination. Our procedure involves the sweeping of volatiles from an aqueous sample of canned tuna press juice with a stream of purified helium. The sample is maintained at 80°C and the volatiles are trapped in a condenser immersed in liquid nitrogen. After a given collection

period the volatiles are released into the chromatograph by warming the condenser. We use a Flame Ionization Detector, a 12-foot, 1/4-inch O.D. column, packed with 2 percent SF96 with Igepal 880 on Chromosorb G. After injection we hold 12 min at initial 20°C temperature, then program 2°/min to 100°C. The end result is a chromatogram consisting of some 50 peaks. This technique appears to offer an improvement over the VRS in that the various volatiles are now separated and we have the choice of either adding them all together again or selecting one or more as suitable indices of spoilage.

We have followed the latter approach and have identified such an index. We can now tell, on the basis of a chromatogram, whether a can of tuna is acceptable, borderline, or not passable. It must be stressed, however, that while our organoleptic parameter is odor, our objective technique does not measure it. Even if we were to take all 50 peaks and add them up, we probably would still come short of measuring odor because our conditions are so different from olfaction. When we smell an object we draw in volatiles on a stream of air and our olfactory apparatus (detector) integrates the odoriferous properties of the various substances, giving each a different weight depending on its sensitivity to it. Furthermore, it is quite probable that substances present in subthreshold concentrations also contribute to the overall impression. The GLC technique, on the other hand, differs in many respects, including gas carrier, completeness of trapping, temperature of sweeping, detector sensitivity, possible transformations on the column, etc.

Thus, what we achieve by means of GLC is only an estimation of the amount of some volatiles, which may or may not be components of the true odor as perceived subjectively. To us this is sufficient, provided at least one of the volatiles shows a close relationship to spoilage.

REFERENCES

1. Farber, L., and M. Ferro, "Volatile Reducing Substances (VRS) and Volatile Nitrogen Compounds in Relation to Spoilage in Canned Fish," *Food Technol.*, *10*, 303-304 (1956).
2. Farber, L., and P. Lerke, "Colorimetric Determination of Volatile Reducing Substances," *J. Food Sci.*, *32*, 616-617 (1968).

POULTRY AND EGG FLAVOR RESEARCH -- Lee-Shin Tsai

POULTRY FLAVOR

The research on the chemistry of poultry flavor can be categorized into two classes. One is to define the poultry flavor in terms of chemical compounds and another to solve by chemical

means the off-flavor problems that occur in the industry. For the former, the main task was to isolate and identify the flavor-contributing compounds of cooked meats and determine the precursors of these compounds in the uncooked meats. The subject has been reviewed by Thomas and coworkers[1] in 1971, by Pippen[2] in 1967, and by Lineweaver and Pippen in 1961[3]. Volatile compounds identified in cooked poultry meat include sulfides, carbonyls, alcohols, ammonia, and amines.

Hydrogen sulfide is known to contribute to the "meaty" odor of chicken. Removal of sulfur compounds from cooked chicken volatiles causes nearly complete loss of "meaty" odor. The principal precursors of hydrogen sulfide are cystine and cysteine residues in muscle proteins.

A great number of volatile carbonyl compounds were isolated and identified from cooked chicken. Removal of them from cooked chicken volatiles causes loss of "chickeny flavor." This indicates that these carbonyls are largely responsible for the characteristic chicken flavor. However, the importance of the individual carbonyls and the interrelationship of the carbonyls to the characteristic falvor is still unknown.

The volatile carbonyl compounds may be derived from the oxidation of unsaturated fatty acids or coverted from amino acids by Strecker degradation. There is little direct evidence linking the volatile carbonyls to specific precursors, except for decadienal whose principal precursor is probably linoleic acid. Specific evidence revealing the identity of precursors of other volatile compounds, such as ammonia, methylamine, and ethanolamine, has not been reported.

POULTRY OFF-FLAVOR

Poultry, chicken, and turkey diets containing high levels of fish oil or linseed oil were known since 1939 to cause off-flavor (fish odor) in cooked meat. A great deal is known about this type of off-flavor. It can be summarized as follows:

1) Linolenic acid and its higher chain homologues deposited in the carcass are the precursors of the off-flavor in cooked meat; therefore, the content of these fatty acids in diets is limited to prevent this type of off-flavor in meat[2].
2) Feeding poultry with a diet containing a high dosage of vitamin E (above the nutritional requirement) concurrently with a high concentration of polyunsaturated fatty acids can prevent the development of off-flavor in cooked meat, though it will not affect the deposition of polyunsaturated fatty acids in the carcasses[4].
3) Poultry meat containing a high level of polyunsaturated fatty acids cooked under an oxygen-free condition will not develop the off-flavor[5].

The oxidative deterioration of refrigerated and frozen poultry products is directly related to the oxidation of its lipid

moiety. Staleness and rancidity are often used to describe the extent of the deterioration. However, the real nature of staleness is not well-defined. It is not certain whether it is due to the loss of characteristic flavor or to the development of off-flavor. The chemical tests used to follow the extent of oxidative lipid deterioration, such as peroxide, carbonyl, and TBA values, correlate inconsistently with the subjective estimation.

EGG FLAVOR

The information in the literature on the chemistry of egg flavor is generally lacking. Sumita (1967)[6] reported the isolation of hydrogen sulfide from boiled eggs. Nath and coworkers (1973)[7] have identified hydrogen sulfide in the exudate of peeled soft boiled eggs and noticed the loss of egg flavor during storage was related to the loss of hydrogen sulfide in the exudate. Recently a patent was issued to M. A. Parret of Rhodia, Inc., (1974)[8] on the formulation of yolk flavor. The formula consists of hydrogen sulfide and a blend of 12 amino acids. The recent development of yolkless egg products in the egg industry has generated a demand for synthetic yolk flavor. At least one synthetic yolk flavor is available on the market. But the chemical nature of this product is a trade secret.

EGG OFF-FLAVOR

Off-flavor in shell eggs is rather rare. The reports of effects of dietary fish oil on the flavor of egg are contradictory. The problem has not been as thoroughly investigated as that in poultry meat. The majority of complaints of off-flavor of shell eggs and egg products have been traced to microorganism spoilage. It could be caused by improper washing of shell eggs and improper storage at elevated temperature, etc.

The oxidative deterioration of dehydrated egg products, specifically yolk products, has been observed at both low and ambient temperatures[9]. Glucose and mositure content, temperature, and pH are important factors in off-flavor development[10]. Generally speaking, deglucosed egg products with less than 3.5 percent moisture, of neutral pH, and stored at refrigerated temperature should have a relatively long storage life. The mechanism of the off-flavor development was postulated to involve the reaction of aldehydes formed from the autoxidation of unsaturated lipids with the amino group of phosphatidylethanolamine.

REFERENCES

1. C. P. Thomas, P. S. Dimick, and J. H. MacNeil, "Sources of Flavor in Poultry Skin," *Food Technol.*, *25*, 407 (1971).

2. E. L. Pippen, "Poultry Flavor," *Symposium on Foods -- Chemistry and Physiology of Flavors*, H. W. Schultz, E. A. Day, and L. M. Libbey, Eds., 251 (AVI Publ. Co., 1967).

3. H. Lineweaver and E. L. Pippen, "Chicken Flavor," Proceedings, Flavor Chemistry Symposium, Campbell Soup Co. (1961).

4. J. Opstvedt, E. Nygard, and E. Olsen, *Acta Agriculturae Scandinavica, 21*, 126-143 (1971).

5. L. Crawford and M. J. Kretsch, "The Effects of Cooking in Air or in Nitrogen on the Development of Fish Flavor in the Breast Meat of Turkeys Fed Tuna Oil With and Without α-tocopherol Supplement or Injection," submitted to *Food Sci.*

6. Aya Sumita, *Rinsko Eiyo, 30* (7), 849 (1967).

7. K. R. Nath, J. M. Darfler, and R. C. Baker, *Poult. Sci., 52*, 1178 (1973).

8. M. A. Perret, U.S. Patent 3,806,608 (1974).

9. O. S. Privett, O. Romanus, and L. Kline, *Food Technol., 18*, 1485 (1964).

10. M. S. Peterson and H. E. Goresline, Eds., *Stability of Dehydrated Eggs* (National Academy of Sciences-National Research Council, 1954).

BEEF FLAVOR PRECURSORS AND QUALITY ASSESSMENT -- Ahmed F. Mabrouk

Freshness is the single quality factor most consistently lacking in processed meats. This is particularly true of beef, which accounts for approximately 16 percent of the military ration dollar value and the food budget of the middle-income American family.

Batzer *et al.* (1960, 1962), Hornstein *et al.* (1960), Hornstein and Crowe (1960), and Landmann and Batzer (1962) reported a water-soluble substance in beef that, upon heating, emitted an aroma and flavor characteristic of freshly cooked beef. Their yield of this material, termed flavor precursor, was so small that detailed characterization was not accomplished. We have been able to improve the flavor precursors yield by one thousand-fold (Mabrouk *et al.*, 1969). Dialysis used in preparation of beef-flavor precursors is a time-consuming and relatively inefficient process at low concentrations. The same effect can be achieved in an ultrafiltration system in a fraction of the time of conventional dialysis, Mabrouk (1973).

Aqueous beef-flavor precursors encompass 15 classes of organic compounds: 1) glycopeptides, 2) nucleic acids, 3) nucleotides, 4) peptide bound nucleotides, 5) nucleotide sugars, 6) nucleotide sugaramine, 7) nucleotide acetylsugaramine,

8) nucleosides, 9) peptides, 10) amino acids, 11) free sugars, 12) sugar phosphates, 13) sugaramines, 14) amines, and 15) organic acids. Compounds belonging to these 15 classes contribute flavor notes to the overall impression of cooked beef.

Cytidine-5'-monophosphate, adenosine-5'-monophosphate, guanosine-5'-monophosphate, uridine-5'-monophosphate, and inosine-5'-monophosphate were identified in beef-flavor precursors. Inosine-5'-monophosphate accounted for 70 percent of the total nucleotides content (200 mg/100 g of fresh beef). Cytidine, adenosine, guanosine, uridine, and inosine are the nucleosides produced upon degradation of beef nucleotides.

Phosphoserine, taurine, aspartic acid, threonine, serine, glutamic acid, proline, glycine, α-alanine, cystine, valine, methionine, isoleucine, leucine, tyrosine, phenylalanine, β-alanine, hydroxylysine, lysine, and arginine were identified in beef-flavor precursors (Jarboe and Mabrouk, 1974). Anserine was the only peptide found in beef extract.

Glucose, fructose, ribose, inositol, glucosamine, glucose 6-phosphate, fructose 6-phosphate, fructose 1,6-diphosphate are the major components of the carbohydrate fraction of beef-flavor precursors. Lactic and succinic acids were identified in beef-flavor precursors (Jarboe and Mabrouk, 1974).

Fractionation of beef aqueous extract by ultrafiltration and preparative gel-permeation chromatography on three consecutive columns of Sephadex G-25, G-15, and G-10, resulted in a fraction that exhibited very intense meaty aroma upon heating dry. Upon testing by thin-layer chromatography and thin-layer electrophoresis, it was found to be heterogeneous. Fractionation by preparative thin-layer chromatography produced seven fractions. Gel-permeation chromatography of the third fraction of the newly produced seven fractions yielded three components. This indicates the complexity of beef-flavor precursors.

The similarity in composition of the free amino acids and reducing sugars in pork, beef, and lamb and the similarity of organoleptic qualities obtained from aqueous extracts of these meats suggest that a basic meaty flavor is common to the lean meat portion of all meats regardless of species, according to Hornstein, *et al.*, (1967). They attributed the characteristic associated with pork, beef, and lamb to their fat content. In 1969, Pippen *et al.* reported that cooked chicken aroma cannot be derived from its fat content alone as fat of raw poultry, free of solid tissues, and washed with water did not develop cooked chicken aroma upon heating. There are a number of reports about the undesirable odor of heated fat from adult male pig (boar), Craig *et al.* (1962), Williams *et al.* (1963), Plimption *et al.* (1962, 1966). Boars generally sell for meat at lower price per pound than barrows or gilts of similar age and weight because the meat has a characteristic unpleasant odor. Patterson (1968) identified 5α-androst-16-ene-3-one as the compound responsible for taint in boar fat. In 1971, Beery *et al.* confirmed Patterson's results.

At present, all quality measurement is geared to subjective evaluation. This situation is not likely soon to change, but we should direct our resources toward more objective evaluation. Gas-chromatographic analysis will permit the development of flavor and odor chemical profiles. When the profile information is available, rather precise flavor characterization will be possible.

Meat flavor must be viewed with respect of desirable and undesirable aspects. The undesirable quality changes caused by lipid hydrolysis or oxidative rancidity have had an important effect on processing and storage practices.

Lipolytic enzymes split off the fatty acids from triglycerides to leave monoglycerides or diglycerides and ultimately free glycerol. This type of deterioration usually proceeds slowly in the fatty tissues of meat, but is accelerated in the presence of lipolytic moulds, yeast, or bacteria. Free fatty acids liberated by enzymatic hydrolysis of animal fats have no serious effect on flavor. The developed "tainted off-flavor" in the fats of chilled beef on storage may be due to protein-degradation products solubilized and concentrated in fats. Titrimetric determination of the free fatty acids appears to be the most reliable method for assessing hydrolytic deterioration.

Both sensory and chemical methods are used to measure "off-flavors" caused by oxidative rancidity. Chemical methods for the measurements of fat deterioration due to oxidative rancidity have been reviewed by Lea (1939, 1962), Watts (1961), Pearson (1968). Attempts to use objective chemical methods: [peroxide value, Kreis test, thiobarbituric acid (TBA), benzidine test, Girard T. Reagent, 2,4-dinitrophenylhydrazine, and anisidine test] are of limited merit in expressing rancidity as the values derived from these tests only indirectly reflect rancidity.

REFERENCES

Batzer, O. F., Santoro, A. T., Tan, M. C., Landmann, W. A., and Schweigert, B. S., *J. Agric. Food Chem.*, *8*, 498 (1960).

Batzer, O. F., Santoro, A. T., and Landmann, W. A., *J. Agric. Food Chem.*, *10*, 94 (1962).

Hornstein, I.,and Crowe, P. F., *J. Agric. Food Chem.*, *8*, 494 (1960).

Hornstein, I., Crowe, P. F., and Sulzbacher, W. L., *J. Agric. Food Chem.*, *8*, 65 (1960).

Landmann, W. A.,and Batzer, O. F. (to American Meat Institute Foundation) U.S. Patent 3,047,399 (July 31, 1962).

Mabrouk, A. F., Jarboe, J. K., and O'Connor, E. M., *J. Agric. Food Chem.*, *17*, 5 (1969).

Mabrouk, A. F., *J. Agric. Food Chem.*, *21*, 942 (1973).

Jarboe, J. K.,and Mabrouk, A. F., *J. Agric. Food Chem.*, *22*, 787 (1974).

Hornstein, I., *The Chemistry and Physiology of Flavors*, Schultz, H. W., Day, E. A., and Libbey, L. M., Eds., 228-250 (AVI Publ. Co., Westport, Connecticut, 1967).

Pippen, E. L., Mecchi, E. P., and Nonaka, M., *J. Food Sci.*, *34*, 436 (1969).

Craig, H. P., Pearson, A. M., and Webb, N. B., *J. Food Sci.*, *27*, 29 (1962).

Williams, L. D., Pearson, A. M., and Webb, N. B., *J. Animal Sci.*, *22*, 166 (1963).

Plimpton, R. F., Jr., Cahill, V. R., Teague, H. S., and Ockerman, H. W., *J. Animal Sci.*, *21*, 984 (1962).

Plimpton, R. F., Jr., Ockerman, H. W., Cahill, V. R., and Teague, H. S., *J. Animal Sci.*, *24*, 867 (1966).

Patterson, R. L. S., *J. Sci. Food Agric.*, *19*, 31 (1968).

Beery, K. E., Sink, J. D., Patton, S., and Zegler, J. H., *J. Food Sci.*, *36*, 1086 (1971).

Lea, C. H., *Rancidity in Edible Fats* (Chemical Publ. Co., New York, 1939).

Lea, C. H., *Symposium on Foods: Lipids and Their Oxidation*, Schultz, H. W., Day, E. A., and Sinnhuber, R. O., Eds. (AVI Publ. Co., Westport, Connecticut, 1962).

Watts, Betty M., *Advan. Food Res.*, *5*, 1 (1961).

Pearson, D., *J. Sci. Food Agric.*, *19*, 553 (1968).

CORRELATION OF CHEMICAL ANALYSIS WITH FLAVOR CHANGES --
Charles Merritt, Jr.

There is relatively no problem today in the qualitative and quantitative determination of the trace volatile components that are normally associated with the various attributes of flavor. Methods employing gas chromatography and mass spectrometry as well as newer developments with liquid chromatography (such as detectors employing atmospheric pressure ionization and chemical ionization mass spectrometry, or fourier transform infrared spectrophotometry, fourier transform nuclear magnetic resonance spectrometry, or microwave spectroscopy together with the use of computers for analytical data processing) now provide very sophisticated means for the determination of the composition of the volatile and not-so-volatile trace components in food. The problem is to correlate these analytical results with the sensory observations that describe the flavor, or perhaps the deterioration of flavor, in a food. It is very difficult -- and probably up to now not possible -- to correlate the flavor of the complex foods with the chemical composition of the volatile or other trace

components. There is a great multiplicity of these components and the relative contribution of each to the sensory response cannot be evaluated.

On the other hand, it is not so difficult to observe changes in the composition of these components, and in many cases these changes can be correlated with changes in flavor. In this context, at least, analytical methods can be utilized to provide an objective evaluation of flavor quality.

A few examples may be cited of correlations of chemical analysis with flavor changes that have been achieved. The simplest case may be illustrated by the loss of flavor from a dehydrated product. The situation for cabbage[1] may be depicted as follows:

$$\text{Cabbage} \xrightarrow{\text{dehydrate}} \text{loss of volatiles}$$

$$\text{Dehydrated Cabbage} \xrightarrow[H_2O]{\text{reconstitute}} \text{loss of flavor}$$

$$\text{Dehydrated Cabbage} \xrightarrow[H_2O + \text{enzyme}]{\text{reconstitute}} \text{flavor restored}$$

Chemical analysis has shown the loss of flavor in dehydrated cabbage to be related to the loss of isothiocyanates. A chromatogram that demonstrates the enzymatic regeneration of isothiocyanate is shown in Figure 1.

A second example is the formation of a particular component that may introduce an off-flavor or otherwise spoil the product. There are 100 or more volatile compounds in a cheddar cheese[2] and the role of each cannot be related rigorously to the flavor. When something goes wrong, however, it is immediately apparent. In this case an undesirable flavor may be attributed to an increase in sulfur compounds, particularly hydrogen sulfide.

Some flavor changes are very complex, but nearly all are due to some kind of spoilage. Freeze-dried eggs[3], for example, undergo a slow deterioration of quality in storage that is reflected in panel scores as a loss of a quality called mildness. Actually, chemical analysis shows that several components are produced in storage that can be related to oxidation of the lipid in the eggs. It is not necessary to correlate any particular individual compound. The volatile compounds as a group are indicative of the changes occurring, and, as seen in Figure 2, can be used to provide an objective measure of storage time and the concomitant flavor loss.

In a similar way spoilage of fish can be followed by the analysis of the trace chemical components. A very simple test for spoilage of fish[4] can be devised by measuring the dinitrophenylhydrazones as a function of storage.

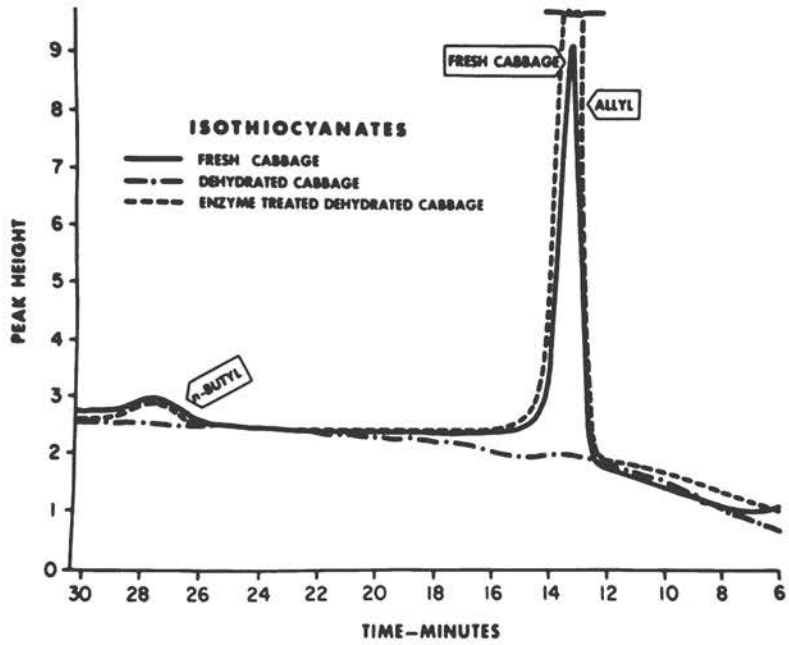


FIGURE 1 Chromatogram of volatile isothiocyanates isolated from cabbage.

Recently, odors of fresh and spoiled chicken have been studied in relation to the chemical composition of the volatiles. The compounds identified are typically characteristic of bacterial contamination. Chicken flesh stored in the refrigerator for 4 days does not normally develop any detectable odor. It has the sweet, odor-free character of fresh chicken. It has no odor or a bland odor up to about 8 days, but by 11 days it begins to develop a slight off-odor, and at 15 days and over, it is putrid.

When the sensory observations are correlated with the analytical results a close relationship is seen. There is essentially no increase in volatiles during the period in which the chicken is observed to retain its fresh character. After about 7 or 8 days, the onset of spoilage is readily detectable by an increase in volatile constituents although it may not be observed by smell until after about 11 days. When the chemical compounds indicating spoilage are first observed, the amount detected is about 80 ppb of total volatile compounds.

From the various studies cited here and many others now known, it may certainly be concluded that chemical analyses may be devised to provide objective methods for the evaluation of flavor quality in foods.

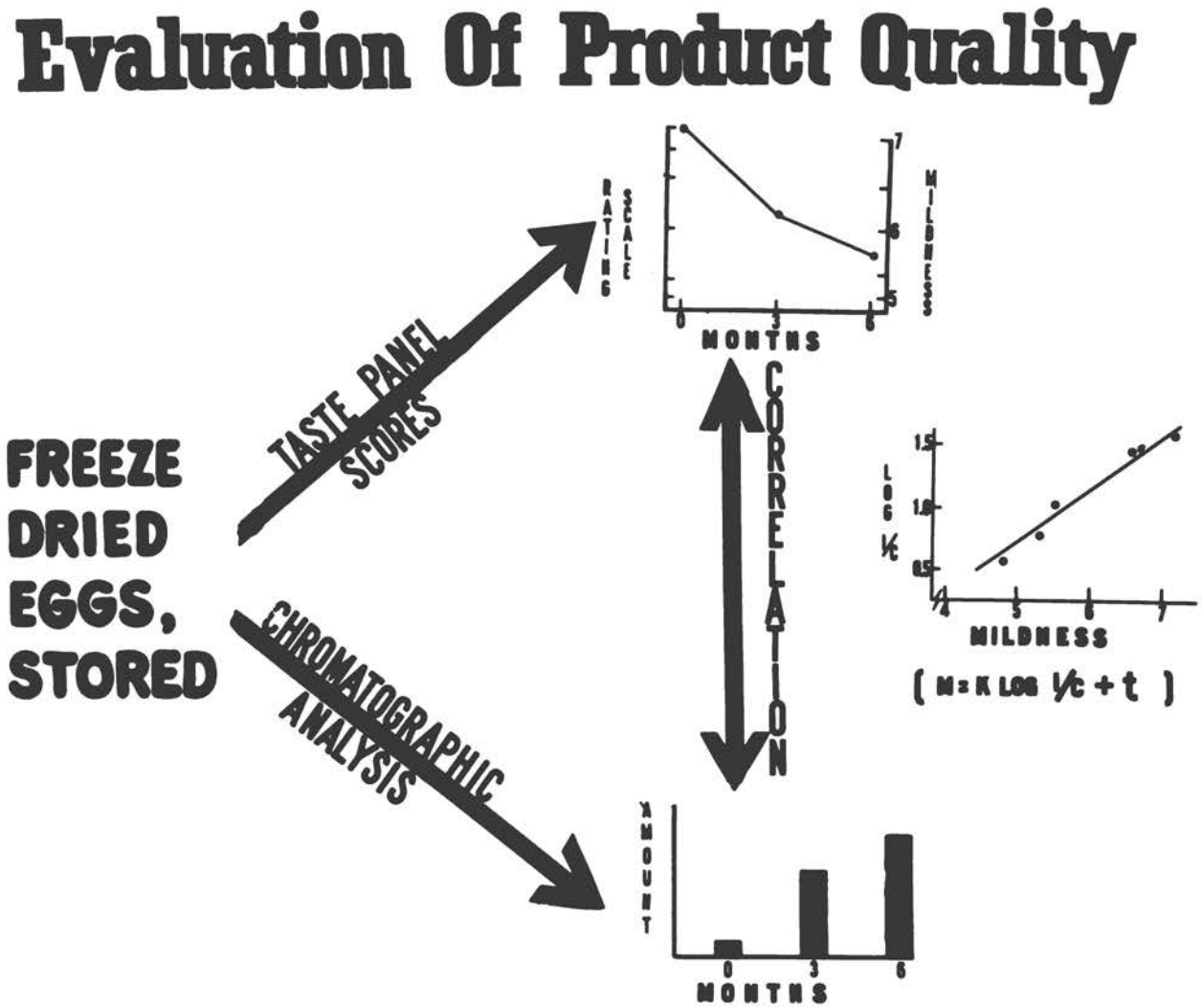


FIGURE 2. Outline of study showing correlation of chemical analysis with sensory evaluation of freeze-dried eggs.

REFERENCES

1. Bailey, S. D., Bazinet, M. L., Driscoll, J. L., and McCarthy, A. K., *J. Food Sci.*, *26*, 163 (1961).

2. Morris, H. A., Angelini, P., McAdoo, D. J., and Merritt, C., Jr., *J. Dairy Sci.*, *49*, 710 (1966).

3. Angelini, P.,and Walts, C. C., Presentation at National Meeting, I.F.T., Portland, Oregon, 1966.

4. Mendelsohn, J. M.,and Steinberg, M. A., *Food Technol.*, *16*, 113 (1962).

OBJECTIVE MEASUREMENT OF FLAVORS IN FATS AND OILS --

Gerard J. Henning

Fats and oils as such are relatively simple substrates for flavor research since volatile flavor components can easily be separated from the much less volatile triglycerides. Isolation and identification of less-volatile components (e.g., flavor precursors) is much more complicated. The same is true for the analysis of flavors in fat-containing products.

Though there is a host of data on the occurrence and origin of flavors and off-flavors in fats and fat-containing products, we are not anywhere near objective measurement of flavor in fats and their derived products. In selected cases, instrumental analysis can be used as a replacement for subjective flavor assessment.

Measurement of the free fatty acid level in oils and fats is an easy method for getting an impression about maltreatment of oil fruits or seeds during harvesting, transport, and storage. In principle, objective measurement of the level of δ-lactones in coconut oil, of hexanal/hexenal in refined soybean oil, and of 2,6-nonadienals in tallow could be used for flavor-quality grading. Similarly, the presence of roast-type flavor components such as pyrazines would indicate that the oil seed was subjected to heat drying prior to extraction[1].

Determination of the level of lower fatty acids or longer chain methylketones would be indicative of the presence of fat-splitting microorganisms, leading to soapy flavor or ketone rancidity.

Much work has been devoted to the characterization of the various flavors and off-flavors in milk and milk products[2,3]. Some of the flavor components identified can be used as markers to indicate the prior history of the products[4].

High amounts of hydroxymethylfurfural, one of the products of non-enzymatic browning, are indicative for excessive heat treatment during production of dried whole milk[5], spray-dried skim milk[6], or sterilized milk[7]. Much higher levels of diacetyl are present in bacteriologically soured milk than in sweet milk,

whereas the occurrence of sulphur compounds like methional would be indicative of light damage[8] or poor souring conditions.

Though the flavor of butter varies according to season, feed of the cattle, and other conditions, sufficient knowledge seems available to allow objective flavor grading of butters. Since subjective grading is done on many other aspects of quality also, objective methods are not applied.

High amounts of free fatty acids (notably butyric) in butter are indicative of improper conditions during souring. The absence of lactones in butter fat would indicate heat treatment, followed by deodorization, while high levels of lactones and methylketones are indicative of hydrolysis of precursors during storage at higher temperatures.

Determination of the level of free fatty acids can be used for following the ripening in cheddar cheese, levels of methylketones would indicate the age of Roquefort. Quality grading will have to be done by determining several classes of flavor compounds simultaneously; e.g., free fatty acids and amino acids in Edam cheese[9].

Since margarine in general is artificially flavored, there is no point in objectively determining either the total level or the presence of certain flavor components. For studying flavor losses during storage, however, objective methods are regularly used.

Finally, taking margarine as an example, a general remark on objective measurement of flavor seems appropriate.

When margarine (or butter) is put in the mouth, the fat crystals will melt. Depending upon the stability of the resulting water-in-oil emulsion, flavor components present in the aqueous phase will be set free to different degrees, leading to differences in the flavor perceived[10].

For objective assessment of flavor, therefore, in addition to the techniques already available, new procedures by which the release of flavor components with time can be measured will have to be developed. Only in this way can we hope to arrive at a really objective assessment of flavor.

REFERENCES

1. J. A. Newell, M. E. Mason, and R. S. Mattock, *J. Agric. Food Chem.*, *15*, 767 (1967).
2. J. E. Kinsella, S. Patton, and P. S. Dimick, *J. Am. Oil Chem. Soc.*, *44*, 449 (1967).
3. D. A. Forss, *J. Am. Oil Chem. Soc.*, *48*, 702 (1971).
4. K. Eichner, *Deutsche Lebensmittelrundschau*, *69*, 4 (1973).
5. M. Keeney and R. Bassette, *J. Dairy Sci.*, *42*, 945 (1959).
6. P. G. Kliman and M. J. Pallansch, *J. Dairy Sci.*, *51*, 498 (1968).

7. E. G. Samuelsson and P. Nielsen, *Milchwissenschaft, 25*, 541 (1970).

8. P. E. Ballance, *J. Sci. Food Agric., 12*, 532 (1961).

9. L. A. M. Ali, *Mededelingen Lanbouwhogeschool Wageningen, 60*, 1-64 (1960).

10. P. B. McMulty and M. Kavel, *J. Food Technol., 8*, 309,415 (1973).

MEAT FLAVOR -- Ira Katz

Before we can use objective methods of evaluation for red meat flavors we first must understand the chemistry of this flavor. Unfortunately there is probably no other major food product for which the lack of scientific knowledge relating to the flavor and its formation is so apparent.

At one time it was thought that our ignorance in this area was due to a lack of intensive scientific research using modern analytical procedures. Once these procedures were brought to bear upon the problem the answers would be forthcoming. After many years of research by different groups it is apparent that this is not the case. This area of research is leading us into new areas of biochemistry, analytical chemistry, and synthetic organic chemistry. It is continually demanding new developments in analytical chemistry as a means of finally solving the problem.

In spite of the difficulty involved in this area of research, the importance of the product has allowed a number of research groups to continue their investigations; as a result, progress is being made.

FATTY FLAVOR

Fat or triglycerides through oxidative degradation undoubtedly contribute to the flavor of meat. This is a specific fat flavor and does not appear to be a basic meat character.

Experiments by Hornstein and Crowe[1,2] and Wasserman and Talley[3] helped to clarify the relative contribution of fatty and meaty tissue. They found that the species-specific flavor of lamb, pork, and beef was attributable to products resulting from heating the fatty tissue; heating aqueous extracts of lean muscle tissue showed that the basic meaty character common to all these species came from non-lipid, water-soluble precursors. This basic meaty character was similar in flavor characteristics regardless of the species from which it was isolated. Addition of the fatty tissue resulted in the development of a species-specific flavor upon heating. Wasserman and Talley[3] presented evidence that panel members found it difficult to discriminate between the meat samples from the various sources. For example, addition of lamb fat to veal significantly increased the identification of veal as lamb.

However, these results have to be reconsidered in light of more recent evidence that indicates the importance of water-soluble components of adipose tissue to flavor formation[4,5].

A myriad of saturated and unsaturated aldehydes and some ketones have been identified in chicken volatiles, the major precursor of which must be unsaturated lipid material[6]. One such example is 2,4-decadienal. It was found that chicken fat had little effect on the taste of chicken broth and meat, but did have an effect on the aroma[7,8]. Further, 2,4-decadienal, an important carbonyl compound in chicken extracts, arose from tissue fatty acids[9]. Earlier, it was shown that 2,4-decadienal was a major compound formed by heating linoleate to high temperatures in the presence of water[10]. And linoleic acid constitutes about 16 percent of the total fatty-acid content of chicken fat[11]. Thus, we can conclude that an important component of chicken flavor, 2,4-decadienal, arises from oxidative breakdown of esterified or free linoleic acid during cooking.

It has been shown that cooking poultry in air results in greater quantities of carbonyls arising from fatty acid oxidation[12]. This was especially true of hexanal and 2,4-decadienal. Several hundred volatile and non-volatile chemicals that have now been identified in chicken have been described[6].

Thus, there is little doubt that the fat of beef, pork, and lamb contributes some of the most characteristic and potent flavor chemicals of the overall melange of flavor constituents formed during cooking.

MEAT-FLAVOR CHEMICALS

In recent years flavor-analyzing chemists have isolated and identified a large number of aliphatic, aromatic, and heterocyclic flavor chemicals in cooked meat products. Most of these chemicals have been found in other cooked foods. Although they probably contribute in some way to cooked-meat flavor, they are undoubtedly not responsible for the basic meaty character. However, in addition to these chemicals, a number of structurally unique chemicals, not found in other foods, have also been found. These chemicals are shown in Table 1.

The two furanones are extremely interesting; van den Ouweland and Peer[19] have reported that the reaction of the dimethyl compound with hydrogen sulfide results in the formation of a meat-like flavor. Some of the chemicals identified in this model system are shown in Figure 1. Although these chemicals have never been identified in meat or other heated foods, their presence can be predicted *a priori* and their discovery is a challenge to modern analytical chemists.

TABLE 1 Chemicals isolated from cooked beef.

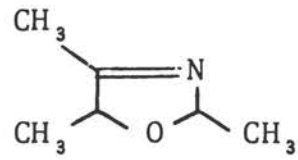
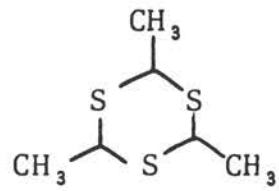
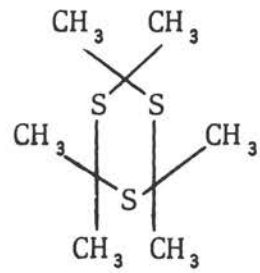
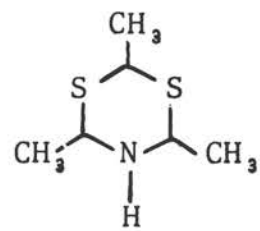
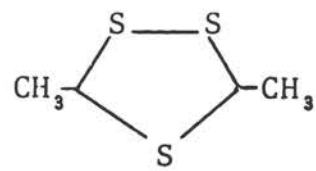
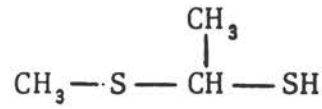
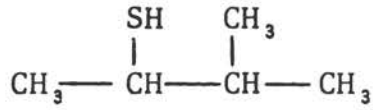
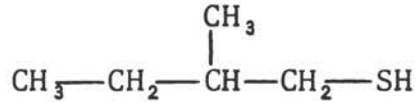
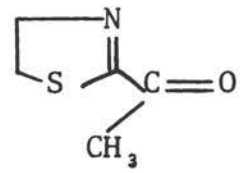
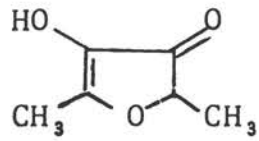
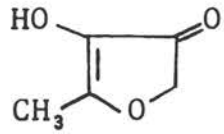
2,4,5-Trimethyl-Δ^3-oxazoline[13,14]	
2,4,6-Trimethyl-S-trithiane[15] (Trithioacetaldehyde)	
2,2,4,4,6,6-Hexamethyl-S-trithiane[15] (Trithioacetone)	
5,6-Dihydro-2,4,6-trimethyl-1,3,5-dithiazine[15,16] (Thialdine)	
3,5-Dimethyl-1,2,4-trithiolane[13,14,15,16]	
1-Methylthioethanethiol[16]	

TABLE 1 Chemicals isolated from cooked beef (continued).

3-Methyl-2-butanethiol[15]	
2-Methyl-1-butanethiol[15]	
2-Acetyl-2-thiazole[17]	
4-Hydroxy-2,5-dimethyl-3(2H) furanone[18]	
4-Hydroxy-5-methyl-3(2H) furanone[18]	

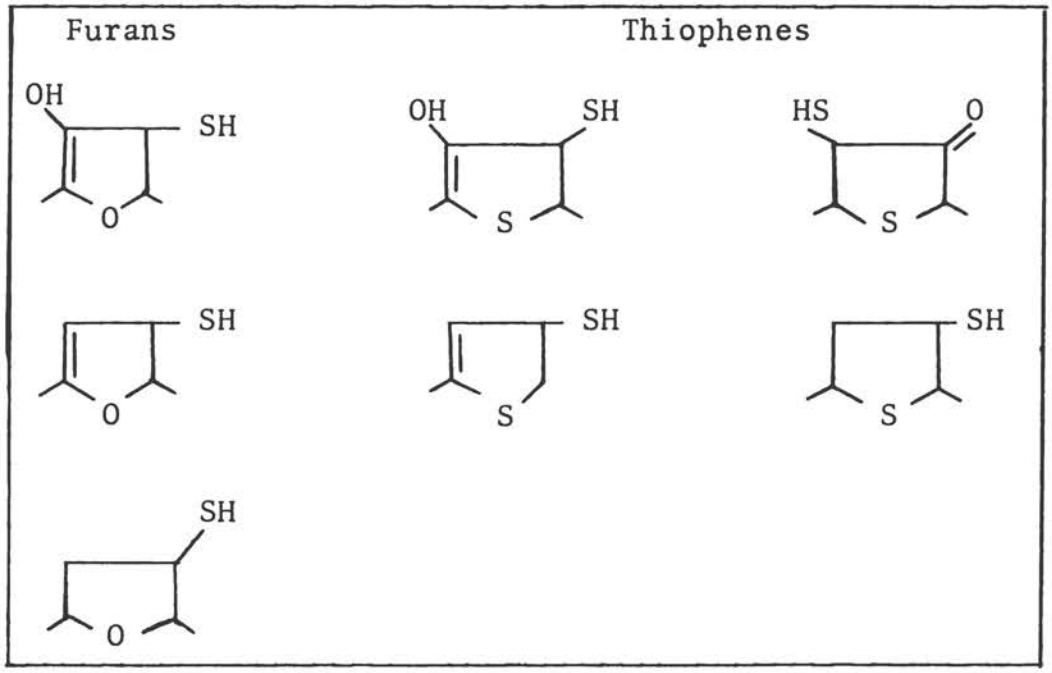


FIGURE 1 Chemicals identified in furanone-H_2S reaction product.

SUMMARY

The current investigations of meat flavor have led to and continue to result in the discovery of new flavor chemicals. As this work continues we will slowly begin to understand the chemistry of meat flavor, including the differences between rare, boiled, roasted, and otherwise cooked meat flavors. Objective methods of evaluation cannot be fully utilized until this information is in hand.

REFERENCES

1. Hornstein, I., and Crowe, P. F., *J. Agric. Food Chem.*, *8*, 494 (1960).
2. Hornstein, I., and Crowe, P. F., *J. Gas Chromatogr.*, *128* (1964).
3. Wasserman, A. E., and Talley, F., *J. Food Sci.*, *33*, 219 (1968).
4. Pepper, F. A., and Pearson, A. M., *J. Agric. Food Chem.*, *19*, 964.
5. Wasserman, A. E., and Spinelli, A. M., *J. Agric. Food Chem.*, *20*, 171 (1972).

6. Wilson, R. A., and Katz, I., *J. Agric. Food Chem.*, *20*, 741 (1972).

7. Pippen, E. L., Campbell, A. A., and Streeter, I. V., *J. Agric. Food Chem.*, *2*, 364 (1954).

8. Peterson, D. W., "The Source of Chicken Flavor", *Chemistry of Natural Food Flavor*, 167 (National Academy of Sciences-National Research Council, Washington, D.C., 1957).

9. Lineweaver, H., and Pippen, E. L., *Proceedings, Flavor Chem. Symposium* (Campbell Soup Co., Camden, New Jersey, 1961).

10. Patton, S., Barnes, I. J., and Evans, L. E., *J. Am. Oil Chem. Soc.*, *36*, 280 (1959).

11. Mecchi, E. P., Pool, M. F., Bekman, G. A., Hamachi, M., and Klase, A. A., *Poult. Sci.*, *35*, 1238 (1956).

12. Pippen, E. L., and Nonaka, M., *J. Food Sci.*, *28*, 334 (1963).

13. Chang, S. S., Hirai, C., Reddy, B. R., Herz, K. O., and Kato, A., *Chem. and Ind.*, 1639 (1968).

14. Hirai, C., Herz, K. O., Pokorny, J., and Chang, S. S., *J. Food Sci.*, *38*, 393 (1973).

15. Wilson, R. A., Mussinan, C. J., Katz, I., and Sanderson, A., *J. Agric. Food Chem.*, *21*, 873 (1973).

16. Brinkman, H. W., Copier, H., de Leuw, J. J. M., and Tjan, S. B., *J. Agric. Food Chem.*, *20*, 177 (1972).

17. Tonsbeek, C. H. T., Copier, H., and Plancken, A. J., *J. Agric. Food Chem.*, *19*, 1014 (1971).

18. Tonsbeek, C. H. T., Plancken, A. J., and von der Weerdhof, T., *J. Agric. Food Chem.*, *16*, 1016 (1968).

19. van den Ouweland, G. A. M., and Peer, H. G., assigned to Unilever, West German Patent, 1,932,800, January 8, 1970.

CONCLUDING STATEMENT -- Roy Teranishi

We are concerned with the problems of getting the best available food with the money allotted for the men in our Armed Forces. A Consumer Evaluation of Air Force Food Service, by L. G. Branch, H. L. Meiselman, and L. E. Symington*, showed that quality of food is by far the most important factor. The quality of various preserved and processed foods has been improved considerably in the last few decades; however, we must continue to improve the quality of the food served under new and different conditions to

---

*L. G. Branch, H. L. Meiselman, and L. E. Symington, "A Consumer Evaluation of Air Force Food Service", Technical Report 75-22FSL (U.S. Army Natick Laboratories, Natick, Massachusetts, 1974).

those who are serving to protect our country. With this goal in mind, this Round Table discussion was held to summarize the current status of objective methods for evaluating quality of meats.

It was the general consensus of the discussants that meat quality, cooked or raw, cannot be measured objectively at this time. More success has been achieved with fruits and vegetables; for example, see the discussion by C. Merritt, Jr., on the enzymatic regeneration of isocyanate in dehydrated cabbages. G. J. Henning pointed out various areas in which instrumental analysis could be used to follow off-flavors or prior history of products. I. Katz has listed some compounds found in cooked meat volatiles. Some of these compounds have very low thresholds. To analyze for such compounds is a severe challenge to analytical chemists.

Although much more research is necessary to understand fully the chemistry of meat flavor, some topics were presented that seem promising for development of quantitative, objective methods that are related to sensory tests. As such methods are developed, we hope that they will be applied quickly and incorporated in specifications to ensure uniformly better food for our Armed Forces.

The chairman of this Round Table discussion of Flavor thanks all the discussants for their enthusiastic participation and hopes that these discussions and references provided will spur on much needed research in this area.

PREDICTION OF FAT STABILITY

LeRoy Dugan, Jr., Chairman

OPENING REMARKS

This session has been convened to bring together a group of scientists who over the years have been seriously concerned with and dedicated to maintaining the stability of fats and oils both as separate entities and as components of food systems. In this group are people who have contributed greatly to knowledge concerning deteriorative changes in fats and relating to flavor and other characteristics developed in foods because of these changes. A major goal of these many studies has been to provide sufficient understanding of the nature of the problems of stability to permit establishment of procedures for processing, storing, and using fats in such ways that foods containing them shall have enhanced keeping quality.

The evaluation of keeping quality has been dependent upon a variety of subjective tests involving taste and smell as well as objective methods based on physical and chemical principles. Many of the tests and procedures used have sought to measure the current status of fat quality; others have sought to determine how long fats and oils and foods containing them will maintain their favorable qualities.

It is for the purpose of discussing the chief elements of knowledge relating to these evaluation studies that this Round Table session on "Prediction of Fat Stability" has been convened. It is hoped that the discussions during the next 2 hr will help to focus on this problem and give us a clearer view of the potentials for and pitfalls in the prediction of the stability of fats.

I should now like to introduce the members of the discussion group and proceed directly into the discussion period. We will plan for each member to have a few minutes for proposing and reporting information relative to his area of interest. Then we will invite questions from the audience and allow the discussions to take the course of most apparent interest.

REMARKS BY RAYMOND H. BOWERS

Thus far at this Symposium we have been exposed to various methods that can be used to study lipid oxidation in foods. Several methods have been presented for objectively determining the extent of lipid oxidation and the applicability of such methods for predictive purposes. Research people understand the problems of lipid oxidation fairly well but even so there are problems of agreement as to the meaning of the results from these various study methods.

As analytical results move from the laboratory and into commercial channels the problems become worse. Non-research-oriented people in the food business expect objectivity from us and are expecting a "magic number" of some kind that will solve all their problems. They can then put this "magic number" on a manufacturing sheet or purchasing specification and everybody will be happy.

Unfortunately, we research people are rather short on our supply of "magic numbers." In the edible oil industry we deal principally with three tests to provide numbers: AOM, Oxygen Bomb, and the Schaal Test. Of course, we back up these numbers with numbers from other tests, such as the iodine value, solid fat index, and various melting points, which also help in predicting fat stability. When we get away from the relatively simple shortenings and edible oil systems and into foods in general, our "magic numbers" are still in short supply. The Oxygen Bomb, Schaal Tests, and, in very special cases, TBA values constitute our supply.

Any of these tests are useful yet limited, for many are attempting to measure components of a system that are apparent organoleptically at extremely low concentration levels due to their low "threshold values." However, the sensitivity of many tests cannot be relied upon to be consistent at such low levels. One test that is generally useful in this respect is the TBA test for malonaldehyde, which has good applicability to meats, fish, and dairy products. Other carbonyl tests frequently lack the sensitivity necessary for detection at early levels of off-flavor development. The AOM test is empirical but useful although it is difficult to correlate with other methods of evaluation. This is probably true of most accelerated methods.

There appears to be a reluctance on the part of the food industry to add to our supply of "magic numbers" by placing meaningful organoleptic criteria on manufacturing or specification sheets. When they are there, they take the form of such unhelpful statements as "typical," "bland," or "free of objectionable flavor and odors."

It is apparently assumed that organoleptic evaluations are too subjective to be used. In research we all use expert, trained, and profile panels as very effective adjuncts to our research programs. Why can't we add to our supply of "magic numbers" by considering more carefully an increased reliance on organoleptic evaluations?

In the dairy industry there is already precedence for such techniques. No one in the dairy industry has any problem understanding the significance of 93 score butter. So why not adapt such a system to other foods?

Very probably this cannot be done for some foods, but wherever it is found that oxidative rancidity is a significant problem in a food, then evaluation by organoleptic means might well be the method of choice.

REMARKS BY ALBERT S. HENICK

Requirements are imposed upon military rations from several quarters and often lead to seeming incompatibility. The Surgeon General prescribes caloric, protein, vitamin, and mineral content, along with qualitative and quantitative limitations on lipids. Users demand high acceptability, low weight and bulk, and long-storage stability. Purchasers and suppliers insist upon keeping costs down by using standard commercial items and procedures. High caloric density means high fat content, which, together with requirements for high polyunsaturated fats, works against good stability. Restrictions on packaging costs also operate against maximum stability.

Fat-stability techniques of an earlier age, such as hydrogenation, inert gas packaging, refrigerated storage, etc., are no longer acceptable. Early development of rancidity in fatty items such as bakery mixes, margarine, peanut butter, salad dressing, and shortenings is the inevitable result. New processing technologies -- dehydration, intermediate moisture -- add to the problem. Lack of reliable test methods for the quality of fatty components in fabricated foods and for predicting stability of the finished products inhibit the development of improved processes and products.

Several methods are available for determining quality and stability in oils and shortenings. These methods, singly or in combination, are fairly useful for predicting shelf life of the oils and shortenings and somewhat less useful for predicting the stability of food products containing them. The major military problem arises from using these methods on lipids separated from food items to predict shelf life of the items. For example, the AOM stabilities of oleo stocks are good predictors of the shelf life of margarines made from them, but the AOM stabilities of the oil separated from the margarines are not.

Similar problems exist in attempting to predict shelf life of peanut butters by AOM test of recovered oil, or by oven or bomb tests on the peanut butters. The gas-chromatographic methods under study by DuPuy at the Southern Regional Research Center, USDA, offer some real possibilities for valid and sensitive measurement of quality and stability in peanut butter and similar products.

Because many systems are complex mixtures of substances and the effects on fat stability are varied, it is essential to develop knowledge about the various changes over a great range of component variables. This by necessity will involve a vast testing program, which has as its major limitation the economics of staff, time, equipment, and supply. Since industry needs to classify products according to stability and quality characteristics, the first line of attack must be at the agencies of management. It is there that decisions must be made to support the programs needed for information acquisition.

REMARKS BY GLEN A. JACOBSON

Prediction is an exercise that may be hazardous to one's reputation. If all the variables necessary for accurate predictions are at hand, then the guesswork can be minimized. However, some of the challenge and excitement is then taken out of the process of prediction.

Although much is known about the prediction of stability in fats, much still remains to be learned, and only a part of the known data can be applied directly to the flavor stability of fats. By "flavor stability" I mean the ability of a fat or fat-bearing product to maintain a desired flavor during a useful specified time under specified conditions. Much of what I'll present will relate to flavor stability. There is no advantage to a 300-hr AOM in a hardened vegetable oil if it develops some 2-pentylfuran off-flavor on standing in the light.

Usually, no one method of predicting fat stability is completely satisfactory in a given application, but let us review some of the commonly used methods of predicting fat stability and cite examples of how some can be important to a food manufacturer.

The AOM stability test remains one of the more important tests for predicting stability in the world of commerce. We find it useful in determining the potential performance of frying fats by heating the fats to 190°C and measuring the decline of AOM stability over a 6-hr period. The AOM test, on the other hand, must be used with caution in predicting flavor stability in products held at room temperature or below. It does, however, offer a general indication of flavor stability of products aged without light at moderate temperatures and limited access to oxygen.

Other forms of induction-period tests can be useful, such as manometric or other devices to measure oxygen uptake from a fat held dry, in an emulsion, in a model system of some type, or in an oxygen bomb. The oxygen uptake measurements can include a catalyst, such as hemin, or metals such as iron and copper. We have found that the oxygen uptake of an emulsified fat in a hemin-catalyzed reaction is useful in predicting the degree of stability conferred on fats, or meats and meat emulsions by antioxidants, especially when these materials are to be stored at freezer or refrigerator temperatures. The oxygen bomb technique

has been reported to be as accurate in predicting shortening shelf life as the AOM, although this technique has not been widely accepted in the fats and oils industry. An oxygen bomb technique was used successfully to evaluate peanut quality by workers at the National Peanut Research Laboratory, and could be applied to the dry and semi-moist, freeze-dried model systems used at MIT, Natick Laboratories, and elsewhere.

Differential scanning calorimetry has been used to predict shortening stability and offers the advantage of being relatively rapid, and the method correlates with other measurements such as conjugated diene values and AOM values.*

We have also aged various lipid-bearing products in an oxygen atmosphere at various temperatures and found that product or fat instability can be quickly demonstrated. For example, sausage that will normally last for 3 mo in frozen storage will reach the same degree of unacceptability in only 1 week under oxygen. Antioxidant performance can also be readily demonstrated by this method. While we have used this accelerated aging technique mainly to study overall flavor to date, the usefulness of the method could be increased greatly by following the fate of key flavor components during the aging process. One example would be by following the dienal and lactone content of fried foods.

Background data for determining key components in given products can be gathered by separating and quantifying the flavor components by GLC and using discriminant analysis to determine which peaks correlate with the flavor evaluation. This approach, used by Powers, Chang, Smouse, Dupuy, and others, in my opinion, offers many opportunities to find which accelerated tests are the most meaningful.

REMARKS BY ALEXANDER E. THOMAS, III

The term "stability" is often thought of as synonymous with oxidative stability. However, oxidative stability is only one factor, albeit an important one, affecting the overall stability of a fat or a fat system. Perhaps, more importantly, it is only one factor affecting the stability of a formulated food product produced from this fat or fat system. After all, few of us consume fats and oils *per se*. Mostly, they are incorporated into the diet as part of another food item.

In the broader sense, any definition of stability must then encompass chemical and physical phenomena that have the potential to produce instability. Oxidative and hydrolytic reactions may be considered important chemical phenomena having the potential

---

*C. K. Cross, *J. Am. Oil Chemists' Soc.*, *47*, 229 (1970).

to produce change or instability in fats. Likewise, crystallization and polymorphic transformation may be considered important physical phenomena with a similar potential to produce change.

Although the extent to which such phenomena affect stability is inherent in the composition of the fat system, the rate at which they affect stability is often dependent upon the presence of other constituents or conditions that act either to retard or accelerate instability; e.g., trace amounts of copper or iron may act as pro-oxidants serving to catalyze oxidation. Acids, bases, and certain enzymes may, in the presence of water, catalyze hydrolysis. Traces of large molecules such as polymers may inhibit or accelerate the rates of crystallization and polymorphic transformation. Finally, processing and environmental conditions such as time and temperature may have a significant impact on stability.

Methodology for predicting the approximate stability of an oil or oil system to oxidative or hydrolytic degradation is today available, as is methodology for other physical and chemical phenomena. Because of the empirical nature of such tests and because of the influence of other trace constituents or conditions, results are approximate at best. They are, however, useful in comparative tests, provided:

1) A sufficient number of different lots of the same formulation are evaluated to eliminate variability due to factors other than composition.
2) The tests are performed by an experienced technician under carefully controlled conditions.

Obviously, solutions to these problems reside in properly designed and executed methodology applied in a good experimental design to test samples.

As mentioned earlier, few of us consume very much fat and oil *per se*. Mostly, they are incorporated into the diet as part of a more complete food item. It logically follows, then, that predictive tests on vegetable oils or other ingredients will have their greatest utility if the conclusions reached can be extrapolated to the complete food. Unfortunately, existing predictive stability tests for fats and oils often bear little relationship to their stability in a more complete food system; e.g., two fats differing in fatty acid composition may exhibit similar or even identical quantitative oxidation and hydrolysis tendencies. Yet, if one of these fats contained appreciable quantities of lauric glycerides and the other did not, a potential for flavor degradation may exist. This becomes evident if the lauric acid-based product is incorporated into a food that contains lipolytic enzymes which may catalyze hydrolyis. Even though the proclivity of these fats to hydrolyze may be quantitatively identical, the qualitative nature of the hydrolysis products is different. In addition, the catalytic agent was introduced external to the fat system. Consequently, flavor changes and their organoleptic

thresholds will be different. Anyone who has tasted a soapy product will recognize that such flavor changes may range from subtle differences to total unacceptability.

Similar examples can be cited for other physical and chemical phenomena having the potential to produce instability, which, although predictable in the oil, cannot predictably be extrapolated to a formulated food without additional information concerning the nature of the total system. The effects of this uncertainty may result in taste, odor, color, appearance, texture of functional changes, ranging from mere subtlety to total unacceptability.

In short, we have come a long way in developing empirical predictive tests for vegetable oil systems. However, such tests are only of limited value when extrapolated to complete foods.

REMARKS BY THOMAS H. SMOUSE

The remarks made here are derived essentially from 3 different studies that have been made recently concerning the flavor of fats and oils and methods evaluated to predict their stability. These 3 studies include (1) the work of the Flavor Nomenclature and Standards Committee of the American Oil Chemists' Society, (2) a paper entitled "Flavor Components of Soybean Oil and Chemical Tests That are Utilized to Evaluate Present Quality and Predict Future Quality" that was delivered to the International Society for Fat Research at their 11th World Congress Meeting in Göteborg, Sweden, and (3) a paper delivered at the 1974 Spring AOCS Meeting in Mexico City entitled "The Frying Properties of a Series of Vegetable Oils and the Effectiveness of Selected Tests in Predicting Their Frying Stability."

As chairman of the AOCS Committee on Flavor Nomenclature and Standards, I have been directly involved in collecting data from as many as 15 laboratories that are evaluating the flavor of fats and oils on a regular basis. To determine the agreement between these laboratories in evaluating the flavor, 3 samples of soybean oil were organoleptically evaluated by participating laboratories. The results from this study show that judges' scores within laboratories were variable as well as the variation between laboratories being significant. The variations observed in subjectively evaluating the flavor of fats and oils have led to more emphasis upon objective methods.

One of these objective methods is an instrumental method for measuring the flavor of fats and oils. Data from various studies using a gas-liquid-chromatographic method to determine the flavor score of several oils show that the method correlates well with actual flavor scores obtained by organoleptic analysis.

Numerous oxidation studies of fats and oils have shown that many chemical classes are formed by hydroperoxide dismutation reactions. These include alcohols, aldehydes, ketones, hydrocarbons, acids, and even cyclic and aromatic compounds.

Several of the chemical tests have been correlated with flavor scores of aged soybean-oil samples. The chemical tests evaluated were the active oxygen method, peroxide value, TBA value, benzidine value, pentane value, and the octanoic acid value. Results from such studies have been fitted into a correlation matrix table to show their linear correlation with the other chemical tests as well as the flavor scores.

Useful tests involve the pentane/iso-octane ratio, free and bound octanoic acid, and hexanal values. Flavor scores did not correlate well with the AOM test or determination of the pentane value. They did correlate fairly well with the TBA test, with the anisidine value, and the octanoic acid value. On the other hand, the pentane value correlated well with peroxide value and the octanoic acid value correlated well with the TBA and anisidine values.

Since most oils have bland flavor after deodorization, soybean oil was oxidized to various oxidation levels and re-deodorized to bland products with high flavor scores. These samples were then aged under normal shelf lighting at room temperature and samples were evaluated at regular intervals. Chemical tests correlated well with the known oxidation level of each oil prior to the second deodorization.

In many applications, the stability of an oil depends upon its oxidation rate at room temperature. However, for a frying fat, oxidation during frying proceeds at a relatively high temperature usually in the area of 180°C. At these temperatures, other factors together with flavor stability become important. To evaluate effectively the utilization of a frying medium, the medium must be evaluated under actual frying conditions. However, the time necessary to perform an actual frying test can be in excess of 200 hours and the evaluation of flavor stability of the fried product can take several weeks. Since such procedures are impossible to perform on each production lot of frying oil, several abbreviated tests have been evaluated and used to predict the frying stability of some of the refined vegetable oils commonly used in United States.

Frying stability is determined according to five criteria: foam resistance, flavor stability, color stability, resistance to hydrolysis, and resistance to polymerization.

REMARKS BY ROBERT G. ACKMAN

Some recent consumer reactions to frozen and convenience processed fishery products ranged from "delightful dish" through "fish dry and chewy" to "fish should have been buried, not frozen." These comments touch on the two major problems: the need to start with good quality raw material, and the need to optimize all subsequent handling and storage steps.

European and Japanese factory trawlers often process and freeze fish a few hours after they are out of the water, ensuring that a top quality raw material is used at the start of the long

road to the consumer. For economic reasons, larger North American operations are shore-based and typically receive fish for processing which has been several days on ice, so that quality deterioration is already well established. In large volume fisheries (salmon and herring), handling by refrigerated sea water (brine chilled from 0°C to -3°C) is being introduced to facilitate handling rather than to improve quality, and will create new problems such as lengthy exposure to excess sodium chloride, enzymatic degradation (belly-burn), etc.

The marketing of fresh fish is not a problem when sanitation practice and temperature control, usually icing or mechanical storage near 0°C are adequate and the fish is disposed of once bacterial spoilage leads to an offensive odor, including trimethylamine odor which is often cited as the classic "fishy" odor. Unfortunately, it is the latter factor which leads to the poor image of fish and fishery products in the eye (or nose) of the contemporary consumer. Why then is there not a complete transition to handling fish in frozen form where at worst a mild cooking odor might be noticed? One reason is tradition; also, there is a lucrative but limited market for high-quality fresh fish. The second reason follows from some bad post-war experience with frozen fish which was very poorly handled leading to low quality products at the consumer level, so that there is some resistance to a complete switchover to a frozen fish industry.

A middle ground is beset with political, regulatory, and technical arguments as to whether frozen fish should be permitted to be sold as "fresh fish" after thawing. Common sense dictates that if the shelf life of fish fillets is 14 days and 7 days are taken up in storage, distribution, storage, and retail sale of a product already half-ruined, then it would probably be better to freeze the fish as soon as possible to stable conditions (discussed below), store and distribute frozen, and thaw at the retail level to suit customer demand. The consumer would, in fact, generally receive a product superior to the fresh fish now marketed.

Two attempts have been made to bypass this "frozen" versus "fresh" problem. In one, "superchilling" is the term used to describe holding the fish muscle in the -1° to -4°C (30° and 25°F) range. Part of the muscle water is converted to ice. This temperature effectively controls bacterial spoilage and improves storage quality in many species, although not in some gadoids such as cusk, hake, and pollock. Advantages of superchilling are that the product can be handled by present freezing and shipping equipment, ice can be dispensed with in favor of insulated containers acceptable to transportation firms, product quality is often superior to conventional "fresh" fish because initial cooling is usually very rapid, and the product is delivered to the retailer in or near a readily salable form. Because enzymatic and other processes affecting quality retention occur during temperature fluctuations, one distinguished fisheries scientist coined the term "damn poor freezing" for superchilling. A

currently used practice involves packing solid carbon dioxide in the top of the container prior to closing it for shipment. Despite the fact that the fish in the top of the box are frozen by this process, they can be sold without question as "fresh" fish.

We are then reduced to considering that freezing to -40°C (-40°F) could be ideal in that most biochemical processes are slowed to an almost infinitely low rate, but that in practice -20° to -25°C (-4° to -13°F) is more likely to be used. A stable temperature is very much to be preferred to a mean temperature based on large cyclical variations. The quality problems in lean fish such as cod were based essentially on thaw-drip and texture toughening. Although taste panels and organoleptic assessment remain the ultimate criteria, the need for chemical or physical tests has long been recognized. Protein extractibility and lipid hydrolysis with free fatty acid formation have been the basis of extensive studies, as have trimethylamine and more recently dimethylamine analyses. In the gadoids, trimethylamine oxide can break down to give dimethylamine and formaldehyde. The latter chemical has unexpectedly emerged as the villain leading to lowering of extractable protein. At the same time there is still apparent evidence for linking free fatty acids from hydrolysed lipids to protein denaturation. Lipid hydrolysis (basically phospholipid) is non-selective, and recent studies have also shown a parallel in the decrease in free polyunsaturated acids (i.e., those with 5 and 6 double bonds) with quality changes, rather than the total fatty acids. Undoubtedly, this decrease is due to oxidation, and either peroxides or the peroxide degradation products (e.g., aldehydes) could be implicated in protein denaturation. Further work is needed in some gadoids to determine if the origin of this oxygen *in situ* could be in trimethylamine oxide, thus extending the relationship between trimethylamine oxide and loss of protein quality.

For several species the potential for protein quality deterioration of fillets on storage can be rapidly evaluated by a Cu^{++} induced development of oxidation combined with the TBA assay.

The last 4 years have seen an effective integration among various aspects of fish quality, chemical indices, and biochemical reactions. However, owing to the differences between species and families of fish, fishing conditions and primary product handling, and product development and handling, a quality panacea remains to be found for frozen fish.

REMARKS BY HAROLD S. OLCOTT

Fats have different modes of behavior during the induction period. Some show essentially no oxygen uptake or other measurable evidence of oxidation while others, with an essentially equivalent period of time to become "rancid," may give evidence for a slow but progressive oxidation in the system.

Long induction periods are induced by antioxidants. Many recent observations suggest that the effective state of the antioxidant is that of a free radical. Studies of EPR (electron paramagnetic resonance) spectra in lipid systems containing added free radical reveal that rapid oxidation does not proceed until the EPR signals for the free radical have essentially disappeared from the sample. This offers a promising technique for predicting stability of fats containing free-radical antioxidants.

Further, evidence is accumulating that all antioxidants may be effective through free-radical intermediates. Hence there is a possibility of developing methods using sensitive EPR measurements to evaluate lipid stability.

DISCUSSION PERIOD

Various topics were discussed after the session was opened to the audience. These brought out several elements of interest and emphasized many factors mentioned in the initial remarks. One was: stability problems in foods involve serious consideration of the composition and concentration of the polar lipids present in the food. These may well be the "flavor limiting lipids" in a food because of the high polyunsaturated fatty acid content in many polar lipids.

Problems in frozen fried chicken relate to breading, skin lipids, and frying fat. Chicken fat, particularly from the skin, cooks out into the frying medium so that the concentration of chicken fat in the frying medium ultimately reaches an equilibrium state. It was noted that the flavor score of fried chicken increases with frying lot number up to some optimum then decreases. This was explained by the fact that frying generates lactones and other flavor enhancing substances up to some point in the frying sequence when oxidation begins to influence the development of flavors and causes a loss of quality.

Oil changes are made in fryers, which do not have a gross and rapid turnover due to volume of food involved, to counteract foaming, hydrolysis, off-flavor, and color. Polymer formation may be a factor in some systems although there is some question regarding the extent to which polymers may influence flavor. It is possible to develop a substantial correlation between the diene and triene content of a frying fat and the formation of polymers.

The rate of formation and the rate of decomposition of peroxides are of utmost importance to the tests which depend upon

measurement of secondary products of oxidation. It is here that components of a food system may exert a strong effect on the nature and concentration of products being measured; these may be unique to the particular food system.

GENERAL OBSERVATIONS AND CONCLUSIONS

There are a number of tests and measurements which are applied to fats and oils and to food systems containing lipids which are useful in assessment of the status of the quality of fat and to some degree may be predictive of the stability characteristics. These stability characteristics relate not only to oxidative stability but also to hydrolytic stability and stability of polymorphic forms of solid fats.

The most frequently encountered problem, and the one with the greatest magnitude, is that of oxidative stability so that most of the methods of concern relate to some assessment of the oxidative status of the fats.

Methods widely used are the AOM or Swift stability test, determination of the peroxide value, and the Schaal or oven stability test. Other tests rely on measurement of functional groups and involve the TBA test for malonaldehyde, other carbonyl determinations, the benzidine or anisidine test, and measurement of formation of diene conjugation. Oxygen uptake tests, as measured manometrically at atmospheric or elevated pressures in a "bomb," are useful. The application of differential scanning calorimetry may also be useful. The measurement of octanoic acid as an indicator of the oxidation history of a fat is gaining as an indicator of the extent of the prior stress to which a fat has been subjected. A growing body of knowledge is found in the application of gas-liquid chromatrography and regression analysis to the various substances capable of being resolved in this manner. This is particularly applicable to the measurement of short chain hydrocarbons such as pentane although it is not limited to these.

Problems relate to the complex nature of many of the foods in which fat stability is important. The stability of the fat in the food may be quite different from the stability of the same fat extracted from the food and then subjected to some test to determine its stability. The extraction process and the completeness of extraction of lipids from a food system also place limitations on assessment of the status of a fat in a food.

Certain elements of the problem of prediction of fat stability stand out. A number of methods exist for measuring stability conditions in a fat or a food system. Some of these are empirical and are useful usually for comparing conditions in one fat or food system with those in another. Thus, certain relative approximations may be made. Some tests are more definitive and measure specific components which are or may be an indicator of the conditions of age and stress placed on the system.

Limiting to all of these are the sensitivity of the various tests. The tests applied may not be able to function at concentrations as low as "threshold values" of the substances they are intended to evaluate. Thus, an organoleptic evaluation may yet be the final criterion by which the status of a fat or food is to be evaluated.

When the many methods now used for evaluating fats are assessed, it becomes apparent that prediction of fat stability depends upon a knowledge of the composition of the fat, of the system in which it resides, of the nature of the stress on the system, and of the mechanisms by which changes occur which are important to evaluation and/or prediction. This knowledge, combined with an increasing sophistication of instrumentation and methodology allowing measurements at lower and lower concentration levels of chemical species, permits the development of data which can be evaluated for the prediction of some features of fat stability. Finally, we must ask the question: do we have the means, the methods, and the information which will permit the prediction of fat stability? With many special recommendations, reservations, and provisos as discussed in the foregoing report, the answer may be a qualified favorable response. However, there is much yet to be learned and it is anticipated that continuing progress will be made only at the expense of much time and dedicated effort on the part of those whose role it is to assure that the consumer, whether civilian or military, will have wholesome, palatable, and nutritious foods available to him through the channels of commerce.

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

A P P E N D I X E S

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

APPENDIX A

Dinner Address

PROBLEMS IN CONSUMER PERCEPTION OF QUALITY

Virgil O. Wodicka

Problems in the consumer perception of quality begin when we consider what we mean by "quality." The relevant definition in Webster's Third New International Dictionary Unabridged is "degree of excellence." It stems from the Latin word qualitas, which is the noun form of the interrogative qualis, meaning "of what kind." We see immediately that the concept of degree of excellence is a derived one in that the basic meaning distinguishes kind rather than level. It is fairly obvious, however, that when we talk about kinds, we are sorting some kind of a population into groups such that the members of a group are more like each other than they are like members of another group. Such a sorting process obviously implies comparisons. When we make these comparisons to sort objects into groups or kinds, it is hard for us to avoid applying value judgments whereby one group or kind becomes better than another. Once we have done this, we have made the transition from "what kind" to "degree of excellence."

In this brief consideration of the fundamental nature of quality, we have really identified two dimensions of quality. One of these is the distinction between kinds, which indeed we tend to talk about as a "qualitative" difference, and the other is the application of value judgment to determine degree of excellence. There are problems with both these dimensions but they are different kinds of problems.

One of the basic problems in determining kinds may be shown by an example. Let us say that we want to classify apples. If we say that one class consists of red apples and the other consists of yellow or green apples, we generally do not have too much difficulty with this distinction. On the other hand, if we want to set up one class which constitutes large apples and another which constitutes small apples, we start running into problems. It immediately becomes obvious that for the purpose of grading apples, we must define the terms "large" and "small." In other words, we need a standard of quality. Physically, this is not too difficult. We may make one or more holes of a certain size in a sheet of rigid material, such as metal, and pass the apples over the sheet. We say that all which fall through are

small and all which do not fall through are large. As we do this, however, we must be aware of the fact that there is really no important difference between an apple that just barely makes it through the hole and one that just barely does not. This boundary value problem remains only a topic for philosophical discussions or essays until we bring in the question of value judgment. Once we have separated large apples from small apples, we discover that the volume of the core does not increase as fast as the volume of the apple. Accordingly, large apples as a class have more edible portion than small apples. In other words, they give us more pounds of fruit per pound of apples. This difference is soon reflected in the price paid by consumers for large apples versus small apples, be that price in money or barter. With an economic difference between the two categories, therefore, the location of the boundary between the categories becomes a much more emotional topic. Any movement of the boundary will lead to a gain for one group and a loss for another, but there is no independent criterion that can be used to determine where the boundary really should be. This fictional problem is close enough to real life to illustrate the basic problem of the standard-setter. More often than not, he finds himself caught between opposing factions with no scientific or absolute way of resolving the conflicts between their interests.

If we define the large apple/small apple problem as the "boundary value" problem, we may then look at what I shall call the "multiple value" problem. Following through our apple illustration, let us assume that red apples are better for eating out-of-hand than green apples, but green apples are better for cooking than red apples. In such case, the degree of excellence of red apples as opposed to green apples would depend on whether the purchaser wants to eat his apples raw or cooked. This obviously varies from purchaser to purchaser. If, for some reason, it is now necessary to assign a single value or grade hierarchy to apple color, it becomes very difficult to determine whether red shall be better than green or green better than red. It is obviously possible to find out which type of purchaser is predominant in the marketplace and, in effect, take a vote, but if the numbers are anything approaching even, this will not be a popular decision.

The next problem we might examine is what we might call the "marginal value" problem. Going back to our apple example let us assume that some vendors polish their apples and some do not. The polished apples obviously give no greater yield than the unpolished apples and they have no difference in flavor or texture that would cause them to be relatively more suitable for raw or cooked consumption. On the other hand, when purchasers are offered a choice, they tend to select the polished applies over the unpolished apples. The difference may even get built into the price, even though the difference is esthetic rather than utilitarian. The informed consumer may object to paying more money for the polished apple because he says it has no more value for him, but he may not have a choice. The dealer from whom he

buys may observe early in the game that polished apples sell better than unpolished apples and, having only limited display space, chooses to buy only polished apples. In such a situation, the consumer who would rather pay less money for the unpolished apple, which meets all his needs, is forced to pay extra for a value he does not desire.

The next problem we might address, we may call the "hidden value" problem. Going back to our apple example, let us assume that a particular group of consumers desires an apple with a high ratio of sugar to acid. This is a property that can be measured in the laboratory, or even with portable field equipment. It may also be measured in a practical way by eating a portion from a sample of the apples. In this case, let us assume that this is a property not included in the official grade standard for the apples and that the apples are normally marketed in plastic or net bags so that the consumer has no practical way of sampling them. For all practical purposes, therefore, this becomes a hidden value. He can associate this property with different varieties through experience and guide his purchasing by selecting on a variety basis. If he encounters an unfamiliar variety or if there is considerable variation within a variety, he must be prepared to gamble.

Next we may look at the "method of measurement" problem. To illustrate this with our apple example, let us assume that there is a group of consumers that desires a special level of sourness in the apple. Whether this is high, low, or in between has no bearing on our problem. Let us further assume that we have evidence showing that sourness correlates only to a very limited degree with either pH or titratable acidity or any known combination of the two. If we then want to communicate the quality of apples in this respect to this group of consumers, we must either describe them with a measurement that is easy to make but communicates limited information or fall back on a sensory method with its difficulties of standardization and precision.

Finally, let us look at what I may call the "imputed value" problem. Going back to our apple example, let us assume that some major public figure, such as an athlete or an astrologer or a literary prize winner or a politician, announces his discovery that yellow apples, but only yellow apples, are a tremendous aphrodisiac. It will take many months to subject this hypothesis to scientific test to determine its validity. It will take years before the production of yellow apples can be increased. Meanwhile, the demand for yellow apples skyrockets, and the price along with it. Millions of people become convinced that this is the food that they have needed all their lives and demand that the government require all labels to be changed forthwith to declare which foods are made with yellow apples. They are imputing a value into yellowness in apples which other less suggestible consumers do not find.

With further study, we could probably identify other problems arising from consumer perception of food quality, but

this assortment of 6 should suffice for now. Let us then pause for a moment to look at the 3 dimensions of quality that really should determine a food's value. These are safety, nutritive value, and sensory appeal. Let us now look at each of these parameters in turn.

Certainly, all 6 of the problems occur in assigning value to safety. The boundary value problem is with us all the time. Safety is inherently a property of level of exposure, so we must always face the problem of how much is too much, and the answers are not always at hand. By way of example, the glycolalkyloids in potatoes are at such a level that potatoes would fail the criteria applied to the safety of food additives. In view of the fact that potatoes are classified as generally recognized as safe, they do not pose a regulatory conflict. On the other hand, a little genetic tinkering could easily turn them into a problem.

Perhaps a better example of the boundary value problem is given by aflatoxin. This is an extremely potent carcinogen that occurs in many foods that depend on low moisture content for their keeping properties. It is difficult to say how much is too much because of the delayed onset of liver cancer and the problems in translating results from experimental animals to man. On the other hand, if we were to insist on levels below the point of detection, we would rule out of the food supply many of our important crops.

The mixed value problem may be illustrated by the situation with milk. Certainly with respect to safety, milk would rate well on a scale of excellence. On the other hand, to those who are intolerant of lactose or allergic to milk, it would not do so well.

The problem of marginal value is also with us in the safety dimension. For example, foods can be contaminated with insects. It is well-known that many insects are disease vectors and can be very dangerous indeed. On the other hand, the particular species of insects that characteristically attack fresh or stored foods are not generally vectors of human disease, and very few of them are directly toxic in their own right. Nevertheless, the consumer unquestionably assigns higher value to a food which is as free as possible from insect contamination than to a food that is known to be infested.

There are many examples of the hidden value problem with respect to safety. A food that is contaminated with environmental pollutants or excess pesticide residues or many types of toxic bacteria or mycotoxins may be difficult or impossible for the consumer to detect. Someone else must protect the consumer in these areas.

The method of measurement problem is all too frequent in the safety dimension. Just to pick one example, the T-2 Toxin, which is known to be produced by certain species of Fusarium on corn, can cause various disorders, some of which are fatal, and very probably occurs in this country when harvest conditions are

unfavorable. The only measurement procedure available now, however, is biological and is neither very sensitive nor very specific.

The imputed value problem is also with us in the safety dimension. For example, there are many foods now being sold on the basis of the fact that they are "natural" or "organic." These words have different meanings to different people, but it has so far been impossible to attach any significance to any of the meanings with respect to safety by any known means of measurement. Nevertheless, for thousands and perhaps millions of people in this country, foods to which these designations are attached have more value because they are safer.

In the same way, all 6 problems can be identified in the dimension of nutritive value. Boundary value problems are painfully apparent in that the recommended dietary allowances change at least slightly every 5 years. Mixed value problems are present in that different people differ in their nutritive requirements, for genetic reasons if no other. Marginal value problems arise in that people will often give higher value to a commodity that gives more of a particular nutrient when they already have enough. The hidden value problem is with us, partly because it is impossible for the ordinary consumer to assess the nutritive value of a particular food, and partly because we do not yet know the identity of all the essential nutrients and therefore, cannot measure their presence. The method of measurement problem is with us in part because many of our existing methods of measuring nutritive value lack both accuracy and precision, and, in part, because with respect to certain nutrients we do not yet know what to measure. The imputed value problem is certainly with us in that many people set considerable value by materials such as bioflavenoids, amygdalin, lecithin, and others, for which the nutritional scientist has not yet been able to identify any effect in spite of many careful investigations.

When we move now to the third dimension of sensory appeal, the profile changes somewhat. Here the method of measurement problem becomes dominant. We must either measure some related physical or chemical property that is easy to measure, or we must make a direct sensory measurement which is relatively slow, cumbersome and costly. The cost and imprecision of the sensory methods and the limited correlation of the physical and chemical methods give us the boundary value problem. On the other hand, when the consumer can make his own assessment on this dimension, he does not have the hidden value problem. This exists only when he is depending upon someone else to make assessment for him. The imputed value problem also is largely absent. The consumer knows whether a product appeals to his senses or not, and does not usually rely on any outside mystique to guide his decisions. The mixed value problem obviously does not apply to an individual consumer, but if we try to build a system of grades based on sensory appeal, it applies very strongly. The marginal value problem applies also in a way that I shall develop later.

Now that we have looked at the catalogue of problems associated with consumer perception of food quality and the fact that they run through the major true parameters of quality, it is time to see how they bear on the problems of the Armed Forces. Consumer perceptions of food quality influence two major aspects of military feeding. One of these is menu selection and the other is specification for procurement. The major one of our six problems that bears on menu selection is that of mixed values. With a large population in the Armed Forces of citizens of widely diverse origins, there is a tremendous gamut of food habits, which, in turn, influence the standard of sensory appeal. Menus must be built on those foods which fewest people dislike.

The Armed Forces probably have the smallest problem on the dimension of nutritive value. The nutritional needs of the military personnel can be met in part by menu selection and in part by specifying nutritive value for those foods that might otherwise be a problem. Accordingly, quality needs along this dimension have long since been met and taken care of.

Of the safety dimension, on the other hand, the Armed Forces have, to a large extent, passed their problem over to the Food and Drug Administration and the U.S. Department of Agriculture (USDA). Specifications seldom single out safety attributes for special attention but rather handle the safety problem by requiring the commodities to comply with the requirements of the Federal Food, Drug, and Cosmetic Act. Other problems, such as those associated with animal disease, are controlled through USDA inspection.

The Food and Drug Administration, on the other hand, has genuine problems associated with the consumer perception of quality associated with the dimension of safety. Consumer quality concerns in the safety area focus first of all on those minor constituents of formulated foods which are delcared on labels by their chemical names, which I may loosely call food additives, and on matters of contamination by insects and rodents. Problems of the intrinsic toxicity of many foods, problems arising from contamination with bacteria and molds, and problems of environmental contaminants do not stimulate nearly as much public attention, even though they are greater hazards. Accordingly, the Food and Drug Administration must continue to do its best to protect without public notice against hazards for which the probability is small while it is being berated for not providing sufficient protection against hazards for which the probability is remote.

Finally, we may move to the problems of the Armed Forces and the regulatory agencies in the area of sensory appeal. Procurement specifications of the Armed Forces are heavily based on United States' standards for grade developed by the USDA to provide common terms of reference for the marketplace. These are based on sensory appeal to the consumer but they are fraught with our six problems.

In order to be practical in facilitating commerce, United States' standards for grades must be based on properties that are

easy and quick to measure. They would otherwise require an Army of inspectors and would be likely to introduce unacceptable delays in shipment. Accordingly, the attributes measured are not necessarily the most important. Nutritive value and safety, for instance, must be otherwise provided for. As a practical matter, flavor cannot enter in a positive way but only in terms of freedom from foreign flavors. Texture is often a factor of grade but usually indirectly in terms of some related attribute. Accordingly, in the important characteristics of flavor and texture, there is something of a hidden value problem. This is largely because of the method of measurement problem.

When a buyer for a retailer finds it necessary to select among a number of lots offered and cannot make his decision based on important properties, he will be guided by unimportant properties. This, of course, is the marginal value problem. United States' standards for grade assign values to uniformity of size and color, whereas marketing men would probably consider these of little importance, and there are at least some panel tests that would confirm this judgment. Perhaps this is one manifestation of the mixed value problem; namely, that these factors would be of much more importance to some consumers than to others.

Certainly, in the activities of the Food and Drug Administration to establish standards of identity and quality, the mixed value problem and the boundary value problem have been much evident. FDA has found it virtually impossible to establish a standard that satisfies all with respect to the relative importance of parameters of quality and the boundary values established. Indeed, there may be some suspicion that if there is not controversy over a standard, the standard is not needed.

The one problem out of the six that is not a serious one in procurement specification for the Armed Forces or in standards for grade or identity or quality is that of imputed value. The technical agencies, both military and civilian, have strenuously tried to avoid imaginary values.

There has been historically and there still is strong pressure among the consumers to have various foods labelled with United States' standards for grade. This has been the case particularly with processed foods. To the extent that these grades are truly reflective of consumer preference, this is understandable pressure. To the extent that these grades do not reflect consumer preference, it is pressure misapplied. In view of the fact that military procurement is also based on these grades, however, whatever discrepancy there is between the grades and true consumer appeal represents inefficient and ineffective procurement.

One problem that currently looms large in the civilian economy but not in the Armed Forces is that of pull dating. There is a widespread belief among consumers that there is a date for each food at which the food loses not only sensory appeal, but also nutritive value, and even safety. Consumer spokesmen want these dates marked on food packages. They refuse to be distracted

by the fact that nutritive value and safety are seldom actually involved and the date at which consumer appeal diminishes markedly is strongly affected by characteristics of a particular lot of food, microbial load, nature of processing, storage temperature and humidity, and other factors. It is likely that pull-date marking on many foods will be required by law before many years are out.

The Armed Forces have probably done more than anybody else on the scientific sensory assessment of food, particularly as affected by storage. You are well aware of the fact that a reliable measurement of the sensory quality of a particular sample of food requires a test by a panel of at least 40 people under rather well-controlled conditions. This immediately makes it clear that we have a method-of-measurement problem. In view of the fact that most foods lose quality gradually and there is not a sharp break in their quality level, we also have a boundary value problem because it becomes necessary to make an arbitrary decision at what point the loss of quality has become unacceptable and the shelf-life is therefore over. Needless to say, the necessity for using a panel of 40 people is an illustration of the mixed value problem. From the standpoint of the consumer, the expectation that a food with little storage is better than a food with longer storage presents a hidden value problem. He is now unable to tell which is which. From the standpoint of the purveyor, this represents an imputed value problem to the extent that there is no measurable difference between products of differing ages, which is frequently the case.

To the federal agencies concerned with food quality, the real problems associated with safety and nutritive value attach to fear of the unknown. The problems we know about are under reasonably good control. The problems we worry about are the ones that haven't surfaced yet. The problems with consumer perception of quality in these areas lie mostly in the fact that the consumers do not know which problems are important and which are not.

In the area of sensory appeal, the situation is totally different. Here every consumer knows the right answer for him. The problem is to get a reasonable consensus of what constitutes quality when there is no reference standard and quality is whatever the consumer says it is. The next problem is to establish practical means of measurement of this quality that will give consistent results at different times, in different places, with different people, and, of course, quickly and cheaply. With all we know today, this appears to be a wish-dream. The only feasible course of action that appears open to us now appears to be to continue the efforts such as those discussed at this meeting to establish primary standards based on sensory measurement that can give the same results at different times in different places with different people and then to calibrate against these quick and cheap measurements of other properties well enough correlated with the sensory properties to be useful indicators.

In summation, we may view problems in consumer perception of quality to be in the areas of definition of quality, measurement of quality, and communication of quality. The spokesmen of the consumer movement have concentrated on the problems of communication, glossing over the problems in what to communicate. We must all realize that the definition of quality will entail much hard work in behavioral science and that the result will be statistical with a wide dispersion. The measurement of quality, to the extent that it considers sensory components, will probably be indirect, with a correlation coefficient annoyingly short of one. The communication of quality, therefore, will always involve a vague and often garbled message. The demand will always exist, however, and efforts to improve in all three respects will continue to challenge our best efforts.

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

APPENDIX B

Attendance List

Miss Judith A. Abbott
Agricultural Research Center
(West), ARS, USDA
Beltsville, Maryland 20705

Dr. Robert G. Ackman
Halifax Laboratory
P. O. Box 429
Halifax, Nova Scotia

Dr. David M. Alabran
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Edward E. Anderson
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Pio Angelini
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. S. David Bailey
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Derek H. Ball
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Michael J. Bayusik
R. T. French Co.
One Mustard Street
Rochester, New York 14609

Mr. Maurice Bazinet
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Emil Becker
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Norman E. Bednarcyk
Nabisco, Inc.
2111 Route 208
Fair Lawn, New Jersey 07410

Dr. Fred W. Billmeyer, Jr.
Rensselaer Polytechnic
Institute
Troy, New York 12181

Mr. Dan Biran
Rutgers University
Cook College, P. O. Box 231
New Brunswick, New Jersey
08903

Mr. Solomon J. Bishov
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Raymond H. Bowers
Swift and Co.
R&D Center
Oak Brook, Illinois 60152

Dr. Maxwell C. Brockmann
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Eugene Brotsky
Merck and Co., Inc.
Building 123
Rahway, New Jersey 07065

Dr. William L. Brown
American Bacteriological and
Chemical Research Corp.
P. O. Box 1557
Gainesville, Florida 32601

Dr. Ari Brynjolfsson
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Robert E. Buck
H. J. Heinz Co.
P. O. Box 57
Pittsburgh, Pennsylvania 15230

Mr. Marvin Byer
Oregon Freeze Dry Foods, Inc.
P. O. Box 1048
Albany, Oregon 97321

Dr. William K. Calhoun
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Robert J. Carroll
Eastern Regional Research
Center, ARS, USDA
600 East Mermaid Lane
Philadelphia, Pennsylvania 19118

Dr. James R. Cavanaugh
Eastern Regional Research
Center, ARS, USDA
600 East Mermaid Lane
Philadelphia, Pennsylvania 19118

Mr. Klemens Ceputis
William Wrigley, Jr., Co.
3535 South Ashland
Chicago, Illinois 60609

Dr. Harold K. Chandler
U.S. Army Medical Research
and Development Command
1000 Independence Avenue
Washington, D.C. 20314

Mr. Peter H. Chen
R. T. French Co.
One Mustard Street
Rochester, New York 14609

Mr. Robert L. Childs, Sr.
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Fergus M. Clydesdale
University of Massachusetts
Amherst, Massachusetts 01002

Mr. Samuel H. Cohen
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Joseph Colaruotolo
Werby Laboratories
88 Broad Street
Boston, Massachusetts 02110

Dr. Morton S. Cole
Archer Daniels Midland Co.
P. O. Box 1470
Decatur, Illinois 62525

Dr. John C. Colmey
ITT Continental Baking Co.
P. O. Box 731
Rye, New York 10580

Dr. Winifred M. Cort
Hoffmann-La Roche, Inc.
340 Kingsland Street
Nutley, New Jersey 07110

Mr. Thomas Crosby
Procter and Gamble Co.
6071 Center Hill Road
Cincinnati, Ohio 45224

Dr. H. Russell Cross
U.S. Department of
Agriculture, ARS
8703 Montpelier Drive
Laurel, Maryland 20811

Dr. Paul S. Dimick
The Pennsylvania State University
116 Borland Laboratory
University Park, Pennsylvania
16802

Mrs. Sandra L. Donatoni
Foremost Foods Co.
6363 Clark Avenue
Dublin, California 94566

Dr. Delbert M. Doty (Retired)
Fats & Proteins Research
Foundation, Inc.
3150 Des Plaines Avenue
Des Plaines, Illinois 60018
(Route 2, Box 132-A
Buchanan, Tennessee 38222)

Dr. Stephen R. Drake
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. LeRoy Dugan, Jr.
Michigan State University
302A Food Science Building
East Lansing, Michigan 48824

Dr. Harold P. Dupuy
Southern Regional Research
Center, USDA
P. O. Box 19687
New Orleans, Louisiana 70179

Miss Paula J. Ebbert
National Academy of Sciences
2101 Constitution Avenue
Washington, D.C. 20418

Mr. Daryl L. Eisenmenger
Swift Processed Meats Co.
Kearny, New Jersey 07032

Lt. Col. Dennis S. Farley
Headquarters
Department of Army
Washington, D.C. 20310

Dr. Frank R. Fisher
National Academy of Sciences
2101 Constitution Avenue
Washington, D.C. 20418

Mr. Walter J. Fitzmaurice
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Thomas E. Florine
U.S. Army Troop Support Agency
Fort Lee, Virginia 23801

Col. James L. Fowler
Letterman Army Institute
of Research
San Francisco, California
94129

Dr. Charles V. Fulger
Kellogg Co.
Porter Street
Battlecreek, Michigan 49016

Miss Esther B. Garber
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Felix J. Germino
Quaker Oats Co.
617 West Main Street
Barrington, Illinois 60010

Mrs. Margaret Y. Gerwin
Stouffer Foods Corp.
5750 Harper Road
Solon, Ohio 44139

Dr. J. Walter Giffee
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Myer Glickstein
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Richard E. Goldstrand
Geo. A. Hormel and Co.
1816 - 4th Street, N.E.
Austin, Minnesota 55912

Capt. John C. Gordon
U.S. Army Medical Department
Veterinary School
Fort Sheridan, Illinois 60037

Mr. Harold Gorfien
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mrs. Germaine G. Gotshall
Air Force Services Office
2800 South 20th Street
Philadelphia, Pennsylvania 19101

Mr. Arthur E. Guest
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Robert E. Gunnerson
Campbell Soup Co.
Campbell Place
Camden, New Jersey 08101

Dr. G. A. Halaby
Stokely-Van Camp, Inc.
6815 East 34th Street
Indianapolis, Indiana 46226

Dr. Kenneth N. Hall
University of Connecticut
Storrs, Connecticut 06268

Mr. Roger G. Hamel
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Norm Harris
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. C. Gene Haugh
Purdue University
Food Sciences Institute
Lafayette, Indiana 47907

Dr. Norman D. Heidelbaugh
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Fred Heiligman
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Richard L. Helmer
U.S. Army Troop Support Agency
Fort Lee, Virginia 23801

Mr. Albert S. Henick
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Gerard J. Henning
c/o Unilever Research
Vlaardingen
P. O. Box 114
Vlaardingen, The Netherlands

Dr. Harold K. Herring
Armour & Company
801 West 22nd Street
Oak Brook, Illinois 60521

Dr. Larry C. Hinnergardt
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Herbert A. Hollender
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Richard S. Hunter
Hunter Associates
Laboratory, Inc.
9529 Lee Highway
Fairfax, Virginia 22030

Dr. Glen A. Jacobson
Campbell Institute for
Food Research
Campbell Place
Camden, New Jersey 08101

Mr. Jerry K. Jarboe
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Karl R. Johnson
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mrs. Susan B. Jones
Eastern Regional Research
Center, ARS, USDA
600 East Mermaid Lane
Philadelphia, Pennsylvania
19118

Dr. Edward S. Josephson
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. John G. Kapsalis
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Ira Katz
International Flavors
& Fragrances
1515 Highway 36
Union Beach, New Jersey 07735

Mr. Morris H. Katz
Pillsbury Co.
311 S.E. 2nd Street
Minneapolis, Minnesota 55414

Mrs. Marjorie L. Kehoe
Navy Food Service Systems Office
Washington Navy Yard
Washington, D.C. 20374

Mr. David A. Kendall
Arthur D. Little, Inc.
15 Acorn Park
Cambridge, Massachusetts 02140

Mr. Kenneth K. Keneaster
Uncle Ben's, Inc.
P. O. Box 1752
Houston, Texas 77001

Sfc. Robert E. Kilburn
Academy of Health Sciences
Fort Sam Houston, Texas 78234

Mr. John J. Killoran
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Frederick J. King
Northeast Utilization
Research Center, F26
Emerson Avenue
Gloucester, Massachusetts 01930

Mrs. Mary V. Klicka
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Alvin A. Klose
Russell Research Center
P. O. Box 5677
Athens, Georgia 30604

Mr. Robert A. Kluter
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Amihud Kramer
University of Maryland
F1122
College Park, Maryland 20742

Dr. Donald H. Kropf
Kansas State University
110 Weber Hall
Manhattan, Kansas 66502

Dr. Glen Lee
Peavey Co. Technical Center
11 Peavey Road
Chaska, Minnesota 55318

Dr. Peter A. Lerke
University of California
1950 - 6th Street
Berkeley, California 94710

Col. Rufus E. Lester, Jr.
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Daryl B. Lund
University of Wisconsin
1605 Linden Drive
Madison, Wisconsin 53706

Dr. Ahmed F. Mabrouk
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Lt. Col. J. A. Martin
Navy Food Service Systems
Office
Washington Navy Yard
Washington, D.C. 20374

Mrs. Wilda H. Martinez
Southern Regional Research Center, USDA
110 Robert E. Lee Boulevard
New Orleans, Louisiana 70124

Dr. Morris G. Mast
The Pennsylvania State University
226 Animal Industries Building
University Park, Pennsylvania 16802

Mr. Yoshito W. Masuoka
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Miss Shirley M. McCarthy
U.S. Army Troop Support Agency
Fort Lee, Virginia 23801

Mrs. Jessie McNutt
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Ferdinand P. Mehrlich
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Charles Merritt, Jr.
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Miss Alice I. Meyer
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Nuri N. Mohsenin
The Pennsylvania State University
University Park, Pennsylvania 16802

Mrs. Mary Alice Moring
Headquarters, U.S. Marine Corps
ATTN: Code LFS-4
Washington, D.C. 20380

Dr. Charles T. Morrow
The Pennsylvania State University
249 Agricultural Engineering Building
University Park, Pennsylvania 16802

Dr. Howard R. Moskowitz
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Wassef W. Nawar
University of Massachusetts
Amherst, Massachusetts 01002

Mrs. Sarah W. Nelson
Headquarters
Department of Army
(DALO-TST)
Washington, D.C. 20310

Mr. E. Nesom
Swift and Co.
115 West Jackson Boulevard
Chicago, Illinois 60604

Mr. Ivor T. Nii
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Ann C. Noble
University of California
Wickson Hall
Davis, California 95616

Dr. Harold S. Olcott
University of California
Institute of Marine Resources
Davis, California 95616

Dr. James K. Palmer
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Alonso S. Pereira
Universidade Federal de Viçosa
Escola Superior de Agricultura
Departamento de Tecnologia de Alimentos
36.570 - Viçosa - Minas Gerais
Brasil

Dr. William L. Porter
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Akiva Pour-El
Archer Daniels Midland Co.
P. O. Box 1470
Decatur, Illinois 62525

Miss Patricia Ann Prell
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Abdul R. Rahman
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. James O. Reagan
University of Georgia
Food Science Building
Athens, Georgia 30602

Mr. Robert E. Ricles
W. J. Schafer Associates, Inc.
14 Lakeside Office Park
Wakefield, Massachusetts 01880

Dr. Frederick M. Robbins
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Franklin P. Rorer
Eastern Regional Research
Center, ARS, USDA
600 East Mermaid Lane
Philadelphia, Pennsylvania 19118

Dr. Daniel Rosenfield
Miles Laboratories, Inc.
1127 Myrtle Street
Elkhart, Indiana 46514

Mr. Glenn R. Salter
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Glenn R. Schafer
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Hilton T. Schlup
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Glenn R. Schmidt
University of Illinois
124 Animal Sciences Laboratory
Urbana, Illinois 61801

Mr. John L. Secrist
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Ronald A. Segars
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Michael R. Sevenants
Procter and Gamble Co.
6071 Center Hill Road
Cincinnati, Ohio 45224

Miss Doris E. Sherman
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Philip Sherman
Queen Elizabeth College
(University of London)
Campden Hill Road
London W87AH, England

Dr. Arthur J. Siedler
University of Illinois
567 Bevier Hall
Urbana, Illinois 61801

Dr. Dale H. Sieling
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Thomas H. Smouse
Anderson Clayton Foods
3333 North Central Expressway
Richardson, Texas 75080

Dr. Myron Solberg
Rutgers University
Cook College, P. O. Box 231
New Brunswick, New Jersey
08903

Mr. Otto J. Stark
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Robert C. Strong
Kroger
1212 State Avenue
Cincinnati, Ohio 45204

Dr. Gene G. Suess
Oscar Mayer and Co.
P. O. Box 1409
Madison, Wisconsin 53701

Mr. Wayne M. Swantak
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Stephen M. Swift
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Alina S. Szczesniak
General Foods Corp.
Technical Center
White Plains, New York 10625

Dr. Roy Teranishi
Western Regional Research
Center, USDA, ARS
800 Buchanan Street
Albany, California 94710

Dr. Bob Terrell
Griffith Laboratories, Inc.
1415 West 37th Street
Chicago, Illinois 60609

Mr. George E. Tesko
RJR Foods, Inc.
Bowman Gray Development Center
Winston-Salem, North Carolina
27102

Dr. Alexander E. Thomas, III
Glidden-Durkee
16651 Sprague Road
Strongsville, Ohio 44136

Mrs. Miriam H. Thomas
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Earl A. Tillinghast, Jr.
Dunkin' Donuts of
America, Inc.
P. O. Box 317
Randolph, Massachusetts 02368

Dr. Lee-Shin Tsai
Western Regional Research
Center, USDA, ARS
Berkeley, California 94710

Mr. Justin M. Tuomy
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Anthony P. Umina
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mrs. Patricia F. Vernon
Hershey Foods Corp.
P. O. Box 54
Hershey, Pennsylvania 17033

Mr. Peter W. Voisey
Engineering Research Service
Agriculture Canada
Ottawa, Ontario K1A 0C6
Canada

Dr. George C. Walker
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Stephen J. Wallner
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Henry C. Warmbier
American Can Co.
333 North Commercial Street
Neenah, Wisconsin 54956

Dr. Aaron E. Wasserman
Eastern Regional Research
Center, ARS, USDA
600 East Mermaid Lane
Philadelphia, Pennsylvania 19118

Miss Day Waterman
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Lt. Col. Jerry L. Welbourn
Department of Defense
OASD(I&L), Pentagon
Room 3B724
Washington, D.C. 20301

Dr. Phillip R. Wells
Best Foods Research Center
CPC International
P. O. Box 1534
Union, New Jersey 07083

Dr. Donald E. Westcott
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mrs. Judith A. Westerling
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mrs. Virginia M. White
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Mr. Fred G. Whitson
Keebler Co.
One Hollow Tree Lane
Elmhurst, Illinois 60126

Dr. Eugen Wierbicki
U.S. Army Natick Laboratories
Natick, Massachusetts 01760

Dr. Harold L. Wilcke
Ralston Purina Co.
Checkerboard Square
St. Louis, Missouri 63188

Dr. Robert L. Winslow
Safeway Stores, Inc.
4th and Jackson Streets
Oakland, California 94660

Dr. Virgil O. Wodicka (Retired)
Food and Drug Administration
Washington, D.C. 20204
(1307 Norman Place
Fullerton, California
92631)

Miss Kathleen Wolfe
Searle Biochemics
2634 South Clearbrook Drive
Arlington Heights, Illinois
60005

Mr. John N. Yeatman (Retired)
Food and Drug Administration
200 C Street, S.W.
Washington, D.C. 20204
(11106 Cherry Hill Road
Adelphi, Maryland 20783)

Mr. William E. Young
William E. Young and Co.
Jumping Brook and Essex Roads
Neptune, New Jersey 07753

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.

Objective Methods for Food Evaluation: Proceedings of a Symposium
http://www.nap.edu/catalog.php?record_id=20027

Copyright © National Academy of Sciences. All rights reserved.