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The Radiochemistry of Tellurium

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Subcommittee on Radiochemistry
National Academy of Sciences — National Research Council

FOREWORD

The Subcommittee on Radiochemistry is one of a number of subcommittees working under the Committee on Nuclear Science within the National Academy of Sciences - National Research Council. Its members represent government, industrial, and university laboratories in the areas of nuclear chemistry and analytical chemistry.

The Subcommittee has concerned itself with those areas of nuclear science which involve the chemist, such as the collection and distribution of radiochemical procedures, the establishment of specifications for radiochemically pure reagents, availability of cyclotron time for service irradiations, the place of radiochemistry in the undergraduate college program, etc.

This series of monographs has grown out of the need for up-to-date compilations of radiochemical information and procedures. The Subcommittee has endeavored to present a series which will be of maximum use to the working scientist and which contains the latest available information. Each monograph collects in one volume the pertinent information required for radiochemical work with an individual element or a group of closely related elements.

An expert in the radiochemistry of the particular element has written the monograph, following a standard format developed by the Subcommittee. The Atomic Energy Commission has sponsored the printing of the series.

The Subcommittee is confident these publications will be useful not only to the radiochemist but also to the research worker in other fields such as physics, biochemistry or medicine who wishes to use radiochemical techniques to solve a specific problem.

W. Wayne Meinke, Chairman
Subcommittee on Radiochemistry

INTRODUCTION

This volume which deals with the radiochemistry of tellurium is one of a series of monographs on radiochemistry of the elements. There is included a review of the nuclear and chemical features of particular interest to the radiochemist, a discussion of problems of dissolution of a sample and counting techniques, and finally, a collection of radiochemical procedures for the element as found in the literature.

The series of monographs will cover all elements for which radiochemical procedures are pertinent. Plans include revision of the monograph periodically as new techniques and procedures warrant. The reader is therefore encouraged to call to the attention of the author any published or unpublished material on the radiochemistry of tellurium which might be included in a revised version of the monograph.

The Radiochemistry of Tellurium

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I. GENERAL REFERENCES ON THE INORGANIC AND ANALYTICAL CHEMISTRY OF TELLURIUM

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II. RADIOACTIVE NUCLIDES OF TELLURIUM

The radioactive nuclides of tellurium that are of interest in the radiochemistry of tellurium are given in Table I. This table has been compiled from information appearing in reports by Strominger, et al.,⁽¹⁾ and by Hughes and Harvey.⁽²⁾

*Operated for U. S. Atomic Energy Commission by Union Carbide Corporation.

Table I. The radioactive Nuclides of Tellurium

| Radio-nuclide | Half-life | Mode of Decay | Energy of Radiation, Mev | Produced by |
|--------------------|-----------|----------------------|---|---|
| Te ¹¹⁸ | 6.0 d | EC | | Sb-d-5n |
| Te ¹¹⁹ | 4.5 d | EC | γ : 1.6 | Sb-d-4n |
| Te ^{121m} | 154 d | IT | γ : 0.082, 0.213 | Te-n- γ , Sn- α -n, Sb-d-2n, Sb-p-n |
| Te ¹²¹ | 17 d | EC | γ : 0.506, 0.573 | d Te ^{121m} , d I ¹²¹ , Sb-d-2n, Sb-p-n |
| Te ^{123m} | 104 d | IT | γ : 0.0885, 0.159 | Te-n- γ , Sb-d-2n |
| Te ^{125m} | 58 d | IT | γ : 0.110, 0.0353 | Te-n- γ , d Sb ¹²⁵ |
| Te ^{127m} | 115 d | IT | γ : 0.0885 | Te-n- γ , Te-d-p, I-n-p, Uranium fission, d Sb ¹²⁷ |
| Te ¹²⁷ | 9.3 h | β^- (only) | β^- : 0.7 | Te-n- γ , Te-d-p, Te-n-2n, I-n-p, Uranium fission, d Te ^{127m} , d Sb ¹²⁷ |
| Te ^{129m} | 33.5 d | IT | γ : 0.106 | Te-n- γ , Te-d-p, Te-n-2n, Te- γ -n, Uranium fission |
| Te ¹²⁹ | 72 m | β^- , γ | β^- : 1.8 γ : 0.3, 0.8 | Te-n- γ , Te-d-p, Te- γ -n, Te-n-2n, Uranium fission, Thorium fission, d Te ^{129m} , d Sb ¹²⁹ |
| Te ^{131m} | 30 h | IT | γ : 0.177 | Te-n- γ , Te-d-p, Uranium fission, d Sb ¹³¹ |
| Te ¹³¹ | 24.8 m | β^- , γ | β^- : 2.0, 1.4 | Te-d-p, Te-n- γ , Uranium fission, d Te ^{131m} , d Sb ¹³¹ |
| Te ¹³² | 77.7 h | β^- , γ | β^- : 0.22 | Te- α -2p, Thorium fission, d Sb ¹³² |
| Te ^{133m} | 63 m | IT | γ : 0.4 | Uranium fission, d Sb ¹³³ |
| Te ¹³³ | 2 m | β^- , γ | β^- : 2.4, 1.7 γ : 0.6, 1.0 | d Te ^{133m} |
| Te ¹³⁴ | 44 m | β^- | | Thorium fission |

III. THE CHEMISTRY OF TELLURIUM AND ITS APPLICATION TO THE RADIOCHEMISTRY OF THE TELLURIUM RADIONUCLIDES

Radiochemistry is probably best described as being an analysis technique used primarily either (1) to assist in obtaining a pure radionuclide in some form so that an absolute measurement of its radioactivity, radiation energies and half-life can be made, or (2) to determine the amount of a particular radioelement in a radionuclide mixture, or (3) to complete a radioactivation analysis being used to determine the concentration of a specific stable element in a particular sample material. In order to be

an aid in accomplishing any one of the above interests, radiochemistry usually considers the isolation of the desired radionuclide by either carrier or carrier-free separation methods.

Generally, carrier methods are used most frequently in radiochemistry. They involve the addition of a small amount of inactive stable element to a solution of the irradiated material to serve as a carrier of the radionuclide of that element through the separation method. In carrier-free separations, i.e., radiochemical techniques used mostly for absolute radioactivity measurements, it is required that the radioelement be isolated in a manner capable of giving either no amount or a minimal amount of stable element in the final form to be used in the radioactivity measurements.

In most instances, analytical radiochemistry is dependent upon more conventional ideas in analytical chemistry involving separations by such methods as precipitation, solvent extraction, chromatography, volatilization, and/or electrolysis and the subsequent presentation of the isolated radioelement in a form suitable for a measurement of the radioelement's radioactivity. One major difference exists between carrier radiochemistry and more conventional techniques in that it is not always necessary to recover completely the added amount of carrier element, since a radiochemical analysis is designed to assure that the atoms of a radioactive element achieve an isotopic state with the atoms of the inactive element, and any loss of the radioactive species is proportional to the "loss" of carrier during the separation process.

Colorimetric, polarographic and similar analysis techniques are seldom used in radiochemistry, because they do not separate the desired radionuclide from contaminants (either radioactive or stable) in the mixture being analyzed. However, some of the developments used in these analysis techniques may be useful in radiochemistry.

The information that follows generally describes the chemical behavior of tellurium and its compounds. Many of these reactions have been used or

can be used in devising radiochemical analysis methods for the tellurium radionuclides. More detailed information on the inorganic and analytical chemistry of tellurium can be obtained either from the references cited in this section or from the general references given in Section I of this monograph.

A. The General Chemistry of Tellurium

Tellurium is less widely distributed than selenium; however, it is more often segregated in specific mineral deposits. Its chief minerals are hessite, Ag_2Te , altaite, PbTe , coloradoite, HgTe , and silver-gold tellurides, such as sylvanite, AgAuTe_4 . Tellurium also is found in its native state and in combination with oxygen, e.g. tellurium ocher, TeO_2 . It also is found mixed with sulfur and selenium. An important ore of tellurium is nagyagite, which is a mixture of lead, gold, copper, silver, and antimony sulfides and tellurides.

Tellurium is most often recovered from the anode slimes of copper refining and from the mineral, nagyagite. These materials are usually decomposed by boiling with a mixture of concentrated hydrochloric and nitric acids. Following an evaporation to dryness, the residue is dissolved in hydrochloric acid and the tellurium precipitated by means of sulfur dioxide. If selenium and tellurium are combined in this mixture, they can be separated by boiling the mixture with sulfuric acid. In this process, tellurium is precipitated as TeO_2 and the selenium, as selenite, is transformed to selenious acid which remains in solution.

1. Metallic Tellurium

Tellurium metal is isomorphous with grey crystalline selenium. It is silver white and has a metallic luster. It is very brittle and can be powdered easily. When tellurium is deposited from solution by the reduction of tellurous acid with sulfurous acid, it is obtained as a voluminous brown powder, or "amorphous" tellurium.

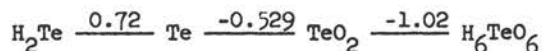
Tellurium metal will burn when heated in air, and it will combine energetically with the halogens and many metals. However, unlike selenium,

it will not combine directly with hydrogen. It will not dissolve in non-oxidizing acids, but it is soluble in concentrated sulfuric acid, nitric acid and caustic alkalis.

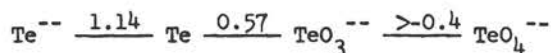
2. The Compounds of Tellurium

Tellurium, like selenium, can form compounds having oxidation states of -2, +4, and +6. A +2 oxide, TeO, exists and will form TeCl₂ in cold hydrochloric acid, but when heated it decomposes into free tellurium and a +4 complex chloride. Latimer⁽³⁾ reports that the following oxidation-reduction potentials exist for tellurium:

a) In Acid Solution



b) In Base Solution



Tellurium (like selenium and sulfur) reacts with other elements in a manner similar to oxygen. However, it will not combine directly with selenium or sulfur but will form mixed crystals with them. The oxides, the chlorides and the fluorides are the most stable compounds of tellurium. The acids and salts derived from the dioxides are also stable.

Tellurium (like selenium) is electronegatively bivalent towards hydrogen and the metals. In its electropositive behavior, tellurium exhibits a valence of +4 in its compound formation with most elements. An exception exists in its combination with fluorine; here a valence state of +6 exists. In general, the compounds of tellurium are analogous to those of sulfur and selenium; the principal difference is that they have a lower stability than the sulfur and selenium compounds.

The most important compounds formed by selenium include oxides, oxyacids, oxyhalides, halide compounds, hydrogen compounds and alkyls. Table II lists the more important of these compounds and gives information about their solubilities.

Table II. Solubility of Tellurium Compounds

| <u>Compound</u> | <u>Formula</u> | <u>Water Solubility</u> | <u>Solubility in Other Solvents</u> |
|------------------|--------------------------------|---|--|
| <u>Acids</u> | $H_6TeO_6 \cdot 4H_2O$ | Soluble in cold water | Soluble in alkalis and dilute acids; slightly soluble in strong acids, insoluble in alcohol. |
| | H_6TeO_6 | Soluble in cold and hot water | Soluble in alkalis and acids; insoluble in alcohol. |
| | $(H_2TeO_4)_x$ | Slightly soluble in cold water; soluble in hot water. | Soluble in alcohol; slightly soluble in KOH. |
| | $TeBr_4 \cdot HBr \cdot 5H_2O$ | Decomposes | |
| | H_2TeO_3 | Slightly soluble in cold water; decomposes in hot water | Soluble in NaOH and acids; slightly soluble in NH_4OH ; insoluble in alcohol. |
| <u>Bromides</u> | $TeBr_2$ | Decomposes | Decomposes in NaOH; slightly soluble in mineral acids and tartaric acid. |
| | $TeBr_4$ | Decomposes | Soluble in NaOH; mineral acids and tartaric acids. |
| <u>Chlorides</u> | $TeCl_2$ | Decomposes | Decomposes in NaOH; slightly soluble in mineral acids and tartaric acid. |
| | $TeCl_4$ | Decomposes slightly | Soluble in HCl, benzene, alcohol, chloroform, and CCl_4 ; insoluble in CS_2 . |

| | | | |
|-----------------------|--|---|---|
| <u>Fluorides</u> | TeF_4 | Decomposes | |
| | TeF_6 | Decomposes | Decomposes in acids and alkalis. |
| <u>Iodides</u> | TeI_2 | Insoluble | |
| | TeI_4 | Slightly soluble in cold water; decomposes in hot | Soluble in HI, alkalis, and NH_4OH . |
| <u>Oxides</u> | TeO | Insoluble | Soluble in HCl, dilute acids, H_2SO_4 and NaOH. |
| | TeO_2 | Insoluble | Soluble in HCl, HNO_3 and alkalis; insoluble in NH_4OH . |
| | TeO_3 | Insoluble | Decomposes in conc. HCl; soluble in hot KOH; insoluble in acids and alcohol. |
| | TeSO_3 | Decomposes | Soluble in H_2SO_4 . |
| <u>Sulfides</u> | TeS_2 | Insoluble | Soluble in alkaline sulfides; insoluble in acids. |
| <u>Metal-Organics</u> | $(\text{C}_2\text{H}_5)_2\text{Te}$ | Slightly soluble in cold water | |
| | $\text{C}_2\text{H}_6\text{Br}_2\text{Te}$ | | Soluble in alcohol and ether. |
| | $\text{C}_2\text{H}_6\text{Cl}_2\text{Te}$ | Soluble | Soluble in alcohol and ether. |
| | $\text{C}_2\text{H}_6\text{I}_2\text{Te}$ | Insoluble in cold water; slightly soluble in hot water. | Soluble in chloroform and benzene |
| | CH_2Te_2 | Insoluble | Insoluble |

a) The Oxide, Oxyacid and Oxyhalide Compounds of Tellurium

The most stable oxide of tellurium, tellurium dioxide, TeO_2 , is formed either by the combustion of tellurium or by oxidizing tellurium with cold concentrated nitric acid. The crystal structure of TeO_2 resembles that of SnO_2 , PbO_2 and MgF_2 . It is usually colorless and will turn yellow when heated. It melts and vaporizes at an incipient red heat and will solidify from the melt in rhombic needles. It is only sparingly soluble in water but will dissolve in concentrated strong acids and concentrated alkali hydroxides. In its reaction with water, it forms tellurous acid, H_2TeO_3 . Tellurous acid cannot be obtained in the pure state because it has a tendency to form high-molecular complexes as it loses water. If the water is driven off at high temperatures, TeO_2 will be formed. The corresponding salts of tellurous acid are the tellurites, which, in their simplest form, M_2TeO_3 , resemble sulfites and normal selenites. Colorless, water-soluble alkali tellurite salts are formed if TeO_2 is fused with alkali hydroxides, or carbonates, or if it is put into solution with caustic alkalis:

H_2TeO_3 (like selenious acid, H_2SeO_3) can be oxidized with strong oxidizing agents like chlorine or bromine to produce telluric acid, H_6TeO_6 . The anhydrides of telluric acid correspond to the sulfuric acid anhydrides. The water content of telluric acid differs from sulfuric acid and the ordinary telluric acid, orthotelluric acid, has the formula $\text{H}_6\text{TeO}_6 \cdot 4\text{H}_2\text{O}$. The salts of telluric acid are the tellurates.

Telluric acid is a very weak acid, and its solubility in water is quite high. As a rule, only a portion of the hydrogen atoms of telluric acid can be replaced by metals to form tellurates; however, some metal tellurates, such as As_6TeO_6 and Hg_3TeO_6 , in which all of the hydrogen atoms are replaced, are known. Other acid anhydride molecules can be linked to the oxygen atoms of telluric acid to form heteropolyacids like $\text{H}_6[\text{Te}(\text{O}\cdot\text{MoO}_3)_6]$ and $\text{H}_6[\text{Te}(\text{O}\cdot\text{WO}_3)_6]$.

Orthotelluric acid, as it is heated, loses water and if the temperature exceeds 300° , yellow tellurium trioxide, TeO_3 , will be formed. TeO_3 is

partially soluble in water and when heated at red heat, it loses oxygen and is transformed into TeO_2 . Allotelluric acid is produced when orthotelluric acid is heated to its melting point in a sealed tube. It is formed as a colorless syrup mass that is miscible with water. Unlike orthotelluric acid, its water solutions are distinctly acid in reaction. Upon standing, a solution of allotelluric acid slowly reverts to orthotelluric acid.

Tellurium does not form oxyhalide compounds as do sulfur and selenium.

b) Halogen Compounds of Tellurium

Tellurium forms bivalent and quadrivalent compounds with bromine and chlorine. It forms both quadrivalent and hexavalent compounds with fluorine and a tetravalent compound with iodine. Monohalide compounds of tellurium do not exist.

Tellurium dichloride, TeCl_2 , can be prepared by a direct combination of the elements or by the reaction between TeCl_4 and elemental tellurium. Tellurium dibromide, TeBr_2 , is formed by heating TeBr_4 . The tetrahalides, TeCl_4 , TeBr_4 , and TeI_4 are formed by a direct combination of the elements. Tellurium tetrafluoride, TeF_4 , is prepared by heating tellurium hexafluoride, TeF_6 , at 180° in the presence of elemental tellurium. All of the tellurium tetrahalides, except TeCl_4 , are thermally unstable. TeBr_4 will dissociate above its boiling point (414°) into TeBr_2 and Br_2 ; TeI_4 when heated above 100° will decompose into the elements; TeF_4 will disproportionate above 190° to form elemental tellurium and TeF_6 . All of the tellurium tetrahalides react with water to form TeO_2 (or the corresponding acid) and the halide acid.

Tellurium hexafluoride, TeF_6 , produced in the disproportionation of tellurium tetrafluoride, TeF_4 , is an active hexafluoride compound (more active than the corresponding sulfur and selenium hexafluorides). At room temperature, it rapidly decomposes in water.

Tellurium tetrachloride, TeCl_4 , tellurium tetrabromide, TeBr_4 , and tellurium tetraiodide, TeI_4 , can form complex halide acids with hydrogen halide compounds. For tellurium, the complex acids $\text{H}(\text{TeCl}_5)$, $\text{H}(\text{TeBr}_5)$ and

$H(TeI_5)$ are known. Hexabromotellurates, hexachlorotellurates and hexaiodo-
tellurates have been formed by the reaction of alkali halide salts upon
these complex acids. The solubilities of these salts resemble the solu-
bilities of the tellurates.

c) Hydrogen Compounds of Tellurium

Hydrogen telluride, H_2Te , corresponds in composition to hydrogen sul-
fide and is best prepared either by decomposing metal tellurides, like
 Al_2Te_3 , with water or acids or by the electrolytic liberation of hydrogen
at a tellurium electrode at low temperatures.

H_2Te is a colorless gas that is less stable than hydrogen sulfide. It
decomposes very easily and can be readily attacked by even weak oxidants.
At ordinary temperatures, it will react with atmospheric oxygen to deposit
elemental tellurium, and it will burn in air with a bluish flame, forming
 TeO_2 . H_2Te is very soluble in water; the solutions are very unstable and
elemental tellurium is rapidly deposited on the walls of the containing
vessel. The acid strength of hydrogen telluride is comparable to the acidity
of phosphoric acid.

The alkali metal tellurides are soluble in water; the solutions are
colorless when pure, but when exposed to air, oxidation causes the solution
to turn red and polytellurides to form. The tellurides of the heavy metals
are dark in color; some of them, like Al_2Te_3 , are readily decomposed in
water while others are decomposed only by acids.

d) The Sulfur Compounds

Tellurium can combine with sulfur to form tellurium disulfide, TeS_2 .
 TeS_2 is a red-black amorphous powder that is insoluble in water and acids
but very soluble in ammonium sulfide. The interaction of tellurium with
sulfur also forms tellurium sulfur oxide, $TeSO_3$, an amorphous red-colored
solid which decomposes in water but is very soluble in H_2SO_4 .

e) Alkyl Compounds of Tellurium

The distillation of neutral or acid alkali tellurides with potassium
alkyl sulfates produce alkyl tellurides, TeR_2 (where R = the alkyl group)

and alkyl telluromercaptans, TeRH . The alkyl tellurides (like the alkyl selenides) are volatile liquids with pungent and repulsive odors. They readily combine with, or add on, halogens or oxygen to form such compounds as $(\text{C}_2\text{H}_5)_2\text{TeCl}_2$ or $(\text{C}_2\text{H}_5)_2\text{TeO}$. In addition, they can add on alkyl iodides to form such compounds as alkyl telluronium, $[(\text{C}_2\text{H}_5)_3\text{Te}]\text{I}$. These compounds are strong bases and, when heated in the presence of moist silver oxide, will form hydroxide compounds.

B. The Analytical Chemistry of Tellurium

As it has already been pointed out elsewhere in this monograph, the use of a known amount of inactive tellurium carrier in a separation method almost always makes it practical to obtain the tellurium carrier in a weighable form in the final stage of the separation procedure used. If this is done, the radionuclide can be concentrated into a small mass for the radioactivity measurements and any loss of the carrier during the analysis can be accounted for.

Tellurium (like selenium) can be qualitatively detected by reducing its salts contained in strong acid solutions to elemental tellurium with such reagents as sulfur dioxide and hydrazine hydrochloride,⁽⁴⁾ hydrazine hydrachloride,⁽⁵⁻⁸⁾ hydrazine,⁽⁹⁻¹¹⁾ hypophosphite,⁽¹²⁾ potassium iodide and sulfur dioxide,⁽¹³⁾ and vandyI sulfate.⁽¹⁴⁾ Many other reducing agents, such as titanous chloride, aluminum, and phosphorus and hypophosphorus acids, can be used to reduce tellurides (and selenides) to the metal in an acid solution.⁽¹⁵⁾ For example, Challis⁽¹⁶⁾ has used hypophosphorus acid to determine tellurium (and selenium) in copper. Evans⁽¹⁷⁾ has used hypophosphate as a reductant in similar analyses.

In addition to its gravimetric determination as the metal, tellurium can be precipitated as tellurium dioxide, TeO_2 , with either pyridine^(18,19) or hexamine.^(12,20) Meyer⁽⁹⁾ and others^(21,22) report that a precipitate of lead tellurate will be formed by the action of lead nitrate on a solution of sodium tellurate. Bersin⁽²³⁾ has shown that a hexamminochromium(III)

tellurate precipitate can be produced by the action of an amine salt of chromium upon an alkali solution of tellurium. Duval,⁽²²⁾ however, reports that none of the methods used in drying these precipitable forms are good for the gravimetric determination of tellurium. He points out that elemental tellurium is much more rapidly oxidized than selenium and that it must be dried at low temperatures when in the reduced state. The vanadyl sulfate method⁽¹⁴⁾ is probably the best method for precipitating elemental tellurium. If TeO_2 is to be the final form, Duval⁽²²⁾ considers that its precipitation by hexamine^(12,20) is to be preferred. The lead tellurate method^(9,21,22) is also considered as a usable method. As far as the hexaminochromium(III) tellurate method⁽²³⁾ is concerned, Duval⁽²²⁾ suggests that this method be rejected, because at very low temperatures ($50^\circ\text{-}60^\circ\text{C}$) ammonia is given off and at temperatures above 300°C , a mixture of Cr_2O_3 plus TeO_2 is formed. The choice of either the elemental tellurium or the lead tellurate or tellurium dioxide compounds to obtain the tellurium carrier and the tellurium radionuclide(s) in a form suitable for a radioactivity measurement, after they have been isolated from a stable and/or radioactive nuclide mixture, would appear to be practical.

The above remarks on the gravimetric determination of tellurium should not be regarded as a prerequisite that it is always necessary to radiochemically separate the desired tellurium radionuclide in a precipitable form before the radioactivity measurements. Sometimes it would be sufficient to accept and use, for example, one of the phases obtained in solvent extraction, or an aliquot from the eluate obtained from an ion-exchange separation column, or a portion of a paper chromatogram, in the radioactivity measurements. Thus, the information that follows generally reports on current ideas used in isolating and determining nonradioactive tellurium.

1. Separation by Precipitation

Tellurium is usually separated from most elements by use of various reducing agents in acid solutions. Hillebrand, et al.,⁽²⁴⁾ report that

tellurium (as well as selenium) can be initially separated from most elements by reduction with sulfur dioxide in 3.7 to 4.8 N hydrochloric acid solution. Gold, palladium and small amounts of antimony, bismuth, copper, and other elements are also reduced under the same conditions and by other reducing agents. Gold can be separated by filtration after the mixed metal precipitate has been digested for some time in a dilute nitric acid solution. Quadrivalent tellurium (and selenium) can be separated from the other elements in the filtrate by saturating the acid solution with hydrogen sulfide.⁽²⁴⁾ The tellurium can then be separated from selenium by precipitating the selenium with sulfurous acid or hydroxylamine hydrochloride.⁽²⁵⁾

In their studies, Seath and Beamish⁽²⁶⁾ and Noakes⁽²⁷⁾ showed that nitrates, Cu^{+2} and Au^{+3} interfered in the reduction of tellurium to elemental tellurium. The nitrates were removed by evaporation with HCl and NaCl; Cu^{+2} and Au^{+3} were separated with hydroquinone before the reduction.^(28,29) Treadwell and Hall⁽⁸⁾ also report on the separation of tellurium from the metals of Groups II, III, IV, and V.

Tellurium (and selenium) can be absorbed on a ferric hydroxide precipitate.⁽³⁰⁾ At least 0.1-0.2 gram of Fe^{+3} must be present and concentrated ammonium hydroxide added in excess to effect this separation of tellurium from other elements. Schoeller⁽³¹⁾ also has used ammonium hydroxide and ferric nitrate to separate tellurium (and selenium) from ores and metals. The ammonium hydroxide precipitation eliminated interferences from copper and nitrates. Tellurium and selenium are separated from iron by a reduction of an acid solution with stannous chloride. Tellurium can be separated from selenium by brominating a hydrochloric acid solution of the metals and precipitating selenium with sulfur dioxide. Plotnikov⁽³²⁾ gives information that Te^{+6} can be quantitatively carried on $\text{Fe}(\text{OH})_3$ in a strong ammonia solution and that it can be effectively separated from Se^{+6} in this manner since Se^{+6} is not co-precipitated with $\text{Fe}(\text{OH})_3$ under these conditions.

Keller⁽³³⁾ reports that tellurium can be separated from selenium by reducing a mixture of tellurous and selenious acids in strong hydrochloric acid solution with sulfur dioxide gas. Only elemental selenium will precipitate under these conditions. Bode⁽³⁴⁾ reports that tellurium can be separated from selenium by precipitating it as Te^{+6} from a 5 N HCl solution with tetraphenylarsonium chloride. Bromide, iodide, fluoride and nitrate ions and Mo^{+6} and W^{+6} will interfere. Lenher and Kao⁽⁴⁾ have used a sulfur dioxide saturated hydrochloric acid solution to separate selenium from tellurium. After filtration, the tellurium was recovered from the filtrate as elemental tellurium following a hydrazine hydrochloride precipitation.

2. Separation by Volatility

Tellurium (and selenium) can be separated as Te^{+4} (and Se^{+4}) from metals whose chlorides are nonvolatile by passing chlorine gas into a hot hydrochloric acid solution.⁽³⁵⁾ Lenher and Smith⁽³⁶⁾ report that tellurium is not volatilized from a sulfuric acid solution when hydrochloric acid gas is passed into the solution. A similar reaction occurs if carbon monoxide gas is passed into a hydrobromic-phosphoric acid solution.⁽³⁷⁾ Se^{+4} and Se^{+6} are volatile and can be separated quantitatively from tellurium in either of these systems.

TeOCl_2 and TeOBr_2 (as well as SeOCl_2 and SeOBr_2) are volatile from 6 N HCl⁽³⁸⁾ or 6 N HBr⁽³⁹⁻⁴¹⁾ solutions at temperatures above 100°C. Arsenic, antimony, tin, and germanium are also volatile under these conditions.

3. Separations by Electrolysis

Only a few applications of electroanalysis techniques exist for the determination of tellurium. Te^{+4} , but not Te^{+6} , is reduced to the metallic state by electrolyzing acidic solutions.^(42,43) Haissinsky⁽⁴⁴⁾ has shown that normal hydrochloric or hydrobromic acid solutions can be used in the electrolysis of 10^{-3} N solutions of Te^{+4} . A smooth well-adhering macro-deposit of tellurium on the platinum cathode was obtained in this work. Mazumdar⁽⁴⁵⁾ recently used a similar electrolysis technique to

obtain a carrier-free tellurium-129m (34d) tracer. Te^{+4} ions in concentrations ranging from 10^{-3} to 10^{-11} N were deposited upon a rotating cathode of gold at a current density of 20 mA/sq. cm. The Te^{129m} tracer was removed from the gold with a HBr solution. Lee and Cook⁽⁴⁶⁾ have removed Te radioactivities from a fission product solution by electrodepositing them upon a platinum cathode at a voltage of 0.15 volt.

4. Separations by Solvent Extraction

Morrison and Freiser⁽⁴⁷⁾ have recently reviewed the applications of solvent extraction involving ion association and chelate complex systems to the determination of most of the elements. Some of these systems are applicable for use as separation processes in the radiochemistry of the tellurium radionuclides.

a) Ion Association Systems

Bock and Hermann⁽⁴⁸⁾ have shown that only 23% of tellurium as Te^{+4} can be extracted from a 20 M HF solution by ethyl ether. Nb^{+5} , Ta^{+5} , and Re^{+6} extract about 50%; Se^{+4} , Sn^{+2} , Sn^{+4} , As^{+3} , As^{+5} , Ge^{+4} , P^{+5} , V^{+3} , V^{+5} , Mo^{+6} and Sb^{+3} are extracted in lesser amounts. The extraction of Te^{+4} (as well as the other ions) can be increased if higher concentrations of HF are used. Tellurium, as Te^{+6} , and Se^{+6} , Fe^{+3} , Ga^{+3} , Sb^{+5} and As^{+3} can be extracted by diisopropyl ketone from a mineral acid-hydrofluoric acid (6 M HCl-0.4 M HF) aqueous system.⁽⁴⁹⁾ Te^{+4} , Se^{+4} , Sb^{+3} and As^{+5} are only partially extracted under these conditions. In this same study, it was shown that Te^{+6} (and Se^{+4} and Ta^{+5}) could be extracted by diisopropylketone from a 6 M H_2SO_4 -0.4 M HF system.

Noyes, et al.,⁽⁵⁰⁾ report that at least 34% Te^{+4} can be extracted from a 6 M HCl solution with ethyl ether. Te^{+4} will not extract appreciably into ethyl ether from a metal bromide solution (0.1 to 6 M HBr).⁽⁵¹⁾ Au^{+3} , Ga^{+3} , In^{+3} , Tl^{+3} , Sb^{+5} , Sn^{+2} , Sn^{+4} and Fe^{+3} extract quantitatively. Se^{+4} , As^{+3} , Sb^{+3} and Mo^{+6} partially extract under these conditions; Cu^{+2} , Cd^{+2} , Co^{+2} , V^{+4} , Ni^{+2} and Zn^{+2} do not. Te^{+4} is only partially extracted (5.5%) from an iodide complex (6.9 M HI) into ethyl ether.⁽⁵²⁾ Sb^{+3} .

Hg⁺², Cd⁺², Au⁺³, and Sn⁺² are completely extracted in this system; Bi, Zn, Mo⁺⁶ and In are extracted in varied amounts.

Scadden and Ballou⁽⁵³⁾ have shown that less than 5% Te⁺⁴ (and Se⁺⁴), either as carrier-free radioisotopes or as macroquantities of stable element, will extract into 0.06 M or 0.6 M di-n-butyl phosphoric acid (DBPA) from a 1 M HNO₃ solution. An organic to aqueous volume ratio of 1:1 was used in this work. Yttrium, tin, molybdenum, niobium, tantalum, zirconium and indium extract in concentrations varying from 5 to 95% or greater in the same system.

b) Chelate Complex Systems

Te⁺⁴ precipitated from a 5 N acidic solution or from solutions at pH 3.3 or pH 8.5-8.7 with a 2% sodium diethyldithiocarbamate solution will extract into either chloroform, benzene, or carbon tetrachloride.⁽⁵⁴⁻⁵⁹⁾ Bode^(58,59) has found that a 5% EDTA solution added to the system will inhibit the extraction of Se⁺⁴. Te⁺⁴ also forms a brown complex with thiosalicylideneethylenediamine that is chloroform-extractable.⁽⁶⁰⁾ This complex (like the Ni, In, Sn⁺², Cd, Pb, Au⁺³, Pt⁺², Pt⁺⁴, Co, Tl, Bi, Cu, Ag, Hg and Pd⁺² complexes) is very stable in HCl. The Zn⁺² complex is unstable while those of Al⁺³, Fe⁺², Fe⁺³, Mn⁺² and Ga⁺³ are unreactive.

Falciola⁽⁶¹⁾ reports that tellurium xanthate in a thiourea system can be quantitatively extracted into ether.

5. Separations by Ion Exchange

Kraus and Nelson⁽⁶²⁾ report that Te⁺⁴ is strongly adsorbed on Dowex-1 resin from hydrochloric acid. Hicks, et al.,⁽⁶³⁾ in their investigation of the adsorption of elements from hydrochloric acid solutions upon Dowex-2 anion exchange resin have shown that Te⁺⁴ and Te⁺⁶ are strongly adsorbed upon the resin but can be removed by eluting with 1-3 M HCl. Attebury, et al.,⁽⁶⁴⁾ and Aoki⁽⁶⁵⁾ have also reported that tellurium and selenium can be adsorbed upon an anion resin column from 3 M HCl and separated from each other by using a mixture of 1 M HCl and 1 M NH₄CNS as an elutriant. Schindewolf⁽⁶⁶⁾ also reports that tellurium and selenium can be adsorbed

upon Dowex-1 resin in strong HCl and separated from selenium by eluting the selenium from the column with 3 N HCl.

Smith and Reynolds,⁽⁶⁷⁾ using radioactive tracers, have shown that Te^{+4} (and Sn and Sb) in a 0.1 M oxalic acid solution are not adsorbed by Dowex-50 resin. In the same study, Te^{+4} was only slightly adsorbed on Dowex-1 resin and could be separated from Sn (and Sb) when a 0.1 M oxalic acid solution was passed through the column. Following the elution of Sn, antimony was removed from the column with 1 M H_2SO_4 . Stronski and Rybakow⁽⁶⁸⁾ also have recently reported on the separation of radioactive tracers of tellurium, antimony and tin by use of Soviet-produced anion exchangers.

Wish⁽⁶⁹⁾ has quantitatively separated radioactive tellurium from a mixture of fission products and uranium by use of Dowex-2 resin and phosphoric acid solutions. Tellurium is not adsorbed, and uranium can be quantitatively eluted from the column with HCl-HF solution after a conversion of the resin to the chloride form.

6. Separations by Paper Chromatography

Levi and Danon^(70,71) have used paper chromatography to separate mixtures of Bi^{210} , Pb^{210} , Po, Se, and Te and Se, Te, Po, RaD and RaE in nitric acid solutions. A butanol-propanol mixture was used as the solvent, and a good separation of each element was obtained. Crouthamel and Gatrousis⁽⁷²⁾ also report on a similar separation of Se, Te, Po and Bi by paper chromatography. Specific separations of tellurium (as Te^{+4}) from selenium (as Se^{+4}) in HCl, HNO_3 and HBr have been studied by Burstall, et al.,⁽⁷³⁾ Lederer,⁽⁷⁴⁾ and Weatherley.⁽⁷⁵⁾ Mixtures of butanol-methanol, butanol-water and butanol-HCl were used as solvents.

Pluchet and Lederer⁽⁷⁶⁾ have investigated the adsorption paper chromatography behavior of inorganic anions in acetate buffers and have shown that tellurium, either as TeO_3^{-2} or TeO_4^{-2} is retained by the paper and moves slower than the liquid front. WO_4^{-} , CrO_4^{-2} , MoO_4^{-2} , SeO_3^{-2} , VO_3^{-} and BO_3^{-} behave in a similar manner. Kertes and Beck⁽⁷⁷⁾ have studied the chromatographic behavior of metallic nitrates in organic solvents usually used in

radiochemical process extractions. TeO_3 and 57 other ions were run in solvent mixtures of ether and various concentrations of nitric acid. Most of the ions moved slower than the liquid front. The results obtained in this study were compared with another study made by Kertes⁽⁷⁸⁾ using di-isopropyl ether-nitric acid-water. A methyl isobutylketone-nitric acid-lithium nitrate-water system has also been studied by Kertes and Ben-Bassat.⁽⁷⁹⁾ Paper chromatographic conditions for the purification of carrier-free tellurium radioactive tracers from Zr^{95} and macro quantities of inactive copper as well as the separation of Te^{+4} and Te^{+6} ions has been evaluated by Ghosh-Mazumdar and Lederer.⁽⁸⁰⁾ Butanol-HCl, butanol-HBr, acetone-water-HCl, and methanol-water were used as the solvent systems.

Grassini and Lederer⁽⁸¹⁾ have used paper chromatography and an electromigration technique to study inorganic ions in 0.1 N NaOH. Tellurite (and selenite) ions moved, while tellurate ions did not, under the conditions of separation. At least 25 different inorganic ions have been investigated in this study. Lederer and Kertes⁽⁸²⁾ have shown that tellurium, as telluryl ions, can be separated from Ni^{+2} and La^{+3} by adsorbing these ions on a paper chromatogram impregnated with Dowex-50 resin. The separations were effected by eluting with H_2SO_4 solutions.

IV. DISSOLUTION OF SAMPLES CONTAINING TELLURIUM

When a tellurium-bearing sample is to be dissolved, it is necessary to use techniques that will minimize the loss of tellurium by volatilization.⁽⁸³⁾ Tellurium will volatilize from boiling concentrated hydrochloric acid, but it will not be lost from either dilute or strong acids heated below 100°C . The presence of alkali salts in a boiling process does not prevent the volatilization of tellurium.

Tellurium-containing minerals can be put into solution by fusing the mineral with sodium peroxide or a mixture of sodium carbonate and niter in a nickel crucible.⁽⁸³⁾ Fusions with potassium or KCN are not recommended because volatilization of the telluride can occur. Ores containing tellur-

ide can be decomposed by heating at dull redness in a current of chlorine and trapping the volatile tellurium chlorides in 1:1 hydrochloric acid. Biological materials, such as plant and animal tissues, body fluids, and petrochemicals, can most often be brought into solution by a wet-oxidation method using sulfuric, perchloric and nitric acid mixtures.⁽⁸⁴⁾

Any one of these dissolution techniques can be adapted for use in the radiochemistry of the tellurium radionuclides. The addition of tellurium carrier to the mixture before dissolution begins will assist in achieving an exchange of the radioactive and inactive tellurium atoms.

V. SAFETY PRACTICES

No matter what method is used to decompose a sample, adequate safety precautions should be followed. The toxicology of most elemental compounds has been reported by Pieters Creighton,⁽⁸⁵⁾ and this manual should be consulted for information in handling tellurium-containing materials safely.

Safety practices in handling radioactive sample materials is always important in radiochemistry. The discharge of radioactivity by explosion or evolution into a laboratory area can be hazardous and can result in wide-spread contamination. Thus, some source of information on safe-handling practices in processing radioactive samples should be consulted before a radiochemical analysis is undertaken. Safe-handling practices, such as those which appear in the Oak Ridge National Laboratory's Master Analytical Manual⁽⁸⁶⁾ and the International Atomic Energy Agency's publication,⁽⁸⁷⁾ should be followed in processing any radioactive material. Many other similar sources of information exist and should also be consulted.

VI. COUNTING TECHNIQUES FOR THE TELLURIUM RADIONUCLIDES

The analysis of sample materials containing tellurium radionuclides may be completed either by a direct (nondestructive) measurement of the radioactivity of the particular radionuclide or by obtaining the radionuclide in some form by radiochemically processing the radioactive sample. The use of either technique is dependent upon the tellurium radionuclide

being measured; such characteristics as the radionuclide's half-life, the type of radiations it emits as it decays, and the energy of the radiations must be considered in selecting the radioactivity measurement technique to be followed.

Table I of this monograph shows the nuclear characteristics of each of the known radioactive isotopes of tellurium. The radioactivity of any of these tellurium radionuclides can be analyzed and measured by standard Geiger-Mueller, gamma scintillation and proportional counting techniques.⁽⁸⁸⁻⁹¹⁾

VII. COLLECTION OF DETAILED RADIOCHEMICAL PROCEDURES FOR THE TELLURIUM RADIONUCLIDES

Both carrier-free and carrier radiochemical analysis procedures exist for the tellurium radionuclides. Such procedures as these have evolved from each investigator's choice of ideas and techniques similar to those reported in Section III of this monograph. Typical of the carrier-free techniques originated to prepare radioactive tellurium tracers from fission products, are those reported by Garrison and Hamilton,⁽⁹²⁾ Glendenin,⁽⁹³⁾ and Jacobson and Overstreet.⁽⁹⁴⁾ Applications of paper chromatography⁽⁸⁰⁾ and of electrolysis^(45,46) in the preparation of radioactive tellurium tracers have also been reported. Although no other specific carrier-free separations for the tellurium radionuclides appear in the current literature, it should be possible to use any of the volatility, solvent extraction or chromatography methods reported elsewhere in this monograph to serve as a means of obtaining carrier-free radioactive tellurium.

The carrier radiochemical procedures that now exist for the tellurium radionuclides have originated from investigations concerned with either the preparation of radioactive tracers,⁽⁹⁵⁾ or the separation of radioactive tellurium isotopes from fission product mixtures⁽⁹⁶⁻⁹⁸⁾ or radioactivation analysis.^(99,100) With regard to radioactivation analysis, this unique analytical method has been applied to the determination of trace stable tellurium in meteorites;^(66,101) selenium;⁽¹⁰²⁾ arsenic, indium, gallium, and their compounds;⁽¹⁰¹⁾ animal tissue;⁽¹⁰¹⁾ and alloys.⁽¹⁰¹⁾

In the radiochemical procedures that follow, special information, regarding the procedure's use, the type of nuclear bombardment, the type of material analyzed, separation time, etc., appears as part of each procedure. Whenever possible, an evaluation of each procedure is made with regard to its use in the decontamination of other radioactive species from the radioactive tellurium isotopes.

PROCEDURE 1

Procedure Used in: Carrier-free separation of tellurium radioactivity from fission products

Method: Precipitation (on selenium carrier) followed by removal of selenium by distillation.

Type of Material Analyzed: Fission products solutions

Procedure by: Glendenin⁽⁹³⁾

Chemical Yield: 50% (of selenium carrier added)

Separation Time: 1 hour

Degree of Purification: 10^2

Equipment Required: Standard

PROCEDURE

1. To an aliquot of the fission product solution add conc. HBr and evaporate solution to dryness.
2. Take up residue in 3 M HCl and add Se carrier. Saturate solution with SO_2 to precipitate elemental selenium (carries Te radioactivity).
3. Dissolve selenium metal in conc. HNO_3 . Evaporate solution to dryness.
4. Repeat Step 2.
5. Dissolve selenium metal in conc. HNO_3 and add conc. HBr to solution. Evaporate by heating to remove remaining selenium as SeBr_4 .
6. Concentrate solution to small volume, then transfer to suitable mount for radioactivity measurement.
7. Measure radioactivity by G-M counting.

PROCEDURE 2

Procedure Used in: Preparation of carrier-free radioactive tellurium tracers

Method: $\text{CuS-RuS}_2\text{-Fe(OH)}_3$ precipitations

Element Separated: Radioactive tellurium tracer

Type of Material Analyzed: Fission product solutions

Procedure by: Jacobson and Overstreet⁽⁹⁴⁾

Chemical Yield of Carrier: No Te carrier added

Separation Time: Not indicated

Degree of Purification: Not indicated

Equipment Required: Standard

PROCEDURE

1. Add conc. HCl to solution and evaporate. Take up in 3 M HCl and add Cu carrier and H_2S to precipitate CuS, carrying Te. Dissolve in conc. HNO_3 , dilute to 3 M HNO_3 with water, add Ce holdback carrier. Add enough HF to make solution 5 M in HF and to precipitate CeF_3 . Centrifuge; discard precipitate.
2. To the supernatant liquid, add Ba, Sr, Zr, Nb, and Ru carriers. Saturate solution with H_2S to precipitate RuS_2 , carrying Te. Dissolve RuS_2 precipitate in conc. HNO_3 . Add 70% HClO_4 and evaporate to volatilize off RuO_4 .
3. Add Fe carrier to the cooled solution and NH_4OH dropwise until precipitation of Fe(OH)_3 is complete. Fe(OH)_3 will carry Te. Dissolve hydroxide precipitate in 9 M HCl, transfer solution to a separatory funnel and add an equal volume of isopropyl ether to the funnel. Extract Fe^{+3} into organic phase by shaking. Separate the phases; discard the organic phase.
4. Evaporate aqueous phase to near dryness; then transfer solution to counting mount for final evaporation. Count Te beta radioactivity.

PROCEDURE 3

Procedure Used in: Preparation of carrier-free Te^{127} (115 d) and Te^{129} (33.5 d) tracers

Method: Electrolysis

Element Separated: Carrier-free Te^{127} (115 d) and Te^{129} (33.5 d) tracers.
Some contamination with Te^{132} (77 h) apparent

Type of Material Analyzed: Fission product solutions

Procedure by: Ghosh Mazumdar⁽⁴⁵⁾

Chemical Yield of Carrier: No tellurium carrier added

Separation Time: Several hours

Degree of purification: Not determined

Equipment Required: Standard

PROCEDURE

1. Evaporate an aliquot of the fission product solution to dryness. Add 70% HClO_4 and distil solution (Note 1). Take up residue in 3% HNO_3 .
2. Electrolyze the solution at a current density of 20 mA/cm^2 for 2 hours (Note 2).
3. Wash the Pt cathode and remove it from the apparatus. Evaporate the solution to dryness in a tared beaker. Then add a few milliliters of concentrated HBr to the beaker. Concentrate to small volume. Cool beaker and its contents, then weigh (Note 3).
4. Adjust acid content to 1 N HBr with water. Place gold cathode (Note 4) in beaker and then electrolyze the solution at a current density of 0.05 mA/cm^2 for approximately 2-3 hours.
5. After electrolysis, remove electrodeposited tellurium by dissolving it in a small amount of HBr . Aliquot this solution for evaporation prior to mounting for radioactivity measurements.
6. Count radioactivity with a Geiger-Muller counter.

Notes:

1. Used to remove radioactive ruthenium.
2. Pt cathode absorbs copper, salts, etc., from solution. Should be cleaned every 15 minutes with 1:1 HNO_3 .
3. Weighed to obtain quantity of constant boiling HBr in beaker. HBr reduces Te^{+6} to Te^{+4} .
4. 90% of tellurium tracer deposited on gold cathode.

PROCEDURE 4

Procedure Used in: Preparation of radioactive tracer

Method: Precipitation

Element Separated: Tellurium carrier and tellurium radioactivity

Type Material Analyzed: Iodine-131 solution ($\sim 10^8$ counts/minute)

Type of Analysis: Milking experiment

Procedure by: Marquez (Reported by Meinke⁽⁹⁵⁾)

Separation Time: 3-4 hours

Chemical Yield of Carrier: $\sim 80\%$

Decontamination: 4 extractions give a sample with ~ 300 c/m of Te and ~ 100 c/m I

Equipment Needed: Centrifuge tubes

PROCEDURE

1. Add to the I^- soln., 10 mg Te carrier as Na_2TeO_4 , acidify with H_2SO_4 until brown color is obtained then add NO_2 to oxidize to I_2 .
2. Extract the I_2 with equal volume CCl_4 .
3. Add several times more I^- and extract the I_2 with CCl_4 .
4. Ppt Te by bubbling in SO_2 gas.
5. Repeat the cycle by dissolving Te in conc. HNO_3 , adding I^- , etc.
6. Centrifuge the Te ppt and filter.
7. Plate and count as Te.

PROCEDURE 5

Procedure Used in: Preparation of radioactive tracer

Method: Precipitation

Element Separated: Tellurium carrier and tellurium radioactivity

Type Material Bombarded: Bismuth metal

Type of Nuclear Bombardment: 184" cyclotron (388 Mev alphas; 348 Mev protons; 194 Mev deuterons)

Procedure by: Goeckermann (Reported by Meinke⁽⁹⁵⁾)

Separation Time: ~1 hour

Chemical Yield of Carrier: ~60%

Decontamination: $>10^4$ from fission and spallation products

Equipment Needed: Standard

PROCEDURE

1. To aliquot of HNO_3 soln of target add 10 mg Te and Se and concentrated HBr, boil nearly to dryness a few times. Take up residue in 10 ml concentrated HCl, add Se, precipitate Se° with SO_2 in an ice bath.
2. Dilute to 3 N HCl, heat, and precipitate Te° with SO_2 , centrifuge with aerosol. Wash with HCl.
3. Dissolve Te° in a few drops HNO_3 , evaporate excess acid, dilute to 10 ml, add 6 N NaOH dropwise until H_2TeO_3 precipitate forms and then redissolves, 10 drops excess. Scavenge with 1-2 mg $\text{Fe}(\text{OH})_3$.
4. Make supernatant 3 N in HCl and precipitate Te° .
5. Repeat Se° , Te° , and $\text{Fe}(\text{OH})_3$ precipitations several times.
6. Filter last Te° precipitate, wash three times with 5 ml H_2O , three times with 5 ml $\text{C}_2\text{H}_5\text{OH}$, three times with 5 ml ether, dry 10 min. at 110°C . Weigh as Te° .

PROCEDURE 6

Procedure Used in: Preparation of radioactive tracer

Method: Precipitation

Element Separated: Tellurium carrier and tellurium radioactivity

Type Material Bombarded: Antimony

Type of Nuclear Bombardment: 184^m cyclotron deuterons and alphas

Procedure by: Lindner (Reported by Meinke⁽⁹⁵⁾)

Separation Time: ~1 hour

Chemical Yield of Carrier: 90%

Decontamination: At least a factor of 100

Equipment Needed: Standard

PROCEDURE

1. To the target add 15 drops 27 N HF in lusteroid tube in hot water bath. Add conc. HNO_3 dropwise until dissolved (10 min). Dilute to ~20 ml.
2. Add 20 mg each Te, Sn, In, Cd, Ag, Pd, Ru, Mo, Y carriers as soluble salts. Add 2 drops conc. HCl. Centrifuge.
3. Divide supn. into two equal parts.
4. Evaporate one part to near-dryness. Cool, add 20 ml 3 N HCl. Saturate with SO_2 gas 10 minutes. Te ppts.
5. Wash the ppt with water and dissolve in one drop conc. HNO_3 .
6. Add 15 ml 3 N HCl, 1 mg each, Sb, Sn, In, Cd holdback. Saturate with SO_2 gas.
7. Repeat steps 5 and 6.
8. Wash the ppt and weigh as Te.

PROCEDURE 7

Procedure Used in: Preparation of radioactive tracer

Method: Precipitation

Element Separated: Tellurium carrier and tellurium radioactivity

Type Material Bombarded: Thorium

Type or Nuclear Bombardment: 60" cyclotron alphas

Procedure by: Newton (Reported by Meinke⁽⁹⁵⁾)

Separation Time: ~1 hour

Chemical Yield of Carrier: 80%

Decontamination: $\sim 10^6$

Equipment Needed: Standard

PROCEDURE

1. To sample add 10 mg Te and dilute to 3 N HCl. Heat and ppt Te^0 with SO_2 .
2. Dissolve Te in 6 N HNO_3 . Add 10 ml conc. HCl. Evap. to rid of HNO_3 . Repeat evaporation. Dilute to 3 N HCl and ppt Te again with SO_2 .
3. Dissolve Te in 6 N HNO_3 . Evaporate and dilute to 10 ml. Add 6 N NH_4OH dropwise until H_2TeO_3 ppt dissolves. Then add 10 drops excess. Scavenge twice with 5 mg Fe^{+++} . Make 3 N in HCl and ppt Te with SO_2 . Repeat Te pptn (Step 2).
4. Filter final Te ppt. Wash three times with 5 ml H_2O , three times with 5 ml EtOH and three times with 5 ml Et_2O . Dry at 110° and weigh as Te.

PROCEDURE 8

Procedure Used in: Determination of Tellurium activity in fission

Method: Precipitation

Element Separated: Tellurium carrier and Te-132 (77 h)

Type of Material Analyzed: Irradiated uranyl nitrate

Procedure by: Novey, T. B.⁽⁹⁶⁾

Chemical Yield of Carrier: 80 to 90%

Separation Time: 1.5-2.0 hours

PROCEDURE 8 (Continued)

Degree of Purification: Contaminated with Se-81 (59m) and Rh-105 (36.5 h)

Equipment Required: Standard

PROCEDURE

1. Transfer the irradiated uranyl nitrate to a beaker and then add 10 to 50 mg of tellurium carrier and several milliliters of conc. HCl. Evaporate to near dryness to remove NO_3^- . Repeat HCl addition and evaporation at least three times.
2. Dissolve residue in beaker with 3 N HCl. Heat and saturate solution with SO_2 to precipitate tellurium metal. Transfer mixture to centrifuge tube (Note 1); centrifuge and discard supernatant liquid.
3. Dissolve precipitate in several milliliters of 6 M HCl plus a few drops of 6 N HNO_3 . Remove excess NO_3^- by fuming with several portions of HCl.
4. Dissolve residue in 3 N HCl and repeat SO_2 saturation to precipitate tellurium metal.
5. Collect tellurium on a small filter-paper disc in a Hirsch funnel. Wash well with water and alcohol. Dry by suction, then transfer to drying oven at 100°C . Dry for 10 minutes. Weigh.
6. Mount and count. Use Geiger-Muller counter for radioactivity measurements. (Note 2)

Notes:

1. Add Aerosol to solution before centrifugation in order to collect tellurium metal scum from walls of centrifuge tube.
2. Radioactivity measurements usually made 24 hours after separation in order to allow 2.4 h I-132 daughter radioactivity to reach equilibrium.

PROCEDURE 9

Procedure Used in: Determination of tellurium radioactivity in fission

Method: Precipitation

Element Separated: Tellurium carrier and Te radioactivity

Type of Material Analyzed: Irradiated uranyl nitrate

Procedure by: Glendenin, L. E. (97)

Chemical Yield of Carrier: 60%

PROCEDURE 9 (Continued)

Separation Time: . 1 hour

Degree of Purification: 10^4

Equipment Required: Standard

PROCEDURE

1. To the sample of irradiated uranyl nitrate in a 125-ml Erlenmeyer flask add 3 ml of tellurium carrier and 10 ml of conc. HBr. Evaporate to near dryness by swirling over a burner. Repeat the evaporation twice with 5 ml of conc. HBr (Note 1).
2. Take up the residue from the HBr treatment in 20 ml of 3 M HCl and transfer to a 50-ml centrifuge tube. Heat the solution nearly to boiling, pass in SO_2 until the black precipitate of tellurium is well coagulated (1 to 2 minutes). Centrifuge, and wash with 10 ml of H_2O (Note 2).
3. Dissolve the tellurium by heating with 5 to 10 drops of HNO_3 , evaporate nearly to dryness, add 2 to 3 drops of 6 M HCl, and dilute to 10 ml (Note 3). Heat nearly to boiling, neutralize with 6 M NH_4OH , and add more reagent drop by drop until the precipitate of H_2TeO_3 redissolves; then add 10 drops in excess. Add 1 to 2 mg of Fe^{+3} carrier drop by drop with stirring, digest the precipitate of $\text{Fe}(\text{OH})_3$ for a few seconds, centrifuge, and discard (Note 4).
4. Add an equal quantity of 6 M HCl to the supernatant solution, heat nearly to boiling, and precipitate tellurium with SO_2 . Dissolve the tellurium in 5 to 10 drops of 6 M HNO_3 , evaporate nearly to dryness, and reprecipitate with SO_2 in 10 ml of hot 3 M HCl. Filter with suction on a tared filter-paper disc (Note 5) in a small Hirsch funnel, and wash three times with 5 ml of H_2O and three times with 5 ml of ethanol. Dry at 110°C for 10 minutes, weigh as elementary tellurium. Mount for counting.
5. Count on Geiger-Muller counter.

Notes:

1. Evaporation with HBr removes NO_3^- , which interferes with the SO_2 reduction, reduces any Te^{+6} present to Te^{+4} and removes any selenium present by volatilization of SeBr_4 .
2. A few drops of 0.1 percent Aerosol prevent scum formation and aid in centrifugation.
3. A white precipitate of H_2TeO_3 may be formed by hydrolysis at this point but dissolves readily upon heating.
4. Scavenging with $\text{Fe}(\text{OH})_3$ removes general contamination, including rhodium.
5. The filter-paper disc is washed with ethanol and is dried under the conditions of the procedure before the weighing.

PROCEDURE 10

Procedure Used in: Separation of radioactive tellurium isotopes from other fission products

Method: Precipitation

Element Separated: Tellurium carrier and tellurium radioisotopes; includes Te-127m (100 d), Te-127 (9.3 h), Te-129m (33 d), and Te-132 (77 h)

Type of Material Analyzed: Aqueous and organic fission product solutions

Type of Nuclear Bombardment: Uranium fission

Procedure by: Druschel, R. E. (98)

Chemical Yield of Carrier: >70%

Separation Time: 1-1.5 hours

Degree of Purification: Negligible contamination from other fission products

Equipment Required: Standard laboratory equipment

PROCEDURE

1. Pipet an appropriate volume, no larger than 10 ml, of the sample into a 125-ml Erlenmeyer flask, add 2 ml of tellurium carrier and 10 ml of conc. HBr solution. Evaporate the solution to near dryness by swirling it over a burner. (Note 1) Repeat the evaporation twice using 5 ml of conc. HBr solution each time.
2. Dissolve the residue from the HBr treatment in 20 ml of 3 M HCl and transfer the solution to a 50-ml glass centrifuge tube. Heat the solution to near boiling, pass in SO₂ until the black precipitate of the tellurium (Note 2) that is formed is well coagulated, centrifuge the mixture, discard the supernatant liquid, and wash the precipitate with 10 ml of water (Note 3).
3. Dissolve the Te precipitate by heating it with 5 to 10 drops of 6 M HNO₃. Evaporate the solution to near dryness, add 2 to 3 drops of 6 M HCl, and dilute it to 10 ml (Note 4). Heat to near boiling, and add 6 M NH₄OH solution dropwise until the precipitate of H₂TeO₃ redissolves, and then add 10 drops in excess. Add 1 to 2 mg of ferric nitrate holdback carrier dropwise with stirring. Digest the precipitate of Fe(OH)₃ for a few seconds, centrifuge, and discard the precipitate (Note 5).
4. Add an equal quantity of 6 M HCl to the supernatant liquid, heat the solution to near boiling, and precipitate the Te with SO₂. Dissolve the Te in 5 to 10 drops of 6 M HNO₃, evaporate the solution to near dryness, add 10 ml of hot, 3 M HCl, and reprecipitate the Te with SO₂. Filter with suction through a tared filter disk cut from Whatman No. 40 filter paper that is placed in a small Hirsch funnel, wash the precipitate with three 5-ml volumes of water and three 5-ml volumes of ethanol. Dry the precipitate at 110°C for 10 minutes, weigh it as elemental Te, and mount it for counting (Notes 6, 7).

PROCEDURE 10 (Continued)

Notes:

1. Evaporation with HBr removes selenium as selenium tetrabromide and nitrates which interfere in the reduction of tellurates to tellurite.
2. If rhenium is present, it will form rhenium telluride. Readily removed in ferric hydroxide scavenging steps.
3. The addition of a few drops of 0.1% Aerosol solution prevents scum formation and aids in centrifugation.
4. At this point, a white precipitate of H_2TeO_3 may be formed by hydrolysis but dissolves readily upon heating. The presence of a little HCl accelerates the dissolution of the Te.
5. An excessive loss of Te by coprecipitation with $Fe(OH)_3$ is prevented by the favorable ratio of Te(IV) to Fe carrier (20:1) and by the addition of ferric nitrate holdback carrier after the solution has been made alkaline.
6. "Young" material requires special conditions for the counting of 77-h Te. Samples from material that is more than three-weeks old contain only the longer-lived Te activities and can be counted in the usual manner.
7. Beta radioactivity of the mounted precipitation counted on a Geiger-Muller counter.

PROCEDURE 11

Procedure Used in: Separation of Te-132 (77 h) from uranium

Method: Anion exchange resin column

Element Separated: Te-132 (77 h)

Type Material Analyzed: Uranium solutions

Procedure by: Wish⁽⁶⁹⁾

Separation Time: 1-2 hours

Degree of Purification: Complete decontamination from uranium

Equipment Required: Standard

PROCEDURE

1. Prepare resin column by packing Dowex 2 resin into a column 0.2 cm I.D. by 15 cm long.

PROCEDURE 11 (Continued)

2. Condition column with 0.1 N phosphoric acid.
3. An aliquot of the sample was adjusted to 1.0 N H_3PO_4 and loaded on to the column.
4. Elution of the mixture from the column was made with 25 ml of 0.1 N H_3PO_4 at a flow rate of 1 ml per 4 minutes.
5. Eluate containing Te-132 collected in suitable container (Note 1), then aliquots taken for radioactivity measurement.

Note:

1. Te-132 does not absorb on column. Uranium removed from column by treating column with HCl and eluting with 8 ml of 0.1 N HCl-0.06 N HF solution.

PROCEDURE 12

Procedure Used in: Separation of tellurium radioactivity from fission products

Method: Electrodeposition

Element Separated: Tellurium carrier and radioactive tellurium tracer

Type of Material Analyzed: Fission products solutions

Procedure by: Lee and Cook⁽⁴⁶⁾

Chemical Yield of Carrier: 70%

Separation Time: Not indicated

Degree of Purification: Adequate decontamination from other fission products

Equipment Required: Standard

PROCEDURE

1. Add Te and Ag carriers to fission product solution and sufficient HCl to make solution 2-3 M HCl. Saturate solution with H_2S , collect sulfide precipitate by filtration. Discard filtrate.
2. Dissolve mixed sulfide precipitate in aqua regia, add conc. H_2SO_4 and evaporate. Cool solution, then add Ru carrier and $HClO_4$. Evaporate to near dryness to remove RuO_4 .

PROCEDURE 12 (Continued)

3. Cool solution, add a few milliliters of conc. HCl and boil to reduce Te^{+6} to Te^{+4} . Transfer solution to electrolysis beaker.
4. Make solution ammonical with NH_4OH , then deposit Ag on Pt cathode by electrolyzing solution at a voltage of between 0.05 and 0.25 V. Remove cathode from solution and replace with a new Pt cathode.
5. Add conc. HCl and NH_2OH to solution; then deposit Te at Pt cathode by electrolyzing solution at a voltage of between 0.27 and 0.15 V.
6. Remove Pt cathode from electrolysis beaker, wash and dry. Mount for radioactivity measurements.
7. Count tellurium radioactivity by means of G-M counter.

PROCEDURE 13

Procedure Used in: Radioactivation analysis

Method: Fusion, precipitation, and ion exchange column separation

Element Separated: Tellurium carrier and radioactive Te-127 (9.4 h) and Te-132 (77 h). (See Note 1)

Type of Material Analyzed: Stoney meteorites⁽⁶⁶⁾

Type of Nuclear Bombardment: n, γ and uranium fission reaction products (see Note 1)

Procedure by: Schindewolf⁽⁶⁶⁾

Chemical Yield of Carrier: Quantitative

Separation Time: Several hours

Degree of Purification: Decontamination of $>10^4$ for Ag, Ce, Co, Cs, Hg, Ir, Nb, Ru, Sb, Sc, Ta, Zn and Zr

Equipment Required: Neutron source and standard laboratory equipment

PROCEDURE

1. Powdered meteorite samples weighing 0.050 to 0.20 gram were irradiated in the Argonne National Laboratory CP-5 reactor.
2. After irradiation, 0.020 gram each of tellurium and selenium was added to the irradiated sample and the mixture fused in a nickel crucible with 1-2 grams of Na_2O_2 .
3. After cooling, the cake was dissolved in 6 N HCl and the mixture boiled. Tellurium and selenium reduced to elemental form with SO_2 gas.

PROCEDURE 13 (Continued)

4. Precipitate dissolved in aqua-regia and the solution made basic with NH_4OH . Impurity scavenge made with $\text{Fe}(\text{OH})_3$.
5. Solution acidified with HCl and the tellurium and selenium adsorbed on a Dowex-1 (100-200 mesh) resin column. Selenium was separated from tellurium by eluting it from the column with 3 to 5 column volumes of 3 N HCl.
6. The tellurium is then eluted from the column with 0.2-0.5 N HCl and the solution treated with SO_2 . The precipitate of elemental tellurium was collected by filtration, weighed and mounted for the radioactivity measurement.
7. The beta radioactivity of the 9.4 h Te-127 was measured by means of a beta counter.
8. Comparator samples were irradiated and processed in a similar manner to obtain the tellurium concentration of the meteorite.

Note:

1. 77 hr- Te^{132} resulted from fission of uranium, a constituent of the sample material.

PROCEDURE 14

Procedure Used in: Radioactivation analysis

Method: Precipitation

Element Separated: Tellurium carrier and radioactive Te isotopes

Type of Material Analyzed: Metals and alloys; ⁽¹⁰¹⁾ animal tissue; ⁽¹⁰¹⁾ meteorites ⁽¹⁰¹⁾

Type of Nuclear Bombardment:

| | | | |
|-------------------|----------------|--------------------|------------------|
| Te_{120} | (n, γ) | Te_{121m} | 127 d half-life |
| Te_{122} | (n, γ) | Te_{123m} | 104 d half-life |
| Te_{124} | (n, γ) | Te_{125m} | 58 d half-life |
| Te_{126} | (n, γ) | Te_{127m} | 115 d half-life |
| Te_{126} | (n, γ) | Te_{129m} | 33.5 d half-life |
| Te_{130} | (n, γ) | Te_{131m} | 30 h half-life |

Procedure by: Leddicotte, G. W. and Mullins, W. T. (Reported by Leddicotte ⁽¹⁰¹⁾)

Chemical Yield of Carrier: >70%

Separation Time: 1.5-2.0 hours

Degree of Purification: Studies with radioactive tracers of Ag-110, Cd-115, As-76, Zn-65, In-114, Sn-125, and Sb-124 show that decontamination is better than 10^5 for each

PROCEDURE 14 (Continued)

Equipment Required: Neutron source and standard laboratory equipment

PROCEDURE

A. Irradiation of Samples

1. Irradiate known amounts of test (Note 1) and comparator (0.025 to 0.030 g of tellurium metal) samples (Note 2) in a neutron flux of at least 5×10^{11} n/cm²/sec for 1 week (Note 3). Irradiate the samples in either small quartz tubes, polyethylene bottles, or aluminum foil.

B. Preparation of Irradiated Samples for Analysis

1. The Comparator Sample

- a. After the irradiation, quantitatively transfer the comparator sample (Note 2) to a 50-ml volumetric flask, then dissolve it in a small measured volume of conc. HNO₃; then dilute the solution to 50 ml with water.

Mix well, using safe-handling practices for radioactive materials.

- b. Pipet a 1.00 ml aliquot of this solution into a 50-ml centrifuge tube and then add by means of a volumetric pipet to the same tube 3.0 ml of a standard carrier solution of known tellurium concentration. (Note 4). Continue with Part C below.

2. The Test Sample(s)

- a. If the sample is a metal or alloy, quantitatively transfer the irradiated test portion from the quartz tube or aluminum wrap to a 50-ml centrifuge tube, and then add, by means of a volumetric pipet, to the same centrifuge tube 2.00 ml of a standard carrier solution of known tellurium concentration (Note 4). To this mixture, add dropwise, enough concentrated mineral acid to completely dissolve the sample. If necessary, heat the mixture to effect solution. Continue with Part C below.
- b. If the sample is a biological material (tissue ash, etc.), quantitatively transfer the total amount of solid from its irradiation container to a 50-ml centrifuge tube. (Note 5) Then add, by means of a volumetric pipet, to the same centrifuge tube 2.00 ml of a standard carrier solution of known tellurium concentration (Note 4). To this mixture, add 3 ml of H₂O, 1 ml of 6 M HCl and 15 ml of HNO₃-HClO₄ mixture (2 parts conc. HNO₃ to 1 part 70% HClO₄). Digest the mixture at a moderate temperature (60°C) for at least 1-2 hours (Note 6). Cool, then continue with Part C below.

C. Radiochemical Separation of Tellurium

1. To any of the solutions obtained above, add 1 ml each of holdback carriers of selenium, silver, cadmium, arsenic, zinc, indium, tin, and antimony (Notes 7 and 8).
2. Heat this solution to near boiling; then add 3 ml of 6% H₂SO₃ and 1 drop of 1% Aerosol solution. Stir the mixture to coagulate the pre-

PROCEDURE 14 (Continued)

- precipitate. Centrifuge; discard the supernatant liquid. Wash the precipitate once with a 10 ml volume of water. Centrifuge and discard the wash liquid.
3. Dissolve the Te^0 precipitate in 6 N HNO_3 . Transfer the solution to a 125-ml distillation flask and add 10 ml conc. HCl , 10 ml 48% HBr and 2 ml of selenium holdback carrier. Heat to boiling and distil to ~5 ml volume. Cool flask, then add 5 ml conc. HCl and 5 ml 48% HBr , heat and distil to ~2 ml. Discard the distillate.
 4. Transfer the solution from the distillation flask to a centrifuge tube. Add conc. NH_4OH dropwise until a white precipitate forms and then dissolves. Add 1-2 ml of NH_4OH in excess and 1 ml of Fe holdback carrier centrifuge; then add 1 ml more of Fe holdback carrier, swirl and centrifuge.
 5. Filter the supernatant liquid through Whatman 41 H filter paper into a clean 50 ml centrifuge tube. Neutralize the solution with HCl ; adjust the acidity to 3 N HCl .
 6. Heat the solution to near boiling and then add 2-3 ml of 6% H_2SO_3 and 1 drop 1% Aerosol. Stir to coagulate precipitate. Centrifuge; discard the supernatant liquid. Wash the precipitate with two 10-ml volumes of H_2O .
 7. Filter through Munktell (# 00) filter paper. Wash with H_2O , alcohol and ether. Weigh, mount and count.

D. Measurement of the Radioactivity from the Tellurium Radioisotopes and Calculation of Inactive Tellurium Content of the Original Sample

1. The radioactivity from all of the longer-lived tellurium radioisotopes (Te-121m , Te-123m , Te-125m , Te-127m , Te-129m , and Te-131m) must be considered in this analysis. All of them decay with gamma radiation and the measurements can be made by means of either a gamma scintillation counter or a gamma scintillation spectrometer. In the use of the latter instrument, the major gamma radiations are measured.
2. Following the radioactivity measurements, the observed radioactivity is corrected for loss of "carrier" during the experiment, half-life of the tellurium radioisotope measured (if gamma spectrometry is used) and the weights of the test and comparator samples. A comparison of these corrected radioactivities becomes a measure of the stable tellurium content of the test sample:

Percent Stable Te in Test Sample =

$$\frac{\text{Corrected Te Radioactivity in Test Sample}}{\text{Corrected Te Radioactivity in Comparator Sample}} \times 100.$$

Notes:

1. At least 0.10 gram portions of solid samples (metals, alloys, tissues, etc.) should be used.
2. Comparator sample may be either tellurium metal or a solution of a tellurium compound. If a solution is used, it should contain

PROCEDURE 14 (Continued)

at least 40 micrograms of tellurium per aliquot used; an aliquot of the irradiated solution can be pipetted directly to the 50-ml centrifuge tube.

3. The Oak Ridge National Laboratory Graphite Reactor was used for the irradiations. The sensitivity of the method is such that 1×10^{-6} gram of tellurium can be determined. The sensitivity can be enhanced by use of higher neutron fluxes.
4. As tellurium metal in $\text{HNO}_3\text{-HCl}$ mixture; Te^{+4} concentration equals 10 milligrams per milliliter.
5. A 100-ml beaker may be substituted here for the centrifuge tube.
6. Sample should be dissolved in this time. Additional HNO_3 may have to be added during the sample digestion.
7. Holdback carriers should be made up to contain 5 milligrams per milliliter of desired elemental species.
8. Solutions of ions of other elements may also be added as holdback carriers.

PROCEDURE 15

Procedure Used in: Radioactivation analysis

Method: Precipitation

Element Separated: Tellurium carrier and radioactive tellurium isotopes

Type of Material Analyzed: Selenium⁽¹⁰²⁾

Type of Nuclear Bombardment: n, γ reaction products

Procedure by: Zvyagintsev and Shamaev⁽¹⁰²⁾

Chemical Yield of Carrier: 30 to 50%

Separation Time: Not indicated

Degree of Purification: Not indicated

Equipment Required: Standard

PROCEDURE

Authors⁽¹⁰²⁾ indicate that Te has been separated and isolated from radionuclides of Cu, Ga, As, Sb and Cd. Publication should be consulted.

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