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The Radiochemistry of Sodium

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CHEMISTRY

The Radiochemistry of Sodium

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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 sodium 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 material on the radiochemistry of sodium which might be included in a revised version of the monograph.

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II. THE RADIOACTIVE NUCLIDES OF SODIUM

The radionuclides of sodium that are of interest in the radiochemistry of sodium are given in Table I. This Table has been completed from information appearing in reports by Strominger, et al,(1) and by Hughes and Harvey.(2)

TABLE I

THE RADIOACTIVE NUCLIDES OF SODIUM

Radio-Nuclide	Half-life	Mode of Decay	Energy of Radiation, Mev	Produced By
Na^{20}	0.385 s	β^+	β^+ 3.5	Ne-p-m, Na-γ-3n
Na^{21}	23 s	β^+	β^+ 2.5	Ne^{20}-p-γ, Ne-p-n, Ne-d-n Mg-p-α, Mg^{24}-p-α
Na^{22}	2.6 y.	β^+	β^+ 0.542	F-α-n, Ne-d-n, Ne^{21}-p-γ Na-n-2n, Mg-d-α
Na^{24}	15 h	β^-	β^- 1.39, 4.14 γ 1.38, 2.76	Na-d-p, Na-n-γ, Mg-d-α Mg-n-p, Mg-γ-p, Al-n-α Al-d-pα, Al-γ-n2p, Al-p-2-n Si-γ-2p
Na^{25}	60 s	β^-	β^- 3.7, 2.7	Mg-γ-p, Mg-n-p Al-γ-2p

III. THE CHEMISTRY OF SODIUM AND ITS APPLICATION IN ANALYSIS METHODS FOR STABLE SODIUM OR ITS RADIONUCLIDES

Radiochemical analysis methods usually follow the ideas and techniques established in conventional methods, such as precipitation, solvent extraction, chromatography, and electrolysis. Since these methods are dependent upon chemical reactions to bring them to completion, the general information presented below on the formation of sodium compounds attempts to relate these more conventional methods to the radiochemistry methods used in analyzing a radioactive material for the sodium radionuclides.

A. The General Chemistry of Sodium

Sodium composes about 2.83% of the earth's crust and occurs principally in the form of sodium chloride. Other natural compounds of sodium include the nitrate, the sulfate, the carbonate, the bicarbonate, and the tetraborate compounds. Sodium also occurs as the silicate in ingeous rocks. Most of the sodium compounds are found in soils, waters, and in the bodies of plants and animals. Sodium metal is chiefly produced from these compounds by first preparing the chloride and then electrolytically fusing it with sodium carbonate.

1. Metallic Sodium

Sodium metal is a soft, silver-white metal having a density of 0.97 and a melting point of 97.5°. It tarnishes quickly in air and must be kept under a liquid such as kerosene. It will burn in air with a bright yellow flame, and it reacts violently with water. It will unite directly with the halogens, sulfur, selenium, and tellurium. Sodium forms an amalgam with mercury to become a very active reducing agent. Sodium also alloys with lead, tin, and antimony, but it will not alloy with iron.

2. The Chemical Compounds of Sodium

The only oxidation state of sodium is +1. Its reactions with carbon dioxide, hydrogen, oxygen, nitrogen, sulfur, and the halogens are similar to those for the other alkali metals, i.e., lithium, potassium, rubidium, and cesium. Its violent reactions with water and aqueous solutions can be moderated by using an amalgam, i.e., a solution of sodium in mercury. Sodium also forms strong salts with all of the common acids.

Table II shows the relative solubility of many of the sodium compounds in water and other reagents. The following information generally describes the reactions of sodium with other elements.

a. The Hydrogen Compound. Sodium hydride, NaH, can be formed by heating sodium and hydrogen directly. It has a melting point of 800°. The hydride reacts with water to produce hydrogen gas.

b. The Oxide Compounds. Two oxide compounds are formed by sodium in its reactions with oxygen: normal oxide, Na_2O, and the peroxide, Na_2O_2. Sodium monoxide, Na_2O, is formed by heating either sodium hydroxide, or sodium nitrate with excess sodium metal. It can also be formed by heating a mixture of sodium azide, NaN_3, and sodium nitrate at 280°. Na_2O is a colorless compound that combines with water to form NaOH.

Sodium peroxide, Na_2O_2, is formed by burning sodium in an excess of air or oxygen. Na_2O_2 is a pale yellow solid that melts at 460°. If it is added to a mixture of ice and water, sodium peroxide hydrate, $Na_2O_2 \cdot 8H_2O$

is produced. When Na_2O_2 is heated, oxygen is given off and carbon dioxide slowly decomposes it to form sodium carbonate and to evolve oxygen. At 0° C, sodium peroxide reacts with alcohol to produce sodium hydrogen peroxide, $NaHO_2$. Sodium peroxide will react with water, with some violence, to produce sodium hydroxide, oxygen, and hydrogen peroxide. Na_2O_2, either in aqueous solution, or as the fused peroxide, is a powerful oxidizing agent.

c. Sodium Hydroxide. Two general processes, i.e., the action of calcium hydroxide on sodium carbonate and an electrolysis method using sodium amalgam and brine, are used to produce sodium hydroxide, NaOH. Sodium hydroxide is a colorless and wax-like solid. It melts at 319° and boils at about 1390°. NaOH dissolves in water with the evolution of heat to yield a strong basic solution that can be used as a precipitant for several of the chemical elements.

d. The Nitrogen Compounds. Sodium nitride, Na_3N, a salt of hydrazoic acid, HN_3, can be formed by reacting sodium and nitrogen in an electric discharge tube. Sodium azide, NaN_3, can also be formed by passing nitrous oxide, N_2O, over sodamide, $NaNH_2$, at 190°. NaN_3 decomposes when gently heated *in vacuo* to give sodium nitride and nitrogen gas. The sodium nitride compounds are rapidly attacked by water, forming sodium hydroxide and ammonia in the reaction.

Sodium nitrate, $NaNO_3$, is obtained by the action of HNO_3 on sodium metal, sodium hydroxide, or sodium carbonate. $NaNO_3$ is hygroscopic and dissolves more rapidly in hot water than cold. It is anhydrous, and it will melt at 380° and be decomposed. When heated above 400°, $NaNO_3$ will lose oxygen and form sodium nitrite, $NaNO_2$. Sodium nitrite is produced as very small, hygroscopic crystals. $NaNO_2$ is a strong electrolyte, and it will melt upon heating to form a yellow liquid which decomposes at higher temperatures. $NaNO_2$ will hydrolyze to nitrous acid when boiled with water.

e. The Sulfide, Sulfate, and Sulfite Compounds. Sodium will react directly with sulfur to form either sodium monosulfide, Na_2S, or sodium polysulfide, Na_2S_2. Sodium monosulfide is a colorless crystalline solid with a melting point of 1180°. Na_2S is hygroscopic, and it reacts readily

Table II. Solubility of Sodium Compounds

Compound	Formula	Water Solubility		Other Solvents
		Cold	Hot	
Acetates	$NaC_2H_3O_2$	Soluble	Soluble	Soluble in alcohol
	$NaC_2H_3O_2 \cdot 3H_2O$	Soluble	Soluble	Soluble in ether and alcohol
Aluminates	$NaAlO_2$	Soluble	Very soluble	Insoluble in alcohol
	$NaClAlCl_3$	Soluble	Soluble	
Amide	$NaNH_2$	Decomposes	Decomposes	Decomposes in hot alcohol; soluble 0.1 g in liquid NH_3
Antimonates	$NaSb(OH)_6$	Slightly soluble	Soluble	Slightly soluble in alcohol
	Na_3Sb	Decomposes		
	$NaSbO_2 \cdot 3H_2O$	Decomposes		
Arsenates	$Na_3AsO_4 \cdot 12H_2O$	Soluble		Soluble in alcohol
	$Na_2HAsO_4 \cdot 7H_2O$	Soluble	Soluble	Slightly soluble in alcohol
	$Na_2HAsO_4 \cdot 12H_2O$	Soluble	Soluble	Slightly soluble in alcohol; insoluble in liquid Cl
	$NaAsO_2$	Very soluble	Very soluble	Slightly soluble in alcohol
Azide	NaN_3	Soluble	Soluble	Soluble in liquid NH_3 and alcohol; insoluble in ether
Barbital	$NaC_8H_{11}N_2O_3$	Soluble	Soluble	Slightly soluble in alcohol; insoluble in ether
Benzene Sulfonate	$NaC_6H_5SO_3$	Soluble	Very soluble	
Benzoate	$NaC_7H_5O_2$	Soluble	Soluble	Soluble in alcohol
Metabismuthate	$NaBiO_3$	Insoluble	Decomposes	Decomposes in acid
Bismuthide	Na_3Bi	Decomposes		
Borates	$NaBO_2$	Soluble	Very soluble	
	$NaBO_2 \cdot 4H_2O$	Very soluble	Very soluble	
	$Na_2B_4O_7$	Soluble	Soluble	Insoluble in alcohol
	$Na_2B_4O_7 \cdot 5H_2O$	Soluble	Soluble	
	$Na_2B_4O_7 \cdot 10H_2O$	Soluble	Soluble	Soluble in glycerin; very slightly soluble in alcohol; insoluble in acid
	$NaBO_3 \cdot H_2O$	Slightly soluble	Decomposes	Soluble in glycerin and alkali
	$NaBO_3 \cdot 4H_2O$	Soluble	Soluble	Soluble in acid, glycerin, and alcohol
	$NaBH_4$	Decomposes	Decomposes	Decomposes in acid
Bromides	$NaBrO_3$	Soluble	Soluble	Insoluble in alcohol
	$NaBr$	Soluble	Soluble	Slightly soluble in alcohol
	$NaBr \cdot 2H_2O$	Soluble	Soluble	Slightly soluble in alcohol
Carbonates	Na_2CO_3	Soluble	Soluble	
	$NaHCO_3$	Soluble	Soluble	
Chlorates	$NaClO_3$	Soluble	Soluble	Soluble in alcohol and glycerine
	$NaClO_4$	Soluble	Very soluble	Soluble in alcohol
Chloride	$NaCl$	Soluble	Soluble	Soluble in glycerin; slightly soluble in alcohol and liquid NH_3; insoluble in HCl
Fluorides	NaF	Soluble		Soluble in HF; very slightly soluble in alcohol
	$NaF \cdot HF$	Soluble	Soluble	
	Na_2SiF_6	Soluble	Soluble	Insoluble in alcohol
Hydroxide	$NaOH$	Soluble	Soluble	Soluble in alcohol and glycerol
Iodides	$NaIO_3$	Soluble	Soluble	Soluble in acetic acid; insoluble in alcohol

Table II is continued on the following page.

Table II (Continued)

Compound	Formula	Water Solubility		Other Solvents
		Cold	Hot	
	$NaIO_4$	Soluble		Soluble in H_2SO_4, HNO_3, and acetic acid
	NaI	Soluble	Soluble	Very soluble in alcohol; soluble in glycerin
Nitrate	$NaNO_3$	Soluble	Very Soluble	
Oxalate	$Na_2C_2O_4$	Soluble	Soluble	
Oxides	Na_2O	Decomposes	Decomposes	Decomposes in alcohol
	Na_2O_2	Soluble	Decomposes	Decomposes in alcohol, NH_3; soluble in dilute acid; insoluble in alkali
Phosphates	NaH_2PO_4	Soluble	Soluble	
	Na_2HPO_4	Soluble	Soluble	
	Na_3PO_4	Soluble	Soluble	
	$NaNH_4HPO_4 \cdot 4H_2O$	Soluble	Soluble	Insoluble in alcohol
Silicate	$Na_2SiO_3 \cdot 5H_2O$	Soluble	Soluble	
Sulfates	Na_2SO_4	Soluble	Soluble	Soluble in glycerin; insoluble in alcohol
	$Na_2SO_4 \cdot 7H_2O_4$	Soluble	Soluble	Insoluble in alcohol
	$NaHSO_4$	Soluble	Soluble	Decomposes in alcohol; slightly soluble in alcohol; insoluble in NH_3
	$Na_2S_2O_7$	Soluble		Soluble in fuming H_2SO_4
	$Na_2S_2O_8$	Soluble; decomposes		Insoluble in alcohol
	$Na_2Ca(SO_4)_2 \cdot 2H_2O$	Decomposes	Decomposes	
Sulfides	Na_2S	Soluble	Soluble	Decomposes in acids; slightly soluble in alcohol; insoluble in ether
	NaHS	Very soluble		Soluble in alcohol
	Na_2S_4	Soluble		Soluble in alcohol
	Na_2S_5	Soluble	Soluble	Soluble in alcohol
Sulfites	Na_2SO_3	Soluble	Soluble	Slightly soluble in alcohol; insoluble in liquid Cl_2 and NH_3
	$NaHSO_3$	Very soluble	Very soluble	Slightly soluble in alcohol
	$Na_2S_2O_5$	Soluble	Soluble	Insoluble in alcohol

with water to form a strongly alkaline solution. Sodium sulfide solutions are unstable and will break down to form polysulfides, thiosulfates, and sulfates. Sodium hydrogen sulfide, NaHS, can be formed by saturating a sodium hydroxide solution with H_2S.

Sodium sulfates can occur in nature. However, sodium hydrogen sulfate, $NaHSO_4$, is produced by heating NaCl with an excess of H_2SO_4 at temperatures lower than 150°. If $NaHSO_4$ is heated to about 400°, it is converted to normal sodium sulfate, Na_2SO_4, which is readily soluble in water. Sodium thiosulfate is prepared by boiling sodium sulfite, Na_2SO_3, in the presence of sulfur. Sodium thiosulfate, as $Na_2S_2O_3 \cdot 5H_2O$, is used principally in photographic film processing. Here, the excess silver bromide, which remains in photographic film after developing, dissolves very readily in the thiosulfate to form a soluble complex. Sodium thiosulfate is also used as a titrant for the determination of iodine.

Sodium hydrogen sulfite, or sodium bisulfite, $NaHSO_3$, is formed by saturating a solution of sodium carbonate or sodium hydroxide with sulfur dioxide. Normal sodium sulfite, Na_2SO_3, is formed by treating the solution with an additional amount of sodium carbonate. The sulfite solutions are alkaline and are good reducing agents. Both the HSO_3^- and SO_3^{--} ions are readily oxidized to SO_4^{--} ions by atmospheric oxygen or oxygen-containing compounds.

f. The Halogen Compounds. The sodium halide compounds, i.e., sodium chloride, HaCl; sodium fluoride, NaF; sodium bromide, NaBr; and sodium iodide, NaI, may be prepared either by a direct union of the elements or by the reaction of a halogen acid upon sodium hydroxide or sodium carbonate. All of the sodium halides are strong salts and are completely ionized. They are readily soluble in water at room temperature.

Sodium salts of the oxygen acids of the halogens include sodium chlorate, sodium hypochlorite, sodium perchlorate, sodium bromate, and sodium iodate. Sodium chlorate, $NaClO_3$, is prepared by passing chlorine gas into hot concentrated sodium hydroxide. $NaClO_3$ is a colorless crystalline compound that is more soluble in hot water than in cold. It is a strong oxidizing agent and will decompose on heating to form free chlorine and oxygen. The corresponding bromate, $NaBrO_3$, and the iodate, $NaIO_3$, are prepared in a similar manner and have similar properties as $NaClO_3$.

Sodium perchlorate, $NaClO_4$, is formed by the anodic oxidation of $NaClO_3$. It is a colorless compound and a strong oxidizing agent. Sodium hypochlorite, NaClO, is produced by passing chlorine into a cold, dilute solution of sodium hydroxide. It is easily reduced to chloride ions and oxygen.

g. The Phosphorus Compounds. Three classes of sodium phosphate compounds can be prepared by adding theroetical amounts of phosphoric acid, H_3PO_4, to sodium hydroxide: monosodium dihydrogen phosphate, NaH_2PO_4, disodium monohydrogen phosphate, Na_2HPO_4, and trisodium phosphate, Na_3PO_4. NaH_2PO_4 is acidic in aqueous solutions. Na_2HPO_4 is only slightly alkaline while Na_3PO_4 is strongly alkaline in aqueous solutions.

When sodium dihydrogen phosphate, NaH_2PO_4, is heated, a series of sodium metaphosphate compounds are formed. They appear to be all polymeric and are represented by the empirical formula $(NaPO_3)_n$. Calcium and magnesium ions can chelate with these polyphosphates. If sodium monohydrogen phosphate, Na_2HPO_4, is dehydrated by heating, *sodium pyrophosphate*, $Na_4P_2O_7$, is formed. $Na_4P_2O_7$ can be hydrolyzed in an aqueous solution to form $HPO_4^=$ ions. If NaH_2PO_4 and Na_2HPO_4 are mixed and heated to 600°, a more complex phosphate, *sodium triphosphate*, $Na_5P_3O_{10}$, is formed. $Na_5P_3O_{10}$ in solution is slowly hydrolyzed to $H_2PO_4^-$ and HPO_4^{--} ions.

h. *The Carbonate Compounds*. Two salts, *sodium carbonate*, Na_2CO_3, and *sodium hydrogen carbonate*, $NaHCO_3$, are formed by the action of carbon dioxide upon sodium hydroxide. $NaHCO_4$ crystals are readily formed by passing CO_2 into concentrated NaOH and they may be filtered off and dried. Na_2CO_3 can be obtained by heating $NaHCO_3$ at temperatures above 200°. Na_2CO_3 will decompose at temperatures above 800°. The common hydrated form of this salt is the decahydrate, $Na_2CO_3 \cdot 10H_2O$. Solutions of Na_2CO_3 and $NaHCO_3$ are alkaline because of the basic action of the CO_3^{--} ions and the slight hydrolysis of the HCO_3^- ions.

i. *Sodium Cyanide*. NaCN, sodium cyanide, is produced by reacting dry ammonia with a molten sodium-charcoal mixture. It can also be produced by fusing calcium cyanamide and carbon with sodium carbonate or sodium chloride or by heating sodium carbonate, coal, and nitrogen with iron as a catalyst. NaCN is a source of hydrocyanic acid. *Sodium thiocyanate*, NaSCN, can be produced by reacting NaCN either with sulfur, ammonium, polysulfide, or sodium thiosulfate.

j. *Other Sodium Compounds*. *Sodium permanganate*, Na_2MnO_4, can be prepared by reacting manganese dioxide with sodium hydroxide. It is less pure than potassium permanganate, K_2MnO_4, and is very hygroscopic. The heating of sodium hydroxide and chromium oxide in air produces *sodium chromate*, Na_2CrO_4. The chromate salt can be extracted from a heated mixture by a water leach. When the solution is heated with acid and then evaporated, *sodium dichromate*, $Na_2Cr_2O_7 \cdot 2H_2O$, will crystallize out of the solution. When $Na_2Cr_2O_7$ is heated, it first melts and then decomposes into Na_2CrO_4, Cr_2O_3, and free oxygen.

When a solution containing sodium ions is heated with a potassium salt of antimony, a slightly soluble salt, sodium orthoantimonate, Na $Sb(OH)_6$ is produced. Sodium fluosilicate, Na_2SiF_6, is formed in the action of fluosilicic acid, H_2SiF_6, upon a solution of sodium hydroxide. It is also insoluble in water. The addition of a large volume of a saturated solution of uranyl zinc acetate to a small volume containing sodium ions results in the formation of the triple acetate compound, sodium zinc uranylacetate, $NaC_2H_3O_2 \cdot Zn(C_2H_3O_2)_2 \cdot 3UO_2(C_2H_3O_2)_2 \cdot 6H_2O$, results. This compound is a light-yellow crystalline solid that is slightly soluble in water.

If a solution of borax is treated with Na_2O_2 or NaOH and H_2O_2, a precipitate of sodium peroxyborate, $NaBO_2 \cdot 3H_2O \cdot H_2O_2$. It decomposes very slowly in water. It is an active oxidizing agent. Sodium tetraborate, $Na_2B_4O_7 \cdot 10H_2O$, can be extracted from natural sources in the forms of prisms. If it is heated with sodium hydroxide, it forms sodium metaborate, $NaBO_2 \cdot 4H_2O$. Borax is a salt of a weak acid. It can be hydrolyzed and its aqueous solution gives an alkaline solution.

Sodium phenoxide, C_6H_5ONa, is formed in the reaction of sodium hydroxide and carbolic acid. Many other organic compounds of sodium exist.

B. The Analytical Chemistry of Sodium

Sodium, after it has been isolated and separated from other elements, is most frequently determined gravimetrically as sodium chloride, NaCl, sodium sulfate, Na_2SO_4 (3,4) or sodium zinc uranyl acetate, $NaZn(UO_2)_3(C_2H_3O_2)_9 \cdot 6H_2O$. (3) Sodium meta-antimonate, $NaSbO_3$; (5) sodium perchlorate, $NaClO_4$; (6) sodium magnesium uranyl acetate, $Na_2U_2O_7 \cdot MgU_2O_7$; (7) or bismuth cesium sodium nitrite, $Cs_9Na_6\ Bi(NO_2)_{6\ 5}$ (8,9) are also considered to be suitable gravimetric forms for a sodium determination. (6)

Precipitation methods are used most frequently to separate sodium from other elements. However, solvent extraction and chromatographic methods are now being studied extensively as separation methods for sodium. The following generally describes how these methods are being applied in the analytical chemistry of sodium.

1. Separations by Precipitation

The methods first suggested by Berzelius(10) and Smith(11) have wide use in separating sodium and the other alkali metals from the other chemical elements. The Berzelius method(9) and its modifications(12-14) uses mixtures of HF-$HClO_4$-H_2SO_4 to decompose the alkali-bearing material (usually a mineral) and then converts the alkali sulfates to chlorides for the final separation of sodium. Smith's method(11) and its modifications(15,16) has also been used to decompose minerals. The mineral is heated in a mixture of calcium carbonate and ammonium chloride. CO_2 and NH_3 are given off, and the mixture reduces to lime and calcium chloride. This mixture converts most of the metals to low-solubility oxides and sinters the mass so that the alkali metals can be extracted as chlorides from the mass by water leaching. Some calcium is also extracted, but it can be removed from the alkali mixture by a precipitation with an ammonium carbonate-oxalate solution and filtering. The sodium is finally separated from the other alkali metals by more specific techniques.

Barium, strontium, magnesium, borates, and sulfates can interfere in the separation of sodium (and the other alkali metals) by either the Berzelius(10) or Smith method.(11) Barium and strontium (as well as calcium) can be removed by an ammonium carbonate and oxalate precipitation.(17) Magnesium can be separated either as magnesium ammonium phosphate(18) or oxinate.(19) Evaporating the chloride mixture to dryness in the presence of methyl alcohol will remove borates as volatile methyl borate compounds.(17) The sulfate ions can be removed by a precipitation as $BaSO_4$ by adding barium chloride solution to the chloride mixture;(17) the excess barium can then be removed by a precipitation with ammonium carbonate.(17) Phosphates can be removed by a precipitation either with zinc carbonate(20) or an $FeCl_3$ solution at a controlled pH.(17)

Several other precipitation methods can be used to separate sodium from the other alkali metals and other ions. Potassium may be separated from sodium (and lithium) by forming the alkali chloroplatinates(21-24) or the perchlorates.(25-29) In the chloroplatinate method, the insoluble K_2PtCl_6 can be separated from the soluble sodium and lithium chloroplatinates and the precipitating agent, $H_2P_tCl_6$, by extracting the chloroplatinate mixture with 80% ethyl alcohol. Rubidium and cesium also form insoluble chloroplainates and can be separated from sodium in the same way.(30-33)

Sodium (and lithium) perchlorates are soluble in either butyl alcohol-ethyl acetate or ethyl alcohol-0.2% $HClO_4$ and can be separated from the insoluble perchlorate salts of K^{+1}, Cs^{+1}, Rb^{+1}, NH_4^{+1}, and Tl^{+1} by extracting the perchlorate precipitate with mixtures of these agents.(25-29) Sulfate ions must be absent, because sodium sulfate is also insoluble in the solvents used in the extraction method. However, magnesium, the alkaline earths, nitrates, and phosphates do not seriously interfere in the perchlorate method.

The precipitation of sodium as the triple acetate, $NaZn(UO_2)_3(C_2H_3O_2)_9 \cdot 6H_2O$, also serves as a means of separating sodium from other elements.(3,34,35) Lithium also forms the triple acetate salt and must be separated before the precipitation is made.(36) Potassium will interfere only if large amounts of sulfate ions are present.(36) Phosphates,(37-40) molybdates,(41) arsenates,(38,39) oxalates, (38,39) tartrates,(39) and strontium(42) are interfering substances in this method.

Potassium (and NH_4^{+1}) forms an insoluble tetraphenylboron compound when sodium tetraphenylboron solution is added to a mixture of the alkali chlorides and can be separated from sodium in this manner.(43-49) Lithium and magnesium are also soluble and can interfere in a sodium separation by this precipitation method.

Sodium can be separated from lithium following the perchlorate separation of potassium by saturating the n-butanol solution used to extract the alkali perchlorate mixture with hydrochloric acid.(28,50) Lithium can also be separated from sodium by extracting a chloride mixture with acetone.(19) Sodium chloride is insoluble under these conditions. However, barium, strontium, and potassium chlorides are also insoluble and would interfere in this method. Gaseous hydrochloric acid has also been used in the precipitating of sodium chloride.(17)

Rubidium and cesium can be separated from sodium with 9-phosphomolybdic acid.(3,23,51) Sodium can also be separated from cesium by precipitating cesium with silicotungstic acid solution.(31,51) Fluoboric acid has been used to precipitate potassium chloride as potassium fluoborate and separate it from the chlorides of sodium and lithium.(52) Magnesium, copper, zinc, cadmium, cobalt, nickel, manganese, iron, aluminum, chromium, and calcium also remain in solution. Ammonium, barium, and sulfate ions also separate with potassium.

2. Separation By Electrolysis. The alkali metals cannot be separated from solution by electrodeposition; however, it is possible to separate them from other elements by electrolyzing an acid solution with a mercury cathode at a controlled potential.(53) The alkali metals and the alkaline earth elements can be left in solution, while elements such as copper, cadmium, iron, cobalt, nickel, and zinc are deposited on the cathode. The alkali metals can then be separated from the alkaline earths and from each other by one of the techniques similar to those described in this section.

3. Solvent Extractions Separations. Nitromethane has been used to extract the polyiodides of the alkali metals from an acid solution.(54) The distribution ratios are dependent upon the amount of free iodine added to the system and increase in the order $Li < Na < K < Rb < Cs$. The extractions of alkali chloroplatinates with 80% ethyl alcohol to separate sodium and lithium chloroplatinates from the insoluble potassium (and Rb and Cs) chloroplatinates have already been cited.(21-24,30-33) Butyl alcohol - ethyl acetate or ethyl alcohol - 0.2% $HClO_4$ mixtures have been used in separating the alkali perchlorates.(25-29) Additional information on the extraction of the chloroplatinate and perchlorate mixture is given elsewhere.(55,56)

4. Chromatographic Separations

a. With Organic Absorbents. Erlenmeyer and Schoenauer(57) have used chromatographic columns of violuric acid, mixed with barium sulfate, diatomaceous earth or starch, to separate potassium chloride from sodium and magnesium chlorides and ammonia. Sodium has also been separated from potassium by use of a violuric acid column(58) and by use of a column of 5-oxo-5-oximino-3-phenylisoxazoline.(59)

b. With Ion Exchange Resins. Cohn and Kohn(60) have separated chloride mixtures of Na, K, Rb and Cs by use of a Dowex-50 column and 0.15 M HCl as the eluant. Beukenkamp and Rieman(64) also used Dowex-50 and 0.70 M hydrochloric acid to effect a similar separation. Brooksbank and Leddicotte(62) and Kayas(63) also have more complete separations of Na, K, Rb and Cs on Amberlite IR-100 resin. Na and K were eluted with 0.1 M HCl, and Rb and Cs with 1 M HCl. Bouchez and Kayar(64) have also used Amberlite-IR-100 and elutriants of either 0.1 M HCl or 0.1 N $HClO_4$ to separate radioactive Na^{22} and Mg. Kayas(65) has also separated milligram amount of NaCl and KCl on a column of Amberlite-IR-100. Sodium (and potassium) have been separated from the other constituents of silicate rocks by dissolving the sample in HF and H_2SO_4 and expelling the fluorine from the mixture and then absorbing the solution on an Amberlite IR-120 column.(66) The alkali metals were eluted from the column with 0.12 N HCl. Potassium and sodium have been separated from each other by use of a column of Wofatit KS ion exchange resin and 0.1 N HCl as the elutriant.(67) Cation exchange resins have also been used to separate K and Na from each other and from Mg and Cs in milk ash,(68) K from Na in blood,(69) alkali metals from insoluble silicates,(70) and K and Na from lithium.(71) Sodium in urine has also been separated by use of a cation exchange resin.(72,73) Both cation and anion exchange resins have been used to determine sodium and the other alkali metals in potassium niobium fluoride(74). Buser(75) reports that the alkali metal complexes of uramildiacetic acid can be separated by use of a cationic resin. Sodium and lithium form stable complexes but potassium does not. The complexes were separated by use of a sulfonic acid resin, pretreated with either dimethylamine or tetramethylammonium hydroxide, and elutions with alkaline solutions. Lithium and sodium, but not potassium, rubidium, or cesium, form complexes with the chelating agent, ethylenediaminetetraacetic acid, EDTA, and can be separated from K, Rb, and Cs, and from each other by eluting an anion resin column of Dowex-1 (4% crosslinkage) with 0.13 M EDTA solution.(76)

c. By Paper Chromatography. Radioactive Na^{24}(15 h) has been separated from radioactive K^{42} (12.4 h) by use of paper chromatography and a solvent mixture of conc. HCl-water-0.05% tartaric acid (1:10:4).(77) In other chromatographic studies with paper columns, the movement rate of sodium (and K, Rb, and Cs) adsorbed on a paper chromatogram has been investigated using solvent systems containing complex-forming mixtures, e.g., butanol-HNO_3-acetylacetone,(78) alcohols in HCl,(79,80) butanol in HCl,(80) alcohols in HNO_3,(81) ketones in HCl,(82) phenol,(81) or collidine in HNO_3.(83) Although these systems showed no appreciable individual alkali ion movement, they can be used to separate the alkalis from many other cationic species.

Sodium and lithium chlorides have been quantitatively separated from KCl using methanol as a solvent,(84,85) ethanol in water,(85) acetone in water,(85) methyl ethyl ketone in methanol,(86) and with acetone-, ethanol-, butanol-, and pentanol-methanol mixtures.(86,87) Ethanol-2N acetic acid as a solvent can also be used to separate alkali acetates by paper chromatography.(88,89) Sodium (and Mg and Ca) has also been separated from potassium in biological samples by the same solvent mixture.(90-93) Sodium has also been separated from K, NH_4, Mg, Ca, and Li by a paper chromatography method that used propanol-methanol mixtures as solvents.(94,95) These solvent mixtures have also been used to separate alkali hydroxides, citrates, and sulphates by paper chromatography(96,97).

Tracer quantities of Na^{22} (2.2 y) has been separated from gram amounts of magnesium by use of a combined precipitation - paper chromatography method.(98,99) Most of the magnesium is removed by heating the solution to form magnesium oxide and the soluble portion eluted from a chromatographic column with ethanol-water. In this solvent, magnesium travels ahead of sodium. Radioactive Na^{24} (15 h) has been separated from K^{42} by use of a solvent mixture of conc. HCl-water-0.05% tartaric acid (1:10:4)(77)

Electrochromatographic separations of the alkali metal cations with aqueous solvents, such as phenolpthalien, ammonium hydroxide, ammonium carbonate and hydrochloric acid with lactate, citrate, tartrate or EDTA buffers, have been reported.(100-104) Organic solvents, such as ammonium formate and trichloroacetic acid, have also been used in an electrochromatographic separation of the alkali metals.(105)

IV. DISSOLUTION OF SAMPLES CONTAINING SODIUM

Some rocks and minerals are soluble in HCl. If the rock or mineral is not acid soluble, then the material can be solubilized by either the Berzelius method(10) or the Smith method.(11) In Berzelius' method(10) the rock is attacked with HF and H_2SO_4, and the excess fluorine and silicon expelled by distillation. The alkali sulfates are then converted to chlorides. In the Smith method,(11) the powdered rock is heated in a mixture of ammonium chloride and calcium carbonate. Under these conditions, the alkali metals are converted to chlorides and can be extracted from the sinter by water.

Soil samples can be decomposed by treatments with ammonium acetate, HCl, and aqua regia,(106,107) and leachings with either HNO_3,(108) HCl-HF,(109) and H_2SO_4.(110) Biological materials, such as tissues, body fluids, vegetation, etc., can be decomposed with HCl, HNO_3, HNO_3-H_2SO_4, or HNO_3-$HClO_4$ mixtures.(106,107)

If any of these dissolution techniques are used in a radiochemical separation of the sodium radionuclides, the addition of inactive sodium carrier to the solubilizing mixture would greatly assist in achieving an isotopic exchange between the radioactive and inactive sodium atoms. The exchange should be rapid and complete, since sodium exists in only one oxidation state.

V. SAFETY PRACTICES

The decomposition and the processing of any sample material by chemical means can be hazardous. Thus, adequate safety precautions should be followed in processing any sample material. Pieters and Creyghton manual(111) on safe laboratory practices, as well as others on laboratory safety, should be consulted before any analysis is undertaken.

The introduction of a radioactive material into a laboratory area greatly magnifies the needs for safe laboratory practices. The discharge of radioactivity into a laboratory area by evolution or spillage, can result in hazardous conditions for personnel and wide spread contamination. Typical of the information about safe handling practices for radioactive materials appear in such sources as the Oak Ridge National Laboratory's Master Analytical Manual(112) and in the International Atomic Energy Agency's publication, entitled, "Safe-Handling of Radioisotopes."(113) Many other similar sources of information exists and should also be consulted.

VI. COUNTING TECHNIQUES FOR THE RADIOACTIVE SODIUM ISOTOPES

The nuclear characteristics of the radioactive isotopes of sodium are summarized in Table I of this monograph.

The radioactivity of either Na^{24} (15 h) or Na^{22} (2.6 y) can be measured by such counting techniques as Geiger-Mueller counting, proportional counting, or gamma scintillation spectrometry.(116-119) Generally, the sample material containing either of these sodium radionuclides must be processed radiochemically before the radioactivity measurements are made. However, it has been possible to determine small amounts of sodium in a variety of simple materials(118-123) by non-destructive radioactivation analysis methods(124,125) involving gamma scintillation spectrometry to measure the radioactivity of Na^{24}.

VII. RADIOCHEMICAL PROCEDURES FOR THE SODIUM RADIONUCLIDES

Both carrier-free and carrier radiochemical analysis procedures exist for the sodium 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.

Radiochemical procedures capable of giving Na^{22} or Na^{24} in carrier-free amounts exist. The ion exchange studies by Beukenkamp and Rieman(61), Brooksbank and Leddicotte(62), Kayas(63,65) and Bouchez and Kayas(64) will produce carrier-free solutions of the sodium radionuclides. Some of the other chromatography separation methods presented in Section III of the monograph can also be adapted for use in preparing carrier-free sodium radioactive tracers. The carrier radiochemical procedures that now exist for Na^{22} and Na^{24} have been originated to separate them as radioactive tracers,(126) or to separate them from fission products(127) and other radioactive species(128) or for use in radioactivation analysis.(124,125) By radioactivation analysis, trace amounts of sodium have been determined in aluminium(129-137), aluminium alloys,(138) asbestos,(139) biological materials,(140-148) bone,(149) water,(150,151) lead,(152,153) lithium,(154) magnesium,(155) minerals,(156) potassium nitrate,(157) potsherds,(158) refractories,(159) and uranium.(152) Radioactivation analysis has also been used to determine trace sodium in ground waters, reactor cooling water, plastics, soils, vegetation, graphite, lithium iodide, resins, zinc ore concentrates, sand, limestone, cement, trichlorosilane, gallium arsenide, alumina, hafnium oxide, stainless steel, and rubidium carbonate.(67,160-162)

The procedures that follow are typical of the radiochemical separation procedures used in analyzing a sample material either for Na^{22} or Na^{24}. Most of the applications cited above(126-162) have used either one of the specific procedures given in this section or have made an adaption to one of these procedures that would be suitable for the particular analysis problem. In each one of the procedures included in this section, information is given about the specific use of the procedure, the type of material analyzed, the type of nuclear bombardment, etc. Whenever possible, statements are made about the decontamination levels, and the chemical yields achieved by the particular procedure cited.

PROCEDURE 1

Procedure Used In: Preparation of Na^{24} tracer

Method: Precipitation

Element Separated: Na^{24} (15 h)

Type Material Bombarded: Aluminum alloy (95% Al; 2.25% Mg, 0.25% Cr plus Fe and Cu)

Type of Nuclear Bombardment: 184" cyclotron deuterons

Procedure By: D. B. Stewart (Reported by Meinke(126))

Separation Time: ~ 8 hours

Chemical Yield of Carrier: ~ 60%

Decontamination: 10^4 from Al; others unknown

Equipment Required: Standard

Procedure:

1. Dissolve aluminum in 6 N HCl. Add 50 mg Na and 10 - 20 mg Cu carriers.

2. Precipitate $Al(OH)_3$ with slight excess of NH_4OH. Centrifuge, pour off supernatant. Dissolve first precipitate in HCl and repeat, adding the 2nd supernatant to the original.

3. Saturate solution with H_2S to precipitate CuS and remaining Al as Al_2S_3. Filter or centrifuge.

PROCEDURE 1 (Continued)

4. Acidify filtrate with HCl or HNO_3 and evaporate to dryness. NH_4^+ salts can be quickly destroyed by boiling with a 1:4 HCl-HNO_3 mixture. Residue is mixture of NaCl and Na_2SO_4.

PROCEDURE 2

Procedure Used In: Separation of radioactive sodium from fission products.

Method: Precipitation

Element Separated: Na^{24}(15h) on Na^{22}(2.6y)

Type Material Analyzed: Fission product mixture

Probable Origin: $Na^{23}(n,\gamma)Na^{24}$ or $Na^{23}(n,2n)Na^{22}$

Procedure By: Prestwood (Reported by Kleinberg(127))

Separation Time: Several hours

Chemical Yield of Carrier: Quantitative

Decontamination: Radiochemically purity possible

Equipment Needed: Standard

Procedure:

1. Introduction

Radiosodium is separated from fission-product materials by specific precipitation as $Na\,Mg(UO_2)_3 \cdot (C_2H_3O_2)_9 \cdot 6\ 1/2\ H_2O$. The sodium is finally converted to and weighed as the chloride. The chemical yield is 50 to 60% and 2 to 3 hours are required for the analysis of a single sample. Excellent decontamination of radio sodium is obtained by this procedure. For example, a product having only 4 c/min was obtained from a solution containing 3×10^{14} fissions 10 days old and inactive sodium.

2. Reagents

Na Carrier: 10^{o} mg Na/ml (added as c.p. NaCl in H_2O) used as a primary standard

Fe Carrier: 10 mg Fe/ml (added as $FeCl_3 \cdot FeCl_3 \cdot 6H_2O$ in dilute HCl)

Cu Carrier: 10 mg Cu/ml (added as $Cu(NO_3)_2 \cdot 6H_2O$ in H_2O)

HCl: Conc.

PROCEDURE 2 (Continued)

$HClO_4$: 70%

NH_4OH: Conc.

$NH_4C_2H_3O_2$: 6 M

Na precipitating reagent (45 gm $UO_2(C_2H_3O_2)_2 \cdot 2H_2O$. Prepared as 300 gm $Mg(C_2H_3O_2)_2 \cdot 4H_2O$, 60 ml glacial $HC_2H_3O_2$ in 800 ml H_2O, diluted to 1 liter. Stir the mixture constantly for two hours, then permit it to stand for several hours, and filter through a 300-ml sintered glass funnel of medium porosity. (Large amounts of sodium are precipitated from the reagents)

Special wash solution. Prepared as 35 ml glacial $HC_2H_3O_2$, 405 ml of anhydrous acetate, 460 ml anhydrous ethanol.

n-butanol saturated with dry HCl: Prepared by bubble dry HCl into n-butanol until the gas is not longer adsorbed. The process takes several hours and much heat is evolved.

n-butanol: anhydrous

3. Equipment

Note: All of the equipment requirements for this analysis are mentioned in the "Procedure."

4. Procedure

Step 1. To the sample in a 40 ml conical centrifuge tube, add 1.0 ml of NaCl carrier, 2 drops of Fe carrier, and adjust the volume to 15 ml. Add conc. NH_4OH dropwise until the solution is just basic and then add three drops in excess. Centrifuge, transfer the supernate to a 125- ml Erlenmeyer flask, and discard the precipitate.

Step 2. To the supernate add 1 ml of conc. HCl and with constant swirling evaporate nearly to dryness over a Fisher burner. Cool. Add 1 ml of 6 M NH_4OAc and 100 ml of Na precipitating reagent (see reagents). Stir vigorously for 20 minutes using a magnetic stirrer. Centrifuge portionwise using a 40-ml conical centrifuge tube and discard the supernate after each centrifugation. Wash the precipitate with 20 ml of special wash solution (see reagents).

Step 3. To the washed precipitate add 20 ml of n-butanol, suspending the precipitate in the alcohol. Add 2 ml of n-butanol which has been saturated with dry HCl (see reagents). Stir and add 6 ml more of n-butanol saturated with HCl. Centrifuge the NaCl precipitate and discard the supernates. Wash the precipitate with 20 ml of n-butanol plus 2 ml of n-butanol saturated with HCl.

Step 4. Dissolve the NaCl precipitate in 15 ml of H_2O, add 2 drops of Fe carrier, make the solution just basic with conc. NH_4OH, and then add 3 drops in excess. Centrifuge, transfer the supernate to a 125 ml Erlenmeyer flask and discard the precipitate.

Step 5. Repeat Steps 2 and 3.

Step 6. Dissolve the NaCl precipitate in 18 ml of H_2O, add 2 drops of Fe carrier, and make the solution just basic with conc. NH_4OH. Centrifuge and transfer the supernate to a clean 40 ml centrifuge tube. Make the solution approximately 3 M in HCl, add 5 drops of copper carrier and saturate with H_2S. Centrifuge and transfer the supernate to a 50 ml Erlenmeyer flask, discarding the precipitate. Evaporate the solution to dryness over a Fisher burner and heat in a furnace at 600° for 15 minutes. Cool to room temperature.

Step 7. Add 1 ml of 70% $HClO_4$ and fume to dryness over a flame. (The presence of a small quantity of free $HClO_4$ will not interfere in the steps which follows.) Add 15 ml of n-butanol and bring the solution to boil to dissolve the $NaClO_4$. Transfer to a dry 40 ml conical centrifuge tube. Heat almost to boiling and add 8 ml (the first 2 dropwise) of n-butanol saturated with dry HCl. While the mixture is hot, filter through a tared No. 42 Whatman filter circle, 7/8" diameter, using a ground-off Hirsch funnel and a filter chimney. Wash the NaCl precipitate with approximately 20 ml of n-butanol. Dry at 110° for 10 minutes, cool, weigh, and mount (Note 1).

Notes:

1. The active Na isotope usually counted is either 15.1 h Na^{24} or 2.6 y Na^{22}.

PROCEDURE 3

Procedure Used In: Separation of Na^{24} from fission product mixture in sea water

Method: Precipitation and distillation

Element Separated: Na^{24}(15h)

Type Material Analyzed: Sea water

Probable Origin: $Na^{23}(n,\gamma)Na^{24}$

Procedure By: Love and Sam(128)

Separation Time: Short

Chemical Yield of Carrier: Quantitative

Equipment Needed: Apparatus described by author(128).

Procedure:

1. To a hot solution of 75 ml of sea water and 1 ml of lanthanum carrier (10 mg La^{+3}/ml) in a 50 ml centrifuge tube, add ammonium hydroxide until lanthanum hydroxide precipitates. Centrifuge; decant the supernatant liquid through a No. 42 Whatman filter paper into a clean centrifuge tube.

2. Cool the solution in an ice bath. Pass anhydrous hydrogen chloride into the solution by means of the anhydrous hydrogen chloride saturation apparatus (Note 1). Saturate the solution with the gas under pressure (Note 2) for 3 minutes after the sodium chloride precipitates out. Then centrifuge. Discard the supernatant liquid.

3. Wash the sodium chloride precipitate with 10 ml of hydrochloric acid wash solution (Note 3). Centrifuge; discard the wash.

4. Dissolve the NaCl in 10 ml of distilled water. Again precipitate sodium chloride with anhydrous hydrogen chloride. Wash the precipitate once with 5 ml of the hydrochloric acid wash solution. Then filter through a medium-pure sintered glass filter.

5. Wash the NaCl precipitate twice with 5 ml aliquots of ethyl alcohol saturated with hydrogen chloride gas, and twice with 5 ml aliquots of diethyl ether saturated with hydrogen chloride gas.

6. Transfer NaCl precipitate to a preweighed weighing bottle. Count Na^{24} gamma radioactivity (1.368-Mev) in a gamma spectrometer.

7. After counting, dry the NaCl at 105°C in an oven, and cool in a desiccator. Weigh to determine chemical yield. (Note 4)

Notes:

1. Saturation apparatus described by authors.(128)

2. At a pressure of 5 psi.

3. Made by saturating concentrated hydrochloric acid with hydrogen chloride gas.

4. If necessary determine the initial amount of sodium in each sample by flame photometry. However, in almost all cases, the amount of sodium in sea water is a known constant.

PROCEDURE 4

Procedure Used In: Radioactivation analysis

Method: Ion Exchange (carrier-free separation)

Element Separated: Na^{24}(15 h)

Type Material Analyzed: Alkali carbonates and chlorides(62,160-162)

Type Nuclear Bombardment: $Na^{23}(n,\gamma)Na^{24}$

Procedure By: Brooksbank and Leddicotte(62) (Reported in detail by Leddicotte(163))

Separation Time: Several hours

Chemical Yield of Carrier: A carrier-free separation

Decontamination: Excellent from radionuclides of K, Rb, and Cs.

Equipment Required: Ion exchange columns

Procedure:

A. Irradiation of Sample Material

1. Irradiate known amounts of test (Note 1) and comparator samples (Note 2) in a neutron flux of at least 6.5 x 10^{11} n/sec/cm^2 for 16 hours or longer (Note 3). Prepare the test and comparator samples for the irradiation either by wrapping each specimen in aluminum foil or placing it in a quartz ampoule. If the sample is a liquid, small polyethylene bottles can be used to contain the sample during the irradiation (Note 4).

B. Preparation of Irradiated Materials for Analysis

I. The Comparator Sample

1. After the irradiation, quantitatively transfer the comparator sample (Note 2) to a 100-ml volumetric flask. Dissolve the sample in a small measured volume of distilled water; then dilute the solution to 100 ml with water. Mix the solution thoroughly by shaking it carefully.

2. By means of a 1-ml volumetric pipet, transfer an aliquot to a second 100-ml volumetric flask; then dilute the aliquot to 100 ml with water.

3. Shake the solution thoroughly; then pipet an aliquot of this solution directly to the top of an ion-exchange column (Notes 5 and 6). Then proceed in the manner described below in Step 1 of Part C below.

II. Solid Test Samples

1. If the sample is a solid, quantitatively transfer the test portion into a 50-ml glass centrifuge tube, and then add dropwise to the same centrifuge tube enough concentrated HCl to completely dissolve the sample. If necessary, heat the mixture to dissolve the sample. Transfer the solution of the irradiated sample to the top of an ion-exchange column (Notes 5 and 6). Then continue with Step 1 of Part C below.

III. Liquid Test Samples

1. Pipet an aliquot of the irradiated test portion directly on to the top of an ion-exchange column (Notes 5 and 6); then continue with Step 1 on Part C below.

C. Ion Exchange Separation of Potassium

1. Connect the ion-exchange column to a reservoir (Note 7) that contains a supply of 0.1 M HCl. Adjust the flow rate so that the eluate flows from the column at the rate of 1 ml per minute.

2. Continue the flow of the 0.1 M HCl through the column and collect and discard the first 400 ml of eluate that passes through the column (Note 8).

3. Collect the next 500 ml of eluate (Note 9). Process this fraction of the eluate as instructed in Step 1 of Part D below.

4. If potassium, rubidium and cesium are not to be determined, stop the flow of eluate from the column after this collection (Note 10).

D. Preparation of Na^{24} Eluate for Radioactivity Assay

1. Evaporate to dryness the solution that was collected in Step 3 of Part C above. Add 1 to 2 ml of conc. HCl to the beaker, and then wash the solution into a 10-ml volumetric flask. Rinse the beaker with small portions of H_2O, transfer the rinsings to the flask, and dilute the solution to volume. Assay the solution for Na^{24} radioactivity as instructed in Part E below.

E. Measurement of Na^{24} Radioactivity and Calculation of Stable Sodium Content of Test Sample

1. The Na^{24} radioactivity in both the test and comparator samples may be assayed by beta or gamma counting. Use a Geiger-Mueller counter for the beta measurements and a gamma scintillation counter for the gamma measurements (Note 11).

2. Following the radioactivity measurements, correct the observed Na^{24} radioactivity for decay (Note 12), dilution volume(s), and the sample weights (or volumes) of both the test and comparator samples. A simple ratio of these corrected radioactivities becomes a measurement of the amount of stable sodium in the test sample:

$$\%\ \text{Na in Test Sample} = \frac{\text{Corrected } Na^{24} \text{ radioactivity in Test Sample}}{\text{Corrected } Na^{24} \text{ radioactivity in Comparator Sample}} \times 100$$

Notes:

1. Use at least 0.1-0.2 gram portions.

2. Use from 0.025 to 0.030 gram of sodium carbonate.

3. The limits of measurement for this procedure is about 0.005 micrograms of sodium.

4. This type of sample will have to be irradiated in an air-cooled or water-cooled facility of the reactor.

5. The ion exchange column is composed of 100- to 120-mesh IR-1 (or IR-100) resin packed into a glass column 1 cm in diameter and 100 cm long. A small glass wool plug at the base of the column holds the resin in the column. The resin is pretreated with a solution of 0.1 M HCl. The

treatment consists of passing the 0.1 M HCl solution over the column until the desired molarity (0.1 M) is obtained.

6. The transfer may be made by use of a volumetric pipet. If it is necessary to rinse the pipet, use 0.1 M HCl as the rinse liquid. In transferring the radioactive solution on to the column, exercise care in delivering the solution from the pipet so that the resin at the top of the column will not be disturbed.

7. A dispensing bottle of at least 3-liter capacity is suitable for use as a reservoir. Tygon or rubber tubing may be used to connect the reservoir to the column.

8. This solution may contain radioactive anionic constituents. The presence of these should not be confused with Na^{24} which is eluted later.

9. This particular procedure is designed to separate Na, K, Rb, and Cs in alkali carbonates and chlorides. This fraction will contain all of the Na^{24} (15 h).

10. If K, Rb and Cs are to be determined, continue as follows: (a) Collect the next 700 ml of eluate and process for K; (b) collect the next 800 ml and process for Rb; then, (c) strip the column with at least 1 N HCl and process this fraction for Cs. Potassium, rubidium and cesium comparator samples should have been irradiated, if stable K, Rb and Cs are to be determined in a sample.

11. Gamma-ray energy discrimination may be used here. Na^{24}(15 h) emits 1.368- and 2.75- Mev gamma radiations in its decay.

12. Decay measurements may be followed, if required.

PROCEDURE 5

Procedure Used In: Radioactivation analysis

Method: Precipitation

Element Separated: Na^{24} (15 h)

Type Material Bombarded: Water

Type of Nuclear Bombardment: $Na^{23}(n,\gamma)Na^{24}$

Procedure By: Blanchard, Leddicotte, and Moeller(150,151)

Separation Time: Several hours

Chemical Yield of Carrier: 60-70%

Equipment Needed: Standard

Procedure:

1. 30 mls of water was placed in a polyethylene bottle and irradiated for 16 hours in a flux of 6.5×10^{11} n/cm^2/sec.

2. If any specific element, such as sodium is to be analyzed quantitatively, a known amount of Na_2CO_3 is irradiated along with the water sample. The standard is processed in the same manner as the unknown sample.

3. The radiochemical separation is carried out as outlined in "The Chemical Separation Scheme" attached.

4. The Na^{24} is found in the last step (Part II of the "Scheme") with the Soluble Group along with potassium rubidium and cesium. Because Na^{24} decays with gamma radiations of 2.75-Mev (as well as 1.368-Mev), radiochemical separations need not always be made (Note 1).

5. Use a gamma scintillation spectrometer to measure the gamma radiations (either the 1.368- or the 2.75-Mev) being emitted by Na^{24}.

6. Following the radioactivity measurements, correct the observed Na^{24} radioactivity for decay, dilution volume, sample and yield weights for both the sample and standard. A ration of these corrected radioactivities becomes a measurement of the amount of stable sodium in the water sample.

$$\%\ \text{Na in } H_2O \text{ Sample} = \frac{\text{Corrected } Na^{24} \text{ radioactivity in } H_2O \text{ sample}}{\text{Corrected } Na^{24} \text{ radioactivity in standard sample}} \times 100$$

PROCEDURE 5 (Continued)

Notes:

1. However, a further separation of the alkali metals may be made by evaporating the solution to dryness in the presence of perchloric acid and then extracting the perchlorate precipitate with a mixture of butyl alcohol and ethyl acetate. Sodium perchlorate is soluble; the insoluble potassium and rubidium perchlorates can be removed by filtration. The Na^{24} radioactivity in the sodium perchlorate solution may be measured directly or collected as NaCl by treating the solution with anhydrous hydrochloric acid.

Chemical Separation Scheme

1) Add the radioactive sample to a 50 milliliter centrifuge tube. Aerate to remove radioactive gases. Acidify with HNO_3 and add Cl, Br, I, Cu, As, Fe, Y, Co, Mn, Zn, Ba, Sr, Ca, Na, and K carriers. Precipitate Cl, Br, and I with slight excess of Ag. Heat to coagulate the precipitate. Centrifuge.

2) Precipitate: AgCl, AgBr, AgI. Wash with water, filter, mount and count. Designate as the SILVER GROUP.

3) Supernate: Adjust acidity to 0.5 N HCl. Saturate with H_2S. Centrifuge.

4) Precipitate: $CuS + As_2S_3$. Wash with water, filter, mount and count. Designate as the ACID SULFIDE GROUP.

5) Supernate: Transfer to beaker. Add 1 ml conc. HCl. Boil until solution is clear. Oxidize Fe^{++} to Fe^{+++} by boiling with HNO_3. Cool solution and make 1:1 with conc. HCl. Neutralize with conc. NH_4OH to precipitate hydroxide. Centrifuge.

6) Precipitate: Fe + Y hydroxides. Wash with water, filter, mount and count. Designate as the HYDROXIDE GROUP.

7) Supernate: Saturate with H_2S. Centrifuge

8) Precipitate: CoS, MnS, ZnS. Wash with water, filter, mount and count. Designate as the BASIC SULFIDE GROUP.

9) Supernate: Acidify solution with HCl. Boil to remove H_2S. Add $(NH_4)_2H(PO_4)$ and an excess of NH_4OH. Digest. Centrifuge.

10) Precipitate: Ca, Ba, and Sr phosphates. Wash, filter, mount, and count. Designate as the ALKALINE GROUP.

11) Supernate: Soluble ions. Designate as the SOLUBLE GROUP.

PROCEDURE 6

Procedure Used In: Radioactivation analysis

Method: Precipitation

Element Separated: Na^{24} (15 h)

Type Material Bombarded: Aluminum(138)

Type Nuclear Bombardment: $Na^{23}(n,\gamma)Na^{24}$

Procedure By: Plumb and Silverman(138)

Separation Time: 3 hours

Chemical Yield: 75%

Equipment Needed: Standard

Procedure:

1. The aluminum sample along with Na_2CO_3 and $NaHCO_3$ standards were irradiated in positions 14-Z-19 through 14-Z-21 of the Graphite reactor at Oak Ridge National Laboratory (Note 1), for 1 week.

2. After irradiation, the sample was dissolved in HCl and H_2O_2 in the presence of 5 mg of sodium carrier (as NaCl).

3. The Na is precipitated as sodium zinc uranyl acetate. The precipitate is filtered, then redissolved in hot water.

4. The solution is made acid with HCl, copper holdback added, and CuS precipitated with H_2S.

5. The CuS is discarded and Na is again precipitated with Zinc uranyl acetate and is used for counting measurements using an end-window Geiger tube (Note 2).

Notes:

1. The authors used this facility since it is the most thermalized facility in the reactor, in order to get away from the fast neutron reaction of $Al^{27}(n,\alpha)Na^{24}$.

2. With the development of the gamma scintillation spectrometer, it is no longer necessary to make a chemical separation of sodium. It can be measured directly by using the 2.7 Mev energy of Na^{24}.

PROCEDURE 7

Procedure Used In: Radioactivation analysis

Method: Direct (separation sometimes required)

Element Separated: Na^{24} (15.0 h) and others

Type Material Bombarded: Normal biological tissues (male)

Type Nuclear Bombardment: $Na^{23}(n,\gamma)Na^{24}$

Procedure By: Hall$^{(140)}$

Separation Time: 3 hours

Equipment Needed: Two 2 x 1 1/8 in. crystals and a coincidence analyzer modified for two a-d channels

Procedure:

1. The biological material to be analyzed was placed in a nuclear reactor for irradiation, along with pure samples of its constituents.

2. After irradiation, the products are measured by simultaneous pulse-height and coincidence analysis, using two 2 x 1 5/8 in. crystals and a coincidence analyzer modified for two a-c channels.

3. The formulation for coincidence analysis is:

$$S_m = K_a S^1{}_a + K_b S^1{}_b \ . \ . \ . + S_B$$

$$C_B = C^1{}_B + S_B 2t$$

$$C_a = C^1{}_a + C^1{}_B + S_a 2t$$

$$C_m = kaC^1{}_a + k_b C^1{}_b + \ . \ . \ . + C^1{}_b + S_m 2t$$

where a, b, c . . . are constituents of the mixture M_1Sa = (geometrical) average counting rate in one crystal (no coincidence) for the vial containing pure element $a_1 S^1 a = S_a - S_B$ with B denoting background; C = coincidence rate; C^1 = single-event coincidence rate; t = coincidence interval; and k_a = ratio of a in the mixture to a in the pure standard.

According to the author, it is possible to measure Na, K, Fe, Co, Zn, and Cu in normal biological material and Sb, Ag, Br, Cs, Hg, and As when present at 10^{-6} gm/cm^3. Radiation time: 30 days.

PROCEDURE 8

Procedure Used In: Radioactivation analysis

Method: Precipitation

Element Separated: Na^{24} (15 h)

Type Material Bombarded: Magnesium

Type Nuclear Bombardment: $Na^{23}(n,\gamma)Na^{24}$

Procedure By: Atchison and Beamer(155)

Separation Time: Several hours

Chemical Yield of Carrier: Quantitative

Equipment Needed: Standard

Procedure:

A. Irradiation of Sample Material

1. Magnesium turnings were irradiated for 4 weeks at the Oak Ridge National Laboratory in a flux of 5×10^{11} n/sec/cm^2 along with a weighed portion of Na_2CO_3 for a standard (Note 1).

B. Radiochemical Separation of Na^{24}

1. After the irradiation, dissolve a weighed portion of the Mg sample in 16 ml of 12 M HCl in the presence of 60 mg of K and 80 mg of Na carrier and 10 ml of water in a 250 ml beaker.

2. Evaporate the contents to a small volume to remove excess acid.

3. Transfer the solution to a 100-ml mixing cylinder and dilute to approximately 75 ml and add 13 ml of n-butylamine. Dilute to 100 ml and mix well.

4. Centrifuge the slurry and decant the clear liquid through a dry filter paper. Place a 60-ml aliquot in a 150-ml beaker and evaporate to dryness on a hot plate. Sublime the amine hydrochloride away using a Bunsen flame while passing a stream of nitrogen into the beaker.

5. After the beaker cools, add 2 ml of 16 M HNO_3 and evaporate to dryness. Repeat treatment until all organic matter is destroyed. Four or five evaporations are usually required.

6. Add 5 ml of 70% $HClO_4$ and evaporate to dense fumes. Separate the K perchlorate by treatment with ethyl acetate (Note 2). Filter; discard the precipitate.

7. Add n-butyl alcohol; evaporate to expel the ethyl acetate. Precipitate the sodium as NaCl by adding a solution of HCl gas in n-butyl alcohol (Note 3).

8. Collect the precipitate and dry at 110° C for 10 minutes and ignite at 600° C for 5 minutes. Weigh as NaCl to determine the chemical yield.

9. The Na_2CO_3 comparator is carried through the same procedure as the Mg sample.

C. Measurement of Na^{24} Radioactivity

1. The activity of the Na^{24} may be measured by beta or gamma counting. Use a Geiger-Mueller counter for beta measurements and a gamma scintillation counter for the gamma measurements.

2. Following the radioactivity measurements, correct the Na^{24} activity for decay, dilution volume, and sample and yield weights for both the magnesium and standard samples. A ratio of these corrected radioactivities becomes a measurement of the stable sodium in the sample:

$$\%\text{ Na in Sample} = \frac{\text{Corrected Na}^{24}\text{ in Mg sample}}{\text{Corrected Na}^{24}\text{ in standard sample}} \times 100$$

Notes:

1. A 24-hour irradiation would be sufficient if the analyst can obtain the sample within a day after irradiation.

2. Page 8 in "Applied Inorganic Analysis" by W. F. Hillebrand and G. E. F. Lundell - 1929.

3. The analyst must remember that Na^{24} can also be formed by the fast neutron reaction, $Mg^{24}(n,p)Na^{24}$. The authors found that a Mg sample with 10 ppm of stable Na present analyzed 123 and 116 ppm by activation.

PROCEDURE 9

Procedure Used In: Radioactivation analysis

Method: Precipitation

Element Separated: Na^{24} (15 h)

Type Material Bombarded: Dunite mineral ore from Balsam Quarry, North Carolina

Type Nuclear Bombardment: $Na^{23}(n,\gamma)Na^{24}$

Procedure By: Salmon(156)

Separation Time: Not estimated by author

Chemical Yield of Carrier: Approximately 75%

Equipment Needed: Standard

Procedure:

1. Weigh approximately 50 mg of sample into a short length of polythene tubing and heat seal the ends. Similarly, weigh and seal 20 mg of Na_2CO_3 dried to 110° C as a standard. Irradiate sample and standard in BEPO (at Harwell) for about 3 hours.

2. After the irradiation, empty the sample into a platinum dish containing 5 ml of a solution of NaCl as carrier (10 mg of Na^+ per ml). Add a few drops of H_2SO_4 (SpG 1.84) and 10 ml of 40% HF. Evaporate gently to fumes of H_2SO_4, cool and add 10 ml of HF and again evaporate to fumes. Cool the residual solution and transfer it to a 250-ml beaker with a few milliliters of H_2O.

3. Add 10 ml of HNO_3 (SpG 1.32). Evaporate to fumes; cool to room temperature and transfer the solution with 2-3 mls of water to a 50-ml centrifuge tube.

4. At this step, take a suitable aliquot from the irradiated standard and to it add 5 ml of sodium carrier and continue with the separation for both sample and standard.

5. Add 10 mgs of Fe^{+3} or chloride, stir and make basic with NaOH. Centrifuge the $Fe(OH)_3$ and filter the supernate into another 50-ml centrifuge tube.

6. Just acidify the filtrate with glacial acetic acid and chill the mixture in an ice bath. Then add 25 ml of zinc uranyl acetate reagent (Note 1) with stirring and digest for 15 minutes. Allow the precipitate to settle,

then centrifuge. Discard the supernate. Wash the precipitate three times with ethanol.

7. To the sodium zinc uranyl acetate precipitate, add 15 ml of ethanol saturated with hydrogen chloride gas. Digest in an ice bath for 15 minutes. Centrifuge and wash the precipitate 3 times with ethanol.

8. Make a slurry with a small quantity of ethanol (Note 2) and transfer it to a weighed counting container, dry it under an infra-red lamp and weigh for chemical yield determination.

9. Determine the Na^{24} either by beta or gamma measurement. Amount of stable sodium in sample by:

$$\text{Wt of } Na^{24} \text{ in sample} = \frac{(\text{Wt. of Na in standard})\ (Na^{24} \text{ activity in sample})}{Na^{24} \text{ activity in standard}} \quad \text{(Note 2)}$$

Notes:

1. Prepared by dissolving 77 g of uranyl acetate in 410 ml of H_2O and 13.3 ml of glacial acetic acid. Warm to 70° C to aid dissolution. Also dissolve 231 grams of zinc acetate in 262 ml of H_2O and 6.6 ml of glacial acetic acid. Mix the two solutions and allow to stand for 24 hours before filtering the reagent to remove any precipitated solids.

2. NaCl may be dissolved in H_2O; scavenged with Fe^{+3} and ammonia and then reprecipitated as NaCl with ethanol-hydrogen chloride.

PROCEDURE 10

Procedure Used In: Radioactivation analysis

Method of Separation: Precipitation

Element Separated: Na^{24} (15 h)

Type of Material Bombarded: Water, reactor cooling water, plastics, aluminum, stainless steel, soils, vegetation, ore concentrates, clays, limestone, cement, tissue, resins, lithium carbonate, lithium iodide, alumina, etc.(160-162)

PROCEDURE 10 (Continued)

Type of Nuclear Bombardment: $Na^{23}(n,\gamma)Na^{24}$

Procedure By: Mullins and Leddicotte(164)

Chemical Yield of Carrier: At least 70%

Time of Separation: 2 hours

Degree of Purification: Greater than 10^5 from all radionuclides

Equipment Needed: Standard

Procedure:

A. Irradiation of Sample Material

1. Irradiate known amounts of test (Note 1) and comparator (Note 2) samples in a neutron flux of at least 6.5×10^{11} n/cm^2/sec for 16 hours or longer (Note 3). Prepare the test and comparator sample for the irradiation either by wrapping each specimen in aluminum foil or placing it in quartz ampoule. Liquid samples should be irradiated in polyethylene bottles (Note 4).

B. Preparation of Irradiated Materials for Analysis

I. The Comparator Sample

1. After the irradiation, quantitatively transfer the comparator sample (Note 2) to a 100-ml volumetric flask. Dissolve the sample in a small, measured volume of 6 M HNO_3; then dilute the solution to 100 ml with water. Mix the solution well by carefully shaking it.

2. By means of a volumetric pipet, pipet a 1.00-ml aliquot of this solution into a 100-ml volumetric flask; then filute the aliquot to 100 ml with water.

3. Shake the solution thoroughly; then pipet a 1.00-ml aliquot of it into a 50-ml glass centrifuge tube. By means of a volumetric pipet, add to the same centrifuge tube, 2.00 ml of a standard carrier solution of known sodium concentration (Note 5). Also, add 1 ml each of holdback carriers of barium, cadmium, cobalt, copper, iron, manganese, phosphorus, potassium, and zinc (Note 6). Dilute the solution to 20 ml with water, mix it well, and make the solution 0.3 M in HCl. Continue with Part C below.

II. Solid Test Sample

1. 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 glass 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 sodium concentration (Note 5). Also add 1 ml each of holdback carrier of barium, cadmium, cobalt, copper, iron, manganese, phosphorus, potassium, and zinc (Note 6). To this mixture, add dropwise enough concentrated mineral acid to completely dissolve the sample (Note 7). Dilute the solution to 20 ml with water, mix it well, and make the solution 0.3 M in HCl. Continue with Part C below.

III. Liquid Test Samples

1. Pipet an aliquot of the irradiated portion into a 50-ml glass centrifuge tube. By means of a volumetric pipet, add to the same centrifuge tube 2.00 ml of a standard carrier solution of known sodium concentration (Note 5). Also add 1 ml each of holdback carriers of barium, cadmium, cobalt, copper, iron, manganese, phosphorus, potassium, and zinc (Note 6). Dilute the solution to 20 ml with H_2O, mix it well, and make the solution 0.3 M in HCl. Continue with Part C below.

IV. Organic Test Samples

1. If the sample is a tissue, vegetation, or a similar material, quantitatively transfer the irradiated test portion from the irradiation container to a 50-ml beaker. By means of a volumetric pipet, add to the same beaker 2.00 ml of a standard carrier solution of known sodium concentration (Note 5). Also add 1 ml each of holdback carriers of barium, cadmium, cobalt, copper, iron, manganese, phosphorus, potassium, and zinc (Note 6). Then add 3 ml of conc. H_2SO_4 and 4 ml of conc. HNO_3. Cover the beaker with a watch glass; then boil the solution for 15 minutes. Cool the solution, add to it 3 ml more of conc. HNO_3, and boil the solution for 15 minutes (or to fumes of SO_3). Repeat the addition of HNO_3 and the boiling until the sample is dissolved. Add a few ml of conc. HCl, evaporate to remove HNO_3. Repeat several times if necessary. Dilute solution to make it 0.3 M in HCl. Transfer to a 50-ml centrifuge tube; then continue with Part C below.

C. Radiochemical Separation of Na^{24}

1. Saturate the solution with H_2S. Centrifuge the mixture (Note 8). Transfer the supernatant liquid to a new 50-ml centrifuge tube. Wash the CuS precipitate with one 10-ml portion of hot water. Centrifuge and add the wash to the centrifuge tube. Discard the precipitate.

2. Add 3 ml of bromine water to the supernatant liquid-water wash mixture. Boil until all of the excess H_2S has been removed; then add conc. NH_4OH dropwise until no further precipitation of $Fe(OH)_3$ occurs. Centrifuge the mixture (Note 9). Filter the supernatant liquid into a 150-ml beaker and evaporate the solution to dryness. Remove the ammonium salts, NH_3^{1+}, by ignition or by evaporating in an acid solution (Note 10).

3. Dissolve the residue in the beaker in a few milliliters of distilled water and transfer the solution to a 50-ml centrifuge tube. Rinse beaker with additional water and transfer the rinses to the centrifuge tube.

4. Concentrate the solution volume to 1 or 2 ml by boiling. Cool; then add 10 ml of zinc uranyl acetate reagent to the tube for each ml of the concentrated solution. Stir the mixture thoroughly. Cool in a water bath at 20° (± 1°) C for 30 to 45 minutes. Stir occasionally. Centrifuge the mixture; discard the supernatant liquid.

5. Wash the precipitate twice with 5 ml volumes of the zinc uranyl acetate reagent. Centrifuge after each wash; discard the wash liquid. Wash the precipitate at least twice more with two 10-ml volumes of 95% ethyl alcohol and once with one 10-ml volume of ethyl ether. Centrifuge after each wash and discard the wash liquid.

6. Add 20 ml of 12% BuOH-HCl reagent to the precipitate. Stir the mixture thoroughly. Digest at 20° (± 1°) C for 5 minutes; then centrifuge, discard the supernatant liquid.

7. Wash the precipitate once with a mixture of 10 ml of ethyl ether and 10 ml of 12% BUOH-HCl reagent. Centrifuge; discard the supernatant liquid.

8. Filter off the NaCl through a tared filter paper (Munktelle No. 00)

that is held in a Hirsch funnel; wash the precipitate three times with 10-ml portions of ethyl ether. Dry the precipitate for 10 minutes in a drying oven at 110° C. Weigh the NaCl precipitate and filter paper on an analytical balance. Mount the precipitate and count its radioactivity as instructed in Part D below.

D. Measurement of Na^{24} Radioactivity and Calculation of Stable Sodium Content of Test Sample

1. The Na^{24} radioactivity in both the test and the comparator samples may be assayed by beta or gamma counting. Use a Geiger-Mueller counter for the beta measurements and a gamma scintillation counter for the gamma measurements (Note 11).

2. Following the radioactivity measurements, correct the observed Na^{24} radioactivity for decay (Note 12), dilution volume(s), and the sample weights (or volumes) of both the test and comparator samples. A simple ratio of these corrected radioactivities becomes a measurement of the amount of stable sodium in the test sample:

$$\%\ \text{Na in Test Sample} = \frac{\text{Corrected } Na^{24} \text{ radioactivity in test Sample}}{\text{Corrected } Na^{24} \text{ radioactivity in comparator sample}} \times 100$$

Notes:

1. Solid test samples should weigh from 0.10 - 0.20 gram; liquid samples should have a volume of from 5 - 25 milliliters.

2. Use 0.025 to 0.030 grams of spectrographically pure Na_2CO_3.

3. The limits of measurement for stable sodium by this procedure is 5×10^{-9} gram.

4. Irradiations of liquid, vegetation, or similar type sample materials must be made in an air-cooled or water-cooled reactor irradiation facility.

5. Standardized to contain at least 10 milligrams of sodium per milliliter.

6. Solutions of the ions of other elements may also be added as holdback carriers; concentration equal to 5 milligrams of element per milliliter.

7. Soils, clays, and similar materials may require addition of HNO_3 or fusion.

PROCEDURE 10 (Continued)

8. Additional amounts of Cu^{+2} ions may be added here for further scavenging of acid sulfide precipitible elements. Sufficient H_2S is present to cause CuS precipitation. Centrifugation is advisable after each addition of copper holdback carrier.

9. Additional amounts of Fe^{+3} ions may be added here to serve as a scavenger for other hydroxide precipitible elements. Sufficient NH_3 ions are present to cause $Fe(OH)_3$ precipitation. Centrifugation is adviseable after each addition of iron holdback carrier.

10. A 1 part conc. HCl to 4 parts conc. HNO_3 mixture can be used here. Add at least 5 milliliters of this mixture and heat to boiling.

11. Gamma-ray discrimination may be used here.

12. Decay measurements may be followed, if required.

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