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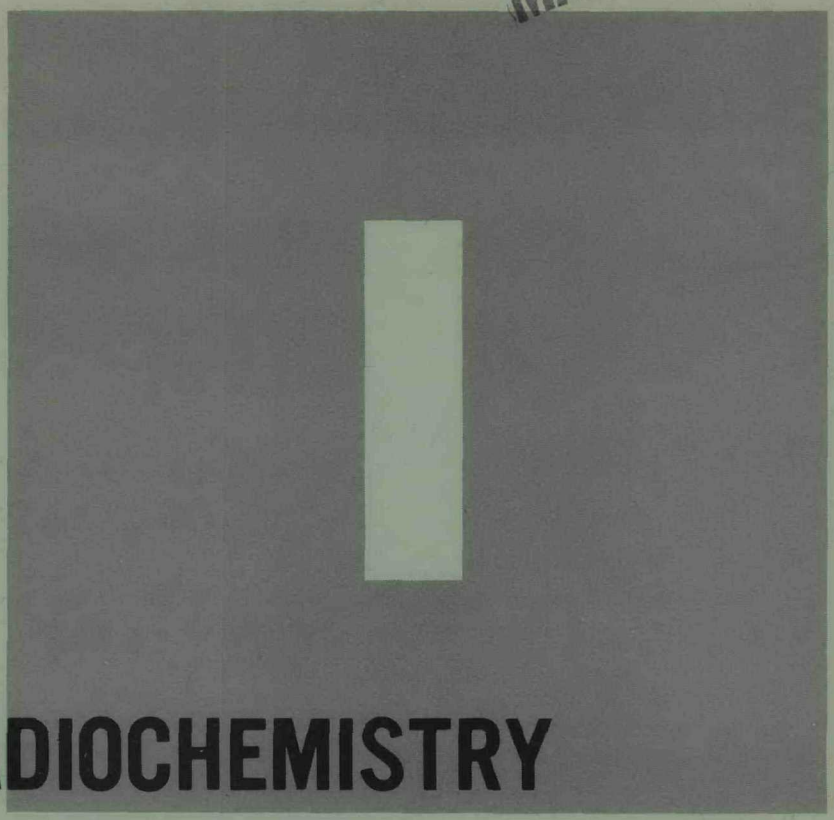

RADIOCHEMISTRY OF IODINE

NUCLEAR SCIENCE SERIES

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Radiochemistry of Iodine

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Foreword

The Subcommittee on Radiochemistry is one of a number of subcommittees working under the Committee on Nuclear Science of the 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 radiochemical purity of reagents, radiochemistry in environmental science and in nuclear medicine, and the role of radiochemistry in college and university programs.

This series of monographs has grown out of the need for compilations of radiochemical information, procedures, and techniques. The Subcommittee has endeavored to present a series that will be of maximum use to the working scientist. Each monograph presents pertinent information required for radiochemical work with an individual element or with a specialized technique.

Experts in the particular radiochemical technique have written the monographs. The Energy Research and Development Administration has sponsored the printing of the series.

The Subcommittee is confident these publications will be useful not only to radiochemists but also to research workers in other fields such as physics, biochemistry, or medicine who wish to use radiochemical techniques to solve specific problems.

G. Davis O'Kelley, *Chairman*
Subcommittee on Radiochemistry

Preface

Following an introductory section containing information on iodine of particular usefulness to the radiochemist, the monograph deals with the preparation of isotopes of the element, with selected procedures for its determination in or separation from various media, and finally with the separation of iodine species from each other. Each part of the introductory section is referenced separately from the remainder of the monograph. For the preparative and analytical sections there is an extensive, indexed Bibliography which was developed from the indexes of Volumes 19-30 inclusive (1965-1974) of Nuclear Science Abstracts (NSA). From these indexes the NSA abstracts of possible pertinent references were selected for examination and a choice was made of those references which were to be included in the Bibliography. The Bibliography has both primary and secondary references. The primary references, which are asterisked, were read in their entirety. The abstracts from NSA were the sole sources of information for the secondary references. Arbitrarily, any article published in a foreign language other than French or German was not used as a primary reference. The style of the references in the Bibliography is essentially that found in NSA. The monograph contains no subject index, but the Contents is sufficiently detailed that each important topic is listed with its page number.

Although the monograph does not cover hot atom chemistry, the kinetics of exchange reactions, decay schemes, or physiological applications, papers in these areas were examined as possible sources of useful preparative and analytical procedures.

No recipes are given for radiochemical procedures. Rather the essential chemical features of the procedures are summarized.

Two other points are worth noting. The term "iodine" is used when no specific oxidation state of the element is indicated, and items indexed under headings of "carrier-free" include both those where it was so stated explicitly and those where there was a strong indication of that condition.

The authors are deeply indebted to the members of Group CNC-11 of the Los Alamos Scientific Laboratory who provided assistance in the preparation of the manuscript. We are particulary grateful to James E. Sattizahn, Jr. for providing an atmosphere in which it was stimulating and pleasant to work, to Patrick M. Grant for the information from which the Table of Isotopes in Section I was constructed, and to William R. Daniels, Jeanne Hasty, and Petrita Oliver for the many hours they spent in collecting literature material. We thank Lynn Holmes and Etheleen Willow for an expert typing job and Kurt Wolfsberg for his assistance in seeing the manuscript through the final publishing steps.

Contents

Radiochemistry of Iodine

I. SOME GENERAL INFORMATION

Table of Isotopes

In this table are listed all the known isotopes of the element iodine. Column 1 gives the mass number of each isotope as a superscript. Column 2 gives atomic masses for the individual isotopes in atomic mass units (^{12}C scale). Half-lives are given in column 3. The symbols used in this column are y = years, d = days, h = hours, m = minutes, s = seconds. Column 4 lists information on modes of decay and radiations emitted and their energies. The numbers following the symbols are the measured energies of the radiations in millions of electron volts; for β^- and β^+ decays the energies listed are the maximum β^- particle kinetic energies for each transition. Also included in this column are the percent absolute (% A) and percent relative (% R) intensities of the gamma rays. The percent absolute intensity is defined as the γ-ray abundance or photons per 100 decays of the parent. The percent relative intensity is defined as the intensity of the γ-ray relative to the most intense γ-line of the nuclide.

Isotope	Isotopic Mass	Half-Life	Decay Modes, Radiations, Major Energies, Absolute and Relative Intensities.	Ref.
^{115}I	114.91738	1.3 m	β^+	1,2
^{116}I	115.91609	2.9 s	β^+ 6.5; γ 0.5402 (11.5% R), 0.6791 (100% R), 1.2193 (3% R)	1,3
^{117}I	116.91322	2.4 m	β^+ 3.5; γ 0.2744 (27% R), 0.3259 (100% R), 0.6831 (2.5% R)	1.4,5
^{118}I	117.91245	8.5 m	Metastable; EC; β^+ 4.9; γ 0.600 (100% R), 0.6052 (100% R), 0.614 (54% R)	1,4,6
		13.7 m	Ground state; EC; β^+ 5.5; γ 0.5448 (12.3% R), 0.6052 (95% R), 1.1499 (5.0% R), 1.2570 (3.8% R), 1.3384 12.4% R)	1,4,6
^{119}I	118.90984	19 m	EC; β^+ 2.4; γ 0.2575 (95% A), 0.3206 (2% A), 0.5570 (1.7% A), 0.6356 (2.7% A)	1,4,5
^{120}I	119.90971	53 m	Metastable; EC;β^+ 3.8; γ 0.5604 (100% A), 0.6011 (87% A), 0.6147 (67% A), 1.3459 (18.9% A)	1,4,7
		1.35 h	Ground state; EC; β^+ 4.6; γ 0.5604 (73% A), 0.6011 (5.8% A), 0.6411 (9.1% A), 1.5230 (11% A)	1,4,7
^{121}I	120.90744	2.12 h	EC; β^+ 1.1; γ 0.2125 (84.3% A), 0.3197 (1.05% A), 0.4750 (1.05% A), 0.5319 (6.16% A), 0.5987 (1.54% A)	1,8
^{122}I	121.90750	3.62 m	EC; β^+ 3.1, γ 0.5640 (17.7% A), 0.6926 (1.31% A), 0.7930 (1.24 % A)	1,9
^{123}I	122.90557	13.2 h	EC; γ 0.1590 (82.9% A), 0.4404 (0.35% A), 0.5290 (1.05% A)	1,10
^{124}I	123.906223	4.18 d	EC; β^+ 2.1, 1.5; γ 0.60272 (61.1% A), 0.72278 (10.1% A), 1.50949 (3.02% A), 1.69102 (10.5% A)	1,11
^{125}I	124.9045852	60.14 d	EC; γ 0.03546 (6.67% A)	1,12

Isotope	Isotopic Mass	Half-Life	Decay Modes, Radiations, Major Energies, Absolute and Relative Intensities.	Ref.
^{126}I	125.905622	13.02 d	Ec; β^+ 1.1; β^- 0.86, 1.2; γ 0.3885 (32% A), 0.4920 (1.9% A), 0.6662 (29.1% A), 0.7537 (3.7% A)	1,13
^{127}I	126.90447	Stable (100% Abundance)		26
^{128}I	127.9058134	24.99 m	EC; β^+; β^- 2.1, 1.7; γ 0.44291 (16% A), 0.52662 (1.5% A), 0.9694 (0.4% A)	1,14
^{129}I	128.904989	1.57×10^7 Y	β^- 0.15; γ 0.03958 (7.50% A)	1,15
^{130}I	129.906723	9.0 m	Metastable; β^- 2.5, 1.9; γ 0.53609 (16.7% A), 0.58605 (1.14% A), 1.12215 (0.18% A), 1.61410 (0.48% A)	1,16
		12.36 h	Ground state; β^- 1.0, 0.62; γ 0.41801 (34.2% A), 0.53609 (99.0% A), 0.66854 (96.1% A), 0.73948 (82.3% A), 1.15747 (11.31% A)	1,15
^{131}I	130.9061269	8.04 d	β^- 0.61, 0.33; γ 0.080183 (2.62% A), 0.284298 (6.06% A), 0.364480 (81.2% A), 0.636973 (7.27% A), 0.722893 (1.80% A)	1,15
^{132}I	131.90800	83.6 m	Metastable; β^- 1.5, 0.8; γ 0.098 (3.8% A), 0.1750 (8.3% A), 0.5998 (13.2% A), 0.6100 (1.4% A), 0.6140 (2.4% A), 0.6677 (13.2% A), 0.7726 (13.2% A)	1,17
		2.30 h	Ground state; β^- 1.2, 2.1; γ 0.52265 (16.1% A), 0.63022 (13.7% A), 0.66769 (98.7% A), 0.77620 (76.2% A), 0.95455 (18.1% A)	1,15
^{133}I	132.90783	9 s	Metastable; γ 0.073, 0.647, 0.913	1,18
		20.8 h	Ground state; β^- 1.2; γ 0.529889 (87.3% A), 0.875370 (4.40% A), 1.29833 (2.27% A)	1,15

Isotope	Isotopic Mass	Half-Life	Decay Modes, Radiations, Major Energies, Absolute and Relative Intensities.	Ref.
^{134}I	133.90985	3.7 m	Metastable; β^- 2.5; γ 0.0444 (10% A), 0.2343 (1.6% A), 0.2719 (79% A)	1,19
		52.6 m	Ground state; β^- 1.3, 1.6; γ 0.595362 (11.4% A), 0.621790 (10.6% A), 0.847025 (95.4% A), 0.88409 (65.3% A), 1.07255 (15.3% A)	1,15
^{135}I	134.910064	6.61 h	β^- 1.4, 1.0; γ 1.131511 (22.5% A), 1.260409 (28.6% A), 1.45756 (8.6% A), 1.67803 (9.5% A)	1,15
^{136}I	135.91474	46 s	Metastable (?); β^- 5.1, 5.3; γ 0.1973 (71.3% A), 0.3700 (16.7% A), 0.3815 (100% A), 1.3132 (100% A)	1,20
		83 s	Ground state (?); β^- 4.3, 5.7; γ 1.3132 (67% A), 1.3213 (24.7% A), 2.2897 (11.3% A), 2.4148 (6.8% A), 2.6355 (6.8% A)	1,20
^{137}I	136.91754	24.7 s	β^- 5.0; γ 0.6009, 1.2167	1,21
^{138}I	-----	6.40 s	β^- 7.4, 6.9; γ 0.4827 (40% A), 0.5890 (100% A)	22
^{139}I	-----	2.4 s	β^-; γ	23
^{140}I	-----	0.86 s	β^-; γ 0.3771, 0.4577	24
^{141}I	-----	0.43 s	β^-; γ	25

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Oxidation Potential Schemes

The potential schemes (1) for iodine species at unit activity and 10^{-7} unit activity in acidic and basic solutions are shown below.

The only expected thermodynamic differences between the behavior of iodine at low concentrations and its behavior at normal concentrations is due to the decreased stability of I_2 at low concentrations, I_2 being both a better oxidizing and a better reducing agent when the concentration of I_2 is low. For example, consider the hydrolysis of I_2.

$$I_2(aq) + H_2O = H^+ + I^- + HIO \quad K = 3 \times 10^{-13} \quad (2)$$

In an aqueous solution at pH 7, I_2 is 8 percent hydrolyzed when the total iodine concentration is 10^{-3} M and 98 percent hydrolyzed when the total iodine concentration is 10^{-7} M. At normal concentrations of iodine, HIO is quite unstable with respect to decomposition into I_2 and IO_3^-. At low concentrations of iodine, HIO is only slightly unstable.

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Potential Schemes for Iodine Species at Unit Activity and 10^{-7} Unit Activity
In Volts at 25°C

Iodine Species at Unit Activity	Iodine Species at 10^{-7} Unit Activity
H^+ Activity = 1	
I^- -0.62 I_2(aq) -1.36 HIO -1.14 IO_3^- (-1.7) H_5IO_6	I^- -0.83 I_2(aq) -1.15 HIO -1.14 IO_3^- (-1.7) H_5IO_6
OH^- Activity = 1	
I^- -0.62 I_2(aq) -0.36 IO^- -0.14 IO_3^- (-0.7) $H_3IO_6^{2-}$	I^- -0.83 I_2(aq) -0.15 IO^- -0.14 IO_3^- (-0.7) $H_3IO_6^{2-}$

Reactions of Iodine Species

Isotopic Exchange Reactions.

The rates of isotopic exchange of iodine atoms between various iodine species in aqueous media are summarized below.

Reactants	Comments on the rate of exchange	Ref.
I^-, I_2	Very rapid at room temperature.	1,2,3
I^-, IO^-	Rapid.	4,13
I^-, IO_3^-	Very slow in neutral solutions.	5,14,15
I^-, IO_3^-	Some exchange in neutral solution at 240°C over 24 hours; an induction period was observed.	6,7
I^-, IO_3^-	Very little exchange over 12 hours in ammoniacal solutions at room temperature.	4,15
I^-, IO_3^-	No exchange in neutral solutions at 50°C in 1600 hours.	15
I^-, IO_3^-	No exchange observed in alkaline solutions.	8,15
I^-, IO_3^-	No exchange in neutral solution at 100°C over 3 hours.	9,15
I_2, IO_3^-	Very slow at 25°C in 1 $\underline{f}$ H^+.	5
IO^-, IO_3^-	Very slow in alkaline solution of pH 12.	10,14
I_2, IO_4^-	No exchange in 1 $\underline{f}$ KNO_3 at pH 10.5.	11
IO_3^-, IO_4^-	Exchange occurs only in acidic solutions and here but slowly.	12,14

Redox Reactions.

The rates of some redox reactions of iodine species at room temperature are given below. A discussion of the mechanisms of some reactions of inorganic iodine compounds may be found elsewhere. (31)

Reaction	Comments	Ref.
$4I^- + O_2 + 4H^+ = 2I_2 + 2H_2O$	Slow in low concentrations of H^+ ion. The rate is increased at high concentrations of H^+ ion. The reaction is induced by light and various ions.	16
$2I^- + 2H^+ + H_2O_2 = I_2 + 2H_2O$	Moderately rapid in 0.1 f H^+ ion. Hastened by molybdate.	17,18
$5I^- + IO_3^- + 6H^+ = 3I_2 + 3H_2O$	Rapid in acidic solution.	5
$2I^- + 2Ce^{4+} = 2Ce^{3+} + I_2$	Rapid in acidic solution.	19
$6I^- + Cr_2O_7^{2-} + 14H^+ = 2Cr^{3+} + 3I_2 + 7H_2O$	Very slow at low concentrations of H^+ ion. Moderately rapid in 0.3 f H^+ ion; the reaction is complete in about five minutes.	20
$2I^- + 2Fe^{3+} = I_2 + 2Fe^{2+}$	Reversible and moderately rapid in acidic solutions; equilibrium is established in several minutes.	16
$2I^- + HClO + H^+ = I_2 + Cl^- + H_2O$	Rapid in dilute acid.	18
$4I^- + 2Cu^{2+} = 2CuI + I_2$	Quantitative and rapid.	18
$2I^- + 2HNO_2 + 2H^+ = I_2 + 2NO + 2H_2O$	Rapid.	18
$4I^- + ClO_2^- + 4H^+ = 2I_2 + Cl^- + 2H_2O$	Very slow in HOAc; rapid in stronger acid.	18
$7I^- + IO_4^- + 8H^+ = 4I_2 + 4H_2O$	Rapid in acid.	21
$2I^- + IO_3^- + 3HCN + 3H^+ = 3ICN + 3H_2O$	Quantitative in 0.25 f H_2SO_4.	18
$I^- + 3HClO = IO_3^- + 3H^+ + 3Cl^-$	Rapid in slightly acidic solution.	22

Reaction	Comments	Ref.
$I^- + ClO_3^- = IO_3^- + Cl^-$	Moderately rapid in 0.1 f HNO_3 at 100°C.	23
$2I^- + IO_4^- + H_2O =$ $IO_3^- + I_2 + 2OH^-$	Moderately rapid at pH 3. Rapid in neutral media. Slow in alkaline solutions; 1 f KOH.	22 14 14
$2I^- + IO_4^- + 2H^+ =$ $IO_3^- + I_2 + H_2O$	Rapid in acidic solution.	21
$2I^- + 6Cl_2 + 6H_2O =$ $2IO_3^- + 12Cl^- + 12H^+$	Rapid in neutral or weakly acidic medium.	18
$2I^- + 6Br_2 + 6H_2O =$ $2IO_3^- + 12Br^- + 12H^+$	Rapid in neutral or weakly acidic medium.	18
$I^- + I_2(aq) = I_3^-$	Rapid and reversible; K = 768 at 25°C.	24
$I_2(aq) + H_2O = H^+ +$ $I^- + HIO$	Fairly rapid. $K = 5.4 \times 10^{-13}$ at 25°C.	25,26
$I_2 + H_2SO_3 + H_2O =$ $SO_4^{2-} + 2I^- + 4H^+$	Rapid.	16,27
$I_2 + 2S_2O_3^{2-} = 2I^- +$ $S_4O_6^{2-}$	Rapid in neutral or acidic solutions.	18
$2I_2 + As_2O_3 + 5H_2O =$ $4I^- + 2H_3AsO_4 + 4H^+$	Completely reversible; at pH between 9 and about 4, the reaction proceeds to the right rapidly; in strongly acidic medium the reaction goes from right to left.	18
$I_2 + Sn^{2+} = 2I^- +$ Sn^{4+}	Rapid.	18
$4I_2 + S_2O_3^{2-} + 10\ OH^-$ $= 2SO_4^{2-} + 8I^- + 5H_2O$	Rapid in Na_2CO_3 solution.	18
$I_2 + H_2S(aq) = S +$ $2H^+ + 2I^-$	Proceeds in acidic media.	18

Reaction	Comments	Ref.
$I_2 + 7XeF_2 + 16OH^- = 2IO_4^- + 7Xe + 14F^- + 8H_2O$	Rapid.	28
$2I_2 + IO_3^- + 6H^+ + 5Cl^- = 5ICl + 3H_2O$	In at least 3 f HCl reaction is quantitative.	18
$I_2 + 5H_2O_2 = 2H^+ + 2IO_3^- + 4H_2O$	Rapid in 0.1 f H^+ if IO_3^- is present.	17
$3OI^- + CH_3COCH_3 = CHI_3 + OAc^- + 2OH^-$	Moderately rapid.	18
$3OI^- = IO_3^- + 2I^-$	Very rapid at pH greater than 8.	18,29
$3H_2SO_3 + IO_3^- 3SO_4^{2-} + I^- + 6H^+$	Rapid in acidic solutions.	27
$2IO_3^- 2H^+ + 5H_2O_2 = I_2 + 6H_2O + 5O_2$	Slow in 1 f H^+.	17
$IO_3^- 2SO_2 + Cl^- + H_2O = 2H^+ + 2SO_4^{2-} + ICl$	Reaction is quantitative when the formality of HCl is 3 or greater.	18
$IO_3^- + N_2H_4 + Cl^- + 2H^+ = ICl + 3H_2O + N_2$	Reaction is quantitative when the formality of HCl is 3 or greater.	18
$As_4O_6 + 2IO_3^- + 4H^+ + 2Cl^- = As_4O_{10} + 2ICl + 2H_2O$	Reaction is quantitative when the formality of HCl is 3 or greater.	18
$IO_3^- + Cl_2 + 2OH^- = IO_4^- + 2Cl^- + H_2O$	Rapid in a strongly basic solution.	22,23
$14\ NH_3\ OH^+ + 4IO_4^- = 2I_2 + 7N_2O + 10H^+ + 23\ H_2O$	Rapid in acidic solution.	30

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Anomalous Chemical Behavior of Carrier-Free Iodine (1).

Inorganic Systems

The difference in behavior of very small amounts of iodine from the known behavior of macro amounts of the element was first suspected because of the difficulty of achieving complete exchange of the minute amounts of radioactive iodine isotopes formed in fission, and by beta decay, with macro amounts of the stable inorganic forms of iodine (I^-, I_2, IO_3^-, IO_4^-) added as carrier (2).

The anomalous behavior of carrier-free iodine (3) is exemplified by the following procedure (4) which gave the results listed in Table 1. Eight milliliters of reaction solution was extracted successively with two 5-ml portions

Table 1

Oxidation of Carrier-Free I^- in 0.5 f H_2SO_4 by Ce(IV) at 25°[a]

(8 ml; 0.015 f Ce(IV), 0.015 f Ce (III); 30 minutes)

Benzene extract, 5 ml	Aqueous extract of benzene, 5 ml	Activity, %		Chemical fraction
	H_2O-A	33		
	H_2O-B	3	40	I_2
A	0.2 f KI	4		
	0.1 f NaOH	9		X
	Left in benzene	11		Y
B	-------	4		
C(I_2 carrier)	-------	8		I^-
Extracted aqueous reaction solution		14		Z
Activity adsorbed on walls of reaction vessel		14		

[a]From Kahn, M. and Wahl, A. C. J. Chem. Phys., 21, No. 7, 1185 (1953). (ref. 4).

of benzene (A and B) and one portion of benzene 0.007 f in I_2 (C). The first benzene extract (A) was back-extracted successively with two 5-ml portions of water (H_2O-A and H_2O-B), 5 ml of 0.2 f potassium iodide, and 5 ml of 0.1 f sodium hydroxide. The following chemical fractions were identified.

I_2: The behavior of the activity extracted into benzene from acidic solution and back-extracted into water or aqueous potassium iodide is expected for molecular iodine at low concentrations where the hydrolysis equilibrium $I_2(aq) + H_2O = I^- + H^+ + HIO$ is shifted to the right. Aqueous iodide, via rapid isotopic exchange with molecular iodine, should complete the back-extraction.

X: The activity in this fraction is extracted from acidic solution by benzene, is not back-extracted by water, or by aqueous solutions of potassium iodide, potassium iodate, iron (II), or sulfurous acid, but is back-extracted by aqueous sodium hydroxide.

Y: Same behavior as X except that it is not back-extracted by aqueous sodium hydroxide.

I^-: The behavior of the activity not extracted from acidic solution by benzene but extracted by a benzene solution of molecular iodine is expected for iodide ion; iodide ion exchanges rapidly with molecular iodine and is not soluble in benzene.

Z: The behavior of the activity that is not extracted into benzene and does not exchange with molecular iodine is expected for iodate ion. However, it turns out that, except under drastic oxidation conditions, only a small fraction of this activity is exchangeable with iodate ion.

With dichromate ion as the oxidizing agent in 0.5 f sulfuric acid at 25°, results similar to those summarized in Table 1 were obtained.

In order to determine whether fraction Z was iodate (or some oxidation state readily exchangeable with iodate), a known amount of potassium iodate was added to a solution containing the Z fraction and successive precipitations of silver iodate were carried out. The fraction of total iodate precipitated was determined in each case as well as the percentage of total activity carried. These experiments showed that oxidation of carrier-free iodide in acidic cerium (IV) solution, at 105°C, for 75 minutes, yielded a Z fraction which was iodate or some intermediate completely exchangeable with iodate; at room temperature, over one-half hour, less than 20 percent of the Z fraction formed was iodate.

Studies of the rates of oxidation of carrier-free iodide by dichromate ion or cerium (IV) at 25° revealed that appreciable amounts of all of the various chemical fractions were rapidly formed. Once formed, fractions X, Y, and Z were destroyed very slowly if at all by prolonged oxidation or by reduction with iron (II). The I_2 fraction initially formed was slowly converted to the Z fraction or to a form adsorbed on the walls of the reaction vessels.

Concentration studies showed that the fractions, X, Y, and Z appeared to a small extent at concentrations of iodine as high as 6×10^{-5} f.

The foregoing results were explained as follows: During the oxidation, in addition to molecular iodine, a reactive form of iodine such as HIO is formed which reacts with trace impurities (molecules or colloids) present in the aqueous solution. At least some of these impurities are organic in nature, and their reaction products with iodine are extracted into organic solvents forming fraction X and fraction Y. Part of fraction Y may result from a reaction between benzene and a reactive form of iodine such as HIO. Fraction Z may be either a product of a reaction between a reactive iodine species and an inorganic-type trace impurity, or a simple inorganic molecule or ion containing iodine in an oxidation state intermediate between +1 and +5 (e.g., HIO_2) which exists only at low concentration. Drastic oxidizing conditions (hot acidic solution of cerium (IV)) are required to break up the iodine-impurity compounds or colloids and complete the oxidation to the +5 state.

To characterize further the non-iodate component of the Z fraction, production of the Z fraction *via* oxidation by nitric acid was investigated (5) in order to establish conditions which would lead to a maximum amount of Z fraction composed chiefly of the non-iodate constituent. The most satisfactory method for the preparation of the Z fraction proved to be oxidation by 0.5 f nitric acid at 125° in the dark for 18 to 24 hours. Under these conditions the ratio of the non-iodate component to iodate was a maximum (8 to 1), the absolute yield of the non-iodate component was appreciable (16%), and no electrolyte other than nitric acid was present. The non-iodate portion of the Z fraction was resolved into two components by exhaustive extraction of the Z fraction from 0.5 f nitric acid with benzene. Because of its chemical behavior and reproducibility of formation, it was suggested that the predominant component, designated as the R species, may be the hypothetical oxide, IO_2.

Similar anomalous behavior has also been observed with carrier free iodine released in the gas phase. For example (6), carrier-free $Na^{131}I$ solution was evaporated to dryness in a Pyrex tube. This was then heated to 400° while a stream of air purified by passage through activated charcoal at -78° was passed over the $Na^{131}I$. The oxidation produced a mixture of molecular iodine, iodine compounds and iodine attached to particulate matter. The molecular iodine was removed by passage through a bed of copper knitmesh; particulate activity was removed by passage through a high-efficiency glass fibre filter; the iodine compounds remaining were then either passed through 0.02 f sulfuric acid or condensed in a liquid-oxygen trap. The radioactivity present in the compound fraction was 1 to 2% of the total taken. Analysis of the activity present in the 0.02 f sulfuric acid via solvent extraction studies yielded results similar to

those summarized in Table 1. The major component of the mixture of iodine compounds was identified as $CH_3{}^{131}I$, using an effusion technique in conjunction with gas chromatography.

Paper-electrophoretic experiments have provided evidence for the formation of an unidentified species of iodine (7). With 1 M sodium hydroxide as a background electrolyte, in conjunction with Toyo Roshi No. 50 1 x 40 cm filter paper, a potential gradient of 200 volts/30 cm was applied for 1 hour to a drop of each solution of neutron-irradiated samples of ammonium iodide, potassium iodate and potassium periodate. The temperature of the migration cell was maintained at 10°. The migration velocities followed the sequence $I^- > IO_3^- > IO_4^-$. When neutron-irradiated telluric acid was dissolved in water and ammonium iodide, potassium iodate and potassium periodate added to the resulting solution of iodine activity the migration pattern corresponded to that already observed. However, when the same active solution was free of carrier iodine an unidentified fourth species was usually found at a position lying between iodate and iodide on the chromatographed paper. It was postulated that this species was iodite (IO_2^-).

More information regarding the production and chemical behavior of inorganic forms of carrier-free iodine-131 may be found elsewhere (8).

Organic Systems

When neutron-irradiated uranium metal was dissolved in 14.5 f nitric acid the total amount of organic iodides formed, as determined by gas chromatography, ranged from 1-4%† at dissolving temperatures from 90° to 115° (9). The percentage of organic iodides formed decreased markedly with decrease in dissolving temperature and was only 0.2% at 70° despite the fact that uranium metal dissolved at a reasonable rate at this temperature. Qualitatively, the percentage yield of organic iodides showed no dependence on the irradiation time. When concentrated hydrochloric acid was used as a dissolving agent, $CH_3{}^{131}I$ amounted to less than 0.1%. It was suggested that a reaction in the acidic solution involving a reactive iodine species is responsible for the formation of organic iodides.

Using gas chromatography it was possible to detect at least four organic iodides (10,11) ($CH_3{}^{131}I$, $C_2H_5{}^{131}I$, $n\text{-}C_3H_7{}^{131}I$, $n\text{-}C_4H_9{}^{131}I$) which formed on the addition of carrier-free iodide-131 to various acids, maintained at 100 ± 5°. The highest total organic yield occurred with 10 f nitric acid; in 14.5 f nitric acid the yield decreased to 1.3%; in 3.6 f nitric acid the yield decreased

†Activities of organic iodides were expressed in percentage yields based on the sum of all the iodine-131 fractions recovered in any one experiment.

to 0.1%. The formation of organic iodides (see Table 2) was found to be largely influenced by the kind of acid used, the concentration of the acid, and the reaction temperature. Because the flow rate of the carrier gas helium had little influence on the yield of organic compounds, it was concluded that the formation of organic iodides occurred in the solutions rather than in the gaseous phase or on the walls of the reaction vessel. It was proposed that species containing iodine in an oxidation state lying between that of elemental iodine and iodate reacts with organic impurities to yield organic species such as methyl iodide. When the total iodide concentration was increased to 2 x 10^{-6} f, the total organic iodide yield in 10.9 f nitric acid was only about 0.05%.

Table 2
Yields of Organic Iodides on Addition of Carrier-Free Iodide-131 to Various Acidic Solutions at 100±5°[a]

Acid	Concentration, f	Total organic yield, %
HNO_3	10.9	10.7
H_2SO_4	36	0.5
H_2SO_4	27	0.15
H_2SO_4	18	0.04
$HClO_4$	5.3	0.01
HCl	10.1	0.01
HCl	8.6	0.01
HCl	5.8	0.01
HNO_3 + HCl	9.5 + 2.9	0.01

[a]Data taken from ref. 11.

Biological Systems

The anomalous behavior of carrier-free iodine was also encountered in studies of biological systems. During a study (12) of the uptake of carrier-free iodide from commercial aqueous solutions by tissue preparations, the presence of an anomalous iodine fraction was observed with the aid of chromatographic and radioautographic techniques. This unknown fraction, labeled U-2, was found to be extremely labile in acidic solutions, reverting almost quantitatively to iodide-131 in a few minutes at pH 5. The U-2 fraction was not destroyed over 2 hours, at 37°, in bicarbonate solutions of such reducing agents as thiosulfate, sulfite or arsenite. Electrophoretic experiments indicated that U-2 was an anionic species. Isotopic exchange between U-2 and carrier iodide was slow in

Krebs-Ringer-bicarbonate buffer at 37°. The formation of U-2 could be inhibited by maintaining anaerobic conditions during the experiments, by adding thiouracil, thiocyanate, thiosulfate, or cyanide to the reaction mixtures, or by heating the tissue preparations at 100° for 1-2 hours prior to their use. It was concluded that U-2 was formed *via* oxidation of carrier-free iodide by tissue enzymes and it was suggested that U-2 was an organic compound which contained a labile N-I bond. Subsequently, the production, in a "purely" inorganic system, of an iodine fraction identical to U-2, was reported (13,14).

Chromatographic analysis of commercially available aqueous solutions of carrier-free iodide revealed the presence of iodine fractions chemically and biologically distinguishable from iodide, particularly in aged preparations (13,14, 15,16,17,18,19,20,21). For example (15), after storage of a given commercial aqueous carrier-free iodide preparation over 4 weeks, at room temperature, only 13% of the original iodide activity was present as iodide; five other fractions were discernible on the radioautographs of chromatograms (24%, 16%, 44%, 1%, 1%). Treatment of these aged solutions with carrier iodide at pH 7.0 over 1.5 hours, at 37°, resulted in complete reduction of, or exchange with, these anomalous iodine fractions. This aging phenomenon was not observed when fresh iodide-131 solutions were inoculated with carrier iodide. On the basis of the R_F values of the extraneous bands, it was concluded that neither iodate nor periodate was present in these fractions. The same results were obtained with activity stored under sterile conditions or in the frozen state.

Commercially available iodide-131 solutions were found to contain some iodine in a form which was identical with that in the U-2 fraction mentioned previously (14,17). Oak Ridge solutions of iodide-131 retained their iodide content (99%) for at least 5 days after receipt (14). However, iodide-131 solutions prepared by dilution of Oak Ridge activity with 0.09% saline solution, followed by the adjustment of pH toward neutrality with hydrochloric acid and the addition of 0.9% benzyl alcohol, did show an aging effect. In one experiment over 50% of the activity was represented by U-2 after 27 days of aging. Appearance of the U-2 component was greatly inhibited when either 3×10^{-5} *f* carrier iodide or 5×10^{-4} *f* 1-methyl-2-mercapto-imidazole was initially present in the solutions prepared from Oak Ridge activity.

The U-2 fraction was found to be biologically as well as chemically distinguishable from the iodide fraction. The most prominent extraneous band obtained by chromatographing aged iodide-131 solutions was eluted and injected into the breast muscle of chicks (15); experiments were also carried out using iodine-131 activity as a control. Analysis of the thyroids 24 hours later revealed that, whereas 60% of the iodide activity was collected, only 20% of the extraneous band was collected. Similar results (16) were obtained when activity was injected

intraperitoneally into day-old chicks. Also, the uptake of iodide-131 by thyroid tissue (14) of hogs or cows was larger than the uptake of the U-2 fraction. It was found (18) that from 41% to 94% of the radioactivity of 24 commercial preparations (from two sources) of iodide-131 supplied without cysteine preservative was non-iodide on chromatographic analysis. The average thyroid uptake of iodide-131 containing extraneous activity was significantly lower than the uptake of iodide-131 in 16 human subjects measured under controlled conditions (18).

At this point it is important to note that the use of chromatographic analysis to detect the presence of all unidentified fractions of iodine-131 in an aqueous solution is open to question. It has been found (22,23) that a significant fraction of iodine activity may volatilize during the development of the chromatogram. Furthermore, during the chromatographic process, the carrier-free activity may be partially oxidized by atmospheric oxygen and/or react with water-soluble organic and inorganic degradation products formed by reaction between the paper and oxygen at the wet-dry interface (24).

Determination of the Specific Activity of Radioactive Iodine.

From the specific activity† of radioiodine in a reaction mixture, the counting rate, and the volume of the mixture, the concentration of total iodine can be determined. Since there is a correlation between concentration of iodine and anomalous behavior, the determination of specific activity in investigations of anomalous behavior is important. It should be pointed out, however, that in most of the studies of the behavior of carrier-free iodine no attempt was made to determine the specific activities of the reaction mixtures; the total concentration of iodine was only estimated.

The determination of the specific activity of an active sample involves two measurements: a) the counting rate of the sample; and b) the total iodine content of the sample. The counting rate is easily determined for a given geometry. For those solutions where the total iodine and iodide content are the same the total iodine content has been obtained in a variety of ways. In one procedure (25), a photocolorimetric microtechnique was developed which made use of the catalytic effect of iodide ion on the reduction of cerium (IV) to cerium (III) by arsenious acid in sulfuric acid solution. Under the conditions described it was possible to determine samples of iodine concentrations ranging from 20 to 160 ng/ml.

Total iodine has also been determined with a radiation-insensitive iodide-sensitive membrane electrode (26). Potentiometric measurements on about 5 ml of

†Specific activity may be defined as the ratio of the number of radioactive atoms to the total number of isotopic atoms.

the active sample were made using a system consisting of an iodide electrode (constructed of a silicone rubber membrane impregnated with silver iodide), a calomel electrode and a pH meter. The potentiometric measurement was usually completed within 5 minutes and over this period the performance of the electrode was independent of the radiation fields associated with as much as 20 curies per 5-ml sample. This procedure was successfully applied to the determination of the specific activity of iodide-125 produced from neutron-irradiated xenon.

Carrier-free iodine-125 for protein iodination is available from different manufacturers in an aqueous solution containing sodium hydroxide or sodium sulfite with the activity claimed to be in the form of iodide. This may be true of freshly prepared solutions but in the course of a few weeks significant amounts of this iodide activity is transformed into unidentified species. These species may be volatile and do not exchange with added iodide even after the addition of sodium sulfite or sodium thiosulfate. The uncertainty of the chemical properties of these species essentially eliminates the direct application of chemical methods for analysis (27). Inasmuch as iodine-125 is produced by neutron-irradiation of xenon the isotopic impurities are limited to iodine-126 and iodine-127. It turns out that the total amount of iodine in an iodine-125 solution is represented primarily by iodine-125 and iodine-127 with only a negligible amount contriuted by iodine-126. The nuclides iodine-125 and iodine-127 were determined by measuring the iodine-126 and iodine-128 formed by thermal neutron activation of a sample.

Stability of Highly Concentrated Carrier-Free Iodide-131 Solutions.

The stability of highly concentrated carrier-free iodide-131 solutions was investigated using paper electrophoresis (28). It was found that when the preparations were allowed to age, oxidation processes took place producing molecular iodine and iodate. The yield of the oxidized species depended largely on the pH and concentration of the activity of the solution. For example, 50% of the iodide-131 in an hydrochloric acid solution at a pH of 2.5, containing 5.2 mCi/ml, was converted to molecular iodine and iodate on standing four days. In alkaline solution, however, no oxidation was observed. Thus, none of the iodide in a sodium hydroxide solution at pH 9, containing 11.3 mCi/ml, was changed to molecular iodine and iodate activities on standing for 13 days.

Exposing a carrier-free iodide-131 solution, pH of 4.5, to an external gamma radiation source (^{60}Co gamma source) transformed 45% of the iodide to molecular iodine and iodate. In this experiment the time of irradiation was four hours; the gamma-integral dose was 1.16×10^6 rad. Repeating the experiment with a solution of pH 10 produced no detectable amount of molecular iodine and iodate.

When iodide solutions of macro concentrations were exposed to ultraviolet light, oxidation processes took place and molecular iodine was liberated. Oxidation processes, however, could not be detected on irradiation of carrier-free iodide-131 solutions in quartz ampoules for 2.5 hours, employing the paper electrophoresis technique for analysis.

It is noteworthy that 5×10^{-3} M potassium iodide containing 1.34 mCi/ml iodide-131 at a pH of 3 did not produce any measureable amount of molecular iodine and iodate, after 3 days; however, where the potassium iodide concentration was 5×10^{-5} M and the activity concentration was 2.01 mCi/ml, 38% of the activity appeared as molecular iodine and iodate.

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II. PREPARATION OF IODINE ISOTOPES

Iodine - 121 (2.12 h)

This isotope has been made by the bombardment of natural antimony powder with 30-MeV ^{3}He particles: $^{121}Sb(^{3}He,3n)\ ^{121}I$ (157a). Iodine-123 is a by-product arising from the same reaction on ^{123}Sb.

The iodine-121 was isolated as iodide, iodate, and periodate species, each with carrier, by the following procedures. The target was dissolved in a hot mixture of chromium(VI) oxide and concentrated sulfuric acid which contained iodide carrier. The iodate formed was reduced by phosphorous acid to iodide and the latter was then oxidized to the elemental state by means of 30% hydrogen peroxide. For the formation of iodide, the iodine was distilled into a solution of hydroxylamine hydrochloride, the excess of that reagent being destroyed by evaporation of the solution to dryness. Distillation of the iodine into chlorine water gave iodate. The excess chlorine was removed by extraction into carbon tetrachloride. For the production of periodate, the element was distilled into dilute sodium hydroxide solution and the iodine oxidized with xenon(II) fluoride. The latter compound is unstable in alkaline solution and decomposes leaving a pure periodate solution.

Iodine - 123 (13.2 h)

Preparative Reactions

Because of its medical applications, iodine-123 has been the most extensively investigated iodine isotope from the preparative viewpoint. Both direct and indirect methods of preparation have been developed, the reactions for the best methods being:

$^{121}Sb(^{4}He,2n)^{123}I$ (88, 247, 260, 302, 325, 332, 333, 373)

$^{122}Te(d,n)^{123}I$ (332, 333)

$^{122}Te(^{4}He,3n)^{123}Xe \xrightarrow[EC]{\beta^{+}} {}^{123}I$ (208, 282, 332, 333)

Other reactions which have been used to obtain the isotope include:

$^{123}Te(p,n)^{123}I$ (164, 305, 332, 373)

$^{123}Sb(^{3}He,3n)^{123}I$ (85, 305)

$^{127}I(p,5n)^{123}Xe \xrightarrow[EC]{\beta} {}^{123}I$ (127, 223)

$^{124}Te(p,2n)^{123}I$ (70, 88)

$^{121}Sb(^{3}He,n)^{123}I$ (247)

The three best preparative methods are discussed below.

$^{121}Sb(^{4}He,2n)^{123}I$. Both natural and enriched antimony metal targets have been used (325, 332). With natural antimony, the purity of the product is limited by two factors. Iodine-125 is formed by the reaction $^{123}Sb(42.8\%)(^{4}He,2n)^{125}I$, and unless the energy of the bombarding particle and the target thickness are controlled so that the $^{121}Sb(^{4}He,2n)^{123}I$ reaction predominates, ^{124}I and ^{126}I may also be significant impurities (332):

$^{121}Sb(^{4}He,n)^{124}I$
$^{123}Sb(^{4}He,n)^{126}I$
$^{123}Sb(^{4}He,3n)^{124}I$

With natural antimony and 25.1-MeV ^{4}He particles, the ^{123}I yield in a 17-mg/cm^2 target is about 150 μCi/μA-hr and the ^{124}I activity is 0.7% that of the ^{123}I activity (325).

A yield of 0.60 mCi/μA-hr has been obtained with α-particles of 25-MeV energy and metallic antimony 98.4% enriched in ^{121}Sb. The product was contaminated by 0.8% ^{124}I(332).

Procedures for the separation of ^{123}I, both carrier-free (85, 170, 260) and with carrier (302, 325), from antimony targets generally include the usual types of redox processes involving iodine species. In one procedure for the isolation of carrier-free ^{123}I(260), the bombarded target was dissolved in concentrated HCl-30%H_2O_2. The iodine was then reduced to the elemental state by iron(II) sulfate and extracted into carbon tetrachloride. The aqueous solution was neutralized with hydrochloric acid and finally concentrated by heating. Other media which have been used to dissolve antimony targets are nitric acid (170), a mixture of concentrated nitric and sulfuric acids or of concentrated hydrochloric and nitric acids(170), and a mixture of sulfuric and oxalic acids ((85), for the $^{123}Sb(^{3}He,3n)^{123}I$ reaction). After the nitric acid treatment, the solution was heated, and iodine distilled into an alkaline solution. Several I_2-I^- redox cycles were carried out and the ^{123}I was finally isolated as iodide. Dissolution of bombarded antimony in concentrated H_2SO_4-HNO_3 or HCl-HNO_3 converts the iodine to a mixture of iodate and periodate and these anions were reduced to the element by means of hydroxylamine hydrochloride. Following extraction of the iodine into carbon tetrachloride, I_2-I^- cycles were performed and the iodine was recovered as iodide. Oxidation of the antimony target by concentrated sulfuric acid in the presence of oxalic acid (a reducing agent) leaves the iodine in the elemental condition (85). In the separation procedure, the iodine was distilled into aqueous sodium hydroxide and then acidified and treated with sodium nitrite. The $^{123}I_2$ was extracted into carbon tetrachloride and finally separated as iodide.

The procedures reported for the separation of ^{123}I with carrier from antimony targets (302, 325) differ little in basic chemistry from those described above.

$^{122}Te(d,n)^{123}I$(332,333). The highest yields for this reaction, about 0.1 mCi/μA-hr have been obtained by bombardment of elemental tellurium, 95.4% enriched in ^{122}Te, with 7-MeV α-particles. The target thickness must be less than 25 mg/cm^2. The product was contaminated with only 0.3% of long-lived radioiodine isotopes and 0.9% ^{130}I.

Iodine-123 has been separated carrier-free from bombarded tellurium targets by the following procedure (164, 170). The target (including the aluminum capsule in which it is held) was dissolved in sulfuric acid and iodine oxidized to the free state by hydrogen peroxide. The ^{123}I was then distilled into sodium hydroxide solution (yield, about 85%). Carrier-free ^{123}I has also been obtained directly as I_2 by distillation from molten tellurium at 700°(170).

A method for the recovery of ^{123}I with carrier has employed the dissolution of the tellurium target (plus iodide carrier) in concentrated sulfuric acid, followed by distillation of the I_2 formed into sodium hydroxide solution (332). The same procedure was used for the separation of the isotope from antimony targets.

$^{122}Te(^4He,3n)^{123}Xe \xrightarrow[EC]{\beta^+} {}^{123}I$(208,331,332,333). With a gas-flow tellurium powder target enriched to the extent of about 96% in ^{122}Te and an α-particle beam of 42 MeV, yields of ^{123}I as high as 0.2 mCi/μA-hr have been obtained. Contamination from ^{124}I was as low as 10^{-3}% and from ^{125}I about 0.4%; no other radioiodine contamination was detected (331). In the process, a helium stream swept volatile products from the water-cooled target into a dry ice trap to remove any iodine formed directly and then into a trap cooled by liquid nitrogen to remove ^{123}Xe. After ^{123}I had grown in for five hours, the liquid nitrogen trap was purged with inert gas to remove remaining ^{123}Xe and gaseous contaminants (mainly ^{125}Xe) and the ^{123}I was washed from the walls of the trap with dilute sodium hydroxide solution.

^{123}I Generators

The one ^{123}I generator currently fully developed and in wide use is based on the nuclear sequence just discussed (210,211). The generator yields the isotope in a state of high purity (± 99.8%). The only radiohalogen contaminant is ^{125}I, which is formed in the reactions

$$^{122}Te(^4He,n)^{125}Xe \xrightarrow[16.8\ h]{EC} {}^{125}I$$

Two types of tellurium targets have been employed---the powdered element and the element plated (in the molten condition) on gold foil. In both instances, the tellurium had a ^{122}Te content of greater than 95%. The target was bombarded with an external α-particle beam of 42-46 MeV (degraded to about 35 MeV on the target).

With the powdered target, helium sweeper gas was passed continuously through the target during bombardment. The xenon was carried by the sweeper gas through a silver furnace at 285°-315° to remove radiohalogen impurities, e.g., ^{124}I. The ^{123}Xe was then collected in glass radiator traps at 77°K and vacuum-transferred to glass storage vessels for use.

When about 90% of the ^{123}Xe had decayed to ^{123}I, the yield and purity of the latter isotope were optimum (208). For the preparation of $Na^{123}I$ solutions, the iodine on the walls of the storage vessels was washed off with a solution containing sodium hydroxide and a reducing agent such as sodium thiosulfate. The resulting solution was then neutralized with hydrochloric acid.

For the recovery of ^{123}I from bombarded tellurium-coated gold foil, the tellurium was dissolved in hot acid (HNO_3?) and the xenon purged from solution with a helium stream (211). The xenon was then scrubbed (silver furnace?) to remove impurities.

Under development is a generator utilizing the reactions:

$$^{127}I(p,5n)^{123}Xe \xrightarrow[EC]{\beta^+} {}^{123}I$$

Proton energies in the range 50-65 MeV have been investigated (211). It is claimed that the method provides a product with a low contamination of ^{125}I.

Iodine - 124 (4.18 d)

This isotope has been produced by the reaction:

$$^{121}Sb(^4He,n)^{124}I.$$

Both natural and enriched (98.4% ^{121}Sb) antimony have been used as targets (313, 301). An alpha beam energy of 30 MeV was employed for bombardment of the enriched target(301); the beam energy for the natural antimony target was not given(313).

Carrier-free ^{124}I was separated from the natural antimony by dissolving the target (along with the copper on which it was soldered) in hot concentrated sulfuric acid, and distilling the iodine, and adsorbing it on silver.

The isotopically enriched antimony and the aluminum foil in which it was wrapped were dissolved in hydrochloric acid. Iodide carrier was added and oxidized to a mixture of iodate and periodate by means of nitric acid. Reduction of the oxyanions to elemental iodine with hydroxylamine hydrochloride was then

effected and the iodine extracted into carbon tetrachloride. After a number of I_2-I^- redox cycles, silver iodide was precipitated and ^{123}I impurity allowed to decay.

Iodine-125 (60.14 d)

Iodine-125 has been obtained by the irradiation of Xe with thermal neutrons (169, 190),

$$^{124}Xe^{\dagger}\ (n,\gamma)^{125}Xe \xrightarrow[16.8\ h]{\beta^+} {}^{125}I$$

and by the bombardment of ^{125}Te with protons or deuterons (169),

$$^{125}Te(p,n)^{125}I$$

$$^{125}Te(d,2n)^{125}I$$

Elemental xenon(169) and solid XeF_2(190) have been used as targets in the first reaction. The element was either in the form of a gas under pressure (about 130 atm. at the time of reaction) or as an adsorbate on charcoal. The difluoride has a number of advantages as a target. It is a solid with a comparatively high melting point (130°) and large amounts can be irradiated. Moreover, owing to the nuclear properties of fluorine, no radiocontaminants other than those of iodine can be formed.

When XeF_2 was irradiated in an integral thermal neutron flux of 2.5 x 10^{19} - 5.1 x 10^{19} yields of 26-43 mCi of $^{125}I/gXeF_2$ were obtained. The product was contaminated with less than 1% ^{126}I from the $^{125}I(n,\gamma)^{126}I$ reaction. The cross-section for reaction of ^{125}I with thermal neutrons is high (about 1100 barns) and the extent of contamination by ^{126}I was kept low because large amounts of XeF_2 were irradiated for short periods of time.

The ^{125}I was isolated carrier-free merely by dissolving the irradiated XeF_2 in water at 0°. In the slow reaction which occurs, gaseous xenon and oxygen are evolved and ^{125}I and hydrogen fluoride remain in solution.

Elemental xenon was permitted to stand for several days after irradiation to permit the ^{125}I to grow in. The xenon was then allowed to evaporate at dry-ice-acetone temperature and the carrier-free ^{125}I in the irradiation vessel was dissolved in dilute potassium hydrogen sulfite solution at pH 7-8. The resulting solution was passed through a Dowex 50 cation exchange resin column to remove ^{137}Cs (produced from ^{136}Xe).

Elemental tellurium and the dioxide, with natural abundance of isotopes or enriched in ^{125}Te, have been the targets employed for ^{125}I preparation by bom-

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†The natural abundance of ^{124}Xe is 0.096%.

bardment with protons or deuterons (169). The energies of the bombarding particles were not given.

For the separation of ^{125}I with carrier from deuteron-irradiated tellurium, the target was dissolved in a mixture of chromium(VI) oxide and concentrated sulfuric acid. Solid oxalic acid and iodide carrier were added to the solution and the molecular iodine formed was swept from the solution by the carbon dioxide liberated from the oxalic acid. The iodine was converted to iodide by collection in an alkaline solution of sodium sulfite.

For the separation of carrier-free ^{125}I from irradiated tellurium dioxide, the following procedure was used. The aluminum capsule containing the target was dissolved in a mixture of aqueous sodium hydroxide and sodium nitrate. The dioxide was then dissolved in concentrated nitric acid and iodine distilled into sodium hydroxide solution. The iodine was oxidized to iodate (no reagent given), the solution evaporated, and iodate reduced to elemental iodine ($NH_2OH \cdot HCl$?) which was distilled (collector not given).

Iodine-126 (13.02 d)

This isotope has been produced from natural iodine by the following reactions:

$$^{127}I\ (\gamma,n)^{126}I\ (182,\ 327)$$

$$^{127}I\ (n,2n)^{126}I\ (315)$$

For the first reaction, lithium iodide (327) and iodic acid (182) targets were irradiated with 20- and 25-MeV bremsstrahlung, respectively. No yields of product were given. The only other isotope formed was ^{128}I, which arose from the action of photoneutrons on the natural iodine.

The second reaction has been effected with fast neutrons. Compounds containing iodine in the +5 and +7 oxidation states, KIO_3, $KH(IO_3)_2$, I_2O_5, $NaIO_4$, were irradiated at fluxes between 10^6 and 10^7 $n/cm^2/sec$. Solutions of the irradiated compounds were shown to contain iodine in both lower and higher oxidation states.

Iodine-128 (24.99 m)

Iodine-128 has been made by the bombardment of natural iodine with thermal neutrons (103, 235); $^{127}I(n,\gamma)^{128}I$.

The isotope has been obtained in a condition of high specific activity by the utilization of hot atom effects (235). Reagent grade ammonium iodate was irradiated at a flux of 1.2×10^{12} $n/cm^2/sec$, permitted to cool for a short time, and then dissolved in dilute sulfuric acid. Iodine was immediately extracted into carbon tetrachloride and back-extracted into aqueous sodium hydroxide.

The radiochemical purity of the product was found by measuring both the decay curve and γ-spectrum immediately after irradiation. After the decay of ^{128}I, the major long-lived impurity detected was ^{126}I formed by the (n,2n) reaction on the iodine target. Active impurities were present to the extent of less than 1 ppm.

The specific activity of the ^{128}I was determined from the amount of inactive iodine (I_2) formed by the radiation decomposition of a given weight of the sample. The enrichment factor of the ^{128}I was found to be 1.3×10^4 and the specific activity 4.2 Ci/mgI.

Solid potassium iodide has also served as a target for the reaction (103). Following irradiation (10^{12} n/cm^2/sec), the target was dissolved in water and the solution passed through two Dowex 50 cation exchange resin columns, the first in the H^+-form and the second in the Na^+-form. The γ-spectrum of the effluent showed no apparent radioisotopic contamination of the ^{128}I.

Iodine-129 (1.57×10^7 y)

This nuclide can be obtained either as a fission product of ^{235}U or as a product of the neutron irradiation of tellurium (107). The former method is preferable, since contamination of the product with ^{127}I occurs to a smaller extent.

In the preparation of ^{129}I, natural uranium was irradiated for two days in a neutron flux of 10^{13} n/cm^2/sec, after which it was stored for a year and a half. The sample was then dissolved in nitric acid and iodine distilled into an alkaline (pH 10.1) solution of sodium bisulfite. The distillate was acidified with sulfuric acid and excess sulfur dioxide boiled off. The iodide in the distillate was oxidized to the elemental state by hydrogen peroxide, the element extracted into carbon tetrachloride, and back-extracted as iodide into aqueous sulfur dioxide. The redox cycle was repeated three times. After the last reduction to iodide, excess sulfur dioxide was boiled off.

For each gram of uranium irradiated, 3.13×10^{-8} g of ^{129}I and 9.78×10^{-9} g of ^{127}I were formed.

Iodine-130: ^{130m}I (9.0 m); ^{130g}I (12.36 h)

Three reactions have been utilized for the preparation of ^{130}I:

$$^{129}I(n,\gamma)^{130}I \ (160, 374)$$

$$^{130}Te(p,n)^{130}I \ (160)$$

$$^{133}Cs(n,^4He)^{130}I \ (109)$$

The irradiation of aqueous $^{129}I_2$ solutions at a flux of 10^{12} n/cm^2/sec for 30 seconds or less has been a source of ^{130m}I (374). At the time of irradiation,

^{131}I had decayed in the target. The product was separated carrier-free by extraction into hexane and reduction of $^{130m}I_2$ to iodide by sodium sulfite solution. No ^{128}I (from $^{127}I(n,\gamma)^{128}I$) was found in the irradiated $^{129}I_2$ sample, permitting an upper limit of 3% to be set on ^{127}I impurity.

Both ^{130m}I and ^{130g}I have been isolated by coprecipitation with silver chloride following neutron-irradiation of $Na^{129}I$ dissolved in alkaline sodium sulfite solution. The ^{129}I was 99% radiochemically pure and the flux was 2 x 10^{13} $n/cm^2/sec$. (160).

In the $^{130}Te(p,n)^{130}I$ reaction (160), the target was elemental tellurium and 14MeV protons were used as bombarding particles. Following irradiation, ^{130m}I and ^{130g}I were separated with carrier by the procedure outlined below.

The target was treated with sodium hydroxide solution, iodide carrier added, and the target dissolved with 30% hydrogen peroxide. After evaporation of the solution to near dryness, the iodine was converted to ICl by treatment with hydrochloric acid and sodium chlorate, and the monochloride extracted into butyl acetate. The iodine was back-extracted into water with sulfurous acid and the iodide in the aqueous phase oxidized to the elemental state by acidified sodium nitrite. It was then extracted into toluene. Following back-extraction of the iodine with aqueous sulfurous acid, $La(OH)_3$ and $Fe(OH)_3$ scavenges were carried out and the iodine was finally precipitated as PdI_2.

For the production of ^{130}I from ^{133}Cs, the target, $^{133}CsIO_3$, was irradiated with fast neutrons obtained from the reaction of deuterons on beryllium (109). After irradiation, the cesium iodate was dissolved in an aqueous solution of molecular iodine and the iodine extracted into carbon tetrachloride. Several I_2I redox cycles were then carried out and the iodine was finally precipitated as silver iodide. The product was contaminated with ^{128}I and ^{126}I (the extent of contamination was not given) formed from iodate.

Iodine-131 (8.04 d)

The common sources of this isotope are the fission of ^{235}U and the (n,γ) reaction on ^{130}Te:

$$^{130}Te(n,\gamma)^{131}Te \xrightarrow[24.8\ m]{\beta^-} {}^{131}I$$

Uranium targets have included the metal and its alloy with aluminum (169) and enriched UO_3 (3, 169). The irradiated metal was dissolved in nitric acid and the iodine-131 removed with steam or air. The condensate was concentrated and the iodine distilled (yield, about 85%). The irradiated alloy was treated with aqueous sodium hydroxide to bring the aluminum and iodine into solution. The solution was then separated from the uranium, treated with sulfuric acid, and the iodine distilled into a solution of base. The ^{131}I was purified by

acidifying the solution, oxidizing the iodine to the elemental form, and distilling it.

Following irradiation of the enriched UO_3 and a 15-day period of waiting, iodine was volatilized ("dry distilled") at 800° in a current of air and collected in an alkaline sodium sulfite solution. Prior to collection of the iodine, the vapors were passed through iron shavings heated to 700° in order to remove ^{103}Ru and ^{104}Ru. Iodine-131 with a purity greater than 99% was obtained in about 70% yield.

Various targets have been employed in the (n,γ) reaction on ^{130}Te. Among them have been elemental tellurium (169), TeO_2(169, 196), Te_5O_{11} (322), TeO_3 (321), H_6TeO_6(169, 243), H_2TeO_4(149, 169, 355), Mg_3TeO_6(360), K_2TeO_3(149), and K_2TeO_4(149). Neutron fluxes were 4.7 x 10^{12} n/cm^2/sec for the irradiation of K_2TeO_4, K_2TeO_3, and H_2TeO_4(149) and 1 - 2 x 10^{13} n/cm^2/sec for Mg_6TeO_6(360). For the other targets, fluxes were not cited.

The types of procedures which have been used for the separation of ^{123}I from irradiated tellurium targets are also suitable for processing such targets for ^{131}I: the so-called dry distillation methods in which molecular iodine is volatilized from the target at high temperatures (600-800°) and wet methods in which the target is dissolved in some appropriate medium and the iodine is converted to the elemental form. The free iodine is then treated in the usual fashion. In addition to these general methods, a number of special procedures have been reported. Selected procedures are described below.

Recovery from Elemental Te(169). The tellurium was dissolved in a mixture of chromium(VI) oxide and concentrated sulfuric acid or in one of potassium dichromate and the acid. The periodate formed was reduced to elemental iodine by oxalic acid and the iodine distilled into a solution containing sulfite or thiosulfate. The iodide in solution was oxidized with permanganate and then reduced to the free condition with oxalic acid. The element was again distilled into a reducing solution. Yields up to 90% were obtained.

Alternatively, the oxidizing medium in the first step of the procedure can be a mixture of 30% hydrogen peroxide and concentrated sulfuric acid. In this case, iodine is converted directly to the free element.

Recovery from TeO_2 (169). (a) The dioxide was dissolved in aqueous sodium hydroxide, the solution acidified, and iodide ion oxidized to the element by iron(III) sulfate or by hydrogen peroxide in the presence of sodium molybdate or sodium metavanadate. The iodine was then distilled. Yield, 90%.

(b) The dioxide was heated at 600° for several hours in an oxygen stream or under vacuum (10^{-2} mm Hg pressure). The iodine (I_2) evolved was trapped in alkaline solution or on a cold finger at liquid air temperature. Yield, 90%.

(c) (196) In this method, I_2 was distilled from phosphoric acid solution. The irradiated dioxide was treated with phosphoric acid (sp. gr. 1.84), air was passed into the mixture at a rate of 1-2 ℓ/min., and the iodine distilled into sodium hydroxide solution at about 240°. Yields, 70-90%. Analysis of the distillate by electrophoresis in a phosphate buffer (pH 7.1 - 7.2) showed less than 10^{-2}% contamination by tellurium isotopes and not more than 8×10^{-3}% by ^{75}Se. At least 95% of the ^{131}I in the sodium hydroxide solution was in the form of iodide.

Recovery from Mg_3TeO_6 (360). The irradiated tellurate was dissolved in dilute sulfuric acid and the solution was passed through a column of platinum black at a flow rate of about 1 ml./min. About 98% of the ^{131}I was adsorbed on the platinum. The platinum black was washed repeatedly with distilled water and then the iodine was desorbed by an alkaline solution of sodium sulfite. Multicurie quantities of ^{131}I were separated by this procedure.

Iodine-132: ^{132m}I (83.6 m); ^{132g}I (2.30 h)

Because of the medical applications of the isotope, the development of ^{132}I generators is of considerable interest and such generators are discussed below. First, however, the preparation and isolation of ^{132m}I is described.

^{132m}I (101)

The 83.6-m isomer of ^{132}I has been made by the fission of uranium by protons in the energy range 30-85 MeV and by the $^{130}Te(^4He,pn)^{132m}I$ reaction. The only details given for the (^4He,pn) reaction on ^{130}Te were that the isotope was 99.49% enriched and that sources with less than 1 mg. of iodine were prepared. No chemical separation of the isomer was described.

For the fission process, natural uranium foils and solid $UO_2(NO_3)_2.6H_2O$ were used as targets. The isomer was separated with carrier from irradiated uranium foils in the following manner. The uranium was dissolved in concentrated hydrochloric acid which contained a little concentrated nitric acid. Iodide carrier was added and oxidized to periodate by means of sodium hypochlorite after the solution had been made basic with sodium carbonate. Then the usual redox steps were carried out: reduction of IO_4^- to I_2 with $NH_2OH.HCl$; extraction of I_2 into carbon tetrachloride; back-extraction into aqueous sodium bisulfite; repetition of I_2-I^- cycles until the γ spectrum showed the presence only of iodine products or their xenon decay products.

For the isolation of ^{132m}I carrier-free from irradiated $UO_2(NO_3)_2.6H_2O$ the target was simply dissolved in dilute nitric acid and the iodine adsorbed (exchanged) on silver bromide.

^{132g}I Generators

These generators have ^{132}Te as their source of ^{132}I:

$$^{132}Te \xrightarrow[78\ h]{\beta^-} {}^{132}I$$

The tellurium isotope was made by the fission of ^{235}U, with an enriched uranium (>90% ^{235}U)-aluminum alloy usually serving as the target (133). The isomer ^{131m}Te (30-h) is also produced and gives rise to ^{131}I. The $^{131m}Te/^{132}Te$ ratio was about 0.16 at the end of a 3-day irradiation period.

For the preparation of ^{132}I generators, ^{132}Te has been isolated both with and without carrier from fission products. In one process (133), the uranium-aluminum alloy was dissolved in nitric acid, tellurium carrier added, and the tellurium (and ^{99}Mo also) adsorbed on an alumina column. The molybdenum was eluted from the column with dilute aqueous ammonia and then the tellurium with dilute sodium hydroxide. Following acidification of the tellurium-containing eluate with hydrochloric acid, the tellurium was reduced to the free state by sulfur dioxide. It was then dissolved in nitric acid and again placed on an alumina column. The column was milked for ^{132}I with 0.01 M aqueous ammonia. (A preliminary milking of the column 10-12 h before its use as a source of ^{132}I optimizes the $^{132}I/^{131}I$ ratio.)

An alumina column was also used in a procedure for the separation of ^{132}Te without carrier (63). The irradiated uranium-aluminum alloy was dissolved in aqueous sodium hydroxide, the solution acidified with nitric acid, and iodine removed by distillation. The ^{132}Te and ^{99}Mo in solution were adsorbed on an alumina column and then the molybdenum was eluted with dilute aqueous ammonia.

In another generator (133), the ^{132}Te was adsorbed on a cation exchange resin column (H^+-form). Prior to the adsorption process, the resin had been saturated with tin(II) chloride. Iodine-132 was milked from the resin with water.

A generator for oil-soluble systems has been reported (291). The ^{132}Te was obtained as a carrier-free fission product in nitric acid solution. The solution was adjusted to a pH of 3, made 0.01 M in hydrochloric acid, and passed through a Bio-rad HZO-1 resin column which had been equilibrated with the same acid. It was found that at least 95% of the ^{132}Te was adsorbed on the resin. The column was washed with methyl ethyl ketone to remove excess aqueous phase, and ruthenium-103 contamination of the resin was reduced to a negligible level by washing with 0.01 M hydrochloric acid. The resin was milked for ^{132}I with methyl ethyl ketone which contained 5% (by volume) of the acid.

Iodine-133 (20.8 h)

This isotope has been obtained by the 24-h irradiation of natural uranium or U_3O_8 enriched in ^{235}U(93%) at a thermal neutron flux of 1.6 x 10^{13} n/cm^2/sec (336). For the isolation of the ^{133g}I, the sample was dissolved in dilute nitric acid, iodine carrier added, and the iodine reduced by sulfite. Following oxidation of iodide to the element, the latter was distilled into an alkaline sulfite solution. A number of I_2-I^- cycles were then carried out and iodine was finally converted to iodide. Chemical yield, 60%.

The product was contaminated with iodine isotopes of mass numbers 131, 132, 134, and 135; the activities of the last three dropped rapidly. The ^{135}Xe formed from ^{135}I was swept from solution by means of a stream of air.

Iodine-134g (52.6 m)

The source of ^{134g}I is its precursor ^{134}Te produced in the fission of ^{235}U (351a).

Solid $UO_2(C_2H_3O_2)_2$ (natural uranium) was irradiated with thermal neutrons for 10 min (flux not given) and the target was dissolved in concentrated hydrochloric acid. The solution was boiled for several minutes and was then made 2-3 molar in the acid. A copper foil was introduced into the solution and the fission product tellurium plated out on the foil. The coated foil was dissolved in 1 M nitric acid and passed through a cation exchange resin column (resin not designated) to remove the copper. The eluate was made basic with ammonia water, iodide carrier added, and ^{134}I permitted to grow in for 70 min. The iodide was oxidized to the free element and a number of I_2 - I^- redox cycles were carried out. The iodine was finally precipitated as the silver salt.

The product was contaminated with ^{132}I, ^{133}I, and ^{133}Xe (from ^{133}I). No other radioactive impurities were observed.

Iodine-136 (83 s)

Iodine-136 has been separated from a solution of natural uranium which was irradiated for 30 sec at a flux of about 5 x 10^{13} n/cm^2/sec (62). Iodide carrier and sodium carbonate were added to the irradiated solution and the iodine was oxidized with sodium hypochlorite. It was then reduced to the free state by means of hydroxylamine in acidic solution. An I_2-I^- redox cycle was then performed and elemental iodine extracted into carbon tetrachloride. The radiochemical purity of the ^{136}I at this stage was described as excellent.

III. PROCEDURES FOR THE DETERMINATION OF RADIOACTIVE IODINE IN VARIOUS MEDIA

In Fission Products

Most procedures for the separation of radioiodine from fission products involve the addition of iodide carrier. To insure complete exchange between added carrier and tracer, the mixture is subjected to a series of oxidation-reduction cycles. For example, in an early procedure (Procedure 15, ref. 192), iodide carrier was added to the fission product solution and oxidized to periodate by hypochlorite in alkaline solution. Periodate was reduced to molecular iodine by hydroxylamine hydrochloride in acidic solution. The iodine was extracted into carbon tetrachloride, back-extracted as iodide into water with sodium acid sulfite, and then purified by another carbon tetrachloride extraction cycle in which sodium nitrite was used for the oxidation of iodide to free iodine and sodium acid sulfite again for the reduction of iodine to iodide. The iodine was finally precipitated as silver iodide.

This general procedure, often with variations, is still in common use. One significant variation eliminates the $I^- - IO_4^- - I_2$ sequence; the iodide is oxidized directly to elemental iodine by hydrogen peroxide in hydrochloric acid medium (Wahl, A. C., Phys. Rev. 99, No. 3, 730 (1955)).

A number of other procedures for the separation of radioiodine from fission products have been designed and some of these are described below.

One method which gave a purification of iodine-131 from a 16-day-old solution of 10^{12} fissions had the following steps (224). Iodide, iodate, or periodate carrier and sodium chlorate were added to the sample which contained only inorganic substances (but no gold) and no reducing agents. The solution was made 6-10 M in hydrochloric acid in order to produce iodine monochloride (yellow-green solution). The monochloride was extracted into butyl acetate and then back-extracted into water as iodide by means of sulfurous acid. Iodide was oxidized to elemental iodine with iron(III) chloride in dilute sulfuric acid and the iodine extracted into toluene. The element was back-extracted into water as iodide by sulfurous acid and palladium(II) iodide was precipitated.

If gold was present in the fission product solution, the iodine was converted to the monochloride as described above and the solution was boiled to expel excess chlorine. Then sufficient sulfurous acid was added to reduce gold (III) to the free element and iodine monochloride to iodide. After removal of the gold and excess sulfur dioxide, sodium chlorate was added and the solution made 6 M in hydrochloric acid. Then the procedure was taken up at the point where the iodine monochloride was extracted into butyl acetate.

The chemical yield was 80-90%. The separated product showed no evidence of contamination by longer-lived nuclides after 10 half-lives.

A procedure for the separation of carrier-free iodine from fission products has employed the exchange of iodide on a column of silver chloride on silica gel as its main decontamination step (220). The sample in dilute nitric acid solution was treated with calcium hypochlorite solution at pH 7-8 to convert the iodine to iodate. The latter was then reduced to iodide by sulfite and the solution placed on the silver chloride-silica gel column. Iodine was eluted from the column as iodate by means of calcium hypochlorite solution (pH 7-8) and the iodate then reduced to molecular iodine with hydroxylamine hydrochloride in acidic solution. The iodine was finally extracted into carbon tetrachloride. The chemical yield was about 90%.

Curie quantities of fission-produced iodine have been purified by adsorption on platinum (64). The irradiated target, enriched uranium alloyed with aluminum, was dissolved in aqueous sodium hydroxide. The solution was acidified with sulfuric acid and sparged with air which was then passed through a sodium hydroxide scrubber. The scrubber solution, which now contained the radioiodine in the form of iodide, was acidified with sulfuric acid and the radioiodide was permitted to be adsorbed on a specially prepared platinum felt (directions for preparation of the felt not given). The iodine was removed from the felt by washing with dilute sodium hydroxide solution. Chemical yields were 65-75% and decontamination factors were greater than 10^6 for removal of other fission products. The original solution contained about 250 curies of radioiodine.

An interesting final step has been reported in a procedure for the determination of fission iodine in the water coolant of nuclear reactors (200). After some of the usual chemical steps: oxidation of iodide carrier to periodate by means of hypochlorite; reduction of periodate to elemental iodine with hydroxylamine hydrochloride; and extraction of iodine into carbon tetrachloride, the iodine was adsorbed from the carbon tetrachloride solution onto activated carbon. The chemical yield for the procedure was 92%.

A method has been described for the separation of iodine isotopes 131-135 from the spontaneous fission of uranium - 238 (241). A large sample of U_3O_8 (samples were reported to contain 1000 and 2500 g of natural uranium) was dissolved in nitric acid and the uranium precipitated as the peroxide. The latter was dissolved in hydrochloric acid and uranyl chloride, UO_2Cl_2, was crystallized from solution. The chloride was dissolved in water and the pH of the solution adjusted to 0.5 with hydrochloric acid. Periodate carrier was added and the iodine reduced to iodide with a saturated aqueous solution of sulfur dioxide.

Iodide was then oxidized to free iodine by dichromate and the iodine was extracted into carbon tetrachloride. It was back-extracted as iodide into water by treatment with acid sulfite and silver iodide was precipitated. The latter was reduced with zinc dust, the free silver separated, and the iodide oxidized to the molecular form by arsenate. The iodine was distilled into aqueous sulfite and silver iodide was precipitated. Chemical yield: 80-90%.

In Water

Three types of methods for the determination of radioiodine in water in concentrations greater than 100 pCi/ℓ have been published by the American Society of Testing Materials (ASTM): ion exchange; distillation; and extraction (17).

In the procedure utilizing ion exchange, the sample solution was first subjected to the usual redox treatment to insure exchange, with the iodine ultimately being converted to iodide. The solution was then passed through a cation exchange resin of the phenol-sulfonic acid type and then iodide was adsorbed on preformed silver iodide. The recovery of radioiodine was greater than 97%.

In the distillation method, iodide carrier was added to the sample, an oxidation-reduction cycle was carried out, and molecular iodine was distilled into carbon tetrachloride. (In the oxidation-reduction cycle, iodide was oxidized to iodate by permanganate in sulfuric acid solution and any elemental bromine and chlorine activities formed were distilled off.) Iodine was back-extracted as iodide into water from the carbon tetrachloride by means of acid sulfite and silver iodide was precipitated.

The extraction procedure made use of the usual chemistry: addition of carrier; redox cycle; extraction of molecular iodine into carbon tetrachloride; back-extraction of iodide; and precipitation of silver iodide.

Other procedures reported for the analysis for radioiodine in water (e.g., 102, 128, 143, 168) make use of chemistry similar to that in the ASTM methods.

In Milk

Radioactive iodine in milk is predominantly (greater than 90%) in iodide form and milk ordinarily requires no pretreatment before separation of iodine (128). When formaldehyde is added to milk as a preservative, most of the iodide becomes bound to the protein in the milk. Trichloroacetic acid is an effective precipitant for the protein.

In one analytical procedure (376, p. 102), fresh milk was treated with formaldehyde, the mixture was allowed to stand for 1 hour to permit all the nonionic iodine to become bound to protein, and trichloroacetic acid was added to precipitate protein. The iodide left in solution was coprecipitated with silver

chloride by the addition of silver nitrate. The precipitated protein and silver halides were combined and counted.

For the determination of picocurie concentrations of radioiodine in whole milk containing no hydrogen peroxide or formaldehyde (181), iodide carrier was added to the milk sample stored at 0°-1° and the mixture was passed through a Dowex 1-X8 anion exchange resin column (Cl^- form). Iodide was eluted from the column with dilute sodium perchlorate and precipitated as the silver salt. Any coprecipitated silver chloride was removed by treatment of the precipitate with concentrated aqueous ammonia. The minimum detectable concentrations for 1-ℓ samples were at the 1 pCi level and the decontamination factor from interfering fallout radionuclides was greater than 4×10^4.

A method claimed to be applicable for a minimum detectable activity of less than 0.05 pCi/ℓ of sample at the time of counting has been reported (86). Carrier was mixed with the milk sample and the mixture was heated to about 75°. Iodide was collected on an anion exchange resin (resin not specified) and then eluted as iodate by means of hypochlorite. Iodate was converted to free iodine by treatment with hydroxylamine hydrochloride and sodium nitrite. An I_2-I^- cycle was performed and the iodine extracted into toluene in which medium it was determined photometrically for chemical yield. 2-Methylbutene was added to the toluene solution and the liquid sample was mixed with a toluene-based liquid scintillation counting solution and β-gated γ coincidence counting was carried out.

In instances where a high concentration of natural iodide in the milk interfered with the chemical yield determination, iodide, before the addition of carrier, was analyzed for with the use of an iodide selective electrode. (See ref. 51a for the determination of chemical yield by means of the iodide selective electrode. Use of the electrode makes standardization of iodide carrier unnecessary. The electrode has also been employed in determining the specific activity of iodine-131 solutions (14).)

A rapid, sensitive separation of radioiodine from milk has as its major step exchange of iodide ion on silver chloride (119). Iodide carrier was added to the homogenized sample and the sample was stirred. (Raw milk samples were cavitated in an ultrasonic unit for homogenization.) The mixture was passed through a silver chloride column. (The exchange of iodide on silver chloride was said to be sufficiently selective that the latter could be counted directly.) A saturated solution of chlorine in dilute sulfuric acid was allowed to come in contact with the column long enough for iodide to be oxidized to iodic acid. The iodic acid effluent was neutralized with sodium hydroxide solution, boiled, and acidified with concentrated nitric acid. Iodate was reduced to free iodine by hydroxylamine hydrochloride and the iodine extracted into benzene. Two I_2-I^-

cycles were performed and silver iodide was precipitated. The chemical yields were 90-95% for homogenized milk and 85-95% for raw milk. Less than 2 pCi of radioiodine could be detected in 1 ℓ of milk.

Iodine-129 and iodine-131 concentrations of 0.2 pCi/ℓ (with 4-ℓ samples) were measured in milk with a standard deviation of better than ±10% by the following method (38). Formaldehyde, iodide carrier, and acid sulfite were added to the sample which then either was passed through a Dowex 1-X8 anion exchange resin column (form not given) or added to a batch of the resin. Treatment of the resin with hypochlorite converted iodide to iodate which came off the resin. Iodate was reduced to elemental iodine by hydroxylamine in acidic solution. The usual extraction and reduction of the iodine were then carried out and palladium (II) iodide was precipitated. The chemical yield was about 82% for the column method and 75% for the batch method.

In Biological Materials

In a procedure for the analysis of radioiodine in vegetation (ref. 376, pp. 94ff), the following reagents were added to the sample: carrier; copper foil; cerium(IV) sulfate; sulfuric acid; and potassium permanganate. The mixture was heated until the sample was completely oxidized. Then oxalic acid was added to convert the iodine to the elemental form and the iodine was distilled into aqueous sodium hydroxide. The alkaline solution was treated with hydroxylamine hydrochloride, sodium nitrite, and nitric acid and the free iodine was extracted into carbon tetrachloride. The iodine was back-extracted as iodide with sulfurous acid and precipitated as the silver salt.

For thyroid glands (ref. 376, pp. 94 ff), most of the organic matter was first destroyed by fusion with sodium hydroxide in the presence of iodide carrier. The destruction process was completed by the addition of solid potassium nitrate and ignition. Then the iodine was converted to the molecular condition by the addition of hydroxylamine hydrochloride, sodium nitrite, and nitric acid, and the usual extraction and reduction process was carried out.

Urine samples were also prepared for analysis for radioiodine by oxidative destruction (105). Iodide carrier, sulfuric acid, and solid potassium permanganate were added to a sample and the mixture was boiled under reflux to oxidize the carrier to iodate. Then the following steps were performed: reduction of iodate to free iodine by oxalic acid; distillation of the iodine into water; reduction to iodide by sulfurous acid; and precipitation of silver iodide. The chemical yield was 84%; the standard deviation ±4%.

In the Atmosphere

In a method for the determination of radioiodine in the atmosphere (20), samples were collected on wool-rayon filter pads impregnated with a $Ba(OH)_2$-BaI_2-$Ba(IO_3)_2$ mixture. The mixture was prepared by dissolving a known amount of elemental iodine in a glycerol-water solution and adding barium hydroxide. (The barium hydroxide-glycerol solution is a favorable medium for the hydrolysis of alkyl iodides.) The barium filter, after collection of sample, was processed for iodine-131 content as follows. The filter was cut into several pieces which were added to dilute sulfuric acid in a distillation flask. Iodide and iodate reacted in the acidic medium to give molecular iodine. Any iodide remaining in the flask was then oxidized by the addition of sodium nitrite solution and iodine was distilled into a receiver containing sodium hydroxide solution. The distillate was acidified and iodine extracted into carbon tetrachloride.

IV. PROCEDURES FOR THE DETERMINATION OF IODINE BY NEUTRON ACTIVATION

The great bulk of the work which has been done on neutron activation analysis for iodine has dealt with natural iodine-127 ($^{127}I(n,\gamma)^{128}I$), and the procedures described below are concerned primarily with that isotope. Because of the growing interest in the long-lived, fission-produced iodine-129 (1.57×10^7 y), the section is concluded with a method for the sequential analysis of low levels of iodine-131, iodine-129, and natural iodine in environmental samples based on chemical separation and neutron activation analysis techniques.

Iodine in Water (230)

Aqueous ammonia was added to the water sample prior to neutron irradiation. Following irradiation, the indicated reagents were added to the sample: sodium nitrate; sulfuric acid; Cl^-, Br^-, Cu^{2+}, and Mn^{2+} holdback carriers; ethylenediamine (to complex Cu^{2+}); and $^{125}I^-$ (for the determination of chemical yield). The solution was then passed through a Dowex 1-X10 resin column on which molecular I_2 had been irreversibly fixed. (The resin was prepared by converting the resin originally in the hydroxide form to the iodide and oxidizing the latter to the free element by means of sodium nitrite in acidic solution. The iodinated resin was preconditioned with aqueous sodium nitrate.) Iodine-128 was counted directly on the resin bed. Calibrated potassium iodide standards were also counted. The limit of sensitivity of the method was determined to be 3.5×10^{-4} µg of iodine and recoveries of iodine were 95-100%.

Iodine in Biological Materials

The neutron activation technique is particularly useful for the analysis of bound iodine in biological materials, since essentially all the radioiodine is present as iodide after irradiation.

In a procedure for the determination of natural iodine in solid samples of biological materials (230), $^{125}I^-$ and iodide carrier were added to the irradiated sample and the mixture was heated in a solution of chromium (VI) oxide in concentrated sulfuric acid. The mixture was cooled and diluted, and phosphorous acid was added to convert the iodine to iodide. The latter was then oxidized to elemental iodine by the addition of sodium nitrite, and the iodine was distilled into aqueous sodium hydroxide which contained Dowex 2-X8 resin (OH^- form). The mixture was shaken, the liquid decanted and the resin counted. Chemical yields of 80-90% were obtained. Where further purification was desired, the resin was washed with sodium nitrate solution to elute chlorine-38 activity, the most likely contaminant. Such treatment decreased the yields to 60-70%.

In a method of analysis for iodine-127 in blood serum (80), the sample was lyophilized (freeze-dried) prior to irradiation. (Such treatment reduces sample size to about one-tenth of its original weight. Losses of iodine during lyophilization are negligible.) After irradiation, ^{131}I-labeled thyroxine (for chemical yield determination) was added to the sample and the mixture was burned in a combustion apparatus, the bottom of which contained a solution of ammonium iodide and sodium hydrogen sulfite. The gases evolved in the combustion process were absorbed in traps of aqueous potassium hydroxide and hydrochloric acid. The burned sample and the solutions of the absorbed gases were added to the solution in the bottom of the combustion flask and the resulting solution was transferred from the flask, treated with concentrated aqueous ammonia, and boiled. A substoichiometric amount of silver nitrate in concentrated aqueous ammonia was added to precipitate silver iodide, which was counted. Ammonium iodide standards were also prepared and irradiated. A 3.5% relative standard deviation was obtained in analysis for the iodine content of a given serum and the sensitivity of the method was a few nanograms. About 99% of the iodine in the ^{131}I-labeled thyroxine was accounted for in the combustion process. Decontamination factors from sodium-24, chlorine-38, bromine-80, and bromine-82 were greater than 10^4.

In a procedure specifically designed for the determination of bound iodine in blood serum or plasma (225), iodide was removed prior to the irradiation of the sample by passage through a Dowex 2-X8 anion resin (NO_3^- form) column. The column was washed with water and the washings were combined with that part of the sample which had passed through the column. Potassium pyrosulfite was then added and irradiation was carried out. Standards, using standardized commercially lyophilized serum, were prepared in the same manner. The irradiated resin-treated sample and standard were transferred to iodinated resins (made by passing a saturated solution of $I^- - I_2$ through Dowex 2-X8 resin). Before counting, the resins were washed with dilute sodium chloride solution to eliminate traces of sodium-24 and chlorine-38 contamination. Measurable interferences of these isotopes were not observed after such treatment.

A procedure for the analysis of natural iodine in aqueous solutions of iodohippuric acid (57) has utilized a freezing technique for the preparation of the sample for irradiation. Irradiation of a frozen sample rather than a solution has a number of advantages: the pressure in irradiation containers caused by the radiolysis of water is reduced substantially; losses of iodine due to vaporization and also to adsorption on container walls are greatly minimized; and interference from nuclides from container walls is avoided.

In the procedure in question, the sample was irradiated at -40° and transferred in the solid phase to a glass container after a thin surface layer was

allowed to melt. Iodide was adsorbed on an anion exchange resin (Cl^- form) from a slightly alkaline solution containing ammonium iodide carrier and the resin was counted directly. Excellent results were obtained.

Iodine in Atmospheric Samples

For the determination of iodine-127 in the atmosphere, samples are generally collected on activated charcoal. The charcoal must have an extremely low iodine content so that the activity of irradiated blanks is minimal. Two procedures for the chemical treatment of airborne samples collected on charcoal are described below.

In one instance (246), the sample on charcoal was transferred after irradiation to a hot solution of sodium hydroxide, iodate carrier was added, and the radioiodine oxidized to iodate by means of hypochlorite. Following reduction of iodate to iodide with bisulfite in acidic medium, an I^--I_2 redox cycle was carried out in an acetate buffered solution (pH 4). Iodide was finally precipitated as the silver salt. Appropriate iodide standards were prepared and analyzed.

In the other case (238), $^{131}I^-$ solution (for chemical yield determination) was added to the sample on charcoal prior to irradiation. The irradiated sample was simply leached with concentrated aqueous ammonia and palladium(II) iodide was precipitated. The error in the procedure was estimated as ±20%. Although it was demonstrated that the aqueous ammonia leached iodine present in the iodide, molecular iodine, and iodate forms, no attempt was made to convert the iodine leached completely to iodide.

Sequential Analysis of Low Levels of ^{131}I, ^{129}I, and ^{127}I(50)

Carrier-free iodine-125 tracer (form not given) was added to the sample which was then burned in oxygen (details not given). The iodine set free was trapped on activated charcoal and the charcoal counted for iodine-131 and -125 (for chemical yield). The charcoal was transferred to a vacuum system and heated to liberate the trapped iodine, which was caught in a quartz tube at liquid nitrogen temperature. The iodine was irradiated along with a comparator standard of mixed iodine-125, -127, and -129. Following irradiation, carrier was added and the iodine was further purified by distillation and solvent extraction (details not given). Iodine was finally precipitated as silver iodide, which was mounted on thin plastic scintillators for counting.

A word of caution regarding the neutron-activation analysis of fission-produced iodine-129 is in order. When that isotope is subjected to a neutron flux, 61% of the activated atoms are generated as iodine-130m (9.0 m) (209). By waiting until the short-lived metastable isotope has decayed (about 10 half-lives) before beginning chemical operations on the sample, complications resulting from isomeric transition induced reactions are eliminated.

V. PROCEDURES FOR THE RAPID SEPARATION OF RADIOACTIVE IODINE FROM FISSION PRODUCTS

A number of the procedures developed for the rapid separation (lower limit 1-2 sec) of radioiodine from fission products for the study of short-lived isotopes are derivative of the standard separation procedure described in section III and only one of those procedures (215) is given here. Iodide in the fission product solution was oxidized to iodate by means of permanganate in acidic solution. This step was followed by reduction of iodate to molecular iodine with hydroxylamine hydrochloride and extraction of the free element into carbon tetrachloride. Iodine was back-extracted as iodide into an ammoniacal solution of sodium pyrosulfite and iodide was precipitated as the silver salt. The time required for the procedure was about 150 sec and the separation yield was approximately 95%.

In a method requiring less than 10 sec (139, 140, 140a) the mixture irradiated consisted of uranium peroxide ($UO_4 \cdot 2H_2O$), paraperiodic acid (H_5IO_6), and activated carbon. The peroxide and the periodic acid were present in a weight ratio of 1:14. After irradiation, the mixture, in its graphite capsule, was transferred to an analytical apparatus maintained at 125° and the capsule was broken open. (At 125°, the paraperiodic acid decomposes to molecular iodine, oxygen, and water, and there is rapid exchange between fission product iodine and carrier iodine.) The molecular iodine formed was collected on a column of glass beads wet with carbon tetrachloride and the I_2-CCl_4 solution was washed from the beads into a volumetric flask by additional solvent. Aliquots of the solution were used for photometric determination of iodine. The chemical yield was 70-80%. The procedure gave excellent decontamination from tin, antimony, tellurium, molybdenum, ruthenium, xenon, cesium, and barium. The only serious contaminant was bromine, the decontamination factor from that element being 11.

Extremely rapid (2 sec or less) automated separations of iodine (and bromine) making use of ion exchange on preformed silver halide precipitates have been reported. In one procedure (96, 297), the irradiated solution contained uranyl nitrate (90% ^{235}U), iodine-131 tracer, sulfurous acid, sodium nitrate, carriers for iodide, tellurium (IV), tin (II) and antimony (III), and oxalic acid to prevent hydrolysis of the last carrier. Immediately after irradiation, the solution in its sealed container was projected into a filtering unit and the container broken by impact. The solution was sucked through a thin layer of silver chloride and then a wash solution of oxalic acid-nitric acid was injected onto the filter. Because of the high filtration speed and the small volume of wash solution, which had to be used to obtain a fast and sharp separation of

iodine, the silver halide precipitate was contaminated by adsorbed fission product solution or by chemically-retained precursors of iodine. Also, the filtrate was contaminated by break-through of iodine. Correction for cross-contamination was made through the use of measurements with various tracers.

In another automated procedure utilizing ion exchange on preformed silver chloride (110), the irradiated uranyl nitrate (natural uranium) was dissolved in dilute nitric acid containing sulfur dioxide as reducing agent and iodide carrier and immediately filtered through a bed of the silver halide. Iodide was reported to be essentially quantitatively adsorbed on the silver chloride.

In a later work (317) than that just noted, the irradiated uranium was dissolved in dilute nitric acid solution which also contained sulfur dioxide and iodide carrier. The solution was filtered successively through beds of silver iodide and silver chloride. The combined iodine yield on the two preformed precipitates was 80-90%. Use of appropriate tracers showed that no selenium, tellurium, or antimony was adsorbed on the precipitates.

A novel gas-chromatographic procedure for the rapid separation of fission iodine (or bromine) from the other fission-produced elements has been reported (97). The method is based on the formation of methyl iodide when uranium is irradiated in an atmosphere of methane. A thin uniform layer of "Krylon"-lacquered uranium dioxide (natural uranium or enriched uranium-235) was deposited on an aluminum sheet and the sheet was placed in a plastic irradiation vial which was then filled with methane, closed, and placed in an irradiation rabbit. (The "Krylon" lacquer slows down the recoil fragments before they enter the gas phase.) Following irradiation, the gaseous content of the vial was injected into a gas chromatograph. Recoil-produced CH_3I^*, in chemical yield of about 1% based on the iodine formed by fission, was characterized within 1 min after the end of irradiation. Smaller amounts of $C_2H_3I^*$ and $C_2H_5I^*$ were also identified.

VI. PROCEDURES FOR THE SEPARATION OF IODINE SPECIES FROM EACH OTHER

Iodide and Iodate

Mixtures of radioiodide and radioiodate have been cleanly separated by ascending paper chromatography (135, 391) and high voltage paper electrophoresis (391). With the first technique and the anion exchange cellulose papers Whatman AE 30 and DE 20 in the free-base form, the R_F values shown in Table 1 were obtained when aqueous sodium carbonate and sodium citrate were used as developing solutions (135).

Table 1

Chromatography of Iodide and Iodate on Whatman AE 30 and DE 20

	Whatman AE 30				Whatman DE 20			
	Sodium Carbonate		Sodium Citrate		Sodium Carbonate		Sodium Citrate	
	0.05 M	0.25 M	0.05 M	0.25 M	0.05 M	0.25 M	0.05 M	0.25 M
Anion	R_F	R_F	R_F	R_F	R_F	R_F	R_F	R_F
I^-	0.63	0.71	0.58	0.67	0.40	0.50	0.42	0.42
IO_3^-	0.80	0.89	0.82	0.78	0.75	0.88	0.88	0.82

It should be noted that the R_F values for iodide on Whatman DE 20 paper are much lower than they are on AE 30; DE 20 is more basic than AE 30. With Whatman No. 1 paper and methanol-water (85:15) as the developing solvent, R_F values of 0.70 for iodide and 0.40 for iodate were found (391). The mobility of the ions was unaffected by the pH of the solvent in the range from 2 to 12.

In the electrophoretic method (391), the electrolyte was 0.025 M sodium barbiturate, 0.05 M sodium acetate, or 0.025 M sodium octoate, with the pH adjusted to 8.6 by means of 0.1 M hydrochloric acid. The sample solution was applied on Whatman No. 1 paper which had been previously equilibrated with electrolyte. After 30 min at 5° and 3000 V, R_M values for iodide and iodate were 0.47 and 0.20, respectively.

Molecular Iodine, Iodide, and Iodate

These species, in carrier-free form, have been separated by low-voltage and high-voltage electrophoresis on Whatman No. 1 paper (252). Voltages of 600 and 1200 V were employed. In the former instance, the electrolyte was 0.1 M sodium chloride, the time of electrophoresis 90 min, and the voltage gradient 10 V/cm; at 1200 V, the electrolyte was 0.01 M sodium chloride, current was passed for 20 min, and the voltage gradient was 30 V/cm. The sample to be analyzed (about

0.01 ml) was applied at the center of the paper strip wetted with electrolyte and the strip was subjected to electrophoresis between hydrophobized glass plates. Separation data are summarized in Table 2.

Table 2

Distances of Migration of Carrier-Free I_2, I^-, and IO_3^- from the Starting Point in the Direction of the Anode

Species	600V/1.4-1.6mA	1200V/2.0-2.5mA
I_2	0.6-1.2 cm	2-4 cm
I^-	11-14 cm	14-16 cm
IO_3^-	4.5-7.0 cm	8-10 cm

Iodate and Periodate

Carrier amounts of iodate and periodate have been separated conveniently on an anion exchange resin and by liquid-liquid extraction (189). In the first method, a neutral solution containing the anions was added to a resin column prepared from 200-400 mesh, 8% cross-linked Amberlite IRA-400 or Dowex 1 in the acetate form. Following a water wash, iodate was selectively removed from the column by means of 0.1 M ammonium chloride.

In the liquid-liquid extraction procedure, the water-insoluble quaternary ammonium salt tricaprylmethylammonium chloride dissolved in trichlorotrifluoroethane (Freon-TF, $Cl_2FCCClF_2$; b.p. 47°) was used to extract periodate from neutral aqueous salt solutions. More than 99% of the periodate appeared in the organic phase after a single extraction of an iodate-periodate sample with an equal volume of the quaternary ammonium salt-Freon solvent system; >94% of the iodate remained in the aqueous phase. High concentrations of sodium chloride (0.2-0.5 M) afforded clear, clean phase separation.

Iodide, Iodate, and Periodate

The separation of carrier-free iodide, iodate, and periodate has been performed by chromatographic and fractional precipitation techniques.

In one procedure (282), good separation was achieved using a thin layer (0.13 mm thick) of silica gel containing a starch binder. The best results were obtained with the solvent system: methanol-25% ammonium hydroxide-water-10% acetic acid (volume ratios, 9:1:1:0.5) and a chromatographic development time of 50 min. This combination gave the R_F values: I^-, 0.83; IO_3^-, 0.51; IO_4^-, 0.00. The influence of the amount of carrier on the separation of the anions by the solvent system noted above was investigated over the concentration range 10^{-4} to 10^{-1} M. In this range, the R_F values for iodide and periodate did not change,

whereas the value for iodate decreased at concentrations exceeding 10^{-3} M and the ion moved with a diffuse tail from the origin. These facts made the separation of iodate from periodate difficult at concentrations greater than 10^{-3} M.

Excellent chromatographic separation of the carrier-free anions has also been carried out with the use of glass fibre paper (Schleicher and Shull No. 6) (250). Of the various solvent systems investigated, the best proved to be n-butanol-acetone-water (volume ratios, 5:2:3); R_F values: I^-, 0.95; IO_3^-, 0.37; IO_4^-, 0.01. No reduction of periodate was detected. Such reduction (probably to iodate) has been shown to occur on cellulose papers.

In the fractional precipitation method for the separation of radioiodide, radioiodate, and radioperiodate (27,45), the sample was dissolved in ammonia water and iodine carriers were added. (The basic nature of the solution prevents redox reactions which might occur between the oxo species and iodide in an acidic or neutral medium.) Silver iodide was then precipitated from the ammoniacal solution. The solution which remained was acidified with nitric acid and silver iodate precipitated. The periodate, which was left in solution, was reduced to iodide by means of sulfite and the iodide precipitated as the silver salt. The separation is not entirely satisfactory; high results for periodate are generally obtained because some iodate appears in the periodate fraction as a result of the slight solubility of silver iodate.

Molecular Iodine, Iodate, and Periodate

The separation of these species has been described in a report on the chemical states of radioiodine (^{128}I, ^{126}I, and ^{130}I) formed by fast neutron activation of solid cesium periodate and cesium perchlorate (108). For the latter salt, the irradiated solid was dissolved in a solution containing molecular iodine, cesium iodate, and sodium periodate carriers. Molecular iodine was extracted into carbon tetrachloride, back-extracted into a sulfite solution, and precipitated with silver nitrate and nitric acid. The iodate fraction was precipitated as silver iodate by the addition of excess silver nitrate and nitric acid. The periodate fraction was reduced to iodide by sulfite and precipitated as silver iodide.

The cesium periodate chemistry was similar to that described above, with cesium iodate and molecular iodine in aqueous solution being used as carriers.

Iodide from Organically-Bound Iodine

Radioiodide has been separated from organically-bound radioiodine mainly by chromatographic methods.

In a procedure (123) for the removal of iodide from iodine-labeled bovine IgG-2, the sample solution (in phosphate buffer, pH 7) was put on a Dowex 1-X4 resin column, Cl^- form. (The resin had been previously equilibrated with a sodium chloride solution containing Tween 80 (polyoxyethylene sorbitan monooleate) or human serum albumen.) Such equilibration substantially reduces adsorption of iodinated protein on resins and gels. The column was then centrifuged. The iodide remained adsorbed on the resin column and the radioiodinated protein was contained in the centrifugate.

Radioiodide has also been separated from iodine-labeled proteins (human serum albumin and Bovine IgG) by thin-layer gel filtration on Sephadex G-200 and G-75 superfine layers (163). (Sephadex is a modified dextran.) The protein solutions were applied in amounts of 5-10 μℓ to the Sephadex layers and the angle of slope of the layers was 15°. Elution was carried out with 0.2 M Tris buffer (Tris = tris (hydroxymethyl) aminomethane) at pH 8.0. The buffer contained Tween 80. Separations took 45-60 min on Sephadex G-75 and 3-4 h on Sephadex G-200 and were particularly good on the latter.

The method has also been used for the separation of radioiodinated proteins in an iodination mixture, after removal of iodide.

Excellent separation of iodide from ^{125}I-labeled antipyrene (4-iodo-1,5-dimethyl-2-phenyl-3-pyrazolone) has been obtained by paper chromatography, thin-layer chromatography, and high-voltage electrophoresis (381).

Both descending and ascending paper chromatography have been employed on Whatman 3MM paper. The chromatograms were developed with an n-butanol-acetic acid-water (volume ratios, 120:30:50) solvent system.

The thin-layer chromatography was carried out on plates coated with silica gel or cellulose containing a fluorescent indicator. A chloroform-methanol-15 M ammonium hydroxide (volume ratios, 100:30:5) system was used for developing the chromatograms.

The high-voltage electrophoretic experiments were run on Whatman 3 MM paper wetted with barbital buffer at pH 8.6. Electrophoresis at 10°-15° at either 3000 V for 20 min or 2000 V for 30 min resulted in iodide migration of 14-15 cm.

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