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Quick Methods for Radiochemical Analysis

INTERNATIONAL ATOMIC ENERGY AGENCY, VIENNA, 1969

QUICK METHODS
FOR RADIOCHEMICAL ANALYSIS

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QUICK METHODS FOR RADIOCHEMICAL ANALYSIS

INTERNATIONAL ATOMIC ENERGY AGENCY
VIENNA, 1969

QUICK METHODS FOR RADIOCHEMICAL ANALYSIS
(Technical Reports Series, No. 95)

ABSTRACT. A report compiled by the IAEA based on information provided by several Member States at the invitation of the Agency.

Contents: Determination of caesium-137; Determination of iodine-131; Determination of phosphorus-32; Determination of plutonium; Determination of strontium-89 and strontium-90; Determination of total activity.

Entirely in English.

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QUICK METHODS FOR RADIOCHEMICAL ANALYSIS
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FOREWORD

Quick methods for radiochemical analysis, of adequate precision for the assay of a limited number of biologically important radionuclides, are important in the development of effective monitoring programs, particularly those that would be applied in emergency situations following an accidental release of radioactive substances.

Methods of this type have been developed in a number of laboratories and are being altered and improved from time to time. They are often not published in the open literature even though they could be of considerable interest to other laboratories and institutes. Several rapid methods are given in a report prepared jointly by the World Health Organization, the Food and Agriculture Organization of the United Nations and the International Atomic Energy Agency. This report was published under the title "Methods of Radiochemical Analysis" by WHO in Geneva, in 1966.

A panel of experts which met in Budapest in 1966 to advise the Agency on the co-ordination of research in radiation protection recommended that particular attention be given to these rapid radiochemical techniques. The Agency thereupon invited several Member States to provide detailed information on any such procedures that might have been developed in their laboratories. From the information thus obtained a number of methods have been selected that appear to meet the criteria of speed and economy in the use of materials and equipment, and seem to be eminently suitable for those radionuclides that might be of major interest from the point of view of assessing the potential dose to persons following a serious dispersal of contamination.

The material provided has been edited with the aim of achieving a more uniform presentation. It is hoped that the booklet will prove to be of general interest, and Member States are invited to send to the Agency any additional material or modified procedures that they would wish to be considered for inclusion in future revised editions.

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DETERMINATION OF CAESIUM-137

DETERMINATION OF CAESIUM-137 IN WATER

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The method is based on the coprecipitation of caesium with Prussian blue. Since the number of operations is small, the procedure makes it possible to achieve a high degree of yield and high radiochemical purity.

PROCEDURE

An amount of 5-10 mg of caesium carrier and 1.6 ml of 0.5M $FeCl_3$ is added to 1 litre of water, acidified with several millilitres of conc. HCl or HNO_3. The solution is stirred vigorously with a stirrer and Prussian blue is precipitated with approx. 1.5 ml of 0.5M $Na_4Fe(CN)_6$. The total volume of the precipitating agent is added in drops within about 1 min. After 1-2 min of stirring the precipitate is allowed to sediment. The clear solution above the precipitate is filtered using a fine filter paper and the remainder is separated by centrifuging. The precipitate is washed by means of decantation with small portions of distilled water till the reaction of the washing liquid is neutral. The precipitate, including the small amount on the filter paper, is completely dissolved in 16 ml of saturated EDTA solution (pH10) and the mixture is then placed on a boiling water bath. The brown-red solution is diluted by adding an equal volume of water, and Prussian blue is re-precipitated by adding conc. HCl until pH0-pH1.

Specimens obtained by coprecipitating caesium-137 with Prussian blue are suitable for gamma spectrometric determinations even without purifying the precipitate by means of re-precipitation. For the determination of the beta activity of the specimens the following refining procedure is necessary:

After dissolving the Prussian blue precipitate several times in alkaline EDTA solution (pH10) two alternative procedures may be followed:

(a) Caesium is extracted twice, using each time 3 ml of 0.1M sodium dipicrylaminate in nitrobenzene; 5 min of extraction are sufficient. After washing the organic phase with strongly diluted EDTA solution, caesium is re-extracted three times, using each time 1 ml of 1M HNO_3. After evaporation of the solution to dryness on a measuring plate, specimens with very low mass-per-unit area are obtained.

(b) Caesium tetraphenyl borate is precipitated from the hot solution with approx. tenfold excess of sodium tetraphenyl borate, the precipitate is separated by centrifuging or filtration and washed with 1% acetic acid solution.

Additional information to this method may be found in the literature quoted below.

REMARKS

The analysis together with the final gamma spectrometric measurement takes about 60 min. The beta activity measurement takes about 30 min more.

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DETERMINATION OF CAESIUM-137 IN LIQUID MILK

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A technique for determining caesium-137 in milk on the basis of caesium-137 sorption has been adapted according to the method of Broadbank. The caesium carrier is not added, because its addition decreases the sensitivity of measurement.

Two procedures were used for the assay of caesium-137 in milk:

(a) Sorption of caesium-137 from the solution of milk ash dissolved in 3M HNO_3

(b) Sorption of caesium-137 from whey.

In both methods the radioactivity of caesium-137 was determined directly on the ammonium molybdophosphate (AMP) bed by means of counting.

PROCEDURE

1. Preparation of ash solution

The milk is evaporated and ashed below 450°C to avoid loss of caesium. 10 g of milk ash is damped with 10 ml of distilled water and then dissolved in 60 ml 0.5N HNO_3 (prepared from HNO_3, sp. gr. 1.4).

The ash solution is filtered through paper pulp into a graduated cylinder. Water is added to adjust the volume of the solution to 150 ml.

2. Preparation of whey from liquid milk

250 ml of milk is poured into a 400 ml beaker. At the same time 25 ml of diluted CCl_3COOH (100 g of CCl_3COOH + 50 ml of H_2O) is added and the sample is mixed well. After about 10 min 50 ml of HNO_3 (sp. gr. 1.4) is added and the sample is stirred and left for 30 min. The mixture is filtered using vacuum through a bed of filter pulp on a Büchner funnel and washed with a small amount of water. The solution is placed in a 500 ml graduated cylinder, 40 ml of conc. HNO_3 (sp. gr. 1.4) is added and the volume of the solution is adjusted to 500 ml with water.

3. Preparation of a bed of ammonium molybdophosphate (AMP)

7 ml of NH_4NO_3 solution (71.4 g of NH_4NO_3 in 134 ml of HNO_3 of sp. gr. 1.4, diluted to 1000 ml with distilled water), 3 ml of 10% solution of $(NH_4)_6Mo_7O_{24}.H_2O$, and 1 ml of $NH_4H_2PO_4$ (3.62 g of $NH_4H_2PO_4$ in 500 ml of H_2O) are mixed in a 50 ml beaker, heated at 80°C for 20 min and then cooled to room temperature. The yellow precipitate of AMP (120 mg) is collected in a radiochemical funnel on a bed of paper pulp and washed with distilled water to remove the acid. The diameter of funnel surface should

fit the surface of the window of a GM counter (about 2.0 cm in diam.). The AMP bed should cover the surface of the filter.

4. Sorption of caesium-137 on the AMP bed and counting

The solution of ash or whey is filtered under reduced pressure through the prepared bed for at least 20 min. The filter is then taken out together with the bed of AMP, placed on a dish and counted, using an end-window GM counter with a mica window of 1.5 mg/cm^2 thickness.

The standard preparation of caesium-137 is also made on a bed of AMP. For this purpose a standard solution of ^{137}CsCl of a known activity is prepared in 3M HNO_3. The solution is filtered through a bed of AMP. The conditions for sorption of caesium-137 on the AMP bed are to be identical with those applied to the unknown solutions examined.

The specific activity of milk is expressed in pCi/litre. It is calculated from the following formulae:

Sorption of caesium-137 from the solution of ash:

$$a_m = \frac{N_p a_w X 100}{N_w b Y}$$

Sorption of caesium-137 from whey:

$$a_m = \frac{N_p a_w 400}{N_w Y}$$

where
N_p = counting rate for the sample (counts/min)
N_w = counting rate for the standard solution (counts/min)
a_w = activity of the standard solution (in pCi)
b = weight of ash
X = content of ash (g/litre)
Y = recovery of caesium-137 (in %).

REMARKS

This method permits the determination of caesium-137 activity greater than 200 pCi in 1 litre of milk. Recovery of caesium-137, as determined by this method, is 75.9 ± 7.8%. The determination of caesium-137 can be completed in 3 h.

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RAPID METHOD FOR DETERMINATION OF CAESIUM-137 IN WATER AND URINE

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The method is selective in the presence of several nuclides and enables us to determine 2.2×10^{-5} μCi and 5.1×10^{-5} μCi of caesium-137 per millilitre of water or urine samples respectively; it is especially convenient for routine measurements of personnel contaminations.

The method is based on ion exchange of caesium-137 on ammonium molybdophosphate (AMP) mats. A modification of the method permits the detection of caesium-137 in water in amounts of approximately 1 pCi. The modification consists in changing the size of AMP mats and omitting the asbestos. This enables the measurements of the samples to be carried out in a low-background counting unit.

PROCEDURE

Prepare the following working solutions:

(a) NH_4NO_3 solution: dissolve 71.4 g of NH_4NO_3 in 95.6 ml of conc. HNO_3 (sp. gr. 1.5) and adjust with H_2O to 1000 ml.

(b) $NH_4H_2PO_4$ solution: dissolve 3.62 g of $NH_4H_2PO_4$ in 500 ml H_2O.

(c) Ammonium molybdate solution: dissolve 26.5 g of $(NH_4)_6Mo_7O_{24}.4H_2O$ in 250 ml H_2O.

Add to a 250 ml beaker 90.3 ml of NH_4NO_3 solution and 38.7 ml of ammonium molybdate solution. Stir well and heat for 17 min in a water bath at 80°C. Add 0.5 g of Gooch asbestos and 12.9 ml of $NH_4H_2PO_4$ solution. Stir and heat at 80°C for 10 min. Then cool to room temperature. This mixture should be freshly prepared before analysis.

Place a soft filter paper on the 9 cm diameter sintered filter funnel 25G2, stir the above mixture well and pour it into the funnel. Wash the beaker with 20 ml of water and add the washings to the funnel. Suck off most of the liquid using vacuum.

Place a hard filter paper on the yellow surface of asbestos with AMP and suck through 50 ml of water. The suction should be strong enough to avoid the separation of layers of the mat. Stop the suction and pour on the mat the water or urine sample prepared as follows:

(a) Urine sample: to 1000 ml of urine add 100 ml of conc. HNO_3

(b) Water sample: acidify 1 litre of water sample with 100 ml of conc. HNO_3 and add 250 mg of NaI.

Drain the sample through the AMP mat and control the suction so as to obtain a flow rate of about 50 ml/min. After draining off most of the sample, wash the mat with 200 ml of diluted HNO_3 (1 + 10). Discard the top filter paper (which should not be coloured by the yellow part of the

mat). Strip the mat from the filter funnel and wrap it around a glass GM counter.

Calculate the results, using blank AMP mat counts per minute as a background.

PROCEDURE FOR A MODIFIED METHOD

The working solutions are prepared in the same way. Add to the 50 ml beaker 7 ml of NH_4NO_3 solution and 3 ml of ammonium molybdate solution. Stir well and heat for 15 min in a water bath at 80°C. Add 1 ml of $NH_4H_2PO_4$ solution, stir and heat at 80°C for 10 min. Then cool to room temperature. This mixture should be prepared freshly as required. Place a hard filter paper on a 25 G2 sintered disc and mount it in a demountable filtration funnel. Stir the mixture well and pour it into the funnel. Wash the beaker with 5 ml of water and add the washings to the funnel. Suck off most of the liquid using vacuum.

On an AMP mat prepared in this way pour rainwater which is acidified with 100 ml of conc. HNO_3 per 1000 ml of water and in which 250 mg of NaI per 1000 ml is dissolved. The volume of the demountable filter funnel is usually much smaller than that of the sample. When adding new portions of the sample, special care should be taken to prevent disturbing the mat surface. After sucking off the last portion of the sample, wash the filter funnel with 10 ml of 96% ethanol. Suck off the mat to dryness and transfer it to the bottom of an inverted beaker. Then cover the mat with collodion film prepared as follows: Mix 50 vol.% of collodion with 25% of amylacetate and 25% of ether and store the mixture in a tightly closed bottle. Drop one drop of the mixture upon the surface of the distilled water in a 300 ml crystallizer. When the collodion film begins to contract, take it out of the water with a wire ring of 4 cm in diam. so that the upper surface of the film remains dry. Such a film is approximately 30 $\mu g/cm^2$ thick. Cover the AMP mat with the dry surface of the film and fold the edges of the film below the paper base of the AMP mat.

Transfer the mat to a circular piece of scotch tape, 2.6 cm in diam., and cover it with a paper ring so that the AMP mat is fully exposed and the edge of the collodion film and scotch tape are completely covered.

Place the AMP mat on the plastic disc (1.8 cm in diam.) of the low-background beta counting unit and measure the beta activity of the sample.

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DETERMINATION OF IODINE-131

RADIOMETRIC DETERMINATION OF IODINE-131 IN WASTE WATERS

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The iodine is extracted by chloroform and this extract is gamma counted if the activity is higher than $10^{-4}\mu$Ci/ml. If the activity is lower, the iodine is re-extracted and the AgI is precipitated and beta counted. The yield of enrichment and separation is determined by spectrophotometry.

PROCEDURE

An amount of 1-10 ml of the sample is placed in a 60 ml separating funnel and the solution is neutralized (using indicator paper) with 4N HNO_3 or 4N NaOH solution. Then 10 ml of 2M Na_2CO_3 solution is added, 0.2 ml of standard solution (13 g NaI per 1 litre of water containing 4-5 mg $NaHCO_3$) if the activity is higher than 1×10^{-4} μCi/ml, or 2 ml if the activity is lower, and 1 ml of NaClO solution (5% of chlorine). The mixture is shaken for 5 min and 5 ml of conc. HNO_3 is added dropwise. After the reduction of iodate with 9 ml hydroxylamine hydrochloride solution (70 g/litre) the iodine is extracted with 10 ml of chloroform and the aqueous phase is rejected. 10 ml of water is added to the separating funnel containing iodine in $CHCl_3$ solution and a few drops of $NaHSO_3$ solution (104 g/litre) until the $CHCl_3$ layer is colourless. It is then rejected.

(a) The activity of the sample is believed to be higher than 1×10^{-4} μCi/ml

1 ml of 6N HNO_3, 4-5 drops of $NaNO_2$ solution (69 g/litre) and 10 ml of $CHCl_3$ are added in turn to the aqueous iodide solution in the separating funnel. After shaking the $CHCl_3$ layer is transferred to a 20 ml volumetric flask. The funnel is washed with 5 ml of $CHCl_3$ which is transferred to the volumetric flask. The volume is then filled up to the mark with $CHCl_3$.

An aliquot of the $CHCl_3$ solution is then gamma counted using a comparative sample with known content of iodine-131.

The recovery of iodine is checked chemically. The comparative solution is prepared as follows: 0.2 ml of standard solution is placed in the 60 ml separating funnel and 1 ml of 6N HNO_3, 10 ml of water and 4-5 drops of $NaNO_2$ solution (69 g/litre) are added. The iodine is extracted three times, each time using 5 ml of $CHCl_3$, and the organic phases are collected in a 20 ml volumetric flask. After filling up to the mark the absorbancy of the comparative solution and that of the sample solution are measured at 510 nm in a cuvette of 1 cm path length. The ratio of absorbancy gives the recovery factor.

(b) The activity of the sample is believed to be lower than 1×10^{-1} μCi/ml

1 ml of 6N HNO_3 is added to the aqueous solution of iodide and the mixture is heated nearly to boiling. Then 2 ml of 0.1N $AgNO_3$ solution is added and the precipitate is filtered through the filter paper on a special demountable type of Büchner funnel. The precipitate is washed three times with 5 ml of water, three times with alcohol, dried at 110°C, beta counted, using a comparative source of known iodine-131 content, and finally weighed.

The chemical recovery is calculated from the ratio of the mass obtained by weighing the AgI recovered from the sample with the theoretical value (37 mg) corresponding to the added volume of standard solution of NaI.

REMARKS

The method is applicable to the following iodine concentrations:
In case (a) - higher than 1×10^{-4} μCi/ml;
In case (b) - ranging from 2×10^{-6} μCi/ml to 10^{-4} μCi/ml.

The time necessary to perform two parallel determinations is about 30 min.

DETERMINATION OF IODINE-131 IN MILK

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PROCEDURE

Improved method of dry-ashing of milk

The simple ignition of the sample at high temperature results in a loss of iodine through evaporation. To minimize the iodine loss, a NaOH solution is added to the milk prior to ignition. It is recommended to add a 1N NaOH solution to the milk in the ratio of 1:19.

Determination of organic and inorganic iodine in milk

According to the method reported by Bergh [2nd Int. Conf. peaceful Uses atom. Energy (Proc. Conf. Geneva, 1958) 18, UN, New York (1958) 508], organic and inorganic iodine are determined separately. However, it is confirmed by our experiment that the inorganic iodine, obtained by oxidizing iodide-ions with $NaNO_2$, combines with casein and fat during the later chemical treatment according to Bergh's method. To determine the individual forms of iodine in milk, it is recommended to adopt the ordinary separation method of fat and protein, such as ether extraction of fat and trichloroacetic acid (TCA) precipitation of protein.

Determination of inorganic iodine

To determine inorganic iodine in fresh milk, a composite system of ion exchange and CCl_4 extraction is recommended. An amount of 3-5 ml of Dowex 1X8 resin (20-50 mesh, NO_3^- form) is charged in a column of 1 cm diam. and 30 cm length. 200 ml of fresh milk containing 10 mg of iodine carrier (NaI form) is passed through it. After washing the resin with warm water, the iodine is eluted with 400-500 ml of 2N $NaNO_3$ (or 200 ml of 4N $NaNO_3$). 100 ml of CCl_4, 1-2 g of $NaNO_2$, and 5 ml of HNO_3 are added to the eluate and shaken vigorously in the separating funnel. The iodine, extracted to CCl_4 layer, is transferred to water layer by shaking with water containing a few drops of SO_2 solution. After the volatilization of SO_2 by heating, AgI is precipitated by adding $AgNO_3$ solution, filtered (or centrifuged) and counted.

A milk sample deteriorated during a long period of transport is hard to pass through an ion-exchange resin column because of its pasty condition. Therefore, a method with TCA treatment and CCl_4 extraction can be recommended. To 200 ml of deteriorated milk 90 ml of 30% TCA solution is added and the mixture is centrifuged. 50 ml of CCl_4 and 2 g of $NaNO_3$ are added to the supernatant and the subsequent treatment of the mixture is the same as that outlined above.

Estimate of total iodine-131 from the amount of inorganic iodine-131 in milk

If the assumption is accepted that about 90% of iodine in milk exists in inorganic form, the total iodine-131 concentration could be easily calculated from the amount of inorganic iodine-131 in milk. The radioisotope tracer experiments carried out on cows confirmed this assumption. Carrier-free iodine-131 (NaI form) was injected into the jugular veins of three Holstein cows. The radioactivities of the fat, protein and whey fractions separated from the milk were counted. During two weeks after injection of a dose of iodine-131 no iodine-131 was detected in the fat fraction, 5-10% of the iodine-131 content in milk was found in the protein fraction and the rest was determined in the whey.

All determinations of iodine-131 for the evaluation of this experiment were made using the method described above, and have confirmed its utility for such purposes.

Rough estimation of total iodine-131 content in milk by gamma spectrometry

The gamma spectrum is determined using 1-2 litre of the milk sample. When a crystal of 7.5 × 7.5 cm NaI(Tl) is used in the above determination, the detectable limit of iodine-131 is 100 pCi/litre.

REMARKS

Two parallel determinations of iodine-131 take about $1\frac{1}{2}$ h for fresh milk samples; for deteriorated milk samples up to about 2 h.

DETERMINATION OF RADIOACTIVE IODINE IN RAINWATER AND URINE

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The method is based on isotopic exchange between stable and radioactive iodine on asbestos mats impregnated with silver iodide. The detection limit of this method is 5 pCi of iodine-131 in a one litre sample and the mean recovery is 97%.

PROCEDURE

Urine analysis

The stock suspension for preparing the isotopic exchange mats is made as follows: To 1500 ml of distilled water add 5 ml of conc. H_2SO_4, 1.86 g of $NaI.2H_2O$ dissolved in 25 ml of 1N H_2SO_4, and 30 g of Gooch asbestos. After thorough mixing, add dropwise, stirring vigorously, 3.75 g of $AgNO_3$ dissolved in 5 ml of distilled water, then introduce the Whatman filter paper of 90 mm diam. into the sintered glass filter funnel No. 25G2, add 100 ml of stock suspension, cover with a second filter paper and apply suction. After draining off most of the liquid, wash the mat twice with 25 ml portions of 0.1N H_2SO_4.

Determination of radioactivity

Pour the urine, acidified with 3 ml of 0.1N H_2SO_4 per 1000 ml, carefully on the mat thus prepared and control the suction so as to obtain a flow rate of about 100 ml/min. When the draining of urine through the mat is completed, wash the mat with two 25 ml portions of 0.1N H_2SO_4 and dry under vacuum. Then discard the top filter paper, wrap the remaining asbestos mat around a cylindrical glass GM tube and count.

Rainwater analysis

The same procedure is used for the determination of radioiodine in rainwater. If the volume of the collected water samples is less than 1000 ml, dilute the sample to 1000 ml with distilled water, filter through the filter paper, add 3 ml of conc. H_2SO_4 and 14 mg of crystalline NaCl.

A higher sensitivity is achieved by this method when 14 g NaCl is dissolved in each litre of the rainwater sample.

The time required for two parallel determinations is about 1 h.

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DETERMINATION OF IODINE-131 IN WATER SAMPLES

UNITED STATES ATOMIC ENERGY COMMISSION,
WASHINGTON, D.C.,
UNITED STATES OF AMERICA

Iodine is extracted from the water sample and counted in a bottle, in a well NaI(Tl) crystal. The determination can be carried out in a very short time.

PROCEDURE

Pour a 500 ml sample into a one litre separating funnel, add 10 mg iodine carrier (1 ml) and acidify with 6 ml conc. H_2SO_4. Add 350 ml CCl_4 and 6 g $NaNO_2$. Shake well.

Take the CCl_4 layer from this extraction, adjust to 500 ml with CCl_4 and count the activity for 10 min in the well of a NaI(Tl) crystal.

DETERMINATION OF PHOSPHORUS-32

DETERMINATION OF RADIOACTIVE PHOSPHORUS, STRONTIUM, LANTHANUM AND CERIUM IN URINE AND WATER

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The method is based on the sorption of nuclides on phosphate gel mats. The recovery ranges from 77.0 to 99.5% and the detection limit is about 10 pCi/litre.

PROCEDURE

Preparation of stock phosphate gel

25 g of $Ca_3(PO_4)_2$ is dissolved in 150 ml of conc. HCl in an 800 ml beaker and an excess of conc. NaOH solution is then added. The resulting gel is filtered through a sintered glass filter funnel 25G2 and washed twice with 500 ml of distilled water. This stock amount of gel should be stored in a tightly closed beaker.

Determination of radioactivity

Assemble the sintered glass filter funnel 25G2 in a 2 litre flask and cover the sinter with filter paper Whatman No. 41. Adjust the 24 hours urine sample to pH9 with 1N NaOH solution and add 0.5 g of stock phosphate gel. Stir the sample well to attain a homogeneous suspension of gel, pour into the filter funnel and adjust the suction to the rate of 0.2 litre/min. After the filtration is completed suck the mat dry under full vacuum. Then strip the mat from the filter funnel with a spatula, wrap it around the cylindrical glass GM counter and count the beta activity in the sample.

DETERMINATION OF PHOSPHORUS-32 IN URINE

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A method for the determination of total phosphorus-32 by direct beta counting of urine is described, together with more sensitive radiochemical procedures for total phosphorus-32 determination and for phosphorus-32 as inorganic phosphate.

PROCEDURES

1. Determination of total phosphorus-32 by liquid beta counting

The phosphorus-32 content of urine may be determined by counting the untreated sample in the low-background liquid beta counter. A 10 ml aliquot is counted for a period of 1 h, and allowance is made for the contribution from natural potassium-40 by measuring the average counter background while it contains 10 ml urine samples from unexposed people. As the method is not specific for phosphorus-32, it may only be used for people who are exposed exclusively to this particular radionuclide. For a counting period of 1 h the method is nine times less sensitive than the radiochemical method, the minimum detectable level being 360 pCi/litre.

2. Radiochemical determination of total phosphorus-32 in urine

Measure 30 ml of urine into a 250 ml round bottom flask. Add 2 g of $KMnO_4$ and 2 ml of H_2SO_4 (sp. gr. 1.84). Fit a water condenser to the flask and heat under reflux for 2 h. Add saturated oxalic acid solution until the solution becomes colourless. Filter into a 250 ml centrifuge tube. Add NH_3 solution (sp. gr. 0.880) dropwise until a slight turbidity appears. Re-dissolve the precipitate by the addition of a few drops of HNO_3 (sp. gr. 1.42). Add a few drops of Aerosol O.T. (dissolve 5 g of sodium dioctyl sulpho-succinate in 100 ml of water) and warm the solution on a water bath to 60°C. Add dropwise with stirring 60 ml of molybdate reagent. (This is prepared by mixing solution A and solution B in a 2:1 ratio. Solution A: dilute 200 ml of nitric acid (sp. gr. 1.42) to 500 ml with water. Solution B: dissolve 50 g of ammonium molybdate in 200 ml of water and add 40 ml of ammonia

solution (sp. gr. 0.880).) Continue to stir for 10 min and allow the solution to cool for a few hours. Alternatively the solution may be cooled in running water for 30 min. Centrifuge and discard the supernate. Wash the precipitate with 0.5M NH_4NO_3 solution. Centrifuge and discard the washings.

Dissolve the precipitate in 10 ml of 9M NH_3 solution. A blue colour will sometimes develop which disappears on standing for about 5 min. Add a few drops of methyl-red indicator and acidify with 3M HCl solution.

Add slowly with stirring a few drops of Aerosol and 25 ml of magnesia reagent. (Dissolve 25 g of magnesium chloride and 50 g of ammonium chloride in 250 ml of water. Add a slight excess of ammonia solution and filter if any precipitate forms. Acidify the solution with 3M hydrochloric acid and add 2 ml of hydrochloric acid (sp. gr. 1.18) in excess. Dilute to 500 ml with water.) Heat to boiling. Add 25 ml of 9M NH_3 solution with stirring. Continue stirring for 10 min and allow to stand for a few hours (or cool in running water for 30 min).

Centrifuge and discard the supernate. Mount the precipitate on a suitable tray. Count in a low-background beta counter calibrated with sources of known activity. The detectable level is 40 pCi/litre.

3. Radiochemical determination of inorganic phosphorus-32 in urine

Measure 50 ml of urine into a 100 ml centrifuge tube. Adjust the pH to a value of 5 by adding either 3M HNO_3 solution or 3M NH_3 solution. Indicator paper may be used to determine the pH. Add 10 ml of $Ca(NO_3)_2$ solution followed by NH_3 solution (sp. gr. 0.880) dropwise with stirring until no further precipitate is formed. Centrifuge and discard the supernate. Wash the precipitate twice with 0.05M NH_3 solution discarding the washings. Dissolve the precipitate in the minimum quantity of 3M HNO_3 solution and adjust the pH to a value of 5 with 3M NH_3 solution. Indicator paper may be used to determine the pH.

Add 50 ml of sodium acetate buffer solution. Heat almost to boiling. Add 10 ml of $Pb(NO_3)_2$ solution with stirring. Boil gently to coagulate the precipitate; cool; centrifuge and discard the supernate. Wash the precipitate with 3M acetic acid. Centrifuge and discard the washings. Mount the precipitate on a suitable tray. Count in a low-background beta counter calibrated with sources of known activity. Alternatively the precipitate may be dissolved in 10 ml of 3M NHO_3 solution and counted in a low-background liquid counter. The minimum detectable level is 25 pCi/litre with conventional beta counting and 70 pCi/litre with liquid beta counting.

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DETERMINATION OF PLUTONIUM

DETERMINATION OF PLUTONIUM IN WASTE WATERS BY ALPHA-PARTICLE MEASUREMENTS

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After oxidation of plutonium to Pu(VI), calcium is precipitated as CaF_2 and separated. The Pu(VI) is then reduced to Pu(III) and precipitated with LaF_3. After dissolution in HNO_3 in the presence of $Al(NO_3)_3$ and oxidation of Pu(III) → Pu(IV), the latter is extracted, using thenoyltrifluoracetone (TTA). The extract is evaporated, ashed and alpha counted.

PROCEDURE

25 ml of the waste water sample is placed in a 50 ml polyethylene tube. 5 ml of conc. HCl and 2 ml of 2N $K_2Cr_2O_7$ solution is added and the solution is heated to 80°C for 30 min. After cooling 5 ml of HF solution (sp. gr. 1.14) is added, the mixture is thoroughly stirred, using platinum wire, and after a few minutes centrifuged for 5 min. The solution is transferred into a second polyethylene tube, the precipitate is washed twice with 1 ml of 1N HCl + 1N HF solution and centrifuged. 4 ml of hydroxylamine hydrochloride solution (69.5 g/litre) are added to the combined solution containing plutonium, and the mixture is heated to 80°C for 15 min. After cooling 0.5 ml of $La(NO_3)_3$ solution (31.2 g/litre) is added. The mixture is stirred, using platinum wire, and after 5 min centrifuged for 5 min. A new portion of 5 ml of $La(NO_3)_3$ solution is added and these operations are repeated. The aqueous solution is rejected and the precipitate is washed twice using 0.5 ml of 1N HCl + 1N HF solution.

After dissolution of the precipitate in 0.3 ml of $Al(NO_3)_3$ solution and 1 ml of 2N HNO_3, 1 ml of hydroxylamine hydrochloride is added and the mixture is then heated to 80°C for 15 min, cooled and transferred into a 30 ml separating funnel. The tube is washed, using 2 ml of 1N HNO_3 and then 2 ml of $NaNO_2$ solution (69 g/litre). After carefully stirring to remove the NO_2 vapours, 5 ml of TTA solution (111 g/1 litre of xylene) is added and the mixture is shaken for 10 min. The aqueous phase is then rejected and the organic phase washed using 5 ml of 1N HNO_3. The plutonium is then re-extracted, using 5 ml of 10N HNO_3 and shaking the mixture for 10 min, and the aqueous phase is separated. The organic phase is washed twice, using each time 2 ml of 10N HNO_3 and shaking for 3 min. All three portions of aqueous solution are combined and evaporated carefully on a watch-glass, calcinated and alpha counted, using a ZnS scintillation counting device.

To determine the recovery of plutonium it is usual to perform four parallel determinations, two with the addition of 1 ml of standard Pu-solution (1×10^{-4} μCi/ml) and two without. If the resulting mean values of counting

numbers are N_1 (without) and N_2 (with), respectively, the concentration of Pu in the sample is given by the formula

$$4 \times 10^{-6} \frac{N_1}{N_2 - N_1} \mu Ci/ml$$

REMARKS

The method is applicable for concentrations of plutonium greater than 5×10^{-8} μCi/ml.

The time required to perform four parallel determinations is about $3\frac{1}{2}$ h.

When the amount of calcium is lower than 200 mg/ml it is not necessary to separate this element and the procedure should start with the coprecipitation of PuF_3 with LaF_3.

DETERMINATION OF PLUTONIUM IN URINE

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The method is based on LaF-precipitation, extraction with thenoyltrifluoracetone (TTA) and electrolysis. The detection limit is 4 pCi Pu/200 ml sample.

PROCEDURE

200 ml of a urine sample in a 1 litre container is boiled with 20 ml of 10M HNO_3 solution and 2.5 ml of La-carrier solution (40 mg La) and evaporated to 50 ml. After cooling in ice water the solution is transferred to a 100 ml polyethylene vessel which contains 25 ml of 2M HF solution. The precipitate is stirred for 1 min. Two minutes later the mixture is centrifuged for 2 min and the supernatant solution is discarded. The precipitate is dissolved in 50 ml of 2M HNO_3 solution, mixed with 5 ml of 20M HF solution and stirred vigorously. After 2 min the precipitate is centrifuged for 2 min and the supernatant solution is discarded. The precipitate is dissolved in 20 ml of (2M) $Al(NO_3)_3$/(0.5M) HNO_3 solution. The solution is transferred to a 150 ml separating funnel which contains 10 ml of 0.45M thenoyltrifluoracetone (TTA) solution in benzene. To extract the plutonium the mixture is shaken for 10 min. The aqueous phase is discarded, the organic phase is washed twice with 5 ml of 2M HNO_3 solution for 2 min each time. The organic phase is evaporated to dryness and incinerated. The residue is dissolved in a few drops of 10M HNO_3 solution and evaporated to dryness. The residue is transferred with 1 ml of water to an electrolysis vessel and rinsed with 4 ml of 6M NH_4Cl solution. To this solution two drops of 10M HCl solution are added, and the plutonium is separated by electrolysis, with a current of 2.5-3 A/cm^2, on a stainless steel disc used as cathode. After 20 min the electrolysis is interrupted by the addition of 1 ml of 15M NH_3 solution, the stainless steel disc is rinsed with distilled water and dried. This sample is counted for 30 min using a solid state detector.

The chemical efficiency is 45%, the detection limit is 4 pCi Pu/200 ml sample.

The whole analysis, including the measurement of the sample, takes approximately 2 h.

DETERMINATION OF PLUTONIUM IN URINE

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The method was developed for application to urine analysis. It may also be used to estimate plutonium in effluents and environmental samples.

PROCEDURE

To the total volume of urine sample add 10 ml of conc. HNO_3 and 10 ml of $NH_2OH.HCl$ solution (50 g/litre). Allow the solution to stand for 15 min. Add 10 ml of conc. H_3PO_4 and heat to boiling.

Add 10 mg of Bi carrier solution dropwise with constant stirring. Allow the precipitate to settle overnight. Decant the supernate and collect the precipitate by centrifuging. Wash the precipitate once with a solution of HNO_3-H_3PO_4 and finally with water.

Add conc. HNO_3 and transfer the precipitate to a centrifuge tube. Heat under infra-red lamp to destroy organic matter. Dilute with distilled water to precipitate $BiPO_4$. (Add 2-3 drops of 10% PO_4^{-3} solution if $BiPO_4$ is not formed readily.) Centrifuge, wash the precipitate with water and finally with acetone, and then dry.

Add 4 ml of conc. HNO_3, 3 ml of distilled water and 1 ml of 1$\underline{M}$ $NaNO_2$ solution. Heat in boiling water bath for 10 min. Pass the solution through Dowex-1 (5 cm × 0.25 cm diam.) conditioned with 8$\underline{N}$ HNO_3 solution, at a flow rate of 0.5 ml/min. Pass a further 15 ml of 8$\underline{N}$ HNO_3 solution through the column followed by 15 ml of 8$\underline{N}$ HCl solution to remove any thorium present in the column. Elute the column with 15 ml of $NH_2OH.HCl$ solution (1.5$\underline{N}$ $NH_2OH.HCl$ in 1$\underline{M}$ HCl) and collect the eluate in a centrifuge tube.

Add 0.5 mg Fe carrier to the eluate and precipitate $Fe(OH)_3$ by the addition of NH_3 solution. Heat to coagulate the precipitate and centrifuge. Wash the precipitate free from ammonia. Transfer to a weighed stainless steel planchet. Dry under infra-red lamp. Flame. Cool, weigh and count for alpha activity.

DETERMINATION OF PLUTONIUM IN WASTE SOLUTIONS

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The procedure is based on a double coprecipitation method which does not require an evaporation process and therefore gives the result very quickly. Plutonium is coprecipitated with iron-lanthanium-hydroxides and then with lanthanium trifluoride. The fluoride precipitate is uniformly deposited on a special counter plate and alpha counted.

PROCEDURE

500 ml of waste water is transferred into a 1000 ml beaker and 30 ml of conc. HCl, 10 ml of 1M $FeCl_3$ solution, 10 ml of 0.01M $La(NO_3)_3$ solution, and 1 ml of 0.01M hydroxylamine hydrochloride solution are added. The mixture is heated on a hot plate. After cooling, $Fe(OH)_3$ and $La(OH)_3$ are precipitated together with plutonium by addition of NH_3 solution. The solution should be sufficiently basic to precipitate all the ferric ions. After 1 h the supernatant is discarded by decantation to leave a volume of less than 50 ml, and the rest is separated by centrifugation.

The precipitate is dissolved in 2 ml of 12M HCl solution and this solution is transferred to a 20 ml polyethylene centrifuge tube.

1 ml of 13M HF solution is added. The precipitate of LaF_3 containing PuF_3 is separated by centrifuging for a few minutes at 2000-3000 rev/min. The supernatant is removed with a pipette, and the precipitate is washed with a small amount of water.

A small amount of water is added to obtain a slurry, which is spread on a counting plate and dried by an infra-red lamp. Counting time is between ten minutes and one hour, depending on the alpha activity.

Usually uranium is also present in a plutonium laboratory, and consequently the liquid wastes contain both elements. However, the uranium is almost completely masked when the LaF_3 precipitates and the alpha emitters on the counting plate are merely plutonium and americium.

As the maximum permissible concentration of Pu and Am in water is of the same order, one can determine the content of both elements and express it as plutonium content.

The concentration is determined using the following formula:

$$\text{Ci/ml} = \frac{C}{(2.22 \times 10^6)\ C_{eff}\ A_{cf}\ W}$$

where

C = alpha counts/min
C_{eff} = counting efficiency of the counter used
A_{cf} = absorption coefficient by the lanthanum trifluoride
W = sample volume (ml).

DETERMINATION OF PICOCURIE AMOUNTS OF PLUTONIUM

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A method for rapid determination of picocurie amounts of plutonium in biological, environmental, filter and other samples.

PROCEDURE

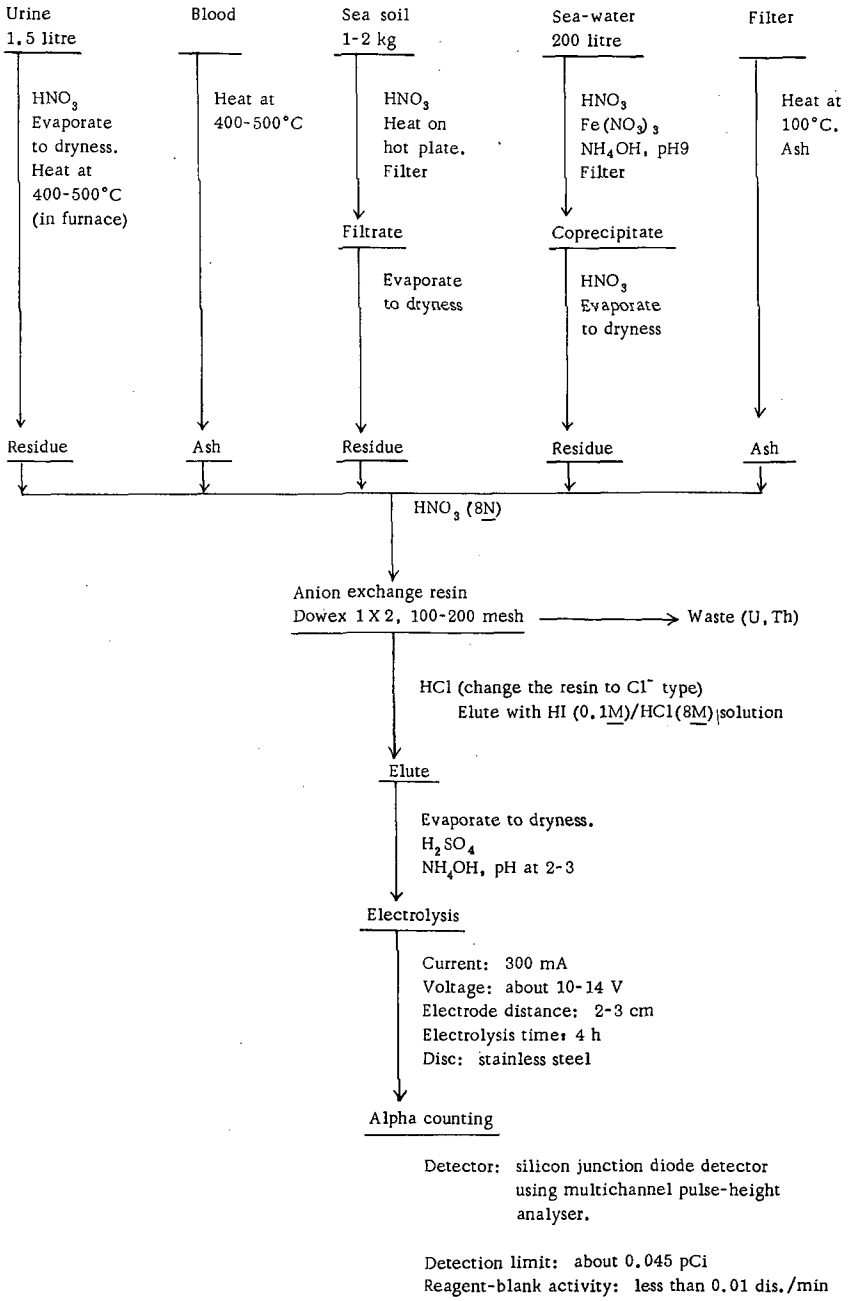
The organic material samples are ashed using wet or dry ashing methods. The environmental samples are first dissolved and then the plutonium is separated by coprecipitation with $Fe(OH)_3$.

Plutonium in the pre-treated sample is then purified with an anion exchange resin (Dowex 1X2) and electro-deposited on a stainless steel disc from an acidic ammonium sulphate electrolyte.

The overall recovery of plutonium with this procedure is $93.4 \pm 7.6\%$.

The spectrum of the electro-deposited plutonium is alpha counted by means of a silicon junction diode detector and a multichannel pulse-height analyser. The low inherent background of the counter is effectively reduced further by counting only alpha particles within the energy range 4.85 to 5.50 MeV.

FLOW DIAGRAM OF PLUTONIUM ANALYTICAL PROCEDURE



DETERMINATION OF PLUTONIUM, NEPTUNIUM AND URANIUM IN URINE

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Chloride complexes of the actinides are extracted from oxidized urine salts with the liquid ion exchanger tri-isooctylamine (TIOA). The actinides are back-extracted and are then counted on low-background solid-state counters.

PROCEDURE

Wet-ash 250 ml of urine with HNO_3 and H_2O_2. Then add 10 ml of 8N HCl solution to the white salts twice, evaporating the solution to dryness each time. Dissolve the salts in 50 ml of 8N HCl solution (with heat) and pour the solution into a separating funnel. Rinse the original container twice with 15 ml of 8N HCl solution and add the rinses to the funnel. Add 10 drops of a 30% H_2O_2 solution.

Add 25 ml of a 10% TIOA-xylene solution to the funnel and shake vigorously for 10 s. Drain and discard the aqueous (lower) layer. (All shaking times are for 10 s except for plutonium stripping.) Rinse the organic phase with 25 ml of 8N HCl solution. Discard the rinse solution. Add 25 ml of warm (50-80°C) 8N HCl - 0.05M NH_4I solution and shake for 1 min to strip plutonium (release pressure after shaking for 5 s). Repeat a second time and combine the two 25 ml solutions.

Strip neptunium with two 25 ml volumes of 4N HCl - 0.02N HF solution and combine the solutions.

Strip uranium with two 25 ml volumes of 0.1N HCl solution and combine the solutions.

Evaporate each strip solution to dryness. Destroy any organic residue by wet ashing.

Dissolve each residue in 1 ml of 4N HNO_3 solution with heat and transfer these solutions and 4N HNO_3 rinse solutions to special stainless steel planchets; evaporate under an infra-red lamp and alpha count each sample using a low-background alpha counting device with large detectors (350 mm^2).

REMARKS

The detection limit of this method is of the order of 0.1 dis./min per 1.5 litre of urine.

Special samples taken after a plutonium-239 incident can be determined rapidly. A 100 ml sample of urine can be oxidized, separated, and counted within four hours to determine uptake and need for further treatment. The TIOA is stripped directly with 0.1N HCl for a total alpha determination.

VERY FAST DETERMINATION OF PLUTONIUM IN URINE

UNITED STATES ATOMIC ENERGY COMMISSION,
WASHINGTON, D.C.,
UNITED STATES OF AMERICA

A method for emergency analysis of plutonium in urine.

PROCEDURE

Place 25 ml of urine sample in a 50 ml centrifuge tube. Add 5 ml of conc. HCl and stir. Add (dropwise with stirring) 0.5 ml of $Ce(NO_3)_3$ solution (2.5 mg Ce/ml). Stir. Add about 2.5 ml of diluted HF solution (1 + 1). Stir for 1 min. Centrifuge for 4 min and pour off supernatant.

Add 5 ml of water to wash the precipitate (do not stir). Centrifuge and pour off supernatant. Transfer the precipitate onto a stainless steel planchet (under heat lamp) with 5 drops of 2N HNO_3 solution. Wash with 5 drops of 2N HNO_3 solution and then with 5 drops of conc. HNO_3. Dry, flame and count in a low-background proportional alpha counter.

DETERMINATION OF PLUTONIUM IN VARIOUS MATRICES

UNITED STATES ATOMIC ENERGY COMMISSION,
WASHINGTON, D.C.,
UNITED STATES OF AMERICA

PROCEDURE

Plutonium is separated as the anionic nitrate complex by batch contact with anion exchange resin. After transfer to a column, the resin is washed first with 7.2N HNO_3 solution and then with 8N HCl - 0.3N HNO_3 solution to remove additional impurities. The plutonium is then eluted with 0.36N HCl - 0.01N HF solution which is evaporated onto a planchet for the assay of plutonium by alpha counting.

The method can be used for the determination of plutonium in samples of water, air, tissue and faeces.

By utilizing an alpha counter with 50% geometry and 0.1 counts/min background, a detection limit of about 1 pCi/sample is obtained with a counting time of 10 min.

DETERMINATION OF STRONTIUM-89 AND STRONTIUM-90

DETERMINATION OF RADIOACTIVE STRONTIUM

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The method described is suitable for milk and water, but is also applicable to ashed biological samples.

PROCEDURES

1. Determination of radioactive strontium in milk

Pour 100 ml of milk into a 250 ml centrifuge bottle. Add 50 g of strontium carrier (5 mg of strontium/ml), 20 mg of barium carrier (10 mg of barium/ml) and, under mechanical stirring, acidify with 100 ml of trichloracetic acid solution (24%), continue stirring for 15 min, centrifuge for 5 min and filter into a second 250 ml centrifuge bottle.

Add 0.5 ml of mixed indicator (0.1 g thymol blue and 0.1 g phenolphthalein dissolved in 100 ml of 50% aqueous ethanol) to the filtrate and neutralize with 6M NaOH solution until the colour changes to yellow, then add 5 ml of 1.5M Na_2CO_3 solution and adjust to pH > 8.5 with NaOH solution (colour changes to red), continue stirring for 15 - 20 min, centrifuge and reject the supernatant liquid.

Dissolve the precipitate in diluted HCl solution, add 3 g of EDTA and 5 g of $(NH_4)_2 SO_4$ and dilute the solution with water to 50 ml. After the addition of conc. NH_3 solution (pH > 10) heat the centrifuge tube in a water bath until the residue is completely dissolved. Then neutralize the solution with 2M and 1M H_2SO_4 solution and adjust exactly to pH4.5 (methyl red). Heat in a boiling water bath for 20 min, stirring occasionally, then cool in ice water, with mechanical stirring, for 15 min, centrifuge and reject the supernatant liquid.

Add 1 ml of 6M NaOH solution and 40 ml of 1.5M Na_2CO_3 solution to the precipitate and heat in a boiling water bath with stirring for 15 min to transform all sulphates to carbonates. Cool in ice water for 10 min, centrifuge and reject the supernatant liquid, wash the precipitate twice with 20 ml of ~0.1M Na_2CO_3 solution.

Dissolve the residue in 1 ml of 2M HCl solution, add 20 ml of water and neutralize with 25% NH_3 solution to adjust to pH4.5 (methyl red), add 1 ml of 6M CH_3COOH solution and 2 ml of 25% CH_3COONH_4 solution and heat in a boiling water bath for 5 min. Add drop by drop with stirring 1 ml of 30% Na_2CrO_4 solution and continue heating for 5 min, cool with stirring in ice water, centrifuge and filter the supernatant liquid through a black ribbon filter paper into a 100 ml centrifuge tube. Reject the residue.

Make the solution alkaline with 25% NH_3 solution, add solid $(NH_4)_2 CO_3$ and heat for 5 min in a boiling water bath with occasional stirring to co-

agulate the precipitate, cool in ice water, centrifuge and reject the supernatant liquid, wash the residue twice with 10 ml of 0.1M Na_2CO_3 solution.

Re-dissolve the precipitate with 1 ml of 2M HCl solution and add 10 ml of 0.1M EDTA solution and 2 ml of yttrium carrier solution (10 mg of yttrium/ml) as hold-back carrier, and make the solution alkaline with conc. NH_3 solution (pH > 10). Add 20 ml of 10% $(NH_4)_2SO_4$ solution and add dropwise with stirring 2M and 1M H_2SO_4 solution to adjust to pH4.5 (methyl red). Note the time of precipitation for the calculation of the yttrium-90 ingrowth! Then allow to stand for 20 min in a boiling water bath, cool in ice water for 10 min, centrifuge and filter through a fine filter paper (blue ribbon), wash with 2 ml of ice-cooled water, with a small amount of sulphuric ions, 5 ml of methanol and 5 ml acetone. Dry in an oven at 120°C for 20 min. Then transfer the filter plus precipitate immediately to a counting dish and count in a low-background beta counter.

If the strontium-90 content is determined by counting the yttrium-90 daughter product, continue as follows: Transfer the filter with the final precipitate of $SrSO_4$ to a 100 ml beaker, add 2 ml of yttrium carrier solution and dissolve with 10 ml of 0.1M EDTA solution and 2 ml of conc. NH_3 solution. Warm gently in a water bath until a clear solution is obtained.

Transfer the solution into a plastic bottle; the strontium is stored for the desired time of yttrium-90 ingrowth.

To separate yttrium-90, the solution is transferred again to a 100 ml centrifuge tube with water and diluted to a volume of about 25 ml.

Add 20 ml of 10% $(NH_4)_2SO_4$ solution and 3 drops of methyl red indicator. Add dropwise with stirring 2M and 1M H_2SO_4 solution to adjust to pH4.5, heat in a boiling water bath for 20 min, cool in ice water for 10 min, centrifuge and filter through a filter paper (blue ribbon). This precipitate is used for the determination of the strontium-90 yield (see "Remarks").

Make the filtrate alkaline by adding 1 ml of conc. NH_3 solution and 20 ml of 5% (wt/vol.) ammonium oxalate solution, stir with a mechanical stirrer, heat to about 80°C and add drop by drop 10% HCl solution until yttrium oxalate precipitates, continue stirring for several minutes, cool and allow the precipitate to settle, filter and wash with 5 ml of cooled water, then with methanol and finally with acetone. Dry for 20 min at 120°C and prepare for counting procedure. Determine the yttrium yield as described under "Remarks".

2. Determination of radiostrontium in water

Pour 100 ml of water into a 600 ml beaker. Add 50 mg of strontium carrier, 20 mg of barium carrier and, with stirring, 55 ml of 2M H_2SO_4 solution and 220 ml of 95% ethanol.

Continue stirring for 15 min, centrifuge in a 250 ml centrifuge tube and wash twice with 10 ml of H_2SO_4 solution in ethanol (0.5M H_2SO_4 solution:ethanol = 3:4).

Dissolve the precipitate by adding 3 g of EDTA and 5 g of $(NH_4)_2SO_4$ and continue from the separation of strontium and barium as sulphates as described in Procedure 1 above.

For many other nuclides present in fall-out the separation of barium as chromate is changed as follows: Dissolve the residue in 1 ml of HCl solution and transfer with 20 ml of water to a 100 ml centrifuge tube, add 1 ml of ferric ion carrier (5 mg Fe/ml) and heat in a boiling water bath for 5 min to remove CO_2. Make alkaline by dropwise adding carbonate-free NH_3 solution and heat for 2-3 min to complete precipitation. Filter through a black ribbon filter paper into a second 100 ml centrifuge tube and neutralize the solution with HCl solution (methyl red indicator), add 1 ml of 6M CH_3COOH solution and continue as described in Procedure 1.

REMARKS

1. Determination of strontium yield

Detach the filter with the $SrSO_4$ from the planchet and place it in a 100 ml beaker, add 10 ml of 0.1M EDTA solution and 4 ml of conc. NH_3 solution and re-dissolve the precipitate by gently heating in a water bath. Add 1 ml of 1M NH_4Cl solution, Erio T indicator and titrate with 0.1M $ZnSO_4$ solution until the colour changes from blue to wine red.

Every millilitre difference between the volume of 0.1M EDTA solution added and 0.1M $ZnSO_4$ solution used in the back-titration corresponds to 8.763 mg of strontium.

2. Determination of yttrium yield

Transfer the filter with the precipitate of yttrium oxalate into a 100 ml beaker, add 5 ml of 0.1M EDTA solution and 25 ml of 22.4% (wt/vol.) triethanolamine solution and re-dissolve the precipitate by gently heating in a water bath. After addition of Erio T indicator titrate with 0.1M $ZnSO_4$ solution until the colour changes from blue to wine red.

Every millilitre difference between the volume of 0.1M EDTA solution added and 0.1M $ZnSO_4$ solution used in the back-titration corresponds to 8.892 mg of yttrium.

DETERMINATION OF STRONTIUM-89 AND STRONTIUM-90 IN WATER SAMPLES

UNITED STATES ATOMIC ENERGY COMMISSION,
WASHINGTON, D.C.,
UNITED STATES OF AMERICA

PROCEDURE

Accurately measure 50-75 ml of sample and place in a 100 ml centrifuge cone. Add Fe carrier and 20 mg Sr carrier. Adjust the pH to ~ 12 with 12N NH_3 solution. Centrifuge, decant, saving precipitate for gamma count if needed. Add saturated $(NH_4)_2 CO_3$ solution or saturated $Na_2 CO_3$ solution until no more precipitation forms. Repeated centrifugings and additions of saturated $(NH_4)_2 CO_3$ and Na_2CO_3 solutions may be necessary to ensure complete precipitation. Centrifuge for about 2 min. Wash with a little water and centrifuge for about 2 min. Decant, saving precipitate. Place centrifuge tube containing precipitate in an ice bath. Carefully add fuming HNO_3 dropwise to dissolve precipitate. Continue adding fuming HNO_3 to a volume of 16 ml. Let stand in the ice bath for 5-10 min.

Centrifuge for about 2 min. Discard supernate. Dissolve precipitate with a few drops of water, adding up to 10 ml. Add 1 ml 12N NaOH solution, then about 25 ml saturated Na_2CO_3 solution, swirl, let stand for 5-10 min and then centrifuge for about 2 min. Decant, discard supernate. Wash once with water. Transfer precipitate into a tared 2.5 cm stainless steel counting dish. Dry under a heat lamp. Reweigh, noting the weight. Count for 10 min in a low-background counter.

The time required to perform the determination is about 1 h.

DETERMINATION OF STRONTIUM IN URINE

UNITED STATES ATOMIC ENERGY COMMISSION,
WASHINGTON, D.C.,
UNITED STATES OF AMERICA

PROCEDURE

Place 25 ml of urine sample in a 50 ml centrifuge tube. Adjust pH to 4 with conc. NH_3 solution/or conc. HCl solution. Add dropwise with stirring 3 ml of Sulkowitch Reagent. (Add 25 g of oxalic acid, 25 g of ammonium oxalate, and 50 ml of glacial acetic acid to 600 ml of distilled water at 40-50°C and stir until dissolved. Cool, make up to 750 ml and filter.) Continue stirring with electric stirrer for 15 min. Remove stirrer. Wash with a few drops of distilled water. (Use a rubber policeman to remove adhering precipitate.) Centrifuge and discard supernatant liquid.

Wash precipitate twice with 3 ml of 0.05% NH_3 solution. (Slurry precipitate with a thin glass rod before centrifuging.) Wash with 3 ml of acetone and discard supernatant liquid. Transfer the precipitate to a 5 cm counting planchet (under heat lamp) using a total of $\approx \frac{1}{2}$ cm^3 water followed by a few drops of acetone. Dry and count in beta counter.

DETERMINATION OF TOTAL ACTIVITY

DETERMINATION OF BETA ACTIVITY INDICES OF WATER

COMMISSION D'ETABLISSEMENT DES METHODES D'ANALYSE,
COMMISSARIAT A L'ENERGIE ATOMIQUE,
PARIS, FRANCE

The procedure determines two conventional indices of water beta activity, the so-called beta radioactivity indices of the soluble and of the insoluble fractions of the sample.

PROCEDURE I

Preparation of standard

2 ml of a standard ($^{90}Sr + ^{90}Y$) solution of about 10^{-4} μCi/ml activity is evaporated on a proper dish and covered with a collodion layer. The radioactive equilibrium of strontium-90 and yttrium-90 is checked.

Determination

The water sample is filtered through a hard filter paper. The volume of the filtrate is noted. The filter paper is dried, placed in a quartz crucible and ashed in an electric furnace (500°C). The ash is transferred to a tray (not more than 50 mg on a 30 mm diam. tray), covered with collodion layer and counted for beta activity using the same geometry factor as for the standard.

100 ml of filtrate is placed in a crystallizer (100 mm in diam.), acidified with conc. HNO_3 up to pH1 and evaporated to about 2 ml on a heating plate (60-70°C). The remainder is transferred to a tray, evaporated (60-70°C) to dryness and kept in a desiccator. The procedure is repeated, using an equal volume of the sample, to obtain two results.

Both dry residues (not more than 50 mg on a 30 mm diam. tray) are counted for beta activity using a GM counter.

PROCEDURE II

The water sample is filtered under pressure through a cellulose-membrane filter. The volume of the filtrate is measured.

The filter is placed in a beaker, 20 ml of conc. HNO_3 is added and then evaporated (avoiding boiling) on a heating plate to dryness. The residue is then dried to constant weight. A small amount of water is added and the mixture is transferred to a tray, dried, covered with collodion layer and beta counted.

The filtrate is treated as in Procedure I.

The indices of beta radioactivity of insoluble and soluble fractions are given by the following formulae:

insoluble	soluble
$\frac{A}{V} \cdot \frac{N_1}{N_0} \cdot \frac{M}{M'}$ μCi/ml of ^{90}Sr equivalent	$\frac{A}{V'} \cdot \frac{N_2}{N_0}$ μCi/ml of ^{90}Sr equivalent

where A = total activity of standard
V = volume of sample
N_0 = counting result of standard
N_1 = counting result of ash
M = weight of total ash
M' = weight of part of ash used for counting
N_2 = counting result of dry residue
V' = volume of part of sample used for evaporation.

RAPID COMPARATIVE METHOD OF IDENTIFICATION OF BETA EMITTERS

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This rapid method of identification of beta emitters is based on the comparison of the counting rates from an unknown emitter and from a standard at the same discrimination levels. Measurements are made using a conventional scintillation counter, fitted with a plastic scintillator and provided with a variable discrimination level. The thickness of the scintillator should be about equal to the maximum range of beta particles of the standard emitter.

PROCEDURE

The identification of beta emitters consists in the determination of their energy E_β. For this purpose it is necessary to:

(a) Measure the counting rates of the sample and of the standard at a few discrimination levels;
(b) Plot the comparative curve of the sample standard and determine the slope of that curve; and
(c) Estimate the energy E_β from the slope of the energy curve.

For a rapid identification of simple beta emitters, the slopes of the comparative curves can be determined with measurements at only two discrimination levels (with ^{204}Tl as a standard). If the energy measured differs very much from beta energy of ^{204}Tl, more exact results can be obtained by the use of ^{99}Tc or ^{32}P as standard or by the use of the emitter to be determined, if available. For the identification of a mixture, however, an accurate knowledge of the shape of the comparative curves is necessary.

REMARKS

The time required for the identification procedure is about 10 min (including 5 min for calculation of the results of the measurements) at an activity level of 10^{-8} Ci, and 4-8 h at an activity level of 10^{-10} Ci.

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DETERMINATION OF TOTAL ALPHA ACTIVITY IN URINE

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The incorporation of alpha emitting substances such as plutonium, americium and others can be assessed by the analysis of urine. As the daily excretion rate amounts to 0.1% or less of the body content, it is necessary to concentrate these isotopes before measurements can be performed. As the method is intended for urine monitoring, the following conditions have to be met:

(a) Concentration of the alpha emitters must be simple;
(b) For the alpha measurement thin samples have to be prepared;
(c) The lowest detectable value must be below the investigation level (1/10 of excretion at maximum permissible body burden (MPBB)).

All samples with activities below the investigation level can be eliminated and thus an important reduction of the work-load may result.

PROCEDURE

Put 500-1000 ml urine and 1 ml of conc. H_3PO_4 in a 1.5 litre beaker. Bring the solution to pH9 by addition of conc. NH_3 solution and let the precipitate settle for 10 h. Centrifuge and discard the solution. Wash once with water containing NH_3. Dissolve the precipitate in 20 ml of conc. HNO_3 and dry carefully on a sand bath.

Wet the residue with conc. HNO_3 and 30% H_2O_2 solution and dry. To obtain a white ash, repeat this step if necessary. Dissolve the ash in 25 ml of 10N HCl solution and transfer the solution into a separating funnel of 100 ml.

Add 10 ml of 6% Aliquat 336 solution in xylol, shake for 5 min, transfer the aqueous solution into a second separating funnel of 100 ml. Repeat this extraction and collect organic phases in the first funnel.

Extract the organic phase twice with 10 ml of water. Collect the water extracts and evaporate to dryness. Add conc. HNO_3 and 30% H_2O_2 solution to oxydize organic matter and dry the mixture again. Dissolve the residue in 0.3 ml of conc. H_2SO_4 and transfer the clear solution into a cell for electrolysis with a stainless steel plate using 10 ml of water. Add conc. NH_3 solution to pH6 (methyl red-yellow) and acidify with one drop of 2N H_2SO_4 solution.

Electrolyse for 2 h at 300 mA, add 1 ml of conc. NH_3 solution and stop the current. Dry the electrode plate and measure the alpha activity.

REMARKS

With this method 90-98% of the actinides in urine can be recovered. One analysis takes about 1 day; several samples can be treated simultaneously. If desired, the electrode plates which are covered with a very

thin layer of the deposit can be used for alpha spectroscopy. It can be assumed that the maximum body burden for insoluble plutonium is 0.02 μCi. The daily excretion rate in urine is 0.05%. Thus the daily urinary excretion at MPBB is 1 pCi (2.22 dis./min). The lowest detectable amount by this method is 0.1 dis./min per 1.4 litre of urine, which means a level corresponding to half the investigation level.

The determination of plutonium or americium would take one day for each. By isolating practically all alpha emitters in one operation, the working time can be reduced. All samples with an activity below 0.2 dis./min per 1.4 litre can be eliminated from further specific analysis.

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DETERMINATION OF GROSS ALPHA ACTIVITY IN URINE

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The phenomenon of surface adsorption on glass fibre filter papers can be used to advantage as part of a routine analytical procedure for the determination of "gross alpha" activity in urine.

PROCEDURE

Transfer the urine sample (normally about 1500 ml) to a 3 litre beaker. Add 200 ml of HNO_3 (sp. gr. 1.42), a few glass beads and 5 ml of $CaHPO_4$ solution. Heat for 3 h at 80-90°C, then add 900 ml of 7M NH_3 solution to precipitate calcium/magnesium phosphates. Allow to settle, preferably overnight. Decant the supernatant solution and filter the phosphate precipitate through a 24 cm Whatman No. 40 filter paper.

Transfer the filter paper and precipitate to a 150 ml silica beaker and ash in a muffle furnace at 500°C. Remove from the furnace, allow to cool and add 5 ml of HNO_3 (sp. gr. 1.42) to the ash. Heat for a further period and repeat the HNO_3 treatment if a pure white residue is not obtained.

Dissolve the white phosphate residue in 100 ml of 1M HNO_3 solution and heat on a hot plate for 40 min. The hot plate should be on "High" and already at a high temperature before the beaker is placed on it. Otherwise ensure that the HNO_3 actually boils for 20 min. Remove the silica beaker from the hot plate and add 5 ml of 1M Na_2SO_3 solution. If the presence of neptunium is suspected in the sample, 5 ml of 50% (wt/vol.) $NH_2OH.HCl$ solution should be added instead of Na_2SO_3 and the solution boiled for a further 10 min. Allow to stand for 5 min. Transfer the solution to a 600 ml beaker and dilute to 500 ml with water. Adjust the solution to pH5.0 with NH_3 solution. Use NH_3 solution (sp. gr. 0.88) until the pH is between 2 and 3, then add dropwise 1M NH_3 solution for the final pH adjustment.

Pour the solution through a previously prepared glass fibre filter paper. Preparation of the filter paper: A Whatman 9.0 cm GF/A glass fibre filter paper is placed in a Büchner funnel and washed with 100 ml of HCl solution. It is then washed twice with about 10 ml of water and finally with 200 ml of water before use.

Wash the filter paper twice with 200 ml of water. Elute the alpha activity with 50 ml of 6N HCl solution and two 10-ml water washes into a 250 ml beaker. Add two glass beads to the solution in the beaker and boil down to a few millilitres. Evaporate the solution to dryness on a platinum counting tray, using several water washes to ensure quantitative transfer

to the tray. Flame the source and count in a suitably calibrated alpha counter.

REMARKS

The use of glass fibre filter papers for surface adsorption has three advantages over conventional ion exchange methods:

(a) Ease of handling of the papers compared with ion exchange resins;
(b) Cheapness;
(c) Rapidity.

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