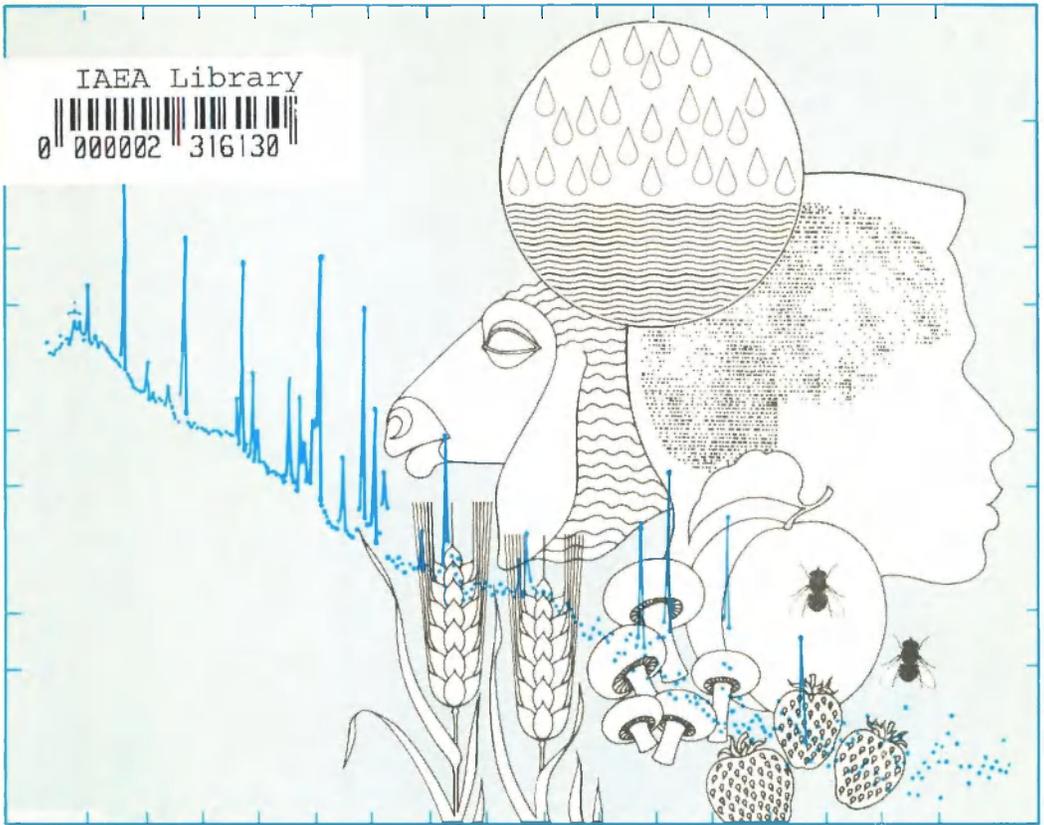


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Measurement of Radionuclides in Food and the Environment

A Guidebook



INTERNATIONAL ATOMIC ENERGY AGENCY, VIENNA, 1989

**MEASUREMENT OF RADIONUCLIDES
IN FOOD AND THE ENVIRONMENT**

A Guidebook

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The Agency's Statute was approved on 23 October 1956 by the Conference on the Statute of the IAEA held at United Nations Headquarters, New York; it entered into force on 29 July 1957. The Headquarters of the Agency are situated in Vienna. Its principal objective is "to accelerate and enlarge the contribution of atomic energy to peace, health and prosperity throughout the world".

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FOREWORD

Assessment of any release of radioactivity to the environment is important for the protection of public health, especially if the released radioactivity can enter the food chain. Assessment demands rapid, reliable and practical techniques for analysis of various radionuclides. After a recent nuclear accident, several Member States requested the assistance of the IAEA in conducting radioanalyses on a large number of food and environmental samples and in developing their own laboratory capabilities for this purpose. In the course of the Agency's response to these requests it became evident that there was a serious need for laboratories capable of performing rapid and reliable measurements of radionuclides on a variety of sample materials and capable of handling large numbers of samples.

"Methods of Radiochemical Analysis", published in 1966 under the auspices of FAO, the IAEA and WHO, is no longer available. Since the book was published, several new methods have been developed and current methods have been improved considerably. In many cases analyses can be carried out more quickly and accurately by the newer methods. This is especially true for gamma ray spectrometry, which has enabled many of the radiochemical separation procedures to be replaced by non-destructive measurements.

In response to the needs of the Member States, the Agency created a new programme entitled Fallout Radioactivity Monitoring in Environment and Food (MEF) as part of the Supplementary Programme on Nuclear Safety (SPNS). A joint meeting of FAO, UNSCEAR, WHO, WMO and IAEA representatives was held in Vienna in July 1986, at which it was agreed that a comprehensive document would be compiled to describe the facilities, equipment and analytical methods required to determine the concentrations of various radionuclides in several environmental materials and foodstuffs.

The overall aim of the MEF Programme is to provide national authorities of Member States and other International Organizations (e.g. FAO, UNSCEAR, WHO, WMO) with reliable analytical methods to obtain data capable of comparison in the case of radioactive releases. This guidebook is therefore published in the Agency's Technical Reports Series as a guide to the measurement of radionuclides in food and environmental samples, for training courses, and to help in the establishment of central and local environmental laboratories.

This guidebook does not contain *rapid* monitoring methods for detecting radioactivity in food and environmental samples, although such methods, in the event of accidental releases of radioactivity, are essential for radiation protection authorities and are needed as a basis for making judicial decisions on the control in food consumption and international trade in food and environmental materials.

The original draft of this document was prepared, at the request and with the assistance of the IAEA, by Ms. C. Klusek, Environmental Measurements Laboratory, New York, and Mr. O. Paakkola, Finnish Centre for Radiation and Nuclear Safety, Helsinki, in October 1986. It was subsequently reviewed at two IAEA Consultants Meetings by thirteen participants from nine Member States. Their modifications and additions have improved the document. A list of the consultants and participants from the IAEA is given at the end of the guidebook. A number of staff members from various Divisions of the IAEA also contributed to the final document.

Mr. T. Scott, retired from Oak Ridge National Laboratory, Oak Ridge, Tennessee, and Mr. R.F.W. Schelenz, IAEA, contributed to several sections and edited the material.

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Section 1

INTRODUCTION

This guidebook is a production of the Fallout Radioactivity Monitoring in Environment and Food (MEF) Programme created by the IAEA in response to requests from Member States. During the course of the Supplementary Programme on Nuclear Safety, it became evident that there was a serious lack of analytical capabilities within IAEA Member States to cope with nuclear releases. The IAEA therefore drew upon experts and specialists in the radioanalytical field to provide guidelines for collecting, preparing and analysing relevant environmental material and basic food for radionuclides of interest. It is recognized that the reliability of the methods depends to a great extent on the laboratory techniques. It is therefore recommended that laboratories institute training programmes in radionuclide analyses, develop quality control procedures, and participate in interlaboratory comparisons to establish and maintain the validity of their results.

This guidebook may be considered as a back-up document of other Agency publications, e.g. Safety Series No. 81, *Derived Intervention Levels for Application in Controlling Radiation Doses to the Public in the Event of a Nuclear Accident or Radiological Emergency* (1986). The guidebook is divided into two parts. The first contains seven sections, including this introduction. Sections 2 and 3 provide general information on samples and radionuclides of interest. Laboratories new to the field of radioanalysis will find Section 4 particularly useful as it describes the laboratory equipment, space and personnel needed for radioanalyses. General instructions for sample collection and preparation are given in Section 5. Sections 6 and 7 briefly discuss analytical methods and analytical quality control, respectively. A bibliography related to the physics, analysis and measurement of radionuclides is given at the end of the first part of the document.

The second part of the guidebook is composed of ten annexes. The first four contain detailed methods for determination of gamma emitters collectively, strontium isotopes, tritium, and the isotopes of plutonium, americium and curium. These procedures are considered reliable for the types of samples that particularly concern food intake. Other sources of documented methods are listed at the end of each section. Annex V provides a list of useful units and symbols; Annex VI gives information on radionuclide spectra in four types of nuclear accidents; nuclear data tables are listed in Annex VII; an example of a sample collection programme is presented in Annex VIII; some examples of NaI- and HP-Ge gamma spectrometric systems are given in Annex IX; and potential suppliers of calibration sources and reference materials are listed in Annex X.

Inquiries concerning the availability of documents cited in the references and of the background material used in the guidebook may be made to the Agency.

Section 2

PATHWAYS AND SAMPLES OF INTEREST

2.1. PATHWAYS

This section describes samples and pathways relevant to the analysis of radionuclides in foods and those environmental materials that are part of the immediate pathways leading to contamination of food.

The source of a release and the conditions at the site at which it occurs determine the one or more critical pathways in the environment between the point of discharge and man. The season of the year determines to a great extent the magnitude of contamination of different foods or environmental components. Figure 1 illustrates the major pathways of radionuclides to man [1].

The main purpose of analysis should be fast identification of the most critical samples and the most important radionuclides so that the necessary rapid actions can be carried out.

2.2. FOOD

Only those foods should be sampled and only those radionuclides analysed the consumption of which contributes significantly to population exposure. If, for example, ^{131}I is being released in proximity to cow pastures, its concentration in the milk produced will provide far more meaningful information than its concentration in air, deposition or forage samples. Nevertheless, measurements of ^{131}I in pasture grass may be very important in providing an indication of the expected concentration in milk. For other circumstances, the need for food sampling should be based on a thorough understanding of agricultural practices and food consumption in specific areas of interest. This information is available in national food sheets [2, 3] or from competent local specialists. Careful attention should be given to the design of the sampling programme in regard to the manpower requirements, the expense, and the need to avoid overloading limited radiochemical laboratory facilities.

It is recommended that food analyses be based on the determination of radionuclides in individual food items rather than a mixed diet sample. Only the analysis of individual foodstuffs can indicate whether and which countermeasures should be taken to reduce doses. Food sampling for estimation of total consumption should be carried out at the retail level when appropriate; otherwise, it should be carried out at the consumption level. The selection of foods to be sampled can be based on individual diet or food consumption statistics. Analyses of individual foodstuffs should preferably be performed after preparation, taking into account the effect of kitchen activities such as washing, cleaning and cooking.

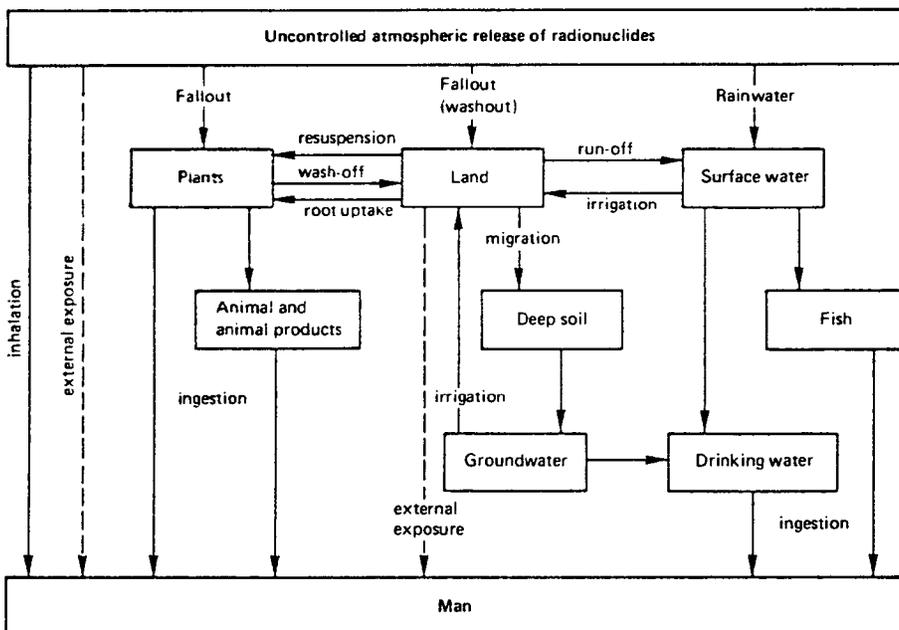


FIG. 1. Major pathways of radionuclides to man in the event of an uncontrolled release of radioactivity [1].

If there is concern about short term effects, it is necessary to go back to the point of production in order to avoid the effect of the lapse of time between production and consumption. The individual foods most likely to be contaminated must always be considered in relation to the nature of the release of the radionuclides.

2.2.1. Milk

Milk and milk products are important components of diet in many countries. Milk is one of the few foods produced over large areas and collected on a daily basis. Its composition is almost identical all over the world, and it is easy to collect a representative sample that can be analysed in liquid or dried form.

Milk is likely to be contaminated by radioactive iodine and caesium within the first days after a release of volatile radionuclides. Contamination of milk will be greatest when cows are grazing during the fallout period, but even when cows are kept indoors, contamination of milk may occur by inhalation of radionuclides or ingestion of radionuclides in drinking water and contaminated feed. Milk from goats and sheep, because of their grazing habits, should be checked periodically over a longer period.

2.2.2. Grain and rice

After harvesting, grain and rice are subjected to contamination only during storage, and only the outer layers would be contaminated. If fallout occurs during the growing season, radionuclides will be transported into the grain and rice through the plant growth process. It is relatively easy to select representative samples of grain and rice at harvest time. If the fallout occurs during the winter, grain will be contaminated only through root uptake in the next growing season.

2.2.3. Meat

Following an accidental release of radiocaesium, meat becomes one of the main sources. Contamination of meat is mainly the result of animal grazing, but contaminated drinking water might also be an important pathway. Inhalation of radiocaesium is not likely to be a significant pathway to meat. Meat sampling should normally be done in such a way that the composite sample is representative of a large number of animals, although after heavy fallout screening measurements of individual animals may be necessary.

2.2.4. Aquatic organisms

Following an accident, contamination of fish in nutrient deficient lakes may constitute a particularly significant pathway for the uptake of radiocaesium by man. Obtaining a representative sample from an area containing many lakes may require some compromise since collection of samples from a large number of the lakes may be impracticable. Ocean fish will not take up as much radiocaesium as fresh water fish because of the dilution through the depth of the ocean and the effective dilution associated with the high potassium content in the water, but particulate-associated radionuclides can be enriched to high levels. Mussels like *Mytilus edulis*, some species of macro algae, and other filter feeders quickly take up the contaminants from the sea water and can also be used as biological indicators.

2.2.5. Vegetables

Green leafy vegetables are very prone to external contamination during their growing season. Other vegetables, including root vegetables, may also become contaminated. It is important to obtain representative vegetable samples, and sampling should be planned carefully. In the early stages of fallout, green vegetables can be a very significant pathway for short lived radionuclides.

2.2.6. Other foods

Game and foods such as mushrooms and berries can be contaminated markedly, although only in very rare cases would they contribute significantly to the ingestion dose. It may still be advisable to analyse these foods in order to decide whether the levels comply with international export regulations.

2.3. ENVIRONMENTAL SAMPLES

2.3.1. Air

Measurement of airborne radioactivity provides the first opportunity to identify the spectrum of radionuclides making up the contamination. Radionuclides will very rapidly appear in ground level air, and air samples can give the first indication of the nature of the contamination. Radioactive materials in the air may result in exposure to man by inhalation or ingestion of particulate matter deposited on vegetation or by ingestion of products derived from animals which were exposed to radioactive materials through inhalation or ingestion.

2.3.2. Water

Rain water and snow are also early indicators of radioactive contamination. In some places drinking water and rain water can be significant pathways of short lived radionuclides, e.g. radioiodine, to man or animals. Drinking water and household water are potentially important pathways, directly or through their use in food preparation and processing, although dilution, time delays and water treatment can reduce the contamination levels markedly. Water consumed by livestock and/or used for irrigation purposes can also be a source of radionuclides in foods. Sea water can be a contamination source for seafoods (e.g. mussels, shellfish, fish, algae). Water from streams, lakes and ponds should also be considered as a source of contamination.

2.3.3. Soil

Contaminated soil serves as a direct source of radionuclides leading to the contamination of all agricultural products. Contaminated soil used in greenhouses could add significantly to the contamination of vegetables.

2.3.4. Grass

Grass is a direct pathway of radionuclides to animals and then to man through meat and/or milk. The radionuclide content of grass can provide a basis for deciding whether cattle can be permitted to graze in a given area.

2.3.5. Sediment

Sediment in all types of water (sea, lake, pond and large or small streams) may be a source of contamination to aquatic organisms. Contaminated sedimentary materials used as fertilizers may increase the radioactivity levels of soil.

Section 3

RADIONUCLIDES OF INTEREST

Although several hundred radionuclides are produced by nuclear explosions or are present in irradiated reactor fuel, only a limited number contribute significantly to human exposure. These would normally include fission products and activation products. Radioactive noble gases, e.g. ^{85}Kr , ^{133}Xe , are not considered since they are unlikely to contribute significantly to internal exposure via the food chain. Radionuclides produced in fission and activation processes which may contribute significantly to human exposure in the event of an accident are listed in Table I [4].

3.1. FISSION PRODUCTS

The primary source of radionuclides produced in the fission process and found in the environment is atmospheric testing of nuclear weapons. The public has been exposed to these and other radionuclides for the past three decades or longer, but there has been a substantial decline in atmospheric testing in the past two decades. Therefore the major source of fission product radionuclides in recent years has been from nuclear accidents. A nuclear reactor meltdown could release a spectrum of radionuclides similar to that of a nuclear bomb explosion, but the ratios of nuclides would greatly differ for the two cases. The reason for the differences in ratios of radionuclides is that during the reactor operation the long lived radionuclides tend to build up progressively, whereas the short lived radionuclides tend to reach an equilibrium state at which the rate of decay equals the rate of production. The proportion of various radionuclides produced in the operation of a nuclear reactor changes with operating time and with fuel burnup. Table II shows radionuclides reported to be present in air and deposition samples shortly after the Chernobyl nuclear power plant accident, including both fission products and activation products [5].

3.2. ACTIVATION PRODUCTS

Radionuclides classified as activation products are created in nuclear reactors and other nuclear devices by the reactions of neutrons with fuel and construction materials. Activation products include the isotopes of the transuranic elements and radioisotopes of hydrogen, carbon, caesium, cobalt, iron, manganese, zinc, and a host of other radionuclides, all of which should be recognized and considered in determining the environmental pathways to human exposure.

TABLE I. FISSION AND ACTIVATION PRODUCTS WHICH MAY BE OF CONCERN IN HUMAN EXPOSURE [4]

	Nuclide ^a	Half-life ^b	Fission yield %	Major decay
Fission products	Sr-89	50.5 d	4.77	β^-
	Sr-90*, Y-90	28.7 a, 64.1 h	5.76	β^-, β^-
	Zr-95, Nb-95	64.09 d, 35.0 d	6.51	$\beta^-\gamma, \beta^-\gamma$
	Mo-99*, Tc-99m*	2.747 d, 6.006 h	6.09	$\beta^-\gamma, \beta^-\gamma$
	Ru-103*, Rh-103m*	39.272 d, 56, 116 min	3.03	$\beta^-\gamma, \beta^-\gamma$
	Ru-106, Rh-106*	372.6 d, 29.92 s	0.4	$\beta^-, \beta^-\gamma$
	Te-129m	33.6 d	0.661	$\beta^-\gamma$
	I-131*	8.021 d	2.875	$\beta^-\gamma$
	Te-132*, I-132	76.856 h, 2.3 h	4.282	$\beta^-\gamma, \beta^-\gamma$
	Cs-137, Ba-137m	30.0 a, 2.55 min	6.136	β^-, γ
	Ba-140*, La-140*	12.751 d, 1.6779 d	6.134	$\beta^-\gamma, \beta^-\gamma$
	Ce-144*, Pr-144	284.45 d, 17.28 d	5.443	$\beta^-\gamma, \beta^-\gamma$
	Activation products	H-3*	12.35 a	
C-14		5730 a		β^-
Fe-55*		2.75 a		EC
Fe-59*		44.53 d		$\beta^-\gamma$
Mn-54		312.5 d		EC, γ
Co-60		5.27 a		$\beta^-\gamma$
Zn-65*		243.9 d		EC, γ
Cs-134*		754.2 d		$\beta^-\gamma$
Np-239*		2.355 d		$\beta^-\gamma$
Pu-241, Am-241*		14.35 a, 432.0 a		$\beta^-, \alpha\gamma$
Cm-242*		162.94 d		α
Pu-238*		87.7 a		α
Pu-239*		2.411×10^4 a		α
Pu-240*		6.563×10^3 a		α
Pu-242*		3.735×10^5 a		α

^a An asterisk indicates that half-life has been revised according to Ref. [VII.2].

^b Half-life is given in minutes (min), hours (h), days (d) and years (a). One year = 365.25 days.

TABLE II. RADIONUCLIDES REPORTED IN AIR AND DEPOSITION SAMPLES SHORTLY AFTER THE CHERNOBYL NUCLEAR POWER PLANT ACCIDENT [5]

Nuclide ^a	Half-life ^b	Major decay
H-3	12.35 a	β^-
Sr-89	50.5 d	β^-
Sr-90*	28.7 d	β^-
Zr-95	64.09 d	$\beta^- \gamma$
Nb-95	35.0 d	$\beta^- \gamma$
Mo-99*	2.7476 d	$\beta^- \gamma$
Ru-103*	39.272 d	$\beta^- \gamma$
Ru-106	372.6 d	β^-
Ag-110m	249.79 d	$\beta^- \gamma$
Cd-115	2.2 d	$\beta^- \gamma$
Sb-125	1008.1 d	$\beta^- \gamma$
Sb-127	3.9 d	$\beta^- \gamma$
Te-129m	33.6 d	$\beta^- \gamma$
Te-131m	30.0 d	$\beta^- \gamma$
Te-132*	3.204 d	$\beta^- \gamma$
I-131	8.021 d	$\beta^- \gamma$
I-133	20.3 h	$\beta^- \gamma$
Cs-134*	754.2 d	$\beta^- \gamma$
Cs-136	13.0 d	$\beta^- \gamma$
Cs-137	30.0 a	β^-
Ba-140*	12.751 d	$\beta^- \gamma$
Ce-141	32.50 d	$\beta^- \gamma$
Ce-144*	284.45 d	$\beta^- \gamma$
Np-239*	2.355 d	$\beta^- \gamma$
Am-241*	432.0 a	$\alpha \gamma$
Cm-242*	162.94 d	α
Pu-238*	87.70 a	α
Pu-239/240*	2.411×10^4 a/ 6.563×10^3 a	α/α
Pu-241*	14.35 a	β^-
Pu-242*	3.735×10^5 a	α

^a An asterisk indicates that half-life has been revised according to Ref. [VII.2].

^b Half-life is given in hours (h), days (d) and years (a). One year = 365.25 days.

3.3. NATURAL RADIOACTIVITY

The major naturally occurring radionuclides include the isotopes of uranium and thorium plus their daughters and ^{40}K . Measurements of these natural activities are not considered in this guidebook except in regard to their influence on the backgrounds of counting instruments. Information on the natural radioactivity in man and his environment has been reviewed by the United Nations Scientific Committee on the Effects of Atomic Radiation (UNSCEAR) [6].

3.4. RADIONUCLIDES OF SPECIFIC INTEREST IN FOODS AND THE ENVIRONMENT

In regard to internal exposure from ingestion of food and water and to the contamination of environmental materials which are part of the immediate pathways leading to contamination of food, the most important radionuclides to be assessed following a release of radionuclides from a uranium-fuelled reactor to the environment are ^{134}Cs , ^{137}Cs ($^{137\text{m}}\text{Ba}$), ^{131}I and other gamma emitters, the beta emitters ^{89}Sr , ^{90}Sr and tritium, and the alpha emitters ^{238}Pu , $^{239+240}\text{Pu}$, ^{241}Am and ^{242}Cm .

The levels of radionuclides in the environment and food have been extensively compiled by UNSCEAR [6, 7]. In general, the radionuclides of major importance in the contamination of food and environmental samples (materials which are part of the pathways leading to food) are:

Air	^{131}I , ^{134}Cs , ^{137}Cs
Water	^3H , ^{89}Sr , ^{90}Sr , ^{131}I , ^{134}Cs , ^{137}Cs
Milk	^{89}Sr , ^{90}Sr , ^{131}I , ^{134}Cs , ^{137}Cs
Meat	^{134}Cs , ^{137}Cs
Other foods	^{89}Sr , ^{90}Sr , ^{134}Cs , ^{137}Cs
Vegetation	^{89}Sr , ^{90}Sr , ^{95}Zr , ^{95}Nb , ^{103}Ru , ^{106}Ru , ^{131}I , ^{134}Cs , ^{137}Cs , ^{141}Ce , ^{144}Ce
Soil	^{90}Sr , ^{134}Cs , ^{137}Cs , ^{238}Pu , $^{239+240}\text{Pu}$, ^{241}Am , ^{242}Cm

This group of radionuclides is most likely to be of concern in terrestrially produced foods. Biological concentration processes in fresh water and marine systems can result in very rapid transfer and enrichment of specific radionuclides. The radionuclides which enter such systems can in certain cases be rapidly accumulated by plankton and algae. These organisms serve as food for higher trophic levels; thus the radionuclides become concentrated in organisms such as oysters, clams, shrimp, etc. Radionuclides of particular concern in fresh water and marine food

chains include: ^{54}Mn , ^{55}Fe , ^{59}Fe , ^{60}Co , ^{65}Zn , ^{95}Zr , ^{95}Nb , ^{103}Ru , ^{106}Ru , $^{110\text{m}}\text{Ag}$, ^{125}Sb , ^{131}I , ^{134}Cs , ^{137}Cs , ^{141}Ce , ^{144}Ce and some of the transuranic elements.

Many other radionuclides would be present in debris from a nuclear accident, and their potential contribution to human exposure depends on the type of accident and the circumstances at the time of the accident. Since there are several types of fuel, the spectra of radionuclides that would be present in accidental releases could be somewhat different. In Annex VI, four nuclear accident scenarios are considered, and the radionuclides which would be of concern during varying periods of time are presented. The scenarios considered in Annex VI are:

- Reactor meltdown with failed containment,
- Reactor meltdown with particle containment,
- Nuclear fuel reprocessing plant release,
- Plutonium fuel fabrication plant release.

Section 4

REQUIREMENTS FOR LABORATORIES, EQUIPMENT AND PERSONNEL

An environmental programme for monitoring radioactivity should include adequately equipped and staffed laboratories. A good plan is to have a fully equipped central laboratory and local monitoring laboratories capable of screening materials that can be subsequently sent to the central laboratory for more detailed evaluation.

4.1. CENTRAL ENVIRONMENTAL LABORATORY

A central laboratory should have the facilities, equipment and personnel necessary to perform detailed analyses on all types of environmental materials and food for the relevant radionuclides associated with any type of nuclear operation which could accidentally release radioactivity to the environment. This means that the laboratory would be capable of performing the required chemical separations and nuclear measurements for determination of:

- Gamma emitters by non-destructive gamma ray spectrometry as described in Annex I;
- ^{90}Sr and/or ^{89}Sr by the method outlined in Annex II;
- Tritium by liquid scintillation counting as presented in Annex III;
- Transuranic elements by chemical separations and alpha spectrometry as outlined in Annex IV.

This laboratory could also serve as a national central reference laboratory for low level radioactivity measurements.

The size of the facilities needed for these functions depends on the number of local nuclear installations (e.g. reactors, reprocessing plants), on food import controls and on the overall need of the country's radiation protection programmes. Table III lists the number of rooms and the area recommended for a central laboratory's activities.

Obviously, organizations that are just beginning to set up monitoring facilities may not be able to establish a full-scale operation as outlined here. However, good judgement in selecting the appropriate facilities and equipment will facilitate the establishment of a scaled down and adequate monitoring laboratory. As an example, the plan for a national central environmental laboratory that is being constructed by the Sudan Atomic Energy Commission is given in Fig. 2. Another option may be to combine the facilities of several organizations in a co-operative effort to establish a monitoring laboratory.

TABLE III. ROOMS AND AREA RECOMMENDED FOR A CENTRAL ENVIRONMENTAL LABORATORY

Operations	No. of rooms	Area (m ²)
Sample registration, storage and preparation: Evaporation and drying Grinding Ashing Handling of higher activity samples	7-8	130-180
Gamma ray spectrometry laboratory: Preparation and storage of samples Counting room Computer room ^a	3-4	90-120
Alpha spectrometry laboratory: Radiochemical laboratory Counting room Computer room ^a	3-4	60-70
Tritium laboratory: Sample preparation by distillation (electrolytic enrichment) Liquid scintillation counting room	3-4	60-70
Strontium laboratory: Radiochemical laboratory Beta counting room Computer room ^a	3-4	80-100
Offices, administration: Social rooms, library, etc.	4-10	80-150
	23-34	500-690

^a One computer room can serve the needs of all the counting rooms, which can also be combined. The size of the operation will determine how practical combining these facilities would be.

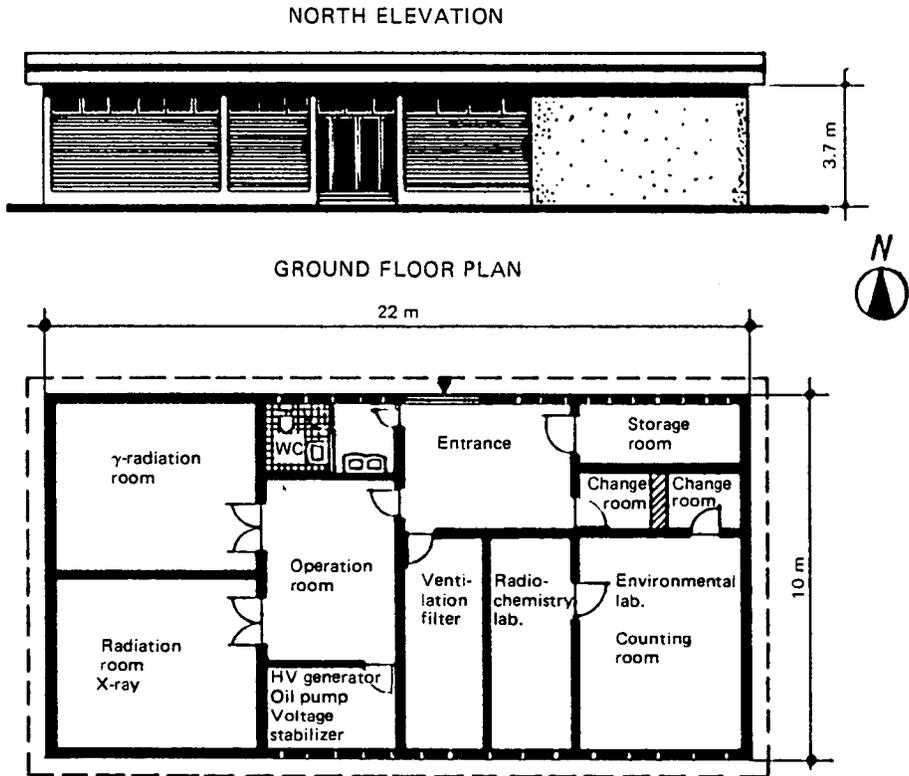


FIG. 2. Sudan Atomic Energy Commission's plan for a central environmental laboratory.

4.1.1. Laboratory design

The central laboratory should be designed and built according to the requirements of a normal analytical laboratory with a few additional features such as special ventilation, specially designed rooms for receiving and storing samples, and specially constructed nuclear measurement facilities (counting room(s)).

The ventilation system should be designed to filter incoming air to all areas and to maintain the lowest pressure in areas used for handling high activity samples and for preparing the samples. The sample receiving room(s) should be at low pressure to prevent any possible contamination of other laboratories by incoming samples. Wet chemistry laboratories should have well ventilated fume hoods. Air supplied to the counting room(s) should be passed through additional charcoal filters (when necessary) to remove gaseous radionuclides. The (filtered) air should not be recycled. This type of filtration is important in the event of a nuclear accident in which

radioiodine and radioactive noble gases may present serious contamination problems. Counting rooms should be under positive pressure at all times. Constant temperature and humidity should be maintained in counting and computer rooms and should be set according to the recommendations of the manufacturers of instruments and equipment. Normal temperatures recommended for counting rooms are between 20°C and 26°C.

Special consideration should be given to the design and construction of counting rooms. Construction materials should be selected so as to have a minimum of background from natural radioactivity, and the floors should be capable of carrying a load of at least 2000 kg/m² in order to support heavy detector shields.

Sample receiving rooms should be equipped with radiation monitoring instruments for surveying incoming samples. There should be adequate labelling and registering equipment so that each sample can be precisely identified. Storage space should be available to accommodate all types of sample materials. Storage shelves or bins should be constructed of materials that are easily decontaminated in case of spills, etc.

4.1.2. Laboratory equipment

Several items of instrumentation and equipment will be required in addition to those of a normal laboratory:

A. Radiation measurement instrumentation

Instruments for nuclear measurements can be divided into three general classes: those for measuring alpha, beta and gamma emitters.

(1) Instrumentation required for alpha measurement:

- Portable survey instrument(s) for monitoring incoming samples.
- Alpha spectrometer system consisting of:
 - (a) Silicon surface-barrier detector in a vacuum chamber with all necessary electronics such as power supply, preamplifier, amplifier and analog-to-digital converter (ADC) (the efficiency of an adequate alpha detector should approach 25% and the detector should have an active surface area of 3–5 cm²);
 - (b) Multichannel analyser (MCA) with a minimum of 400 channels, with a display screen and keyboard;
 - (c) A high-speed printer.

This system could consist of a 4096-channel analyser with four or more separate inputs from four or more detectors routed through a multiplexer.

- (2) Instrumentation required for beta measurement:
- Portable, thin-window, Geiger-Müller, beta-gamma survey instrument for monitoring incoming samples.
 - Low background, gas-flow, anti-coincidence beta counter(s). Some beta counting systems of this type are capable of making multiple simultaneous measurements (as many as 16 or more). Also, these systems have a variety of input and output capabilities; for example, some are programmable and equipped for printing full reports in selected units.
 - Liquid scintillation counter. A cooled, low background liquid scintillation counter is recommended, but modern benchtop scintillation counters are nearly as good for measuring tritium and other low energy beta emitters and (under certain conditions) alpha emitters.
- (3) Instrumentation required for gamma measurement:
- Portable gamma measurement systems to monitor incoming samples. Usually the beta/gamma survey instruments are adequate. However, several portable germanium detector systems which could be used for this purpose as well as for field measurement are available.
 - A fully integrated gamma spectrometry system for qualitative and quantitative determination of gamma emitters. The system should comprise a germanium detector, shield, multichannel analyser (MCA), high-speed printer, and (optionally) a plotter. The system should be equipped with a germanium diode semiconductor detector with a relative efficiency of about 20% and a resolution of about 2.0–2.2 keV. The detector should be contained in a lead shield, with wall thickness of about 5–10 cm. Germanium detectors require liquid nitrogen for cooling. Figure 3 shows typical shields with liquid nitrogen Dewars. With the proper ADCs and multiplexers, a single MCA system can be used for both alpha and gamma spectrometry. Figure 4 shows a typical spectrometer system with MCA and detectors. Further examples of equipment are given in Annex IX.
 - A gamma ray counter and/or spectrometer equipped with a 7.62 cm × 7.62 cm NaI(Tl) detector (8% resolution), a 2048-channel analyser and a lead shield with an inside diameter of about 20 cm and a wall thickness of 5–10 cm.

B. Other laboratory equipment

- (1) Special equipment will be required for sample collection and preparation and for laboratory use:
- Truck or other type of vehicle and/or boat for sampling according to the environmental programme;

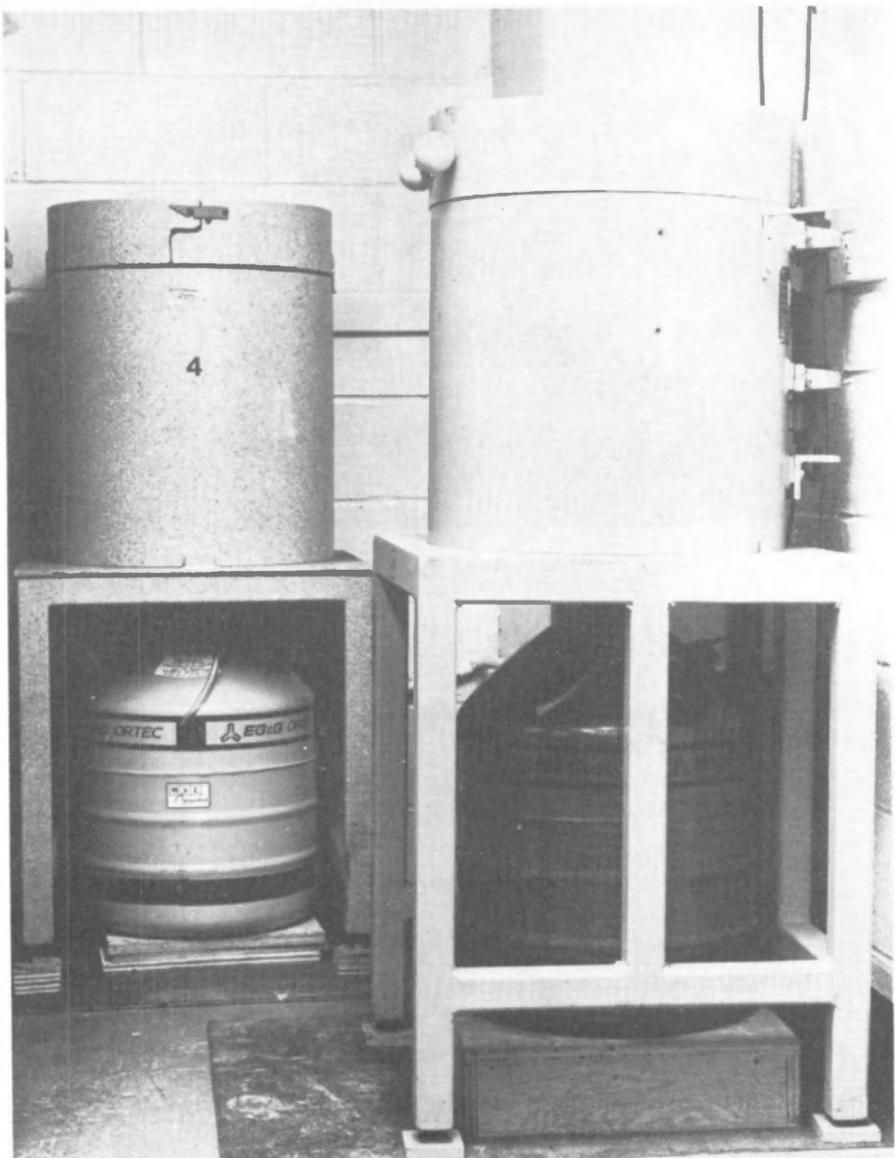


FIG. 3. Germanium detector shields with liquid nitrogen Dewars for cooling.

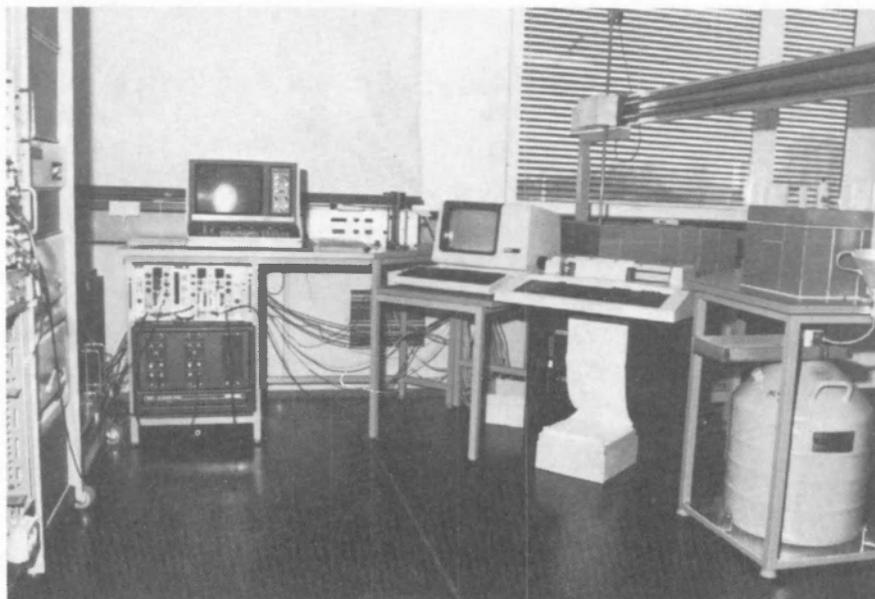


FIG. 4. Typical spectrometer system with multichannel analyser detector.

- Large refrigerator for preserving samples;
- Freezer for storing samples;
- Crusher and/or grinder (see Fig. 5);
- Evaporator or evaporation lamps;
- Large drying oven;
- Large muffle furnace (min. requirement 600°C, preferably 800°C) (see Fig. 6);
- Freeze dryer (as an option).

(2) Additional items needed for the laboratory are:

- Electrolytic enrichment unit;
- Multicelled electrodeposition system (four or more cells) with controlled (constant current) power supply;
- Large centrifuge with at least four positions of 250 mL capacity each.

4.1.3. Personnel requirements

The following list gives an idea of the personnel required to staff a central environmental laboratory:

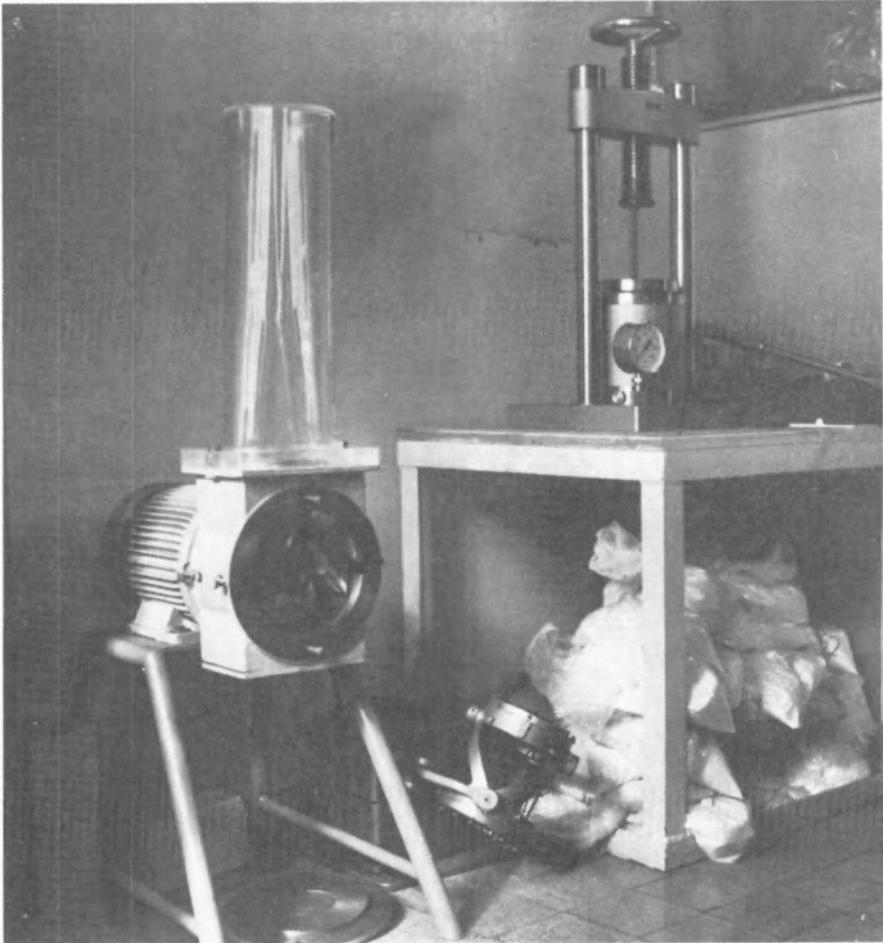


FIG. 5. Typical grinder used in preparation of samples.

- 1 manager (chief scientist);
- 3 scientists;
- 3 senior technicians;
- 6 technical assistants;
- 1 secretary;
- 1 typist;
- 1 field technician to operate trucks, other vehicles and boats for sample collection;
- 1 maintenance and service technician familiar with electronic equipment.

Further assistance personnel according to the customs of the country.

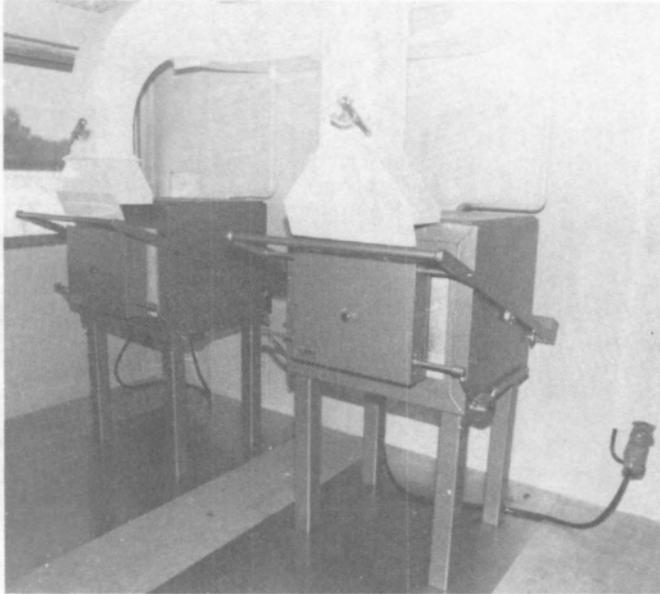


FIG. 6. Muffle furnaces with exhaust systems.

4.2. LOCAL MONITORING LABORATORIES

4.2.1. General description

A local laboratory should service a relatively restricted geographical area. It can be located in a food or environmental laboratory that is routinely analysing food, water, or other environmental samples. Such a laboratory need not necessarily be normally engaged in measuring radioactivity but may have been designed for chemical, microbiological, and/or other analyses. These laboratories could be placed at customs facilities, food import entry locations, or large food processing installations.

A local laboratory needs only relatively simple counters for radioactivity measurement. To obtain reliable results their measurements must be based on the standards and calibrations provided by the central environmental laboratory. This co-operation between the local and central laboratories must be actively maintained, and a system of measuring and reporting to the central laboratory must be appropriately organized.

The staff of local laboratories must undergo continuous training in order to maintain the radioactivity measurement capability adequate for routine measurements and to be ready for any possible emergency. The work of these laboratories

is mainly screening, but some better equipped local laboratories may also carry out more advanced measurements. Laboratories that are already established should cooperate with a central laboratory. It is recommended that new local laboratories consult with a central laboratory so that uniform equipment is obtained.

The area needed for these activities is approximately two rooms with a floor space of 20–30 m². In an emergency it should be possible to expand this working space to a larger area. The area required for radioactivity measurements can normally also be used for other laboratory activities.

4.2.2. Equipment for radiation measurement

Local laboratories should be equipped with a simple gamma ray spectrometric system. They should also have a beta/gamma monitor for checking possible laboratory contamination and for sorting samples into groups according to their radiation levels.

The radiation measurement and/or counter instruments should include:

- Gamma ray counter and/or spectrometer equipped with a 7.62 cm NaI(Tl) detector (8% resolution), a 2048-channel analyser and a lead shield with an inside diameter of about 20 cm and a wall thickness of 5–10 cm;

- Beta/gamma contamination monitor equipped with a Geiger-Müller tube, a plastic scintillator, or other radiation sensor.

4.2.3. Other equipment

If the local monitoring laboratory is situated within an operative chemical or microbiological laboratory, some of the latter's standard equipment and instruments will be useful for emergencies. Since the need to analyse a large number of samples may arise, the laboratory should reserve sampling equipment and plastic bottles and bags exceeding normal needs.

4.2.4. Personnel

Staff members of analytical laboratories within which the local monitoring laboratories are situated can be trained to perform the required measurements.

Section 5

COLLECTION AND PREPARATION OF SAMPLES

Samples received in the laboratory may not be in the proper physical form for analysis. They may require reduction in size, drying or some form of homogenizing before aliquots can be taken for analysis. Some general considerations for handling and pretreatment of samples are presented in this section. Specific methods for individual matrices are described in Section 5.2. Detailed collection and preparation procedures for several matrices can be found in Ref. [7]. To avoid contamination, it is important that samples with high levels of activity be processed in a separate area from low level samples.

5.1. GENERAL CONSIDERATIONS

5.1.1. Collection of samples

Double identities should be placed on samples at collection time. It is advisable to fill in a standard form with all relevant information (date, location, fresh weight, weather, collector's name, etc.). Care should be taken that the sample is representative and suitable for the specific purpose of the monitoring procedures. The design and organization of sampling programmes have been considered by others (see, for example, Refs [4] and [8]).

5.1.2. Storage

After collection, the samples must be properly stored to avoid degradation, spoiling, or other decomposition, and to avoid contamination. Proper care must be taken to avoid loss of volatile radionuclides. Short periods of storage before analysis may require refrigeration, freezing, or the addition of a preservative such as sodium bisulphite, alcohol or formaline (as in the case of milk) for preservation of biological samples. When long periods of storage are needed, it may be preferable to convert the samples to a more stable form immediately after sampling. Drying or ashing the sample will allow extended storage; however, the temperature must be carefully controlled in these operations to avoid loss of radionuclides (see Section 5.1.4 and Table IV [9]).

Sample containers must be suitable for storage without degradation especially when acids are added to liquid samples. Adsorption of most radionuclides from solution is less on polyethylene than on glass. With a few exceptions, almost all sorption losses can be eliminated by the addition of acid, a carrier solution containing stable elements, or a complexing agent.

5.1.3. Cleanliness

The sample collection equipment, containers and sample preparation areas must be kept clean to avoid contamination. Disposable containers should be used whenever possible (plastic bags, aluminium trays, etc.).

5.1.4. Drying, evaporation and ashing

Drying reduces the weight and volume of the samples and may also permit longer storage time. Samples may be dried in a low temperature oven at 105°C or at room temperature without a significant loss of any radionuclides except radioiodines. Samples should be dried for a sufficient period of time at a fixed temperature to acquire a constant dry weight. Measurements of the fresh or wet weight and the dry weight are required. It is important to prevent contamination during the drying procedure. If necessary, freeze drying may be used to further reduce the loss of volatile radionuclides from the sample. However, this process is very time consuming and is therefore not highly recommended.

Evaporation is the normal method of concentrating liquid samples. Reasonable care is required when evaporating liquids with a hotplate, particularly milk, in order to avoid spattering and loss of sample. Evaporation lamps usually eliminate the problem of spattering. An evaporating system as illustrated in Fig. 7 can be employed. The evaporation bowl should be made of material that will not absorb the radionuclides. Some radionuclides, such as radioiodine, tritium and radioruthenium, may be lost during the evaporation process. A fast evaporation can be satisfactorily performed with a rotating evaporation system that operates under reduced pressure. Different volumes of the rotating spheres of up to 30 L are available. Figure 8 illustrates a typical rotating evaporator.

Where samples require ashing, low carbon nickel trays are adequate for the ashing operations. However, other trays lined with thin-sheet aluminium which is discarded after each use may be entirely satisfactory. Trays are easily cleaned with detergents or dilute mineral acids (usually HCl). The temperature for dry ashing varies but an upper limit of 450°C is recommended. If the sample is not completely dry at the start, an initial drying step at 105°C should be introduced. The ashing time depends on the type and quantity of the material; large samples may require 16–24 hours. Dry ashing should be used only for radionuclides that do not vaporize at the ashing temperature. Significant loss of radiocaesium will occur above 400°C. A study on the loss of radionuclides during ashing is reported in Ref. [10]. If only radiostrontium is to be analysed, temperatures up to 600°C may be used. Carrier elements and radioisotope tracers should be added to all sample types before ashing. Measurements of the ashed weight are necessary for calculations of radionuclide concentrations and yield.



FIG. 7. Evaporation lamps for drying samples.

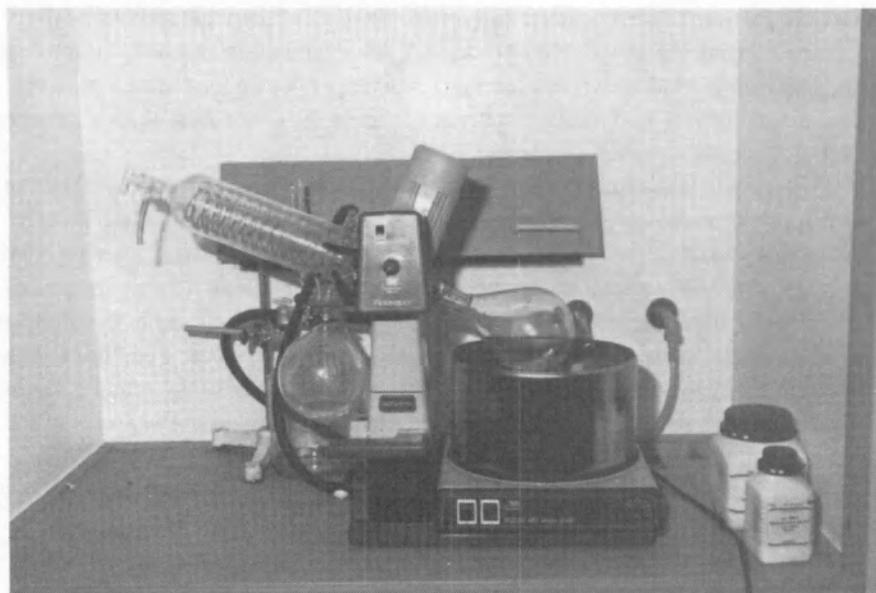


FIG. 8. Rotary evaporator for fast evaporation of samples.

5.1.5. Homogenization and subsampling

Sample materials are usually homogenized after drying and/or ashing. The process can be carried out by a variety of means, such as “V” blenders, mixer-blenders, ball mills, etc. It is extremely important that samples are thoroughly homogenized when subsamples are to be used for analyses.

Regardless of the care taken to homogenize the sample materials, a subsample may not always be representative of the whole sample, for example, material in which certain radionuclides are attached to or adsorbed on fine- or coarse-grained particles which may not be evenly divided throughout the sample. The recommended procedure for materials of this type is to take a subsample as large as is practicable to obtain a sample which is representative of the whole material.

5.2. SAMPLING AND PREPARATION OF SAMPLES

5.2.1. Air

Airborne particulate samples are usually collected on a filter with the aid of an air pump. In situations involving airborne ^{131}I , an activated carbon cartridge or a specially treated chemical absorbent can be placed between the filter and the pump. The air sampler should be supplied with a vacuum gauge and a flow meter and be mounted in an all-weather station. It should be located in a manner similar to a meteorological instrumentation station (a sample station is shown in Fig. 9). The discharged-air duct should be positioned to prevent recirculation of air. Air should be drawn through the filter at a known rate for a known period of time, preferably using an integrating flow meter to give the total volume. In particular, when high dust concentrations occur during sampling it may not be possible to maintain a constant air flow through the filter. The radionuclide concentrations are reported per unit volume of air (Bq/m^3).

Several types of filter material are used for collecting aerosol materials. For normal operations, glass, PVC or Microsorban filters are used. Collection efficiencies and other characteristics of air filters are available [11, 12].

All commercial filter media, when used properly, have adequate efficiencies. The filters are usually compressed to provide a standard counting geometry and are measured by gamma spectrometry, after which they may be dry- or wet-ashed for radiochemical analysis.

5.2.2. Grass

When collecting pasture grass or other types of animal forage, it is essential that sampling be carried out from a considerable area to ensure that the sample is

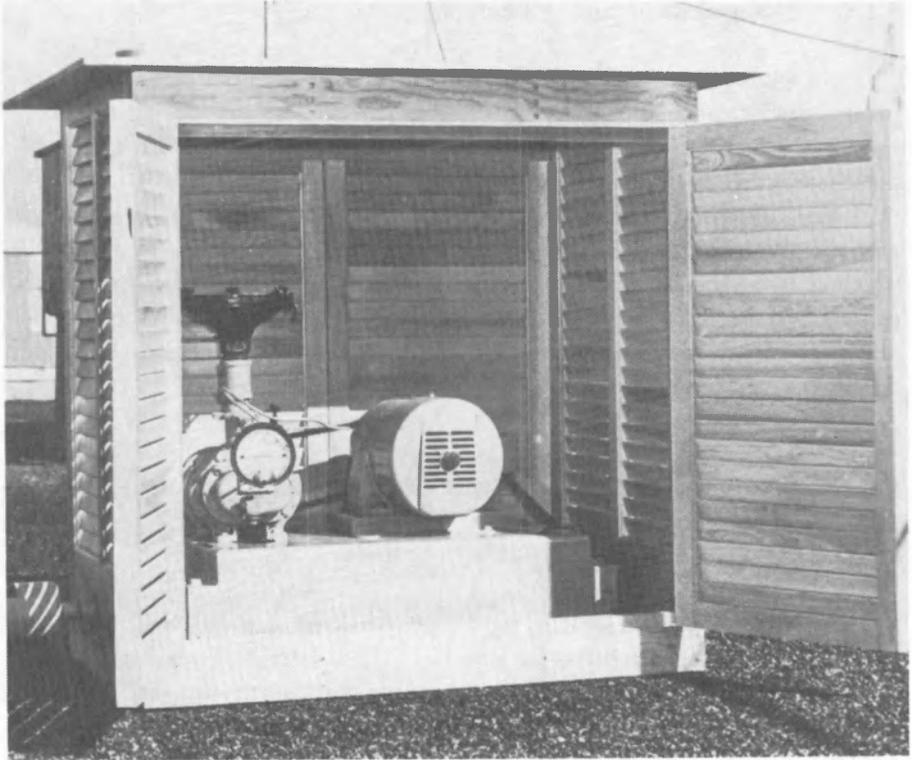


FIG. 9. All-weather sampling station used by the Environmental Measurements Laboratory, New York.

representative. Care should be taken to avoid contamination with soil. Slopes on which abnormal runoff may occur should be avoided. It is recommended not to rake the plot being sampled. The vegetation should be held in the hand while being cut, or an apron (grass catcher) should be used if the forage is cut mechanically. Vegetation within each area should be cut to a height of 5 cm from the ground. Under normal growing conditions this will simulate the height of grass eaten by cows. In drier climates or during sparse growing seasons, it may be necessary to sample nearer to the soil surface. After collection, samples can be composed for analysis. Larger samples can be reduced in the field to a more usable size by standard quartering techniques. A specific grass ecosystem sampling procedure as used by the IAEA is outlined in Annex VIII.

In the laboratory the vegetation may be analysed in a fresh state or, if time permits and the loss of radioiodine is not of concern, it may be dried. Generally, grass is dried at temperatures not exceeding 105°C for 24 hours. The dried matter

should be ground so that all particles pass through a 2-mm sieve. Care should be taken to use dust-tight containers. Alternatively, the samples can be dried and ashed. Activity on grass is reported on the basis of both weight (Bq/kg wet mass or Bq/kg dry mass) and area (Bq/m²).

5.2.3. Soil

Soil samples are usually collected for studying either total deposition or the availability of radionuclides to crops grown in cultivated agricultural land.

For deposition studies, samples should be collected in undisturbed areas to a depth of 25–50 cm with a coring tool. Sampling depths vary with the purpose of the study and the soil characteristics. The collection area should be flat, open terrain preferably covered with grass but without sheltering vegetation or structures. The soil should have moderate to good permeability. There should be little or no runoff during heavy rains or overwash. There should be a minimum of earthworm or rodent activity which could cause mixing of the soil. The sample should include the overlying grass and mat.

For radionuclide availability studies, samples should be taken in crop areas with a coring tool to a depth of 5 cm or to the depth of the plough line.

Sampling tools vary, but any device that will cut a straight core will be satisfactory. Samples should be taken with coring tools of known diameter so that the ground area represented by the sample is accurately known. In general, about 10 cores (or a total surface area of at least 200 cm²) are taken and composed to make a single sample.

Samples containing relatively high levels of activity should be kept separated from other samples from the time of collection throughout all subsequent processes to avoid cross contamination. At the laboratory, soil samples should be spread on a suitable surface, such as trays or plastic sheets, and allowed to dry at room temperature for several days. A slow-airflow, low temperature (50°C) drying cabinet will accelerate the drying process without loss of radionuclides from the soil. The dry mass of the material should be recorded. Whether the vegetation and organic debris are removed from the soil samples depends on the purpose of the study. If they are not discarded, the roots, mat and vegetation should be cut into very fine particles so as to be distributed evenly; if they are to be discarded, these materials should be collected and weighed. Stones should be collected, weighed and discarded. The mass of the discarded material should be taken into account, but not in calculating the specific activity of the soil. It provides, however, additional information to indicate the overall make-up of the growing area. In either case, the soil should be crushed, ground or pulverized to a particle size predetermined by the analytical requirements. The need for preparing the material will also depend on the nature of the soil; for example, beach sand may not need grinding. In general the soil should pass a mesh

size of 2 mm. Activity in soil samples is reported on a dry weight basis in Bq/kg *and* on an area basis in Bq/m² simultaneously.

5.2.4. Milk

Milk should preferably be collected at processing centres. It also can be collected from individual farms or from individual animals. For short periods, milk is usually stored in a refrigerator. A preservative, such as formaline or sodium azide (3.5 mL of 5% aq. solution per litre), can be added to prevent souring if a longer period of storage is anticipated.

It is, of course, essential to note the date of collection of the milk. At large processing centres, milk is pooled from several sources with different collection days at the local farms. The date of processing should therefore be taken as the reference date for sampling.

5.2.5. Other foods

Foods that contribute 5% or more to the total diet intake should be collected for analyses. Information on what foods are in this category is available from compilations of consumption statistics discussed in Section 2.2. Individual food samples are preferable for analyses rather than composite mixed diet samples. If, however, a mixed diet sample is to be used, a separate mixed diet of baby foods should also be analysed.

Regional food items that are highly contaminated will contribute substantially to the total radioactivity intake even though the amount of food intake is small. These foods are generally well known to the national health authorities from previous experience. Where few data are available, a screening procedure should be adopted to identify such items for analyses in the case of an accident.

Food sampling should be undertaken first at the producer's and subsequently at the retail market. In general, food products should be prepared as for home use. The samples should be cleaned and the edible portions retained for analyses. Masses are determined by subtracting the inedible portion from the total since loss of moisture from the edible portion during the time of preparation may amount to a sizeable fraction of the mass. For fish, poultry and meat samples, the bones are easily separated after heating for one hour at 150°C. The mass of the sample must be referred to the mass of the genuine material after subtracting the mass of the bones. For direct measurements, 1 kg of fresh sample is generally sufficient.

When ashing foods, the temperature should be raised slowly until the upper limit of the temperature prior to ignition has been reached. The preliminary ashing temperatures of some standard food materials are listed in Table IV. For those foods not listed, it is important to recall that foods with high phosphorus content tend to ignite.

TABLE IV. PRELIMINARY ASHING TEMPERATURES [9]

Material	Temperature °C
Eggs	150–250
Meat	150–250
Fish	150–250
Fruit (fresh/canned)	175–325
Fruit juices	175–225
Milk	175–325
Vegetables (fresh/canned)	175–225
Root vegetables	200–325
Flour	175–250
Dry beans	175–250
Grains	225–325
Macaroni	225–325
Bread	225–325

When the upper limit has been reached, the temperature can be raised more rapidly to 450°C and samples can be ashed for about 16 hours. Temperatures higher than 450°C may result in losses of volatile radionuclides, e.g. radiocaesium. With proper adjustment, ignition can be avoided for all materials except those with large amounts of fat. Usually, 10–25 g of ashed samples are sufficient for analyses. Table V lists the fresh weight required for 10 g of ash for some standard foods [13].

5.2.6. Water

Tap water should be collected at the water processing (filtration/purification) plants just prior to discharge into the distribution system. If the water is to be collected from a residence, then the pipes should be flushed sufficiently (two or three minutes) prior to sample collection.

Rain collectors 0.1 m² to 1 m² in area provide adequate collection of rain water (Fig. 10 shows a typical rain water collector). Automatic sampling devices are commercially available which protect the collector from dry deposition prior to the rainfall. These samplers start to open the collection area when the rain begins to fall and close it when the rain stops. High-walled vessels with smooth surfaces are equally suitable. Some loss of the less soluble radionuclides will occur on either of

TABLE V. MASS OF FOOD REQUIRED FOR 10 GRAMS OF ASH [13]

	% ash ^a	Weight of original food (kg)
Beans (dry)	3.8	0.3
Eggs (shelled)	1.0	1.0
Fish	1.3	0.8
Flour	0.48	2.1
Fruit (canned)	0.27	3.7
Fruit (fresh)	0.62	1.6
Juices (fruit)	0.61	1.6
Macaroni	0.7	1.4
Meat	0.92	1.1
Potatoes	1.1	0.9
Poultry	0.81	1.2
Rice	0.65	1.5
Shellfish	1.8	0.6
Vegetables (fresh)	0.75	1.3
Vegetables (canned)	1.1	0.9
Vegetables (root)	0.76	1.3
Bread (white)	2.1	0.5
Bread (whole wheat)	2.4	0.4
Milk (liquid)	0.7	1.4
Milk (powder)	6	0.2
Milk (buttermilk powder)	11	0.1
Wheat	1.7	0.6

^a % ash is an average value found in routine work. Variations have been found as large as 25% and are dependent upon particular sample composition and ashing conditions.

these collectors but the loss can be largely recovered (if desirable) by washing with dilute acid (0.1N HCl). An alternative method is to filter the water directly through a mixed bed ion exchange column, after which the water is drained away. Contamination of rain water samples by airborne soil and surface dust can be minimized by locating the sampling stations on the roofs of buildings. Overhanging vegetation should be avoided. The most suitable size for the collector depends upon the amount and frequency of precipitation in the area, as well as the frequency of collection. For



FIG. 10. Typical rain water collector.

areas receiving 5–25 cm precipitation per month, a collecting area of 0.1–0.2 m² is suggested. All materials in the collector must be transferred with great care to ensure that all the radioactive material is collected. The sides and bottom of the container should be scrubbed several times with a rubber spatula and rinsed with distilled water. If the containers are re-used, care must be taken to avoid a build-up of contamination that will influence subsequent samples.

In areas receiving significant amounts of snow during periods of radioactive deposition, the precipitation may be sampled. This may be done by collecting a square metre area of snow to a depth sufficient to include the new snowfall.

In collecting surface water from rivers, care should be taken to avoid stagnant areas. Lake water samples should not be taken near the shore line.

If water samples have to be stored for any length of time, hydrochloric acid (11M) should be added at the rate of 10 mL per litre of sample to sample bottles either prior to sampling or as soon as possible after sampling to avoid adsorption of radionuclides on the walls of the container. The longer the storage time before analysis the more important it is to acidify water samples.

Section 6

ANALYTICAL METHODS

It is assumed that normal analytical laboratory procedures are applied. Special attention must be paid to the problem of contamination when both low and high level samples have to be analysed in the same facility. Direct instrumental analysis without pretreatment can be performed on highly contaminated samples and perhaps on all samples for determination of gamma emitters.

It will be sufficient if the local monitoring laboratories are capable of measuring gamma emitting radionuclides such as ^{137}Cs at concentrations of 5–10 Bq/kg (or per litre) by direct measurement. It is recommended that counting geometries be uniform at all the local laboratories but this may not be practical where these facilities are used for other purposes (see Section 4.2.1). However, all local laboratories should be calibrated against a central laboratory. The detection level for the central laboratory should be much lower, 0.1–1 Bq/kg, because of more sophisticated equipment, longer counting time and/or more extensive sample preparation. In general the detection limit should be a small fraction of the action level (1–10%) for a specific radionuclide.

Action levels are specific to the event and the radionuclide and will not be discussed in detail in this guidebook. In general, monitoring results can be used to estimate potential doses to man. The need for further protective measures can then be determined from a comparison with the intervention levels of dose or with the levels of dose expressed in terms of levels of radionuclides present in appropriate environmental materials, i.e. the derived intervention levels. The international health and radiation organizations have published [14-21] guidance on the principles for establishing intervention levels of dose at which it may be necessary to introduce appropriate measures for the protection of the public or to control the level of radionuclides in foods moving in international trade.

6.1. GAMMA SPECTROMETRY

The outstanding advantage of gamma ray spectrometry is the ability to measure gamma emitters directly in the original sample without the need for chemical separations. Gamma ray spectrometry allows both qualitative identification and quantitative determination of the radionuclides in the sample. A list of the more important gamma emitters, from a human exposure standpoint, together with their energies and those of interfering radionuclides, is given in Annex VII.

The form in which the sample is presented to the gamma ray detector depends on the sample type, the available equipment, the composition of radionuclides and the level of activity. Some standard sample containers include nylon planchets, aluminium cans and moulded Marinelli beakers. Measuring time varies according to

sample type, required detection limits, detection efficiency, and radionuclides of interest. For a description of gamma ray spectrometric methods for the analysis of foods see Annex I.

Some specific recommendations for the effective and accurate use of gamma ray spectrometry for analyses of matrices of interest are as follows:

- Sample geometries must be selected for the matrices of interest including air filters, water, vegetation, milk, fresh vegetables and other foods, and fresh water and marine organisms.
- The geometries must be calibrated for the densities of the sample of interest as a function of gamma ray energy. This involves the preparation of calibration curves of gamma ray counting efficiency versus energy.
- In preparing the calibration curves, standard preparations of radionuclides from an organization such as the National Bureau of Standards (USA) or other reliable sources must be used (see Section 7.2).
- Calibration curves for unit density materials including water and meat can be made by using known amounts of radioisotopes in aqueous solution in the sample containers of interest. Samples of greater or less than unit density should be radiolabelled with the appropriate radionuclide(s), and calibration curves should be prepared on the labelled matrix.
- Radionuclide standards such as ^{137}Cs and ^{60}Co should be counted daily to ensure that the gamma ray spectrometer is operating correctly (see Section 7.4).

6.1.1. Evaluation of the spectra

When purchasing new gamma ray spectrometry equipment it is advisable to purchase computer software to analyse photopeak areas and process the data in terms of radionuclide content [22]. Software programs should be checked by manual calculations for accuracy when first implemented and occasionally thereafter. Figure 11 illustrates the type of information that can be obtained from modern gamma ray spectrometry analysis systems.

The Analytical Quality Control Services (AQCS) of the IAEA provides a set of data comprising nine Ge gamma ray spectra in different forms. The performance of computer software can be evaluated, using these data, for the following parameters:

- Singlet peaks: detectability, position and area
- Doublet peaks: position and area.

If it is necessary to process the gamma-ray spectra manually, the techniques needed are well described in Refs [23] and [24].

P E A K A N A L Y S I S

PK	CENTROID CHANNEL	ENERGY KEV	FWHM KEV	BACKGND COUNTS	NET AREA COUNTS	ERROR %	NUCLIDES
17	27.43	27.42	1.9	34122.	105339.	1.7	
27	31.25	31.24	1.9	33229.	51142.	3.1	
37	35.57	35.56	1.9	32220.	23511.	5.2	
PEAK AT CHANNEL 42.2 DROPPED FROM MULTIPLT ANALYSIS							
4	46.48	46.49	2.3	32336.	1141.	21.1	
5C	59.47	59.49	2.2	37406.	6894.	3.9	AM-241
6N	103.00	103.08	3.6	125492.	703.	42.1	
7N	108.39	108.47	3.6	127856.	1661.	21.5	
8N	112.86	112.95	3.6	129659.	2541.	17.2	
9N	117.19	117.29	3.6	131405.	2515.	17.4	
10N	121.92	122.02	3.6	133313.	2949.	15.9	SE-75,CO-57
11N	166.78	166.96	3.7	133613.	6570.	6.9	U-235
12N	190.98	191.17	3.7	136095.	5889.	7.0	NA-141,FE-59
13	351.69	352.98	3.3	35703.	467.	57.4	PF-214
14C	427.51	428.01	2.2	32889.	1927.	19.8	SH-125
15	463.47	464.01	2.6	34509.	775.	33.0	CS-138
16C	562.41	563.09	2.0	9313.	527.	19.6	CS-134, SH-122
17C	568.81	569.50	2.0	8908.	923.	17.3	CS-134, BI-207
18C	600.01	600.74	2.1	7984.	817.	13.5	SH-125, SH-124
19C	604.06	604.80	2.1	7817.	6148.	3.4	CS-134, SH-125
20	635.22	636.01	1.9	7960.	495.	25.9	SH-125,I-131
21?	656.05	656.86	2.2	4241.	1656.	36.3	AG110M,RP-89
22?	666.88	661.70	2.2	4743.	447601.	0.2	CS-137
23	723.11	724.02	3.4	2070.	196.	33.6	SH-124,I-131, ZR-95
24C	794.84	795.85	2.3	1704.	4551.	2.6	CS-134
25C	800.90	801.93	2.3	1605.	416.	12.9	CS-134
26	833.91	834.99	2.7	1610.	798.	7.9	GA-72,KR-88, MN-54,I-135
27	935.97	937.21	2.2	1451.	114.	48.2	AG110M
28	1113.93	1115.45	2.6	1025.	2612.	2.7	ZN-65
29	1171.51	1173.13	2.7	664.	8829.	1.2	CO-60,CO-56
30	1272.72	1274.50	1.9	276.	102.	27.1	NA-22
31	1330.51	1332.39	2.8	195.	7994.	1.1	CO-60
32	1363.26	1365.20	2.8	95.	115.	15.8	CS-134
33	1382.27	1384.24	3.7	87.	59.	27.4	SK-92,AG110M
34C	1398.18	1400.18	2.5	60.	23.	39.2	I-132
35C	1405.70	1407.72	2.5	75.	79.	23.8	
36C	1458.64	1460.74	2.6	54.	95.	15.2	K-40
37	1761.72	1764.38	2.6	18.	70.	15.4	BI-214

ERROR QUOTATION AT 1.00 SIGMA
PEAK CONFIDENCE LEVEL AT 68.0%

C - MULTIPLT ANALYSIS CONVERGED NORMALLY
N - MULTIPLT ANALYSIS DID NOT CONVERGE
? - MULTIPLT ANALYSIS CONVERGED BUT GFIT > 4

SAMPLE: FZS SCHLAMM
 DATA COLLECTED ON 11-MAR-88 AT 12:10:00
 DECAYED TO 0. DAYS, 0.0000 HOURS BEFORE THE START OF COLLECT.

R A D I O N U C L I D E A N A L Y S I S R E P O R T

NUCLIDE	ACTIVITY CONCENTRATION IN BQ /ML		DL-TOTAL COUNTS
	MEASURED	ERROR	
AG-110M	LLD<9.95E-02	LLD<9.95E-02	905.82
AM-241	6.23E-01 +-2.60E-02	6.23E-01 +-2.60E-02	
AR-41	LLD<5.20E-03	LLD<5.20E-03	28.25
AS-76	LLD<4.23E-02	LLD<4.23E-02	206.03
AU-198	LLD<2.62E-02	LLD<2.62E-02	344.83
BA-133	LLD<3.69E-02	LLD<3.69E-02	355.50
BA-139	LLD<9.39E-02	LLD<9.39E-02	455.63
BA-140	LLD<8.62E-02	LLD<8.62E-02	230.55
BA-141	LLD<6.09E-02	LLD<6.09E-02	512.22
BE-7	LLD<2.61E-01	LLD<2.61E-01	330.27
BI-207	LLD<1.92E-02	LLD<1.92E-02	202.19
CO-109	LLD<4.04E-01	LLD<4.04E-01	473.97
CE-139	LLD<2.07E-02	LLD<2.07E-02	455.63
CE-141	LLD<3.35E-02	LLD<3.35E-02	483.10
CE-144	LLD<1.53E-01	LLD<1.53E-01	495.70
CO-56	LLD<9.49E-03	LLD<9.49E-03	76.05
CO-57	1.11E-01 +-1.77E-02	1.11E-01 +-1.77E-02	
CO-58	LLD<9.48E-03	LLD<9.48E-03	77.99
CO-60	1.41E+00 +-2.05E-02	1.41E+00 +-2.05E-02	
CR-51	LLD<2.22E-01	LLD<2.22E-01	364.33
CS-134	6.09E-01 +-2.09E-02	6.09E-01 +-2.09E-02	
CS-136	LLD<9.61E-03	LLD<9.61E-03	78.97
CS-137	5.47E+01 +-2.46E-01	5.47E+01 +-2.46E-01	
CS-138	LLD<4.61E-03	LLD<4.61E-03	15.23
FE-59	LLD<1.59E-02	LLD<1.59E-02	58.05
HF-181	LLD<3.16E-02	LLD<3.16E-02	320.48
H6-203	LLD<2.47E-02	LLD<2.47E-02	374.27
I-131	LLD<2.81E-02	LLD<2.81E-02	341.01
I-132	LLD<1.00E-01	LLD<1.00E-01	895.11
I-133	LLD<2.75E-02	LLD<2.75E-02	239
I-137	LLD<1.17E-02	LLD<1.17E-02	245.57
AE-138	LLD<8.54E-02	LLD<8.54E-02	393.91
Y-88	LLD<1.06E-02	LLD<1.06E-02	76.98
Y-91M	LLD<3.72E-02	LLD<3.72E-02	209.44
ZN-65	7.91E-01 +-2.18E-02	7.91E-01 +-2.18E-02	
ZR-95	LLD<1.74E-02	LLD<1.74E-02	82.99
ZR-97	LLD<1.05E-02	LLD<1.05E-02	86.12
TOTAL	5.92E+01 +-2.78E-01	5.92E+01 +-2.78E-01	

ERROR DUPLICATION AT 1.00 SIGMA
 LLD CONFIDENCE LEVEL AT 68.02

FIG. 11. Typical data obtained from a modern gamma ray spectrometer system.

6.2. RADIOCHEMISTRY

Reliable methods are presented in the annexes for ^{89}Sr and ^{90}Sr (Annex II), for tritium (Annex III) and for plutonium, americium and curium (Annex IV). It is obviously impracticable to include in this guidebook all the reliable methods currently available. The methods described here have been selected on the basis of experience. Sources for related information and other reliable methods are given in the bibliography of each annex.

6.2.1. Analysis of strontium

The most commonly used method for separating strontium is by nitrate precipitation. With some modifications this method can be applied to all kinds of environmental samples and foods. Methods for determination of strontium in milk and cheese, other foods and water are given in Annex II-1, and a modified method for soils in Annex II-2. Methods of calculating the ^{90}Sr , ^{89}Sr and Ca contents of the samples are given in Sections II-3, II-4 and II-6, respectively, of Annex II.

The chemical yield varies according to the type of material. The use of ^{85}Sr tracer to determine chemical yield is discussed in Annex II-7. When determining yield in this manner, it is important that the tracer is pure ^{85}Sr , i.e. free from ^{89}Sr and ^{90}Sr .

Although the method is time consuming, it is reliable and safe. Rapid methods for ^{90}Sr analysis exist, and it has been shown that they can be used after short lived nuclides have decayed. In fresh fallout situations, the nitrate precipitation method has been shown to be more reliable. Also, during periods of fresh fallout, the amount of ^{89}Sr is of interest and the rapid methods can only analyse for ^{90}Sr . In the case of higher contamination with ^{90}Sr , the daughter ^{90}Y can be separated without waiting for equilibrium. Within 10 hours the activity concentration of ^{90}Y will be approximately 10% of the equilibrium value and may be sufficient for a reliable ^{90}Sr analysis.

A special application of liquid scintillation counters is the measurement of Cerenkov radiation produced by beta emitters with beta energies greater than 260 keV. This application can be used for screening samples for ^{90}Sr [25].

6.2.2. Analysis of tritium

A method for determination of tritium is presented in Annex III. Tritium is measured by liquid scintillation counting of a portion of a distilled sample. Several reagents (such as sodium sulphite and silver iodide) can be added in the distillation to prevent interference of radioiodine. The allowed concentration of tritium in water is relatively high for human consumption; thus the method presented in Annex III is normally adequate for routine determinations. However, if required, lower

concentrations of tritium in water can be determined by electrolytic enrichment. Some reference methods on the topic of tritium enrichment by electrolysis are presented in Ref. [26].

6.2.3. Analysis of the transuranic elements

A method for determination of transuranic elements (plutonium, americium and curium) by alpha spectrometry after sequential radiochemical separation is presented in Annex IV. This method has been applied to a variety of environmental samples.

The technique of isotopic dilution is incorporated in the method for quantitative determination of plutonium isotopes ($^{239/240}\text{Pu}$ and ^{238}Pu), curium isotopes (^{242}Cm and ^{244}Cm) and ^{241}Am . Chemical recoveries are also determined by this technique by the use of either ^{236}Pu or ^{242}Pu as the plutonium tracer and ^{243}Am as the tracer for americium and curium. The chemistry of curium is so similar to that of americium that there is no need of a curium tracer, i.e. the americium tracer serves as a yield monitor for both elements. Quantitative determinations are derived by alpha spectral analyses for these elements.

Section 7

ANALYTICAL QUALITY CONTROL

Quality control measures are necessary to provide documentation to show that the analytical results are reliable. This is very important since analytical results can be the basis upon which economic, administrative, medical and/or legal decisions are made.

Reliability of results is a function of precision (reproducibility) and accuracy (true value). The precision of results can easily be determined by internal measurement. The determination of accuracy, however, in most cases requires more detailed procedures such as:

- Analysis by as many different methods, analysts and techniques as possible. In cases where agreement is good, results are assumed to be accurate.
- Control analysis with reference materials that are as similar as possible to the materials to be analysed. Agreement between certified and observed values is then a direct measure of accuracy for that particular determination.
- Participation in an interlaboratory comparison. Samples used in such an inter-comparison should be, as far as possible, similar in composition and concentration to the samples to be analysed on a routine basis. The agreement of results received from a particular laboratory with the most probable mean value obtained from statistical evaluations of all the results will be a measure of the accuracy for that particular determination.

For practical reasons, such as the following, most analytical laboratories are not in a position to check accuracy internally without an external source of reference materials:

- Frequently resources are available for only one method and/or technique;
- With few exceptions, reference materials, particularly in the case of trace analysis, are not normally prepared by the institutes themselves;
- Intercomparisons are organized on a rather limited basis and many important materials and/or analytes have not yet been covered.

7.1. ANALYTICAL QUALITY CONTROL SERVICES (AQCS) PROGRAMME

To overcome some of the difficulties in checking the accuracy of analytical results, the IAEA provides the Analytical Quality Control Services (AQCS) Programme to assist laboratories to assess the quality of their work. AQCS coordinates intercomparison studies and supplies reference materials [27,28]. Participation is on a voluntary basis and at minimum cost. Information supplied by laboratories taking part in the intercomparisons is treated as confidential.

The Agency's AQCS Programme provides three main types of material:

- Materials that can be used in analytical laboratories working in the fields of nuclear technology and isotope hydrology. These include uranium ore reference materials and other substances relevant to nuclear fuel technology as well as stable isotope reference materials for mass spectrometric determination of isotope ratios in natural waters.
- Materials with known content of uranium, thorium and/or transuranic elements or fission products for determination of environmental radioactivity or control of nuclear safety.
- Materials for use in the determination of stable trace elements in environmental or biomedical research. Radiochemical methods such as neutron activation or isotope dilution analysis are often used in the determination of such trace elements and constitute an important contribution of nuclear techniques to applied science.

7.2. CALIBRATION AND STANDARDS

It is desirable and often necessary that calibrations of measurement systems be carried out with standards of the radionuclides to be determined. Standards should be accompanied by certificates which specify activity, purity and accuracy. Standards and/or reference materials are available from several organizations and institutions (for names and addresses see Annex X).

It is important that calibration be prepared with standard samples that are of similar chemical composition, are of similar concentration for the relevant radionuclides, and are in the same sample geometry and counting configuration as the real samples. Calibration sources are available in standard (such as Marinelli beakers) and custom-made configurations.

When the desired standards are not available, a reasonable approximation of counting efficiency can be made by determining the efficiency of the detectors for beta particle and gamma ray energies which are both higher and lower than the energies of interest and interpolating to the energy of interest.

7.3. INTERCOMPARISONS

As discussed in Section 4.2.1, it is important that the local and central laboratories should be intercalibrated on a routine basis. In addition, participation in inter-laboratory comparisons, such as those carried out by the Agency (see Section 7.1), is strongly recommended. It is important that the newer laboratories adopt the view

that participation in international intercomparisons is a normal part of the laboratory's operations. Participation on a regular basis will detect systematic errors, i.e. those due to improper calibration, contamination or incorrect methods of calculation.

7.4. INTERNAL CONTROL PROGRAMMES

The laboratory should operate a routine programme to maintain the equipment and the validity of the results.

7.4.1. Equipment performance

In addition to intercomparisons, the laboratory should routinely check the status of equipment by checking background and standard samples. It is suggested that quality control charts be kept for each system on a routine basis.

7.4.2. Analytical control samples

The validity of the analytical results should be checked by an internal standards programme which incorporates the use of blind duplicates, blanks and standards. These results often give the first indication of analytical difficulties. Analytical control samples, submitted with each group of samples to be analysed, generally constitute 10–15% of the total samples.

Laboratory control standards can be developed by preparing large, homogeneous batches of material for various sample types. By analysing these controls in duplicate or triplicate, sufficient data will be available in a short time to establish the "standard" value. This material can also be run by several other reliable laboratories as a check.

7.5. DATA PRESENTATION

The results of the sample analysis should be presented in such a way that the reader can evaluate the data and the conclusions. The presentation should discuss as a minimum:

- Location, collection method and preparation procedures;
- Analytical method and specifications of the equipment.

In the ideal case, a report on environmental radioactivity should contain tables in which the data have the following characteristics:

- (a) The International System (SI) units and symbols should be used (see Annex V). Some standard units of presentation are:
- Air in Bq/m^3
 - Water and milk in Bq/L
 - Deposition in Bq/m^2
 - Soil in Bq/kg dry mass and Bq/m^2
 - Grass in Bq/kg dry mass and Bq/m^2
 - Foods in Bq/kg fresh mass.
- (b) The value should be given to the correct number of significant figures [29].
- (c) The precision of the data should be indicated and the form of expressing this data should be clearly explained, e.g. the standard error, the two-sigma error, the counting error. Where possible the accuracy of the data should be indicated, generally by reference to the supporting data for the particular method used.
- (d) Where low levels of activity are indicated, the lower limit of detection should be indicated and the method used for its calculation should be referenced.

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Annex I

METHOD FOR DETERMINING GAMMA EMITTERS ¹

This method can be used to determine gamma-emitting radionuclides with energies ranging from 60 keV to 2000 keV in a large variety of sample matrices. However, the user is expected to have some previous training and experience in gamma ray spectroscopy, since it is beyond the scope of this guidebook to provide the necessary details for an inexperienced user.

I-1. PRINCIPLE AND APPLICATION

I-1.1. Principle

This method describes the use of gamma spectrometry for the measurement of gamma photons emitted from radionuclides in a sample without the need to separate the radionuclides from the sample matrix. The simultaneous detection of several gamma emitters in the sample material is carried out with a shielded germanium semiconductor detector of high resolution connected to a multichannel analyser (MCA) with at least 4096 channels and associated data input and output units. Automatic processing of the collected spectral data can be conveniently controlled by a computer system with selected software and is the recommended means of processing data.

I-1.2. Application

The method can be applied to a large variety of environmental and biological materials such as air, water, soil, sediments, vegetation (grass, hay, etc.) and, particularly, individual foods of vegetable and animal origin as well as total diet mixtures. The method is suitable for the surveillance and monitoring of radioactivity originating from the operation of nuclear plants, tests of nuclear weapons, and releases from nuclear accidents.

¹ Taken from laboratory procedures by R.F.W. Schelenz, reported in KANISCH et al. (1984) (see Bibliography, Annex III).

I-2. COLLECTION AND PREPARATION OF SAMPLES

I-2.1. Sample collection

The type and quantity of samples to be collected as well as the collection procedures are described in the first part of the guidebook, in Sections 2 and 5. It is always advisable to put all sample materials into tight containers such as polyethylene containers with screw caps or into sealable polyethylene bags. The gamma measurements can often be made directly on the samples in the original container without any sample processing other than crushing and compacting the material to achieve a calibrated geometry.

I-2.2. Sample preparation

Procedures for the preparation of various materials are discussed in the first part of the guidebook, in Sections 5 and 6. Particular care should be taken not to exceed a temperature of 400°C in the ashing process in order to avoid loss of volatile radionuclides, e.g. ¹³⁷Cs. It is recommended that the ashed material be crushed in a ball mill to obtain as homogeneous a sample as possible. The uncombusted carbon which appears as black particles in the ash does not interfere in the gamma ray measurements.

I-3. REAGENTS AND MATERIALS NEEDED

I-3.1. Reagents

Nitric acid, concentrated
Formaline
Acetone, technical grade
EDTA (ethylenediaminetetraacetic acid, tetra sodium salt)

I-3.2. Apparatus

Wide-mouth polyethylene bottles, various sizes with screw caps
Plastic bags, freezer type
Marinelli beakers, one-litre size with lids
Ball mill
Grinder
Sample cutter or shredder
Sample mixer or blender

I-3.3. Standards

Standard solutions of mixed radionuclides and solid standards of mixed radionuclides in several geometrical configurations identical to the sample containers are necessary for calibrating the counting system.

I-4. GAMMA SPECTROMETRY SYSTEM

It is recommended that the spectrometry system be a fully integrated data acquisition and computation system comprising the following items:

- A vertical high purity germanium (HPGe) or a lithium-drifted germanium (Ge(Li)) detector is recommended. The detector should have an efficiency of 18–20%. Generally, the efficiency of germanium detectors is specified as the photopeak efficiency relative to that of a standard 7.62 cm × 7.62 cm cylindrical NaI(Tl) scintillation crystal and is normally based on the measurement of the 1.33 MeV gamma ray photopeak of a ⁶⁰Co source with a source detector spacing of 25 cm used in both measurement systems. The resolution of the detector — which is normally specified for germanium detectors as the full width (in keV) at half maximum (FWHM) of the full energy peak of the 1.33 MeV peak of ⁶⁰Co — should be between 1.8 keV and 2.2 keV. It is recommended that the peak-to-Compton ratio of the detector be greater than 46:1. The peak-to-Compton ratio is defined as the ratio of the count in the highest photopeak channel to the count in a typical channel just below the associated Compton edge and is conventionally quoted for the 1.33 MeV gamma ray photopeak of ⁶⁰Co.
- A preamplifier is necessary. This is normally an integral part of the detector unit and is located very near the detector in order to take advantage of the cooling which is necessary for the operation of the detector and which aids the preamplifier to operate with low noise.
- A bias high voltage power supply is required to supply high voltage to the detector through the preamplifier. A power supply of 1500–5000 V is adequate for the operation of germanium detectors.
- A linear amplifier to process the output signals from the preamplifier is required.
- A detector shield will be needed with a cavity adequate to accommodate large (up to 4 litres) samples and constructed of either lead or steel with some type of graded liner to degrade X-rays. Lead shields have a much lower backscatter effect than steel shields. The back scatter effect also decreases as the internal dimensions of the shield increase. Typically, lead shields have walls 5–10 cm

thick lined inside with graded absorbers of cadmium (~ 1.6 mm) and copper (~ 0.4 mm). Other materials such as plexiglass and aluminium are also used for absorbers.

- A multichannel analyser (MCA) with a minimum of 4096 channels should be connected to a keyboard and display screen for input and output of data and interaction with a computer. Several kits are available for the conversion of personal computers (PCs) into MCAs. Basically there are three types of conversion kits. One type makes use of a board with analog-to-digital converter (ADC) that simply clips in the PC; a second type uses a clip-in board with an external ADC; and the third type uses a multichannel buffer (MCB) connected to the PC. All of these PC-based MCA systems are relatively inexpensive and very suitable for use in germanium and sodium iodide gamma ray spectrometry.
- A rapid data storage and recovery system is needed. It can consist of magnetic tape, hard disc, floppy disc, or a combination of these media. This system can be used for programming, short term storage of data, and archiving data.
- A high speed printer for data output is required. Useful, but not absolutely necessary, is a plotter for archiving spectral drawings.
- Software for system operation and data reduction is usually supplied with the MCA system. Software packages with varying features and capabilities are available for MCAs based on PCs (see Annex IX). A comparison of available PC software has been presented by Sanderson in Ref. [I.1].

Figure 12 is a block diagram of a typical gamma ray spectrometry system.

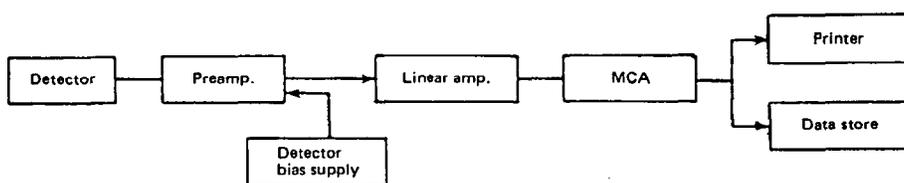


FIG. 12. Block diagram of a typical gamma ray spectrometer system.

I-5. INTERFERENCES AND PROBLEMS

I-5.1. Sources of error

Interferences associated with gamma determinations may be caused by improper spectral identities, changes in background, errors in calibration and/or geometry, and lack of homogeneity in samples.

I-5.2. Problems

Many of the problems in gamma ray spectrometry are due to malfunctions of electronic components. An extensive presentation of the problems associated with the determination of gamma-emitting nuclides with germanium semiconductor detectors under practical working conditions was published by Debertin (Ref. [I.2]).

I-6. CALIBRATION

I-6.1. Energy calibration

An essential requirement for the measurement of gamma emitters is the exact identity of photopeaks present in a spectrum produced by the detector system. The procedure for identifying the radionuclides within a spectrum relies upon methods which match the energies of the principal gamma rays observed in the spectrum to the energies of gamma rays emitted by known radionuclides. This procedure can be performed either by manual inspection or by computer analysis. In either case, one must have an accurate energy calibration for the germanium detector system so that correct energies may be assigned to the centroid of each full energy peak (FEP) in a sample spectrum. The energy calibration of a germanium detector system (i.e. establishing the channel number of the MCA in relation to a gamma ray energy) is made by measuring mixed standards sources of known radionuclides with well-defined energies within the energy range of interest, usually 60 keV to 2000 keV. The use of the lower energy photons emitted by ^{241}Am may indicate changes in the intercept. Mixed gamma ray standards are available in various forms and containers from reliable suppliers (see Section 7.2). A partial list of radionuclides usually available with gamma energies in the range of interest is presented in Table VI. The energy calibration source should contain a selection of radionuclides with at least four different gamma ray energies. It is recommended that one of the nuclides should be ^{137}Cs . The gain of the system should be adjusted so as to position the 662 keV photopeak of ^{137}Cs at about one-third full scale. It is also recommended that the gain of the system be adjusted to 0.5 keV/channel. Once these adjustments are made, the gain of the system should remain fixed.

The energy calibration source should be counted long enough to produce well defined photopeaks. The channel number that corresponds to the centroid of each FEP on the MCA should be recorded and plotted on linear graph paper on the X-axis versus the gamma ray energy on the Y-axis. A linear curve will result in the plot of these data if the system is operating properly. The slope and intercept of the energy calibration line should be determined by least squares calculations. Computerized systems are usually equipped to perform their slope and intercept calculations automatically during the calibration routine. The system should be

TABLE VI. RADIONUCLIDES SUITABLE FOR ENERGY CALIBRATIONS

Nuclide	E_{γ} (keV)
Am-241	59.54
Cd-109	88.03
Co-57	122.06 136.47
Ce-139	165.85
Cr-51	320.08
Na-22	511.00 1274.54
Cs-137	661.66
Mn-54	834.84
Co-60	1173.24 1332.50

checked each day of operation for the stability of the slope and intercept by the measurement and plot of at least two different gamma energies. An excellent source for routine checks of the system's performance is a sealed source of ^{226}Ra in equilibrium with daughter activities. This type of source will produce four major photopeaks with the following energies: 351.9 keV, 609.3 keV, 1120.3 keV and 1764.5 keV.

I-6.2. Efficiency calibration

An accurate efficiency calibration of the system is necessary to quantify radionuclides present in a sample. It is essential that this calibration be performed with great care because the accuracy of all quantitative results will depend on it. It is also essential that all system settings and adjustments be made prior to determining the efficiencies and be maintained until a new calibration is undertaken. Small changes in the settings of the system components may have slight but direct effects on counting efficiency.

Some points to consider in determining the efficiency calibration are: (1) sample counting configuration (geometry), (2) calibration method, (3) calibration sources, and (4) analytical efficiency expressions.

I-6.2.1. Sample counting configuration

For routine, reproducible sample analyses, the containers used for counting must be selected taking into account both the quantity of sample material available and the sensitivity acquired by the geometry of the sample in the container. In practice, it is recommended that several containers with practical geometries be selected in accordance with the sample matrices to be measured. Some examples of sample containers are sealable plastic bags for filters, Marinelli beakers for both liquids and solids, cylindrical plastic containers with screw caps (bottles and jars), petri dishes and aluminium containers of various dimensions for small volumes of soils and ashed materials (see Section 6.1). In general, the dimensions of the container should be well suited to the dimensions of the detector and lead housing, e.g. not too tall or too thin.

I-6.2.2. Calibration method

Several theoretical efficiency calibration methods are in use today. It is recommended, however, that efficiency calibration be determined experimentally for environmental measurements even though this method takes more effort and is time consuming. Practical calibration standards must be prepared for each counting configuration from appropriate certified radionuclides. The composition of these standards should approximate as closely as possible, with respect to density and attenuation factors, to the actual samples to be analysed. The volume and/or height within each configuration must be the same for standards and samples.

I-6.2.3. Calibration sources

Appropriate radionuclides must be selected for use as standards in efficiency calibration. Solutions of certified mixed radionuclides with reasonably long half-lives are available from several reputable suppliers (see Section 7.2). Accurate absolute gamma ray emission rates should be stated in the certificates supplied with the standards. The certificate should also state the following characteristics of the standard:

- Uncertainty associated with activity
- Reference date
- Purity
- Mass or volume
- Chemical composition

TABLE VII. RADIONUCLIDES USED FOR EFFICIENCY CALIBRATION

Nuclide	Half-life ^a	E _γ (keV)	Photon per decay
Na-22	950.4 d	511.00 1274.54	1.807 0.9994
Sc-46	83.80 d	889.28 1120.55	0.99984 0.99987
Cr-51	27.71 d	320.08	0.0985
Mn-54	312.5 d	834.84	0.99975
Co-57	271.84 d	122.06 136.47	0.8559 0.1058
Co-60	1925.5 d	1173.24 1332.50	0.9990 0.999824
Cd-109	436 d	88.03	0.0368
Cs-137	30.0 a	661.66	0.850
Ce-139	137.65 d	165.853	0.800
Ce-141	32.50 d	145.44	0.489
Hg-203	46.612 d	279.20	0.813
Am-241	420.0 a	59.54	0.360

^a The half-life is given in days (d) and years (a).
One year = 365.25 days.

- Values of half-lives
- Branching fractions for all modes of decay
- Method(s) of measurement of the radionuclides in the standard.

If radionuclides, such as ⁶⁰Co, ⁸⁸Y, ¹³³Ba or ¹⁵²Eu, that decay by cascade transitions and produce multilined spectra, are used in the efficiency calibration, great care must be taken to correct for the counting losses created by coincidence summing effects (Ref. [1.3]).

Radionuclides that are used for determinations of efficiency can be classified into two groups: those radionuclides with only a few prominent gamma rays, and those with many prominent gamma rays. The radionuclides listed in Table VII belong to the first group and have been used extensively for efficiency calibrations. By the proper selection and combination of these radionuclides, an efficiency curve can be determined over the energy range of interest (usually 60 keV to 2000 keV). The energy range must be adequately covered by calibration points so that interpolation between the points is accurate. It is recommended to take calibration points approximately every 50 keV from 60 keV to 300 keV, approximately 200 keV from 300 keV to 1400 keV, and at least one calibration point between 1400 keV and 2000 keV. This spacing of calibration points is adequate to define the selected energy range, particularly the region from 100 keV to about 2000 keV. In the region of 60 keV to 300 keV in which large variations in efficiency are prevalent, it may be necessary to calibrate with a specific radionuclide when the purpose of the measurement is to determine that specific radionuclide more precisely.

I-6.2.4. Analytical efficiency expressions

Once a sufficient number of data are acquired experimentally in the energy region of interest, a means of representing the efficiency as a function of energy should be chosen. A graphic log-log plot of energy on the X-axis and efficiency on the Y-axis can be used, although it is frequently more useful and desirable to express the efficiency in some analytical mathematical form as a function of gamma ray energy. An expression of this type can be readily programmed and is adaptable to automatic data analyses.

Least squares fitting procedures are used to fit the efficiency data to an analytical expression. Such a technique has the advantage of yielding an unbiased estimate of the fitting errors. Care must be taken in selecting the analytical expression in order to avoid one which would introduce systematic divergences from the observed data. The literature contains numerous expressions for determining efficiency, but to present even a few is beyond the scope of this document. A generally accepted and simple expression for efficiency determination is as follows:

$$\ln \epsilon = a_1 + a_2 \ln E \quad (I-1)$$

where: \ln is the natural log
 ϵ is the absolute FEP efficiency
 a_1 and a_2 are fit parameters
 E is the energy (keV) of the corresponding gamma line.

This expression is adequate for determining efficiency of gamma energies from 200 keV to 2000 keV. A typical efficiency curve for a germanium detector is given in Fig. 13.

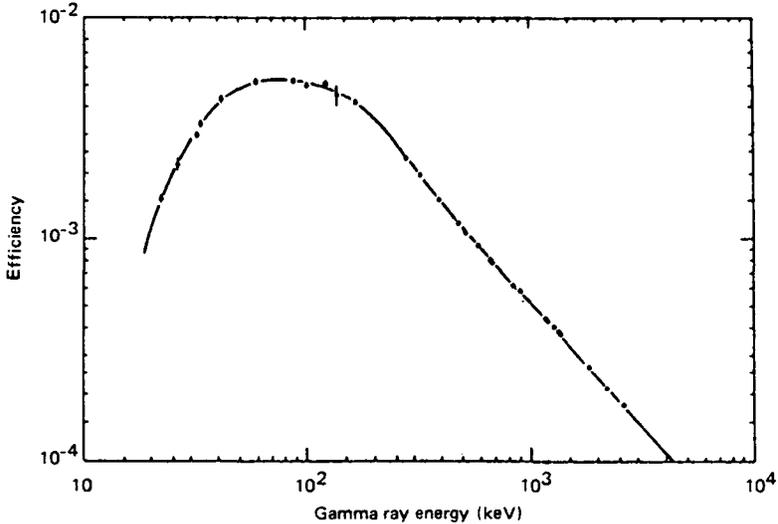


FIG. 13. Full energy peak efficiency as a function of gamma ray energy for a typical Ge(Li) detector.

If radionuclides with energies below 200 keV need to be quantified, more terms in the polynomial expression would improve the fit of the data in this energy region.

I-7. COUNTING CONSIDERATIONS

I-7.1. Counting geometry

Samples should be counted only in the types of container used to acquire the counting efficiencies, as stipulated in Section I-6.2.1. The density, volume and height of the sample in the container must be the same as that of the standards used for calibration. Any change of these factors will require additional calibrations to match the characteristics of the sample.

I-7.2. Background

The background of the system has a very significant influence on the detection limit and accuracy of the measurement of low levels of activity. The counting system must have a background as low as is attainable with a minimum of spectral lines originating from natural radionuclides which may be present in the system components and the surrounding environment, i.e. the walls, floor, etc., of the counting

TABLE VIII. TYPICAL BACKGROUND FROM NATURAL RADIONUCLIDES OBSERVED WITH A SHIELDED GERMANIUM DETECTOR

Radionuclide	E_{γ} (keV)	Count rate (counts/1000 s)
Pb-212	75.0	
Pb-214	74.8	26.9
Tl-208	75.0	
Th-234	92.9	10.3
Ra-226	186.2	13.5
Ac-228	209.3	4.9
Pb-212	238.6	7.3
Ac-228	270.2	6.2
Tl-208	278.0	1.7
Pb-214	295.2	3.7
Ac-228	328.0	1.1
Ac-228	338.3	16.4
Pb-214	351.9	16.7
Ac-228	463.0	4.9
Tl-208	511.0	} (double line)
β^+	511.2	
Tl-208	583.0	34.5
Bi-214	609.3	16.4
Bi-212	727.0	8.8
Ac-228	794.7	4.2
Tl-208	860.4	4.5
Ac-228	911.1	27.0
Bi-214	1120.3	4.3
Bi-214	1238.1	2.4
Bi-214	1377.7	1.0
K-40	1460.8	107.0
Tl-208	1592.5	(double escape)
Bi-212	1620.6	1.1
Ac-228	1630.4	1.1
Bi-214	1729.6	1.1
Bi-214	1764.5	6.4

Measuring time: 3×10^5 s (5000 min).

Detector: Ge(Li) — 100 cm³ vol.; 23% efficiency; 1.9 keV resolution.

Shield: 30 mm thick consisting of (from outside inward) lead, steel, aluminium and plexiglass. Inner dimensions — 500 mm × 500 mm × 500 mm.

Location: Counting room with walls made of barytes concrete and located in the basement of a building.

facility. Construction materials such as concrete, plaster, and paints which contain barytes (barium sulphate) will tend to cause elevated backgrounds due to natural activities. Other materials that contain large quantities of potassium (which has a natural radioisotope, ^{40}K) will also cause elevated backgrounds.

Background count rates for natural radionuclides measured on a shielded gamma spectrometer system are given in Table VIII. Table XV in Annex VII provides similar data for an unshielded system.

Interfering spectral lines of the radioactive daughters of ^{222}Rn (which is always present in varying concentrations) can be lessened considerably by purging the chamber of the detector shield with an aged gas such as argon, helium, breathing air, or with low-pressure compressed air that is first passed through a filter system composed of a drying agent and charcoal.

Background measurements should be taken as frequently as is practicable and for counting times as long as possible in order to obtain good counting statistics. A good practice is to record the background measurements on a control chart with statistically fixed limits. This provides a means both of checking the stability of the electronics of the system and of checking for contamination of the detector and/or shield. Should the background exceed the control limits, an immediate and thorough investigation should be made and appropriate steps taken to maintain a minimum background.

Great care should be taken to prevent any contamination of the detector, because the decontamination process is difficult, tedious, and time consuming. The detector should always be covered with a thin polyethylene film (foil) held in place over the detector by either Scotch tape or a rubber band. The polyethylene film can be easily replaced when contamination is suspected to be present. If the detector does become contaminated, the following cleaning procedure is recommended. Carefully wipe all external parts of the detector with tissue dampened with water; wipe with tissue dampened with 1M HCl acid; wipe with a 0.1% aqueous solution of tetrasodium EDTA; repeat the water cleaning step; and, finally, wipe with a tissue dampened with acetone. In cleaning a detector the wipes should be changed very frequently to avoid distributing a contamination. Extreme care must be taken that none of the solutions drip on to other parts of the detector system. It is always advisable to use EDTA to clean a detector contaminated with corrosion elements (activation products) such as the radioisotopes of zinc, manganese, iron and cobalt. The shield can be decontaminated by the same procedure as used for the detector. The background of the system should always be checked thoroughly after cleaning.

I-7.3. Detection limits

Detection limits is a term used to express the detection capability of a measurement system under certain conditions (see Refs [I.4,5]). An estimate for the lowest amount of activity of a specific gamma-emitting radionuclide that can be detected at

the time of measurement can be calculated from several different expressions. A generally accepted expression for the estimate of the detection limits, which is frequently referred to as the lower limit of detection (LLD) and which contains a preselected risk of 5% of concluding falsely that activity is present and a 95% degree of confidence for detecting the presence of activity, is as follows (see Ref. [I.6]):

$$LLD = \frac{4.66 S_b}{\epsilon P_\gamma} \quad (I-2)$$

where S_b is the estimated standard error of the net count rate;
 ϵ is the counting efficiency of the specific nuclide's energy; number ≤ 1 ;
 P_γ is the absolute transition probability by gamma decay through the selected energy as for ϵ ; number ≤ 1 .

The LLD of Eq. (I-2) provides a means of determining the operating capability of a gamma measuring system without the influence of a sample and is applicable on the assumption that the count rate in the energy area taken for the specific nuclide and the count rate in the region(s) taken for background are essentially equal.

When a sample is introduced into the gamma measurement(s), the term usually associated with detection limits is the minimum detectable concentration (MDC) which can be expressed by:

$$MDC = \frac{4.66 S_b}{\epsilon P_\gamma W} \quad (I-3)$$

where W is the mass of the sample (kg).

As can be seen in Eq. (I-3), the factors that tend to influence the detection limits are the counting efficiency, the quantity of sample (mass or volume), the counting time associated with S_b , and the background. To obtain low detection limits the efficiency should be high, the sample should be as large as practicable, the counting time should be as long as practicable, and the background should be as low as attainable. The efficiency is strongly influenced by the sample geometry and tends to decrease as the sample height (distance away from the detector) increases. Therefore, an optimum sample size and geometry may be used to obtain low detection limits. Doubling the counting time improves the detection limits by only a factor of $\sqrt{2}$, so, in order to lower the detection limits by a factor of two, the counting time would need to be four times as long. The background of a measurement system is usually kept as low as possible. However, the number and type of radionuclides in a gamma spectrum can influence the level of background in the Compton continuum region. Consequently, the detection limits for radionuclides with lower energies (i.e. energies in the Compton continuum region) will be higher. The

TABLE IX. AN EXAMPLE OF DETECTION LIMITS OF SOME RADIONUCLIDES IN VEGETABLE ASHES^a

Radionuclide	E_{γ} (keV) ^b	Detection limit (Bq/kg)
Nd-147	91.1	241
Co-57	122.1	62
Ce-144	133.5	514
Ce-141	145.4	115
Ba-140	162.9	936
	537.4	281
Cr-51	320.1	659
I-131	364.5	97
Sb-125	427.9	222
Be-7	477.6	654
La-140	487.0	147
	1596.5	76
Ru-103	497.1	74
Sb-124	602.7 ^c	70
	1691.0	91
Cs-134	604.7 ^d	75
	795.9 ^c	47
Ru-106	621.8	226
Ag-110m	657.8	74
	884.7	100
Cs-137	661.7	78
Zr-95	756.7	137
Nb-95	765.8	77
Co-58	810.8	69
Mn-54	834.8	79
Fe-59	1099.3	134
Zn-65	1115.6	157
Co-60	1173.2	91
	1332.5	78

Counting geometry: 100 mL volume in a cylindrical plastic dish placed directly on the surface of the detector.

Counting time: 9×10^4 s (1500 min).

Detector: Ge(Li) — 100 cm³ vol.; 23% efficiency; 1.9 keV resolution.

Shield: 30 mm thick consisting of (from outside inward) lead, steel, aluminium and plexiglass. Inner dimensions: 50 cm × 50 cm × 50 cm.

Location: Counting room with walls made of barytes concrete and located in the basement of a building.

TABLE IX. (cont.)

- ^a Determined by measurement of 15 g kale ash (about 1.5 kg fresh kale).
- ^b Gamma energies and transition probabilities and possible interferences were taken from Table XIV (Annex VII).
- ^c Possible interference from Cs-134.
- ^d Possible interference from Sb-124, Sb-125, Bi-214.
- ^e Possible interference from Ac-228, Pa-234.

concentration of potassium in samples has a direct influence on the detection limits for many radionuclides because of the Compton scattering caused by ⁴⁰K.

Table IX lists typical detection limits for several radionuclides measured in a sample of ashed kale. The detection limits presented can be achieved routinely but only with the specified spectrometer system and under the specified measuring conditions.

I-8. SPECTRAL EVALUATION

I-8.1. Computer evaluations

The computer-controlled evaluation of a complex gamma spectrum is of special importance (see Ref. [I.7]). Computer programs can be used to resolve overlapping peaks by fitting analytical curves to collected data. They can also be used to calculate activities in complicated spectra in which certain spectral lines can be attributed to several nuclides by comparing peak height ratios. Computers can also handle very large amounts of data very rapidly and accurately.

Computer programs used for automated spectral analyses can be developed by the user, adapted from published routines, or bought from commercial suppliers of software or vendors of nuclear instruments. It is recommended that whenever possible the software used for the computer system and gamma spectral analyses be purchased from the supplier of the pulse height analysis system in order to assure compatibility. Computer programs can vary in final output from simple energy identifications to full reports which include identification and quantification of all major nuclides present in the spectrum, the uncertainties associated with the determinations, and the detection limits for nuclides found and those not found in the spectrum.

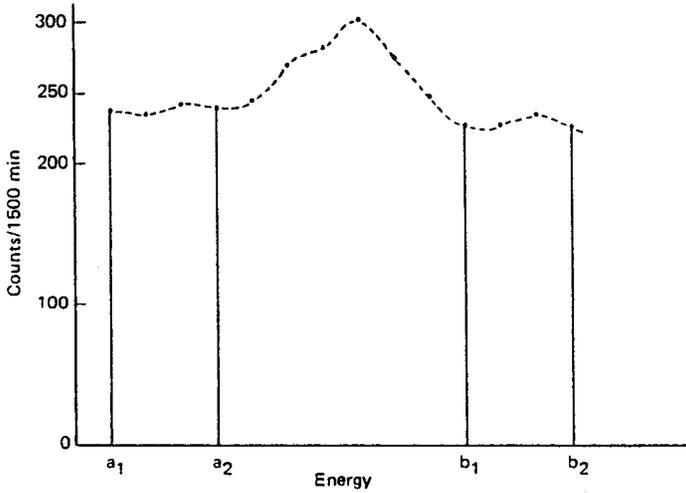


FIG. 14. Gamma ray spectrometry: typical peak of low level radioactivity.

The tasks usually performed by the computer programs are as follows:

- Automatic peak search,
- Evaluation of the peak position(s) in energy,
- Identification of radionuclides by use of a nuclide library,
- Calculation of the net peak area(s),
- Calculation of activity concentrations in selected units,
- Calculation of the detection limits for specific nuclides.

I-8.2. Manual calculations

Despite the advantages and attractive features of computerized systems, care must be taken to check the validity of data output, especially in the initial set-up (see Ref. [I.1]). Manual spectra evaluation should be well understood, and the computer data output should be checked from time to time to ensure the quality of determinations. A typical peak resulting from low level environmental radioactivity gamma measurement is given in Fig. 14. For manual calculations the following procedure may be used:

$$n_t = \sum_{i=a_2}^{b_1} n_i \quad (\text{I-4})$$

$$n_b = \left(\sum_{i=a_1}^{a_2-1} n_i + \sum_{i=b_1+1}^{b_2} n_i \right) \frac{(b_1 - a_2 + 1)}{(a_2 - a_1 + b_2 - b_1)} \quad (\text{I-5})$$

$$n = n_t - n_b \quad (\text{I-6})$$

where n = net counts
 n_b = background counts
 n_i = counts in channel i
 n_t = total counts
 a_1, a_2, b_1, b_2 are the numbers of the respective channels (energy)

Note: The regions a_1 to a_2 and b_1 to b_2 must be representative of the background under the peak. If nearby peaks interfere, these regions have to be taken farther from the peak (see Section 6.1.1 for additional references on manual techniques).

I-9. CALCULATIONS

I-9.1. Activity at measuring time

If in the gamma spectrum the peak of a radionuclide with the count rate R_n was detected, the mean activity per kilogram of wet mass at the beginning of the measurement can be calculated as follows:

$$R_n = R_T - R_b \quad (\text{I-7})$$

$$A_n = \frac{R_n m_f}{\epsilon m_\mu m_F P_\gamma} \quad (\text{I-8})$$

where A_n is the mean activity concentration of nuclide n (Bq/kg wet mass),
 R_n is the net count rate of nuclide n (counts/s),
 R_T is the gross count rate of nuclide n (counts/s),
 R_b is the background count rate for nuclide n (counts/s),
 m_F is the mass of wet sample used for measuring or ashing (kg),
 m_f is the mass of sample after ashing (kg),
 m_μ is the mass of ashed sample used for measurement (kg).

Note: When measuring a sample without pretreatment, m_f and m_μ take a value of 1.

I-9.2. Corrections for decay

As long as the counting time t is short compared to the half-life $T_{1/2}$ of a radionuclide ($t < 0.01T_{1/2}$), the disintegration rate may be considered constant. However, if the counting time t is greater than 1% of the half-life $T_{1/2}$ ($t > 0.01T_{1/2}$), the decrease in the disintegration rate during the measurement must be taken into consideration and a correction made in the determination. For example, this type of correction would be necessary in the determination of ^{131}I , ^{147}Nd , ^{140}Ba and ^{140}La with counting times of 1500 min or longer. Correction of the activity of a relatively short lived nuclide to the start of the measurement can be made by use of the following equations:

$$A_s = A_n f \quad (\text{I-9})$$

$$f = \lambda t / (1 - e^{-\lambda t}) \quad (\text{I-10})$$

$$\lambda = \ln 2 / T_{1/2} \quad (\text{I-11})$$

where A_s is the mean activity concentration of the specified nuclide at the start of the measurement (Bq/kg wet mass)
 A_n is the mean activity concentration of the measurement (Bq/kg wet mass)
 f is the decay factor over the counting time t
 t is the counting time (s)
 λ is the decay constant (s^{-1})
 $T_{1/2}$ is the half-life (s).

The activity of a specified nuclide can be corrected to the sampling time by the following decay equation:

$$A_{st} = A_s e^{\lambda t_1} \quad (\text{I-12})$$

where A_{st} is the mean activity concentration at the sampling time (Bq/kg wet mass)
 A_s is the mean activity concentration at the start of the measurement (Bq/kg wet mass)
 t_1 is the time between sampling and the start of the measurement(s).

Coincidence losses during counting due to cascade nuclear transitions (see Section I-6.2.3) have to be taken into account. The correction for coincidence summing is becoming more important with the improvement of efficiencies of the detectors. These corrections are very sensitive to counting geometry, individual radionuclides, gamma emission intensity and specific activity. It is recommended

that each user should study and determine these effects for his/her individual gamma spectrometry system (see Refs [I.3, 8]).

For radionuclides with cascade transitions, counting losses due to summing effects are to be expected. The simplest solution to this problem is to calibrate the spectrometer with a standard containing the same radioisotope. The measurements are then comparative and the fraction of the events lost due to coincidence summing is the same for both the standard and the test sample. If a suitable standard of the measured radionuclide is not available then the correction must be calculated. The methods for doing the calculations are too complex to be given here, but they can be found in Refs [I.9–12]. It should also be mentioned that coincidence summing can lead to an apparent increase in photopeak counts in some cases, but the correction is generally small.

If the count rates from the sample are very high then losses can also occur due to random summing of gamma rays originating from separate decaying nuclei. Random summation is usually insignificant for environmental and food samples because the count rates are generally very low.

I-9.3. Complete calculation

The complete calculations of the activity of a specified nuclide at the time of sampling with a time difference of t_1 between sampling and starting the measurement are made by means of the following equation:

$$A_{st} = \frac{R_n m_f \lambda t e^{\lambda t_1}}{\epsilon P_\gamma m_\mu m_F (1 - e^{-\lambda t})} \quad (I-13)$$

I-9.4. Standard deviation

The standard deviation (S_n) of the mean net count rate R_n can be calculated from the following expressions:

$$S_n^2 = S_T^2 + S_B^2 \quad (I-14)$$

$$S_T^2 = \frac{R_T}{t} \quad (I-15)$$

$$S_B^2 = \frac{R_B}{t} \quad (I-16)$$

where S_n is the standard deviation of net count rate R_n (counts/s)
 S_T is the standard deviation of gross count rate R_T (counts/s)
 S_B is the standard deviation of the background count rate R_B (counts/s).

TABLE X. EXAMPLE OF STATISTICAL COUNTING ERRORS FOR CAESIUM-137 AS A FUNCTION OF COUNTING TIME AND ACTIVITY CONCENTRATION^a

Counting time (min)	Activity measured (mBq/kg)	Statistical counting error (mBq/kg)	Relative statistical counting error (%)
500	180	90.8	50.4
1500	180	52.4	29.1
3000	180	37.0	20.6
500	360	93.4	25.9
1500	360	53.9	15.0
3000	360	38.1	10.6
500	900	100.3	11.1
1500	900	57.9	6.4
3000	900	40.9	4.5
500	1800	110.9	6.2
1500	1800	64.0	3.6
3000	1800	45.3	2.5

^a The gamma spectrometric system is described in Table VIII.

I-10. ACCURACY

The total error in a determination of radionuclides (exclusive of sampling error) depends on the error in the determination of the nuclide specific counting efficiency (3–5%) and the statistical counting errors (1–5%). Therefore, the concentrations of activities of the various radionuclides in the standards used for calibration of the gamma spectrometer system should be known to at least 3%. The counting time should be optimized to accommodate the operating conditions and should be for a period long enough to minimize the statistical counting error.

An increase of the counting time affects the statistical counting error of a radioactivity measurement. The relative counting error will vary inversely as the square root of the counting time; for example, by doubling the counting time the relative error will be reduced by $\sqrt{2}$.

The statistical counting error decreases in direct proportion to increasing sample activity if the counting time is constant when the background B is much larger than the sample counts n , e.g. $B \gg n$. This is typical for low level counting. The relative counting error is then given by the approximation \sqrt{B}/n . Doubling the activity will thus result in a 50% reduction of the relative counting error. In cases where the background is much smaller than the sample counts ($B \ll n$), the estimate of the relative error is given by the approximation \sqrt{n}/n . This is typical for high activity samples, e.g. after accidental releases. Doubling the activity will thus result only in a 30% reduction of the relative counting error.

An example of statistical counting errors as a function of counting time and activity of samples containing ^{137}Cs is given in Table X.

An average error of 10% for the determination of gamma emitters in foods and environmental samples should be assumed, but if the activity levels are very low then larger errors can be expected.

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Annex II

METHODS FOR RADIOCHEMICAL ANALYSIS OF STRONTIUM

Strontium-90 is normally determined by separating it from the matrix material and then separating and counting its daughter product yttrium-90 from the strontium-90 after it has grown in at or near radioactive equilibrium. These methods are deemed to give results with the best attainable accuracy for a wide range of materials.

II-1. DETERMINATION OF RADIOSTRONTIUM IN VARIOUS MATERIALS BY NITRATE PRECIPITATION (Ref. [II.1])

A. *Outline of method*

The ashed material is dissolved in nitric acid in the presence of strontium and barium carriers. The nitric acid concentration is then increased to precipitate all the strontium and barium (and part of the calcium) as nitrates. After further nitric acid separations, barium chromate and iron hydroxide scavengers are carried out. The subsequent treatment depends somewhat on the circumstances but the following is normal practice.

Yttrium carrier is added to the purified strontium solution and, after a delay of about 14 days for the growth of ^{90}Y , the yttrium is separated, mounted and counted. The storage period for the growth of ^{90}Y can be reduced if sufficient ^{90}Sr is known to be present, and the appropriate growth factor applied. For samples of very low activity, as well as for measurement of ^{89}Sr , strontium is precipitated from the solution remaining after the removal of yttrium and mounted for counting. In many cases the determination of the natural inactive strontium content of the material is required so that the strontium chemical yield can be corrected.

In the case of milk, direct application of the nitric acid separation to a solution of the ash usually gives low strontium yields. The calcium, strontium and barium are therefore concentrated by an initial phosphate precipitation. The mixed phosphates are then dissolved in an acid and the general procedure continued from that point.

In the case of cereals, and vegetation generally, the ash is very variable in composition and contains numerous elements other than calcium, some (e.g. silicon) in considerable amounts. Consequently, a more vigorous treatment that uses a mixture of hydrofluoric and perchloric acids is necessary to decompose and dissolve the ash. After heating to remove the hydrofluoric and most of the perchloric acid, the residue is dissolved in dilute acid and the alkaline earths precipitated as phosphates. The general procedure is then taken up at this point.

The analysis of soil presents special difficulties. For this reason a somewhat modified method is used (see II-2).

B. Reagents

Fuming nitric acid, 95-96%, w/wt
Nitric acid, 6M
Hydrochloric acid, 6M
Acetic acid, 6M
Oxalic acid solution, 8%
Ammonium hydroxide, concentrated (17M) and 6M
Ammonium hydroxide, carbonate-free
Ammonium carbonate
Ammonium acetate solution, 25%
Sodium chromate solution, 30%
Hydrogen peroxide, 30%
Acetone
Strontium carrier solution, 5 mg of strontium per mL, as nitrate, standardized
Yttrium carrier solution, 10 mg of yttrium per mL, as nitrate, standardized
Barium carrier solution, 10 mg of barium per mL, as nitrate
Ferric iron carrier solution, 5 mg of iron per mL, as nitrate

C. Preparation of samples for analysis

In general, the fresh, dry and ash weights of all samples are recorded. Samples are dried in ovens at a temperature of 80-100°C, and ashed, in all but a few special cases, at 600-800°C in electric muffle furnaces.

D. General procedure

The initial treatment of the ashed sample depends upon the nature of the material being analysed. These preparatory steps are given in detail later (see Section II-1.1(c)). After this treatment, a nitric acid solution is usually obtained which contains essentially all the radiostrontium. The subsequent procedure is as follows:

1. Make the solution in the centrifuge tube alkaline with ammonium hydroxide, add solid ammonium carbonate and heat in a boiling water bath to coagulate the carbonate precipitate. Centrifuge and reject the supernate.

Note: The carbonate precipitation serves to concentrate the strontium and calcium since more water is used in transferring the material to the centrifuge tube than is required in the next separation.

2. Add 10 mL of water to the residue in the centrifuge tube and then 22.5 mL of fuming nitric acid cautiously to precipitate the strontium. Cool in running water with stirring for 30 minutes, centrifuge and reject the supernate.
3. Repeat Step 2. Dissolve the residue in 10–15 mL of water, add 1 mL of barium carrier solution and 1 drop of methyl red indicator. Neutralize excess acid with 6M ammonium hydroxide and add 1 mL of 6M acetic acid and 2 mL of 25% ammonium acetate. Dilute the solution to 30 mL and heat in a boiling water bath. Add 1 mL of 30% sodium chromate and continue heating for 5 minutes. Centrifuge and filter the supernate through a filter paper into a centrifuge tube. Reject the residue.

Note: The solution is diluted to reduce the loss of strontium that occurs in a more concentrated solution.

4. Make the solution alkaline with concentrated ammonium hydroxide, add solid ammonium carbonate and heat in a boiling water bath to coagulate the carbonate precipitate. Centrifuge and reject the supernate.
5. Dissolve the residue in dilute nitric acid, add 1 drop of 30% hydrogen peroxide and then 1 mL of iron carrier solution. Heat and stir to remove carbon dioxide. Dilute to 15–20 mL and make alkaline with carbonate-free ammonium hydroxide, heating for 2–3 minutes to complete precipitation. Centrifuge and transfer the supernate to another 40-mL centrifuge tube. Reject the precipitate.

Note: The carbonate precipitate contains occluded chromate which is removed by reducing the chromium to the trivalent state and precipitating with the iron as hydroxide.

6. Precipitate the strontium as carbonate from the supernate by adding ammonium carbonate and heating in a water bath. Cool and filter on a tared, fine-porosity filter paper in a demountable filter unit (“filter stick”).

Note: Two filter papers are placed together in the filter unit, the lower one being subsequently used as a counterpoise when weighing the strontium carbonate.

Wash with water and methanol successively, transferring the precipitate quantitatively to the paper. Separate the paper from its counterpoise, allow it to come to equilibrium with the atmosphere for 2 hours and weigh the strontium carbonate. Calculate the strontium yield and mount the paper and source on an aluminium planchette using an adhesive, for example a 3% solution of polyvinyl acetate in methanol. Count the strontium source after storing for at least 14 days.

7. Pipette 1 mL of yttrium carrier solution into a 40 mL centrifuge tube and add 10 mL of acetone. Detach the paper with the source from the planchette and

place in the centrifuge tube. Add sufficient 6M hydrochloric acid to dissolve the carbonate precipitate and dilute with water to 20 mL.

Note: Application of gentle heat to the tray will soften the adhesive sufficiently to enable the paper complete with source to be removed.

8. Warm carefully, remove the paper and any lumps of the adhesive and make the solution alkaline with carbonate-free ammonium hydroxide. Centrifuge and transfer the supernate to a second tube. Redissolve the precipitate in 6M nitric acid, dilute to 10–15 mL and reprecipitate with ammonium hydroxide. Centrifuge and add the supernate to that from the previous precipitation. Note the time of the first precipitation.

Note: The combined supernates should be retained in case further counting of the strontium and/or yttrium is required.

9. Heat the tube in a boiling water bath, redissolve the precipitate in a minimum of 6M nitric acid and add 20 mL of 8% oxalic acid solution. Heat in a water bath for 10–15 minutes to produce a granular precipitate. Cool and filter through a fine-porosity filter paper in a demountable filter. Wash three times with methanol and mount on a planchette with adhesive. Commence counting immediately, noting the time.
10. When counting of the yttrium source is completed, including any check on residual activity, remove the paper with the source from the planchette (see Note to Step 7). Transfer to a tared crucible, burn off the paper, ignite at 800–900°C, and weigh as Y_2O_3 .
11. Calculate the yttrium recovery. Calculate the radiostrontium content as described in Sections II-3 and II-4.

II-1.1. Determination of radiostrontium in milk or cheese

A. Additional reagents required

Nitric acid, concentrated (16M)

Syrupy phosphoric acid, 15M

Wash solution: Add 3 mL of syrupy phosphoric acid and 20 mL of concentrated (17M) ammonium hydroxide to 2 litres of water

B. Preparation of samples for analysis

Dried milk powder is ashed directly in porcelain or silica basins. Liquid milk may be poured into stainless steel trays and heated to 100°C in a well ventilated oven until well charred. The carbonaceous material is transferred to basins and ashing

completed in a muffle furnace. The original volume of milk and the weight of ash are recorded.

C. Procedure

1. Weigh out 20 g of the milk or cheese ash into a 600-mL beaker. Add 10 mL of strontium carrier solution, 1 mL of barium carrier solution, 200 mL of distilled water and 40 mL of concentrated nitric acid. Warm and stir until a clear solution is obtained. Add 1 mL of syrupy (15M) phosphoric acid and neutralize with concentrated ammonium hydroxide until ammonia is in excess. Transfer to two 200-mL centrifuge bottles and centrifuge. Remove the supernate and add 100 mL of wash solution to the contents of each bottle. Stir up the precipitate to obtain a slurry and centrifuge. Reject the washings.
2. Dissolve the precipitate by adding 20 mL of fuming nitric acid to each bottle and stirring. Combine the two solutions in a measuring cylinder and measure the volume (2V mL). Return half the solution (V mL) to each centrifuge bottle and add to each (2.5V – 40) mL of fuming nitric acid. Cool in water with stirring for 30 minutes. Centrifuge and reject the supernate.
3. Transfer the contents of one bottle to the other with 40 mL of distilled water. Add 90 mL of fuming nitric acid and cool with stirring for 30 minutes. Centrifuge and reject the supernate. Transfer the residue to a 40-mL centrifuge tube with 20–30 mL of water.
4. Continue as described under General Procedure above (Section II-1 (D)).

II-1.2. Determination of radiostrontium in cereal, vegetables, herbage and other foodstuffs

A. Additional reagents required

Hydrochloric acid, 11M

Hydrofluoric acid, 40%

Perchloric acid, 60%

Syrupy phosphoric acid, 15M

Wash solution: Add 3 mL of syrupy phosphoric acid and 20 mL of concentrated ammonium hydroxide to 2 litres of water

B. Preparation of samples for analysis

Samples are weighed out from dry homogenized material. A preliminary reduction in bulk may be effected by igniting the dry material with a bunsen burner

on a stainless steel tray. The charred material is transferred to porcelain or silica basins and ashing completed in a muffle furnace. The ash is lightly brushed through a sieve (0.125 mm mesh) where any soil particles are retained and can be removed. Particular care is required in the ashing of cereals (including rice) and potatoes as the yield of ash from these materials is low and material with high potassium content may attack porcelain or similar crucibles. A lower ashing temperature (about 400°C) is recommended.

C. Procedure

1. Weigh the required amount of ash (see Note below) into a 10-cm platinum dish. Add 10 mL of strontium carrier solution, 1 mL of barium carrier solution and 10 mL of water and then cautiously add about 40 mL of 40% hydrofluoric acid. Add 30 mL of 60% perchloric acid and evaporate almost to dryness. Cool, add a further 30 mL of perchloric acid and evaporate as before. Dissolve the residue in water with the addition of 5 mL of 11M hydrochloric acid and transfer to a 600-mL beaker; adjust the volume to 250–300 mL and boil. Cool and filter through a coarse-porosity filter paper into a second 600-mL beaker.

Note: Suggested weights of ash required for analysis are as follows:

Herbage 10–20 g
Whole grain 10 g
Flour 20 g
Bran 50 g
Vegetables 10–20 g
Other 10–15 g

2. Add 5 mL of syrupy phosphoric acid to the filtrate and then stir in 17M ammonium hydroxide until an excess of ammonia is detected.
3. Transfer the contents of the beaker to a 200-mL centrifuge bottle, centrifuging and rejecting the supernate, until all the precipitate has been collected in the bottle. Wash the precipitate by stirring with 100 mL of wash solution, centrifuge and reject the supernate.
4. Dissolve the precipitate by the addition of 20 mL of fuming nitric acid and transfer to a measuring cylinder. Note the volume (V mL) and return the solution to the centrifuge bottle. Add $(2.5V - 40)$ mL of fuming nitric acid and cool in running water for 30 minutes, stirring occasionally.
5. Centrifuge and reject the supernate. Dissolve the residue in 40 mL of distilled water, add 90 mL of fuming nitric acid and cool with stirring for 30 minutes. Centrifuge and reject the supernate. Transfer the residue to a 40-mL centrifuge tube with 20–30 mL of water.

6. Centrifuge and transfer the supernate to a second 40-mL centrifuge tube. Wash the precipitate with 10 mL of distilled water, centrifuge and add the supernate to that in the second tube. Reject the residue and continue as described under General Procedure (Section II-1 (D)).

II-1.3. Determination of radiostrontium in water (Ref. [II.2])

As the ^{90}Sr activity concentrations in water appear to be comparatively low, a preconcentration of the sample (up to 100 litres) is recommended using a rotation evaporator and dissolving the residue in nitric acid.

A. Additional reagents required

Nitric acid, concentrated (16M), 8M and 4M
Hydrochloric acid, 1M
Perchloric acid, 60%
Sodium hydroxide, 5M
Sodium carbonate
Calcium carrier solution: 200 mg of calcium per mL, as chloride

B. Procedure

1. Thoroughly shake the sample and filter it under reduced pressure through a double thickness of ashless rapid filter paper into a polyethylene container. Measure the volume of the filtrate.
2. Wash the original sample container with 8M nitric acid. Transfer the washings to a 100-mL silica basin and evaporate to dryness. Place the filter paper containing the residue in the basin and ignite at 500–600°C until free from carbonaceous matter.
3. Add 20 mL of 60% perchloric acid and 20 mL of 16M nitric acid and evaporate the solution to dryness. Cool and leach the residue twice with 20-mL portions of 1M hydrochloric acid, warming on a water bath to assist in dissolving the residue. Combine the leachings and filter through an ashless rapid filter paper. Discard the residue.
4. Combine the filtrate from leaching the insoluble material with the initial sample filtrate and add 10 mL of strontium carrier solution and 1 mL of barium carrier solution. Add 2 mL of calcium carrier solution for every litre of the original sample and transfer the solution to a beaker.

5. Heat the sample almost to boiling and add 5M sodium hydroxide until the solution is alkaline. Add solid sodium carbonate, with stirring, until precipitation of the mixed strontium and calcium carbonate is complete.
6. Boil for about 20 minutes to coagulate the precipitate, cool and allow to stand for at least 2 hours.
7. Decant or syphon off the bulk of the supernate, dissolve the carbonate precipitate in the minimum amount of 4M nitric acid and, if necessary, dilute to at least 40 mL with water. Measure the volume of the solution (V mL) and transfer to a centrifuge bottle.
8. Cautiously add (2.25V - 40) mL of fuming nitric acid, cool in running water with stirring for 30 minutes. Centrifuge and discard the supernate.
9. Dissolve the precipitate in 40 mL of water and cautiously add 90 mL of fuming nitric acid. Cool in running water with stirring for 30 minutes. Centrifuge and discard the supernate.
10. Dissolve the precipitate in a little water and transfer to a centrifuge tube, washing the centrifuge bottle with 20 to 30 mL of water and adding the washings to the tube. Continue as described in General Procedure above (Section II-1 (D)).

II-2. MODIFICATION OF METHOD II-1 AS REQUIRED FOR ANALYSIS OF STRONTIUM-90 IN SOILS

A. *Outline of method*

Because of the low specific activity of soils, large sample weights are usually necessary. In the method given here, the radiostrontium is extracted from the soil with 6M hydrochloric acid. By this means, up to 500 g of soil can be treated without making the subsequent separations too unwieldy.

The soil is extracted in the presence of strontium carrier and filtered. Calcium and strontium are then concentrated by precipitation as oxalates. With clay soils, kieselgur is added as a filter aid and, because of the presence of appreciable amounts of aluminium in the extract, the preliminary concentration of calcium and strontium is best effected by carbonate precipitation from sodium hydroxide solution. The carbonates are then dissolved in acid and the alkaline earths precipitated as oxalates.

The oxalates are ignited, the residue dissolved in dilute acid, any residual iron and aluminium removed as hydroxides, and the calcium and strontium precipitated as carbonates and weighed.

The strontium is then separated from calcium by successive precipitations as nitrate and purified by two barium chromate scavenges and an iron hydroxide scavenge. Yttrium carrier is added and, after storage for a known length of time, preferably at least 14 days, separated, mounted and counted.

Because of the presence of significant amounts of natural activity in soils, two barium chromate scavenges are included. Even so, the decay and any residual activity in the yttrium source must be carefully measured to check its radiochemical purity.

If it is necessary to attempt a determination of ^{89}Sr , a suitable weight of strontium carbonate can be counted, but any growth or decay should be carefully checked before calculating the ^{89}Sr content.

The natural stable strontium present in soil may introduce errors in the gravimetric determination of the chemical yield of strontium. To overcome this difficulty, ^{85}Sr is frequently used in chemical yield determinations, and this procedure is recommended.

B. Reagents

Nitric acid, fuming, 95-96% w/wt

Nitric acid, concentrated (16M), 6M and 3.5M

Hydrochloric acid, concentrated (11M)

Acetic acid, 6M

Ammonium hydroxide, carbonate-free, concentrated (17M) and 6M

Ammonium acetate solution, 50%, 25%

Sodium chromate solution, 30%

Oxalic acid solution, 8%

Hydrogen peroxide, 30%

Yttrium carrier solution, 10 mg of yttrium (as nitrate) per mL, standardized

Barium carrier solution, 10 mg of barium (as nitrate) per mL

Ferric iron carrier solution, 5 mg of iron (as nitrate) per mL

Strontium carrier solution, 40 mg of strontium (as nitrate) per mL, standardized

C. Preparation of samples for analysis

After removal of the root mat and herbage, the soil samples are spread on trays to dry. The dry soil is weighed, crushed and sieved through a 4-mm mesh. Any stones are collected, weighed and discarded. The dry soil is treated directly without ashing.

D. Procedure

1. Weigh 500 g of dry soil into a 1-litre beaker. Add 250 mL of distilled water and 10 mL of strontium carrier solution. Stir and add 250 mL of concentrated hydrochloric acid (see Note below). Allow to stand with occasional stirring for at least 8 hours. Transfer the contents of the beaker to a large Buchner funnel provided with a fine-porosity filter paper. Filter and wash the soil with about 500 mL of distilled water. Return the soil to the original beaker and treat with a further 250 mL of water and 250 mL of concentrated hydrochloric acid. After completion of the second period of extraction, filter and wash as before. Reject the residue. Combine the two filtrates in a 4-litre beaker.

Note: Caution should be used during the addition of hydrochloric acid to soils of high carbonate content.

2. Add 100 g of oxalic acid and 25 mL of 50% ammonium acetate solution. Warm to dissolve the oxalic acid and neutralize by the addition of ammonium hydroxide to pH4.0 (see Note below). Stand the solution in a warm place for at least 4 hours. Remove the supernate by suction and filtration. If the precipitate is discoloured owing to the presence of iron, dissolve it in 6M nitric acid, dilute to about 1 litre, add 50 g of oxalic acid and reprecipitate at pH4.0.

Note: If a brown precipitate is obtained before pH4 is reached, there is insufficient oxalate present to convert all the iron to a complex. Add a further 50 g of oxalic acid and again adjust the pH. The pH is conveniently determined with pH papers.

3. Transfer the oxalate precipitate to a silica dish, dry in an oven and ignite in a well ventilated area in a muffle furnace at 700–800°C for 30 minutes. Remove from the furnace and allow to cool completely. Add 50–60 mL of water and dissolve the residue by addition of concentrated nitric acid. Transfer the solution to a 400-mL beaker and boil to remove carbon dioxide. Dilute to about 200 mL. Add 6M ammonium hydroxide to the hot solution to precipitate any iron and aluminium. Filter through a 15-cm coarse-porosity filter paper and wash the precipitate with hot water.
4. Add solid ammonium carbonate to the filtrate until precipitation of calcium and strontium is complete. Filter the carbonates on a filter paper of suitable size, dry and weigh (see Note below). This weight gives the calcium carbonate content of the soil plus the weight of strontium carbonate carrier recovered.

Note: If the calcium content is not required, the carbonates need not be dried and weighed. If the calcium content of the soil is low and the precipitate is not much greater than that expected from the strontium carrier alone, the drying and weighing may be omitted and the calcium determined later (see Step 5, Note (b)).

5. Dissolve the mixed carbonates in the minimum of 3.5M nitric acid. Measure 58-mL portions of this solution into 200-mL centrifuge bottles. To the contents of each bottle add 120 mL of fuming nitric acid with stirring. The residual volume of solution is treated with a proportionate amount of fuming nitric acid (see Note (a)). Cool in running water for 30 minutes, stirring occasionally. Centrifuge and remove the supernatant acid (see Note (b)).

Note (a): In 58 mL of 3.5M nitric acid dissolve approximately 10 g of calcium carbonate.

Note (b): If the weighing in Step 4 has been omitted, this supernate and the subsequent one must be retained for calcium determination if required.

6. To the contents of each centrifuge bottle add 40 mL of water, dissolve the residue and then add 90 mL of fuming nitric acid. Cool in water for 30 minutes, centrifuge and remove the supernatant acid.
7. Repeat Step 6. Transfer the residues to a 40-mL centrifuge tube with 10–20 mL of water. Add 1 mL of barium carrier solution and 1 drop of methyl red indicator. Neutralize excess acid with 6M ammonium hydroxide and add 1 mL of 6M acetic acid and 2 mL of 25% ammonium acetate. Dilute the solution to 30 mL and heat in a boiling water bath. Add 2 mL of 30% sodium chromate solution and continue heating for 5 minutes. Centrifuge and filter through a 7-cm ashless rapid filter paper into a second centrifuge tube.
8. Heat the second tube in the boiling water bath, add 1 mL of barium carrier solution, stir immediately, and heat for 5 minutes. Centrifuge and filter as before into another tube. Reject the two residues. Make the solution alkaline with concentrated ammonium hydroxide, add solid ammonium carbonate and heat to coagulate the carbonate precipitate. Centrifuge and reject the supernate.
9. Dissolve the residue in dilute nitric acid, add 1 drop of 30% hydrogen peroxide and 1 mL of iron carrier solution. Heat and stir to remove carbon dioxide. Dilute to 15–20 mL and make alkaline with carbonate-free ammonium hydroxide. Heat and stir to complete precipitation. Centrifuge and transfer the supernate to another 40-mL centrifuge tube. Reject the precipitate.
10. Acidify the solution with 6M nitric acid and add 1.00 mL of yttrium carrier solution. Cover the tube and store for at least 14 days.
11. Make the solution alkaline with carbonate-free ammonium hydroxide and heat in a boiling water bath to coagulate the precipitate. Centrifuge and transfer the supernate to a second tube. Redissolve the precipitate in 6M nitric acid, dilute to 10–25 mL and reprecipitate with ammonium hydroxide. Centrifuge and add the supernate to that from the previous precipitation. Note the time of the first precipitation.

12. Heat the tube in a boiling water bath, redissolve the precipitate in the minimum of 6M nitric acid and add 20 mL of 8% oxalic acid solution. Heat in a water bath for 10–15 minutes to produce a granular precipitate. Cool and filter through a fine-porosity filter paper in a demountable filter unit, wash three times with methanol and mount on a planchette with adhesive. Commence counting immediately.
13. Precipitate the strontium as carbonate from the combined supernates from Step 11 by adding ammonium carbonate and heating in a water bath. Cool and filter the solution in a tared sintered-glass crucible (porosity 4). Wash with water and methanol, and dry in an oven at 110°C. Weigh and calculate the strontium yield.
14. When the counting of the yttrium source is completed, remove the paper with the source from the planchette (see Note below). Transfer to a tared crucible, burn off the paper, ignite at 800–900°C and weigh as Y_2O_3 .

Note: Application of gentle heat will soften the adhesive sufficiently to enable the paper complete with source to be removed from the planchette. Alternatively, the adhesive can be dissolved with methanol.

15. Calculate the yttrium recovery. Calculate the ^{90}Sr content of the soil as described in Section II-3.

E. Modification for soils of high aluminium content

Additional reagents: Sodium hydroxide solution, 1M
Hydrochloric acid, 5M

Clay soils are difficult to filter without some filter aid. Before filtering (see Step 1 in Section II-2 (D) above) mix the soil with a suitable filter aid (e.g. Hyflo Supercel — flocculated cellulose — or kieselgur), which has been extracted previously with 5M hydrochloric acid. Combine the filtrates and neutralize with 10M sodium hydroxide solution. Dissolve 50 g of sodium hydroxide and 50 g of sodium carbonate in water and add to the solution. Allow to stand with occasional stirring for an hour and remove the supernate by suction and by filtration on a Buchner funnel through a hardened fast-filtering paper of great wet strength. Dissolve the precipitate in dilute hydrochloric acid and continue from Step 2 of Procedure in Section II-2 (D) above.

F. Determination of calcium

For soils of high calcium content the weight of the mixed carbonates (see Step 4 in Section II-2 (D) above) is corrected for the weight of strontium recovered (Step 13) and the calcium content calculated.

To determine the calcium content of soils low in calcium, evaporate the nitric acid supernates (see Steps 5 and 6 in Section II-2 (D) above) to low bulk and dilute to 40–50 mL. Add 50 mL of 8% oxalic acid solution and proceed as described in Section II-6 below.

II-3. CALCULATION OF STRONTIUM-90 CONTENT

The ^{90}Sr content is determined whenever possible by separating the ^{90}Y daughter and following its decay. If the total activity of the aged strontium source is low (2 counts/min or less above the background and blank), the counting of separated yttrium is not practicable. In such cases an estimate of the ^{90}Sr can be obtained by measuring the strontium source at intervals of six to eight weeks and assuming that any decay is due to the presence of ^{89}Sr . This method should not be used if at all possible as the almost unavoidable presence of small amounts of long lived activity other than that due to strontium leads to erroneous results. The errors can sometimes be reduced if the strontium source is counted three times at intervals of six to eight weeks and the appropriate corrections made.

II-3.1. Calculation based on the yttrium count

Count the yttrium source for three periods spaced at approximately three-day intervals. Each count should be at least 3000. If the total count does not reach this figure in two days, the count is recorded and the source left in the counter, readings being taken at intervals of two days.

Calculate the mean count rate for each counting period, add the correction for paralysis losses, and subtract the background of the counter. Evaluate the ^{90}Y decay factor, f , for each counting period from the expression:

$$f = \exp \frac{0.69315 t}{2.69} \quad (\text{II-1})$$

where t is the time in days from the yttrium separation to half-way through the particular counting period.

Calculate the count rate at the time of separation from the three count rates. A series of values, f_1C_1 , f_2C_2 , f_3C_3 , will be obtained, where

f_1 , f_2 , f_3 are the decay factors for counting periods 1, 2, 3;
 C_1 , C_2 , C_3 are the mean count rates for counting periods 1, 2, 3.

The three fC values should agree, showing that the ^{90}Y is radiochemically pure. The presence of a long lived activity gives values of fC which increase steadily. In this case, subtract a correction:

$$\frac{f_3 C_3 - f_1 C_1}{f_3 - f_1} \quad (\text{II-2})$$

from each mean count and calculate the corrected count rate at the time of yttrium separation. The ^{90}Sr activity (Bq) is calculated from the expression:

$$^{90}\text{Sr} = \frac{C \times 10^6}{60 \times S \times Y \times E} \quad (\text{II-3})$$

where: C is the corrected count per minute
 S is the strontium yield (%)
 Y is the yttrium yield (%)
 E is the counter efficiency for ^{90}Y (%).

II-3.2. Calculation based on the strontium count

Count the source without an absorber, after at least 14 days storage, for sufficiently long to obtain a count of at least 3000. If the amount of activity is judged to be too low to permit the counting of the separated ^{90}Y , put the source aside and recount in the same counter after six to eight weeks.

Correct the count rates for the background of the counter (paralysis losses will normally be negligible). If the two corrected count rates, C_1 and C_2 , are equal, no detectable ^{89}Sr is present and the ^{90}Sr activity (Bq) is calculated from the expression:

$$^{90}\text{Sr} = \frac{C_1 \times 10^4}{60 \times S \times E} \quad (\text{II-4})$$

where: S is the strontium yield (%)
 E is the counter efficiency for ^{90}Sr and ^{90}Y (%) (i.e. counts/min of $^{90}\text{Sr} + ^{90}\text{Y}$ per 100 disintegrations/min of ^{90}Sr).

If C_1 is greater than C_2 , calculate the ^{89}Sr decay factor, f , for the time interval between the two counts (t days) from the expression:

$$f = \exp \frac{0.69315 t}{50.5} \quad (\text{II-5})$$

Then the ^{90}Sr activity (Bq) is calculated from the expression:

$$^{90}\text{Sr} = \frac{10^4}{60 \times S \times E} \frac{fC_2 - C_1}{f - 1} \quad (\text{II-6})$$

II-4. CALCULATION OF STRONTIUM-89 CONTENT

The ^{89}Sr content, which is normally calculated back to the date of collection of the sample, may be determined by one of the following methods, depending on the level of the activity and the $^{89}\text{Sr}/^{90}\text{Sr}$ ratio.

In the first method, the strontium source is mounted and counted immediately after the yttrium separation through a 100 mg/cm^2 absorber. The method is applicable only to samples in which the $^{89}\text{Sr}/^{90}\text{Sr}$ ratio exceeds 10 and there is sufficient activity to obtain a count of 3000 through the absorber in 24 hours.

For samples of lower activity or those in which the ratio $^{89}\text{Sr}/^{90}\text{Sr}$ is less than 10, the strontium is stored for at least 14 days for the ^{90}Y to grow to equilibrium and then counted without an absorber. The contribution made by ^{90}Sr and ^{90}Y to the observed count can be calculated from the ^{90}Sr content determined by counting the yttrium daughter; the count due to ^{89}Sr is then obtained by subtraction. In some laboratories, the measurement is repeated at intervals of three weeks to follow the decay of ^{89}Sr . This method is suitable for samples of low activity or with low $^{89}\text{Sr}/^{90}\text{Sr}$ ratios. If this ratio is less than one, the ^{89}Sr content may be an overestimate because of the presence of other radioactive nuclides.

II-4.1. Calculation based on the strontium count through a 100 mg/cm^2 absorber

Count the strontium source through a 100 mg/cm^2 aluminium absorber as soon as possible after the yttrium has been separated. The count should not be less than 3000 and should be finished within 24 hours.

Correct the count rate for paralysis losses and background; then the ^{89}Sr activity (Bq) is calculated from the expression:

$${}^{89}\text{Sr} = \frac{C \times 10^4}{60 \times S \times e} - \frac{A \times E_1}{e} \quad (\text{II-7})$$

where: C is the corrected count rate per minute
 S is the strontium yield (%)
 A is the ${}^{90}\text{Sr}$ content of sample in Bq
 E_1 is the counter efficiency for ${}^{90}\text{Sr}$ through the absorber (%)
 e is the counter efficiency for ${}^{89}\text{Sr}$ through the absorber (%).

This calculation is based on the assumption that the amount of ${}^{90}\text{Y}$ present is negligible. If sufficient ${}^{90}\text{Y}$ grows in to affect the count rate, calculate the decay factor f from the expression:

$$f = \exp \frac{0.69315 t}{2.69} \quad (\text{II-8})$$

where t is the time in days from the yttrium separation to half-way through the counting period. Calculate the ${}^{89}\text{Sr}$ activity in Bq from the expression:

$${}^{89}\text{Sr} = \frac{C \times 10^4}{60 \times S \times e} - \left(\frac{A}{e} E_1 + \left(\frac{f - 1}{f} \right) E_2 \right) \quad (\text{II-9})$$

where E_2 is the counter efficiency for ${}^{90}\text{Y}$ through the absorber (%).

II-4.2. Calculation based on the count of the aged strontium source

Count the source, after at least 14 days storage without an absorber to obtain a count of at least 3000. The ${}^{89}\text{Sr}$ activity in Bq is then calculated as follows:

$${}^{89}\text{Sr} = \frac{C \times 10^4}{60 \times S \times e} - \frac{A \times E_3}{e} \quad (\text{II-10})$$

where: C is the corrected count per minute
 S is the strontium yield (%)
 A is the ${}^{90}\text{Sr}$ content of sample in Bq
 E_3 is the counter efficiency for ${}^{90}\text{Sr}$ and ${}^{90}\text{Y}$ (%)
 e is the counter efficiency for ${}^{89}\text{Sr}$ (%).

II-4.3. Calculation based on repeated counting of the strontium source

Count as described in Section II-3.2; then the ^{89}Sr activity in Bq is calculated as follows:

$$^{89}\text{Sr} = \frac{10^4 \times f (C_1 - C_2)}{60 \times S \times e (f - 1)} \quad (\text{II-11})$$

where: C_1 is the corrected original count per minute at time t_1
 C_2 is the corrected second count per minute at time t_2
 f is the ^{89}Sr decay factor for period t_1-t_2
 S is the strontium chemical yield (%)
 e is the counter efficiency for ^{89}Sr (%).

II-5. CALIBRATION OF COUNTERS

II.5.1. For strontium-89

Add known aliquots of standardized ^{89}Sr solution to centrifuge tubes containing 10 mg, 20 mg, 30 mg and 40 mg of strontium carrier. Precipitate the strontium as carbonate, mount and weigh as described 7 in Step 6 of General Procedure in Section II-1 (D) above. Count the source with and without a 100 mg/cm² absorber between the source and counter tube.

Correct the count rates for paralysis losses, background and chemical yield, allowing for any strontium in the standard solution.

Calculate the counter efficiency, E , where

$$E = \frac{\text{Corrected count rate} \times 100}{\text{Disintegration rate of radionuclide in the aliquot}} \quad (\text{II-12})$$

The appropriate correction must be applied for any decay of the radionuclide in the standard solution after the date of standardization and for any ^{90}Sr in the standard solution (this may exceed 4% in fresh material).

II-5.2. For strontium-90 through 100 mg/cm² absorber

To centrifuge tubes containing 10 mg, 20 mg, 30 mg and 40 mg of strontium and 10 mg of yttrium carrier add known aliquots of standardized ^{90}Sr solution.

Separate the yttrium from one tube as hydroxide as described in Step 8 of General Procedure in Section II-1 (D) above, and proceed immediately to the precipitation, mounting and weighing as described under Step 6 of General Procedure. Count the source at once through a 100 mg/cm^2 absorber.

Record the count rate at hourly intervals for three hours and extrapolate back to the time of yttrium separation to obtain the count rate for ^{90}Sr alone. Process and count the strontium sources from the other tubes in turn.

Correct the count rates and calculate the efficiency as in Section II-5.1 above.

II-5.3. For strontium-90 and yttrium-90

Add known aliquots of standardized ^{90}Sr solution and treat as described in Step 6 of General Procedure in Section II-1 (D) above to centrifuge tubes containing 10 mg, 20 mg, 30 mg and 40 mg of strontium carrier. Count the sources after storing for at least 18 days.

Correct the count rates and calculate the efficiency as in Section II-5.1 above.

II-5.4. For yttrium-90

To a centrifuge tube containing 10 mg of strontium and yttrium carriers add a known aliquot of standardized ^{90}Sr solution. Store for a minimum of 18 days and then treat as described in Steps 8–10 of General Procedure in Section II-1 (D) above. Repeat the calibration but count through a 100 mg/cm^2 absorber. Calculate the count rate of the yttrium source to time of separation (see Section II-3.1), correct for the yttrium yield and calculate the efficiency as in Section II-5.1.

Prepare calibration curves for strontium sources by plotting counter efficiency on a log scale against source weight on a linear scale. These curves can be used to obtain the counter efficiencies at intermediate source weights.

Calibrations should be checked routinely from time to time.

II-6. DETERMINATION OF CALCIUM

Normally, the fresh, dry and ash weights of all samples will have been recorded.

II-6.1. Milk ash

Weigh accurately 0.5 g of ash into a 250-mL beaker and dissolve in about 40 mL of water and 5 mL of 11M hydrochloric acid. Add 40 mL of 8% oxalic acid solution and heat to about 80°C . Neutralize the solution by the dropwise addition of 6M ammonium hydroxide to pH4. Stand the beaker on a boiling water bath for

3–4 hours and filter on a tared sintered-glass crucible (porosity 4). Wash the precipitate three times with distilled water and twice with methanol, and dry in an oven at 110–115°C for 30 minutes. Weigh the precipitate as calcium oxalate monohydrate and calculate the calcium content (factor 0.2743). Retain the precipitate for the determination of natural strontium, if required (see Ref. [II.3]).

II-6.2. Vegetation and vegetable ash

Weigh about 1 g of ash accurately into a 10-cm platinum dish. Add 20 mL of distilled water and then cautiously about 20 mL of 40% hydrofluoric acid and 20 mL of 60% perchloric acid. Warm on a hot plate and evaporate almost to dryness. Cool, add a further 20 mL of perchloric acid and evaporate as before. Dissolve the residue in 40–50 mL of water, transfer to a 400-mL beaker and add 40 mL of 8% oxalic acid solution. Proceed as described in Section II-6.1 above.

II-7. USE OF STRONTIUM-85 TRACER TO DETERMINE CHEMICAL YIELD

Radiometric determination of the recovery of strontium, using ^{85}Sr as tracer, is advantageous for three reasons:

- The method avoids the possibility of error from contamination of the strontium precipitate with calcium or other impurities;
- The method avoids the possibility of an erroneously high estimate of the recovery of strontium due to the presence of appreciable amounts of naturally occurring stable strontium in the material analysed;
- When the final determination is to be made by counting ^{90}Y , the separation of calcium and strontium does not have to be complete.

In this method, a known amount of ^{85}Sr (~10 Bq) is added along with the stable strontium carrier at the start of the analysis, and the amount remaining in the final strontium precipitate is subsequently determined by gamma counting.

II-7.1. Recommended procedure

1. The purified strontium carrier, after recovery at the end of the analytical procedure, is gamma counted using a sodium iodide crystal detector. The strontium may be in either solid or solution form, but must be in a well defined geometry with respect to the detector.
2. Immediately afterwards, an aliquot of the standard ^{85}Sr solution equal in volume to that added to the strontium carrier at the start of the analysis is treated to bring it into the same form and volume as the sample in Step 1 above. This standard is gamma counted in exactly the same geometry.

If the count rate in Step 1 is S, the count rate in Step 2 is P, and the background is B, then:

$$\text{Chemical yield (\%)} = \frac{S - B}{P - B} \times 100 \quad (\text{II-13})$$

Note: In cases where use of the ^{85}Sr method is inconvenient, it is recommended that the stable strontium be determined by means of a flame photometer. This is often more convenient and accurate than the gravimetric method, although it lacks the additional advantage of the ^{85}Sr tracer method with respect to the elimination of the error that may arise from the presence of naturally occurring strontium in the sample material.

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Annex III

METHOD FOR RADIOCHEMICAL ANALYSIS OF TRITIUM¹

III-1. INTRODUCTION

Tritium present in the environment is of both natural and man-made origin. As a result of nuclear weapons testing in the atmosphere, emissions from nuclear engineering installations, and the application and processing of radioisotopes, significant amounts of tritium are released to the environment.

III-2. SCOPE AND FIELD OF APPLICATION

The method is specified for determination of tritiated water ($[^3\text{H}]\text{H}_2\text{O}$) activity concentration in water by liquid scintillation counting. The method is applicable to all types of water including sea water with tritium activity concentrations of up to 10^6 Bq/m³ when using 20-mL counting vials.

Notes:

- (a) Below tritium activity concentrations of about 2×10^4 Bq/m³, a prior enrichment step and/or measurement of larger sample volumes can significantly improve the accuracy of determination and lower the limit of detection (see Section III-12.1 below for calculation of the minimum detectable activity concentration). However, enrichment means an additional step in the analytical procedure and thus a potential source of error, quite apart from the greater analytical effort involved. A scatter of about 1–3% occurs as a result of the inherent variability of the enrichment cells. Direct scintillation counting with commonly used liquid scintillation counters (see Ref. [III.1]) is therefore preferable for tritium activity concentrations higher than approximately 2×10^4 Bq/m³.
- (b) Tritium activity concentrations higher than 10^6 Bq/m³ may be determined after appropriate dilution with distilled water of proven low tritium content. An alternative method for determination of these higher activities involves increasing the tritium activity concentrations of the internal standard solution (see Section III-6 below).

¹ Taken after revision from INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Provisional Draft International Standard, ISO/DIS 9698, Status November 1987, Geneva.

- (c) The method is not applicable to the analysis of organically bound tritium, which requires for its determination an oxidative digestion. Organically bound tritium and tritium gas are assumed to be in equilibrium with tritiated water.

III-3. PRINCIPLE

Tritium decays to helium by emitting beta radiation with a maximum energy of 18.6 keV. Its half-life is 12.35 years (see Ref. [III.2]). The water sample is distilled to remove non-volatile quenching materials and non-volatile radioactive materials. Prior to distillation, sodium carbonate (Na_2CO_3) and sodium thio-sulphate ($\text{Na}_2\text{S}_2\text{O}_3$) are added to the sample. The majority of the constituents of the sample that might interfere remain in the residue together with any radioactive iodide and bicarbonate that might be present. If the tritium content of non-aqueous biological samples is required, the sample can be converted to water by oxidation (Ref. [III.3]). The distillation is carried out to dryness to ensure complete transfer of the tritium to the distillate. An aliquot of the distillate is mixed with a scintillation solution in a counting vial. The mixture is cooled and counted in a liquid scintillation spectrometer (coincidence type).

In this sample (usually an emulsion) the kinetic energy of the tritium beta particles is partly converted into light photons. When certain boundary conditions are satisfied (e.g. simultaneous detection by two or more photomultiplier tubes connected in coincidence, discrimination of pulses by preset measurement channels), these photons are counted as pulses.

Standard tritium and background samples are prepared and counted identically to minimize errors produced by aging of the scintillation medium or instrument drift. The counting rate is a measure of the tritium activity concentration. The sensitivity (counting time 100 minutes) is generally of the order of 20–200 Bq/L.

III-4. REAGENTS

1,4-dioxane naphthalene, solid
2,5-diphenyloxazole (PPO)
1,4-di [2-(phenyloxazoly)] benzene (POPOP)
Sodium carbonate, anhydrous (Na_2CO_3)
Sodium thiosulphate, anhydrous ($\text{Na}_2\text{S}_2\text{O}_3$).

Note: For analysis use only reagents of recognized analytical grade.

III-5. BLANK WATER

Obtain water with a tritium activity concentration as low as possible, e.g. (deep) groundwater. Distill the water in accordance with Section III-10.1 below. Keep the distillate in a well stoppered borosilicate glass bottle in the dark at a constant temperature. Determine (see Note (b)) the tritium activity concentration of this water and note the date ($t = 0$) of this determination $c_b(t=0)$, in Bq/m^3 .

Notes:

- (a) It is advisable to keep an adequate quantity of blank water in stock and to prepare small subsamples from it for immediate use as required. Contamination with tritium (e.g. from water vapour in the air and from tritium sources such as luminous watches and gas chromatographs) or other radioactive species should be avoided.
- (b) The tritium activity concentration in the blank water can be determined by enrichment, followed by, for example, liquid scintillation counting. Blank water of known low tritium activity concentration may be obtained upon request from the IAEA (see Annex X). When the stock of blank water is sufficiently large, e.g. 10–20 litres, and well sealed it will be stable for years, although it is advisable to redetermine the tritium activity concentration at predetermined intervals, e.g. every year.
- (c) It is preferable to use blank water with a tritium activity concentration $\leq 500 \text{ Bq/m}^3$. If the tritium activity concentration of the blank water is $\geq 500 \text{ Bq/m}^3$, a correction must be made using the calculation procedure given in Section III-11.1.2 below. The tritium activity concentration $c_b(t)$ at the time t at which the samples are measured (see Section III-10.3 below), corrected for radioactive decay, is given by:

$$c_b(t) = c_b(t=0) \times e^{-\lambda t} \quad (\text{Bq/m}^3) \quad (\text{III-1})$$

where:

- $c_b(t)$ is the tritium activity concentration of the blank water at the time t at which the samples are measured, in becquerels per cubic metre;
- $c_b(t=0)$ is the tritium activity concentration of the blank water at the time of its preparation, in becquerels per cubic metre;
- λ is the decay constant, in reciprocal years (0.0561);
- t is the time between preparation of the blank water and the measurement of the samples, in years.

For a blank water with a tritium activity concentration of up to about 500 Bq/m^3 correction for radioactive decay is not necessary.

III-6. INTERNAL STANDARD SOLUTION

In a location remote from the area where the tritium analyses are to be done, weigh out into a weighed 100-mL volumetric flask the requisite quantity of a concentrated tritium ($[^3\text{H}]\text{H}_2\text{O}$) standard solution (activity concentration 10^{10} – 10^{11} Bq/m³, total inaccuracy $\leq 1\%$) so that the tritium activity concentration will be about 170 Bq/mL after filling to the mark with blank water. Make up to the mark and mix. Calculate the tritium activity concentration of the resulting internal standard solution $c(t=0)$, in Bq/m³. Note the date at which the standard was made up ($t=0$).

Notes:

- (a) The tritium activity concentration of the internal standard solution at time t at which the samples are measured, corrected for radioactive decay, is given by:

$$c_s(t) = c_s(t=0) \times e^{-\lambda t} \text{ Bq/m}^3 \quad (\text{III-2})$$

where:

$c_s(t)$ is the tritium activity concentration of the internal standard solution at the time t at which the samples are measured, in becquerels per cubic metre;

$c_s(t=0)$ is the tritium activity concentration of the internal standard solution at the time of its preparation, in becquerels per cubic metre;

λ is the decay constant, in reciprocal years (0.0561);

t is the time between preparation of the internal standard solution and the measurement of the samples, in years.

- (b) Aqueous soluble tritium standard capsules may be used instead of a concentrated tritium ($[^3\text{H}]\text{H}_2\text{O}$) standard solution.

III-7. SCINTILLATION SOLUTION

Commonly used are scintillation solutions with one or more emulsifiers in which relatively large quantities of water samples can be incorporated (usually in an emulsion or gel). Commercially available cocktails are found to be the most satisfactory in practice. Example of a scintillation solution: mix thoroughly 4 g of PPO, 0.05 g of POPOP and 120 g of naphthalene in 1 litre of 1,4-dioxane (see Section III-4 above).

Store in the dark and — particularly just before use (see Section III-10.2 below) — avoid exposure to direct sunlight so as to prevent interfering luminescence.

III-8. APPARATUS

This method relates to the widely used liquid scintillation counters with vials that hold about 20 mL. When other vials are used with appropriate counters, the described method must be modified.

- A. Liquid scintillation counter, preferably with an automatic sample presentation unit. Operation at constant temperature is recommended. Follow the instructions given by the manufacturer.
- B. Distillation apparatus, dried before use, consisting of:
 - 500-mL round-bottom flask (a larger flask may be used for preparing the blank water (see Section III-5 above))
 - Splash head
 - Vigreux distillation column, length 40 cm
 - Condenser
 - Adaptor, bent type.
- C. Pipette, suitable for the accurate transfer of 100 μL of internal standard solution, with a total inaccuracy $\geq 1\%$.
- D. Counting vials, made from polyethylene or equivalent material, that will hold at least 20 mL, will fit the counting chamber of the liquid scintillation counter, and distortion of which is acceptably small after being filled. Polyethylene counting vials are preferred to glass as they generally give a lower background counting rate.

Notes:

- (a) To prevent interfering luminescence, the counting vials should be kept in the dark and should not be exposed to direct sunlight, particularly just before use.
- (b) Toluene-based scintillation solutions may distort polyethylene and should therefore not be used in combination with polyethylene counting vials.
- E. Borosilicate glass or polyethylene bottles of about 100 mL volume.
- F. Usual laboratory apparatus and accessories.

III-9. SAMPLING AND SAMPLES (Ref. [III-4])

Obtain samples in accordance with ISO 5667. Take a laboratory sample of about 250 mL for the sample preparation (see Section III-10.1 below).

III-10. ANALYTICAL PROCEDURE

III-10.1. Preparation of samples

Place the laboratory sample in the distillation apparatus. Add about 250 mg of sodium thiosulphate to convert iodine into iodide and about 0.5 g of sodium carbonate to make the sample alkaline, and finally some carborundum beads. Assemble the equipment. Distil, discard the first 50–75 mL of distillate, then collect about 100 mL of the middle fraction in a bottle. Discard the residue in the flask.

Note: With this procedure there is no significant isotopic fractionation in this distillation.

III-10.2. Filling the counting vials

For each water sample, preferably in dimmed lighting, fill three counting vials by the addition of a volume V_1 (in mL, see Note (d) below) of scintillation solution; then add a volume $V_2 = 20 - V_1$ (in mL) of distillate (this mixture will hereafter be referred to as scintillation emulsion). Add by pipette 100 μL of internal standard solution into one of the counting vials. Mark the lids of the counting vials; for example, with the designation 1a, 1*, and 1b (for sample 1); 2a, 2* and 2b (for sample 2); etc.

In the same way, fill the appropriate number, as required by the counting procedure, of background counting vials with a volume V_1 (in mL) of scintillation solution followed by a volume $V_2 = 20 - V_1$ (in mL) of blank water. The total inaccuracy of each addition should be $\geq 1\%$. Mark the lids of these counting vials, for example, with the designation B_1 , B_2 , B_3 , etc. Shake the counting vials thoroughly and uniformly, e.g. by a shaking machine.

Notes:

- (a) The above operations should take place in dimmed lighting (preferably from an incandescent source or red light) in view of the possible interference by luminescence in some batches of counting vials.
- (b) For routine determinations of similar distilled samples the internal standard method described above may be replaced by a simplified procedure or by the use of an external standard, the applicability of which should be verified.
- (c) The use of an internal standard is recommended when polyethylene counting vials are used. When using an external standard in polyethylene counting vials interference may arise because the counting rate of the external standard changes as a function of time on account of the loss of components of the scintillation solution by diffusion into the wall of the counting vials. The effects are considerably smaller at lower temperatures (4–10°C) than at higher temperatures (e.g. 20–25°C).

- (d) Under optimal counting conditions many liquid scintillation solutions can hold up to about 40% water; in this case $V_1 = 12$ ml.

III-10.3. Counting procedure

After shaking, clean the counting vials with a damp cloth that will leave no deposit in order to remove, for example, any electrostatic charge. Hereafter, avoid any contact with the light-transmitting parts of the counting vials.

Place the counting vials in a fixed sequence in the liquid scintillation counter: background, sample 1, sample 1 with internal standard added, sample 1, background, sample 2, etc. (B_1 , 1a, 1*, 1b, B_2 , 2a, 2*, 2b, B_3 , 3a, 3*, 3b, B_4 , 4a, 4*, 4b, etc.).

Count the vials for a preset period of time using one or more measurement channels or, for the vials with internal standard, until a preset count is obtained.

Notes:

- (a) A counting time of 100 min per vial is usually sufficient. It is preferable to count the vial series during repeated short counting times rather than during one long counting time; for example, instead of one 100-min count, count five times for 20 min, for which purpose an automatic sample presentation unit is necessary. This provides better control of stability of the samples and reduces the possibility of erroneous counts passing undetected. Low tritium activity concentrations may require a longer counting period, depending on the desired counting accuracy.
- (b) Before counting, it is advisable to equilibrate the counting vials in the liquid scintillation counter for light and temperature adaptation, e.g. overnight, thus reducing the interfering luminescence during counting.
- (c) For samples containing relatively high tritium content a preset count may be used. In this case, Eq. (III-5) is not applicable (see Section III-11.3, Note (b)).

III-11. REPORTING RESULTS: CALCULATION METHOD

III-11.1. Counting efficiency

Calculate the counting efficiency from the equation:

$$\epsilon = \frac{R_s^* - \bar{R}_s}{D} \quad \text{(III-3)}$$

where:

- ϵ is the counting efficiency, (≤ 1), representing the number of counts per second per becquerel;
- R_s^* is the counting rate of the sample with the added standard solution, in counts per second;
- \bar{R}_s is the mean counting rate of the duplicate samples without internal standard solution, in counts per second;
- D is the activity of the added internal standard solution at the time the sample is measured, in becquerels; calculate D from the equation: $D = V \times c_s(t)$, where $c_s(t)$ is defined in Section III-6 above, in Bq/m^3 , and $V = 10^{-7} m^3$, as follows from Section III-10.2 above.

III-11.2. Tritium activity concentration of the sample

Calculate the tritium activity concentration of the sample from the equation:

$$c = \left(\frac{\bar{R}_s - \bar{R}_0}{\epsilon V_2} + c_b(t) \right) e^{\lambda \Delta t} \quad (Bq/m^3) \quad (III-4)$$

where:

- c is the tritium activity concentration, at the time of sampling in becquerels per cubic metre;
- \bar{R}_s is the mean counting rate of the duplicate samples without internal standard, in counts per second;
- \bar{R}_0 is the mean counting rate of both adjacent duplicate blank water samples, in counts per second;
- ϵ is the counting efficiency;
- V_2 is the volume of sample of blank water in the counting vial, in cubic metres ($1 \text{ mL} = 10^{-6} m^3$);
- λ is the decay constant, in reciprocal years ($= 0.0561$);
- Δt is the time elapsed from sampling to counting, in years;
- $c_b(t)$ is the tritium activity concentration of the blank water, in becquerels per cubic metre, at the time the sample is measured.

Notes:

- (a) When using blank water with a low tritium activity concentration compared to the tritium activity concentration of the sample, there is no need to correct the result for the decay of the tritium activity concentration of the blank water (see also Section III-5 above, Note (c)).
- (b) If $\Delta t \leq 0.5$ years the last term of the equation can be deleted.

III-11.3. Inaccuracy due to the statistical nature of radioactive decay and background radiation

Calculate the standard deviation of c (see Section III-11.2 above) due to the statistical nature of radioactive decay and background radiation from the equation:

$$s_c = \left\{ \frac{\sqrt{(\bar{R}_0 + \bar{R}_s)/t}}{\epsilon V_2} \right\} e^{\lambda \Delta t} \quad (\text{Bq/m}^3) \quad (\text{III-5})$$

where:

s_c is the standard deviation of c (see Section III-11.2 above), in becquerels per cubic metre;

\bar{R}_0 , \bar{R}_s , ϵ , V , λ and Δt are as defined in Section III-11.2;

t is the summed counting time of the blank counting vials (equal to the summed counting time of the sample counting vials), in seconds.

Notes:

- (a) In samples with a low tritium activity concentration the statistical nature of radioactive decay and background radiation is the predominant source of inaccuracy, usually designated "statistical counting error". At tritium activity concentrations higher than about 10^5 Bq/m³, other sources of error become noticeable (see Refs [III.1,5]).
- (b) When a preset count is used, calculate the standard deviation of c due to the statistical nature of radioactive decay and background radiation from the equation:

$$s_c = \left\{ \frac{\sqrt{(\bar{R}_0/t_0) + (\bar{R}_s)/t_s}}{\epsilon V_2} \right\} e^{\lambda \Delta t} \quad (\text{Bq/m}^3) \quad (\text{III-6})$$

where:

s_c , \bar{R}_0 , \bar{R}_s , ϵ , V_2 , λ and Δt are as defined in Section III-11.2 above;

t_0 is the summed counting time of the blank counting vials, in seconds;

t_s is the summed counting time of the sample counting vials, in seconds.

III-12. OPTIMIZING COUNTING CONDITIONS

III-12.1. Minimum detectable activity concentration

The minimum detectable activity concentration can be calculated from the following equation:

$$c_{\min} = \frac{k}{\epsilon V_2} \sqrt{(\bar{R}_0/t_s) (1 + t_s/t_0)} e^{\lambda \Delta t} \quad (\text{Bq/m}^3) \quad (\text{III-7})$$

where:

c_{\min} is the minimum detectable activity concentration, in becquerels per cubic metre;

k is the coefficient of confidence; usually 3;

\bar{R}_0 , t_0 , t_s , ϵ , V_2 , λ and Δt are as defined in Sections III-11.2 and III-11.3.

c_{\min} incorporates the most relevant factors that should be taken into account when measuring low activity concentrations. It excludes factors such as long term stability of the electronic apparatus and liquid scintillation emulsion, errors due to cross-contamination, etc., that are difficult to express in an equation.

\bar{R}_0 and ϵ are among other things dependent on the composition of the scintillation emulsion, i.e. also on V_2 , and on the adjustment of the measurement channels.

It is generally advantageous to incorporate as much distillate into the counting vial as possible. The volumes of distillate (V_2 , in mL) and scintillation solution ($V_1 = 20 - V_2$, in mL) should be optimized in relation to \bar{R}_0 and ϵ in such a way that the value of $\epsilon^2 \times V_2^2/\bar{R}_0$ is maximized. The optimal value of V_2 is not identical for all scintillation solutions and should be determined for each cocktail individually (see Ref. [III.6]). An important prerequisite is stability of the scintillation emulsion during the measurement, i.e. the counting rates of samples and blanks should remain constant, apart from the statistical counting error.

It should be emphasized that it is more important to keep the blank counting rate as constant as possible than to keep it as low as possible, particularly for samples in which the activity concentration approaches c_{\min} . A high stable background raises c_{\min} , but a low and unstable background results in an unreliable c_{\min} due to scatter.

III-12.2. Optimal adjustment of the measuring channel

For liquid scintillation counters with selectable thresholds, select the lower threshold of the measurement channel so that the 2-photon pulses lie well above it. For a given scintillation emulsion, adjust the upper threshold so that e^2/\bar{R}_0 takes a maximum value. This is the optimal setting for measurement of samples with low activity concentrations (measurement channel A).

III-13. QUALITY CONTROL

III-13.1. Interference by luminescence

Serious interference of tritium determinations can occur as a result of luminescence. It is advisable to use a liquid scintillation counter capable of tracing these

single-photon events. In the absence of such a provision one can use the method described by Bransome and Grower ([III.7], p. 342) in which tritium is measured in parallel in another measurement channel B with the same lower threshold as measurement channel A, whereas the upper threshold is adjusted so that the tritium counting efficiency is about two-thirds of the tritium counting efficiency of channel A. In the absence of interfering luminescence, e.g. chemiluminescence, phosphorescence, triboluminescence, static electricity, calculation according to Section III-11.2 above should yield the same absolute tritium activity concentration in a sample for both measuring channels A and B, when using appropriate efficiencies for each channel.

In the presence of excessive luminescences the calculation according to Section III-11.2 above will give an apparently higher activity for channel B than for channel A due to random coincident monophoton events (which the apparatus cannot distinguish from the tritium double photon pulses; these pulses lie near the lower threshold and are consequently registered by channels A and B with the same counting efficiency). The occurrence of such a discrepancy points to interference due to luminescences.

III-13.2. Equipment stability

Once the measurement channels A and B have been adjusted, it is advisable to check that the setting is maintained by measuring in each sequence two hermetically sealed unquenched vials, one containing tritium standard solution and the other containing blank water. Drift of the apparatus from its initial setting will then easily be detected. For control purposes the use of control charts is advisable in this context (see Ref. [III.7]).

III-14. ANALYSIS REPORT

The test report shall include the following information:

- (a) A reference to the method used;
- (b) All information necessary for complete identification of the sample (traceability);
- (c) The tritium concentration and the corresponding standard deviation: $c \pm s_c$, in becquerels per cubic metre.

Note: For monitoring purposes, to avoid violating the statistical soundness of data series even negative tritium contents may be reported (see Ref. [III.8]);

- (d) Any noteworthy features observed during analysis of the sample;
- (e) Details of any operations not included in the methods, or regarded as optional, together with any circumstance that may have affected the results.

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Annex IV

METHODS FOR RADIOCHEMICAL ANALYSIS OF PLUTONIUM, AMERICIUM AND CURIUM

IV-1. DETERMINATION OF PLUTONIUM, AMERICIUM AND CURIUM IN BIOLOGICAL SAMPLES, SEDIMENT, SOIL, WATER AND AIR FILTERS¹

A. *Outline of method*

After the sample material has been properly prepared and isotopic tracers added, plutonium is separated from americium and curium by anion exchange. Americium and curium are precipitated with calcium oxalate and extracted into DDCP (dibutyl-N, N-diethyl carbamyl phosphonate). Americium and curium are separated from rare earths by anion exchange from mineral acids-methanol media. Curium follows americium in the procedure and is determined simultaneously. After electrodeposition on to stainless steel discs, the elements are counted by alpha spectrometry.

This procedure has been applied to biological, soil and sediment samples up to 200 grams dry weight, air filters, and water samples up to 1800 litres.

B. *Apparatus*

Stainless steel discs (electropolished)
Electroplating apparatus with cells and power supply
Ion exchange columns
Alpha spectrometry system with solid state detectors or gridded ionization chamber
Hot plates
Glassware, beakers, centrifuging tubes, Teflon beakers, etc.

C. *Reagents*

Hydrochloric acid; conc., 10M
Nitric acid; conc., 2M, 8M, 12M
Sulphuric acid; conc., 1% (vol./vol.), 20% (vol./vol.)

¹ Provided by HOLM, E., BALLESTRA, S., International Laboratory of Marine Radioactivity, Monaco.

Oxalic acid; solid
 Perchloric acid; conc.
 Hydrogen fluoride; conc.
 Sodium nitrite; solid
 Ammonia; conc., 1% (vol./vol.)
 Ammonium nitrate; solid
 Ammonium carbonate; saturated solution
 Fe^{3+} ; 100 mg per mL. Dissolve 47.6 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 100 mL H_2O , slightly acidified
 NaOH; saturated solution in water
 Ammonium oxalate solution; 0.5% wt/vol. in H_2O
 Hydrogen peroxide, 30%
 Ion exchange resin; BIO-RAD AG 1X8, AG 50WX8 and AG 1X4 (100-200 mesh)
 9M HCl-0.1M NH_4I ; mix 1 mL of 1M NH_4I with 9 mL 10M HCl
 Calcium; 100 mg per mL. Dissolve 58.93 g of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ in 100 mL H_2O
 DDCP (dibutyl-N, N-diethyl carbamyl phosphonate)
 1M HNO_3 -93% CH_3OH ; to 72.5 mL of conc. HNO_3 (13.8M) add CH_3OH to 1000 mL
 0.1M HCl-0.5M NH_4SCN -80% CH_3OH ; mix 100 mL of 1M hydrochloric acid and 100 mL of 5M ammonium thiocyanate with 800 mL of pure methanol
 1.5M HCl-86% CH_3OH ; mix 125 mL of conc. (12M) HCl with 860 mL CH_3OH ; add H_2O to 1000 mL
 0.3M sodium sulphate; dissolve 4.26 g Na_2SO_4 in 100 mL of H_2O
 Thymol blue; 0.4% wt/vol. in H_2O
 Acetone.

IV-1.1. Determination of plutonium, americium and curium in biological samples

A. Preparation of sample

1. Weigh out an expected suitable amount of ground dry sample, 1-200 g, into a porcelain crucible. Add a known amount of radiochemical yield determinants, ^{242}Pu (or ^{236}Pu), ^{243}Am and/or ^{244}Cm (about 20 mBq of each).
2. Put the sample into an electric furnace, raise the temperature gradually up to 550°C and ash overnight. If traces of carbon remain, add, after cooling, a few grams of NH_4NO_3 and ash again at 600°C for 15 minutes. Repeat until no carbon remains.
3. Continue with Step A.10, Section IV-1.4 below.

IV-1.2. Determination of plutonium, americium and curium in water samples

A. Preparation of sample

1. Acidify the sample upon collection to pH1 with conc. HCl. Prior to analysis add a suitable amount of yield determinants as described in Step A.1, Section IV-1.1 above and mix for two to four hours.
 - (a) For smaller fresh water samples (1–5 litres): Evaporate on a hotplate in a glass beaker. In evaporating the sample, care should be taken not to boil it dry as this may result in low yields. Wet ash with conc. HNO_3 at the end of evaporation. Transfer the sample to a 100–200-mL beaker with 8M HNO_3 . Add about 100 mg NaNO_2 and heat gently. Proceed with ion exchange procedure I, Section IV-2, below.
 - (b) For larger (> 5 L) fresh water samples: Add 10 mg Fe^{3+} per litre. Precipitate hydroxides by adding ammonia to pH10 during stirring. Collect the precipitate. Acidify the supernate again, repeat the precipitation procedure. Combine the two precipitates. Centrifuge and discard the supernate. Continue with Step A.9, Section IV-1.4 below.
 - (c) For sea water samples: Add 1 mg Fe^{3+} per litre and add saturated NaOH solution during stirring until pH10. Collect the precipitate and dissolve it in conc. HCl. Precipitate hydroxides by adding ammonia until pH5 and then complete the precipitation by adding $(\text{NH}_4)_2\text{CO}_3$ till pH9. Collect the precipitate. Centrifuge and discard the supernate. Continue with Step A.9, Section IV-1.4 below.

IV-1.3. Determination of plutonium, americium and curium on glass fibre filters

A. Preparation of sample

1. Cut the filter (diameter = 135 mm) into small pieces, add yield determinants as in Step A.1, Section IV-1.1 above, and ash at 550°C in a porcelain crucible overnight. If traces of carbon remain, add a few grams of NH_4NO_3 , mix and ash for one hour.
2. Transfer the filter to a Teflon crucible with 10 mL conc. HNO_3 .
3. Add 2 mL conc. HClO_4 and 20 mL conc. HF. Evaporate to *almost* dryness.
4. Add 2 mL conc. HNO_3 and 2 mL conc. HClO_4 and evaporate to *almost* dryness.
5. Repeat Step 4.

6. Dissolve the sample in 10 mL 2M HNO₃. Transfer the sample to a 100-mL centrifuge tube with 10 mL 2M HNO₃.
7. Add 20 mg Fe³⁺ and water to 70 mL. Precipitate hydroxides with ammonia at pH10. Centrifuge and discard the supernatant solution.
8. Wash the precipitate with 1% ammonia. Centrifuge and discard the supernatant solution.
9. Continue with step A.9, Section IV-1.4 below.

IV-1.4. Determination of plutonium, americium and curium in sediment and soil samples

A. Preparation of sample

1. Weigh out an expected suitable amount of ground dry sample, 1–200 g into a Pyrex glass beaker. Add yield determinants as in Step A.1, Section IV-1.1 above.
2. If the sample material contains noticeable organic matter, such as roots, stems, etc., place the sample in an electric furnace and raise the temperature gradually up to 550°C and ash overnight. If traces of carbon remain, add, after cooling, a few grams of NH₄NO₃ and ash again at 600°C for 15 minutes. Repeat until no carbon remains. If the sample does not contain organic matter, continue to the next step.
3. *Cautiously* add 8M HNO₃ and leach the sample during heating and occasional manual stirring (or preferably magnetic stirring) for four hours using a total volume of 50 mL 8M HNO₃ per 20 g sample and about 2 mL of H₂O₂. The beaker should be covered by a watch glass during leaching.
4. Transfer the sample to a centrifuge tube and centrifuge (2000 rev./min for 10 minutes). Transfer the supernate to a 250-mL glass beaker.
5. Return the sample to the beaker, repeat Steps 3 and 4, and combine the two supernates.
6. Evaporate the leach solution on a hotplate to a volume of 10–20 mL or until turbidity starts.
7. Dilute with water to 100 mL and transfer to a centrifuge tube.
8. Precipitate hydroxides by adding ammonia to pH5, then complete the precipitation by adding (NH₄)₂CO₃ to pH9. Centrifuge and discard the supernate. Wash the precipitate with 25 mL 1% ammonia, centrifuge again and discard the supernate.
9. Dry the precipitate in a drying oven at 105°C overnight.
10. Dissolve the sample in 8M HNO₃ 20–50 mL (or more if necessary). Add about 100 mg of NaNO₂ per 50 mL of acid and heat gently. Proceed with ion exchange, Step A.1, Section IV-2 below.

Note: If high-fired Pu oxides are present in the sample or produced during ignition, this leaching procedure may give low recoveries and incomplete exchange between the Pu in the sample and the added Pu tracer. The reader may refer to C.W. Sill (1975) for more details (see Bibliography).

IV-2. PLUTONIUM SEPARATION

A. *Ion exchange procedure I*

1. Prepare an ion exchange column with anion exchange resin (AG 1X8, 100–200 mesh) diameter = 1 cm, height = 8 cm. The resin is added to the column slurried in water. Let the water drain to the surface of the resin.
2. Pass 60 mL of 8M HNO₃ through the column.
3. Pass the sample through the column at a speed of 1–2 mL/min. Wash the column and beaker with portions of 8M HNO₃ up to 100 mL with the same speed. Collect effluents and washings for americium and curium separation.
4. Wash the column with 100 mL 10M HCl. Discard the washings.
5. Elute plutonium with 80 mL 9M HCl–0.1M NH₄I (freshly prepared). Collect the eluate in a 100-mL glass beaker and evaporate to near dryness. Care should be taken to avoid evaporating the sample to complete dryness. Proceed with electrodeposition, Section IV-4.

IV-3. AMERICIUM AND CURIUM SEPARATION

A. *Oxalate precipitation procedure*

1. Evaporate the effluent and washings from Step A.3, Section IV-2 above to a few mL or until turbidity occurs. Dilute to 300 mL with H₂O.
2. Add 100 mg of calcium in the form of CaCl₂ and 20 g of oxalic acid to the sample. Heat on a hotplate for 20 min.
3. Precipitate oxalates by adding ammonia to pH1.5 (white precipitate occurs).
4. Filter through paper filter by gravity. Wash the filter with 0.5% ammonium oxalate solution.
5. Acidify the filtrate to pH1 with HCl. Add 100 mg calcium to the sample and repeat the oxalate precipitation. Filter and combine the two filters.
6. Ash the filters overnight in a porcelain crucible at 550°C.

B. Ion exchange procedure II (clean-up step)

1. Dissolve the residue from Step A.6, Section IV-3 above in 10 mL 10M HCl.
2. Prepare a 'double ion exchange column' (diameter: 1 cm) consisting of 2 cm each of ion exchange resins AG 1X8 and AG 50WX8 (100–200 mesh). An ion exchanger (AG 501X8) is available which contains both resins along with an indicator that changes the colour when the exchange capacity is saturated. Pass 40 mL of 10M HCl through the column.
3. Pass the sample through the column. Wash crucible and column with portions of 10M HCl up to 40 mL. Collect effluent and washings. Evaporate to dryness.

C. Extraction procedure

1. Dissolve the the residue from Step B.3, Section IV-3 above in 10–50 mL 12M HNO₃. Transfer the sample solution to a 100-mL separation funnel with 10 mL 12M HNO₃.
2. Add 1 mL DDCP (dibutyl-N, N-diethyl carbamyl phosphonate) and shake for 30 seconds.
3. Let the phases separate and transfer the aqueous (lower) phase to another separation funnel.
4. Add 1 ml of DDCP to the sample in the second funnel and extract again.
5. After separation of the phases, discard the aqueous layer and combine the DDCP fractions. Use 20 mL 12M HNO₃ to wash one of the funnels. Collect the HNO₃ together with the DDCP in the other one. Shake for 10 seconds.
6. Add 20 mL of toluene and let the phases separate. Discard the aqueous (lower) layer.
7. Back extract Am/Cm with two 20-mL portions of 2M HNO₃ (shake for 2 min). Collect the HNO₃ in a 50-mL beaker and evaporate to almost dryness.

D. Ion exchange procedure III: Am/Cm rare earth separation

1. Prepare an ion exchange column as in Step A.1, Section IV-2 above but use AG 1X4 anion exchange resin instead (height 4 cm).
2. Dissolve the sample from Step C.7, Section IV-3 above in 5–10 mL 1M HNO₃–93% CH₃OH. Add 0.5 cm³ of AG 1X4 ion exchange resin to the beaker from Step C.7, Section IV-3 and mix. Let the beaker stand for 0.5 hour covered with plastic film. Mix occasionally by swirling the beaker.
3. Generate the column with 50 mL 1M HNO₃–93% CH₃OH.
4. Let the sample pass through the column at a speed of 0.5 mL/min.

5. Wash beaker and column with portions of the feed solution up to 50 mL. Try to quantitatively transfer the resin added to the beaker. Discard the effluents and washings. Drain to the level of the resin.
6. Wash the column with 80 mL of 0.1M HCl-0.5M NH₄SCN-80% CH₃OH. Discard the washings. Use a portion of this to wash the beaker if the transfer of resin has not been completely successful.
7. Wash the column with 20 mL of 1M HNO₃-93% CH₃OH. Discard the washings.
8. Elute Am/Cm with 70 mL 1.5M HCl-86% CH₃OH into a 150-mL beaker.
9. Evaporate the sample to almost dryness. Destroy thiocyanates with a few drops of conc. HNO₃ during heating. Proceed with electrodeposition, Section IV-4.

IV-4. ELECTRODEPOSITION

A. *Plating preparation*

1. Add 1 mL 0.3M Na₂SO₄ to the samples from Step A.5, Section IV-2 above and/or Step D.9, Section IV-3 above and evaporate to dryness.
2. Add 300 μL of conc. H₂SO₄. Heat until the sample is dissolved and white fumes appear.
3. Add 4 mL H₂O and 1 drop of 0.4% thymol blue. Adjust pH to 2 (orange) with ammonia.
4. Transfer the sample to an electrodeposition cell (mounted with a stainless steel disc) with 5 mL 1% H₂SO₄ and adjust pH to 2.1-2.4 with ammonia. Use indicator paper for this determination. If the end point is exceeded, use 20% H₂SO₄ for correction.

B. *Electroplating*

1. Adjust the anode (platinum wire of horizontal helix shape) distance to the disc to about 3 mm.
2. Electrolyse at 1 A for 1 h. At the end of electrolysis, add 1 mL conc. ammonia and switch off the current after 1 min.
3. Discard the liquid and dismount the cell. Wash the disc with 1% ammonia and acetone.
4. Touch the edge of the disc with a paper to absorb the acetone. Dry the disc by heating it gently. The sample is now ready for alpha spectrometry.

TABLE XI. ALPHA-EMITTING RADIONUCLIDES OF INTEREST

	E (MeV)	Fraction	T _{1/2}
Pu-236	5.77	0.69	2.85 a
	5.72	0.31	
Pu-238	5.50	0.71	87.7 a
	5.46	0.29	
Pu-239	5.16	0.73	24 110 a
	5.14	0.15	
	5.10	0.12	
Pu-240	5.17	0.73	6563 a
	5.12	0.27	
Pu-242	4.90	0.77	373 500 a
	4.86	0.23	
Am-241	5.49	0.86	433 a
	5.44	0.13	
Am-243	5.28	0.88	7370 a
	5.23	0.11	
Cm-242	6.11	0.74	163 d
	6.07	0.26	
Cm-243	6.06	0.05	28.5 a
	5.99	0.05	
	5.79	0.74	
	5.74	0.11	
Cm-244	5.81	0.76	18.1 a
	5.76	0.24	

IV-5. CALCULATIONS

A. Activity

1. The activity in the sample is simply calculated as:

$$A = \frac{Xp}{YW} \text{ or } A = \frac{Xp}{YV} \quad (\text{IV-1})$$

where:

- A is the sample activity in Bq/kg for solid samples or Bq/L for water;
- X is the number of counts in the peak from the sample after subtraction of background;
- Y is the number of counts in the peak from the yield determinant after subtraction of background;
- W is the mass of sample analysed (kg);
- V is the volume of sample analysed (L);
- p is the activity of yield determinant added (Bq).

IV-6. GENERAL REMARKS

A. Alpha spectrometry

1. The counting efficiency of the detector system is normally invariant with energy within the range 4–8 MeV.
2. The energy resolution is normally 20–50 keV and counting times 1–20 days. The alpha-emitting radionuclides are seldom monoenergetic but not all the peaks are normally resolved (see Table XI).
3. The alpha energies of ^{239}Pu and ^{240}Pu overlap and it is therefore impracticable to attempt reporting these isotopes separately. A good practice is to report data as ^{239}Pu plus ^{240}Pu .
4. The analyst must be aware of possible interference from natural alpha-emitting radionuclides such as:

^{228}Th for ^{238}Pu and ^{241}Am

^{210}Po for ^{243}Am

^{227}Ac — ^{227}Th and decay products for ^{242}Cm .

5. Shorter procedure:

The oxalate precipitation step (see Section IV-3A above) and the extraction step (see Section IV-3C) used in the americium and curium method can be omitted for certain samples. The omission of these steps depends on the type and quantity of material being analysed and on the experience of the analyst.

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Annex V

UNITS, PREFIXES AND SYMBOLS

V-I. RELATIONSHIP BETWEEN OLD AND NEW RADIATION UNITS

Quantity	Old unit	Symbol	New unit	Symbol	Dimensions	Relationship
Activity	curie	Ci	becquerel	Bq	s^{-1}	1 Ci = 3.7×10^{10} Bq
Absorbed dose	rad	rad	gray	Gy	$J \cdot kg^{-1}$	1 rad = 0.01 Gy
Dose equivalent	rem	rem	sievert	Sv	$J \cdot kg^{-1}$	1 rem = 0.01 Sv

V-II. UNITS OF COLLECTIVE DOSE

Old	New
Man-rad	Man-Gy
Man-rem	Man-Sv

Note: Roentgen is the unit of exposure.

$$1 \text{ Roentgen (R)} = 258 \mu\text{C} \cdot \text{kg}^{-1}$$

(microcoulomb per kilogram).

V-III. PREFIXES

Prefix	Symbol		Multiplier
Exa	E	1 000 000 000 000 000 000	10^{18}
Peta	P	1 000 000 000 000 000	10^{15}
Tera	T	1 000 000 000 000	10^{12}
Giga	G	1 000 000 000	10^9
Mega	M	1 000 000	10^6
Kilo	k	1000	10^3
Milli	m	0.001	10^{-3}
Micro	μ	0.000 001	10^{-6}
Nano	n	0.000 000 001	10^{-9}
Pico	p	0.000 000 000 001	10^{-12}
Femto	f	0.000 000 000 000 001	10^{-15}
Atto	a	0.000 000 000 000 000 001	10^{-18}

Note: When the symbol for a unit is shown with a superscript -1 on the right, it signifies that the quantity is being used in a fractional context or to present rate. Thus Sv^{-1} means per sievert, and $\text{Sv} \cdot \text{h}^{-1}$ means sievert per hour.

V-IV. COMMON SYMBOLS

Symbol	Term
α	Alpha
β	Beta
γ	Gamma
e	Electron
p	Proton
n	Neutron
Z	Atomic number
A	Mass number
eV	Electronvolt
Bq	Becquerel
Gy	Gray
Sv	Sievert
man-Sv	Man-sievert
$T_{1/2}$	Half-life

Annex VI

NUCLEAR ACCIDENT SCENARIOS

In this annex four possible nuclear accidents which would release different radionuclide spectra are listed together with the time dependent change in these spectra. In addition, other potential nuclear accidents are listed.

VI-1. SUMMARY OF TYPES OF ACCIDENTS

The four possible accidents which are considered along with their time dependent radionuclide spectra are the following:

- Reactor meltdown with or without failed containment
- Reactor meltdown with particle containment
- Nuclear fuel reprocessing plant release
- Plutonium fuel fabrication plant release

The radionuclides released in each of the above scenarios are listed in the sections below.

VI-1.1. Reactor meltdown with or without failed containment¹

A. *Of importance in the first day:*

- (a) Radionuclides with noble gas precursors;
- (b) Volatile radionuclides;
- (c) Less volatile and refractory radionuclides (fine particles, aerosols):

Radionuclides with half-lives of 6 hours and greater²:

⁹⁰Y, ⁹¹Sr, ⁹³Y, ⁹⁶Nb*, ⁹⁷Zr, ⁹⁹Mo,
¹⁰⁵Rh, ¹⁰⁹Pd, ¹¹¹Ag, ¹¹²Pd, ¹¹⁵Cd, ¹²¹Sn,
¹²⁵Sn, ¹²⁶Sb, ¹²⁷Sb, ¹³¹I, ¹³²I, ^{131m}Te,
¹³²Te, ¹³³I, ¹³⁵I, ¹⁴⁰La, ¹⁴²Pr*, ¹⁴³Ce,
¹⁴³Pr, ¹⁴⁶Ba, ¹⁴⁷Nd, ¹⁴⁹Pm, ¹⁵¹Pm, ^{152m}Eu*,
¹⁵³Sm, ¹⁵⁶Sm, ¹⁵⁷Eu, ²³⁹Np.

¹ The presence of high levels of the radionuclides of cerium, zirconium, ruthenium and transuranic elements in foods and environmental materials indicates the presence of hot particles which may be of special importance in considering exposure by inhalation and/or ingestion.

² Italic type denotes radionuclides are of major concern. Asterisks denote shielded fission products.

B. *Of importance in the first week:*

- (a) Volatile radionuclides;
- (b) Less volatile or refractory radionuclides:

Radionuclides with half-lives of about 1 day and greater:

^{86}Rh , ^{89}Sr , ^{90}Y , ^{91}Y , ^{95}Nb , ^{95}Zr ,
 $^{96}\text{Nb}^*$, ^{99}Mo , ^{160}Tb , ^{103}Ru , ^{105}Rh , ^{111}Ag ,
 ^{112}Pd , ^{115}Cd , $^{115\text{m}}\text{Cd}$, ^{121}Sn , ^{124}Sb , ^{125}Sn ,
 ^{127}Sb , ^{131}I , $^{131\text{m}}\text{Te}$, ^{132}Te , ^{133}I , ^{136}Cs ,
 ^{140}Ba , ^{140}La , ^{141}Ce , ^{143}Ce , ^{143}Pr , ^{147}Nd ,
 ^{149}Pm , ^{151}Pm , ^{153}Sm , ^{239}Np .

C. *Of long term importance:*

^3H , ^{89}Sr , ^{90}Sr , ^{91}Y , $^{93\text{m}}\text{Nb}$, ^{95}Nb ,
 ^{103}Ru , ^{106}Ru , $^{110\text{m}}\text{Ag}$, $^{113\text{m}}\text{Cd}$, $^{115\text{m}}\text{Cd}$, $^{121\text{m}}\text{Sn}$,
 ^{123}Sn , ^{124}Sb , ^{125}Sb , ^{129}I , ^{134}Cs , ^{137}Cs ,
 ^{141}Ce , ^{144}Ce , ^{147}Pm , ^{160}Tb , ^{238}Pu , ^{239}Pu ,
 ^{240}Pu , ^{241}Am , ^{241}Pu , ^{242}Cm , ^{242}Pu , ^{243}Am ,
 ^{244}Cm .

VI-1.2. Reactor meltdown with particle containment

A. *Of importance in the first day:*

^3H , ^{88}Rb , ^{89}Sr , ^{90}Sr , ^{90}Y , ^{91}Sr ,
 ^{91}Y , ^{103}Ru , ^{105}Ru , ^{106}Ru , ^{121}I , ^{123}I ,
 ^{132}I , ^{134}I , ^{135}I , ^{136}Cs , ^{138}Cs , ^{139}Cs ,
 ^{139}Ba , ^{140}Ba , ^{140}La .

B. *Of importance in the first week:*

^3H , ^{89}Sr , ^{90}Sr , ^{103}Ru , ^{105}Ru , ^{106}Ru ,
 ^{131}I , ^{133}I , ^{140}Ba , ^{140}La .

C. *Of long term importance:*

^3H , ^{89}Sr , ^{90}Sr , ^{99}Tc , ^{103}Ru , ^{106}Ru ,
 ^{129}I , ^{131}I , ^{137}Cs .

VI-1.3. Nuclear fuel reprocessing plant release:

^{90}Sr , ^{95}Nb , ^{95}Zr , ^{99}Tc , ^{103}Ru , ^{106}Ru ,
 ^{129}I , ^{131}I , ^{134}Cs , ^{137}Cs , ^{141}Ce , ^{144}Ce ,
 ^{238}Pu , ^{239}Pu , ^{240}Pu , ^{241}Am , ^{241}Pu , ^{242}Cm ,
 ^{242}Pu , ^{243}Am , ^{244}Cm .

VI-1.4. Plutonium fuel fabrication plant release:

^{238}Pu , ^{239}Pu , ^{240}Pu , ^{241}Am , ^{241}Pu , ^{242}Pu .

VI-2. POTENTIAL RADIONUCLIDE RELEASES

Other nuclear accidents which may result in major atmospheric emissions but which are not specifically discussed here include the following:

- Plutonium fuelled reactor meltdown
- Breeder reactor meltdown
- High flux radionuclide production reactor meltdown
- Fast flux reactor meltdown
- Nuclear powered ship/submarine reactor meltdown
- Satellite re-entry and burnup of satellite nuclear power source
- Nuclear weapon destruction by chemical explosion
- Criticality at nuclear materials processing plant
- Fusion reactor fuel loss.

Each of these possible accidents may release a unique spectrum of radionuclides and this should be considered in developing radioanalytical capabilities.

Nuclear weapon detonation would be a major source of fission products. Some of the possible scenarios for atmospheric release are:

- Venting from underground tests
- Venting from underwater nuclear tests
- Above ground nuclear testing
- Nuclear war.

Annex VII

RADIONUCLIDE DATA¹

For alpha-, beta- and gamma-emitting radionuclides the following nuclear data are given (Tables XII–XVII):

Half-life

Energy E_r of decay r

Absolute emission probability P_r of decay r

The half-life is given in years (a), days (d), hours (h) and minutes (min); the energy E_r in keV. One year equals 365.25 days.

For beta-emitters that show a continuous emission spectrum, the average energy E_β and the maximum energy E_β^m are given. In Table XIII, for beta-emitters, data are given for some nuclides which emit X-rays only.

Measurement uncertainties are attached in brackets to the respective value (in units of the last significant digit). They correspond to the one-fold standard deviation. In Table XIV, representing decay schemes for gamma-emitters (marked with a + next to the nuclide symbol), the lines for daughter nuclides are also given. For the emission probabilities, radioactive equilibrium is assumed.

The emission probabilities of the radionuclides originating from the ²³⁵U chain and partly from the ²³⁸U chain are not recalculated for radioactive equilibrium, and the radionuclides are listed with the respective half-life. To refer to radioactive equilibrium, the decay branching ratios (within the decay chain) have to be considered.

¹ Taken from:

KANISCH, G., RÜHLE, H., SCHELENZ, R. (Eds), Messanleitungen für die Überwachung der Radioaktivität in der Umwelt, Bundesminister des Inneren, Karlsruhe (1984), α -, β -emitters updated 1987 (Tables XII and XIII).

SCHÖTZIG, U., SCHRADER, H., Halbwertszeiten und Photonen-Emissionswahrscheinlichkeiten von häufig verwendeten Radionukliden, Rep. PTB-16/2, 2nd edn., Physikalisch-Technische Bundesanstalt, Braunschweig (1986).

TABLE XII. ALPHA EMITTERS

Nuclide	Half-life	E_{α} (keV)	P_{α}
U-234	2.457 (3) 10^5 a	4774.8	0.725 (15)
		4722.6	0.275 (15)
U-235	7.037 (7) 10^8 a	4400	0.57 (3)
		4374	0.061
		4368	0.123
		4218	0.062
U-238	4.468 (5) 10^9 a	4197	0.77 (4)
		4150	0.23 (4)
Pu-238	87.7 (3) a	5499.07	0.715 (2)
		5456.3	0.285 (2)
Pu-239	2.411 (3) 10^4 a	5156.6	0.733 (2)
		5143.8	0.151 (2)
		5105.0	0.115 (2)
Pu-240	6.563 (7) 10^3 a	5168.17	0.7351 (36)
		5123.68	0.2639 (21)
Pu-242	3.735 (11) 10^5 a	4900.5	0.770 (30)
		4856.2	0.230 (20)
Am-241	432.0 (2) a	5485.60	0.852 (8)
		5442.90	0.131 (3)
Cm-242	162.94 (6) d	6112.77	0.738 (5)
		6069.42	0.262 (5)
Cm-244	18.10 (2) a	5804.82	0.764 (2)
		5762.70	0.236 (2)

TABLE XIII. BETA EMITTERS

Nuclide	Half-life	E_{β}^m (keV)	E_{β} (keV)	E_x (keV)	P_x
H-3	12.35 (1) a	18.6	5.68		
C-14	5730 (40) a	156.48	49.47		
P-32	14.29 (2) d	1710.4	695.0		
S-35	87.44 (7) d	167.47	48.80		
Fe-55	2.75 (2) a			K_{α} 5.9 K_{β} 6.5	0.278 (10)
Ni-63	96 (4) a	65.87	17.13		
Sr-89	50.5 (1) d	1492	583.1		
Sr-90	28.7 (3) a	546.0	195.8		
Y-90	64.1 (1) h	2284	934.8		
I-125	59.3 (2) d			K_{α} 27.4 K_{β} 31.0	1.140 (29) 0.258 (9)
Pb-210	22.3 (2) a	16.5 63.0	4.15 (80%) 16.13 (20%)		
		E total:	6.51		

TABLE XIV. GAMMA EMITTERS

Nuclide	Half-life	E_γ (keV)	P_γ
Be-7	53.17 (7) d	477.61	0.1032 (4)
Na-22	950.4 (4) d	511.00 (3)	1.807 (2)
		1274.542	0.9994 (2)
Na-24	0.62323 (12) d	1368.63 (3)	0.99994 (2)
		2754.030	0.99876 (8)
K-40	1.277 (8) 10^9 a	1460.81	0.1067 (11)
Ar-41	1.827 (7) h	1293.64	0.9916 (2)
Sc-46	83.80 (3) d	889.280	0.99984 (1)
		1120.55	0.99987 (1)
Cr-51	27.71 (3) d	320.08	0.0985 (9)
Mn-54	312.5 (5) d	834.84	0.99975 (3)
Mn-56	0.10744 (3) d	846.75	0.989 (3)
		1810.72	0.272 (8)
		2113.05	0.143 (4)
Co-56	77.3 (3) d	846.75	0.9993 (1)
		977.42	0.0144 (2)
		1037.820	0.1411 (11)
		1175.09	0.0227 (3)
		1238.26	0.667 (6)
		1360.21	0.0427 (2)
		1771.40	0.1550 (14)
		2015.35	0.0302 (5)
		2034.91	0.0788 (8)
		2598.55	0.1720 (13)
		3202.24	0.0324 (9)
		3253.52	0.0798 (26)
3273.20	0.0189 (9)		
3451.42	0.00954 (36)		
Co-57	271.84 (4) d	122.06	0.8559 (19)
		136.47	0.1058 (8)
Co-58	70.78 (10) d	511.00	0.300 (5)
		810.78	0.9945 (1)
Fe-59	44.53 (3) d	142.54	0.0100 (3)
		192.35	0.0270 (7)
		1099.25	0.561 (12)
		1291.57	0.436 (8)

TABLE XIV. (cont.)

Nuclide	Half-life	E_γ (keV)	P_γ
Co-60	1925.5 (8) d	1173.24	0.9990 (2)
		1332.50	0.999824 (5)
Zn-65	243.9 (3) d	511.00	0.0286 (2)
		1115.55	0.504 (3)
Se-75	119.76 (5) d	121.12	0.173 (3)
		136.00	0.590 (8)
		198.60	0.0147 (3)
		264.65	0.591 (8)
		279.53	0.252 (4)
		303.910	0.0134 (2)
		400.65	0.1156 (14)
Kr-85	3.909 (8) 10^3 d	514.01	0.00434 (10)
Kr-85m	4.48 (8) h	151.18	0.753 (16)
		304.87	0.141 (5)
Sr-85	64.85 (3) d	514.01	0.984 (5)
Kr-87	1.272 (9) h	402.58	0.495 (16)
		673.87	0.0191 (10)
		845.43	0.073 (4)
		1175.40	0.0112 (6)
		1740.52	0.0205 (10)
		2011.88	0.0290 (14)
		2554.8	0.093 (6)
2558.1	0.039 (4)		
Kr-88	2.84 (2) h	165.98	0.0310 (15)
		196.32	0.260 (13)
		362.23	0.0225 (12)
		834.83	0.130 (7)
		1518.39	0.0215 (12)
		1529.77	0.109 (6)
		2029.84	0.0453 (23)
		2035.41	0.0374 (21)
		2195.84	0.132 (7)
		2231.77	0.0339 (17)
2392.11	0.346 (16)		
Y-88	106.66 (5) d	898.04	0.946 (5)
		1836.06	0.9924 (7)

TABLE XIV. (cont.)

Nuclide	Half-life	E_γ (keV)	P_γ
Kr-89	3.16 (4) min	220.90	0.200 (17)
		497.5	0.066 (7)
		576.96	0.056 (5)
		585.80	0.166 (14)
		738.39	0.042 (4)
		867.08	0.059 (5)
		904.27	0.072 (6)
		1324.28	0.0306 (25)
		1472.76	0.069 (6)
		1530.04	0.033 (3)
		1533.68	0.051 (4)
		1693.7	0.044 (4)
		2012.23	0.0156 (14)
		2866.23	0.0174 (14)
3532.9	0.0134 (11)		
3923.0	0.0041 (4)		
Sr-89	50.5 (1) d	909.2	0.0000976 (20)
Zr-95	64.09 (10) d	724.20	0.440 (5)
		756.73	0.543 (5)
Nb-95	35.0 (1) d	765.80	0.9980 (2)
Mo-99 +	2.7476 (5) d	140.47	0.0495 (9)
		181.06	0.0603 (7)
		366.42	0.0122 (2)
		739.50	0.1231 (9)
		777.92	0.0433 (4)
Tc-99m	0.25025 (8) d	140.47	0.8897 (24)
Ru-103	39.272 (9) d	497.08	0.909 (7)
		610.33	0.0565 (7)
Ru-106 +	372.6 (10) d	511.85	0.2047 (23)
		616.17	0.00735 (13)
		621.84	0.0995 (8)
		1050.47	0.01452 (13)
Ag-108m	127 (21) a	433.93	0.905 (6)
		614.37	0.898 (19)
		722.95	0.908 (19)

TABLE XIV. (cont.)

Nuclide	Half-life	E_γ (keV)	P_γ
Ag-110m	249.79 (20) d	657.75	0.9465 (16)
		677.61	0.1068 (11)
		706.670	0.166 (2)
		763.93	0.224 (2)
		884.67	0.734 (8)
		937.48	0.346 (4)
		1384.27	0.247 (3)
		1475.76	0.0397 (4)
		1505.00	0.1316 (20)
Cd-109	463 (1) d	88.03	0.0365 (7)
In-111	2.8049 (5) d	171.28	0.9093 (22)
		245.39	0.9417 (11)
Sn-113 +	115.1 (2) d	255.12	0.0193 (10)
		391.69	0.649 (7)
Te-123m	119.7 (3) d	158.96	0.840 (5)
Sb-124	60.20 (3) d	602.72	0.9783 (5)
		645.82	0.0744 (4)
		722.78	0.1078 (6)
		1691.02	0.4752 (23)
		2091.0	0.0547 (3)
Sb-125	1008.1 (8) d	176.33	0.0679 (7)
		380.44	0.01520 (19)
		427.89	0.294 (3)
		463.38	0.1045 (11)
		600.56	0.1778 (18)
		606.64	0.0502 (9)
		635.90	0.1132 (20)
		671.41	0.0180 (5)
I-125	59.3 (2) d	35.49	0.0667 (22)
I-131	8.021 (1) d	364.48	0.816 (6)
		636.97	0.0712 (6)
		722.89	0.0178 (2)
Xe-131m	11.84 (7) d	163.93	0.0196 (6)
Xe-133	5.245 (6) d	79.62	0.0026 (2)
		81.00	0.377 (8)

TABLE XIV. (cont.)

Nuclide	Half-life	E_γ (keV)	P_γ
Xe-133m	2.19 (1) d	233.18	0.103 (3)
Ba-133	3842 (18) d	53.16	0.0220 (4)
		79.62	0.0261 (7)
		81.00	0.340 (8)
		276.39	0.0710 (1)
		302.85	0.1833 (22)
		356.01	0.623 (7)
		383.85	0.0892 (9)
Cs-134	754.2 (5) d	475.35	0.0151 (3)
		563.23	0.0834 (12)
		569.32	0.1538 (22)
		604.70	0.976 (1)
		795.85	0.854 (3)
		801.93	0.0864 (12)
		1038.57	0.00998 (13)
		1167.94	0.0180 (20)
Xe-135	0.3796 (8) d	1365.15	0.0302 (3)
		249.79	0.9013 (8)
Xe-135m	15.36 (14) min	608.19	0.029 (9)
		526.57	0.812 (6)
Xe-137	3.83 (1) min	455.51	0.312 (5)
Cs-137	30.0 (2) a	661.66	0.850 (3)
Xe-138	14.13 (5) min	153.75	0.0595 (25)
		242.56	0.0350 (14)
		258.31	0.315 (13)
		396.43	0.063 (3)
		434.49	0.203 (9)
		1768.26	0.167 (7)
		2004.75	0.0535 (23)
		2015.82	0.123 (5)
Ce-139	137.65 (7) d	2252.26	0.0229 (11)
		165.85	0.800 (2)
Ba-140	12.751 (5) d	537.38	0.2439 (22)

TABLE XIV. (cont.)

Nuclide	Half-life	E_{γ} (keV)	P_{γ}
La-140	1.6779 (12) d	328.77	0.2074 (18)
		487.03	0.4594 (38)
		815.83	0.2364 (17)
		1596.49	0.9540 (8)
Ce-141	32.50 (1) d	145.44	0.489 (4)
Ce-144 +	284.45 (14) d	133.54	0.1109 (16)
		696.51	0.0134 (2)
		1489.15	0.00279 (3)
		2185.66	0.00700 (10)
Nd-147	10.98 (1) d	91.11	0.282 (13)
		531.03	0.123 (4)
Eu-152	4939 (6) d	121.78	0.2837 (24)
		244.69	0.0751 (6)
		344.27	0.2658 (18)
		411.11	0.02234 (13)
		443.91	0.0280 (18)
		778.89	0.1296 (7)
		963.38	0.1462 (6)
		1085.78	0.1016 (5)
		1112.02	0.1356 (6)
1407.95	0.2085 (9)		
Yb-169	32.032 (19) d	109.78	0.175 (2)
		118.19	0.0186 (2)
		130.52	0.1128 (10)
		177.21	0.2244 (21)
		197.95	0.360 (5)
		261.07	0.0168 (3)
		307.73	0.1010 (22)
Hf-180m	0.2300 (2) d	215.25	0.817 (12)
		332.31	0.945 (5)
		443.18	0.831 (29)
		500.71	0.139 (4)
Ta-182	114.43 (5) d	84.68	0.0263 (10)
		100.11	0.1423 (42)
		113.67	0.0187 (6)
		116.41	0.00445 (15)
		152.43	0.0695 (9)
		156.38	0.0263 (5)

TABLE XIV. (cont.)

Nuclide	Half-life	E_{γ} (keV)	P_{γ}
Ta-182	114.43 (5) d	179.39	0.0309 (4)
		198.35	0.0144 (2)
		222.10	0.0750 (1)
		229.32	0.0364 (5)
		264.07	0.0362 (6)
		1121.28	0.3530 (32)
		1189.04	0.1644 (15)
		1221.42	0.2717 (25)
		1230.87	0.1158 (11)
Ir-192	73.831 (8) d	295.96	0.286 (3)
		308.46	0.298 (3)
		316.51	0.828 (7)
		468.07	0.477 (4)
		588.59	0.0451 (4)
		604.41	0.0819 (6)
		612.47	0.0531 (4)
Au-198	2.696 (2) d	411.80	0.9547 (8)
Hg-203	46.612 (19) d	279.20	0.813 (2)
Bi-207	32.2 (20) a	569.70	0.977 (4)
		1063.66	0.7408 (25)
		1770.24	0.0687 (3)
Pb-210	22.3 (2) a	46.50	0.0418 (9)
Ra-226 +	1600 (7) a	186.21	0.0351 (6)
		241.98	0.0712 (11)
		295.21	0.1815 (22)
		351.92	0.351 (4)
		609.31	0.446 (5)
		768.36	0.0476 (7)
		934.06	0.0307 (4)
		1120.29	0.147 (2)
		1238.11	0.0578 (7)
		1509.23	0.0208 (5)
		1764.49	0.151 (3)
		2118.55	0.0117 (3)
		2204.22	0.0498 (12)
		2293.36	0.00301 (9)
2447.86	0.0155 (4)		

TABLE XIV. (cont.)

Nuclide	Half-life	E_γ (keV)	P_γ
Th-232 +	1.405 (6) 10^{10} a	59.0	0.0019 (3)
		105.0	0.016 (7)
		129.08	0.0223 (14)
		146.1	0.0021 (6)
		154.2	0.0090 (3)
		209.28	0.0381 (11)
		238.63	0.435 (12)
		240.98	0.0404 (17)
		270.23	0.0344 (9)
		278.0	0.0233 (7)
		300.09	0.0327 (9)
		321.7	0.00245 (23)
		328.0	0.0310 (9)
		338.32	0.1126 (27)
		409.51	0.0195 (7)
		463.00	0.0450 (12)
		562.3	0.0089 (5)
		570.7	0.00213 (26)
		583.0	0.307 (8)
		727.0	0.0735 (20)
		755.18	0.0104 (6)
		763.13	0.0073 (5)
		772.17	0.0145 (6)
		785.46	0.0107 (5)
		794.70	0.0434 (11)
		835.5	0.0153 (9)
		860.37	0.0455 (12)
911.07	0.266 (7)		
964.6	0.052 (6)		
969.11	0.1623 (38)		
1459.30	0.0078 (4)		
1588.00	0.0326 (10)		
2614.66	0.356 (11)		
Am-241	432.0 (2) a	59.54	0.360 (3)

TABLE XV. BACKGROUND LINES FOR GAMMA EMITTERS USING Ge(Li) SEMICONDUCTOR DETECTORS

Measuring time: 3×10^5 s (5000 min)

Volume of detector: approx. 100 cm^3

Shielding: none

Location: measuring room in the basement of a building

Explanation:

DC = decay chain in which the nuclide appears

U = decay chain of ^{238}U

Th = decay chain of ^{232}Th

E_γ (keV)	Radionuclide	(DC)	E_γ (keV)	Radionuclide	(DC)
53.2	Pb-214	(U)	338.3	Ac-228	(Th)
75.0	Pb-212	(Th)	351.9	Pb-214	(U)
75.0	Pb-214	(U)	409.5	Ac-228	(Th)
75.0	Tl-208	(Th)	438.8	{ K-40 }	
				{ (double escape) }	
77.1	Pb-212	(Th)	463.0	Ac-228	(Th)
87.2	Pb-212	(Th)	511.0	Tl-208	(Th)
	Pb-214	(U)	511.2	(annihilation)	
92.9	Th-234		562.3	Ac-228	(Th)
99.5	Ac-228	(Th)	583.0	Tl-208	(Th)
129.1	Ac-228	(Th)	609.3	Bi-214	(U)
154.2	Ac-228	(Th)	665.5	Bi-214	(U)
186.2	Ra-226	(U)	703.1	Bi-214	(U)
	U-235				
209.3	Ac-228	(Th)	727.0	Bi-212	(Th)
238.6	Pb-212	(Th)	755.2	Ac-228	(Th)
242.0	Pb-214	(U)	763.1	Tl-208	(Th)
270.2	Ac-228	(Th)	768.4	Bi-214	(U)
278.0	Tl-208	(Th)	772.1	Ac-228	(Th)
295.2	Pb-214	(U)	782.0	Ac-228	(Th)
300.1	Pb-212	(Th)	785.5	Bi-212	(Th)
328.0	Ac-228	(Th)	785.9	Pb-214	(U)

TABLE XV. (cont.)

E_γ (keV)	Radionuclide	(DC)	E_γ (keV)	Radionuclide	(DC)
794.7	Ac-228	(Th)	1512.8	Bi-212	(Th)
806.2	Bi-214	(U)	1538.5	Bi-214	(U)
830.5	Ac-228	(Th)	1543.4	Bi-214	(U)
835.5	Ac-228	(Th)	1556.9	Ac-228	(Th)
840.0	Ac-228	(Th)	1580.2	Ac-228	(Th)
860.4	Tl-208	(Th)	1583.2	Bi-214	(U)
893.4	Bi-212	(Th)	1588.0	Ac-228	(Th)
904.5	Ac-228	(Th)	1592.5	{ Tl-208 }	(Th)
911.1	Ac-228	(Th)		{(double escape)}	
934.1	Bi-214	(U)	1599.3	Bi-214	(U)
950.0	{ K-40 }		1620.6	Bi-212	(Th)
	{(single escape)}				
964.1	Bi-214	(U)	1624.7	Ac-228	(Th)
964.4	Ac-228	(Th)	1630.4	Ac-228	(Th)
1000.7	Pa-234m	(U)	1638.0	Ac-228	(Th)
1035.5	Ac-228	(Th)	1661.3	Bi-214	(U)
1052.0	Bi-214	(U)	1667.4	Ac-228	(Th)
1078.6	Bi-212	(Th)	1684.0	Bi-214	(U)
1120.3	Bi-214	(U)	1693.1	{ Bi-214 }	(U)
1155.2	Bi-214	(U)		{(single escape)}	
1238.1	Bi-214	(U)	1729.6	Bi-214	(U)
1281.0	Bi-214	(U)	1764.5	Bi-214	(U)
1377.7	Bi-214	(U)	1838.3	Bi-214	(U)
1385.3	Bi-214	(U)	1847.4	Bi-214	(U)
1401.5	Bi-214	(U)	2103.5	{ Tl-208 }	(Th)
1408.0	Bi-214	(U)		{(single escape)}	
1460.8	K-40		2118.6	Bi-214	(U)
1495.8	Ac-228	(Th)	2204.2	Bi-214	(U)
1501.5	Ac-228	(Th)	2447.9	Bi-214	(U)
1509.2	Bi-214	(U)	2614.7	Tl-208	(Th)

TABLE XVI. POSSIBLE INTERFERENCES IN GAMMA SPECTROMETRY
 $(\Delta E_\gamma = \pm 5 \text{ keV}; t_r = > 1 \text{ d}; P_\gamma = > 0.01)$

Nuclide	E_γ (keV)	P_γ	t_r (d)	Interfering nuclide	E_γ (keV)	P_γ	t_r (d)
Cr-51	320.084	0.0985	27.71	Np-239	315.88	0.013	2.35
				Rh-105	319.24	0.196	1.50
				Nd-147	319.4	0.022	11.06
				Ra-223	324.1	0.040	U-235
				Rn-219	324.1	0.040	U-235
Mn-54	834.843	0.99975	312.5	Bi-211	831.8	0.033	U-235
				Pa-234	831.8	0.057	U-238
				Pb-211	831.8	0.030	U-235
				Ac-228	835.6	0.015	Th-232
Co-57	122.0614	0.8559	271.84	Np-239	117.7	0.063	2.35
				Np-239	120.7	0.023	2.35
				Ra-223	122.4	0.011	U-235
				Rn-219	122.4	0.011	U-235
Co-58	810.775	0.9945	70.78	Pa-234	806.2	0.033	U-238
Fe-59	1099.251	0.561	44.53	—	—	—	—
	1291.569	0.436	44.53	—	—	—	—
Co-60	1173.238	0.999	1925.5	—	—	—	—
	1332.502	0.999824	1925.5	—	—	—	—
Zn-65	1115.546	0.504	243.9	Bi-214	1120.4	0.136	U-238
				Sc-46	1120.545	1.000	83.80
Zr-95	724.199	0.440	64.09	Sb-126	720.4	0.560	12.5
				Ce-143	722.0	0.045	1.40
				Sb-124	722.78	0.1126	60.2
				Sb-127	723.0	0.018	3.85
				Bi-212	727.17	0.065	Th-232
				La-140	751.79	0.0441	1.6779
Nb-95	765.8	0.998	35.0	Ag-110m	763.928	0.224	249.79
				Bi-214	768.7	0.042	U-238

TABLE XVI. (cont.)

Nuclide	E_γ (keV)	P_γ	t_r (d)	Interfering nuclide	E_γ (keV)	P_γ	t_r (d)
Mo-99/ Tc-99m	140.466	0.8896	(2.7476)	Co-57	136.4743	0.1058	271.84
				U-235	143.78	0.097	2.57×10^{11}
				Rn-219	144.3	0.032	U-235
				Ra-223	144.3	0.032	U-235
				Ce-141	145.4442	0.489	32.5
	181.057	0.0603	2.7476	Sb-125	176.334	0.0689	1008.1
				Cs-136	176.75	0.132	13.7
				U-235	185.72	0.54	2.57×10^{11}
				Pa-234	186.0	0.019	U-238
				Ra-226	186.211	0.0351	584 400
	366.421	0.0122	2.7476	I-131	364.48	0.816	8.021
				Pa-234	369.8	0.034	U-238
	739.5	0.1231	2.7476	Pa-234	742.8	0.029	U-238
	777.921	0.0433	2.7476	Ag-110m	744.26	0.0464	249.79
Te-131m				773.7	0.46	1.25	
			Te-131m	782.7	0.067	1.25	

Ru-103	497.080	0.909	39.272	Cd-115	492.29	0.081	2.23

Ru-106/ Rh-106	621.84 1050.47	0.0995 0.01452	(372.6) (367)	Ag-110m Cs-136	620.35 1048.1	0.0277 0.805	249.79 13.7

Ag-110m	657.749	0.94652	49.79	Sb-126	656.2	0.028	12.5
				Cs-137	661.66	0.850	10 958
	884.667	0.734	249.79	Pa-234	880.8	0.130	U-238
				Pa-234	883.2	0.120	U-238
	1384.27	0.247	249.79	Sc-46	889.277	1.000	83.8

Sb-124	602.72	0.9792	60.2	Sb-125	600.557	0.178	1008.1
				Sb-127	603.6	0.042	3.85
				Ir-192	604.414	0.0819	73.831
				Cs-134	604.699	0.976	754.2
				Sb-126	605.0	0.024	12.5
	Sb-125	606.641	0.0502	1008.1			
	1691.02	0.488	60.2	—	—	—	—

TABLE XVI. (cont.)

Nuclide	E_γ (keV)	P_γ	t_r (d)	Interfering nuclide	E_γ (keV)	P_γ	t_r (d)
Sb-125	176.334	0.0689	1008.1	Cs-136	176.75	0.132	13.7
				Mo-99	181.057	0.0603	2.7476
	427.889	0.2933	1008.1	Ba-140	423.69	0.0315	12.751
				Bi-211	426.9	0.019	U-235
				Pb-211	427.1	0.019	U-235
				La-140	432.55	0.0299	1.6779
	600.57	0.178	1008.1	Sb-124	602.72	0.9792	60.2
				Sb-127	603.6	0.042	3.85
				Ir-192	604.414	0.0819	75.1
				Cs-134	604.699	0.976	754.2
Sb-126				605.0	0.024	12.4	
635.895	0.1132	1008.1	I-131	636.973	0.0712	8.021	
I-131	364.48	0.816	8.021	Tl-210	360.0	0.040	U-238
				Mo-99	366.421	0.0122	2.7476
				Pa-234	369.8	0.034	U-238
Cs-134	604.699	0.976	754.2	Sb-125	600.557	0.178	1008.1
				Sb-124	602.72	0.9792	60.2
				Sb-127	603.6	0.042	3.85
				Ir-192	604.414	0.0819	73.831
				Sb-126	605.0	0.024	12.5
				Sb-125	606.641	0.0502	1008.1
				Bi-214	609.3	0.412	U-238
	Te-131m	793.6	0.159	1.25			
	795.845	0.854	754.2	Tl-210	795.0	1.000	U-238
				Ac-228	795.0	0.039	Th-232
				Pa-234	796.6	0.039	U-238
Sn-125				800.5	0.010	9.62	
Cs-137	661.66	0.850	10958	Ag-100m	657.749	0.9465	249.79
				Ce-143	664.0	0.050	1.40
				Te-131m	665.0	0.035	1.25
				Bi-214	666.0	0.022	U-238
				Sb-126	666.2	1.000	12.5

TABLE XVI. (cont.)

Nuclide	E_γ (keV)	P_γ	t_τ (d)	Interfering nuclide	E_γ (keV)	P_γ	t_τ (d)
Ba-140/ La-140	162.9	0.0621	12.751	Te-123m	158.96	0.840	119.7
				U-235	163.36	0.045	2.57×10^{11}
				Cs-136	164.04	0.045	13.7
	328.77	0.2074	(12.751)	Ra-223	324.1	0.04	U-235
				Rn-219	324.1	0.04	U-235
				Ac-228	328.3	0.026	Th-232
				Th-227	329.7	0.023	U-235
				Pa-231	329.9	0.01	U-235
				Ir-192	484.578	0.032	73.831
	487.03	0.4594	(12.751)	—	—	—	—
	537.38	0.2439	12.751	—	—	—	—
	815.83	0.2364	(12.751)	Ag-110m	818.02	0.073	249.79
				Cs-136	818.48	1.000	13.7
Pa-234				819.7	0.027	U-238	
1596.49	0.954	(12.751)	—	—	—	—	
Ce-141	145.4442	0.489	32.5	Tc-99m	140.466	0.8896	2.7476
				U-235	143.78	0.097	2.57×10^{11}
				Rn-219	144.3	0.032	U-235
				Ra-223	144.3	0.032	U-235
				Te-131m	149.7	0.242	1.25
Ce-144	133.544	0.1109	284.45	Ac-228	129.1	0.021	Th-232
				Pa-234	131.2	0.200	U-238
				Co-57	136.4743	0.1058	271.84

TABLE XVII. GAMMA LINES: LISTING BY ENERGY^a

Energy (keV)	Nuclide	Intensity /100 decays	Energy (keV)	Nuclide	Intensity /100 decays
13.60	Pu-239	4.40	97.43	Gd-153	27.60
13.85	Ba-140	1.20	98.44	Pu-239	0.59
14.41	Co-57	9.54	100.10	Ta-182	14.10
22.16	Cd-109	86.00	103.18	Gd-153	19.60
24.94	Cd-109	17.00	103.18	Sm-153	28.30
26.35	Am-241	2.40	105.31	Eu-155	20.50
27.40	Sb-125	61.92	106.12	Np-239	22.86
29.97	Ba-140	10.73	112.95	Lu-177	6.40
31.00	Sb-125	12.89	121.12	Se-75	17.32
31.82	Cs-137	1.96	121.78	Eu-152	28.32
32.19	Cs-137	3.61	122.06	Co-57	85.59
35.50	Sb-125	4.28	123.14	Eu-154	40.50
36.40	Cs-137	1.31	123.80	Ba-131	29.05
42.80	Eu-154	28.47	129.30	Pu-239	0.64
46.52	Pb-210	4.05	133.02	Hf-181	41.00
49.41	Np-239	0.10	133.54	Ce-144	10.80
51.62	Pu-239	0.27	134.25	W-187	8.56
59.54	Am-241	35.90	136.00	Se-75	58.98
59.54	U-237	33.48	136.25	Hf-181	6.90
60.01	Eu-155	1.14	136.48	Co-57	10.61
63.29	Th-23	43.83	140.51	Tc-99m	88.90
67.75	Ta-182	42.30	142.65	Fe-59	1.02
67.88	Np-239	0.90	143.21	Np-237	0.42
72.00	W-187	10.77	143.76	U-235	10.93
79.62	Xe-133	0.60	145.44	Ce-141	48.44
80.11	Ce-144	1.60	151.17	Kr-85m	75.08
80.18	I-131	2.62	158.20	Xe-135	0.29
81.00	Ba-133	32.92	162.64	Ba-140	6.21
81.00	Xe-133	37.00	163.33	U-235	5.00
86.50	Np-237	12.60	163.93	Xe-131m	1.96
86.54	Eu-155	30.80	164.10	Ba-139	22.05
86.79	Tb-160	13.20	165.85	Ce-139	79.95
88.03	Cd-109	3.61	172.62	Sb-125	0.18
91.10	Nd-147	27.90	176.33	Sb-125	6.79
92.38	Th-234	2.73	176.56	Cs-136	13.59
92.80	Th-234	2.69	181.06	Mo-99	6.52
94.67	Pu-239	0.37	185.72	U-235	57.50
97.43	Sm-153	0.73	186.21	Ra-226	3.28

^a Data primarily derived from "Nuclear Data Sheets" published by Academic Press.

TABLE XVII. (cont.)

Energy (keV)	Nuclide	Intensity /100 decays	Energy (keV)	Nuclide	Intensity /100 decays
192.35	Fe-59	3.08	302.85	Ba-133	18.71
196.32	Kr-88	26.30	304.84	Ba-140	4.30
205.31	U-235	5.03	304.86	Kr-85m	13.70
208.01	U-237	21.67	312.40	K-42	0.18
208.36	Lu-177	11.00	314.20	Pb-214	0.79
209.75	Np-239	32.70	319.41	Nd-147	1.95
216.09	Ba-131	19.90	320.08	Cr-51	9.83
220.90	Kr-89	20.40	328.77	La-140	20.50
228.16	Te-132	88.20	329.43	Eu-152	0.15
228.18	Np-239	10.79	333.03	Au-196	22.85
233.18	Xe-133m	10.30	334.31	Np-239	2.04
234.68	Zr-95	0.20	338.40	Ac-228	11.40
236.00	Th-227	11.05	340.57	Cs-136	48.55
238.63	Pb-212	44.60	344.28	Eu-152	22.67
240.98	Ra-224	3.95	345.95	Hf-181	12.00
241.98	Pb-214	9.00	351.92	Pb-214	38.90
244.70	Eu-152	7.51	355.73	Au-196	86.90
248.04	Eu-154	6.59	356.01	Ba-133	62.58
249.44	Ba-131	2.80	358.39	Xe-135	0.22
249.79	Xe-135	89.90	361.85	I-135	0.19
252.45	Eu-154	0.10	362.23	Kr-88	2.28
255.06	Sn-113	1.82	363.50	Kr-88	0.49
256.25	Th-227	6.71	363.93	Cs-138	0.24
258.41	Xe-138	31.50	364.48	I-131	81.24
258.79	Pb-214	0.55	365.29	Cs-138	0.19
264.66	Se-755	9.10	367.79	Eu-152	0.87
273.70	Bi-214	0.18	373.25	Ba-131	13.30
274.53	Pb-214	0.33	375.05	Pu-239	0.16
276.40	Ba-133	7.32	380.44	Sb-125	1.52
277.60	Np-239	14.20	383.85	Ba-133	8.89
279.19	Hg-203	81.55	387.00	Bi-214	0.37
279.54	Se-75	25.18	389.10	Bi-214	0.41
282.52	Yb-175	3.10	391.69	Sn-113	64.16
284.29	I-131	6.06	396.32	Yb-175	6.50
293.26	Ce-143	42.00	400.66	Se-75	11.56
295.21	Pb-214	19.70	402.58	Kr-87	49.60
295.94	Eu-152	0.45	405.74	Bi-214	0.17
298.57	Tb-160	26.90	407.99	Xe-135	0.36
300.09	Pb-212	3.41	411.12	Eu-152	2.27

TABLE XVII. (cont.)

Energy (keV)	Nuclide	Intensity /100 decays	Energy (keV)	Nuclide	Intensity /100 decays
411.80	Au-198	95.51	533.69	Pb-214	0.19
413.71	Pu-239	0.15	537.32	Ba-140	24.39
414.70	Sb-126	83.3	546.94	Cs-138	10.76
416.05	Eu-152	0.11	551.52	W-187	4.92
426.50	Bi-214	0.11	554.32	Br-82	70.60
427.89	Sb-125	29.44	555.61	Y-91m	56.10
433.95	Ag-108m	90.70	557.04	Ru-103	0.83
434.56	Xe-138	20.30	559.10	As-76	45.00
439.90	Nd-147	1.20	563.23	Cs-134	8.38
443.98	Eu-152	3.12	563.23	As-76	1.20
454.77	Bi-214	0.32	564.00	Sb-122	71.20
462.10	Pb-214	0.17	564.02	Eu-152	0.49
462.79	Cs-138	30.70	566.42	Eu-152	0.13
463.38	Sb-125	10.45	569.32	Cs-134	15.43
469.69	Bi-214	0.13	569.67	Bi-207	97.80
474.38	Bi-214	0.12	580.15	Pb-214	0.37
477.59	Be-7	1.03	583.19	Tl-208	85.77
479.57	W-187	21.13	585.80	Kr-89	16.90
480.42	Pb-214	0.34	586.29	Eu-152	0.46
482.16	Hf-181	83.00	591.74	Eu-154	4.84
487.03	La-140	45.50	595.36	I-134	11.16
487.08	Pb-214	0.44	600.56	Sb-125	17.78
488.66	Eu-152	0.42	602.73	Sb-124	97.80
496.28	Ba-131	43.78	604.70	Cs-134	97.56
497.08	Ru-103	89.50	606.64	Sb-125	5.02
497.50	Kr-89	6.80	608.19	Xe-135	2.87
503.39	Eu-152	0.16	609.31	Bi-214	43.30
510.57	I-133	1.84	610.33	Ru-103	5.64
511.00	Co-56	18.60	616.20	Ru-106	0.70
511.00	Cu-64	37.10	618.28	W-187	6.07
511.00	Na-22	90.00	619.07	Br-82	4.31
511.00	Y-88	0.40	621.79	I-134	10.59
511.00	Zn-65	2.83	621.84	Ru-106	9.81
511.85	Ru-106	20.60	635.90	Sb-125	11.32
513.99	Kr-85	0.43	636.97	I-131	7.27
513.99	Sr-85	98.30	645.86	Sb-124	7.38
526.56	Xe-135m	80.51	652.30	Sr-91	2.97
529.89	I-133	87.30	652.90	Sr-91	8.02
531.02	Nd-147	13.09	653.00	Sr-91	0.37

TABLE XVII. (cont.)

Energy (keV)	Nuclide	Intensity /100 decays	Energy (keV)	Nuclide	Intensity /100 decays
656.48	Eu-152	0.15	776.49	Br-82	83.40
657.05	As-76	6.17	777.88	Mo-99	4.62
657.71	Rb-89	10.10	778.91	Eu-152	12.96
657.76	Ag-110m	94.64	785.46	Bi-212	1.26
661.66	Cs-137	85.21	785.91	Pb-214	1.10
665.45	Bi-214	1.25	786.10	Bi-214	0.32
666.31	Sb-126	99.60	793.75	Te-131m	13.82
667.69	I-132	98.70	795.85	Cs-134	85.44
671.15	Eu-152	0.23	801.93	Cs-134	8.73
675.89	Au-198	0.80	810.77	Co-58	99.45
685.74	W-187	26.39	815.80	La-140	23.50
685.90	Nd-147	0.81	818.50	Cs-136	99.70
692.60	Sb-122	3.90	834.83	Mn-54	99.98
695.00	Sb-126	99.60	834.83	Kr-88	13.10
696.49	Ce-144	1.48	839.03	Pb-214	0.59
697.00	Sb-126	29.00	845.44	Kr-87	7.34
697.49	Pr-144	1.48	846.70	Co-56	99.93
698.33	Br-82	27.90	846.75	Mn-56	98.87
702.63	Nb-94	100.00	847.02	I-134	95.41
703.11	Bi-214	0.47	852.21	Te-131m	20.57
715.76	Eu-154	0.18	856.70	Sb-126	17.60
719.86	Bi-214	0.41	860.56	Tl-208	12.00
720.50	Sb-126	53.80	863.96	Co-58	0.68
722.79	Sb-124	10.76	871.10	Nb-94	100.00
722.89	I-131	1.80	873.19	Eu-154	11.50
722.95	Ag-108m	91.50	875.37	I-133	4.40
723.30	Eu-154	19.70	879.36	Tb-160	29.50
724.20	Zr-95	44.10	884.09	I-134	64.88
727.17	Bi-212	7.56	884.69	Ag-110m	72.68
739.50	Mo-99	13.00	889.26	Sc-46	99.98
749.80	Sr-91	23.60	898.02	Y-88	9.50
752.84	Bi-214	0.13	904.27	Kr-89	7.30
756.73	Zr-95	54.50	911.07	Ac-228	27.70
763.94	Ag-110m	22.2	925.24	La-140	7.09
765.79	Nb-95	99.79	937.49	Ag-110m	34.36
768.36	Bi-214	5.04	954.55	I-132	18.10
772.60	I-132	76.20	964.13	Eu-152	14.62
772.91	W-187	3.98	966.16	Tb-160	25.00
773.67	Te-131m	38.06	969.11	Ac-228	16.60

TABLE XVII. (cont.)

Energy (keV)	Nuclide	Intensity /100 decays	Energy (keV)	Nuclide	Intensity /100 decays
989.03	Sb-126	6.80	1260.41	I-135	28.60
996.32	Eu-154	10.30	1274.45	Eu-154	35.50
1001.03	Pa-234m	0.59	1274.51	Na-22	99.95
1004.76	Eu-154	17.89	1291.60	Fe-59	43.20
1009.78	Cs-138	29.80	1293.64	Ar-41	99.16
1024.30	Sr-91	33.40	1298.33	I-133	2.27
1031.88	Rb-89	59.00	1317.47	Br-82	26.90
1037.80	Co-56	14.09	1318.00	Fe-59	Pair peak
1043.97	Br-82	27.40	1332.50	Co-60	99.98
1048.07	Cs-136	79.72	1345.77	Cu-64	0.48
1050.47	Ru-106	1.73	1368.53	Na-24	99.99
1063.62	Bi-207	74.91	1377.82	Bi-24	45.06
1072.55	I-134	14.98	1384.30	Ag-110m	24.28
1076.70	Rb-86	8.78	1408.01	Eu-152	20.85
1087.66	Au-198	0.16	1420.50	Ba-139	0.30
1099.25	Fe-59	56.50	1435.86	Cs-138	76.30
1112.12	Eu-152	13.56	1457.56	I-135	8.60
1115.52	Zn-65	50.74	1460.75	K-40	10.70
1120.29	Bi-214	15.70	1472.76	Kr-89	7.00
1120.52	Sc-469	9.99	1489.15	Ce-144	0.30
1121.28	Ta-182	35.00	1524.00	K-42	17.90
1128.00	Ru-106	0.40	1529.77	Kr-88	11.10
1131.51	I-135	22.50	1573.73	Nb-94	0.15
1140.20	Sb-122	0.57	1596.48	Eu-154	1.67
1167.94	Cs-134	1.81	1596.49	La-140	95.49
1173.24	Co-60	99.90	1642.40	Cl-38	32.80
1177.94	Tb-160	15.20	1674.73	Co-58	0.52
1189.05	Ta-182	16.30	1678.03	I-135	9.50
1212.92	As-76	1.44	1690.98	Sb-124	47.30
1216.08	As-76	3.42	1740.52	Kr-87	2.04
1221.42	Ta-182	27.10	1764.49	Bi-214	17.00
1228.52	As-76	1.22	1768.26	Xe-138	16.70
1230.90	Ta-182	11.50	1770.23	Bi-207	6.85
1235.34	Cs-136	19.78	1771.40	Co-56	15.51
1236.56	I-133	1.44	1791.20	I-135	7.70
1238.11	Bi-214	5.94	1810.72	Mn-56	27.19
1238.30	Co-56	66.95	1836.01	Y-88	99.35
1248.10	Rb-89	43.00	2004.75	Xe-138	12.30
1257.00	Sb-122	0.77	2015.82	Xe-138	5.35

TABLE XVII. (cont.)

Energy (keV)	Nuclide	Intensity /100 decays	Energy (keV)	Nuclide	Intensity /100 decays
2090.94	Sb-124	5.58	2323.10	Sb-124	0.24
2113.05	Mn-56	14.34	2392.11	Kr-88	35.00
2167.50	Cl-38	44.00	2554.80	Kr-87	9.23
2185.70	Ce-144	0.77	2558.10	Kr-87	3.92
2195.84	Kr-88	13.30	2570.14	Rb-89	10.00
2196.00	Rb-89	13.60	2598.50	Co-56	16.74
2204.22	Bi-214	4.98	2614.53	Tl-208	99.79
2218.00	Cs-138	15.20	2753.90	Na-24	99.84

Annex VIII

GRASS SAMPLE COLLECTION

The Agency's Grass Ecosystem Sampling and Analysis Project has been designated as part of the Fallout Radioactivity Monitoring in Environment and Food (MEF) Programme and provides a good example for the collection and preparation of environmental samples. This annex presents the protocol, sampling information and forms provided to participants in the project as guidelines for collection and preparation of similar sample materials requiring radioanalyses in a nuclear incident.

For the Grass Ecosystem Sampling and Analysis Project, samples are collected once each year from approximately 20 locations distributed throughout Europe and Asia by volunteers from various institutions. The samples are packed in containers provided by the Agency and sent to the Agency's laboratory at Seibersdorf for preparation. Agency personnel perform the required preparations by the procedures outlined in Section VIII-2 below. After preparations are completed and the sample materials thoroughly homogenized, the materials are subdivided and sent to eight volunteer laboratories for radioanalyses by both non-destructive and destructive methods. Results of the analyses are sent to the Agency where they are compiled into a report for distribution. It is planned to continue the Grass Ecosystem Project for five years following the 1986 accident at Chernobyl.

VIII-1. PROTOCOL FOR GRASS ECOSYSTEM SAMPLES

The objective in collecting and analysing grass ecosystem samples is to determine the total radionuclide deposition per unit area of grassland and to determine what fraction of the radionuclides appears in the grass. Ideally, we would like to make these measurements on pasture grass where cattle feed and the radionuclides would thus enter the food chain by appearing in beef and milk.

Because of the difficulty in obtaining pasture grass samples before grazing occurs, we suggest a compromise programme that will sample lawn grass and its associated ecosystem. We believe that the uniformity permitted by the use of lawn grass will outweigh the uncertainties in extrapolating these grasses to pasture grass.

The grass ecosystem protocol requires collection of samples from three principal compartments of the grass ecosystem as illustrated in Fig. 15. It is desirable to sample the grass proper, the mat area, and the root/soil system. Grass samples are composed of the leaf and stem material that is devoid of mat material.

The mat itself would include some green leafy grass which is mixed with the mat material and overlies the root/soil system. The root/soil system would extend beneath the mat to a depth of approximately 2 cm and should include most of the root material. This comprehensive collection protocol will provide samples which

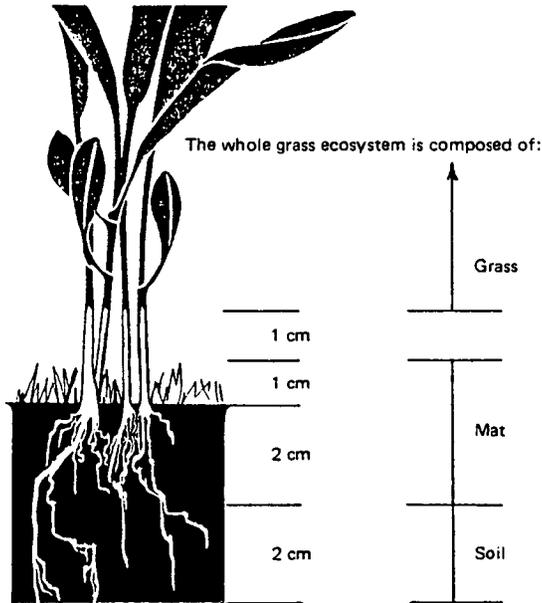


FIG. 15. Diagram for sampling a grass ecosystem.

will allow the behaviour of the specific radionuclides to be characterized. Details for sampling each of the three compartments are as follows (and see Section VIII-3.2).

VIII-1.1. Grass

It is desirable that the grass collected be about 1 cm above the mat region (2 cm above the surface). Only that part of the grass should be collected which is separated from the underlying mat since radionuclide concentrations in the mat are expected to be far greater than that in the grass, and we wish to avoid contamination of the grass samples with mat material. The grass samples could be collected by means of a lawn mower with a catcher which permits collection over a precisely measured and recorded area. This grass should be weighed while fresh and air-dried before being transferred to the plastic bag which will be provided by the Agency. A total of about 5 kg of grass should be collected. The size of the area sampled and the height of the grass should be recorded.

VIII-1.2. Mat

As illustrated in Fig. 15, the mat section includes a portion of the green leafy material and the dry mat which lies above the soil layer. This should be collected

by using a flat-edged scraping device, and care should be taken to avoid collecting the underlying soil material. The mat should be collected in the same area from which the grass was taken. It will require a much smaller but precisely measured and recorded area. A total of about 5 kg of mat material should be collected. Again this should be air-dried before being transferred to the plastic bag.

VIII-1.3. Root/soil

The root/soil depth to be sampled is illustrated in Fig. 15 and represents a depth of approximately 2 cm. This should be collected from beneath the area where the mat was taken and a total of about 5 kg should be taken. Again this should be air-dried before transferring to the plastic bag.

Note: Since sample collections of this type will be required annually for up to five years, it is requested that a sampling site be selected from which adjacent sampling can be conducted over this period of time.

VIII-1.4. Packaging and shipment

All three types of samples are to be placed, after sufficient air-drying, in plastic bags provided by the Agency. The loss of most of the moisture prior to shipment is desirable. All samples should be placed in cardboard boxes which are provided by the Agency. The following material will be shipped to each of the participants:

- Protocol for grass ecosystem samples,
- Laboratory Invoice/Packing Note for customs clearance only,
- Cardboard box for return shipment,
- Return label for cardboard box,
- Three plastic sample bags (one each for grass, mat and root/soil samples),
- Sampling forms.

VIII-2. SAMPLE PREPARATION

VIII-2.1. Grass samples

After the grass samples have been thoroughly dried at 105°C, they should be ground and then passed through a 2-mm sieve so that particles greater than this would be retained on the sieve. If necessary, the finer material could be separated by sieving two or three times during the grinding process so that only the larger particles are exposed to a longer grinding time. Once the material has been sieved, it should be homogenized and used for preparing aliquots for analysis. Table XVIII lists drying temperatures and particle sizes required for samples.

TABLE XVIII. DRYING TEMPERATURES AND PARTICLE SIZES REQUIRED FOR GRASS ECOSYSTEM SAMPLES

	Temperature	Particle size	Drying time
Grass	105°C	2-mm sieve	24 hours ^a
Mat	105°C	2-mm sieve	24 hours ^a
Soil	Room temperature	1-mm sieve	As required

^a A longer drying period can be used if the material does not appear to be dry after 24 hours.

VIII-2.2. Mat samples

The mat materials ought to be reasonably free of soil. It is not, however, necessary that all the soil be removed since it should contain a rather small amount of radioactivity compared to the mat material. The mat should be air-dried for several days prior to drying at 105°C. At this time, large amounts of soil, if present, could be removed by simply manipulating the materials by hand, although this would only be required if excessive amounts of soil were present. Once the soil is removed, the mat should be further dried at 105°C, pulverized, and sieved in the same manner as the grass.

VIII-2.3. Soil samples

Once the soil samples have been thoroughly dried at room temperature, they should be pulverized carefully with a large mortar and pestle in a manner that does not grind the rocks and roots that may be associated with the soil. Once this has been accomplished, the material should be sieved through a 1-mm mesh sieve. The separated soil, roots and rocks remaining should each be weighed; the roots and rocks can then be discarded. Soil aliquots should then be taken and the fraction they represent of the total aliquots and its area should be indicated.

VIII-3. EXAMPLES OF INFORMATION FORMS FROM THE IAEA

VIII-3.1. Collection information

The following is a typical memorandum to a collector:

TO: Collectors of grass samples

SUBJECT: Collection of grass ecosystem samples

We have been informed that you agreed to participate in our programme for collection of grass ecosystem samples to obtain data on the characteristics and behaviour of the fallout debris resulting from the Chernobyl accident, and we therefore enclose the following items:

- (a) Protocol for grass ecosystem samples (modified 88-05-07),
- (b) Laboratory Invoice/Packing Note for customs clearance only,
- (c) Cardboard box for return shipment,
- (d) Return label to be put on cardboard box,
- (e) Three sample containers (one each for grass, mat and root-soil samples),
- (f) Sampling form

You are requested to collect samples following the sampling instructions in the protocol for grass ecosystem samples (a). Packed samples with reporting forms should be sent back by mail (Cash on Delivery/C.O.D.) to the following address (return label enclosed):

International Atomic Energy Agency
Seibersdorf Laboratory
LAB/G6.05.2
Wagramerstrasse 5
P.O.Box 100
A-1400 Vienna, Austria

VIII-3.2. Sampling form

GRASS ECOSYSTEM SAMPLING FORM (GESF)

SEND TO:	IAEA SAMPLE CODE
International Atomic Energy Agency	DATE OF RECEIPT
Analytical Quality Control Services	DO NOT FILL IN BELOW HERE:
Chemistry Unit	
Seibersdorf Laboratory	
P.O. Box 100	
A-1400 Vienna, Austria	

**COLLECTOR'S
ADDRESS:**

**PLEASE INDICATE CHANGE OF ADDRESS IN SPACE PROVIDED UNDER
"COLLECTOR'S ADDRESS"**

GES FORM (Cont.)

1. COLLECTOR'S SAMPLE CODE
(Give the sample a code of your own choice, if desired, but not exceeding 9 characters or numbers)
2. SAMPLING DATE (YEAR/MONTH/DAY)
3. LAST HARVESTING DATE (YEAR/MONTH/DAY)
4. SPECIES OF SAMPLE (LAWN/PASTURE/OTHER)
(Specify as precisely as possible)
5. HEIGHT OF GRASS BEFORE CUTTING FOR SAMPLING (cm)
6. HEIGHT OF GRASS AFTER CUTTING FOR SAMPLING (cm)
7. GRASS SAMPLING AREA (m²)
(Whole area cut, from which grass sample is taken)
8. WEIGHT OF GRASS COLLECTED FROM SAMPLING AREA
_____ FRESH (g)
(Weight of freshly cut grass for sample from total sampling area taken immediately after cutting, at least 5 kg)
9. MAT SAMPLING AREA (cm²)
(Whole area from which mat sample is taken)
10. WEIGHT OF MAT SAMPLE COLLECTED FROM MAT SAMPLING AREA
_____ FRESH (g)
(Weight of freshly sampled mat from total mat sampling area taken immediately after sampling, at least 5 kg)
11. ROOT SOIL SAMPLING AREA (cm²)
(Whole area from which root soil sample is taken)
12. WEIGHT OF ROOT SOIL SAMPLE COLLECTED FROM ROOT SOIL SAMPLING AREA
_____ FRESH (g)
(Weight of freshly sampled root soil from the root soil)

GES FORM (Cont.)

INFORMATION ABOUT SENDER

COLLECTOR'S CODE

PERSON RESPONSIBLE

INSTITUTION

MAILING ADDRESS

TELEX

TELEFAX

PLACE OF COLLECTION

PERSON RESPONSIBLE FOR COLLECTION

INSTITUTION

ADDRESS

ALTITUDE

LONGITUDE

LATITUDE

REMARKS (if any):

Annex IX

GAMMA SPECTROMETRIC SYSTEMS

A. Food monitoring and environmental samples NaI gamma spectrometric system; stand-alone bench unit:

1. NaI detector (7.62 cm × 7.62 cm) including preamplifier, amplifier with gain control, high voltage supply (+), single channel analyser and ratemeter or included in one unit. Minimum resolution 8%.
2. Personal computer (PC) including hardware needed for operation of the system:

- 640-kB RAM
- 20-MB hard disc
- 1.2-MB floppy disc
- 360-kB floppy disc
- colour monitor, EGA card (graphics adapter),
- 10-MHz co-processor

3. One MCA card (2k), one ADC (2k) suitable for PCs including software package for food monitoring, e.g. system calibration and operation, peak search and net area determination, support for plotting, spectrum display.
4. Plotter compatible with the system.
5. Printer compatible with the system.
6. Lead shielding (5 cm wall) to house the detector including preamplifier as well as polyethylene bottles (1 L) and Marinelli beakers (1 L).
7. Radionuclide standards:

- Marinelli beaker (1 L) and polyethylene bottle (1 L) containing resin with spiked radionuclides for NaI measurement:

- Two Ba-133
- Two Cs-134
- Two Cs-137
- Two Cs-134/Cs-137 (optional)

B. High purity Ge spectroscopic multichannel analyser system for measurement of environmental radioactivity:

1. Detector:
High purity coaxial Ge detector (p-type) system with sealed preamplifier and high voltage filter. To be mounted in different configurations (vertical/horizontal) including appropriate cryostat (30 L) for installation in a suitable lead shielding for Marinelli beakers (1 L) as well as polyethylene bottles (1 L) and dipstick extension.

Specifications: Efficiency (1.33 MeV): 20% relative

Resolution: 2.0 keV

Peak (Compton): 45:1 or better

Automatic high voltage shut down

High voltage supply (bias); 5 kV

Spectroscopy amplifier (research grade for high resolution gamma spectrometry to include: active baseline restorer, pile-up rejector, shaping time 0.5–6 μ s or better, automatic pole zero adjustment)

BIN and power supply

Cables necessary to connect a pulse height analysis system and the various modules to the detector.

2. Multichannel analyser system:

Multichannel buffer with dual port memory, PC interface

ADC (8k memory)

Set of cables

PC including hardware needed for operation of the system (same as A.2).

3. Software:

MCA operating software

Spectra evaluation (peak search, net area calculation, etc.)

Radionuclide library

Quality assurance

Environmental analysis

4. Lead shielding (5 cm, copper lining) to house the detector, including pre-amplifier as well as Marinelli beakers (1 L) and polyethylene bottles (1 L), preferably top loading (e.g. inner diameter 250 mm, height 300 mm).

5. Printer compatible with the system.

6. Plotter compatible with the system.

7. Radionuclide standards:

(a) Mixed radionuclide solution covering the energy range 100–2000 keV;

(b) Marinelli beaker (1 L) and polyethylene bottle (1 L) containing spiked resin of radionuclides covering the same energy range as B.7(a) above; dimensions suited to the detector and the lead housing.

8. Sample containers:

— Marinelli beakers (1 L); dimensions suited to the detectors (NaI, Ge) and the lead housing.

— Polyethylene bottles (1 L) with screw caps; dimensions suited to the lead housing.

— Portable multichannel analyser (minimum memory 1024 channels) supporting a NaI detector (5.08 cm \times 5.08 cm) including data storage capability, high voltage supply, built in spectroscopy amplifier, serial RS 232, etc.

- Portable multichannel analyser (minimum memory 4096 channels) supporting a HP-Ge detector including data storage capability, high voltage supply, built in spectroscopy amplifier, serial RS 232, etc.
- Gamma gauge cryostat supplied with 5 L Dewar supporting a HP-Ge detector (see B.1 above).
- Precision tail pulse generator.
- Spare parts (diskettes, paper, set of cables, ribbon cartridges, etc.).

ANNEX X

Potential Suppliers of Calibration Sources and Reference Materials

This annex contains a list of potential suppliers of calibration sources and reference materials for radioactivity measurements. The list is based mainly on information provided by the Agency's Member States who were surveyed in 1988. Additional information from suppliers' catalogues and published reports is also included. Although the list is extensive, it may not be complete. Calibration sources are also available from a number of suppliers of nuclear counting instruments. These have not been included in the list, but the reader can contact the manufacturers of instruments of interest for further information. The list contains names and addresses of commercial suppliers as well as national institutions. Some of the larger commercial suppliers have branch offices or local representatives in many countries; further information should be available from the Head Offices of these suppliers.

Inclusion of a supplier in the list does not indicate an endorsement of that supplier by the Agency. The onus is on the reader to ascertain whether the products offered by a supplier are adequate for the purpose intended. To ensure accurate analysis of radioactivity, the standards used to calibrate the instruments must be traceable to a competent standards institution. The reader is cautioned further that radionuclides that are supplied as unstandardized sources have only a nominal activity value assigned to them. Since the nominal value usually differs markedly from the true value, such sources cannot be used for calibration purposes unless they are first calibrated by a competent standards institution.

The Agency assumes no responsibility for errors in the entries, nor for the completeness of the list, since the information was supplied by other parties.

AUSTRALIA

Amersham (Australia) Pty Ltd.,
28-36 Foveaux St. (8th Floor),
Sydney, NSW 2010

Australian Nuclear Science and Technology Organisation,
Private Mail Bag 1,
Menai, NSW 2234

Australian Radiation Laboratory,
Lower Plenty Road,
Yallambie, VIC 3085

AUSTRIA

International Atomic Energy Agency,¹
Analytical Quality Control Services,
P.O. Box 100,
A-1400 Vienna

Laborex GmbH,
Damböckgasse 4,
A-1060 Vienna

New England Nuclear GmbH,
Lasallestraße 2/20,
A-1020 Vienna

BELGIUM

Amersham Belgium NV,
Vorstsesteenweg 171-173,
Postbus 1,
B-1060 Brussels

Du Pont de Nemours (Belgium),
Biotechnology Systems Division,
Mercure Centre,
Raketstraat 100,
100 Rue de la Fusée,
B-1130 Brussels (Haren)

¹ Reference materials only.

BRAZIL

Comissão Nacional de Energia Nuclear,
Calibration of Radionuclides Division,
Rua General Severiano 90,
Botafogo,
22.294 Rio de Janeiro, RJ

Instituto de Pesquisas Energeticas e Nucleares,
COURP/AFN — Laboratorio de Metrologia Nuclear,
Cx. P. 11049, Pinheiros,
CEP 01000, São Paulo, SP

CANADA

Amersham Canada Ltd.,
505 Iroquois Shore Road,
Oakville,
Ontario L6H 2R3

Canada Centre for Mineral and Energy Technology,
555 Booth St.,
Ottawa, Ontario K1A 0G1

Chalk River Nuclear Laboratories,
Neutron and Solid State Physics Branch,
Chalk River,
Ontario K0J 1J0

EDA Instruments Inc.,
4 Thorncliffe Park Drive,
Toronto, Ontario M4H 1H1

CHINA

Institute for Radiation Protection,
P.O. Box 120,
Taiyuan, Shanxi

CZECHOSLOVAKIA

Chemapol,
Foreign Trade Co.,
Kodanská 46,
Prague 10

Ústav pro výzkum, výrobu a využití radioisotopů,
Radiová 1,
Prague 10

DENMARK

Amersham Denmark,
Abildgardsparken 2,
DK-3460 Birkerød

Du Pont Denmark,
Biotechnology Systems Division,
Park Allee 292,
DK-2605 Brøndby

FRANCE

Amersham France,
B.P. 144,
Avenue du Canada,
F-91944 Les Ulis Cedex

Du Pont de Nemours (France),
Biotechnology Systems Division,
137, rue de l'Université,
F-75334 Paris Cedex 07

Laboratoire de métrologie des rayonnements ionisants,
CEA/ORIS/DAMRI,
B.P. 21,
F-91190 Gif-sur-Yvette

Transnucléaire,
Rue Christophe Colomb,
F-75008 Paris

GERMAN DEMOCRATIC REPUBLIC

Amt für Standardisierung,
Messwesen und Warenprüfung,
Bereich Messwesen,
Fürstenwalder Damm 388,
Berlin 1162

GERMANY, FEDERAL REPUBLIC OF

Amersham Buchler GmbH,
Gieselweg 1,
D-3300 Braunschweig

Du Pont de Nemours GmbH,
Biotechnology Systems Division,
NEN Research Products,
Postfach 40 12 40,
D-6072 Dreieich

New England Nuclear GmbH,
Postfach 40 12 40,
D-6072 Dreieich

Physikalisch-Technische Bundesanstalt, Abt. 6,
Bundesallee 100,
Postfach 3345,
D-3300 Braunschweig

HUNGARY

Izinta Isotope Trading Enterprise,
P.O. Box 77,
Konkoly Thege Miklos ut 29/33,
H-1525 Budapest

National Office of Measures,
P.O. Box 19,
Németvölgyi ut 37/39,
H-1124 Budapest

Pharmatrade,
P.O. Box 126,
H-1808 Budapest

INDONESIA

National Atomic Energy Agency,
Centre for Standardization and Radiological Safety Research,
JL. Cinere Pasar Jumat Kotak,
P.O. Box 43 Kby,
Jakarta Selatan

ITALY

Amity Pg. SR2,
Via Mecenate 30/14,
I-20138 Milan

Du Pont de Nemours,
Via Niccolini 3/E,
I-50121 Florence

ENEA,
Laboratorio di Metrologia delle Radiazioni Ionizzati,
C.R.E. Casaccia C.P. 2400,
I-00100 Rome

JAPAN

Amersham Japan Ltd.,
Tokyo Toyama Kaikan,
1-3 Hakusan 5-chome,
Bunkyo-ku,
Tokyo 112

Japan Radioisotope Association,
28-45, 2-chome, Hon-Komagome,
Bunkyo-ku,
Tokyo 112

NETHERLANDS

Amersham Nederland BV,
Sporhaag 134,
Postbus 32,
NL-3990 DA Houten

Du Pont de Nemours (Nederland) BV,
Biotechnology Systems Division,
Postbus 2060,
NL-5202 CB's-Hertogenbosch

PANAMA

Departamento de Salud Radiológica,
Complejo Hospitalario Metropolitano,
Caja de Seguro Social,
Apartado 9732, Zona 4,
Panama

POLAND

Polatom,
Institute of Atomic Energy,
Foreign Trade Office,
PL-05-400 Otwock-Świerk

Institute of Atomic Energy,
Isotope Production and Reactor Centre,
PL-05-400 Otwock-Świerk

ROMANIA

Institute of Physics and Nuclear Engineering,
Radionuclide Metrology Group,
P.O. Box MG 6,
76900 Bucharest

SOUTH AFRICA

Radioactivity Standards and Radiation Safety Division,
National Acceleration Centre, CSIR,
P.O. Box 72,
Faure 7131

SWEDEN

Amersham Sweden AB,
Hagalundsgatan 30,
S-171 50 Solna

Du Pont Scandinavia AB,
Biotechnology Systems Division,
Torshamnsgatan 35-39,
S-163 86 Stockholm

SWITZERLAND

Du Pont de Nemours International SA,
Biotechnology Systems Division,
Pumpwerkstr. 15,
CH-8105 Regensdorf

UNION OF SOVIET SOCIALIST REPUBLICS

All-Union Association "Isotop",
Pogodinskaja ul. 22,
119435 Moscow

Techsnabexport,
121200 Moscow

UNITED KINGDOM

Amersham International plc,
UK Sales Office,
Lincoln Place, Green End,
Aylesbury, Bucks

Du Pont (UK),
Biotechnology Systems Division,
Wedgwood Way, Stevenage,
Herts SG1 4QN

National Physical Laboratory,
Division of Radiation Science and Acoustics,
Department of Trade and Industry,
Teddington, Mdx TW11 0LW

National Physical Laboratory,
Office of Reference Materials,
Teddington, Mdx TW11 0LW

Nuclear Enterprises Ltd,
Bath Road,
Reading, Berks R67 PR

UNITED STATES OF AMERICA

Amersham Corp.,
2636 Clearbrook Drive,
Arlington Heights, IL 60005

Dupont NEN Research Products,
331 Treble Cove Road,
North Billerica, MA 01862

National Oceanic and Atmospheric Administration,
Catalog of Standard and Reference Materials for Marine Science,
US Department of Commerce,
Rockville, MD 20857

New England Nuclear,
575 Albany Street,
Boston, MA 03118

Oak Ridge National Laboratory,
Martin Marietta Energy Systems, Inc.,
Isotope Distribution Office,
P.O. Box 2008-6015,
Oak Ridge, TN 37831

Thermo Electron,
P.O. Box 3874,
7021 Pan America NE,
Albuquerque, NM 87190

SUPPLIERS OF CALIBRATION SOURCES FOR RADIOACTIVITY MEASUREMENTS

Analytics Inc.,
1094 Hemphill Avenue, NW,
Atlanta, GA 30318

Isotope Products,
1800 North Keystone St.,
Burbank, CA 91504

National Bureau of Standards,
Radioactivity Group,
Center for Radiation Research (Bldg 245),
Gaithersburg, MD 20899

SUPPLIERS OF NATURAL MATRIX REFERENCE MATERIALS

Environmental Measurements Laboratory,
Analytical Chemistry Division,
US Department of Energy,
376 Hudson St.,
New York, NY 10014-3621

National Bureau of Standards,
Office of Standard Reference Materials,
Room B311, Chemistry Building,
Gaithersburg, MD 20899

New Brunswick Laboratory (Bldg 350),
Chicago Operations Office,
US Department of Energy,
9800 South Cass Avenue,
Argonne, IL 60439

Nuclear Radiation Assessment Division,
US Environmental Protection Agency,
P.O. Box 93478,
Las Vegas, NV 89193-3478

National Bureau of Standards,
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Center for Radiation Research (Bldg 245),
Gaithersburg, MD 20899

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