

Determination of Radioactivity in Water by Multinuclide Gamma Ray Spectrometry 1989

Methods for the Examination of Waters and Associated Materials

This document
contains 47 pages

Determination of Radioactivity in Water by Multinuclide Gamma Ray Spectrometry 1989

Methods for the Examination of Waters and Associated Materials

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About This Series

This booklet is part of a series intended to provide both recommended methods for the determination of water quality, and in addition, short reviews of the more important analytical techniques of interest to the water and sewage industries.

In the past, the Department of the Environment and its predecessors, in collaboration with various learned societies, have issued volumes of methods for the analysis of water and sewage culminating in 'Analysis of Raw, Potable and Waste Waters'. These volumes inevitably took some years to prepare, so that they were often partially out of date before they appeared in print. The present series will be published as series of booklets on single or related topics; thus allowing for the replacement or addition of methods as quickly as possible without need of waiting for the next edition. The rate of publication will also be related to the urgency of requirement for that particular method, tentative methods and notes being issued when necessary.

The aim is to provide as complete and up to date a collection of methods and reviews as is practicable, which will, as far as possible, take into account the analytical facilities available in different parts of the Kingdom, and the quality criteria of interest to those responsible for the various aspects of the water cycle. Because both needs and equipment vary widely, where necessary, a selection of methods may be recommended for a single determinand. It will be the responsibility of the users—the senior technical staff to decide which of these methods to use for the determination in hand. Whilst the attention of the user is drawn to any special known hazards which may occur with the use of any particular method, responsibility for proper supervision and the provision of safe working conditions must remain with the user.

The preparation of this series and its continuous revision is the responsibility of the Standing Committee

of Analysts (to review Standard Methods for Quality Control of the Water Cycle). The Standing Committee of Analysts is a committee of the Department of the Environment set up in 1972. Currently it has 9 Working Groups, each responsible for one section or aspect of water cycle quality analysis. They are as follows:

- 1.0 General principles of sampling and accuracy of results
- 2.0 Microbiological methods
- 3.0 Empirical and physical methods
- 4.0 Metals and metalloids
- 5.0 General nonmetallic substances
- 6.0 Organic impurities
- 7.0 Biological monitoring
- 8.0 Sewage Works Control Methods
- 9.0 Radiochemical methods

The actual methods and reviews are produced by smaller panels of experts in the appropriate field, under the overall supervision of the appropriate working group and the main committee.

The names of those associated with this method are listed inside the back cover. Publication of new or revised methods will be notified to the technical press, whilst a list of Methods in Print is given in the current HMSO Sectional Publication List No 5.

Whilst an effort is made to prevent errors from occurring in the published text, a few errors have been found in booklets in this series. Correction notes and minor additions to published booklets not warranting a new booklet in this series will be issued periodically as the need arises. Should an error be found affecting the operation of a method, the true sense not being obvious, or an error in the printed text to be discovered prior to sale, a separate correction note will be issued for inclusion in that booklet.

L R PITTWELL
Secretary and Chairman

11 August 1988

Warning to Users

The analytical procedures given in this booklet should only be carried out by competent trained persons, with adequate supervision when necessary.

Local Safety Regulations must be observed.

Laboratory procedures should be carried out only in properly equipped laboratories.

Field Operations should be conducted with due regard to possible local hazards, and portable safety equipment should be carried.

Care should be taken against creating hazards for one's self, one's colleagues, those outside the laboratory or work place, or subsequently for maintenance or waste disposal workers. Where the Committee have considered that a special unusual hazard exists, attention has been drawn to this in the text so that additional care might be taken beyond that which should be exercised at all times when carrying out analytical procedures. Reagents of adequate purity must be used, along with properly maintained apparatus and equipment of correct specifications. Specifications for reagents, apparatus and equipment are given in manufacturers' catalogues and various published standards. If contamination is suspected, reagent purity should be checked before use. Lone working, whether in the laboratory or field, should be discouraged.

The best safeguard is a thorough consideration of hazards and the consequent safety precautions and remedies well in advance. Without intending to give a complete check-list, points that experience has shown are often forgotten include: laboratory tidiness, stray radiation leaks (including ultra violet), use of correct protective clothing and goggles, removal of toxic fumes and wastes, containment in the event of breakage, access to taps, escape routes, and the accessibility of the correct and properly maintained first-aid, fire-fighting, and rescue equipment. Hazardous reagents

and solutions should always be stored in plain sight and below face level. Attention should also be given to potential vapour and fire risks. If in doubt, it is safer to assume that the hazard may exist and take reasonable precautions, rather than to assume that no hazard exists until proved otherwise.

There are numerous handbooks on first aid and laboratory safety. Among such publications are: 'Guide to Safe Practices in Chemical Laboratories' and 'Hazards in the Chemical Laboratory', issued by the Royal Society of Chemistry, London; 'Safety in Biological Laboratories' (Editors Hartree and Booth), Biochemical Society Special Publication No 5, The Biochemical Society, London, which includes biological hazards; and 'The Prevention of Laboratory Acquired Infection', Public Health Laboratory Service Monograph 6, HMSO, London.

It cannot be too strongly emphasised that prompt first aid, decontamination, or administration of the correct antidote can save life; but that incorrect treatment can make matters worse. It is suggested that both supervisors and operators be familiar with emergency procedures before starting even a slightly hazardous operation, and that doctors consulted after any accident involving chemical contamination, ingestion, or inhalation, be made familiar with the chemical nature of the injury, as some chemical injuries require specialist treatment not normally encountered by most doctors. Similar warning should be given if a biological or radio-chemical injury is suspected. Some very unusual parasites, viruses and other micro-organisms are occasionally encountered in samples and when sampling in the field. In the latter case, all equipment including footwear should be disinfected by appropriate methods if contamination is suspected. If an ambulance is called or a hospital notified of an incoming patient give information on the type of injury, especially if poisoning is suspected, as the patient may be taken directly to a specialized hospital.

Determination of Radioactivity in Waters by Multinuclide Gamma Ray Spectrometry

1. Introduction

All natural waters contain small amounts of radioactivity. These arise chiefly from the decay of natural radionuclides and their decay products. Since the early 1940s the naturally-occurring radioactivity has been supplemented by a number of man-made radionuclides which have entered the hydro-sphere as fall-out from nuclear weapons testing, as low-level radioactive waste discharges from the operations of industry including medical and research applications and from nuclear accidents such as that at Chernobyl in 1986.

In the United Kingdom the controlled discharges of low-level aqueous radioactive waste to the environment are made in accordance with authorizations or agreements issued under the Radioactive Substances Act (1960) (1) and EEC regulations relating to the disposal of radioactive wastes. Among other things, these aim to ensure that the levels of radionuclides do not exceed specified limits derived from the recommendations of the International Commission on Radiological Protection (ICRP) which are incorporated in UK national policy(2). The operators authorized to discharge radioactive waste are, where appropriate, required to measure the radioactive content of waters into which they discharge, and Authorizing Government Departments carry out their own check monitoring programmes to ensure compliance with the regulations. Her Majesty's Inspectorate of Pollution (HMIP), Department of the Environment (DOE) and some UK Water Authorities carry out checks on the levels of radioactivity in drinking water supplies, and, as a result of the Chernobyl nuclear accident, a number of Local Authorities have found it helpful to conduct some environmental monitoring of radioactivity levels in their areas.

2. The Measurement of Radioactivity

A previous publication in this series discussed the measurement of radioactivity in water and described, in detail, screening methods for the determination of gross alpha and beta activity(3). Many radionuclides emit gamma photons during decay; these may be detected, identified and quantified using crystals such as (a) sodium iodide activated with thallium (NaI(Tl)) which emit pulses of light (scintillate) when they absorb the energy from gamma photons or (b) detectors made from germanium whose electrical conductivity increase when they absorb the energy of the gamma photons.

Unlike alpha and beta particles, gamma rays interact to a very limited extent with the matrix of most environmental materials. Furthermore, because the primary photons are emitted at discrete energies and are characteristic of the radionuclide from which they arise, they are amenable to multi-radionuclide spectrometric analysis. Gamma spectrometry is thus a very powerful radio-analytical technique and those unfamiliar with the method are recommended to read one or more of the many publications which provide an introduction to the subject(4,5,6,29).

Gamma-emitting radionuclides in the aqueous environment typically include the cosmogenic and other naturally-occurring radionuclides including ^7Be , ^{40}K and the decay products of the uranium and thorium series, plus man-made radionuclides such as ^{54}Mn , ^{60}Co , ^{65}Zn , $^{95}\text{Zr}/^{95}\text{Nb}$, $^{106}\text{Ru}/^{106}\text{Rh}$, $^{134}/^{137}\text{Cs}$, ^{144}Ce , ^{144}Pr , $^{152}/^{154}/^{155}\text{Eu}$ and ^{241}Am . A more complete list of gamma-emitting radionuclides is given in Section 14 and for a complete listing of nuclear data reference should be made to ICRP report number 38(7).

3. Quantities, Symbols and Units

Given below are the quantities, symbols and units and abbreviations as used in this document.

Quantity	Symbol	Unit	Abbreviation
Activity	A	becquerel	Bq
Activity at time t	A(t)	becquerel	Bq
Volume	V	litres	L
Activity concentration	C	becquerel per litre	Bq L ⁻¹
Half-life	t _½	seconds	s
Decay constant*	λ	reciprocal seconds	s ⁻¹
Counting efficiency	ε	counts per becquerel	Cts Bq ⁻¹
Transition probability (abundance)	P _E	fractional proportion	
Counts per second	cps	reciprocal seconds	s ⁻¹
Identifying subscripts:			
Radionuclide	i		
Count	c		
Reference source	r		
Peak	p		
Background	b		

$$*\text{Decay constant } \lambda = \frac{\ln 2}{t_{1/2}} = \frac{0.693}{t_{1/2}}$$

4. Hazards

Two distinct types of hazard need to be considered in the determination of radioactivity by gamma spectrometric procedures—hazards to the operator and hazards to the equipment.

4.1 Hazards to the operator

Proper attention must be paid to the handling of radioactive substances such as radionuclide standards and samples. Inhalation of particulates from dry solids must be avoided. Strict controls are laid down in the Ionizing Radiations Regulations (1985) and the Radioactive Substances Act (1960) which are enforced by the Health and Safety Executive (HSE) and Her Majesty's Inspectorate of Pollution (HMIP) respectively(8). Further information on regulatory control is also contained in another booklet in this series(9).

High voltages are employed in parts of the equipment used for gamma spectrometry; consult manufacturer's instructions.

Liquid nitrogen is used as a coolant (BP – 196°C). Appropriate protective clothing including specialist gloves and full face protection must be worn when handling this material, and adequate ventilation must be provided(10). Care is also needed to ensure that ice does not block either the filling tube or the exhaust vent of the detector cryostat; bursting can result from blockage.

4.2 Hazards to equipment

Germanium detectors must be cooled with liquid nitrogen for some 6 hours before applying bias and high voltages. Care must be taken to ensure that the polarity of the high voltage is correct before switching on, otherwise **irreversible damage** to the detector will occur. The high voltage must be applied by increasing slowly over a period of several minutes.

High voltage supplies must be switched off if the detector starts to warm up. Automatic warning devices are available for this purpose and many detectors are now supplied with in-built warning devices and automatic cutouts. (Consult manufacturer's literature).

Samples must be enclosed to avoid spillage onto the detector.

It is advisable to cover the detector with a thin plastic film to protect it from contamination. An exposed preamplifier must not however be covered or it may be damaged by condensation.

5. Characteristics of the Method

5.1 Scope and Field of Application

The method permits the simultaneous determination of the many environmentally important radionuclides which emit gamma rays during decay. It is intended to provide guidance for monitoring liquid effluents from nuclear establishments, hospitals or research laboratories handling radioactive substances and the aquatic environment for the presence of radionuclides whether the source be natural, authorized, or accidental releases. Use of this method permits the concentrations of appropriate radionuclides in water samples to be checked. A list of the more important gamma-emitting nuclides likely to be encountered is given in Section 14 and a typical gamma spectrum from an environmental sample using a Ge detector is shown in Figure 1.

Radionuclides found in environmental materials emit gamma rays with energies from a few keV (kilo-electron volts) to a few MeV (million-electron volts), however it is usual to limit the range of a gamma spectrometer to cover energies from 40 keV to 2 MeV. If a lower energy range is to be measured then an alternative type of detector is usually employed (silicon or low energy germanium types), but it should be pointed out that at these low energies photon attenuation within the sample can be a severe problem and unequivocal identification of photopeaks becomes a skilled operation. Additional expertise must therefore be established if analyses of gamma ray energies below 40 keV are to be carried out.

The method is applicable to samples in which there is no suspended particulate matter, or in which any solids present will remain suspended in a fixed geometry throughout the measurement period. For other samples, such as fluid sludges, mechanical stirring may provide constant geometry. If this is not practicable, filtration or centrifugation to separate the liquid and solid phases should be carried out and the two presented separately to the detector for measurement(14).

If the concentrations of the radionuclides of interest in the sample are too low for satisfactory assay, then consideration can be given in appropriate cases to the use of larger volume equivalents by concentrating the sample by evaporation prior to gamma spectrometric measurement. Care is however needed to ensure that volatile components such as radio-iodine and radio-ruthenium are not lost during the concentration step. Section 9 gives further guidance for this procedure and discusses the use of stable element carriers to stabilize samples during storage.

5.2 Principal

The technique of gamma spectrometry relies on the ability of the radiation detector material to respond to a gamma photon by generating a signal, the magnitude of which is proportional to the energy of the photon. The material to be examined is usually placed on, in, or around the detector: both sample and detector are enclosed in a lead shield to reduce background radiation. Counts are accumulated for an appropriate length of time in order to establish a statistically valid number of counts in the channels relating to the radionuclides of interest. The concentrations of individual radionuclides are calculated by comparison with accredited standards counted in the same geometry or by using a derived equation of detector efficiency.

5.3 Performance Characteristics

The performance of the method is dependent upon a number of factors. The more important of these include:

- (1) the efficiency of the detector, its resolution and its peak to Compton ratio as discussed in (4) and defined in (15). See also fig 4.
- (2) the same volume, the geometry in which it is presented to the detector and the nature of the radionuclide matrix
- (3) the length of time for which the sample is counted and the adequacy of background shielding. (See Section 7.1)

Thus, no single statement of method performance for any given radionuclide or combination of radionuclides is possible. Table 1 provides an indication of the capabilities of equipment in use at three laboratories when applied to the analysis of the liquid effluent discharges from three nuclear power stations. Differences in the detection limits quoted for the nine radionuclides at the three laboratories reflect differences in relative detector efficiency (20–35%), sample volume (100 mL or 250 mL) and counting times (55,000 seconds to 250,000 seconds).

5.4 Limit of Detection

The limit of detection, like the method performance, depends upon detector characteristics, sample presentation and counting time. It is currently accepted that the most realistic assessment of the limit of detection in gamma spectrometry is as described by Currie (1968) (Ref. 28) (See also Section 12.2.5 and the Appendix).

The Currie procedure has therefore been adopted to produce the following data for a typical present day detector assembly:—

Detector	— High Purity Germanium
Relative efficiency	— 42 per cent
Peak to Compton ratio	— 65:1
Resolution at 1.33MeV	— 1.90 KeV at FWHM
Volume of sample	— 250 mL in 100 mm diameter container
Counting time	— 55,000 seconds

Nuclide	Detection Limit (Bq.L ⁻¹)
⁶⁰ Co	1
¹³⁴ Cs	1
¹³⁷ Cs	1

In a multinuclide mixture, as is usually the case in environmental samples, a more appropriate assessment is given by the minimum Detectable Activity (MDA), fully described in the Appendix

FWHM = Full Width Half Maximum

6 Reagents

Analytical grade reagents are, in general, a minimum requirement.

6.1 Water

The water used for blank determinations in the method itself and in the preparation for other reagents should have a radioactive content which is low compared to that likely to be encountered in any sample.

6.2 Cobalt-60 setting up source

Any source, solid or liquid, should give a dead-time no greater than about 5% (See Section 7.3).

6.3 Single Peak Source (Caesium -137)

Any source, solid or liquid, should give a dead-time no greater than about 5%.

6.4 Mixed Gamma Calibration Standard (Liquid)

It is essential that the calibration of standards be traceable to a national or international measurement standard and certificates issued by the supplier should provide supporting information and data. The National Physical Laboratory is the UK's national standards Laboratory for all physical quantities. Suitable mixed radionuclide standards are obtainable from Amersham International plc (current catalogue number QCY 48) or the National Physical Laboratory. A recently available low level mixed radionuclide NPL standard solution contains Am^{241} , Hg^{203} , Ce^{139} , Cs^{137} , Sn^{113} , Cd^{109} , Y^{88} , Sr^{85} , Co^{60} and Co^{57} . In either case, these will normally need diluting to provide a diluent of suitable composition for the required geometry for calibration. If the correct diluent is not used, some of the radionuclides contained in the standard will almost certainly come out of solution. For diluting the QCY 48, a suitable inactive solution N441 is available from Amersham International. This contains 225 ug ml^{-1} each of Cd, Cs, Ce, Co, Hg, Sn, Sr, and Y in 4M HCl. For diluting the NPL standard the user is advised to contact NPL for information. As with the 'setting up' and 'single peak' sources, the total activity level in the calibration standard solution should be such that it does not lead to a dead-time in the detector system of more than about 5%.

6.4.1 Single radionuclides which emit well documented photo peaks over a range of energies can be used as an alternative. These too must be traceable.

6.5 Carrier Solution for Sample Storage

Details of a suitable carrier solution for stabilizing samples during storage and its preparation is given in Section 9.2.

7 Apparatus

A gamma spectrometer comprises a detector encased in a thin cylindrical metal container with ancilliary equipment. The detector requires high voltage and bias voltage supplies; the signal from the detector is fed via a linear amplifier and pulse shaping electronics to a multi-channel pulse height analyser or micro-processor with display screen, printer and data storage facilities (see Figure 2).

For many years the most common type of detector used in gamma-spectrometry was the thallium activated sodium iodide crystal (NaI(Tl)). Such detectors had the advantage of high efficiency, but suffered from poor resolution and temperature control problems which adversely affected spectral quality. Many of the problems have been overcome with the advent of semi-conductor detectors, initially lithium drifted Ge detectors (Ge/Li), and today hyperpure intrinsic Ge detectors (HPGe), albeit at some loss of relative efficiency. Figure 3 compares the spectrum of Cobalt 60 as measured on the two types of detector.

The method discussed in this booklet considers only the use of intrinsic germanium detectors covering the energy range 40 keV to 2 MeV. Other semi-conductors are available which provide better detection characteristics below 40 keV. Similarly, the use of hard-wired multi-channel analysers (MCA) will not be discussed in detail as it is considered that modern micro-processor controlled systems are readily available, more versatile and no more expensive.

7.1 The detector and shield assembly

A typical specification of a Ge detector would be as follows:

- (a) Energy range 40 keV-2 MeV
- (b) Energy resolution Approximately 1.0 keV at 122 keV and 2.0 keV at 1.33 MeV
at full-width, half maximum (FWHM)
(see E in Figure 4)
- (c) Relative efficiency* 5-75%
*(Relative to a NaI(Tl) crystal under standard conditions described in ANSI/IEE 325 (15).)
- (d) Peak to Compton ratio >50:1 for the ^{60}Co peak at 1.33 MeV. (15)

Note Detectors can be obtained in several forms including planar, coaxial and well configuration. The nature and size of the samples to be measured and the information required from them determines which type and configuration will be most advantageous. Closed end coaxial detectors are popular at the present time, but the development of new detectors is still taking place and up to date information should be sought from manufacturers by prospective purchasers.

The high purity Ge detector is encased in a sealed, evacuated metal cylinder. The vacuum is maintained by a molecular sieve and the whole unit must be held at liquid nitrogen temperature whilst the high voltage is applied. Low temperature operation is necessary because at ambient temperatures thermal noise in the detector is considerably greater than the primary signal; low temperature operation allows a satisfactory signal to noise ratio to be achieved. The high vacuum conditions around the crystal are designed to exclude contaminants, and maintain the intrinsic purity of the detector. Figure 5 shows a typical detector/cryostat assembly.

Both the detector and the sample being counted need to be adequately shielded from background radiation. This is achieved by enclosing both in a 'castle' of heavy metal (normally not less than 100 mm of lead). This shield is designed to attenuate terrestrial and cosmic radiation and where low-level environmental samples are to be counted, particular care is necessary. For such work old (pre-nuclear age) lead may be better. If work below 100 keV is contemplated, steps must be taken to prevent fluorescence X-rays and Bremsstrahlung (16) produced in the lead from reaching the detector. A graded shield built inside the lead castle is used for this purpose and comprises, for example, layers of cadmium, copper and perspex of optimized thickness with the cadmium layer next to the lead and the perspex nearest to the detector.

Careful consideration should also be given to the construction materials, ventilation and air filtration of the room in which the detector assembly is housed to minimize the effect of natural radiation—chiefly radon and its daughters. Radon can creep, and diurnal and weather variation may also occur.

In operation, a high voltage (of the correct polarity) is applied to the crystal and when photons interact with the active region of the crystal charge differences are created. These are sensed by a preamplifier.

It should be noted that the detector is not uniformly efficient over the energy range 40 keV to 2 MeV, peak response occurs at about 100 keV and then gradually reduces as the energy increases (Figure 6).

7.2 Preamplifier

The detector can be thought of as a capacitor into which a charge is deposited. The primary purpose of the preamplifier is to collect and integrate this charge and, with the aid of an applied bias voltage, the preamplifier produces an output voltage pulse that is proportional to the input charge (and hence the energy of the photon).

The preamplifier is located as close to the detector as possible to minimize the effect of stray capacitance. This is one of the most critical components of the whole assembly, because both noise levels and energy resolution are affected by the

characteristics of the preamplifier. This unit therefore determines to a large extent the quality of the entire measuring system.

An equally important function of the preamplifier is to serve as a match between the high impedance of the detector and the low input impedance of the main amplifier. Cables and connectors with the correct impedance (typically 50, 75 or 93 ohms) must be used.

Preamplifiers are normally factory-set and require no adjustment by the user.

7.3 Main Amplifier

For practical reasons the main amplifier and other associated electronics may need to be located some distance away from the preamplifier and detector.

The function of the main amplifier is to shape and amplify the pulses arising from the pre-amplifier for optimal analogue to digital conversion. To this end there may be controls to enable the operator to adjust the gain (amplification), pulse shaping (time constant), pulse tailing (pole zero) and pulse DC level (usually termed a DC restorer). To obtain optimum energy resolving performance from the system these latter two pulse shaping controls must be adjusted correctly; these can be readily set with the aid of an oscilloscope. The pulse should be set as shown in Figure 7d, the Pole Zero will affect the under- and overshoot of the pulse, the DC level will affect the pulse base line level.

The gain controls are set in conjunction with a suitable calibration source such that the full energy range of interest is accommodated within the horizontal energy scale of the MCA. The gain controls should be set so that a suitable interval, compatible with the energy per channel chosen, exists between two well defined photopeaks. The absolute position of the peaks along the MCA axis can be adjusted by the zero control on the analogue to digital converter (ADC).

The amplifier should provide a 'dead time' signal for timing purposes. Dead time occurs in an amplifier because it cannot accept further pulses whilst it is analysing a previous one. The actual time taken to do this depends upon the height of the pulse. An electronically imposed dead time is applied to enable correction to be made. Any pulse which arrives during this processing period may be lost because the analyser is effectively 'dead'.

The period of time during which the amplifier is capable of responding to pulses is known as the 'live time'. It should be clearly noted that this live time differs from the 'real' or clock time according to the following relationship:

$$\text{Live time} + \text{Dead time} = \text{Real time.}$$

Where especially high counting rates are anticipated (say, > 50,000 cps), then a gated biased amplifier may be needed.

7.4 Analogue to digital converter

An analogue to digital converter (ADC) is required to convert the analogue output signal from the main amplifier into a digitized form suitable for feeding into the subsequent spectral analysis equipment.

There are two main types of ADC: (1) the Wilkinson type which digitizes a pulse by charging a capacitor to the amplitude of the input pulse and then discharging the capacitor at a constant rate; (2) the successive approximation ADC where a succession of comparators is used to successively re-estimate the pulse amplitude to the required precision. The latter is inherently faster and therefore more commonly found in systems designed for high count rates. Both output a digital number which corresponds to the input voltage level, and normally also give associated timing signals.

7.5 Multi-channel analyser

The MCA performs the essential functions of collecting the spectral data and timing signals, providing a visual monitor and producing an output either of raw data for

subsequent analysis, or a calculated result. MCAs may be grouped into several categories (details of which may be obtained from appropriate manufacturers) but all 'hard-wired' analysers tend to have limited data handling capabilities.

An alternative to the hard-wired MCA, called a multi-channel buffer (MCB), is now available. This type of unit, when used in conjunction with relatively cheap personal computers (PC), can emulate quite sophisticated MCAs.

Manufacturers now offer an increasing variety of hardware and software packages for gamma spectrometry. These include a detector and its associated cryostat, shielding and electronics, an analyser and spectrum interpretation computer programme with storage and data print-out facility. Such dedicated systems can save much time and effort in choosing and matching the necessary component parts and assembling them into a reliable, working system. They do not however avoid the need to train skilled operators to run the equipment and interpret the gamma spectrometric data produced—a typical one-day 'familiarization with the equipment' course does not constitute 'training'!

7.6 Computer

The facilities available can range from the very simple to the very complex. The computer should ideally be capable of carrying out the following:

1. read the data from the MCA or MCB;
2. reproduce the data on a screen, plotter or printer and store them;
3. determine the relationship between channel number and energy over the entire energy range by using data obtained from appropriate reference sources;
4. determine the energy-dependent counting efficiency over the entire energy range to be studied by making use of the appropriate reference source;
5. store 'background' data to enable correction procedures to be applied—see Figure 8;
6. detect peaks, determine the characteristics of the detected peaks such as the centroid, the FWHM, the number of net counts under the peak and determine the uncertainty associated with this number;
7. recognize interfering peaks and apply appropriate separation procedures; deconvolute if applicable;
8. identify the radionuclides responsible for the observed photopeaks by making use of radionuclide libraries;
9. allow the user to form a library of radionuclides;
10. inform the user about the presence of unidentified peaks;
11. calculate the activity and the activity concentrations of the respective radionuclides based on the number of counts, the count live-time, the counting efficiency, the data in the radionuclide libraries and the sample weight, mass or volume in the chosen units;
12. calculate the standard deviation of the activity of the identified radionuclides or a minimum detectable activity (MDA) or Limit of Detection (LD) of radionuclides sought but not found in the sample and report in the units chosen.

7.7 For Additional Information on Testing Apparatus see References 30 a, b, c.

8 Setting-up Procedure

Gamma spectrometry is a straightforward analytical procedure which allows many different types of material to be assayed in a wide variety of configurations provided that due consideration is given to calibration for material and geometry. The interpretation of gamma spectra from environmental samples, on the other hand, is complex and even computer-calculated results require interpretation by a skilled analyst.

The following procedures, which are designed for optimizing the equipment performance are presented as a guide and to assist understanding of the procedure. Reference

to manufacturers' manuals is essential when carrying out work of this type. Technical improvements may obviate many of these steps.

Step	Procedure	Notes
8.1	Optimizing of equipment	
8.1.1	The detector should be established in a shield (see 7.1) in such a manner that it is electrically and mechanically isolated from the shield (note a)	(a) This is to avoid earth loops
8.1.2	Ensure the detector is mounted on supports designed to dampen vibrations	
8.1.3	Ensure that the liquid nitrogen filling tube is accessible at all times and so fixed as to prevent disturbance of the detector when topping up	
8.1.4	Connect cables to preamplifier, amplifier, etc in accordance with manufacturer's instructions. Ensure that all BNC cables have the same impedance	
8.1.5	Fill the cryostat with liquid nitrogen and leave for 6 hours to stabilize	
8.1.6	Check the bias supply is set to the correct positive or negative bias polarity. With the detector bias OFF and set at zero (0) volts, switch on the high voltage. Set the amplifier coarse gain to 50 and the shaping time to that recommended for the detector.	
8.1.7	Using a 'tee' connector, couple the amplifier unipolar output to the input of the MCA/MCB and to an oscilloscope using BNC co-axial cables (note b).	(b) The oscilloscope must be DC coupled and contribute no distortion to the waveforms
8.1.8	Increase the bias supply voltage slowly and continue to increase the voltage over a period of a few minutes until the recommended operating voltage is reached (note c)	(c) signals should be received on the MCA/MCB and the scope. If there is no signal check that the correct connections have been made. After checking, if there is still no signal, consult the manufacturer
8.1.9	Leave the system to stabilize for approximately 1 hour before continuing with the set-up procedures	
8.1.10	Place a cobalt-60 source close to the detector and, using the gain adjustment on the amplifier, adjust the energy response to give a channel reading of about 2665 for the 1332 keV peak (note d)	(d) Channel 2665 is appropriate for a 4096 channel system. For systems of smaller or greater memory capacity the appropriate channel should be selected
8.1.11	Remove the radioactive source from the vicinity of the detector before proceeding with the baseline checks	
	If the amplifier has a factory-set DC baseline proceed to 8.3	

Step	Procedure	Notes
8.2	DC base line adjustment	
8.2.1	Disconnect the cable between the amplifier and the preamplifier and adjust the DC potentiometer on the amplifier until the DC level is equal to or slightly more negative than the ground level (note e)	(e) There should be a horizontal trace on the oscilloscope showing the ground level for the system
8.2.2	Reconnect the amplifier to preamplifier cable	
8.3	Base line noise check	
8.3.1	Adjust the oscilloscope controls such that the baseline signal can be observed in detail (note f)	(f) There should be no obvious signals on the baseline display and the width should not exceed 5 mV
8.3.2	Check the detector for phonic response by clapping hands nearby with shield open and closed	
8.3.3	Minimize noise effects by adjusting the baseline If the amplifier has an automatic Pole Zero adjustment, follow the manufacturers' instructions. Then proceed to 8.6.	
8.4	Pole zero adjustment	
8.4.1	Place the cobalt-60 source (6.2) close to the detector	
8.4.2	Observe the unipolar output from the amplifier with the oscilloscope	
8.4.3	Adjust the pole-zero potentiometer control so that the trailing edge of the pulses returns to the baseline and thereby minimize over- and under-shoot (note g)	(g) For examples of overshoot and undershoot see Figure 7b and c
8.5	Base line restorer threshold adjustment After the amplifier gain and shaping time have been selected and the Pole-zero adjustment made, the Baseline Restorer Threshold adjustment can be used to establish the correct discriminator threshold for the baseline restorer circuit. For most applications the toggle switch can be set in the Auto position where the threshold level is set automatically just above the noise level. However, if desired the threshold can be set manually as follows:	
8.5.1	Remove the radioactive source from the vicinity of the detector	
8.5.2	Set the Baseline Restorer (BLR) switch on the amplifier to the Threshold position and adjust the potentiometer fully clockwise	

Step	Procedure	Notes
8.5.3	Observe the unipolar output on the oscilloscope using a 100 mV/cm vertical scale and 5 micro-second horizontal deflection	
8.5.4	Connect the 'busy' output on the amplifier to the external trigger input on the oscilloscope	
8.5.5	Adjust the BLR potentiometer until the baseline discriminator begins to trigger on noise (h)	(h) This corresponds to about 200 counts per second from the 'busy' output
8.5.6	Adjust the BLR potentiometer until the trigger level gives a horizontal response on the oscilloscope (note i)	(i) For the correct oscilloscope output refer to Figure 7d
8.5.7	Switch the BLR toggle to the automatic mode and observe whether the automatic mode can cope to the same degree as the manual settings	
8.5.8	Disconnect the cable from the busy output and the 'tee' from the unipolar output of the amplifier	
8.5.9	Reconnect the signal cable from the MCA/MCB to the unipolar output of the amplifier	
8.5.10	The system is now ready for the initial MCA/MCB tests. Step 8.6	
8.6	Initial MCA/MCB tests	
8.6.1	Acquire a spectrum using a single peak source, eg caesium-137 (note j)	(j) Ensure that the peak is a symmetrical singlet and not skewed. Non-symmetry of the peak is the product of poor pole zero adjustment. A noisy baseline will affect the resolution creating peak broadening.
8.6.2	If the peak is skewed, and manual adjustment of pole zero is available then repeat the set-up procedure steps 8.4.1 to 8.5.7. In the case of automatic pole zero adjustment consult the supplier.	
8.6.3	Acquire a spectrum using a mixed gamma calibration standard (note k)	(k) If the gain settings are correct then the caesium-137 peak at 661.6 keV should be at channel 1324 and the 1332 keV peak of cobalt-60 at channel 2664 for a 4096 channel system. See Section 7.3, third paragraph.
8.6.4	The system is now ready for calibration: Step 11.1	

9 Sampling, Pretreatment and Storage

9.1 Sampling

It is important to note that the sample be adequately representative of the whole medium being sampled. General principles for sample collection are discussed in the various parts of ISO 5667 (11), while recommendations specific to the measurement of radioactivity in water are given in 'Methodology in Environmental Radionuclide Analysis' (13). See also General Principles of Sampling 2nd Edition with Supplements (12), especially the advice on sampling for radioactivity measurement in the Sampling Precipitation supplement.

Samples must be homogenous when presented to the detector for gamma spectrometry. The handling of waters containing suspended particulate matter is dealt with in section 5.1.

9.2 Treatment for Storage

In many cases where the sample does not contain radionuclides with short half lives, an appreciable time can elapse between collection and measurement of samples. It is essential that, during this time, the radionuclides of interest are not removed from the sample by physical processes such as adsorption on the collection vessel walls, precipitation, etc. Addition of a carrier solution containing non-radioactive isotopes or analogues can help to keep those radionuclides of interest in solution. The carrier solution should be chosen with the subsequent measurement in mind; for samples requiring gamma counting only, it is usual to use a carrier of greater concentrations than would be the case for samples where further analyses, for example for strontium-90, are expected, and where addition of carrier may interfere with the chemical methods employed. Carrier solutions should be added at the first available opportunity; in the case of rainwater, when collection of the sample is over a number of days or weeks, this would be at the onset of sampling; in most other cases it would be at the time of sampling. The carrier solutions are usually slightly acidified to assist prevention of absorption. However, if measurements are to be made on suspended solids (see 5.1), these must be removed before acidification to minimize dissolution of particulates and/or desorption of radionuclides from them. In this case, filtration or centrifugation should be carried out as soon as possible after sampling, preferably immediately, and the carrier solution then added to the filtrate.

An example of a carrier solutions in common use is:

Barium nitrate $\text{Ba}(\text{NO}_3)_2$	191 mg
Cerium (III) nitrate $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$	309 mg
Caesium nitrate CsNO_3	147 mg
Manganese (II) chloride $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$	360 mg
Ruthenium chloride $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$	259 mg
Antimony oxychloride $\text{Sb}_2\text{O}_3 \cdot 2\text{SbOCl}$	131 mg
Zirconyl chloride $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$	253 mg
Strontium nitrate $\text{Sr}(\text{NO}_3)_2$	1210 mg
Sodium fluoride NaF	400 mg
Yttrium nitrate $\text{Y}(\text{NO}_3)_3$	2150 mg
Sodium iodide NaI	30 mg
Potassium iodate KIO_3	30 mg

Each salt is made up to 100 mL with water and equal volumes of these solutions are mixed together to produce the carrier solution. The ruthenium, antimony and zirconyl salts are first dissolved in a few mL of concentrated hydrochloric acid before diluting to 100 mL. Note that the ruthenium salt may not dissolve fully in acid until the water is added. 10 mL of the mixed carrier solution is used for up to 5 L of sample.

Ideally storage of the samples, where necessary, should be in tightly closed, plastic bottles in the dark at 4°C. On no account must the sample be frozen.

9.3 Pre-concentration

Though seemingly attractive, preconcentration in gamma-spectrometric analysis is something not to be generally encouraged as it has many inherent problems and destroys the fundamental advantage of gamma spectrometry—namely the ability to make measurements on the original material with the minimum of handling or alteration. However, in certain circumstances it may be necessary to preconcentrate the radionuclides in a sample prior to carrying out gamma-spectrometric analysis. The need to pre-concentrate may arise because the required radionuclide concentrations are too low for direct measurement on the original sample, or because of bulk storage problems. Whatever the reason, considerable care is needed if quantitative results are to be achieved.

The method chosen for volume reduction depends on the nature of the sample and the radionuclides of interest. Thus, if several radionuclides are to be determined in a water of low solute concentration, evaporation may be suitable but the conditions

for evaporation must be selected so that potentially volatile determinands are not lost. For example, alkaline evaporation in the presence of sodium hydroxide will prevent the loss of ruthenium and iodine but, if this procedure is followed, steps must be taken at the end of the evaporation to ensure the complete recovery of many other radionuclides from the walls of the container. An acid wash normally achieves this reasonably well.

Where specific radionuclides need to be concentrated, various procedures are possible. Co-precipitation with ferric hydroxide or manganese dioxide, for example, will scavenge numerous carrier-free radionuclides with high efficiency. Complexation/co-crystallization procedures may also be useful, for example, in the collection of cobalt radionuclides from seawater using 1 nitroso-2-naphthol (26). The pre-concentration of caesium radionuclides from fresh or saline waters using ammonium molybdophosphate (AMP) is in common use. Depending on the way in which this is carried out, the process may be considered one of co-crystallization or inorganic ion-exchange, and under the right conditions it can be made very specific for caesium (27). One example of the use of organic ion exchangers to pre-concentrate radionuclides for gamma spectrometry, is the collection of iodine as iodide on Dowex anion exchange resin.

The possibilities for pre-concentration are almost endless; details of a number of useful methods are given in publications such as the Procedures Manual of the US Department of Energy's Environmental Measurements Laboratory (18). Any method must be fully evaluated before use and, where possible, should be checked using some means of determining chemical recovery of the radionuclides of interest. One way to achieve this is to add at the beginning of the concentration step, a known amount of gamma-emitting isotopic tracer for each element, to act as a yield monitor. A possible disadvantage of this approach is that it increases the complexity of the gamma-spectrum and interference problems may be troublesome. Such problems may be avoided by adding an accurately known amount of the stable form of the element to provide yield recovery measurements. In either case, steps must be taken to ensure proper isotopic exchanges between tracer and determined forms of the element before pre-concentration begins. This may involve an oxidation/reduction cycle or the breakdown of complexes in order to ensure that both tracer and determined forms of the element are present as the same species during the pre-concentration.

10. Counting Geometry

It is important to select the counting geometry so as to maximize the counting efficiency of the detector. The optimum would be a point source, but in practice this is not feasible and there are only a limited number of geometries that are commonly used. One typical example is the Marinelli beaker (see Figure 9); this container uniformly presents the maximum useable volume of sample around the sensitive area of the crystal and is particularly useful for water samples (17). Other types of container of smaller volume, designed to sit on top of the detector, are more commonly used for solid materials. With these it is preferable that the container does not overlap the crystal and is not too tall, as both reduce the effective counting efficiency.

11. Calibration

The essential requirements of a calibration are to establish an energy/efficiency/resolution relationship. This is most conveniently achieved by using a commercially-available mixed source standard (6.4) dispersed evenly throughout the calibration material. This should contain radionuclides covering the whole gamma energy range. Dispersion is relatively easy to achieve with liquid samples. However, solids present more of a problem because the calibration medium has to reflect accurately the density of the samples to be measured.

Calibrations should be checked periodically to assure the quality of data. Any change of geometry, density, volume, etc necessitates either an additional calibration or, if suitable software is available, factors may be established to take account of the change.

The following practical instructions for calibrating the counting system are based on the assumption that the user has access to a computer-controlled system. If this is not the case then peak areas, efficiencies, activities, etc can be calculated by hand as

described in section 11.2, but it should be clearly recognized that this can be an exceedingly complex calculation, especially if the effects of interference of one radionuclide upon another, ingrowth of daughter radionuclides, etc are to be accounted for.

Despite all efforts to shield the detector and sample from extraneous radiation, the detector always records a small continuum of background radiation together with some photopeaks over the spectral range.

When counting low-level environmental samples, the number of counts collected from the sample radioactivity is usually low. Under such conditions it is important to make allowances for the background contribution to the gross count. Careful measurements should therefore be made of (a) the environmental background arising from the shield and its surroundings; (b) any additional effects of the blank material used in the calibration.

Containers of the same type as those used for the samples should be filled with blank material and counted for not less than four times longer than that required for the least active sample (see 11.1.11 note s). These data are stored in the system memory and are used to correct the sample counts at a later stage.

Step	Procedure	Notes
11.1	Computer-assisted calibration	
11.1.1	Select with utmost care the counting geometry(ies), choose one container to carry out the initial calibration (note 1)	(l) Keep in mind future supply of containers and costs etc.
11.1.2	Fill the container with the prepared reference source (note m) and double wrap with protective film	(m) (See also 6.4)
11.1.3	Place the container on to the detector in such a way that the position can be reproduced for any other sample	
11.1.4	Acquire a spectrum for a time period such that at least 10^4 counts have been collected for each peak of interest (note m)	
11.1.5	Following the manufacturer's instructions, input reference source data. Then, using the spectral output, calculate each peak efficiency (note n)	(n) It is assumed that a radionuclide library resides within the computer memory
11.1.6	Plot an efficiency/energy relationship	
11.1.7	Using recognized mathematical principles smooth the curve to eliminate any points which cause irregularities (note o)	(o) Any points within the statistical limits may be smoothed. Others outside require careful checking before proceeding further
11.1.8	Recalculate efficiency values (note p)	(p) Figure 6 shows a typical calibration for a popular type of HPGe detector
11.1.9	Follow the required computer protocol to input the new efficiency values, (from 11.1.8) (note q)	(q) These values are only valid for this geometry. All other geometries require new calibration
11.1.10	Repeat the calibration for any other geometry or density (note r)	(r) Some software systems allow a single calibration to be used in conjunction with factors which relate one geometry or density to another

Step	Procedure	Notes
11.1.11	Fill a container of the same geometry with blank material and count for at four times the counting period used for the least active sample (notes s and t)	(s) The Blank should consist of material least of the same matrix composition as the sample but without detectable radioactive content (t) Count for NOT less than 24 hours
11.1.12	Store or keep this background spectrum for subsequent use in the calculation of the activity of samples	
11.1.13	In an identical container to that used in the calibration, count a sample of known radioactive content for sufficient time to accumulate statistically valid counts in each peak of interest (note u)	(u) The known sample may be a reference material or reference standard, preferably with the same matrix composition as the sample
11.1.14	Using the efficiency curve already obtained, calculate the activity in the sample in recognized units, eg BqL ⁻¹ or Bq Kg ⁻¹	(v) The efficiency curve and other related data such as the peak search programmes etc are assumed to be in the computer memory
11.1.15	Compare answers with those given for the known sample. Accept those that fall within say ± two standard deviations of the correct result. If errors greater than this exist, carry out an investigation and carefully check calibration data	
11.2	Manual calibration	
11.2.1	Proceed as for steps 11.1.1 to 11.1.4	
11.2.2	Print out the raw spectral data	
11.2.3	Sum the counts under each peak of interest as shown in Figure 8, to obtain N. Let the net counts corresponding to each energy E be N _E	
11.2.4	Calculate the efficiency for each peak using the following equation (note w)	(w) Values of P _E may be obtained from reference data (7)
	$\epsilon_E = \frac{N_E}{A_i(t) \times t_o(r) \times P_E}$	
	where ϵ_E = the counting efficiency for gamma rays with energy E;	
	N _E = the net counts collected for the peak corresponding to energy E;	
	A _i (t) = the activity (Bq) of the radio-nuclide standard i at the time of measurement;	
	t _o (r) = the live-time in seconds (ie corrected for dead-time) during which the reference source has been counted;	
	P _E = the transition probability of decay for the route giving gamma-ray energy E.	
11.2.5	Plot the efficiency versus energy relationship (note x)	(x) Figure 6 shows a calibration curve for an HPGe detector

Step	Procedure	Notes
11.2.6	Smooth the efficiency curve, either mathematically, or by sketching a best-fit line (note y)	(y) The mathematical principles involved in doing this are complex. The simpler approach of sketching in a best-fit line by hand introduces relatively little error
11.2.7	Repeat steps 11.2.1 to 11.2.6 for any other geometry or density that needs to be used	
11.2.8	Proceed as in steps 11.1.11 to 11.1.15 for blanks and a known sample (note u)	

12. Sample Counting Procedure

Frequently, manufacturers allow the user some control over the degree of uncertainty associated with the counts, in order to set the level of detection and selection of a peak from the accumulated spectrum. Manufacturers describe this as a control of sensitivity.

It must be understood that by using high sensitivity there is a risk that some of the peaks supposedly identified could more properly fall within the random variability of the background continuum.

The activity of identified radionuclides is calculated from calibration data taking into account background contributions, interferences, half-lives, etc and a print-out is produced. These data must then be examined with care by a skilled analyst, as it must be realized that they are mathematically calculated and occasionally may give anomalous results. For example unless the operator predetermines a maximum number of half lives that the software should be allowed to back calculate, a very large factor could be applied in the case of short lived radionuclides. Such results could be totally unreliable.

Note: Manufacturers almost always supply a radionuclide library with spectrometric systems, but users are free to make their own additions or deletions as they find necessary. Reference was made in section 2, to a definitive source of nuclear data (ref 7). These data are revised periodically.

12.1 Sample counting procedure

Step	Procedure	Notes
12.1.1	Count the sample in an appropriate geometry for sufficient time to ensure that the precision with which individual radionuclides are measured is sufficient for the analytical requirements (note z)	(z) For low-level environmental samples this can only be achieved by counting for considerable lengths of time—typically in excess of 60,000 seconds and occasionally much longer
12.1.2	If a pre-set counting time has not been selected, stop the counting when sufficient counts have been obtained in all peaks of interest	
12.1.3	Use calibration data to calculate activity concentrations, or limits of detection as described in 12.2	

12.2 Analysis of spectra

Gamma spectra from environmental samples are often exceedingly complex, and it is almost impracticable to calculate a full range of results from these data without the aid of a computer and specially written software.

12.2.1 Computer-assisted analysis

Spectral information is fed into a computer where initially a comparison is made between peak position and energy and resolution. Having established this relationship, the computer then compares the gamma peak energies with those contained in the computer library and identifies radionuclides which fall within tolerance limits previously specified in the computer software.

Identified radionuclides are quantified in terms of count rate and converted into activity concentration (BqL^{-1}) using counting efficiency, decay data and sample volume.

12.2.2 Calculation of activity for a single radionuclide

To give some simple practical guidance to the method of calculation, the following procedure shows how the activity of a radionuclide is calculated from a well-defined peak which has no associated interferences.

Calculate the activity A_i for each radionuclide i present in the sample by means of the equation:

$$A_i = \frac{N_E \times f}{\epsilon_E \times P_E \times t_0}$$

where A_i is the activity in becquerels of the radionuclide i at the time of sampling; (see note below)

N_E is the net number of counts assigned to radionuclide i ;

E is the detection efficiency at the energy E to which the maximum of the peak corresponds;

P_E is the transition probability for the radionuclide i giving rise to the emission of a gamma-ray with energy E ;

t_0 is the time in seconds during which the spectrum has been recorded, corrected for dead time;

f is the factor required to correct for the decay of the radionuclide i between the time of sampling and the time of measurement.

It is calculated according to the equation:

$$f = e^{-\lambda t} \times \frac{t_0}{1 - e^{-\lambda t_0}}$$

where λ is the decay constant in reciprocal seconds of the radionuclide i ;

t is the time in seconds between the time of sampling and the start of the measurement;

t_0 is the time in seconds, corrected for dead time, during which the spectrum was recorded.

Note: Many radionuclides yield more than one photopeak. On one hand this facilitates the assignment of the peaks to their respective radionuclides, but on the other hand it may yield more than one activity value for each radionuclide. The best estimates are usually derived from the strongest photopeaks, but interferences from other radionuclides might preclude this choice.

12.2.3 Calculation of the activity concentration

Calculate the activity concentration C for each radionuclide i present in the sample by means of equation:

$$C_i = \frac{A_i}{V}$$

where C_i is the activity concentration in becquerels per litre of the radionuclide i at the time of sampling;

V is the volume of the sample in litres.

12.2.4 Calculation of the uncertainty due to the counting statistics

The coefficient of variation of the activity due solely to the statistical nature of the process of nuclear disintegration is calculated by means of the equation:

$$\sigma = \frac{\sqrt{N+b}}{(N-b)} \times 100\%$$

where σ is the percentage uncertainty;

N is the total number of counts under the peak;

b is the number of counts in the background under the peak in the same time.

12.2.5 Calculation of the limit of detection

The limit of detection for each radionuclide is calculated by means of the following equation(18).

$$LD = F \times 2 \times k \times \sqrt{2b}$$

where F = a factor converting counts to activity involving duration of count decay, transition probability efficiency etc;

k = value of the normal probability integral corresponding to $P=0.05$ 95% confidence level, ie 1.645 for a one tailed distribution

$$\begin{aligned} \text{Hence } LD &= F \times 3.29 \sqrt{2b} \\ &= F \times 4.66 \sqrt{b} \end{aligned}$$

See also the Appendix.

12.3 Test Report

The internal test report should include the following information:

- a reference to this SCA method;
- details of any sample pretreatment, eg filtration, acidification (in which order); evaporation, suspended matter etc.
- complete identification of the sample (see 13.3 (iv));
- the activity concentration of each radionuclide which has been identified, with its coefficient of variation and limit of detection;
- the precision and the estimated accuracy of the measurements;
- the limit of detection for each radionuclide which has not been detected but whose presence was sought;
- the geometry of the system including, in particular, the sample holder dimensions and the detector type;
- the relative efficiency of the detector and the energy resolution at 1.33 MeV;
- any unusual features noted during the determination;
- date and time of the analysis (start and finish);
- any deviation from the procedures specified in this method;
- the analyst and the laboratory involved.

13. Quality Assurance 13.1 General

There are many references in the technical literature on the subject of quality assurance and quality control procedures: a short selection is provided in references 12, 13, 19, 20, 21, 22, 23, 24, 25.

13.2 Standards (see Section 5, 6.2–6.4)

13.3 Records

As the analyst acquires experience in the day-to-day use of gamma ray spectroscopy, a great deal of data will be accumulated. The first and an elementary requirement is to establish a system to collate the data. The following headings constitute a guide or starting point for further development:

- (i) An instrument logbook or diary. To record commissioning and maintenance visits, power supply failure, air conditioning excursions, equipment faults and repairs (symptom and cure) and any unusual events.
- (ii) A background spectra log, and efficiency calibration records. A computer-based system may enable these records to be stored on disc, however there are circumstances (legal implications for the presentation of court evidence; Police and Criminal Evidence Act) where hard copy is essential.
- (iii) Instrument stability. Variations in response, drift or resolution can be determined using a suitably diluted reference solution (6.4). Control charts can be set up but it will not prove practicable to cover the energy spectrum in its entirety. Two or three energy peaks suitably distributed will be all that can be managed. If cobalt-60 is included it will at the same time enable instrument resolution to be measured, but as with any other radionuclide, its decay/half-life will have to be taken into account when comparing efficiency of response at different times.
- (iv) Sample identification and traceability of data.

Sample numbering system(s) and analytical data records employed on a day to day basis must be capable of demonstrating traceability through the various stages of sample preparation and analysis.

13.4 Quality Control and Intercomparison exercises

Gamma-ray spectrometry can be a totally non-destructive technique, however for environmental monitoring, the levels of radioactivity are such that concentration steps are frequently necessary if a definitive result is required (as opposed to a limit test). Thus there is scope for variation between individual analysts in the processes employed and in their use of the instrumentation.

As with any analytical technique the uninitiated should develop their skills through replicate analyses from a large bulk sample. The results can be used to develop a quality control chart or computer equivalent to enable the determination of mean values, warning levels and action markers (ref 22, 24, 25).

The performances of different analysts, or of different methods, can be assessed similarly (ref 23).

Participation in interlaboratory analytical comparison exercises once or twice a year will provide an additional quality assurance function to guard against an organization developing a bias of its own. Equally it will serve to establish the credibility of a laboratory in an acceptable independent forum.

14. Some radionuclides for determination by gamma-ray spectrometry
(for a complete nuclide list see reference 7)

14.1 Naturally-occurring gamma emitting radionuclides

Atomic number	Element	Symbol	Mass number	Principal energy (MeV)	Abundance (percent)	Half-life
4	Beryllium	Be	7	4.776E-01	10.3	53.3 d
19	Potassium	K	40	1.461E-00	10.7	1.28 × 10 ⁹ y
81	Thallium	Tl	208	5.831E-01	85.8	3.07 m
82	Lead	Pb	210	4.650E-02	4.05	22.3 y
82	Lead	Pb	211	4.048E-01	3.00	36.1 m
				8.318E-01	2.82	
82	Lead	Pb	212	2.386E-01	44.6	10.64 h
83	Bismuth	Bi	212	7.272E-01	11.8	60.55 m
86	Radon	Rn	219	2.712E-01	9.9	3.96 s
88	Radium	Ra	226	1.860E-01	3.28	1.6 × 10 ³ y
89	Actinium	Ac	228	9.111E-01	29.0	6.13 h
90	Thorium	Th	227	5.020E-02	8.51	18.718 d
90	Thorium	Th	228	8.440E-02	1.19	1.9131 y
90	Thorium	Th	232	5.900E-02	0.191	1.405 × 10 ¹⁰ y
92	Uranium	U	235	1.857E-01	54.0	7.038 × 10 ⁸ y

14.2 Gamma emitting radionuclides arising from routine discharges

Atomic number	Element	Symbol	Mass number	Principal energy (MeV)	Abundance (percent)	Half-life
25	Manganese	Mn	54	8.348E-01	100	312.5 d
27	Cobalt	Co	60	1.173E-00	99.9	5.271 y
				1.332E-00	100	
30	Zinc	Zn	65	1.116E-00	50.7	243.9 d
40	Zirconium	Zr	95	7.567E-01	55.0	63.98 d
41	Niobium	Nb	95	7.658E-01	100	35.15 d
43	Technetium	Tc	99m	1.405E-01	88.9	6.02 h
44	Ruthenium	Ru	103	4.971E-01	86.4	39.28 d
44	Ruthenium	Ru	106	6.218E-01(a)	9.81	368.2 d
47	Silver	Ag	110m	8.847E-01	72.9	249.9 d
51	Antimony	Sb	125	4.279E-01	29.5	2.77 y
53	Iodine	I	125	3.549E-02	6.67	60.14 d
53	Iodine	I	129	3.958E-02	7.51	1.57 × 10 ⁷ y
53	Iodine	I	131	3.645E-01	81.2	8.04 d
55	Caesium	Cs	134	6.047E-01	97.6	2.062 y
55	Caesium	Cs	137	6.616E-01(b)	89.80	30.0 y
58	Cerium	Ce	144	1.335E-01	10.8	284.3 d
63	Europium	Eu	152	3.443E-01	26.6	13.33 y
63	Europium	Eu	154	1.274E-00	35.5	8.8 y
63	Europium	Eu	155	1.053E-01	20.6	4.96 y
88	Radium	Ra	226	1.860E-01	3.28	1.6 × 10 ³ y
				8.654E-02	30.9	
90	Thorium	Th	232	5.900E-02	0.191	1.405 × 10 ¹⁰ y
95	Americium	Am	241	5.954E-02	35.7	4.322 × 10 ² y

Notes

(a) Via ¹⁰⁶Rh daughter (t_{1/2} = 29.9 s)

(b) Via ^{137m}Ba daughter (t_{1/2} = 2.522 m)

14.3 Gamma emitting radionuclides routinely used in hospitals

Atomic number	Element	Symbol	Mass number	Principal energy (MeV)	Abundance (percent)	Half-life
Open use						
24	Chromium	Cr	51	3.201E-01	9.83	27.704d
27	Cobalt	Co	57	1.221E-01	85.60	270.90d
27	Cobalt	Co	58	8.108E-01	99.40	70.80d
31	Gallium	Ga	67	9.331E-02	38.30	78.26h
34	Selenium	Se	75	1.360E-01	60.6	119.8d
36	Krypton	Kr	81m			13s
43	Technetium	Tc	99m	1.405E-01	88.90	6.02h
49	Indium	In	111	2.454E-01	94.00	2.83d
53	Iodine	I	123	1.590E-01	82.80	13.20h
53	Iodine	I	125	3.549E-02	6.67	60.14d
53	Iodine	I	131	3.645E-01	81.20	8.04d
54	Xenon	Xe	133	8.100E-02	37.40	5.245d
81	Thallium	Tl	201	1.674E-01	10.00	3.044d
In sealed sources only						
27	Cobalt	Co	57	1.221E-01	85.60	270.90d
27	Cobalt	Co	60	1.173E-00	99.9	5.271y
				1.332E-00		
55	Caesium	Cs	137	6.616E-01	89.80	30.0y
77	Iridium	Ir	192	3.165E-01	82.80	74.02d
79	Gold	Au	198	4.11E-01	95.50	2.696d

14.4 Gamma emitting radionuclides which might arise from an accident

Atomic number	Element	Symbol	Mass number	Principal energy (MeV)	Abundance (percent)	Half-life
27	Cobalt	Co	60	1.173E-00	99.9	5.271y
				1.332E-00	100	
40	Zirconium	Zn	95	7.567E-01	55.0	63.98d
41	Niobium	Nb	95	7.658E-01	100.0	35.15d
42	Molybdenum	Mo	99	1.405E-01	4.95	66.00h
43	Technetium	Tc	99m	1.405E-01	88.9	6.02h
44	Ruthenium	Ru	103	4.971E-01	86.4	39.28d
44	Ruthenium	Ru	106	6.218E-01(a)	9.81	368.2d
47	Silver	Ag	110m	8.847E-01	72.9	249.9d
51	Antimony	Sb	125	4.279E-01	29.5	2.77y
52	Tellurium	Te	129m	1.055E-01	0.148	33.6d
52	Tellurium	Te	132	2.282E-01	88.2	78.2h
53	Iodine	I	131	3.645E-01	81.2	8.04d
53	Iodine	I	132	6.677E-01	98.7	2.3h
53	Iodine	I	133	5.299E-01	86.3	20.8h
55	Caesium	Cs	134	6.047E-01	97.6	2.062y
55	Caesium	Cs	137	6.616E-01(b)	89.80	30.0y
56	Barium	Ba	140	5.373E-01	24.4	12.74d
57	Lanthanum	La	140	1.596E-01	95.4	40.272h
58	Cerium	Ce	141	1.454E-01	48.0	32.501d
58	Cerium	Ce	144	1.335E-01	10.8	284.3d
93	Neptunium	Np	239	1.061E-01	22.7	2.355d

Notes

- (a) Via ^{106}Rh daughter ($t_{1/2} = 29.9\text{s}$)
 (b) Via $^{137\text{m}}\text{Ba}$ daughter ($t_{1/2} = 2.522\text{m}$)

Short-lived radionuclides in the lists are associated with longer-lived parents with which they are usually in equilibrium. Hence they will appear in the gamma spectrum. Care must be exercised in making decay

corrections for parent/daughter radionuclide combinations. Usually, but not always, (depending on relative length of half-lives) the activity of both parent and daughter is determined by the half-life of the longer lived parent.

15. Note on the handling of suspended solids, sludges, sediments and soils.

Suspended Solids and Sludges can contain appreciable amounts of the total radioactive material in a sample; similarly, sediments and soils may be of interest. There are many analytical methods in use, dependent on the type of sample, level of activity and radionuclides expected or sought.

15.1 Samples where the bulk of the sample is liquid and the solid particles fine and easily suspended may be measured by the foregoing procedure (Section 11), but with good stirring to ensure a relatively homogeneous sample.

15.2 It is sometimes possible to homogenize a sample with a high solid content (such as a sludge or sediment) and completely fill a sample container without leaving any voids. The (Section 11) procedure is followed.

15.3 If the sample is intermediate in fluid content and segregates too readily, either filter sufficient sample using a filter of suitable porosity and analyse as two samples, one liquid and the other homogenized solid (as in 15.2), or add a suitable nonradioactive absorbent such as pure cellulose, homogenize thoroughly and analyse as in 15.2

15.4 Completely solid samples can be analysed, after homogenization either by suspending in clean water with negligible radioactivity, or packed into a suitable container, without voids. Standardization of such analyses is difficult as this entails preparing similar standards with known activity. Usually, the results will only be qualitative, or semiquantitative, but such information may suffice.

15.5 CAUTION. Adequate precautions must be taken to contain all dust so as to prevent inhalation. Furthermore, it is essential that facilities are available to decontaminate all equipment and to dispose of waste without creating any hazard.

Appendix Gamma—Spectrometry: Limits of Detection

1 Introduction

This statement is based on L A Currie's paper 'Limits for Qualitative Detection and Quantitative Determination—Application to Radiochemistry' (Ref 28), but Currie's work has been extended to cover simple aspects of Gamma-spectrometry.

The theoretical treatment that follows is undertaken knowing that this probably is limited to the best possible case, and that the truth is probably worse.

Single peak assessment has been assumed.

2 Simple imprecision assessment

Consider a peak over a continuum (figure 10):

Let the total counts: = T
the counts in the peak = A
the counts in the continuum = B

(See also Figure 8 where A is N and B is b).

If the assumption is then made that the uncertainties associated with the measurement of the peak area are solely due to the randomness associated with radioactivity decay then:

$$\begin{aligned} \text{because } A &= T - B \\ \sigma_A &= (T + B)^{1/2} \\ &= (A + 2B)^{1/2} \end{aligned}$$

When low levels of activity are being considered:

$$A \rightarrow 0 \text{ and } \sigma_A \rightarrow \sqrt{2B}$$

Hence the uncertainty associated with very low or zero levels of activity can be taken to be the same as that of the nett zero signal.

3 The Nett Zero Signal (Nzs) distribution

Consider a series of γ -spectrometric measurements attempting to measure activity where none is present: a Gaussian pattern of distribution around the nett zero will result:

The shape of the distribution around the nett zero will depend on its standard deviation, $\sqrt{2B}$, which is the theoretical value. It should be remembered that the actual value of σ_{Nzs} may be worse.

4 Evidence of Activity above the Nett Zero Signal (Nzs)

It is usual (but not obligatory) to start any consideration of statistical events by stating a null hypothesis, in this case 'The counts recorded are indistinguishable from the nett zero signal'. We then have to set a critical count level such that the probability of being wrong (ie that we say there is activity when there is not) is less than a certain value (eg < 1 in 20 or $< .05$).

The selection of a specific probability limit will result in the selection of a specific value of gamma peak area count as being the critical count level; above this level we may reject our null hypothesis and say 'activity has been detected'—but always remembering that we may be wrong.

Such a critical count level is shown on figure 11 and is situated a certain number, k, of standard deviations above the nett zero mean. The value of k depends on the

probability requirements for not making a mistake (and is obtained from tables of one-tailed Gaussian distribution data):

Probability of making a mistake	k
< .05	1.645
< .01	2.326
< .005	2.576

So critical count = $k\sqrt{2B}$

and minimum activity based on critical count (MACC) = $F k\sqrt{2B}$

where F is the factor converting counts to activity (which involves duration of count, decay, decay scheme factor, efficiency etc).

5 Minimum detectable activity (MDA)

An analyst is often asked 'How small a quantity of activity could you have detected?' There is no answer to this question, but there is to the question 'What amount of activity has to be present such that it can be detected with a probability of for example < .05 of saying it is not there when it is?' This type of error is the opposite from that discussed in the previous sections, and the analyst must reply with both types of error in mind thus producing a different limit of detection: the minimum detectable activity (MDA).

The distribution patterns of the nett zero signal and the counts from the MDA are shown in Figure 12. (Distribution patterns for nett zero signal and counts from minimum detectable activity.)

Obviously, if probability requirements are the same (ie < a certain value) for making both types of error then

$$\text{MDA} = F \times 2 \times k \times \sqrt{2B}$$

and $\text{MDA} = 2 \times \text{MACC}$

6 Probability level to be used

The use of $p < .05$ (and hence $k = 1.645$) is often applied to radiometric data and probably (!) is the reason why so much emphasis is placed on the $\pm 2\sigma$ concept (for in a 2-tailed distribution only one result in 20 lies outside the $\pm 1.96\sigma$ limits). It would seem reasonable to use this criterion for the rejection or acceptance of the null hypothesis to establish 'detected' or 'not detected'.

7 Summary of 'Detection Limits' so far

We have now defined two values

(a) MACC—a minimum activity based on a critical count, such that the analyst can say that above this critical level there is a probability < p of the result coming from the nett zero signal distribution:

$$\text{MACC} = F \times k \times \sqrt{2B}$$

(b) MDA—a minimum detectable activity—this is the amount of activity that must be present such that there is a probability < P_1 of saying there is not any activity when there is and there is a probability < P_2 of saying there is activity present when there is not. If the probability $P_1 = P_2$ then:

$$\text{MDA} = F \times 2k \times \sqrt{2B}$$

The use of $p < .05$ as the requirement for making an incorrect judgement results in:

$$\begin{aligned} \text{MACC} (p < .05) &= F \times 1.645 \times \sqrt{2B} = F2.33\sqrt{B} \\ \text{MDA} (p < .05) &= F \times 3.29 \times \sqrt{2B} = F4.66\sqrt{B} \end{aligned}$$

One objection to the use of the MDA is that counts can quite properly arise and be regarded as detected at half the level of the MDA counts. Our Nuclear Data system originally used MDA ($p < .05$) and this caused concern to the data user who sees, eg < 200 and 110 on the same sample type and count time. We have altered our software to report MACC ($p < .05$) using $2.33\sqrt{B}$; the user can always multiply by 2 to get the MDA if he wishes.

8 Extension to obtain one sigma imprecision information

The theoretical imprecision associated with counts at the MACC and MDA levels can easily be calculated, for:

$$\begin{aligned} \sigma \text{ of the distribution of the nett zero signal counts} &= \sqrt{2B} \\ \text{and counts at the MACC level} &= k\sqrt{2B} \end{aligned}$$

$$\text{so at MACC, } \sigma = \frac{100\sqrt{2b}}{k\sqrt{2b}} = \frac{100}{2k}\%$$

$$\text{and similarly, at MDA, } \sigma = \frac{100}{2k}\%$$

Hence, using $k = 1.645$ we see that any result with a calculated imprecision (one σ) of worse than 30.4% is below the MDA ($p < .05$), and any result with an imprecision worse than 60.8% is below the MACC ($p < .05$) and is therefore 'not detected'. A graph of imprecision of result (one sigma) at the MACC level versus probability is attached—Figure 13.

9 Possible extension to practical situations

The theoretical limits of detection are worsened by two factors: systematic errors and added imprecision from random experimental errors.

One way to assess the added random experimental error is to determine the theoretical MACC in say a mixture of Cs-137, Cs-134 and Sb-125, and then to add quantities of radionuclides of interest such as Mn-54, Fe-59, Zn-65, Ag-110m, Ru-106, Ce-144 etc at levels between the calculated MACC and MDA. By replicate counting the actual distribution of results can be assessed and subsequently compared with the theoretical distribution. In this way some information on systematic error may also be obtained.

Acknowledgement

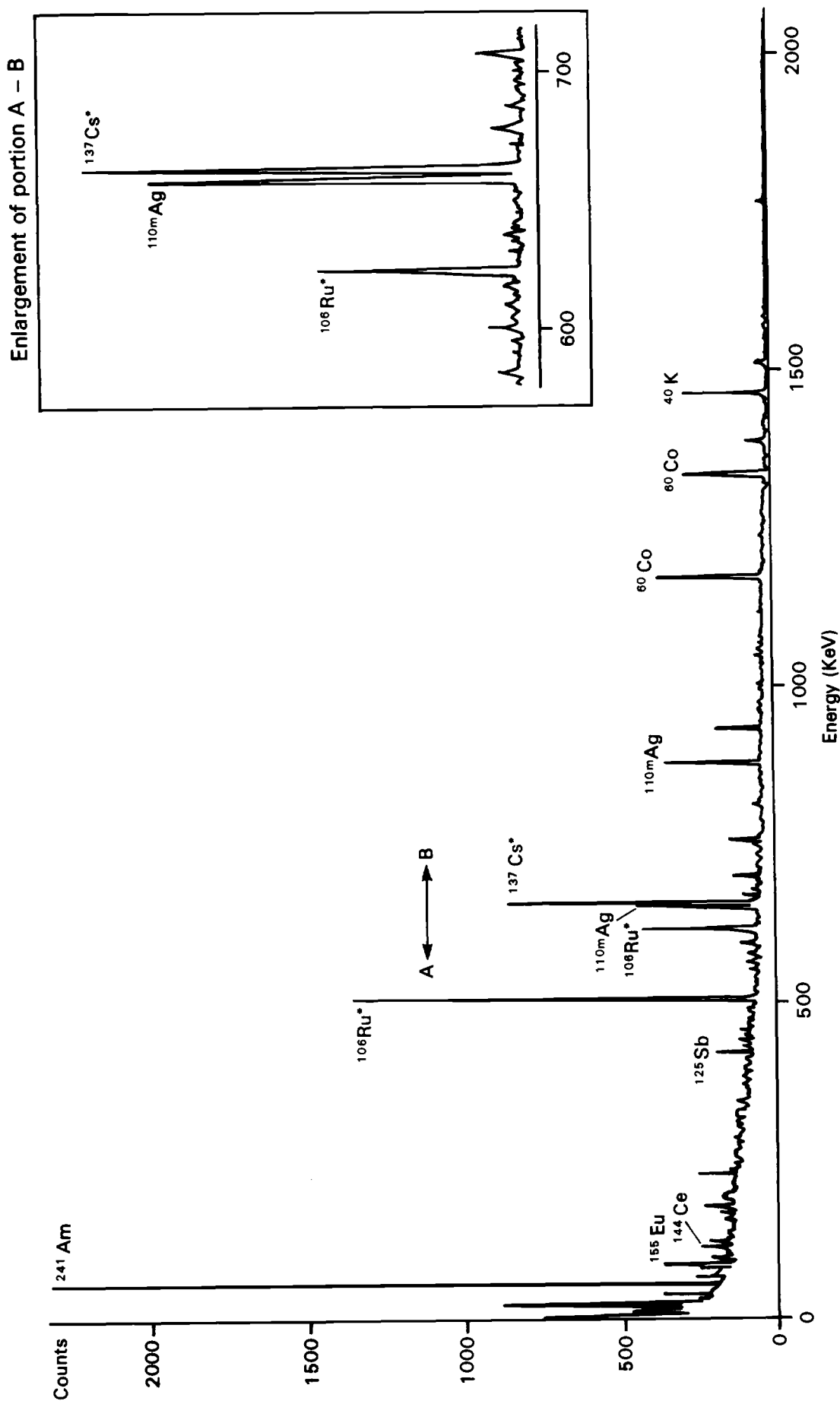
The statistical treatment reproduced here is that given in an unpublished document prepared by J W R Dutton, in 1982, for use at the Ministry of Agriculture, Fisheries and Food, Directorate of Fisheries Research, Lowestoft, England, who contributed it to this booklet.

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Figure 1 Gamma spectrum of sample counted on a 26% relative efficiency HPGe detector



*Via daughters. See 14.2

Figure 2 Schematic diagram of a typical gamma spectrometer

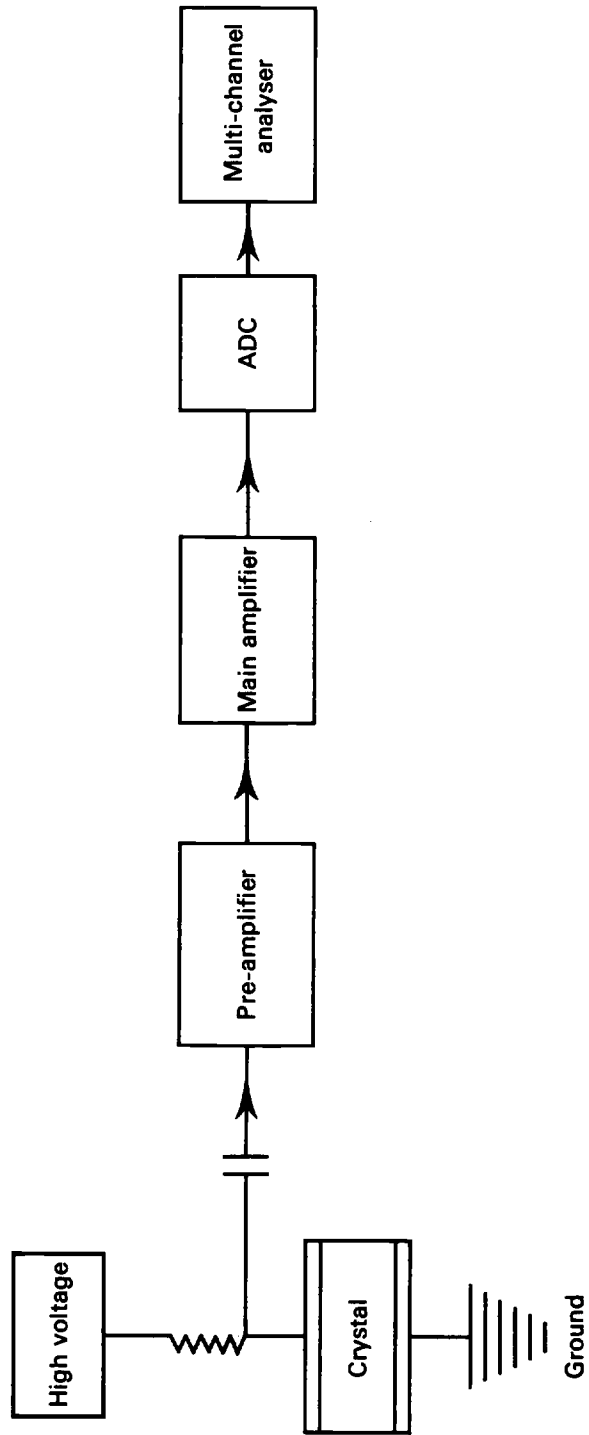


Figure 3 Gamma spectrum of ^{60}Co as measured by a) NaI(Tl) crystal b) HpGe detector

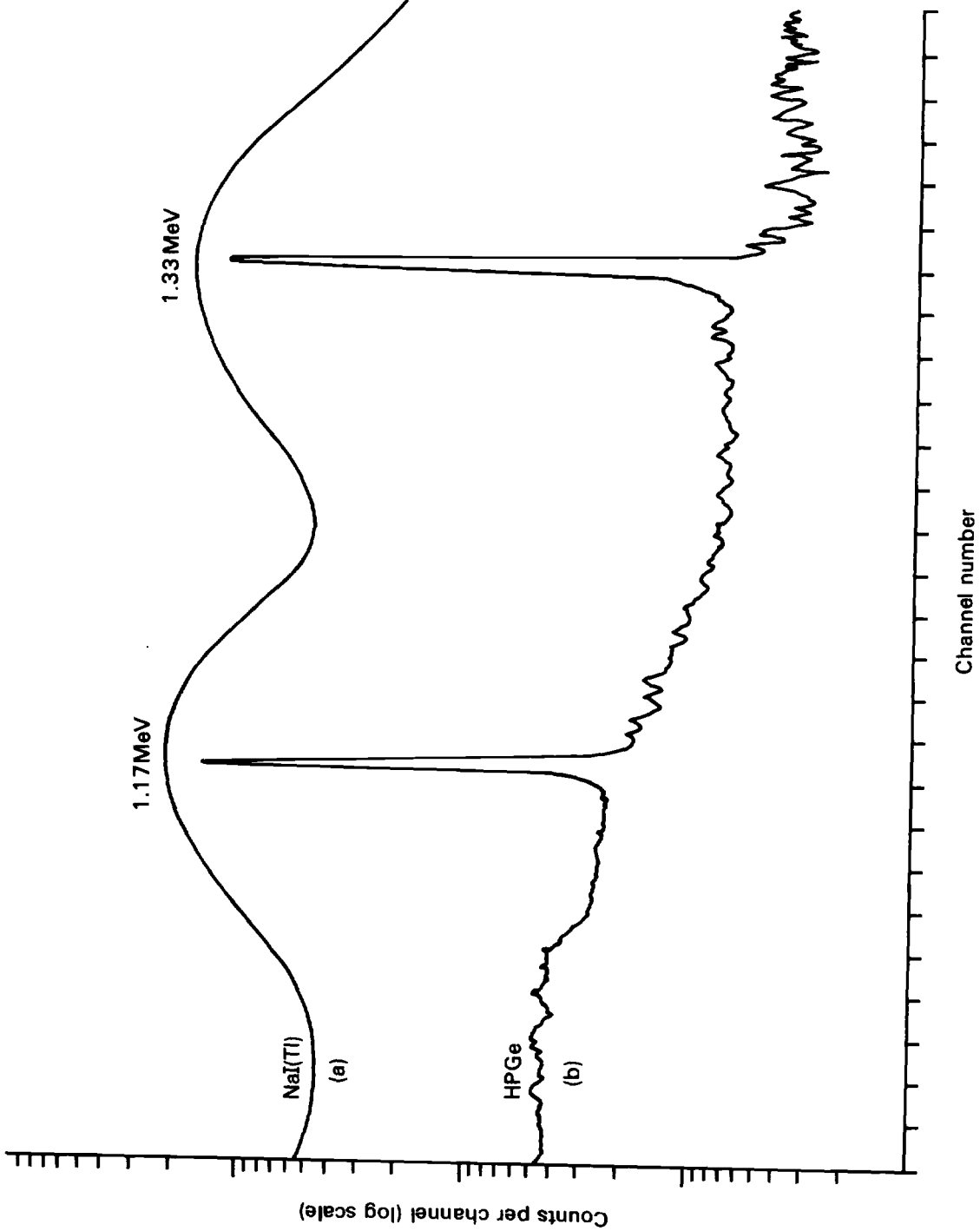


Figure 4a Definition of detector resolution

(Photopeak resolution is given by ΔE , where ΔE is the peak width in energy units at half maximum/pulse height)

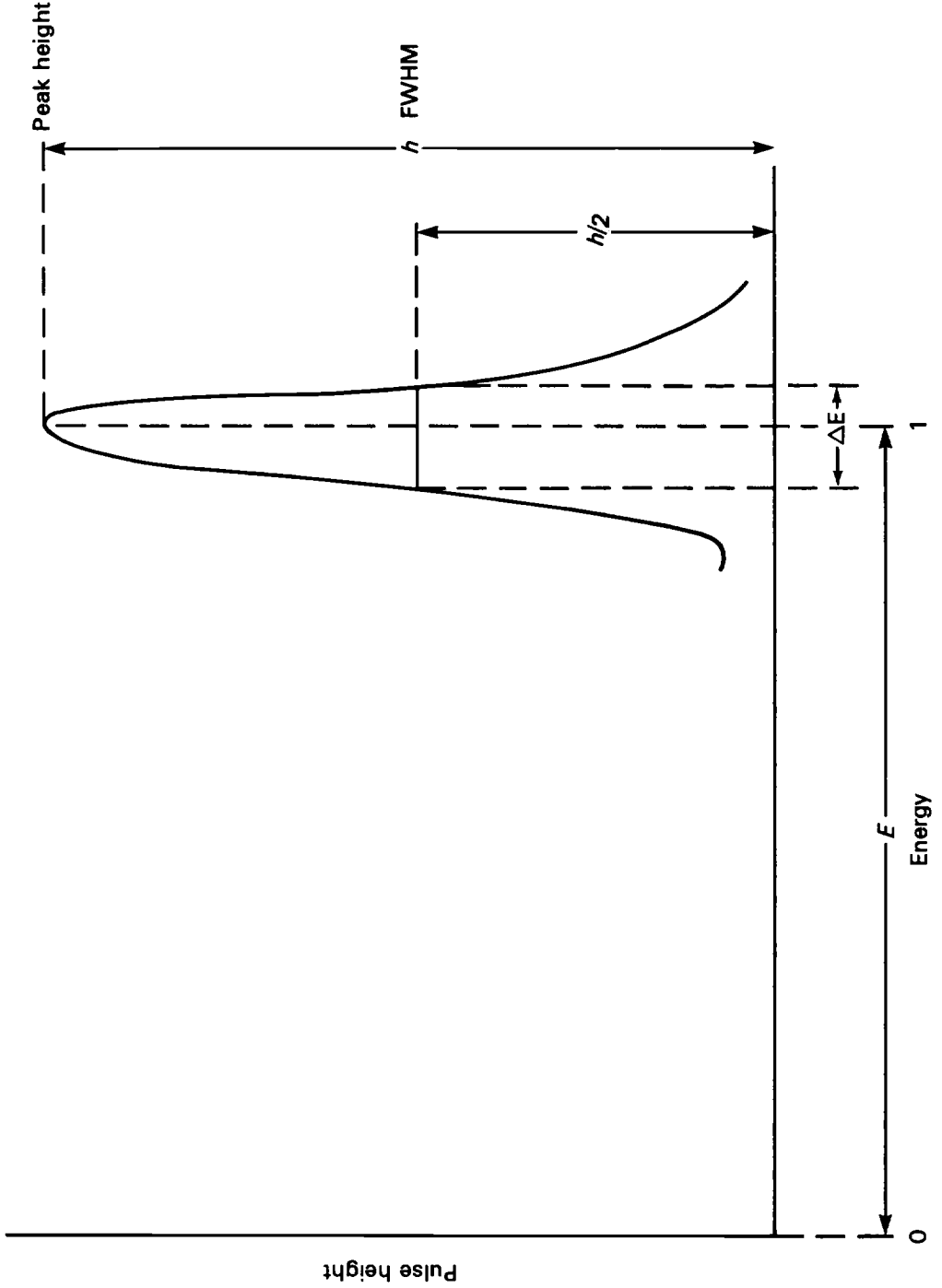


Figure 4b Definition of peak to Compton ratio

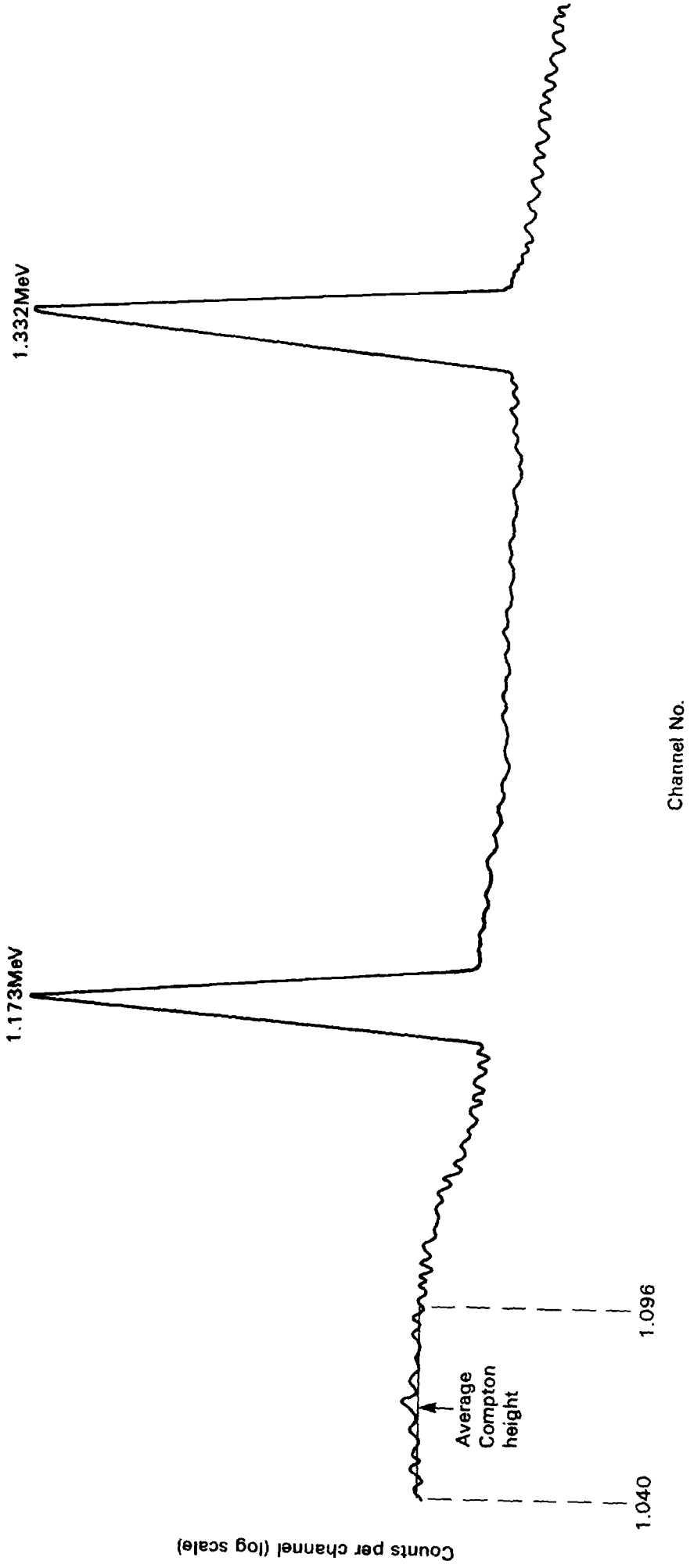


Figure 5 Ge detector assembly showing cryostat and shielding

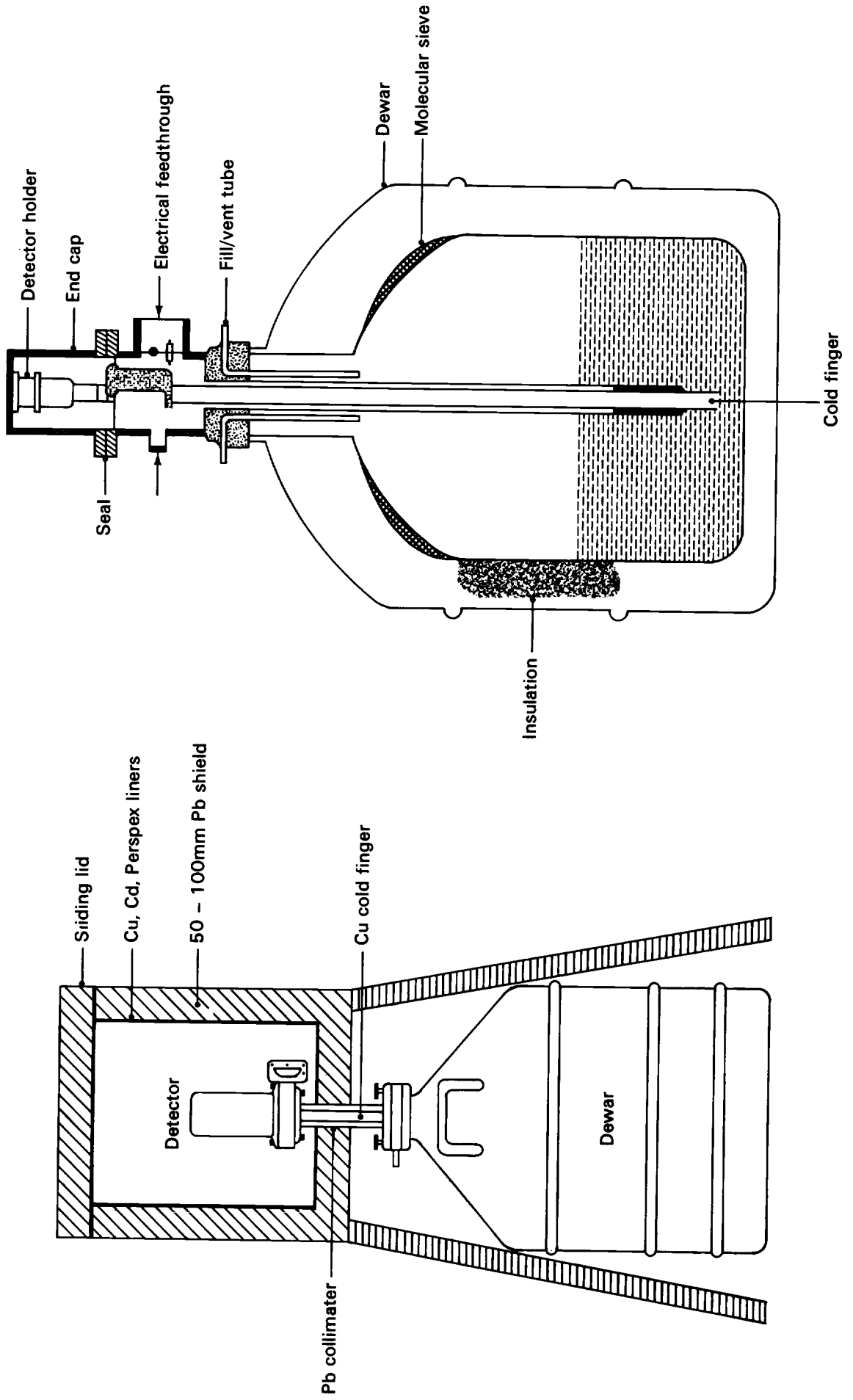


Figure 6 Efficiency v. energy calibration for a typical HPGe detector

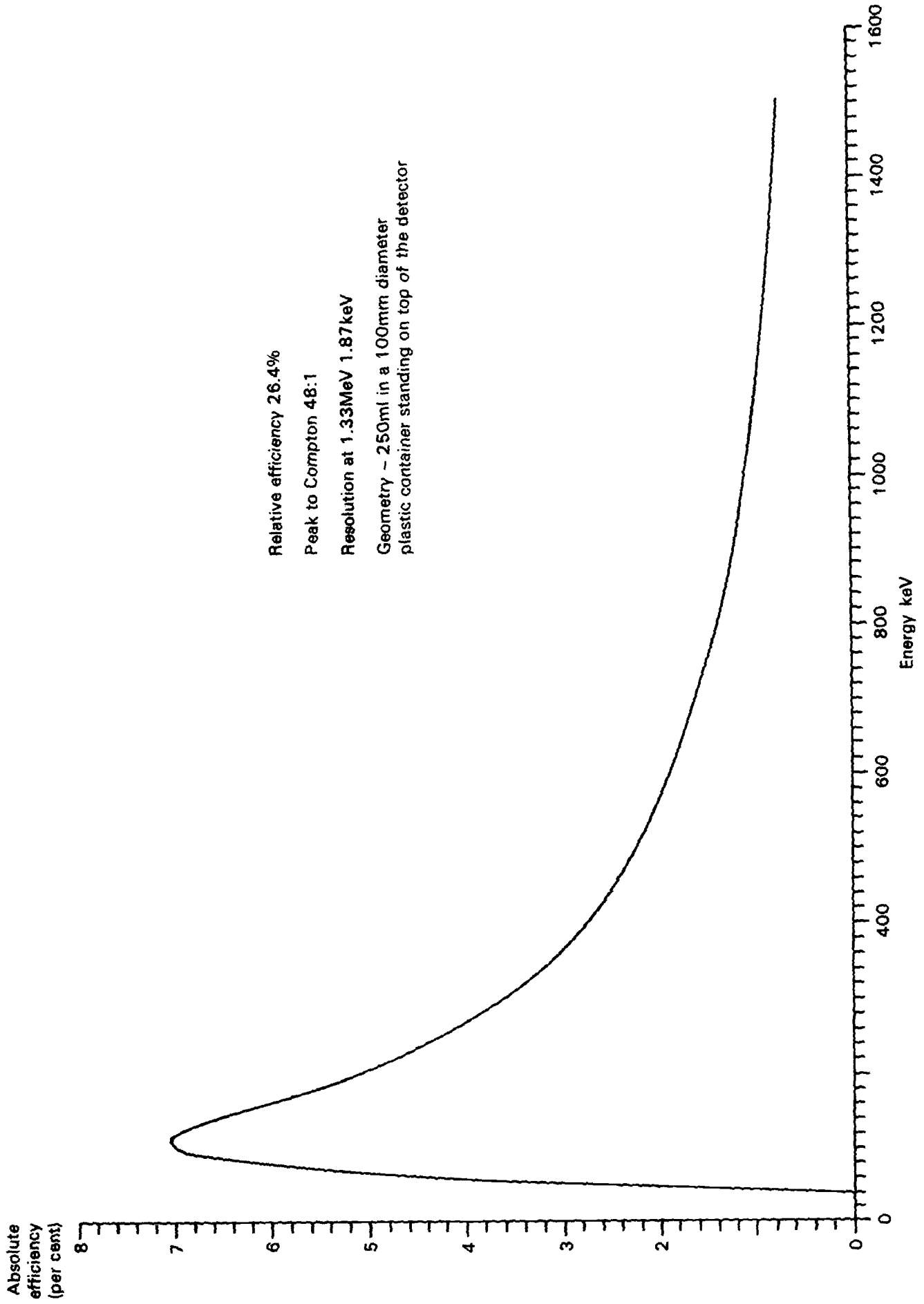
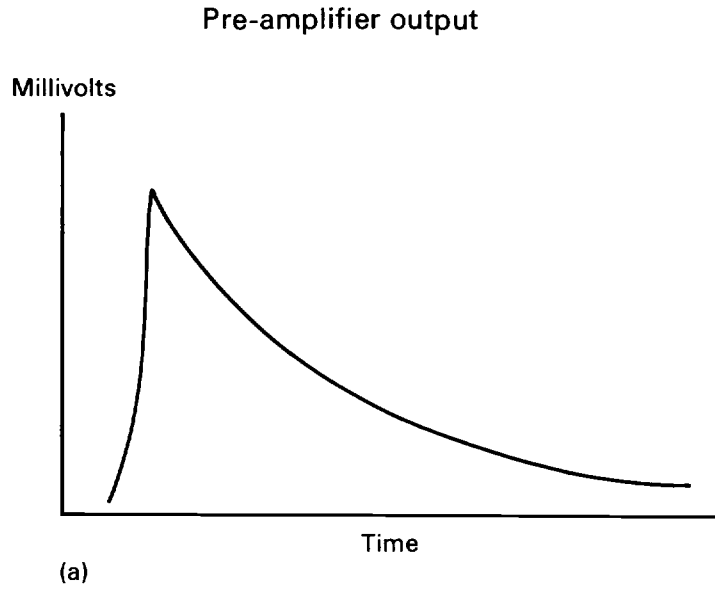
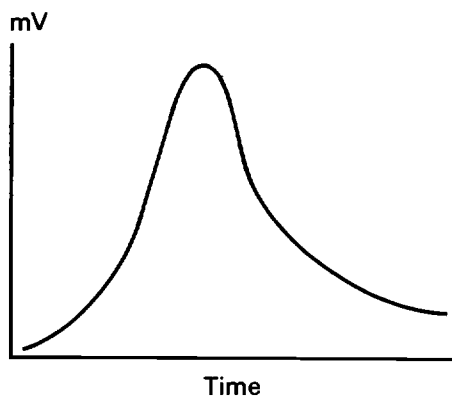


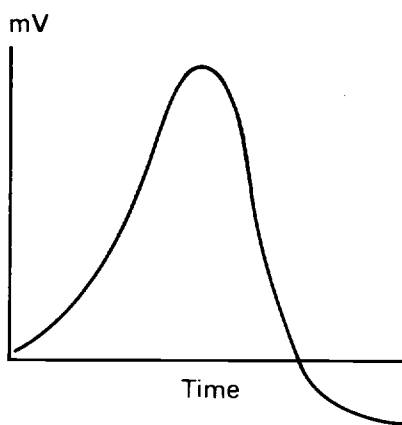
Figure 7 Pulse characteristics in a gamma spectrometer (Oscilloscope displays)



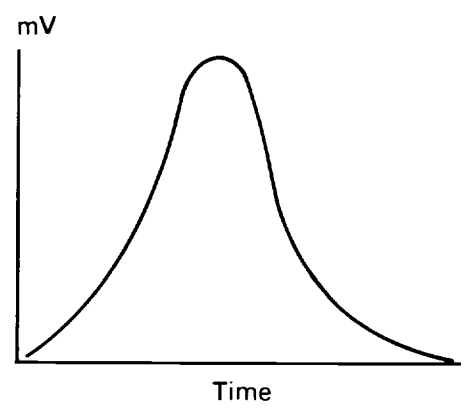
Main amplifier output



(b) Overshoot



(c) Undershoot



(d) Correct

Figure 8 Composition of a peak in a gamma spectrometry

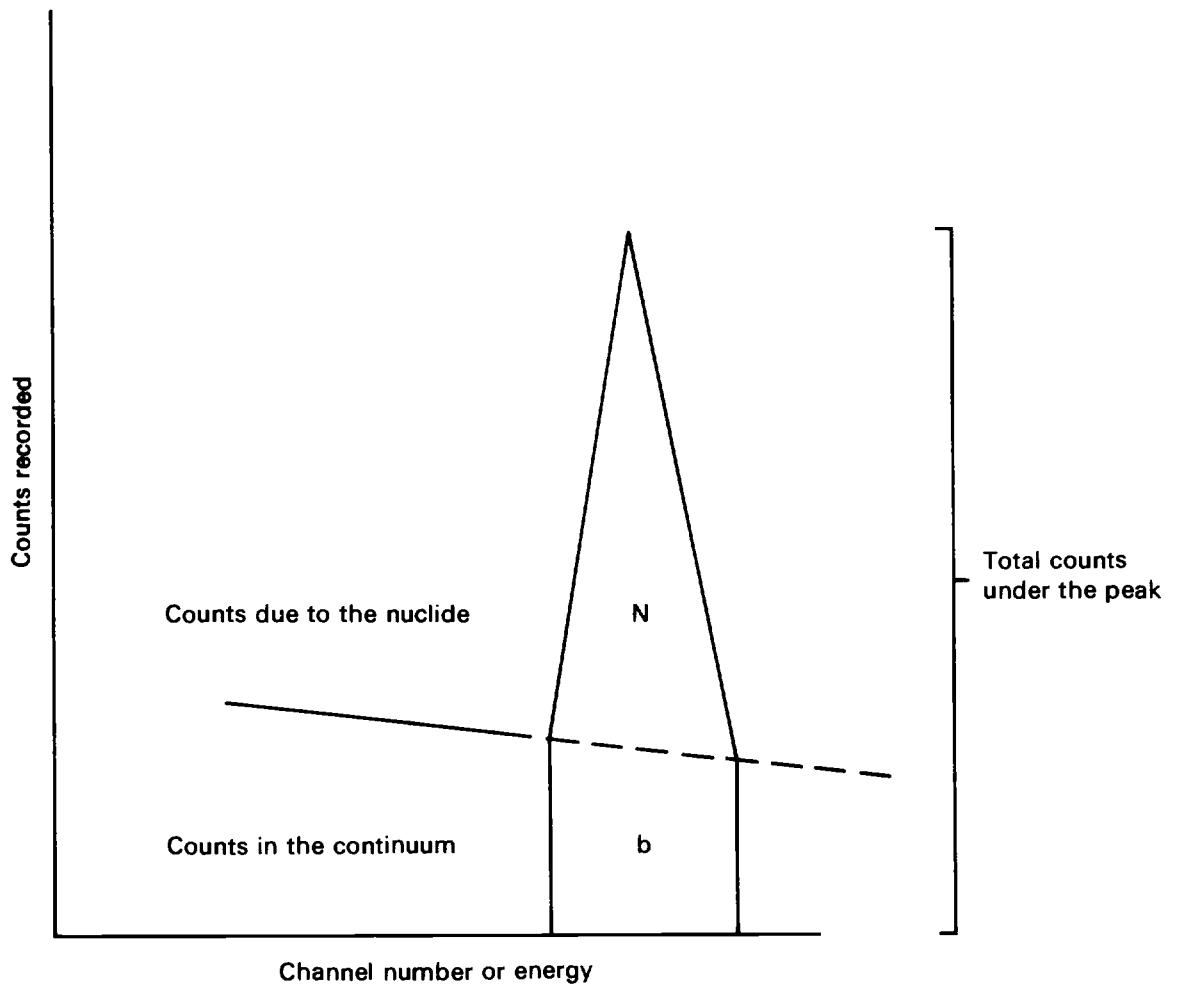


Figure 9 Marinelli type counting beaker

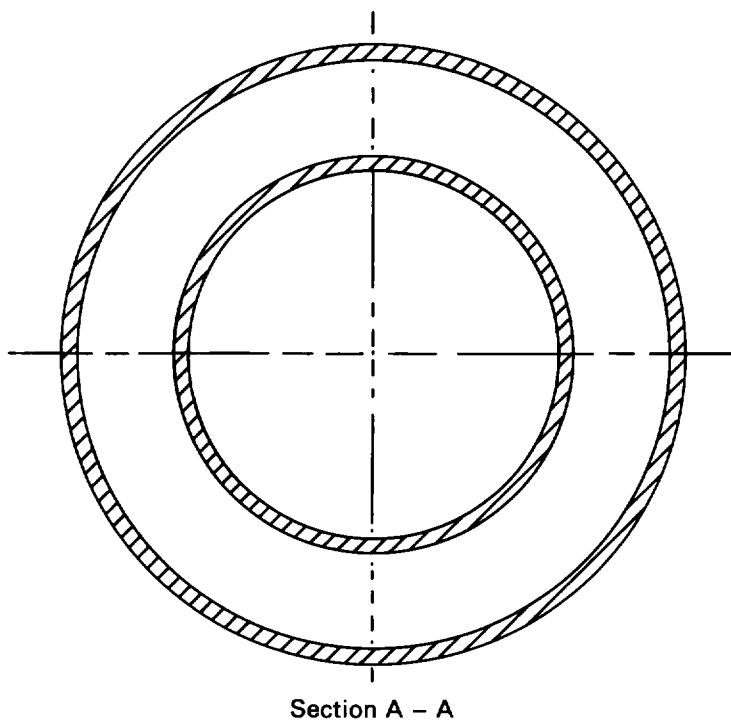
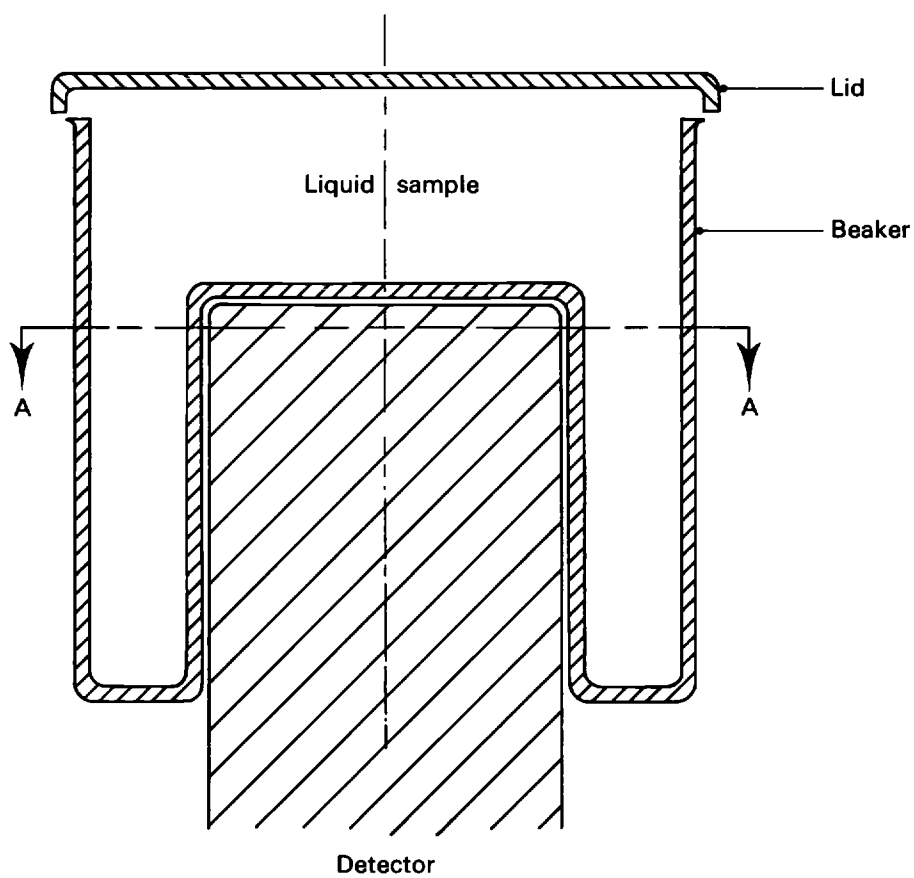


Figure 10

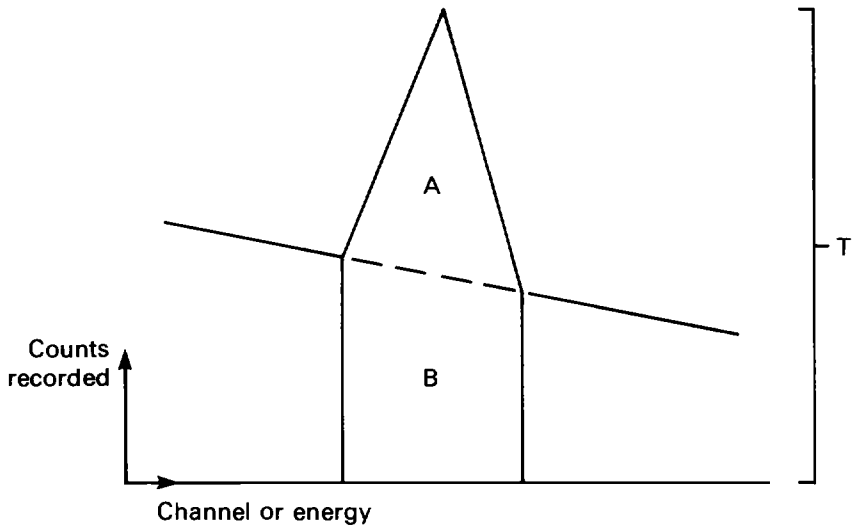


Figure 11

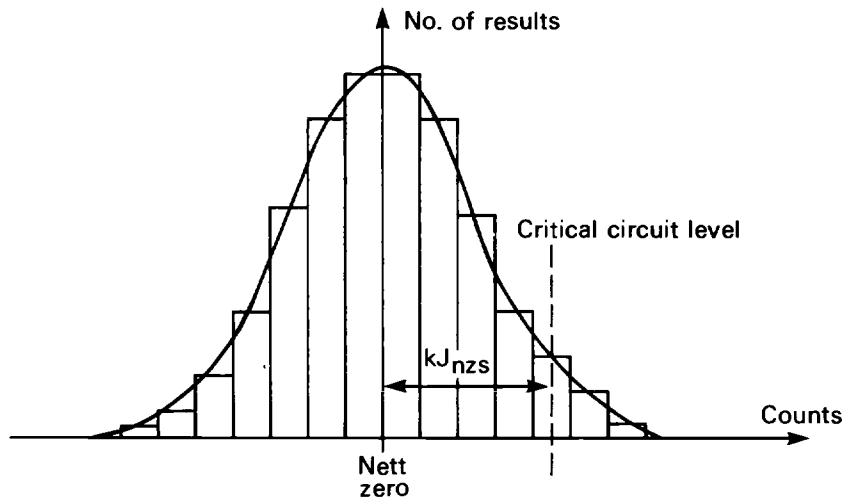


Figure 12

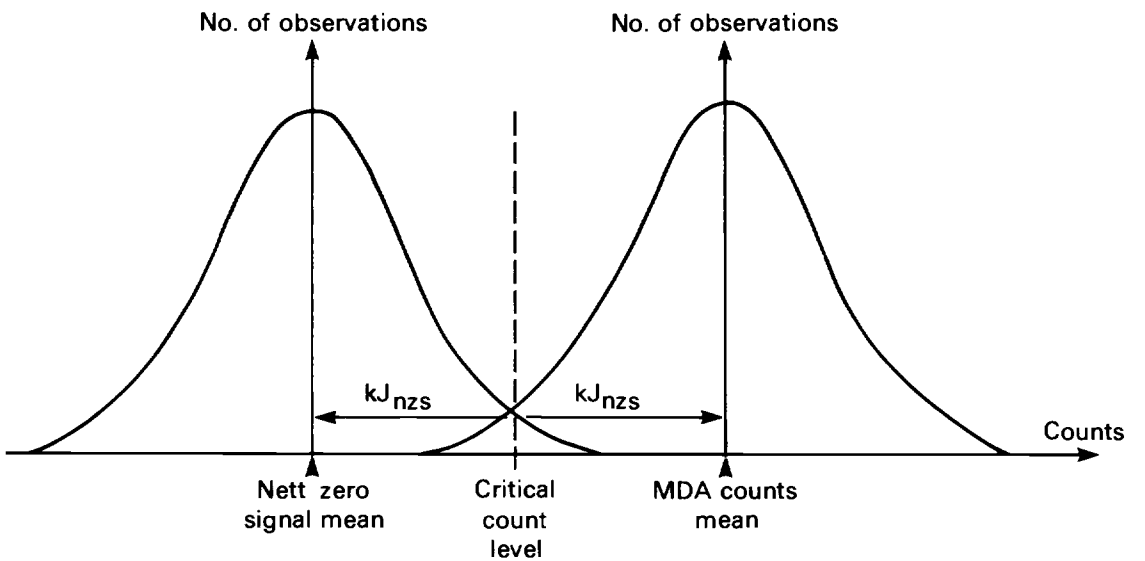


Figure 13 MACC: Critical count for a decision: "detected" or "not detected"

Associated imprecision – one Sigma – %

30 40 50 60 70 80 90

↑
Probability of being wrong

<.3
<.2
<.1
<.05
<.03
<.02
<.01
<.005

Imprecision

K value

Critical count = $K \sqrt{B}$ (B = background count)

Associated imprecision = $\frac{100 \sqrt{2}}{K} \%$

$K = k \alpha \sqrt{2}$

1 2 3 4

K →

Address for Correspondence

Users with comments, queries or suggestions about this method should write to:

The Secretary
The Standing Committee of Analysts
Department of the Environment
Romney House
43 Marsham Street
LONDON
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Department of the Environment

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