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Measurement of radioactivity — Gamma emitting radionuclides — Reference measurement standard specifications for the calibration of gamma-ray spectrometers

Mesurage de la radioactivité — Radionucléides émetteurs gamma — Caractéristiques des étalons de mesure de référence pour l'étalonnage de spectromètres gamma

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Contents

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 2, *Radiological protection*.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Everyone is exposed to natural radiation. The natural sources of radiation are cosmic rays and naturally occurring radioactive substances existing in the Earth itself and inside the human body. Human activities involving the use of radiation and radioactive substances cause radiation exposure in addition to the natural exposure. Some individual activities, such as the mining, use of ores containing naturally radioactive substances and the production of energy by burning coal that contains such substances, can simply enhance the exposure from natural radiation sources. Nuclear installations use radioactive materials and produce radioactive effluent and waste during operations. The use of radioactive materials in industry, medicine, agriculture and research is expanding around the globe.

All these human activities generally also give rise to radiation exposures that are only a small fraction of the global average level of natural exposure. The medical use of radiation is the largest and a growing man-made source of radiation exposure in developed countries. It includes diagnostic radiology, radiotherapy, nuclear medicine and interventional radiology.

Radiation exposure also occurs as a result of occupational activities. It is incurred by workers in industry, medicine and research using radiation or radioactive substances, as well as by passengers and crew during air travel and space travel. The average level of occupational exposures is generally similar to the global average level of natural radiation exposure[10].

As the uses of radiation increase, the potential health risk and the public's concerns may increase. Thus, ionizing radiation exposures are regularly assessed in order to improve the understanding of regional levels and temporal trends of public and worker exposure, to evaluate the components of exposure to provide a measure of their relative importance, and to identify emerging issues that may warrant more attention and scrutiny. While doses to workers are usually directly measured, doses to the public are usually assessed by indirect methods using radioactivity measurements results performed on various sources, including waste, liquid or air effluent, and environmental samples. Environmental samples may include ambient air, soil, surface water, ground water, treated water, vegetation, livestock and game or other biota.

Surveillance programs require financial and technical resources. The program should be designed to acquire data to adequately monitor potential risks. To ensure that the data obtained from radioactivity monitoring programs support their intended use, it is essential in the dose assessment process that stakeholders (the operators, the regulatory bodies, the local information committee and associations, etc.) agree on appropriate data quality objectives, methods and procedures for

— the acquisition, handling, transport, storage and preparation of test samples;

— the test analytical method, and

— for calculating measurement uncertainty.

As reliable, comparable and 'fit for purpose' data are an essential requirement for any public health decision based on radioactivity measurements, international standards of tested and validated radionuclide test methods are an important tool for the production of such measurement results. The application of standards serves also to guarantee comparability over time of the test results and between different testing laboratories. Laboratories apply them to demonstrate their technical qualifications with successful completion of proficiency tests during laboratory intercomparison, two prerequisites to obtain national accreditation. Today, over a hundred international standards, prepared by Technical Committees of the International Organization for Standardization, including those produced by ISO/TC 85, and the International Electrotechnical Commission, are available for application by testing laboratories to measure the main radionuclides.

A reliable determination of the activity concentration of gamma-emitting radionuclides in various matrices is necessary for the assessment of any potential human exposure (public and workers) to the radioactivity of these sources.

Gamma-ray spectrometry is commonly used to determine the activity of gamma-emitting radionuclides. ISO 20042 describes the generic requirements and instrumentation to quantify the activity concentration of gamma-emitting radionuclides in samples after proper sampling, sample handling and test sample preparation in a testing laboratory or directly on site. ISO 20042 also helps testing laboratories to manage the measurement process by setting out the general requirements and methods to calibrate and validate techniques. It forms the basis for measurement tasks using gamma-ray spectrometry, such as those set out in ISO 18589-3, ISO 18589-7, ISO 10703, ISO 13164-2 and ISO 13165-3.

According to ISO 20042 and the above-mentioned ISO standards, reference measurement standards that are traceable to International Standards or national standards are required to calibrate gamma-ray spectrometry systems. The necessity for developing this document originated from the need for standardized specifications for radioactive reference measurement standards used to calibrate gamma-ray spectrometers, as mentioned in those International Standards. Accordingly, traceability of measurement standards to International Standards or national standards is established by use of sources of reference measurement standards that comply with this document.

Measurement of radioactivity — Gamma emitting radionuclides — Reference measurement standard specifications for the calibration of gamma-ray spectrometers

1 Scope

This document specifies the characteristics of solid, liquid or gas sources of gamma emitting radionuclides used as reference measurement standards for the calibration of gamma-ray spectrometers. These reference measurement standards are traceable to national measurement standards.

This document does not describe the procedures involved in the use of these reference measurement standards for the calibration of gamma-ray spectrometers. Such procedures are specified in ISO 20042 and other documents.

This document specifies recommended reference radiations for the calibration of gamma-ray spectrometers. This document covers, but is not restricted to, gamma emitters which emit photons in the energy range of 60 keV to 1 836 keV. These reference radiations are realized in the form of point sources or adequately extended sources specified in terms of activity which are traceable to national standards.

Liquid standards that are intended to be used for preparing extended standards by the laboratories are also within the scope of this document. Reference materials (RMs) produced in accordance with ISO 17034 are out of scope of this document.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2919, *Radiological protection — Sealed radioactive sources — General requirements and classification*

ISO 9978, *Radiation protection — Sealed sources — Leakage test methods*

ISO 12749-2, *Nuclear energy, nuclear technologies, and radiological protection — Vocabulary — Part 2: Radiological protection*

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

IEC 60050-395, *International Electrotechnical Vocabulary — Part 395: Nuclear instrumentation: Physical phenomena, basic concepts, instruments, systems, equipment and detectors*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 12749-2, IEC 60050-395, ISO/IEC Guide 99 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1
activity

<of a source> quantitative indication of the radioactivity of an amount of each radionuclide in a source of reference measurement standard, at reference date

Note 1 to entry: It is expressed as $A = -\mathrm{d}N/\mathrm{d}t$ where $\mathrm{d}N$ is the mean change in the number of nuclei in that energy state due to spontaneous nuclear transformations in the time interval $\mathrm{d}t$.

Note 2 to entry: The special name for the unit of activity is becquerel (Bq), where $1\ \mathrm{Bq} = 1\ \mathrm{s}^{-1}$ and $1\ \mathrm{Ci} = 3{,}7 \times 10^{10}\ \mathrm{Bq}$.

3.2
reference measurement standard
RMS

<of activity>measurement standard designated for the calibration of other measurement standards for quantities of a given kind in a given organization or at a given location

Note 1 to entry: Reference measurement standard (RMS) for activity of a quantity of radionuclide(s) is provided as radioactive material sealed in a container or associated with a material to which it is closely bonded, this capsule or bonding material being strong enough to maintain leak-tightness of the sealed source under the conditions of use and wear for which it was designed.

3.3
working measurement standard
WMS

<of activity>measurement standard used routinely to calibrate or verify measuring instruments or measuring systems

Note 1 to entry: A working measurement standard (WMS) is usually calibrated with respect to a reference measurement standard.

Note 2 to entry: In relation to verification, the terms “check standard” or “control standard” are also sometimes used.

3.4
transfer measurement device

device used as an intermediary to compare measurement standards

3.5
cascade summing

simultaneous detection of two or more photons originating from a single nuclear disintegration that results in only one observed (summed) pulse

3.6
detection efficiency

ratio of the count rate of detected photons to the photon emission rate of the same energy E from a *RMS* (3.2) at actual measurement geometry

3.7
self-absorption

<of a source> absorption of radiation which occurs within the material of the source itself

3.8
uncertainty

standard uncertainty ($k = 1$) unless otherwise stated

Note 1 to entry: The treatment of uncertainties is in accordance with ISO/IEC Guide 98-3.

3.9
homogeneity

<of a volume in respect of a given property> indication of the lack variation of that property over the radioactive region in a volume standard

3.10

instrument check source

single or mixed radionuclide source which have been developed to check the correct functioning of radiation measurement equipment involving gamma-ray spectrometers

Note 1 to entry: Instrument check sources are not used for the calibration of gamma-ray spectrometers.

3.11

metrological traceability

property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement *uncertainty* (3.8)

3.12

bulk density

value in g cm^{-3} obtained by the mass of radioactive matrix material filling a container divided by the volume of the container

4 Metrological traceability of reference measurement standards (RMSs)

National metrology institutes (NMIs) or calibration laboratories should, at their discretion, provide the means whereby reference measurement standards of a specified range of radionuclides may be certified by them. In other words, the NMIs or calibration laboratory should be able to provide traceability documentation for RMSs that they create, use, or measure. NMIs and calibration laboratories that provide measurement standards should be accredited in accordance with ISO/IEC 17025 requirements for calibration of measurement standards.

For those countries signatory to the Mutual Recognition Arrangement (MRA), a certificate of calibration from another participating institute in a second country is recognized as valid in the first country for the quantities, ranges, and measurement uncertainties specified in Annex C of Reference [11].

The RMS provided as a solid sealed source should be fabricated using liquid standards of radionuclides. The activity concentration of such a solution named “standardized solution” is determined by absolute activity measurement (e.g. $4\pi\beta - \gamma$ coincidence counting technique) by NMIs.

Pressurized ionization chambers or germanium semiconductor detectors, which were previously calibrated by use of standardized solutions of several kinds of nuclides are also commonly used to determine the activity concentration of the liquid standard by secondary calibration laboratories.

For a point source RMS [see 5.1 a)], the well-mixed liquid standard shall be gravimetrically deposited onto an infinitesimal small substrate (e.g. ion-exchange resin) and the radioactive material is sealed in the source capsule in accordance with ISO 2919.

The well-mixed liquid standard can also be gravimetrically deposited into the capsule and after the drying process, it is covered to avoid leakage of radioactive material.

The activity of the point source can be determined from the activity concentration of the solution source and deposited mass of the liquid standard expressed in grams. Such sources are also used as the RMS to determine the activity of other point sources of the same nuclide by the comparative measure in the secondary calibration laboratory.

In case of the RMS of wide area sources [see 5.1 b)], the well-mixed liquid standard shall be gravimetrically deposited uniformly onto filter paper and after the drying process, sealed in the source capsule[12]. The activity of the wide area RMS can be determined from the activity concentration of the liquid standard and the deposited mass of liquid standard in grams.

For solid source RMS [see 5.1 c)], the well-mixed liquid standard shall be gravimetrically added to a sorbent (e.g. resin, agar, various adsorbents). After a homogenizing process, the radioactive matter shall be gravimetrically deposited in a container and sealed to prevent leakage. The resulting activity can be determined from the mass of radioactive matter in grams, dilution factor (mass of liquid standard per total volume of liquid standard plus sorbent) and the activity concentration of the master solution.

The uncertainty of the calibrated activity should be estimated taking into consideration the uncertainty caused by the loss of activity during the source fabrication process and the homogeneity of the activity distribution across the radioactive matter.

Organizations that type test or require calibrated instruments to measure radioactivity (e.g. testing laboratory of environmental radioactivity measurements) need to have access to suitable RMSs.

The purpose of a working measurement standard is for in-house routine calibration or confirmation of the detection efficiencies of gamma-ray spectrometers. It shall not be confused with instrument check sources which are only intended to verify that a monitor is operating.

Organizations with a requirement to provide WMSs for the routine calibration or confirmation of the detection efficiencies of gamma-ray spectrometers require access to a transfer measurement device with which to calibrate such sources in terms of activity against a RMS. Where only a few monitors need calibration or a high degree of accuracy is required, RMSs may be used as WMSs.

5 Specification of measurement standards

5.1 General

RMSs are of the following kinds:

a) point source RMSs consisting of an infinitesimal substrate of a known small size (e.g. <1 mm in diameter) comprising the radioactivity homogenously inside of it. The size of the radioactive part is generally less than 10 mm in diameter,

b) wide area source RMSs consisting of a planar radioactive area source (e.g. a filter standard), wherein the activity is uniformly distributed in the radioactive area,

c) solid source RMSs filled with a sealed solid substrate with a known density wherein the activity is homogeneously distributed,

or

d) liquid source RMSs sealed in glass ampoules which contain a liquid, wherein the activity is homogeneously distributed.

Point sources RMSs are used to infer efficiencies for a counting geometry different from measured efficiency obtained by a point source geometry. When calculations are used to determine efficiencies in the use of gamma-ray spectrometry systems based on numerical models, point sources might be also used to characterize individual detectors of users.

Wide area source RMSs and volume sources RMSs are used to determine full-energy peak efficiency as a function of energy, or detection efficiency of a specific gamma-ray or radionuclide at a certain detector-sample geometry that are employed by the user.

Liquid source RMSs sealed in glass ampoules for calibration of instruments are commonly referred to as reference solutions. The reference solutions are used to prepare reference material or measurement standards by the users themselves after opening ampoules. Prepared reference materials or measurement standards are used to determine full-energy peak efficiency as a function of energy, or detection efficiency of a specific gamma-ray or radionuclide at a certain detector-sample geometry that are employed by the user.

RMSs shall be fit for purpose and it shall be the responsibility of the manufacturer to determine and report the radioactive impurities to the extent necessary to ensure that the use of the source is not compromised by emissions from any impurity.

At a minimum, all radioactive impurities in a measurement standard with an activity of at least 1 % of the total source activity, shall be determined and reported. For those sources which might contain radioactive impurities, users should take due account that the relative level of the impurity changes with time and could produce a significant effect on the efficiency calibration.

5.2 Reference measurement standard

5.2.1 General requirements

The general method used to calibrate a spectrometer is to establish the detection efficiency as a function of energy for a defined geometry and energy range. One or more RMSs containing single or multiple radionuclides are used for this purpose. The activity or the activity concentration of the radionuclide(s) in the RMS(s) shall be traceable to national or international standards (see ISO 20042).

In order to comply with the requirements specified in this document, volume source RMSs should match, as closely as possible, the geometry, density and matrix of the samples to be measured in testing laboratories.

RMSs shall be accompanied by a calibration certificate involving a product specification giving the following information:

a) unique RMS identification number;

b) manufacturer and year of manufacture;

c) mass or volume (in grams or litres respectively) of the radioactive solution or matrix in the container or the ampoule (for liquid source RMS or solid source RMS);

d) nature of radioactive matrix (elemental composition) (for liquid source RMS or solid source RMS);

e) bulk density of radioactive matrix (for solid volume source RMS);

f) homogeneity (for solid volume source RMS);

g) Results of the leak test according to ISO 9978;

h) Then, radionuclide;

NOTE Half-life values and other current nuclear data values are provided by Reference [13].

1) activity or activity concentration and its expanded uncertainty ($k = 2$);

2) impurities of an activity of at least 1 % of the total radionuclide activity;

3) reference date [shall be identical for 1) and 2)];

Manufacturers may decide to give further useful information. Additionally, markings on the RMS itself shall indicate the radionuclide and the RMS identification number.

In order to maintain history and owner inventory information, the following optional descriptive information can be appended to the certificate by the owner, including:

- physical description of source;
- owner inventory identification number;
- date of purchase;
- purchased from.

5.2.2 Container and matrix of the solid reference standards

A solid source RMSs should match, as closely as possible, the nature, the volume and the shape of the samples to be measured. The composition or the attenuation coefficient of radioactive matrix shall be determined. Epoxy resin, adsorbent or agar are commonly used to simulate sample materials.

The container of the solid RMS and of the test samples shall be matched as similar as possible. Use of the same container is strongly recommended. Containers made from Polymethyl methacrylate (PMMA), Polyethylene (PE) or Polypropylene(PP) are commonly used to hold samples. The following factors for solid RMS containers shall be considered (see ISO 20042):

- reproducibility of measurements;
- the effect of sample geometry to self-absorption;
- optimization of detection efficiency;
- water-tight containers to reduce the risk of evaporation of solutions;
- hermetically sealed containers for radioactive materials;
- the risk of chemical reactions with the radioactive materials;
- ease of filling (a wide-necked opening is recommended);
- transparency and a clear fill line to ensure consistent fill height;
- robustness if dropped accidently;
- robustness to expansion of the resin;
- absorption of low energy gammas and X-rays in the container material.

NOTE 1 Self-attenuation coefficients for low energy photons (e.g ^{210}Pb (Eγ=46,5 keV)) can become significant for reference standard with high atomic number radionuclides.

NOTE 2 Epoxy resin or agar in polytetrafluoroethylene (PTFE) containers can be peeled off from the inner surface of the container over time.

NOTE 3 In case that users calibrate detectors using solid standards calibrated for "water equivalent activity" and measure water sample, transparent containers cannot be required.

5.2.3 Activity

The source which can provide a spectrum with sufficient counts within a short period is useful. The selection of activity for the RMS should take into consideration the background level of the apparatus, the statistical uncertainty and the dead-time error (see ISO 20042:2019, 8.2). For the calibration of the detector efficiency versus energy with small sample-to-detector distances, an activity of the volume source of 0,1 Bq g^{-1} to 1 000 Bq g^{-1}, depending on the volume of the container, is recommended. In the use of RMSs for efficiency calibration of the spectrometer, activity strength of the source does not affect an accuracy of the calibration as long as it is not so high as to cause pulse pile-up, when pulses arrive closer in time than the pulse resolution time for the system.

The activity or the activity concentration of all types of RMSs shall be stated with a relative expanded uncertainty (k = 2) not exceeding 10 %.

Overall source activity of volumetric sources has to be related to the source volume when the sources are used to calibrate detectors. The source might need sufficient activity concentration for a given detector with a radioactive volume of 5 ml but not so much activity concentration, to preclude overloading a detector, when a volume of 2 000 ml is used.

5.2.4 Homogeneity of radioactivity in the solid reference measurement standard

The homogeneity of solid RMS larger than 100 ml shall be determined by the measurement of activities of at least 4 samples randomly fractionated from the same batch as the radioactive matrix. The mass of samples should be comparable and less than one fourth of the filled mass of the RMS. The homogeneity of a solid RMS shall be expressed as 1 minus the standard deviation of the measured activities (u_n) of the individual samples divided by the mean value of these activities (n), as given by Formula (1):

$$\left(1-\frac{u_n}{n}\right)\times 100(\%) \qquad (1)$$

The homogeneity of a RMS shall be greater than 95 %. In the homogeneity measurement, activities of each fractionated samples should be determined with relative standard uncertainty of counting statistics not exceeding 5 %.

5.2.5 Radionuclides

The general method used to calibrate a spectrometer establishes the detection efficiency as a function of energy for a defined geometry and energy range. One or more liquid RMSs containing single or multiple radionuclides are used for this purpose. Radionuclides with gamma-ray energies ranging from 20 keV to 2 000 keV are selected for multi-radionuclide RMSs.

Selection of the nuclides in the measurement standard may be application-specific. For mixed-gamma detection, a multi-radionuclide standard containing 9 or 10 nuclides from those listed in Table 1 as preferred nuclides is recommended for a measurement standard. For example, measurement standards are usually prepared from a mix of ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{88}Y, and ^{60}Co (^{241}Am may be included in this mix).

As an alternative to the use of a short-lived nuclide, long-lived multiple gamma emitters that are provided as possible alternatives in Table 1 are also applicable to the combination of nuclides employed for a RMS. In this case, users shall be required to carry out true coincidence summing correction to determine the detection efficiency as a function of energy.

For direct comparison with a RMS, the same radionuclide contained in the test sample shall be prepared. This approach is appropriate if the aim of the measurement is to determine the activity of a single radionuclide or if the photon energy is in an energy region that is difficult to calibrate (typically 20 keV to 100 keV). For this approach, the appropriate nuclides shall be requested for the RMS.

For determining the activity of only specific radionuclides or radionuclides emitting gamma or X-rays in the energy region less than 60 keV, a RMS containing the radionuclide of interest should be used. The decay data given in Table 1 are for indicative information only. For those countries signatory to the Mutual Recognition Arrangement (MRA), the data used for calibrations and calibration certificates shall be taken from Reference [13].

Table 1 — Radionuclides for reference measurement standards

Radionuclide	Half-life	Principal photon energy keV	Photons per 100 disintegrations	Comments
Preferred (single or double gamma emitters)				
^{109}Cd	461,9 d	88,033 6	3,66	
^{57}Co	271,81 d	122,060 65 136,473 56	85,49 10,71	γ-X summing may be present
^{139}Ce	137,641 d	165,857 5	79,90	
^{203}Hg	46,594 d	279,195 2	81,48	Volatile
^{51}Cr	27,704 d	320,083 5	9,89	Short half-life

Table 1 *(continued)*

Radionuclide	Half-life	Principal photon energy keV	Photons per 100 disintegrations	Comments
^{113}Sn	115,09 d	391,698	64,97	
^{85}Sr	64,850 d	514,004 8	98,5	Close to 511 keV[1]
^{137}Cs	30,05 years	661,657	84,99	Possible single escape of 1173,23 keV at 662,3 keV
^{54}Mn	312,19 d	834,848	99,975 2	
^{88}Y	106,63 d	898,042 1836,070	93,7 99,346	Cascade summing
^{65}Zn	244,01 d	1115,539	50,22	
^{60}Co	5,271 1 years	1173,228 1332,492	99,85 99,982 6	Cascade summing
^{241}Am	432,6 years	59,540 9	35,92	
Possible alternatives (multiple gamma emitters)				
^{125}Sb	2,758 55 years	427,874 600,597	29,55 17,76	Cascade summing
^{129}I	$16{,}1 \times 10^6$ years	39,578	7,42	For low energy photon calibration
^{133}Ba	10,539 years	356,012 9 79,614 2 80,997 9	62,05 2,63 33,31	Cascade summing
^{152}Eu	13,522 years	121,781 7 344,278 5 1408,013	28,41 26,59 20,85	Cascade summing
^{134}Cs	2,064 4 years	604,720 795,86	97,63 85,47	Cascade summing
^{59}Fe	44,494 d	1099,245 1291,590	56,51 43,23	

NOTE Annihilation quanta at 511 keV, corresponding to the interaction of annihilation photons consecutive to the production of pairs, are often present in spectra due to positron decay, or pair production from the sources or from the background, and may interfere with the measurement of the 514 keV gamma ray of ^{85}Sr due to poor resolution of the detector.

5.3 Working measurement standards (WMSs)

5.3.1 General requirements

The detailed requirements specified for WMSs are the responsibility of the user. Such WMSs may often be prepared by the testing laboratories and due recognition shall be given to any relevant national regulations.

The preparation of custom WMSs shall be performed by technical staff who are experienced in the preparation of radioactivity standards. All quantitative subsequent radioactivity measurements are directly affected by the accuracy of the standard(s) used in the calibration of a spectrometer. Some useful examples of WMSs preparation procedures are available in Reference [12]. A typical example of a WMS preparation procedures is shown in Annex A.

When specifying characteristics of a WMS, the following aspects shall be considered. WMSs shall be:

a) provided in a quantity and variety of sizes to meet the needs of the organization in respect of the routine calibration of its spectrometers,

b) provided with information including the activity at a reference date, the radionuclide and the serial number, and shall be accompanied by a note detailing the geometry for which they have been calibrated and hence should be used,

c) sufficiently robust to withstand day-to-day handling,

d) in compliance as far as possible, with the requirements specified for RMSs in 5.2, avoiding any other conflicting requirements.

5.3.2 Activity

The activity of a WMS should be agreed between the user and the manufacturer. The activity of a WMS shall be stated by the manufacturer and shall be traceable to the national or international standard.

The activity shall have been measured on a reference transfer instrument that has been calibrated using a RMS of the same construction or the similar method of the source preparation.

The activity of a working source needs to be known to the uncertainty specified by the appropriate instrument calibration regulations.

5.3.3 Homogeneity

The homogeneity of a volume source WMS should preferably be the same as specified for a RMS (see 5.2.4).

5.3.4 Radionuclides

WMSs shall be prepared with photon-emitting radionuclides required by the user.

6 Transfer devices

6.1 Transfer measurement device

The recommended type of transfer measurement device is a calibrated gamma-ray spectrometer system using a germanium detector. A single transfer device for multi-radionuclide standards would cover the full range of photon energies proposed in this document.

Scintillation detectors such as NaI(Tl) may also be suitable for calibration of a single-radionuclide standard.

6.2 Calibration

A transfer device shall be calibrated both initially (prior to first use) and at regular time intervals during its working life in accordance with regulatory requirements, codes of practice, or other recommendations. It is recommended that the transfer device be re-calibrated at least annually or, if less frequently, calibrated before any use as a transfer measurement device.

Calibration of a transfer measurement device shall be the responsibility of the organization. Where gamma-emitting radionuclides not available as reference measurement standard are required as WMSs, traceability may be maintained by interpolation using the transfer measurement device.

Annex A
(informative)

Procedures for preparation of working measurement standards from liquid source reference measurement standard

A.1 General

It is recommended that WMSs are prepared using reliable and tested procedures. Sometimes RMSs are not available as an off-the-shelf product in the geometry or matrix required. Under these conditions, it is necessary either to purchase custom-prepared RMSs or to prepare them at the laboratory from liquid source RMSs that are documented to be traceable to NMI. The preparation of aliquots by gravimetric techniques using a calibrated micro-balance that is traceable to NMI of mass is required. Whenever possible, it is recommended that multiple samples are prepared and cross-checked.

This annex shows some examples of procedures for preparation of WMSs based on Reference [12]. Other useful information about WMSs preparation technique are also available in NCRP Report No. 58[14] or other published papers.

A.2 Preparation of liquid source WMSs

a) Obtain a liquid source RMSs volume of at least 5 ml whose radionuclide activity concentration is known. A solution containing stable carrier ions of the various radioelements is required.

b) Dilute the carrier solution, using pure filtered or distilled water, an aqueous solution that has been pH adjusted with a suitable acid or base, as recommended by the supplier of the liquid source RMSs. Chemical data on some typical liquid source RMSs is available in Reference [14].

c) From the activity concentration, calculate the amount of liquid source RMS needed to prepare the custom-made WMSs. In order to avoid unacceptably large uncertainty or unexpected errors, minimum amount of mass of master solution (RMS) to be transferred is determined taking into account the performance of the micro-balance used. A five or six decimal-place balance is preferred since smaller aliquots can be accurately measured.

d) Obtain the tare mass of a small, flat-bottom, plastic container from which small aliquots can be easily dispensed without loss due to splattering or droplets sticking to the lip of the container. If there is a lid, it should be on during all mass measurements. [A miniature plastic squeeze vial (5 ml to 20 ml), with the top heated and drawn out to a capillary, is ideal for removing radioactive solutions from a container and for dispensing small aliquots without evaporation loss. Such a miniature plastic squeeze vial is named pycnometer.] A volumetric flask or plastic (e.g. polyethylene, polypropylene) bottle is one of the best containers for larger sizes.

e) Transfer the master solution (RMS) to the small plastic container. Obtain the mass of the container plus the master solution. The mass from this step minus that from step d) is the mass of the transferred solution.

f) Obtain the tare mass of the sample container that holds the WMS. (Be sure that the container is identical in dimension to the containers to be used for the samples to be analysed.)

g) Transfer to the sample container enough carrier solution to half-fill it.

h) Transfer to this container an amount of master solution that results in a sample that is neither too high or too low in activity for the counting geometry to be used (a counting rate of less than