IEC 61452

Edition 2.0 2021-06

INTERNATIONAL STANDARD

colour inside

Nuclear instrumentation – Measurement of activity or emission rate of gamma-ray emitting radionuclides – Calibration and use of germanium-based spectrometers

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 17.240 ISBN 978-2-8322-9813-8

Warning! Make sure that you obtained this publication from an authorized distributor.

CONTENTS

FOREWORD ... 6
INTRODUCTION ... 8
1 Scope ... 9
2 Normative references ... 9
3 Terms, definitions and symbols ... 10
3.1 Terms and definitions ... 10
3.2 Symbols ... 15
4 Installation of instrumentation ... 16
5 Peak analysis and calibration procedures ... 16
5.1 Energy calibration ... 16
5.2 Energy resolution calibration ... 17
5.3 Peak-finding algorithm ... 17
5.4 Peak position and area measurement ... 17
5.5 Efficiency calibration measurement ... 18
5.5.1 General ... 18
5.5.2 Standardization coefficient for specific radionuclides ... 18
5.5.3 Detector efficiency as a function of energy ... 18
5.5.4 Efficiency function ... 19
6 Gamma-ray measurements with HPGe spectrometers ... 21
6.1 Measurement of gamma-ray energies ... 21
6.2 Measurement of gamma-ray emission rates and radionuclide activities ... 21
6.2.1 General ... 21
6.2.2 Subtraction of interference peaks in the background ... 22
6.2.3 Radioactive decay ... 23
6.2.4 Pulse pile-up (random summing) ... 25
6.2.5 True coincidence (cascade) summing ... 26
6.2.6 Efficiency transfer corrections ... 26
7 Performance tests of the spectrometry system ... 29
7.1 General ... 29
7.2 Multichannel-analyser and digital signal processing clocks ... 29
7.3 DC offset and pole-zero settings ... 29
7.4 Energy calibration ... 29
7.5 Spectrometer efficiency and energy resolution ... 29
7.6 Pulse pile-up (random summing) ... 30
8 Performance tests of the analysis software ... 31
8.1 General ... 31
8.2 Test of automatic peak-finding algorithm ... 31
8.3 Test of independence of peak-area from the gross peak-height to continuum-height ratio ... 33
8.4 Test of the doublet-peak finding and fitting algorithms ... 34
9 Verification of the entire analysis process ... 37
9.1 Assessment of the magnitude of true coincidence summing ... 37
9.2 Deviations in the relative full-energy-peak efficiency ... 40
9.3 Accuracy of the full-energy-peak efficiency ... 41
10 Radionuclide identification ... 41
10.1 General ... 41

10.2 Identification through multipeak analysis and correction for interference from other radionuclides ... 42
10.3 Detection limits ... 42
11 Uncertainties and uncertainty propagation ... 42
12 Mathematical efficiency and correction factors modelling ... 45
12.1 General ... 45
12.2 Mathematical full energy peak efficiency calculations ... 46
12.2.1 General ... 46
12.2.2 Construction of the detector model ... 46
12.2.3 Creation of sample geometries ... 47
12.2.4 Validation of the detector and sample container ... 47
12.2.5 Estimation of uncertainties for Monte Carlo codes for full energy peak efficiencies ... 47
12.3 Estimation of uncertainties from geometry variations ... 48
12.4 Efficiency transfer ... 49
12.5 True coincidence summing corrections ... 49
Annex A (informative) Procedures for characterization of a HPGe gamma-ray spectrometer ... 51
A.1 General ... 51
A.2 Adjustment of the pole-zero cancellation and direct current level ... 51
A.2.1 Rationale for systems using analog electronics ... 51
A.2.2 Adjustment of the pole-zero cancellation ... 51
A.2.3 Adjustment of the direct current (DC) level ... 51
A.3 Adjustment of the lower-level discriminator (LLD), ADC zero and initial energy scale ... 53
A.3.1 Rationale ... 53
A.3.2 Adjustment of the lower-level discriminator ... 53
A.3.3 Adjustment of the ADC zero and initial energy scale ... 53
A.4 Check of the multichannel analyser (MCA) real-time clock ... 54
A.4.1 Rationale ... 54
A.4.2 Instructions ... 54
A.5 Digital electronics ... 55
A.6 Measurement of energy resolution and peak-to-Compton ratio ... 55
A.6.1 Rationale ... 55
A.6.2 Measurement of the energy resolution at 122 keV and 1 332 keV ... 56
A.6.3 Measurement of the peak-to-Compton ratio for ^{60}Co ... 57
A.7 Correction for losses due to counting rate ... 57
A.7.1 Rationale ... 57
A.7.2 Empirical or source method ... 58
A.7.3 Live-time extension method (see [18]) ... 60
A.7.4 Pulser method (see [10], [14] and [17] to [22]) ... 61
A.7.5 Virtual pulser and add "N" counts method ... 65
A.8 Measurement of the full-energy peak efficiency curve ... 65
A.8.1 Rationale ... 65
A.8.2 Measurement of standardization coefficients for specific radionuclides ... 65
A.8.3 Measurement of the detector efficiency versus energy for large sample-to-detector distances ... 66
A.8.4 Measurement of the detector efficiency versus energy for small sample-to-detector distances ... 69
A.9 Preparation of reference sources from standard solutions ... 70

A.9.1 Rationale ... 70
A.9.2 Preparation of standard sources ... 70
A.9.3 Preparation of soil sources ... 71
A.9.4 Preparation of filter sources ... 72
Annex B (informative) Measurement of peak position, net area and their uncertainties ... 73
B.1 General ... 73
B.2 Non-fitting technique ... 73
B.3 Fitting techniques ... 74
Annex C (informative) Formulas for the true coincidence summing correction of cascade gamma-rays ... 76
C.1 Formulas for true coincidence summing correction factors ... 76
C.1.1 General ... 76
C.1.2 True coincidence summing correction factors for a simple decay scheme ... 77
C.1.3 Correction factor for the 591 keV gamma-ray emitted in the decay of ^{154}Eu ... 79
C.1.4 General case ... 84
C.1.5 Total efficiency calculation ... 84
Annex D (informative) Construction of shields for HPGe spectrometers ... 86
D.1 Construction materials ... 86
D.2 Shield design ... 86
D.2.1 General ... 86
D.2.2 Shield design (for detectors counting a variety of low or high activity level samples) ... 86
D.2.3 Shield design for detectors counting only environmental samples of the same size and shape ... 87
D.2.4 Active shielding ... 91
Bibliography ... 94

Figure 1 – Full-energy-peak efficiency as a function of gamma-ray energy ... 20
Figure 2 – $\boldsymbol{\varepsilon f E f}(\mathbf{keV})\mathbf{0,835}$ as a function of gamma-ray energy ... 21
Figure 3 – Specification of times for decay corrections ... 24
Figure 4 – Deviation in measured net peak area as a function of continuum height ... 34
Figure 5 – Deviation in equally sized doublet peak areas for different separations ... 36
Figure 6 – Deviation in unequally sized doublet peak areas for different pulse-height ratios ... 37
Figure 7 – Cascade-summing corrections for a ^{154}Eu 591 keV gamma-ray ... 39
Figure 8 – Partial HPGe gamma-ray spectrum of a long-lived mix ... 40
Figure 9 – Results of Monte Carlo simulation to compute true coincidence summing correction factors: example of ^{134}Cs in different geometrical conditions (point or volume) (filter or water) source at different distances from the HP-Ge detector window ... 50
Figure A.1 – Amplifier output pulses showing correct and incorrect pole-zero cancellation ... 52
Figure A.2 – Distribution of FWHM of spectral peaks as a function of energy ... 56
Figure A.3 – Specification of times for pulse processing by an ADC ... 58
Figure A.4 – Pulse pile-up correction as a function of integral counting rate ... 60
Figure A.5 – Preamplifier and amplifier pulse shapes resulting from different pulser shapes ... 63

Figure A.6 – Gamma-ray spectrum of a mixed radionuclide standard 69
Figure B.1 – Well-resolved peak with continuum 74
Figure C.1 – A three-transition decay scheme 79
Figure C.2 – Partial decay scheme of ^{154}Eu 80
Figure D.1 – Background spectra normalised to the Ge-crystal mass from two HPGe detectors located in the same laboratory 87
Figure D.2 – Expanded view of the background spectrum from the low-background detector in Figure D.1 89
Figure D.3 – Background spectra from (top) a standard HPGe detector and shield, (middle) a low-background HPGe detector and shield and (bottom) an ultra-low-background and shield located underground at a depth of 500 m water equivalent 89
Figure D.4 – The low energy part of a background spectrum from a HPGe detector with a thin (0,4 μm) top dead layer and a 0,5 mm carbon-epoxy window 91
Figure D.5 – Background gamma-ray spectrum recorded without sample and successsive shielding steps to reduce the counting rates 93

Table 1 – Net-peak areas as a function of continuum height 34
Table 2 – Uncertainty propagation for simple functions 44
Table 3 – Uncertainty contributions 45
Table A.1 – Adjustment of energy channels to yield energy equation with zero intercept 53
Table D.1 – List of typical background peaks from the ^{232}Th and ^{226}Ra decay chains in a HPGe detector 88

INTERNATIONAL ELECTROTECHNICAL COMMISSION

____

NUCLEAR INSTRUMENTATION – MEASUREMENT OF ACTIVITY OR EMISSION RATE OF GAMMA-RAY EMITTING RADIONUCLIDES – CALIBRATION AND USE OF GERMANIUM-BASED SPECTROMETERS

FOREWORD

IEC 61452 has been prepared by IEC technical committee 45: Nuclear instrumentation. It is an International Standard.

This second edition cancels and replaces the first edition published in 1995. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) Title modified;

b) Additional information on digital electronics;

c) Information on Monte Carlo simulations;

d) Reference to detection limits calculations.

The text of this International Standard is based on the following documents:

FDIS	Report on voting
45/921/FDIS	45/925/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

IMPORTANT – The "colour inside" logo on the cover page of this document indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

A typical gamma-ray spectrometer consists of a high purity germanium (HPGe) detector with its liquid nitrogen or mechanically refrigerated cryostat and preamplifier, associated to either analog or digital electronic modules including the detector biasing and signal processing (amplification, multichannel conversion and storage) and data-readout devices. The spectrometers include or are associated with computers and their acquisition software. A radiation shield often surrounds the detector to reduce the counting rate from room background radiation for shield construction guidelines). Primary interactions of the photons (X- and gamma-rays) in the HPGe crystal (by photoelectric absorption, Compton scattering or pair production) impart energy to electrons whose energy is finally released by creation of electron-hole pairs. These electrons and holes are collected to produce a pulse whose amplitude is proportional to the energy deposited in the active volume of the HPGe crystal. These pulses are amplified, shaped and sorted according to pulse height to produce a histogram showing, as a function of energy, the number of photons absorbed by the detector. After the accumulation of a sufficient number of pulses the histogram will display a spectrum with one or more peaks with an approximately normal (Gaussian) distribution corresponding to photons that transferred their entire energy to the detector. These are superimposed on continuum constituted by the events related to the partial deposition of energy.

The recorded peak area depends on the emission rate of the gamma-ray and on the detection efficiency of the detector, which is energy dependent. The emission rate, $R(E)$, for a gamma-ray of energy E is determined by dividing the net area, $N(E)$, in the full-energy peak by the measurement live time, T_{L}, and full-energy-peak efficiency, $\varepsilon(E)$, of the detector for the counting geometry used. A curve or functional representation of the full-energy-peak efficiency permits interpolation between available calibration points. Corrections may be needed for:

a) decay of the source during sampling (e.g., with air filters) and counting and/or ingrowth;
b) decay of the source from a previous time to the counting period and/or ingrowth;
c) attenuation of photons within and/or external to the source that is not accounted for by the full-energy-peak efficiency calibration;
d) solid angle correction that is not accounted for by the full-energy-peak efficiency calibration;
e) true coincidence (cascade) summing;
f) loss of pulses due to pulse pile-up (at high counting rates).

NUCLEAR INSTRUMENTATION – MEASUREMENT OF ACTIVITY OR EMISSION RATE OF GAMMA-RAY EMITTING RADIONUCLIDES – CALIBRATION AND USE OF GERMANIUM-BASED SPECTROMETERS

1 Scope

This document establishes methods for the calibration and use of high purity germanium spectrometers for the measurement of photon energies and emission rates over the energy range from 45 keV to approximately 3 000 keV and the calculation of radionuclide activities from these measurements. Minimum requirements for automated peak finding are stated. This document establishes methods for measuring the full-energy peak efficiency with calibrated sources.

Performance tests are described that ascertain if the spectrometer is functioning within acceptable limits. These tests evaluate the limitations of the algorithms used for locating and fitting single and multiplet peaks. Methods for the measurement of and the correction for pulse pile-up are suggested. A test to ascertain the approximate magnitude of true coincidence summing is described. Techniques are recommended for the inspection of spectral analysis results for large errors resulting from true coincidence summing of cascade gamma-rays in the detector. Suggestions are provided for the establishment of data libraries for radionuclide identification, decay corrections, the conversion of gamma-ray emission rates to decay rates and Monte Carlo simulations.

The measurement of X-ray emission rates is not included because different functional fits are required for X-ray peaks, which have intrinsically different peak shapes than gamma-ray peaks. Further, X-ray peaks are complex multiplets (e.g., the K X-rays of Tl include 10 individual components that form four partially resolved peaks). This document does not address the measurement of emission rates of annihilation radiation peaks or single- and double-escape peaks resulting from partial energy deposition in the detector from pair production. Escape peaks may require different fitting functions than comparable full-energy peaks. Further, annihilation radiation and single-escape peaks have a different and larger width than a gamma-ray peak of similar energy. Discussion of acceptable methods for measuring the lower limits of detection as they relate to specific radionuclides is beyond the scope of this document.

The object of this document is to provide a basis for the routine calibration and use of germanium (HPGe) semiconductor detectors for the measurement of gamma-ray emission rates and thereby the activities of the radionuclides in a sample. It is intended for use by persons who have an understanding of the principles of HPGe gamma-ray spectrometry and are responsible for the development of correct procedures for the calibration and use of such detectors. This document is primarily intended for routine analytical measurements. Related documents are IEC 60973 and ISO 20042.

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.

IEC 60050-395:2014, *International Electrotechnical Vocabulary (IEV) – Part 395: Nuclear instrumentation – Physical phenomena, basic concepts, instruments, systems, equipment and detectors*
IEC 60050-395:2014/AMD1:2016
IEC 60050-395:2014/AMD2:2020

IEC 60973, *Test procedures for germanium gamma-ray detectors*

ISO 11929 (all parts), *Determination of the characteristic limits (decision threshold, detection limit and limits of the confidence interval) for measurements of ionizing radiation – Fundamentals and application*

ISO 20042, *Measurement of radioactivity – Gamma-ray emitting radionuclides – Generic test method using gamma-ray spectrometry*

JCGM 100:2008, *Evaluation of measurement data – Guide to the expression of uncertainty in measurement (GUM)*

JCGM 200:2012, *International vocabulary of metrology – Basic and general concepts and associated terms (VIM), 3rd edition 2008 version with minor corrections*