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



IEC 61452:2021-06(en)



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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 17.240

ISBN 978-2-8322-9813-8

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

FOREWORD

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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.

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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*

3 Terms, definitions and symbols

For the purposes of this document, the following terms and definitions apply, as well as those given in IEC 60050-395 and JCGM 200.

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

- IEC Electropedia available at: http://www.electropedia.org/ and IEC Glossary available at: http://std.iec.ch/glossary
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1 Terms and definitions

3.1.1
accuracy
closeness of agreement between a measured quantity value and a true quantity value of a measurand

[SOURCE: JCGM 200:2012]

3.1.2
activity
A
number dN of spontaneous nuclear transitions or nuclear disintegrations for a radionuclide of amount N produced during a short time interval dt, divided by this time interval

Note 1 to entry: The unit is becquerel (Bq).

[SOURCE: IEC 60050-395:2014, 395-01-05]

3.1.3
analog-to-digital converter
ADC
electronic device used to convert the amplitude of a voltage pulse from analog to digital format

3.1.4
ADC conversion gain
number of channels over which the full amplitude span can be spread

Note 1 to entry: Usually 4 096 to 16 384 channels are used for HPGe gamma-ray spectrometry.

3.1.5
attenuation
net loss at the detector of primary photons of a given energy resulting from their interaction with matter either due to the occurrence of scattering or absorption in the sample or in material between the sample and the active volume of the detector

3.1.6
background
spectral data including peaks not caused by the source but rather resulting from radioactive decay occurring in the surrounding environment or from cosmic ray interactions in or adjacent to the detector (see 3.1.11)

3.1.7
calibration
determination of a value that converts a measured number into a desired physical quantity (e.g., pulse-height into photon energy, or counts per second into emission rate)

3.1.8
cascade transitions
gamma-rays in the radioactive decay of a single radionuclide that are emitted sequentially and within the resolving time of the spectrometer

3.1.9
true coincidence summing
simultaneous detection of two or more photons originating from a single radioactive decay that results in only one observed (summed) pulse (also cascade summing)

3.1.10
combined standard uncertainty
standard uncertainty of the result of a measurement when that result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities

Note 1 to entry: See JCGM 100:2008 GUM 1995 with minor corrections.

3.1.11
continuum
part of the pulse-height distribution lying underneath a peak including contributions associated with the source, detector, and measuring conditions that affect the spectral shape

3.1.12
conventional true value <of a quantity>
commonly accepted best estimate of the value of that quantity

Note 1 to entry: This and its associated uncertainty will normally be determined by a national or international transfer standard, or by a reference instrument that has been calibrated against a national or international transfer standard.

3.1.13
counting rate
number of pulses registered by the detector per unit of time being registered in a selected voltage or energy interval (expressed in s^{-1})

3.1.14
crossover gamma-ray
gamma-ray occurring between two non-adjacent nuclear levels

3.1.15
dead time
time during which a counting system is unable to process an input pulse

3.1.16
direct current (DC) level
input or output voltage level on a DC-coupled instrument when there are no pulses present

3.1.17
direct current (DC) offset
difference between a current or voltage level and a reference level

3.1.18
emission intensity per decay
emission intensity and yield
$P(E)$ or $P_\gamma(E)$
probability that a radioactive decay will be followed by the emission of the specified radiation

Note 1 to entry: Gamma-ray emission intensities are often expressed per 100 decays.

3.1.19
energy resolution, full width at half maximum
FWHM
width of a peak at half of the maximum peak height where the baseline is measured from the continuum

3.1.20
energy resolution, full width at tenth maximum
FWTM
width of a peak at one tenth of the maximum peak height where the baseline is measured from the continuum

Note 1 to entry: For a normal (Gaussian) distribution, FWTM is 1,823 times its FWHM.

3.1.21
error
measurement error
measured quantity value minus a reference quantity value

Note 1 to entry: The concept of ‘measurement error’ can be used both:

a) when there is a single reference quantity value to refer to, which occurs if a calibration is made by means of a measurement standard with a measured quantity value having a negligible measurement uncertainty or if a conventional quantity value is given, in which case the measurement error is known, and

b) if a measurand is supposed to be represented by a unique true quantity value or a set of true quantity values of negligible range, in which case the measurement error is not known.

Note 2 to entry: Measurement error should not be confused with production error or mistake.

[SOURCE: JCGM 200:2012]

3.1.22
full-energy peak
photopeak
FEP
peak in the spectrum resulting from the complete (total) absorption of the energy of a photon in the active volume of the germanium crystal and collection of all of the resulting charge

3.1.23
full-energy-peak efficiency
$\varepsilon(E)$
ratio between the number of counts in the net area of the full-energy peak to the number of photons of that energy emitted by a source with specified characteristics for a specified measurement geometry (i.e., source-to-detector distance, source type)

3.1.24
gamma-ray branching ratio
$f(E)$
for a given excited nuclear state, ratio of the emission rate of a particular gamma-ray to the total transition rate from that state

3.1.25
gamma-ray emission rate
$R(E)$
rate at which a gamma-ray of a given energy from the decay of a particular radionuclide is emitted from a given source

Note 1 to entry: The gamma-ray emission rate is the activity times the gamma-ray emission intensity.

3.1.26
live time
T_{L}
time interval of a count during which a counting system is capable of processing input pulses

3.1.27
multichannel pulse-height analyser
MCA
electronic device that records and stores pulses according to their amplitude. It consists of three function segments:

– an ADC to provide a means of measuring pulse amplitude;
– memory registers (one for each channel of the spectrum) to tally the number of pulses having an amplitude within a given voltage increment; and
– an input/output section that permits transfer of the spectral information to other devices such as a computer, or other display or permanent storage media.

3.1.28
peak-to-Compton ratio for the 1 332 keV ^{60}Co peak
ratio of the full-energy-peak height, for ^{60}Co measured at 1 332 keV, to the average height of the corresponding Compton plateau between 1 040 keV and 1 096 keV

3.1.29
pole-zero cancellation
pole-zero adjustment on the shaping amplifier adjusts the zero location of the pole-zero network to exactly cancel the preamplifier output pole and thus provide single-pole (i.e., no under or overshoot) response of the signal pulse at the amplifier output

Note 1 to entry: This operation converts the long-tailed preamplifier pulse to a short-tailed pulse suitable for signal optimization and subsequent pulse-height analysis.

3.1.30
pulse baseline
average of the level from which a pulse departs and to which it returns in the absence of a following overlapping pulse

3.1.31
pulse pile-up
random summing
occurrence of two successive pulses closely associated in time but from separate decays such that they contribute to each other's pulse height and shape

Note 1 to entry: Usually, the system processes the two inputs as a composite single pulse which is stored in a spectral channel different from that at which either of the component pulses would have been stored.

Note 2 to entry: Pulse pile-up is a function of the square of the counting rate and of the amplifier pulse width.

3.1.32
real time counting period
T_r
counting time uncompensated for periods in which an instrument might be unable to respond

Note 1 to entry: Real time of a count equals live time plus dead time.

3.1.33
relative full-energy-peak efficiency <for detector specification>
ε_r
ratio of full-energy-peak efficiency for a point source of ^{60}Co (1 332 keV photons) to that of a NaI(Tl) crystal of 7,6 cm diameter × 7,6 cm high for a source-to-detector distance of 25 cm (that is 1,2 × 10^{-3}).

$$\varepsilon_r = 0{,}833 \times 10^3 \times \frac{n(1332)}{R(1332)} \quad (1)$$

where

$n(1332)$ is the measured full-energy peak counting rate;

$R(1332)$ is the 1 332 keV gamma-ray emission rate from the ^{60}Co source;

0,833 × 10^3 is the reciprocal of the full-energy-peak efficiency of the NaI(Tl) detector at that energy and distance.

3.1.34
sample
radioactive material measured using the HPGe detector to determine the activity and/or emission rate

3.1.35
shaping-time constant
index
indicator of shaped pulse width (width of a shaped pulse at 50 % of its peak height). Unless otherwise specified, that width is $f_{1/2}$

3.1.36
source
calibrated radioactive material used to determine the HPGe detector response

3.1.37
standardization coefficient
$S_c(i,E)$
factor used for the direct conversion of the counts contained under a net full energy peak of a given energy, E, and from a specific radionuclide, i, to the activity of the radionuclide

3.1.38
total efficiency
$\varepsilon_t(E)$
ratio of the number of pulses recorded in the entire energy spectrum and the number of photons emitted by the source of a given energy, E, for a specified source-to-detector distance

3.1.39
uncertainty
non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

[SOURCE: JCGM 200:2012]

3.2 Symbols

A	activity, or decays per second, of a nuclide (Bq)
$B(E)$	net counting rate of an interfering background peak at energy E
C_i	multiplicative correction factor, where i is: a (attenuation), c (true coincidence summing), d (decay), or r (pile-up)
e	2,71828... base of natural or Naperian (or Napierian) logarithms, ln
$f(E)$	fraction of transitions from a level which produce gamma-rays with energy E
$N(E)$	net counts in a full-energy peak due to a gamma-ray of energy E
$n(E)$	net counting rate in a full-energy peak due to a gamma-ray of energy E
$P(E)$	probability per decay or emission intensity per decay for a gamma-ray with energy E
$R(E)$	emission rate of gamma-rays with energy E by nuclei in a sample
$S_c(i, E)$	fraction of decays of radionuclide, i, in a sample which lead to counts in a full-energy peak corresponding to a gamma-ray of energy E
$f_{1/2}$	shaping-time constant (width of a shaped pulse at 50 % of its peak height)
$T_{1/2}$	half life
λ	decay constant, $\lambda = \ln(2)/T_{1/2}$
T_L	live-time counting period
T_r	real-time counting period
T_d	decay time from a reference time to the start of the counting period
$\alpha_K(E)$	K-shell internal conversion coefficient: the ratio of transitions of energy E that occur by K internal conversion to those that emit gamma-rays
$\alpha_t(E)$	total internal conversion coefficient: the ratio of transitions of energy E that occur by internal conversion to those that emit gamma-rays
$\varepsilon(E)$	full-energy-peak efficiency for gamma-rays of energy E
ε_r	relative full-energy-peak efficiency for detector specification
$\varepsilon_t(E)$	total efficiency for detecting a gamma-ray of energy E and producing a pulse with a height greater than the noise level of the amplifier
ω_K	K-shell fluorescence yield: the fraction of atomic K vacancies which lead to K X-rays
Π	operator that takes the product of the correction factors