SLOVENSKI STANDARD

SIST EN 17462:2021

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Krma: metode vzorčenja in analize - Določevanje radionuklidnega joda-131, cezija-134 in cezija-137 v krmi

Animal feeding stuffs: Methods of sampling and analysis - Determination of the radionuclides Iodine-131, Caesium-134 and Caesium-137 in feed

Futtermittel: Probenahme- und Untersuchungsverfahren - Bestimmung der Radionuklide Jod-131, Cäsium-134 und Cäsium-137 in Futtermittel

Aliments des animaux : Méthodes d'échantillonnage et d'analyse - Détermination des radionucléides iode 131, césium 134 et césium 137 dans les matières premières et aliments composés pour animaux

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Animal feeding stuffs: Methods of sampling and analysis - Determination of the radionuclides Iodine-131, Caesium-134 and Caesium-137 in feed

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COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (EN 17462:2021) has been prepared by Technical Committee CEN/TC 327 “Animal feeding stuffs - Methods of sampling and analysis”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2021, and conflicting national standards shall be withdrawn at the latest by October 2021.

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Introduction

This document describes a method for ^{131}I, ^{134}Cs and ^{137}Cs massic activity determination (Bq/kg) in animal feeding stuffs. It was initiated by Directorate General for Health and Food Safety (DG SANTE) of the European Commission following the accident in the Fukushima Daiichi nuclear power plant in March 2011. The event highlighted the need for standardized measurements of the three most common radioactive contaminants following such type of nuclear accident.

The most commonly used method for identification and quantification of these radionuclides in animal feeding stuffs samples is high-resolution gamma-ray spectrometry. As this is a secondary measurement method based on analysis of photopeaks of the emitted gamma rays, care should be taken to use appropriate energy and efficiency calibrations for the detector and test portion used. This method of massic activity determination is described in the present document.

1 Scope

This document describes a method for determination of the massic activity (Bq/kg) of ^{131}I, ^{134}Cs and ^{137}Cs in animal feeding stuffs in monitoring laboratories.

General guidance on the preparation of feed samples and the measurement of the three radionuclides ^{131}I, ^{134}Cs and ^{137}Cs by high resolution gamma-ray spectrometry is provided. The current document aims to be complementary to existing standards. More information on sample preparation, moisture content determination and gamma-ray spectrometry can be found in specific standards referred to in this document. For example, generic advice on the equipment selection, detectors and quality assurance for gamma-ray spectrometry can be found in ISO 20042 [4].

The method was fully statistically tested and evaluated in a collaborative trial comprising five animal feeding stuff samples for the radionuclides ^{131}I, ^{134}Cs and ^{137}Cs. Details on the successfully tested working range for each of the examined radionuclides are described in Annex C.

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 cited edition applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 662, *Animal and vegetable fats and oils — Determination of moisture and volatile matter content (ISO 662)*

EN ISO 665, *Oilseeds — Determination of moisture and volatile matter content (ISO 665)*

EN ISO 712, *Cereals and cereal products — Determination of moisture content — Reference method (ISO 712)*

EN ISO 6497, *Animal feeding stuffs — Sampling (ISO 6497)*

EN ISO 6540, *Maize — Determination of moisture content (on milled grains and on whole grains) (ISO 6540)*

EN ISO 11929-1:2019, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 1: Elementary applications (ISO 11929-1)*

ISO 771, *Oilseed residues – Determination of moisture and volatile matter content*

ISO 6496, *Animal feeding stuffs — Determination of moisture and other volatile matter content*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN ISO 6497 and the following apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
background
spectrum recorded by the gamma-ray detector when no sample is measured

Note 1 to entry: The spectral data, including full energy peaks, in such a spectrum is resulting from radioactive decay occurring in the environment surrounding the detector (including the cosmic ray interactions) or in the detector.

3.2
background continuum
events in the spectrum that form a smooth curve onto which the photopeaks are superimposed

Note 1 to entry: The continuum may arise from gamma-rays scattered inside the test sample or any surrounding materials, from cosmic radiation or from radionuclides in the surrounding materials.

[SOURCE: ISO 20042:2019, 3.1 [4]]

3.3
blank sample
sample, liquid or solid, with very low to no activity for radiation of the same type and region of interest, with a mass and a composition as close as possible to those of the test sample

[SOURCE: EN ISO 19581:2020, 3.1 [1]]

3.4
dead time
time during spectrum acquisition (real time) during which pulses are not recorded or processed

Note 1 to entry: Dead time is given by real time minus live time.

Note 2 to entry: The time is given in seconds.

[SOURCE: ISO 20042:2019, 3.5 [4]]

3.5
efficiency transfer
detection efficiency transfer
calculation that enables the user to establish the value of the detection efficiency for a given gamma-ray peak in the spectrum of the test portion, when only the detection efficiency from an experimental calibration with a reference source that may have a different composition, density and/or geometry compared to the test portion is known

3.6
high resolution gamma-ray spectrometry
energy resolution obtained with a Ge(Li) or an HPGe detector

Note 1 to entry: This definition is specific to gamma-ray spectrometry.

3.7
laboratory sample
sample as prepared (from the lot) for sending to the laboratory and intended for inspection or testing

[SOURCE: EN ISO 6498:2012, 2.1.2 [2]]

3.8
live time
time during which pulses are processed during an acquisition (real) time

Note 1 to entry: The time is given in seconds.

[SOURCE: ISO 20042:2019, 3.12 [4]]

3.9
photopeak
full energy peak (FEP)
peak observed above the background continuum in a gamma-ray spectrum due to events that deposit the full energy of the photon in the detector material, usually approximately Gaussian in shape

[SOURCE: ISO 20042:2019, 3.17 [4]]

3.10
real time
time taken to acquire a spectrum

Note 1 to entry: The time is given in seconds.

[SOURCE: ISO 20042:2019, 3.19 [4]]

3.11
sample holder
device that is specially designed to enable the placement of a given sample container in a well-defined position on top of a specific detector

3.12
spectrometry system
complete assembly of the sensor and associated pulse-processing electronics that converts the gamma-rays detected by the sensor into a pulse-height spectrum

[SOURCE: ISO 20042:2019, 3.22 [4]]

3.13
test portion
quantity of material drawn from the test sample (or from the laboratory sample if both are the same)

[SOURCE: EN ISO 6498:2012, 2.1.4 [2]]

3.14
test sample
subsample or sample prepared from the laboratory sample and from which test portions will be taken

[SOURCE: EN ISO 6498:2012, 2.1.3 [2]]

3.15
true coincidence summing
TCS
coincidence summing
cascade-summing
simultaneous detection of two or more gamma-rays in the spectrometry system, due to the emission of a cascade of gamma-rays in the decay of a single nucleus in the test sample

[SOURCE: ISO 20042:2019, 3.24 [4]]

4 Symbols and abbreviations

4.1 Symbols

For the purposes of this document, the following symbols apply:

Symbol	Name of quantity	Unit
A	activity of a reference radionuclide emitting photons of energy E in the calibration source, at the time of calibration	Bq
a	annum (year), the tropical year which is approximately equal to 365,2422 d	year
a_{m}	massic activity at energy E of a radionuclide in the sample	Bq/kg
$a_{\mathrm{m,605}}$; $a_{\mathrm{m,796}}$	massic activity of ^{134}Cs obtained using the gamma-ray of energy i, which is either 604,72 keV or 795,86 keV	Bq/kg
a'_{m}	massic activity of ^{134}Cs based on the weighted mean calculation including the two major gamma rays of this radionuclide	Bq/kg
a^*_{m}	decision threshold	Bq/kg
$a^\#_{\mathrm{m}}$	detection limit	Bq/kg
$\tilde{a}$	true value of massic activity at energy E of a radionuclide in the sample	Bq/kg
d	day (1 day = 86 400 s)	day
$f_{\mathrm{att}(E)}$	factor to correct for gamma-ray attenuation within the test portion (self-attenuation)	-
f_{d}	factor to correct for decay between the reference time and the start of the measurement and during the measurement NOTE 1 The latter is important for short-lived radionuclides like ^{131}I.	-
f_{d1}	is the factor to correct for decay between the reference time and the start of the measurement	-
f_{d2}	is the factor to correct for decay during the measurement	-
f_E	composite correction factor for the gamma ray with energy E considering all necessary corrections as shown in Formula (6)	-
f_{g}	factor to correct for geometry differences	-
$f_{\mathrm{tcs},E}$	factor to correct for true coincidence summing effects NOTE 2 In this case, this is only applicable to 134Cs.	-
k	coverage factor	-

Symbol	Name of quantity	Unit
m	quantity of the test portion	kg
m_c	corrected quantity of the test portion	kg
m_d	dry mass of the moisture content determination portion	kg
m_f	fresh mass of the test portion	kg
m_w	fresh mass of the moisture content determination portion	kg
N	number of gamma rays used for the calculation of massic activity for ^{134}Cs	-
$n_{b,E}$	number of counts in the net area of the photopeak at energy E, in the background spectrum	-
$n_{N,E}$	number of counts in the net area of the photopeak at energy E, in the test portion spectrum	-
P_E	probability (per 100 decays) of the emission of a gamma ray with energy E by a radionuclide NOTE 3 The probability can be expressed in percentage (%) or in absolute values.	-
$r_{N,E}$	net count rate in the full energy peak at energy E	s^{-1}
T	temperature	°C
t_b	background spectrum live time	s
t_g	test portion spectrum live time	s
t_i	time elapsed between the reference time and the start of the measurement NOTE 4 It will have a negative value when the measurement was started before the reference time and a positive value when the measurement was started after the reference time.	s
t_r	test portion spectrum real time	s
t_s	calibration spectrum live time	s
$t_{1/2}$	half-life of a radionuclide	s
$U(a_m)$	expanded uncertainty with coverage factor k calculated as U = k × u	Bq/kg
$u(a_m)$	standard uncertainty of the massic activity	Bq/kg
$u(a_{m,605})$; $u(a_{m,796})$	standard uncertainty of the massic activity calculated for the two most intense gamma rays (604,72 keV and 795,86 keV) of ^{134}Cs	Bq/kg
$u(n_{b,E})$	uncertainty of the net number of counts in the photopeak at energy E in the background spectrum	-
$u(n_{N,E})$	uncertainty of the net number of counts in the photopeak at energy E in the test portion spectrum	-
$u(r_{N,E})$	uncertainty of the net count rate	-
u_{rand}	random uncertainty component	-
$u_{rel}(A)$	relative uncertainty of the activity of a reference radionuclide emitting photons of energy E in the calibration source, at the time of calibration	-

Symbol	Name of quantity	Unit
$u_{\mathrm{rel}}(f_d)$	relative uncertainty of the factor to correct for decay between the reference time and the start of the measurement and during the measurement	-
$u_{\mathrm{rel}}(f_E)$	relative uncertainty of the composite correction factor for the gamma ray with energy E considering all necessary corrections as shown in Formula (6)	-
$u_{\mathrm{rel}}(f_{tcs,E})$	relative uncertainty of the factor to correct for true coincidence summing effects	-
$u_{\mathrm{rel}}(m)$	relative uncertainty of the quantity of the test portion	-
$u_{\mathrm{rel}}(P_E)$	relative uncertainty of the probability (per 100 decays) of the emission of a gamma ray with energy E by a radionuclide	-
$u_{\mathrm{rel}}(r_{N,E})$	relative uncertainty of the net count rate	-
$u_{\mathrm{rel}}(\varepsilon_E)$	relative uncertainty of the detection efficiency at energy E for the specific measurement geometry and detector used	-
u_{sys}	systematic uncertainty component	-
u_{tot}	total uncertainty calculated based on random and systematic components	-
$u(w)$	total standard uncertainty for coverage factor w	-
$u_{\mathrm{rel}}(w)$	relative value of total standard uncertainty for coverage factor w	-
$\tilde{u}(\tilde{a}_{\mathrm{m}})$	standard uncertainty of a_{m} as a function of its true value	Bq/kg
v_{605}; v_{796}	weighting factor for the calculation of massic activity of ^{134}Cs using the two most intense gamma rays (604,72 keV and 795,86 keV)	-
w	calibration factor	-
ε_E	detection efficiency at energy E for the specific measurement geometry and detector used	-
λ	decay constant of a radionuclide	s^{-1}

4.2 Abbreviations

ALARA	As low as reasonably achievable
FEP	Full energy peak
FWHM	Full width at half maximum
HPGe	High purity germanium
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
TCS	True coincidence summing