

13.1.06A

AOAC Official Method 996.05 Cesium-134 and Cesium-137 in Foods

γ-Ray Spectrometric Method
First Action 1996
Final Action 1998

(Applicable for determination of long-life radiocesiums in foods at levels >100 Bq/kg or >100 Bq/L. Method is also applicable to foods for which “fresh” fission products [e.g., Ba-La-140, I-131, etc.] have decayed. Method is not applicable to foods with unknown composition of radionuclides and/or not well separated peaks. The method is not appropriate for Cs-134 determinations if activity ratio between Cs-137 and Cs-134 is >10. See Table 996.05 for results of interlaboratory study.)

Caution: See Appendix B, safety notes on radioactive chemicals.

A. Principle

Test portion is placed onto shielded Tl-activated NaI-scintillation detector connected with multichannel *γ*-ray spectrometer. Photons are counted under known geometrical conditions. After defining ranges of photon energy for Cs-134 (peak maximum 796 keV) and Cs-137 (peak maximum 662 keV), area counts for each radionuclide are measured, and Cs-134 and Cs-137 activity concentrations are manually calculated.

B. Apparatus

(a) *Multichannel γ -ray spectrometer.*—Equipped with 4096 memory channels and analog digital convertor (ADC) gain (Canberra Industries, 800 Research Parkway, Meriden, CT 06540, USA, or equivalent).

(b) *Shielded Tl-activated NaI scintillation detector.*—e.g., 3 × 3 in.

(c) *Lead castle.*—Thickness, 5 cm; external diameter, 22 cm; internal diameter, 12 cm.

(d) *Counting vessel.*—Plastic; maximum capacity, 520 mL.

C. Reagents

Use deionized or “aged” H₂O, or tap H₂O free of radon and its decay products. Radionuclide standards are available from AEA Technology, 40 North Ave, Burlington, MA 01803, USA; 329 Harwell, Didcot, Oxfordshire OX11 0RA, United Kingdom.

(a) *Cs-134 stock standard solution.*—Containing known *γ*-ray activity. Composition, concentration, and stability according to manufacturer’s specification. Store in air-tight closed bottle and avoid evaporation of solvent (H₂O).

(b) *Cs-137 stock standard solution.*—Containing known *γ*-ray activity. Composition, concentration, and stability according to manufacturer’s specification. Store in air-tight closed bottle and avoid evaporation of solvent (H₂O).

(c) *γ -Ray reference standard solution.*—Solution of radionuclides with relatively long half-lives, e.g., Cs-137, Co-60, or Y-88 containing ca 185 kBq radioactivity; for detector energy calibration. Composition, concentration, and stability according to manufacturer’s specification. Store in air-tight closed bottle and avoid evaporation of solvent (H₂O).

(d) *HCl-CsCl solution.*—0.1M HCl containing 100 μ g CsCl/mL with stable Cs isotope as carrier reagent.

Dissolve all standards in this HCl-CsCl solution.

D. Determination

(a) *Calibration of γ -ray energy spectrum.*—For manual calibration of *γ*-ray energy spectrum. Perform *γ*-ray energy calibration procedure according to manufacturer’s instructions using photon energy spectrum recorded and visualized with multichannel *γ*-ray spectrometer, **B(a)**. Use at least 2 radionuclides with peak energies sufficiently different (e.g., Co-60 or Y-88 with Cs-137). (*Note:* Identification of some peaks in energy spectrum will be difficult due to low resolution of NaI (Tl) crystal. NaI scintillation detectors should have resolution of Cs-137 measurements of 7–9%. Contact supplier (Canberra, or equivalent) if resolution is >9%. Check resolution periodically.)

Adjust *γ*-ray spectrometer according to manufacturer’s instructions. Adjust spectrometer for each measurement, so one channel is ca 2 keV. The 662 keV peak of Cs-137 must fall in channel 331. If 662 keV peak differs more than 2 channels from channel 331 (i.e., >4 keV), proceed with *γ*-ray energy calibration procedure as follows: Prepare *γ*-ray reference calibrating solution by diluting 1 mL *γ*-ray reference standard solution, **C(c)**, in counting vessel, **B(d)**, to ca 250 mL with H₂O. Place vessel over detector, **B(b)**, and count photons 900 s (15 min). Keep *γ*-ray reference calibrating solution for additional measurements.

(b) *Counter efficiency.*—Prepare calibrating standard solution as follows: Accurately pipet known amount of *γ*-ray activity of radionuclide stock standard solution *i* [e.g., 50 μ L Cs-134 stock standard solution, **C(a)**, and/or 50 μ L Cs-137 stock standard solution, **C(b)**] into counting vessel, **B(d)**. Dilute solution with H₂O to 250 mL. Place vessel in lead castle, **B(c)**, over detector, **B(b)**, and adjust photon energy range *j* of interest. [*Note:* In case of no overlapping peaks, scanning range should cover the entire peak plus 3 channels on each side. In case of overlapping peaks, scanning range should cover both overlapped peaks and 3 channels on each side. For test samples containing Cs-134 and Cs-137, recommended energy ranges are: For Cs-134 with separated peak at 795 keV, channel 358–440 (716–880 keV); for Cs-137, channel 258–357 (516–714 keV) covering range of both Cs-134 and Cs-137 peaks. *γ*-Ray energy maxima for Cs-134 and Cs-137 are 796 and 662 keV, respectively.]

Count photons emitted from calibrating standard solution at least 900 s (*t_s*). Register number of area counts [*N(s)_{ij}*] of working standard solution *i* for photon energy range *j*. Calculate counter efficiency of calibrating standard solution from **D(b)** for photon energy range *j* as in **E, Calculations**. If necessary, repeat calibration for small quantities of standards or test samples using known geometry caused by different counting volume. Check counter efficiency and energy calibration periodically, using *γ*-ray reference calibrating solution from **D(a)**.

(c) *Test sample activity.*—Homogenize and weigh aliquot of test sample into counting vessel, **B(d)**. Determine counter geometry of test sample (i.e., its volume *v*; mL) and record its mass (*m*; kg). Before performing quantitative analysis, adjust energy range *j* for each radionuclide as in (b). Place counting vessel in lead castle over NaI(Tl) detector, and count photons at least 900 s (*t_m*) to obtain desired number of counts. Establish total area count numbers [*N(u)_{ij}*] from radionuclide (*i*) in test sample (*u*) for energy range *j*.

E. Calculations

(a) *Counter efficiency of calibrating standard solution.*—Calculate counter efficiency (ϵ_{ij} ; %) of radionuclide *i* for given count ge-

Table 996.05 Interlaboratory study results for detection of Cs-134 and Cs-137 in foods by γ -ray spectrometric method^a

Sample	Cs level, Bq/kg	Mean, Bq/kg	s_r , Bq/kg	RSD _r , %	s_R , Bq/kg	RSD _R , %	r^b , Bq/kg	R^c , Bq/kg
Cs-134								
Herbs	ca 100	104	21	19.7	49	46.7	57	136
Honey ^d	ca 0–100	121	14	11.7	18	14.9	39	50
Milk ^e	ca 300	337	15	4.3	36	10.7	41	101
Water	ca 65	69	8	11.9	15	21.6	23	42
Cs-137								
Herbs	ca 1110	1130	54	4.8	80	7.1	152	225
Honey	ca 200–250	23	4.4	2.0	8.8	4.1	12	25
		236	17	7.3	17	7.4	48	49
Milk	ca 50	550	37	6.7	37	6.7	104	104
Water	ca 370	369	12	3.3	15	4.0	34	41

^a Based on results received from 13 laboratories.

^b $r = 2.8 \times s_r$.

^c $R = 2.8 \times s_R$.

^d Tested at 2 levels: low and high. Table presents statistical analysis from testing high level, results obtained from testing lower level were below determination level (<100 Bq/kg or <100 Bq/L) under given experimental conditions.

^e Information presented in Bq/L except for RSD values.

ometry (v ; mL) observed in photon energy range j for calibrating standard solutions, as follows:

$$\epsilon_{i,j}, \% = \frac{N(s)_{i,j}}{t_s \times A(s)_i} \times 100$$

where $N(s)_{i,j}$ = number of individual counts of calibrating standard solution i observed in energy range j , in area counts; t_s = counting time corrected for read time of calibrating standard solution i , in s; $A(s)_i$ = activity of calibrating standard solution i with known count geometry (v ; mL) observed in energy range j , in Bq.

(b) *Activity of test sample.*—(1) *One radionuclide.*—Calculate activity $[A(u)_i; \text{Bq}]$ of radionuclide i in test sample (u) for given count geometry observed in photon energy range j for calibrating standard solutions, as follows:

$$A(u)_i = \frac{N(u)_{i,j}}{t_m \times \epsilon_{i,j} \times 0.01}$$

where $N(u)_{i,j}$ = number of individual area counts of radionuclide i observed in photon energy range j ; t_m = time of counting of radionuclide i in test sample, in s.

Calculate activity concentration $[A(u)_i(m) \text{ or } A(u)_i(v); \text{Bq/kg or Bq/L}]$ of radionuclide i in test sample (u) as follows:

$$A(u)_i(m) = \frac{A_i}{m}$$

or

$$A(u)_i(v) = \frac{A_i}{v}$$

where m = mass of test portion, in kg; v = volume of test portion, in L; A_i = activity of radionuclide i for given count geometry (v ; mL), in Bq. Report results to the nearest Bq/kg or Bq/L.

(2) *Two radionuclides.*—Calculate counting rates (R_1 and R_2 ; area count/s) for both radionuclides a and b in their respective photon energy ranges 1 and 2 , as follows:

$$R_1 = \frac{N_1}{t_m}$$

$$R_2 = \frac{N_2}{t_m}$$

where N_1, N_2 = total number of individual area counts of both radionuclides observed in photon energy ranges 1 and 2 , respectively. Calculate activities of radionuclides a and b $[A(u)_a \text{ and } A(u)_b, \text{ respectively; Bq}]$ for given count geometry observed in their respective photon energy ranges 1 and 2 , from following equations:

$$R_1 = [A(u)_a \times \epsilon_{a,1} \times 0.01] + [A(u)_b \times \epsilon_{b,1} \times 0.01]$$

$$R_2 = [(A(u)_a \times \epsilon_{a,2} \times 0.01) + [A(u)_b \times \epsilon_{b,2} \times 0.01]$$

where $\epsilon_{a,1}$ = counter efficiency of radionuclide a for given count geometry observed in photon energy range 1 ; in percent; $\epsilon_{a,2}$ = counter efficiency of radionuclide a for given count geometry observed in photon energy range 2 ; in percent; $\epsilon_{b,1}$ = counter efficiency of radionuclide b for given count geometry observed in photon energy range 1 , in percent; $\epsilon_{b,2}$ = counter efficiency of radionuclide b for given count geometry observed in photon energy range 2 , in percent. [Note: If combination of Cs-134 (radionuclide a) and Cs-137 (radionuclide b) is measured, counter efficiency of one of these radionuclides (Cs-137) will be zero in photon energy range 2 ($\epsilon_{b,2} = 0$).] Calculate activity concentrations $[A(u)_i(m) \text{ or } A(u)_i(v); \text{Bq/kg or Bq/L}]$ of radionuclides a and b in test sample (u) as follows:

$$A(u)_i(m) = \frac{A_i}{m}$$

or

$$A(u)_i(v) = \frac{A_i}{v}$$

where A_i = activity of radionuclide a or b for given count geometry (v ; mL) observed in photon energy ranges 1 and 2, in Bq. Report results to the nearest Bq/kg or Bq/L.

References: *J. AOAC Int.* **78**, 1244(1995); **80**, 545(1997).

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