

SECTION 4
GAMMA EMITTING RADIONUCLIDES IN DRINKING WATER
METHOD 901.1

1. Scope and Application

- 1.1 This method describes the use of gamma spectroscopy for the measurement of gamma photons emitted from radionuclides without separating, them from the sample matrix. This technique makes It possible to ascertain whether a hazardous concentration of a specific gamma emitter is present in a drinking water sample.
- 1.2 The limits set forth in PL 93-523, 40 FR 34324 recommend that in the case of man-made radionuclides, the limiting concentration is that which will produce an annual dose equivalent to 4 mrem/year. This is calculated on the basis of a 2 liter per day drinking water intake using the 168 hour data listed in NBS Handbook 69. If several radionuclides are present, the sum of their annual dose equivalent must not exceed 4 mrem/year.
- 1.3 Two types of gamma detectors are currently widely used, namely, the thallium activated sodium iodide crystal, NaI(Tl), and the lithium drifted germanium detector, Ge(Li). The Ge(Li) detector does not detect gamma photons as efficiently as the NaI(Tl) detector, but its photon energy resolution is far better than that of the NaI(Tl) detector. because of its energy resolution advantage and the availability of large active volume Ge(Li) detectors, a Ge(Li) detection system is recommended for measuring gamma emitting radionuclides in drinking water samples.
- 1.4 The method is applicable for analyzing water samples that contain radionuclides emitting gamma photons with energies ranging from about 60 to 2000 keV. The required sensitivity of measurement for the more hazardous gamma emitters is listed in the National Interim Drinking Water Regulations, Section 141.25. for a method to be in compliance, the detection limits for photon emitters must be 1/10 of the applicable limit. The detection limits for cesium-134 and cesium-137, which are 10 and 20 pCi/l respectively, are met by this procedure.

2. Summary of Method

- 2.1 A homogeneous aliquot of drinking water is put into a standard geometry for gamma counting. The counting efficiency for this geometry must have been determined with standard (known) radionuclide activity. Sample aliquots are counted long enough to met the required sensitivity of measurement, specified by the NIPDWR (see Appendix C).

- 2.2 The gamma spectrum is printed out and/or stored in the appropriate computer-compatible device for data processing (calculation of sample radionuclide concentrations).
3. Sample Handling and Preservation - See Section 3, Method 900.0
4. Interferences
 - 4.1 Significant interference occurs when counting a sample with a NaI(Tl) detector and the sample radionuclides emit gamma photons of nearly identical energies. Such interference is greatly reduced by counting the sample with a Ge(Li) detector,
 - 4.2 Sample homogeneity is important to gamma count reproducibility and counting efficiency validity. When sample radionuclides are adsorbed on the walls of the counting container, the sample is no longer homogeneous. This problem can be lessened by adding 15 ml 1N HNO₃ per liter of sample at collection time.
5. Apparatus - See Appendix D for Details and Specifications
 - 5.1 Large volume (> 50 cm³) Ge(Li) detector or 4" x 4" NaI(Tl) detector.
 - 5.2 Gamma-ray spectrometer plus analyzer with at least 2048 channels for Ge(Li) or 512 for NaI(Tl).
 - 5.3 Standard geometry sample counting containers for either detector. (1-pint cylindrical container or 4-liter Marinelli polyethylene beaker.)
 - 5.4 Access to a computer.
6. Reagents
 - 6.1 Radon free distilled or deionized water for standard preparation and sample dilution.
 - 6.2 Nitric acid, 1N: Mix 6.2ml of 16H HNO₃ (conc.) with distilled water and dilute to 100 ml.
7. Calibration
 - 7.1 A Ge(Li) detector-gamma spectrometer can be calibrated for energy resolution as follows:

NBS or NBS-traceable standard solutions are prescribed for this calibration. Adjust the analyzer amplifier "gain" and analog-to-digital converter "zero offset" to locate each photopeak in its appropriate channel. For a Ge(Li) detector system a 0.5 or 1.0 keV per channel calibration is recommended. For a NaI(Tl) detector system a 10 or 20 keV per channel calibration is satisfactory since the energy resolution of this type detector is lower than that of the Ge(Li) detector.

- 7.2 For NaI(Tl), a library of radionuclide gamma energy spectra is prepared with known radionuclide-water sample concentrations at standard sample geometries; for Ge(Li), a single solution containing a mixture of fission products may be used. These standard solutions are available from NBS or the Duality Assurance Division, EMSL-Las Vegas. Counting efficiencies for the various gamma energies (photopeaks) are determined from the activity counts of those known value samples. A counting efficiency vs. gamma energy curve is determined for each container geometry and for each detector that is to be used for sample analysis. Known amounts of various radionuclides that emit gamma photons with energies well spaced and distributed over the normal range of analysis may also be used for this calibration. These are put into each container geometry and gamma counted for a photopeak spectrum accumulation.
- 7.3 The detector efficiency, E, at a given photopeak energy for a given geometry is determined by using a known quantity or concentration (for a volume geometry) of a gamma emitting radionuclide, as follows:

$$E = \frac{C}{A * B}$$

where:

C = net count rate, cpm, (integrated counts in the photopeak above the base line continuum divided by the counting time in minutes),

A = activity of radionuclide added to the given geometry container (dpm),

B = the gamma-ray abundance of the radionuclide being measured (gammas/disintegration).

8. Procedure

- 8.1 Measure an aliquot of the drinking water sample in a standard geometry (one that has been calibrated).

- 8.2 Place the standard geometry container (with the sample aliquot) on a shielded Ge(Li) or NaI(Tl) detector and gamma count for a period of time that will meet the required sensitivity of measurement, specified by the NIPDWR. (The required counting time can be determined by equations given in Appendix C).
- 8.3 Print the gamma spectrum and/or store the spectrum on the appropriate computer-compatible device.
- 8.4 Calculate the radioactivity of the gamma emitters present in the sample.

9. Calculations

These calculations are for determinations using a Ge(Li) detector system. With a NaI(Tl) detector system, similar calculations can be done by a computer using a library of radionuclide spectra and a least-squares (1,2) or matrix analysis program (J).

- 9.1 The isotopes indicated by the gamma spectrum are determined as follows:
 - 9.1.1 Identify all photopeak energies.
 - 9.1.2 Integrate the photopeak regions of the spectrum and subtract the area under the base line continuum to determine the true photopeak area.
 - 9.1.3 Isotopes are identified by their appropriate photopeaks, and ratios to each other when more than one gamma photon is emitted by an isotope in the sample.
- 9.2 Calculate the sample radionuclide concentrations, A, in pCi/l as follows:

$$A = \frac{C}{2.22 * BEV}$$

where:

C = net count rate, cpm, in the peak area above base line continuum,

B = the gamma-ray abundance of the, radionuclide being measured (gammas/dlsintegration),

E = detector efficiency (counts/gamma) for the particular photopeak energy being considered.

V = volume of sample aliquot analyzed (liters).

2.22 = conversion factor from dpm/pCi.

10. Precision and Accuracy

- 10.1 Precision and accuracy of this test procedure will be determined by a separate collaborative study. However, a summary of the EMSL-Las Vegas cross-check and performance sample studies (six and two respectively) for the period of August, 1978, to October, 1979 gives the following information about acceptable performance in the analyses of water samples for gamma emitting radionuclides by gamma spectroscopy. Some laboratories used Ge(Li) detector/gamma
- 10.2 Six gamma emitting radionuclides were used in those studies, namely, chromium-51, cobalt-60, zinc-65, ruthenium-106, cesium-134, and cesium-137. Samples for the August 1978 and October 1978 cross-check samples and the April 1979 performance samples contained cobalt-60 and cesium-134. The February 1979 cross-check samples contained cobalt-60, zinc-65, cesium-134, and cesium-137. The October 1979 cross-check samples contained chromium-51, cobalt-60, cesium-134, cesium-137. The October 1978 performance samples contained cesium-134 and cesium-137.
- 10.3 Cesium-134 in seven studies was analyzed by an average of 46 laboratories for a 90.8 ± 11.6% average acceptable performance. Cesium-137 in five studies was analyzed by an average of 48 laboratories for a 87.7 ± 11.7% average acceptable performance. Since the radionuclide concentrations in the samples for all studies were well below the maximum allowable concentrations for drinking water, this non-destructive gamma-emitting procedure to ascertain whether cesium-134 or cesium-137 is present is recommended as an alternate to Method 901.0.

BIBLIOGRAPHY

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