



Standard Guide for the Determination of Iodine-129 In Uranium Oxide¹

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1. Scope

1.1 This method covers the determination of iodine-129 (^{129}I) in uranium oxide by gamma-ray spectrometry. The method could also be applicable to the determination of ^{129}I in aqueous matrices.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

C1402 Guide for High-Resolution Gamma-Ray Spectrometry of Soil Samples

D1193 Specification for Reagent Water

D3648 Practices for the Measurement of Radioactivity

D3649 Practice for High-Resolution Gamma-Ray Spectrometry of Water

3. Summary of Practice

3.1 An aliquot of uranium oxide is dissolved in dilute nitric acid and the iodine is selectively extracted via liquid-liquid extraction. The iodine is further purified by selective precipitation and counted by gamma-ray spectrometry.

3.2 Gravimetric tracer recoveries using this method are typically between 75 and 90 %.

3.3 The minimum detectable activity (MDA) will vary with chemical yield, sample size, instrument background, counting time and counting efficiency. For a sample size of 100 mg U oxide, using a well shielded detector, a 1000 minute counting time, and 32 % detector efficiency at 30 keV, a MDA of ≤ 0.74 Bq/g (20 pCi/g) oxide was achieved.

¹ This guide is under the jurisdiction of ASTM Committee C26 on the Nuclear Fuel Cycle and is the direct responsibility of subcommittee C26.05 on Methods of Test.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

4. Significance and Use

4.1 The determination of ^{129}I is not typically requested in nuclear fuel specifications however it is commonly requested for disposal of the spent fuel, or for disposal of excess uranium from national weapon complexes. This practice can provide results of sufficient quality for waste disposal repositories.

5. Interferences

5.1 Incomplete removal of uranium and its $^{234}\text{Th}/^{234\text{m}}\text{Pa}$ daughters could lead to elevated Compton background in the low energy region of the gamma-ray spectrum, where the ^{129}I x-rays are counted.

5.2 Because the iodine yield monitor is added after the oxide dissolution, any loss of ^{129}I during the dissolution step will not be monitored and may lead to results that are biased low. To minimize any iodine loss, avoid prolonged heating of the sample and minimize the time the sample is in an acidic state.

6. Instrumentation

6.1 Extended-range or low-energy gamma ray spectrometry system. See **C1402**, **D3648** or **D3649** for a general description of gamma-ray spectrometry systems. The system used to measure the low-energy x-rays from ^{129}I should have a thin window to allow the efficient penetration and measurement of the low-energy x-rays.

7. Terms and Definitions

7.1 *ROI*: Region-of-Interest; the channels, or region, in the spectra in which the counts due to a specific radioisotope appear on a functioning, calibrated gamma-ray spectrometry system.

7.2 *Reagent blank*: reagent water processed the same as the samples; used in the determination of the minimum detectable activity.

8. Apparatus

8.1 *Plastic bottles*, 30 and 60-ml, or separatory funnels

8.2 *Filter paper*—25-mm diameter, 0.45 μm pore size

8.3 Vacuum filter apparatus

8.4 pH paper with unit resolution

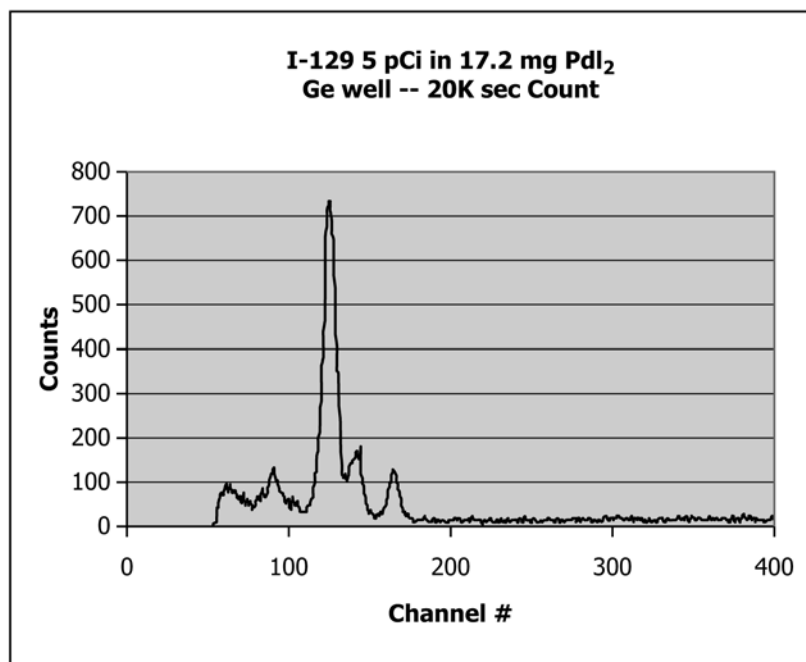


FIG. 1 Low-Energy Photon Spectrum of I-129 on a Ge Well Detector

9. Reagents and Materials

9.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available³.

9.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type I water as defined in Specification D1193.

9.3 *1M Hydroxylamine-hydrochloride*—commercially available solution or dissolve 70 g of the powder in 500 mL of water, dilute to 1 litre final volume.

9.4 Iodide carrier, 20 mg I per millilitre as KI.

9.5 Nitric Acid, concentrated, ~16M

9.6 *0.1M Nitric Acid*—Add ~6 mL of concentrated HNO₃ to 950 mL of water, dilute with water to a final volume of 1 litre.

9.7 *8M Nitric Acid*—Add 500 mL of concentrated HNO₃ to 450 mL of water; dilute with water to a final volume of 1 litre.

9.8 p-xylene.

9.9 *Palladium carrier*—~10 mg/mL, dilute a commercially prepared solution to the correct concentration

9.10 *Sodium bisulfite, 0.1M*—dissolve 10.4 g of powder in 500 mL of water, dilute to a final volume of 1 litre

9.11 *Sodium Carbonate, 2M*—dissolve 212 g of powder in 500 mL of water, dilute to 1 litre final volume.

9.12 *Sodium Hydroxide, 4M*—dilute a commercially prepared solution or dissolve 160 g of pellets in 700 mL of water, dilute to a final volume of 1 litre. This is a very exothermic reaction. The use of an ice bath can mitigate the magnitude of the exothermicity.

9.13 Sodium Hypochlorite.

10. Calibration and Standardization

10.1 The gamma-ray spectrometry system should be calibrated for energy, resolution and efficiency according to the manufacturer instructions. The background counting rate for the instrument should be measured at a frequency determined by the user. See C1402, D3648 or D3649 for additional information. A typical spectrum for ¹²⁹I is shown in Fig. 1.

10.2 Confirm the concentration of the I carrier by adding 1.00 mL of the carrier solution to 15 mL of water. Add 1 mL of the 0.1M NaHSO₃, mix, heat gently and then add 2 mL of the Pd²⁺ carrier. Collect the precipitate (PdI₂) on a tared 25-mm filter paper. Dry and reweigh the filter paper to confirm the expected precipitate weight. Repeat this confirmation several times to increase the precision of the determination.

10.3 Prepare an efficiency curve for the 30 keV x-rays comparing the relative efficiency versus weight of PdI₂ by precipitating equal quantities of ¹²⁹I with various weights of PdI₂. A typical curve for a Ge well detector is shown in Fig. 2; note that this curve shows the net count rate versus weight of PdI₂ rather than calculated efficiency (the ¹²⁹I activity used to prepare this graph was 2.2 Bq (60 pCi)).

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.