



Designation: D 5174 – 97

Standard Test Method for Trace Uranium in Water by Pulsed-Laser Phosphorimetry¹

This standard is issued under the fixed designation D 5174; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of total uranium, by mass, in water in the range of 0.05 ppb or greater. Samples with uranium by mass, levels above the laser phosphorimeter dynamic range may be diluted to bring the concentration to a measurable level.

1.2 This test method was used successfully with reagent water. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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 determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1129 Terminology Relating to Water²

D 1193 Specification for Reagent Water²

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water²

E 131 Terminology Relating to Molecular Spectrometry³

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminologies D 1129 and E 131.

3.1.2 For terms not defined in this test method or in Terminology D 1129 or E 131, reference may be made to other published glossaries.⁴

4. Summary of Test Method

4.1 This test method is based on the utilization of a laser

phosphorimeter to determine total uranium, by mass, in water samples.

4.2 A sample aliquot is pipetted into a pretreated glass vial. Concentrated HNO₃ and hydrogen peroxide are added and the sample heated to near dryness. This step is repeated as necessary and the residue is dissolved in dilute HNO₃. A complexant is added to an aliquot of this sample and analyzed in the phosphorimeter.

4.3 For screening purposes only, an aliquot of the sample may be pipetted directly into the phosphorimeter cell and read. This cannot be done if the sample was preserved with HCl or if the matrix is not known.

5. Significance and Use

5.1 This test method is useful for the analysis of total uranium in water following wet-ashing, as required, due to impurities or suspended materials in the water.

6. Interferences

6.1 *Absorption (Inner Filter Effect)*—This effect is more severe with ultraviolet excitation light (337 nm) than with visible excitation because many prevalent compounds have pi-bonding and absorb strongly in this region. Ferric iron and oxy-anions such as nitrate and organic acids are examples. Visible excitation (425 nm) may be absorbed by yellow solutions, for example, chromate. The consequences of this may be reduced signals and low analysis results.

6.2 *Lumiphors*—Many organic substances, such as humic acids and organic degradation products from incomplete ashing emit luminescence of varying lifetimes after excitation. An advantage of phosphorescence measurement is the ability to determine if interferences are present by observing the lifetime of the decay.

6.3 *Quenching*—Shortened triplet-state lifetime and reduced phosphorescence intensities of the excited uranyl complex result when quenching occurs. Reliable results cannot be obtained when quenching exceeds 80 to 90 %. Reducing agents such as alcohols, halides except fluoride, and metals with electronic energy levels overlapping those of uranyl ion are strong quenching agents. Examples are silver, lead, iron (II), manganese (II), and thallium. Results from single time-gated instruments are particularly sensitive to even mild quenching agents such as aluminum (III), magnesium (II), calcium (II), and strontium (II).

¹ This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

Current edition approved Aug. 10, 1998. Published February 1998. Originally published as D 574 – 91. Last previous edition D 5174 – 91.

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 03.06.

⁴ See *American National Standard Glossary of Terms in Nuclear Science and Technology* (ANSI N1.1), available from American National Standards Institute, 1430 Broadway, New York, NY 10014.