



Designation: D6052 – 97 (Reapproved 2023)

# Standard Test Method for Preparation and Elemental Analysis of Liquid Hazardous Waste by Energy-Dispersive X-Ray Fluorescence<sup>1</sup>

This standard is issued under the fixed designation D6052; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of trace and major element concentrations by energy-dispersive X-ray fluorescence spectrometry (EDXRF) in liquid hazardous waste (LHW).

1.2 This test method has been used successfully on numerous samples of aqueous and organic-based LHW for the determination of the following elements: Ag, As, Ba, Br, Cd, Cl, Cr, Cu, Fe, Hg, I, K, Ni, P, Pb, S, Sb, Se, Sn, Tl, V, and Zn.

1.3 This test method is applicable for other elements (Si-U) not listed in 1.2.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

**C982 Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems (Withdrawn 2008)**<sup>3</sup>

**D1193 Specification for Reagent Water**

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

2.2 *Other ASTM Documents:*

**ASTM Data Series DS 46 X-ray Emission Wavelengths and KeV Tables for Nondiffractive Analysis**<sup>4</sup>

## 3. Summary of Test Method

3.1 A weighed portion of activated alumina and sample are combined in a mixing vessel and shaken until well mixed. The sample mixture is transferred into a disposable sample cup and placed in the spectrometer for analysis.

3.2 The *K* spectral emission lines are used for elements Si-Ba.

3.3 The *L* spectral emission lines are used for elements with atomic numbers greater than Ba.

## 4. Significance and Use

4.1 The elemental analysis of liquid hazardous waste is often important for regulatory and process-specific requirements. This test method provides the user an accurate, rapid method for trace and major element determinations.

## 5. Interferences

5.1 *Spectral Overlaps (Deconvolution):*

5.1.1 Samples containing a mixture of elements often exhibit X-ray emission line overlap. Modern Si (Li) detectors generally provide adequate resolution to minimize the effects of spectral overlap. In cases where emission line overlap exists, techniques of peak fitting exist for extracting corrected analyte emission line intensities. For example, the PbL $\alpha$  “line overlaps with the AsK $\alpha$ .” The PbL $\beta$  line can be used to avoid this overlap and the AsK lines can then be resolved from the PbL $\alpha$  overlap. The actual lines used for any particular element should be such that overlaps are minimized. Follow the EDXRF manufacturer's recommendation concerning spectral deconvolution. Reference should be made to ASTM Data Series DS 46 for detailed information on potential line overlaps.

5.2 *Matrix Interferences (Regression):*

5.2.1 Matrix interference in the measurement of “as received” LHW samples using EDXRF has been the principle limitation in the development and expanding use of this

<sup>4</sup> Available from ASTM Headquarters, Customer Service.

instrumental technique. Using well-understood XRF principles for controlling matrix effects, for example, dilution and matrix modification using lithium borate fusion and addition of heavy absorbers, a matrix can be stabilized. Using calcined alumina and the above principles, matrices are stabilized for quantitative EDXRF analysis.

5.2.2 The response range of this test method should be linear with respect to the elements of interest and their regulatory or process control, or both, action thresholds. Large concentration variations of element or matrix, or both, components in LHW samples can result in nonlinear X-ray intensity response at increasing element concentrations.

## 6. Apparatus

6.1 *Energy-Dispersive X-ray Fluorescence Spectrometer*, capable of measuring the wavelengths of the elements listed in 1.2. Refer to Guide C982 for system specifications.

6.2 *Analytical Balance*, capable of weighing to 0.001 g.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>5</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean meeting the numerical requirements of Type II water as defined by Specification D1193.

7.3 *Aluminum Oxide, Al<sub>2</sub>O<sub>3</sub>*, pre-calcined at 1500 °C, approximately 100 to 125 mesh.

7.4 *Aqueous or Organic-Based Atomic Absorption Standards (AAS)*, 1000 mg/L for the elements Ag, As, Ba, Cd, Cr, Cu, Fe, Hg, K, Ni, Pb, Sb, Se, Sn, Tl, V, and Zn. Standard solutions for elements not listed are also available.

NOTE 1—AAS standards are typically presented in mass/vol units. The density of these solutions can be considered as unity (that is, 1) thus they can be considered as % mass/mass (m/m).

7.5 *1-bromonaphthalene, trichlorobenzene, iodobenzoic acid, triethyl phosphate, and dithiodiglycol* are the recommended standards for the elements Br, Cl, I, P and S, respectively.

7.6 *Low Molecular Weight Polyethylene Glycol (PEG 400, or equivalent) or Water* is used for producing method blank.

7.7 *High-Density Polyethylene (HDPE) Wide Mouth, Round, Screw-Cap Bottles*, 50 to 60 mL capacity.

<sup>5</sup> 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.

TABLE 1 Recommended Standards Ranges

| Analyte | Low Concentration Range, mg/kg | High Concentration Range, mg/kg | Analyte | Low Concentration Range, mg/kg | High Concentration Range, mg/kg |
|---------|--------------------------------|---------------------------------|---------|--------------------------------|---------------------------------|
| Ag      | 5                              | 600                             | Zn      | 5                              | 600                             |
| Ba      | 5                              | 600                             | As      | 5                              | 600                             |
| P       | 0.1 %                          | 5 %                             | Se      | 5                              | 600                             |
| S       | 0.05 %                         | 5 %                             | Br      | 10                             | 5000                            |
| Cl      | 0.05 %                         | 5 %                             | Cd      | 5                              | 600                             |
| K       | 0.1 %                          | 5 %                             | Sb      | 5                              | 600                             |
| V       | 5                              | 600                             | Sn      | 5                              | 600                             |
| Cr      | 5                              | 600                             | I       | 5                              | 600                             |
| Fe      | 5                              | 600                             | Hg      | 5                              | 600                             |
| Ni      | 5                              | 600                             | Tl      | 5                              | 600                             |
| Cu      | 5                              | 600                             | Pb      | 5                              | 600                             |

7.8 *Mixing Balls*, approximately 1 cm diameter, stainless steel or equivalent.

NOTE 2—Potential low level Cr, Fe or Ni (<20 mg/kg<sup>1</sup>) contamination due to the use of stainless steel may exist. Other suitable materials would be tungsten carbide, Zr or Ta.

7.9 *Thin-Film Support*.

NOTE 3—The user should select a thin-film support that provides for maximum transmittance and is resistant to typical components in LHW. The thin-film supports used in the development of this test method were a polypropylene base and a high-purity 4 µm polyester film.

7.10 *Sample Cups*, vented.

7.11 *Helium, He*—Minimum 99.99 purity for use as a chamber purge gas for the analysis of Cl, P, and S. This numerical purity is intended to specify a general grade of helium. Ultra-high purity helium is not required for this test method.

## 8. Sample

8.1 Because of the potential heterogeneous nature of LHW, all possible efforts should be made to ensure that representative samples are taken.

## 9. Preparation of Apparatus

9.1 Follow the manufacturer's instructions for set-up, conditioning, preparation, and maintenance of the XRF spectrometer.

9.2 When required, reference spectra should be obtained from pure element standards for all deconvoluted elements.

9.3 Spectral and matrix interferences as listed in the Interferences section must be addressed per the manufacturer's recommendations.

## 10. Calibration and Standardization

10.1 The spectrometer must be calibrated using an appropriate reference element(s) at a minimum frequency as recommended by the manufacturer.

10.2 Analytical standards should be prepared gravimetrically by blending the solution or pure element standards with Al<sub>2</sub>O<sub>3</sub> to suitable standard concentrations as determined by the user's analytical requirements. Table 1 gives recommended concentration ranges for regression. Standards can be single or