



Standard Test Method for Preparation and Elemental Analysis of Liquid Hazardous Waste by Energy-Dispersive X-Ray Fluorescence¹

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

2. Referenced Documents

2.1 ASTM Standards:²

C982 Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems (Withdrawn 2008)³

D1193 Specification for Reagent Water

2.2 Other ASTM Documents:

ASTM Data Series DS 46 X-ray Emission Wavelengths and KeV Tables for Nondiffractive Analysis⁴

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 α “line overlaps with the AsK α .” The PbL β line can be used to avoid this overlap and the AsK lines can then be resolved from the PbL α 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 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

¹ 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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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.

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from ASTM Headquarters, Customer Service.