



Designation: D8064 – 16

Standard Test Method for Elemental Analysis of Soil and Solid Waste by Monochromatic Energy Dispersive X-ray Fluorescence Spectrometry Using Multiple Monochromatic Excitation Beams¹

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1. Scope

1.1 This test method is based upon energy-dispersive X-ray Fluorescence (EDXRF) spectrometry using multiple monochromatic excitation beams for detection and quantification of selected heavy metal elements in soil and related solid waste.

1.2 This test method is also known as High Definition X-ray Fluorescence (HDXRF) or Multiple Monochromatic Beam EDXRF (MMB-EDXRF).

1.3 This test method is applicable to various soil matrices for the determination of Cr, Ni, As, Cd, Hg, and Pb in the range of 1 to 5000 mg/kg, as specified in [Table 1](#) and determined by a ruggedness study using representative samples. The limit of detection (LOD) for each element is listed in [Table 1](#). The LOD is estimated by measuring a SiO₂ blank sample (see [Table X1.1](#) in [Appendix X1](#)).

1.4 This test method is applicable to other elements: Sb, Cu, Se, Ag, Tl, Zn, Ba, Au, Co, V, Fe, Mn, Mo, K, Rb, Sn, Sr, and Ti.

1.5 *X-ray Nomenclature*—This standard names X-ray lines using the Siegbahn convention.²

1.6 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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.*

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 Jenkins, R., Manne, R., Robin, R., and Senemaud, C., “Nomenclature System for X-ray Spectroscopy,” *Pure & Appl Chem.*, Vol 63, No. 5, pp. 735–746, 1991.

1.8 *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:³

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D4944 Test Method for Field Determination of Water (Moisture) Content of Soil by the Calcium Carbide Gas Pressure Tester

D5283 Practice for Generation of Environmental Data Related to Waste Management Activities: Quality Assurance and Quality Control Planning and Implementation

D5681 Terminology for Waste and Waste Management

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

E1169 Practice for Conducting Ruggedness Tests

E1727 Practice for Field Collection of Soil Samples for Subsequent Lead Determination

E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques

2.2 Other Documents:

ASTM DS46 X-Ray Emission Wavelengths and Kev Tables for Nondiffractive Analysis⁴

ASTM MNL 7 Manual on Presentation of Data and Control Chart Analysis, 8thed.⁴

³ 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 Available from ASTM International, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959, www.astm.org.

TABLE 1 Limit of Detection (LOD) and Method Range

Element	LOD (mg/kg)	Method Range (mg/kg)
Cr	2.3	11 to 500
Ni	1.1	5 to 500
As	0.2	1 to 2000
Cd	0.4	2 to 100
Hg	0.4	2 to 100
Pb	0.6	3 to 5000

US EPA Method, Method 6200 Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment⁵

3. Terminology

3.1 *Definitions*—Definitions of terms applying to XRF, soil, and waste management appear in Terminologies **D653** and **D5681**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *fundamental parameters (FP) model, n*—a model for calibration of X-ray fluorescence response, including the correction of matrix effects, based on the theory describing the physical processes of the interactions of X rays with matter.

3.2.2 *high energy monochromatic beam, n*—a focused monochromatic beam having its selected photon energy between 32 and 40 keV.

3.2.3 *medium energy monochromatic beam, n*—a focused monochromatic beam having its selected photon energy between 15 and 23 keV.

3.2.4 *monochromatic beam, n*—an incident monochromatic beam on a sample having a selected photon energy with a narrow energy bandwidth relative to the selected energy; method precision is achieved with a monochromatic beam having an energy bandwidth (Full Width Half Maximum) less than 15 % relative to the selected energy and containing more than 95 % flux of the spectrum of the excitation beam which is incident on the sample.

3.2.5 *multiple monochromatic excitation beams, n*—two or more monochromatic beams.

3.2.6 *Rayleigh scattering, n*—the elastic scattering of an X-ray photon through its interaction with the bound electrons of an atom; this process is also referred to as coherent scattering.

3.3 *Acronyms:*

3.3.1 *ARV*—accepted reference values

3.3.2 *EDXRF*—energy dispersive X-ray fluorescence

3.3.3 *FP*—fundamental parameters

3.3.4 *HDXRF*—high definition X-ray fluorescence

3.3.5 *LOD*—limits of detection

3.3.6 *MMB*—multiple monochromatic beams

3.3.7 *ND*—non-detected

3.3.8 *RSD*—relative standard deviation

4. Summary of Test Method

4.1 The operating conditions presented in this test method have been successfully used in the determination of Cd, As, Cr, Pb, Hg, Sb, Cu, Ni, Se, Ag, Tl, Zn, Ba, Sn, and Au in soil and related solid waste.

4.2 This technique uses one or more monochromatic excitation beams to quantify elemental concentrations in soil and solid waste samples. The sample is homogenized to a reasonable degree and positioned in front of an aperture where it is exposed to one or more monochromatic X-ray beams that are focused by X-ray optics from an X-ray source. A nearby detector is positioned to collect fluorescent and backscattered X rays. The X rays collected by the detector are converted to electric pulses by a digital pulse processor. A multi-channel analyzer separates the pulses by X-ray energy, forming the measurement spectrum. The spectrum is processed by an FP method to obtain the analysis result.

4.3 The apparatus is calibrated for each monochromatic beam and the detector. The calibration may be performed by the manufacturer or by the user.

5. Significance and Use

5.1 Elemental species such as Cr, Ni, As, Cd, Hg, and Pb are widely used in many industrial processes. These elements have been identified in many former industrial sites driving the need for a quick, easy method for testing on-site at trace levels in soil and solid waste matrices.

5.2 This method may be used for quantitative determinations of Cr, Ni, As, Cd, Hg, and Pb in soil matrices and solid waste. Typical test time is 90 seconds to 15 minutes.

6. Interferences

6.1 *Spectral Interference*—Spectral interferences result from spectral overlaps among the X-ray lines that remain unresolved due to limited energy resolution of the detector. For instance, the arsenic (As) $K\alpha$ peak directly overlaps the lead (Pb) $L\alpha$ peak. The Pb $L\beta$ line can be used to account for this overlap and the As K lines can then be resolved from the Pb $L\alpha$ overlap. The actual lines used for any particular element should be such that overlaps are minimized. Reference ASTM Data Series DS46 for detailed information on potential line overlaps. Interactions of photons and electrons inside the detector result in additional peaks in the spectrum known as escape peaks and sum peaks. These peaks can overlap with X-ray lines of interest, for example, the sum peak of iron (Fe) $K\alpha$ can overlap with the Pb $L\beta$ peak.

6.2 *Matrix Effects*—Matrix effects, also called interelement effects, exist among all elements as the result of absorption of fluorescent X rays (secondary X rays) by atoms in the specimen. Absorption reduces the apparent sensitivity for the element. In contrast, the atom that absorbs the X rays may in turn emit a fluorescent X ray, increasing apparent sensitivity for the second element. Mathematical methods may be used to compensate for matrix effects. A number of mathematical

⁵ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <https://www.epa.gov/hw-sw846/sw-846-test-method-6200-field-portable-x-ray-fluorescence-spectrometry-determination>.

correction procedures are commonly utilized including full FP treatments and mathematical models based on influence coefficient algorithms.

6.3 *Physical Matrix Effects*—Physical characteristics of the sample such as particle size and homogeneity. Effects can be minimized by mixing samples, grinding and sieving them to a uniform particle size prior to analysis, or by increasing the area exposed to the X-ray beam path by rotating the sample during analysis.

6.4 *Moisture Effects*—Soil samples that are in excess of 15 % moisture content may introduce error to the analysis. Samples with high moisture content can be dried in an oven prior to analysis at less than 150°C. If mercury is a target analyte, a separate portion of the sample should be dried without heating and analyzed on its own. Another option is to use the manufacturer's auto-moisture correction for moisture content using FP modeling.

7. Apparatus

7.1 *EDXRF Spectrometer*,⁶ designed for X-ray fluorescence analysis using multiple monochromatic excitation beams with an energy dispersive detector and with a design that incorporates at a minimum the following features (unless otherwise specified):

7.1.1 *Source of X-ray Excitation*—An X-ray tube with a zirconium, molybdenum, rhodium, palladium, silver target or other suitable target can be used.

7.1.2 *X-ray Optics*—X-ray optical elements capable of accepting X rays from a tube and directing monochromatic beams on the sample. Two or more X-ray optical elements are necessary to provide multiple monochromatic beams. At least one optical element provides a medium energy monochromatic beam, and at least one optical element provides a high energy monochromatic beam.

7.1.3 *Beam Shutter*, used to select a monochromatic beam or select a combination of monochromatic beams.

7.1.4 *X-ray Detector*, with energy resolution ≤ 140 eV full width at half maximum of the manganese (Mn) K α line.

7.1.5 *Digital Pulse Processor and Multi-channel Analyzer*—A digital pulse processor for pulse shaping and conditioning, and a multi-channel analyzer for binning the pulses according to X-ray energy.

7.1.6 *Detector Aperture*—An aperture in the beam path between the sample and the detector to limit the field of view of the detector.

7.2 *Analyzer Test Stand*, may have the following accessories:

7.2.1 *Sample Cell Rotator (optional)*, designed to hold a removable sample cell.

7.2.2 *Removable Sample Cell*—An open ended specimen holder compatible with the geometry of the MMB-EDXRF

spectrometer and is designed to use replaceable X-ray transparent film to hold a soil specimen with a minimum depth of 1 cm.

8. Reagents and Materials

8.1 *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.⁷ 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.

8.2 *Calibration Standard(s)*—At least three homogenous reference materials are required for calibration (see **Note 1**). The calibration standards should provide (a) concentrations of Cr, Ni, As, Cd, Hg, and Pb at or near background soil level, (b) some or all of concentrations of Cr, Ni, As, Cd, Hg, and Pb are at low range levels, and (c) some or all of concentrations of Cr, Ni, As, Cd, Hg, and Pb are at high range levels. It is recommended to use calibration standards that are traceable to standard reference materials when such materials are available.

NOTE 1—Additional calibration standards may be used for improved accuracy.

8.3 *Calibration Verification Sample(s)*—At least one homogenous reference material containing Cr, Ni, As, Cd, Hg, and Pb is required for calibration.

8.4 *Drift Correction Monitors (optional)*—Due to instability of the measurement system, the sensitivity and background of the spectrometer may drift with time. Drift correction monitors may be used to compensate for this drift. The optimum drift correction monitor samples are permanent materials that are stable with repeated exposure to X rays.

8.5 *Gloves*—Disposable gloves are recommended for handling reference materials and other samples.

8.6 *Quality Control Sample(s)*—To ensure the quality of the results, a quality control (QC) sample is used for establishing and monitoring the stability and precision of an analytical measurement system (see 17.4). If possible, the QC sample shall be reference material representative of samples typically analyzed. The materials shall be stable under the anticipated storage conditions. The QC sample can be a calibration validation sample.

8.7 *Reference Materials*—Homogenous material with a known elemental composition. Reference materials are available from commercial sources or may be prepared gravimetrically. For purposes of this method, homogenous reference materials in this test method are soils or sludge, unless otherwise specified.

⁶ The sole source of supply of the apparatus known to the committee at this time is XOS, Inc., 15 Tech Valley Drive, East Greenbush, NY 12061. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, D.C. 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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

8.8 *Independent Reference Material (IRM)*—A material of known purity and concentration obtained either from the National Institute of Standards and Technology (NIST) or other reputable supplier.

8.9 *Silicon Dioxide Powder (SiO₂)*—For preparing gravimetric empirical calibration standards (if using calibration method B, see 12.5) and for use as a blank sample to monitor instrument contamination and sample preparation contamination (see 17.3).

8.10 *Single/Multi-Element Aqueous Standards*—Aqueous reference material with known elemental composition for preparing gravimetric empirical calibration standards (if using calibration method B or matrix spiked).

8.11 *X-ray Transparent Thin-Film*—Used as a protective barrier between the sample and the analyzer. See Note 2.

NOTE 2—The user should select a thin film that provides for maximum transmittance. The thin-film used in the development of this test method was high-purity 12 µm polypropylene film.

9. Hazards

9.1 Occupational Health and Safety standards for X rays and ionizing radiation shall be observed. Guidelines for safe operating procedures are also given in current handbooks and publications from original equipment manufacturers. For more information see similar handbooks on radiation safety.

9.2 Use proper personal protective equipment (PPE) when handling contaminated soil and solid waste. Consult chemical safety data sheets for recommended PPE.

10. Sampling and Test Specimen Preparation

10.1 *Sampling*—Collected samples in accordance with Practice E1727 or similar procedure.

10.2 *Preparation for Measurement:*

10.2.1 To ensure a representative sample is used, thoroughly mix the sample in the collection container by stirring, mixing, or kneading. Large agglomerations of soil or solid waste should be broken up. If mixing is not possible, take at least five portions of the sample when transferring.

10.2.2 If sample exceeds 15 % moisture content by wet weight it should be dried. Samples that require drying will appear muddy or have visible excess water. If unable to determine visibly, use Test Method D4944 or other method to determine moisture content. If drying is needed, place sample in drying container. Place drying container in oven until mass is constant. Sample can also be air dried or sun dried at the site alternatively.

10.2.3 Exclude non-representative material such as twigs, leaves, roots, insects, asphalt, and rocks. Completely grind the sample until it will pass through a number 50 sieve or higher (<275 µm particle size). Transfer enough material to fill the sample cell and cover with an X-ray film.

11. Preparation of Apparatus

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

11.2 Allow the apparatus to stabilize for operation according to the manufacturer's guidelines.

11.3 Ensure that the spectral processing operates correctly according to the manufacturer's guidelines addressing spectral and matrix interferences as listed in the interferences section.

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

12. Calibration

12.1 Calibration should, at a minimum, be conducted when a QC sample is out of range or as recommended by the manufacturer. For some applications including regulatory compliance, more frequent calibration may be required.

12.2 *Calibration of the X-ray Detector and Digital Pulse Processor*—Using calibration standards, identify one or more elemental emission line peaks from the spectrum of each monochromatic excitation beam to calibrate the energy to channel relationship of the X-ray Detector and Digital Pulse Processor (see 7.1.4 and 7.1.5). Chosen emission lines should be free from major interferences and should have a statistically significant peak counting area.

12.3 Two methods of calibrations are available:

12.4 *Calibration Method A – Fundamental Parameters Method:*

12.4.1 Fundamental Parameters calibration should be performed using multiple soil calibration standards, consisting of background soil level, low contamination levels and high contamination levels for selected elements to determine the initial sensitivity factors from each monochromatic beam. If appropriate reference soil standards are difficult to be obtained for some elements of interest, a reference standards with adjacent or nearby elements in the periodic table can be used in FP calibration. Soil reference materials may be NIST Soil Standard Reference Materials or similar. Soil reference materials may also be a soil surrogate like SiO₂ powder spiked with known metal contents; see 12.5.2 for spike process. By measuring the X-ray net intensity (cps) for each element and using the determined sensitivity factor for each beam plus a set of equations to account for the X-ray production, the X-ray absorption and enhancement effect, the concentration of all elements present can be determined. Due to the use of monochromatic beams for excitation, the FP calculation is much more accurate compared to the polychromatic excitation.

12.4.2 Solid waste samples may have a matrix quite different from a soil matrix. At least two reference materials with lighter matrix and heavier matrix shall be used to refine the FP model to account for significant matrix variation other than soil matrix. A solid waste sample that has organic type of matrix is considered to be a light matrix. A solid waste sample that contains more than 10 % of metals, like Fe, is considered as a heavy matrix. This step is not needed if only soil samples are tested in an application.

12.4.3 Most manufacturers provide FP calibration software to perform FP calibration and FP refinement. Follow the manufacturer's FP set-up recommendations. The manufacturer may have an optional setup to use a standard that has similar

matrix to the testing sample that can be used to refine FP parameters for the best accuracy.

12.5 Method B – Empirical Calibration Method:

12.5.1 Empirical calibration for soil matrix is performed using the following calibration standards: a blank and at least four or more concentration levels that bracket between the low range and the high range defined in **Table 2**. The calibration standards should provide a linear response of element intensity to concentration. If extended range is needed, then an additional calibration shall be performed to cover between the high range and extended range. For the additional calibration, use four or more concentration levels that bracket between the high concentration range and the extended range.

12.5.2 Calibration standards may be prepared by gravimetrically mixing SiO₂ powder with single/multi-element in aqueous standard. The mixing weight ratio for aqueous standard and SiO₂ is about 1:1. For example, prepare a 200 mg/kg Pb in SiO₂ standard by mixing 5 g of 100 mg/kg Pb aqueous standard solution, and 5 g of SiO₂. Dry the standard in a 60°C to 100°C oven or allow to air dry.

12.5.3 The matrix effect between the SiO₂ calibrants and soil samples should be corrected for by using a correction factor that is determined by measuring a spiked uncontaminated soil sample. An uncontaminated soil sample should be prepared and spiked (see **12.5.2** and use the uncontaminated soil in the place of the SiO₂ powder) to a known high range concentration to obtain a matrix correction factor between the calibrant and the real soil sample. The correction factor is typically recorded in a software tool provided by the manufacturer.

12.5.4 Empirical calibration for a specific solid waste matrix is performed using the following calibration standards: a blank and at least four or more concentration levels that bracket between the low range and the high range defined in **Table 2**. The calibration standards should provide a linear response of element intensity to concentration. If extended range is needed, then an additional calibration shall be performed. For the additional calibration, use four or more concentration levels to cover between the high range and extended range. The calibration standards shall have similar matrix to the solid waste matrix. If a matrix match between the calibrants and solid waste batch samples cannot be found, FP calibration method shall be used. This step is not needed if only soil samples are tested in an application.

12.5.5 For elements such as Hg, empirical calibration may produce measurement results which are biased due to its high volatility. Suitability of the empirical calibration for a volatile element can be verified using a commercially available refer-

ence material (see **12.4**) that has been stored as recommended by the manufacturer and is within expiration date. In this case, a FP calibration method may be preferred over empirical calibration.

12.6 After calibrating, verify analyzer calibration by using one or more calibration validation samples (see **8.4**). Results shall have a percent deviation less than 20 % from the known concentration for the target elements. If results for elements of interest are not within acceptable limits, the analyzer must be recalibrated.

12.7 Calibration should be verified periodically per **12.6**. The initial validation frequency shall be weekly. If there is sufficient data to demonstrate that the calibration is stable, the calibration validation frequency can be reduced to monthly or longer period.

13. Procedure

13.1 Measurement of Unknown Sample:

13.1.1 Prepare the sample according to Section **10**, and prepare the instrument according to Section **11**. Place the sample in the X-ray beam path and perform the measurement as directed by the manufacturer.

13.1.2 Process the spectrum using the same procedure as outlined in Sections **11** and **12**.

13.2 Measurement of Reference Materials:

13.2.1 When using reference materials, measure them in the same manner as an unknown sample (**13.1**) and before measuring any unknowns.

13.2.2 Analysis of result(s) from these samples must be carried out following Section **17**. When the sample results exceed the laboratory's control limits, drift correction or instrument calibration may be required.

13.3 Drift Correction:

13.3.1 Drift is corrected by normalizing or standardizing the new measured count rates to make them comparable to count rates obtained at the time of calibration. Use the manufacturer's tools, if provided, or another suitable procedure.

14. Interpretation of Results

14.1 Using the net count rates for a sample and the calibration from Section **12**, the result is calculated by the instrument software in units of mg/kg for each element that is reported.

14.2 For samples measured on a SiO₂ empirical calibration curve, apply the matrix correction by multiplying the measurement results by the matrix correction factor. Typically a software tool provided by the manufacturer can do the matrix correction automatically.

15. Report

15.1 Report the mass content of each element of the test sample calculated from Section **14** using units of mg/kg, rounded to the nearest 0.01 mg/kg for mass fractions ≤100 mg/kg, and rounded to the nearest 0.1 mg/kg for mass fractions >100 mg/kg. Include a description of the sample type, and indicate that the test results were obtained using Test Method D8064.

TABLE 2 Empirical Calibration Standard Ranges

Element	Low Concentration Range (ppm)	High Concentration Range (ppm)	Extended Range (ppm)
Cr	5	500	5000
Ni	5	500	5000
As	1	1000	5000
Cd	1	500	5000
Hg	1	500	5000
Pb	1	1000	5000

16. Precision and Bias

16.1 *Precision*—The precision of this test method was determined from data from a single site study in accordance with Practice E1169. Five NIST SRM soil samples and two NIST SRM sludge samples (see Table X1.2 in Appendix X1) were measured ten times each to determine the relative standard deviation (RSD) and bias. The results obtained for the study are summarized in Table 3. Appendix X1 provides the detailed results for single site blank, and precision and bias study from each NIST SRM.

16.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the repeatability values only in one case in twenty. The final repeatability of this test method will be available following the completion of the interlaboratory or intersite study. A preliminary repeatability is indicated by the single site RSD given in Table 3 and Appendix X1.

16.1.2 *Reproducibility*—The reproducibility of this test method is not currently available. The reproducibility of this test method will be available following the completion of the interlaboratory or intersite study.

16.2 *Bias*—Between-lab bias will not be available until the interlaboratory or intersite study is complete. Single laboratory bias has been determined as shown in Table 3. No significant bias was exhibited for all seven NIST samples in the reporting ranges.

17. Quality Control

17.1 The following quality control requirements shall be performed to ensure the analyzer is within control:

17.1.1 Verify analyzer calibration by using one or more reference materials (see 8.4). Calibration verification shall be performed after calibrating the analyzer and at least once weekly initially. If the calibration is stable for a longer period of time, the calibration validation frequency can be reduced to monthly or a longer period. See 12.4 for calibration verification acceptance criteria.

17.1.2 An initial demonstration of site capability is necessary to prevent errors as a result of unfamiliarity with the test method. See 17.2.

17.1.3 Analysis of a blank is performed to ensure measurement results are not due to contamination. Analysis should be performed at least once daily or for each batch analysis. See 17.3.

17.1.4 Analysis of a Quality Control (QC) sample is performed to ensure that the measurement system is in control at the time samples are being analyzed. Analysis should be performed at least once weekly or for each batch analysis. See 17.4.

17.1.5 A duplicate analysis of a sample or a control sample is performed periodically to ensure precision of the sample analysis within the repeatability of the method. See 17.5.

17.1.6 Analysis of a Matrix Spiked (MS) sample is performed to ensure no significant matrix interference for each batch analysis. Alternatively, an IRM sample can be measured. See 17.6.

17.2 *Initial Demonstration of Site Capability*—If a site has not performed the test method before or if there is a change in the measurement system, for example new analyst, new instrument, etc., a demonstration of laboratory capability shall be performed. Analyze an IRM sample or reference material sample seven times. Calculate the mean, standard deviation,

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<https://standards.iteh.ai/catalog/standards/sist/514028a1-72ce-4204-92d5-6d460e56589a/astm-d8064-16>
TABLE 3 Repeatability and Bias Results for Single Site Study (mg/kg)

NIST SRM		Cr	Ni	As	Cd	Hg	Pb
1646a	Mean	41.66	22.07	6.66	11.83
	ARV	40.9	23	6.23	0.148	0.04	11.7
	RSD	4.39 %	8.33 %	2.05 %	1.58 %
	Bias	1.86 %	-4.04 %	6.90 %	1.11 %
2586	Mean	313.2	72.91	8.16	2.89	0.63	422.80
	ARV	301	75	8.7	2.71	0.367	432
	RSD	2.56 %	0.78 %	9.49 %	12.31 %	37.7 %	0.65 %
	Bias	4.06 %	-2.79 %	-6.21 %	6.64 %	71.66 %	-2.13 %
2709a	Mean	133.70	87.01	11.63	...	0.96	18.07
	ARV	130	85	10.5	0.37	0.9	17.3
	RSD	2.18 %	0.78 %	5.10 %	...	27.47 %	1.02 %
	Bias	2.82 %	2.36 %	10.76 %	...	6.67 %	4.45 %
2710a	Mean	23.25	8.85	1506.5	11.68	9.44	5422.5
	ARV	23	8	1540	12.3	9.88	5520
	RSD	11.44 %	6.12 %	0.79 %	3.27 %	11.49 %	0.15 %
	Bias	1.09 %	10.63 %	-2.17 %	-5.04 %	-4.45 %	-1.77 %
2711a	Mean	54.30	18.76	104.54	55.04	7.49	1404.6
	ARV	52.3	21.7	107	54.1	7.42	1400
	RSD	5.06 %	2.32 %	2.27 %	1.50 %	4.37 %	0.11 %
	Bias	3.82 %	13.55 %	-2.30 %	1.74 %	0.94 %	0.33 %
2782	Mean	109.0	153.4	159.8	4.31	...	594.7
	ARV	109	154.1	166	4.17	1.1	574
	RSD	10.58 %	2.43 %	1.23 %	14.58 %	...	0.59 %
	Bias	0.00 %	-0.47 %	-3.76 %	3.43 %	...	3.61 %
2781	Mean	202.3	80.29	8.14	12.69	3.78	201.6
	ARV	202	80.2	7.82	12.78	3.64	202.1
	RSD	1.95 %	1.27 %	3.78 %	14.36 %	13.78 %	0.36 %
	Bias	0.15 %	0.11 %	4.10 %	-0.72 %	3.83 %	-0.25 %