

Designation: E3203 - 21

Standard Test Method for Determination of Lead in Dried Paint, Soil, and Wipe Samples by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES)¹

This standard is issued under the fixed designation E3203; 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 specifies a procedure for analysis of dried paint, soil, and dust wipe samples collected in and around buildings and related structures for lead content using inductively coupled plasma-optical emission spectroscopy (ICP-OES).

1.2 This test method should be used by analysts experienced in the use of ICP-OES, the interpretation of spectral and matrix interferences, and procedures for their correction. For determination of lead (Pb) and other metals in air by ICP-OES, see Test Method D7035.

1.3 This test method cites specific methods for preparing test solutions of dried paint, soil, and wipe samples for analysis.

1.4 It is the user's responsibility to ensure the validity of this test method for sampling materials of untested matrices.

1.5 No detailed operating instructions are provided because of differences among various makes and models of suitable ICP-OES instruments. Instead, the analyst shall follow the instructions provided by the manufacturer of the particular instrument. This test method does not address comparative accuracy of different devices or the precision between instruments of the same make and model.

1.6 This test method contains notes that are explanatory and are not part of the mandatory requirements of this test method.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7.1 *Exception*—The inch-pound and SI units shown for wipe sampling data are to be individually regarded as standard for wipe sampling data.

¹ This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.12 on Sampling and Analysis of Lead for Exposure and Risk Assessment.

1.8 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.

1.9 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:²
- D1193 Specification for Reagent Water
- D1356 Terminology Relating to Sampling and Analysis of Atmospheres
- D6785 Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry
- D6966 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals
- D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D7440 Practice for Characterizing Uncertainty in Air Quality Measurements
- E631 Terminology of Building Constructions
- E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
- E1583 Practice for Evaluating Laboratories Engaged in Determination of Lead in Paint, Dust, Airborne Particulates, and Soil Taken From and Around Buildings and Related Structures

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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.

E1605 Terminology Relating to Lead in Buildings

- E1613 Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques (Withdrawn 2021)³
- E1644 Practice for Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead
- E1645 Practice for Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis
- E1726 Practice for Preparation of Soil Samples by Hotplate Digestion for Subsequent Lead Analysis
- E1727 Practice for Field Collection of Soil Samples for Subsequent Lead Determination
- E1728 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination
- E1729 Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination
- E1792 Specification for Wipe Sampling Materials for Lead in Surface Dust
- E1908 Practice for Sample Selection of Debris Waste from a Building Renovation or Lead Abatement Project for Toxicity Characteristic Leaching Procedure (TCLP) Testing for Leachable Lead (Pb)
- E1979 Practice for Ultrasonic Extraction of Paint, Dust, Soil, and Air Samples for Subsequent Determination of Lead
- E2115 Guide for Conducting Lead Hazard Assessments of Dwellings and of Other Child-Occupied Facilities
- E2271/E2271M Practice for Clearance Examinations Following Lead Hazard Reduction Activities in Multifamily Dwellings
- E2913/E2913M Practice for Hotplate Digestion of Lead from Composited Wipe Samples
- E2914/E2914M Practice for Ultrasonic Extraction of Lead from Composited Wipe Samples
- E3074/E3074M Practice for Clearance Examinations Following Lead Hazard Reduction Activities in Single Family Dwellings, in Individual Units of Multifamily Dwellings, and in Other Child-Occupied Facilities
- 2.2 ISO and European Standards:⁴
- ISO 1042 Laboratory glassware One-mark volumetric flasks
- ISO 3585 Borosilicate glass 3.3 Properties
- **ISO 8655** Piston-operated volumetric apparatus (6 Parts)
- ISO 15202 Workplace air Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry (3 Parts)
- **ISO/IEC** 17025 General requirements for the competence of testing and calibration laboratories

2.3 Other Standards:

EPA SW-846 Test Method 311 Toxicity Characteristic Leaching Procedure⁵

JCGM 100 Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement⁶

3. Terminology

3.1 For definitions of pertinent terms not listed here, see Terminologies D1356, E631, and E1605.

3.2 Definitions:

3.2.1 *atomic emission*, *n*—characteristic radiation emitted by an electronically excited atomic species.

3.2.1.1 *Discussion*—In atomic (or optical) emission spectrometry, a very high-temperature environment, such as a plasma, is used to create excited state atoms. For analytical purposes, characteristic emission signals from elements in their excited states are then measured at specific wavelengths.

3.2.2 *axial plasma*, *n*—a horizontal inductively coupled plasma that is viewed end-on (versus radially; see 3.2.27).

3.2.3 *background correction*, *n*—the process of correcting the intensity at an analytical wavelength for the intensity due to the underlying spectral background of a blank. **ISO 15202**

3.2.4 *background equivalent concentration, n*—the concentration of a solution that results in an emission signal of equivalent intensity to the background emission signal at the analytical wavelength. ISO 15202

3.2.5 *batch*, n—a group of field or quality control (QC) samples that are collected or processed together at the same time using the same reagents and equipment. **E1613**

3.2.6 *blank solution, n*—solution prepared by taking a reagent blank or field blank through the same procedure used for sample dissolution.

3.2.7 *calibration blank solution, n*—calibration solution prepared without the addition of any stock standard solution or working standard solution. **ISO 15202**

3.2.7.1 *Discussion*—The concentration of the analyte(s) of interest in the calibration blank solution is taken to be zero.

3.2.8 *calibration solution, n*—solution prepared by dilution of the stock standard solution(s) or working standard solution(s), containing the analyte(s) of interest at the concentration(s) suitable for use in calibration of the analytical instrument. ISO 15202

3.2.8.1 *Discussion*—The technique of matrix matching is normally used when preparing calibration solutions.

3.2.9 *continuing calibration blank (CCB), n*—a solution containing no analyte added, that is used to verify blank response and freedom from carryover. **E1613**

3.2.9.1 *Discussion*—The absolute value of the measured concentration of the CCB is to be not more than 50 % of the lowest regulatory limit for the sample matrix analyzed or minimum level of concern.

 $^{^{3}\,\}text{The}$ last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

⁵ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

⁶ Available from Bureau International des Poids et Mesures (BIPM), Pavillon de Breteuil F-92312, Sèvres Cedex, France, http://www.bipm.org.

3.2.10 *excitation interferences*, *n*—non-spectral interferences that manifest as a change in sensitivity due to a change in inductively coupled plasma conditions when the matrix of a calibration or test solution is introduced into the plasma.

ISO 15202

3.2.11 *field blank, n*—sampling media (for example, a sampling wipe) that is exposed to the same handling as field samples, except that no sample is collected (that is, no surface is wipe sampled). **D6785**

3.2.11.1 *Discussion*—Analysis results from field blanks provide information on the analyte background level in the sampling media, combined with the potential contamination experienced by samples collected within the batch resulting from handling.

3.2.12 *inductively coupled plasma (ICP), n*—a high-temperature discharge generated by a flowing conductive gas, normally argon, through a magnetic field induced by a load coil that surrounds the tubes carrying the gas. **ISO 15202**

3.2.13 *inductively coupled plasma (ICP) torch, n*—a device consisting of three concentric tubes, the outer two usually made from quartz, that is used to support and introduce sample into an ICP discharge. **ISO 15202**

3.2.14 *injector tube*, *n*—the innermost tube of an inductively coupled plasma torch, usually made of quartz or ceramic materials, through which the sample aerosol is introduced to the plasma. **ISO 15202**

3.2.15 *inner (nebulizer) argon flow, n*—the flow of argon gas that is directed through the nebulizer and carries the sample aerosol through the injector and into the plasma.

3.2.15.1 Discussion—Typically 0.5 to 2 L/min. ISO 15202

3.2.16 *instrumental detection limit (IDL), n*—the lowest concentration at which the instrumentation can distinguish analyte content from the background generated by a minimal matrix. **E1613**

3.2.16.1 *Discussion*—The IDL pertains to the maximum capability of an instrument and should not be confused with the method detection limit (MDL).

3.2.17 *interelement correction, n*—a spectral interference correction technique in which emission contributions from interfering elements that emit radiation at the analyte wavelength are subtracted from the apparent analyte emission after measuring the interfering element concentrations at other wavelengths. **ISO 15202**

3.2.18 *intermediate (auxiliary) argon flow, n*—the flow of argon gas that is contained between the intermediate and center (injector) tubes of an inductively coupled plasma torch.

3.2.18.1 Discussion-Typically 0.1 to 2 L/min. ISO 15202

3.2.19 *internal standard*, *n*—a non-analyte element, present in all calibration, blank, and sample solutions, the signal from which is used to correct for non-spectral interference or improve analytical precision. **ISO 15202**

3.2.20 *linear dynamic range, n*—the range of concentrations over which the calibration curve for an analyte is linear. It extends from the detection limit to the onset of calibration curvature. **ISO 15202**

3.2.21 *load coil, n*—a length of metal tubing (typically copper) which is wound around the end of an inductively coupled plasma torch and connected to the radio frequency generator. **ISO 15202**

3.2.21.1 *Discussion*—The load coil is used to inductively couple energy from the radio frequency generator to the plasma discharge.

3.2.22 *matrix interference, n*—interference of a non-spectral nature which is caused by the sample matrix.

3.2.22.1 *Discussion*—Matrix matching involves preparing calibration solutions in which the concentrations of acids and other major solvents and solutes are matched with those in the test solutions. ISO 15202

3.2.23 *method quantitation limit (MQL), n*—the minimum concentration of an analyte that can be measured with acceptable precision, ordinarily taken to be at least ten times the standard deviation of the mean blank signal (1).⁷

3.2.23.1 *Discussion*—The MQL is also known as the limit of quantitation.

3.2.24 *nebulizer, n*—a device used to create an aerosol from a liquid. ISO 15202

3.2.25 *outer (plasma) argon flow, n*—the flow of argon gas that is contained between the outer and intermediate tubes of an inductively coupled plasma torch.

3.2.25.1 *Discussion*—Typically 7 to 15 L/min. **ISO 15202** 3.2.26 *pneumatic nebulizer*, *n*—a nebulizer that uses highspeed gas flows to create an aerosol from a liquid. **ISO 15202**

3.2.27 *radial plasma*, *n*—an inductively coupled plasma that is viewed from the side (versus axial).

3.2.28 *sample dissolution*, n—the process of obtaining a solution containing the analyte(s) of interest from a sample. This may or may not involve complete dissolution of the sample. **D6785**

3.2.29 *sample preparation*, *n*—all operations carried out on a sample, after transportation and storage, to prepare it for analysis, including transformation of the sample into a measurable state, where necessary.

3.2.30 *spectral interference, n*—an interference caused by the emission from a species other than the analyte of interest. **ISO 15202**

3.2.31 *spray chamber*, *n*—a device placed between a nebulizer and an inductively coupled plasma torch whose function is to separate out aerosol droplets in accordance with their size, so that only very fine droplets pass into the plasma, and large droplets are drained or pumped to waste. **ISO 15202**

3.2.32 *stock standard solution, n*—solution used for preparation of working standard solutions and/or calibration solutions, containing the analyte(s) of interest at a certified concentration(s) traceable to primary standards (National Institute of Standards and Technology (NIST) or international measurement standards).

 $^{^{7}\,\}mathrm{The}$ boldface numbers in parentheses refer to a list of references at the end of this standard.