



Designation: E 2120 – 00

Standard Practice for the Performance Evaluation of the Portable X-Ray Fluorescence Spectrometer for the Measurement of Lead in Paint Films¹

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1. Scope

1.1 This practice covers portable x-ray fluorescence (XRF) instruments intended for the measurement of lead in paint. It is intended that manufacturers apply this practice to one unit of a particular model of an instrument when that model is initially available. Replicate tests on additional units of the same model of an instrument are to be performed at the discretion of the manufacturer. This practice also is intended for use by third parties performing independent evaluation of portable x-ray fluorescence instruments.

1.2 All performance evaluation data are to be in International System of Units (SI) units.

1.3 Tests of performance are based on replicate measurements of certified reference paint films on a variety of substrate materials. Tests are performed to determine: bias, precision, linearity, limit of detection, interferences, substrate affects, and stability.

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 3332 Test Methods for Mechanical Shock Fragility of Products, Using Shock Machines²
- E 344 Terminology Relating to Thermometry and Hydrometry³
- E 456 Terminology Relating to Quality and Statistics⁴
- E 1605 Terminology Relating to Abatement of Hazards from Lead-Based Paint in Buildings and Related Structures⁵

2.2 ANSI Standards:

- American National Standard N538–1979, “Classification of Industrial Ionizing Radiation Gauging Devices”⁶
- American National Standard N542–1977, “Sealed Radioactive Sources, Classification”⁶

2.3 Underwriters Laboratory Standards:

- Underwriters Laboratory Standard 544, “Medical and Dental Equipment”⁷
- Underwriters Laboratory Standard 3101–1, “Chemical Analyzers”⁷

3. Terminology

3.1 Definitions:

3.1.1 *accuracy, n*—the theoretical maximum error of a measurement, expressed as the proportion of the amount being measured without regard for the direction of the error, that is achieved with a given probability (typically 0.95) by the method.⁸

3.1.2 *bias, n*—the discrepancy between the mean of the distribution of measurements from a method and the true concentration being measured.

3.1.3 *limit of detection, n*—the smallest (true) signal that will be detected with a probability $1 - \beta$ (β is the probability of an error of the second kind, failing to decide that a substance is present when it is), where the a posteriori decision mechanism has a built-in protection level, α (α is the probability of an error of the first kind, deciding that the substance is present when it is not), against falsely concluding that a blank observation represents a “real” signal. The β and α terms typically are 5 % or 1 %, depending on the requirements of the testing program.⁹

3.1.4 *precision, n*—the closeness of agreement between repetitive test results obtained under prescribed conditions (see

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² *Annual Book of ASTM Standards*, Vol 15.09.

³ *Annual Book of ASTM Standards*, Vol 14.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ *Annual Book of ASTM Standards*, Vol 04.11.

⁶ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁷ Available from Underwriters Laboratories, Inc., Research Triangle Park, NC.

⁸ Kennedy, E.R., T.J. Fischbach, R. Song, P.M. Miller, and S.A. Shulman, *Guidelines for Air Sampling and Analytical Method Development and Evaluation* DHHS (NIOSH) Publication No. 95–117. National Institute for Occupational Safety and Health, Cincinnati, OH 45226, May 1995.

⁹ Currie, L.A., “Limits for Qualitative Detection and Quantitative Determination,” *Anal. Chem.*, 40 (3), pp. 586–593, 1968.

Terminology E 456). The precision of a single instrument is the random component of its accuracy and is usually indicated by the value of the standard deviation.

3.2 The definitions given in Terminologies E 344 and E 1605 shall apply to this practice.

3.3 Definitions of Terms Specific to This Standard:

3.3.1 *battery charger, n*—a means for recharging a portable instrument's self-contained battery pack, usually converting 110 V AC to low level DC power.

3.3.2 *cycle or reading time, n*—a period of x-ray data collection (counting) performed automatically by some instruments. Such time may be established by a standardization procedure that would adjust for variation in the strength of the radioactive source. Also, it might be adjustable on some instruments to achieve different levels of measurement precision. Depending on the instrument model, one "cycle time" may be equivalent to one "measurement time" or several "cycles" may be automatically or manually averaged to equal one "measurement time." The time begins with the opening of the XRF instrument shutter to expose the paint film surface to the source radiation and is concluded when the source shutter is closed.

3.3.3 *display unit, n*—an electronic device that presents the results of the measurement to the user. Other parameters such as total measurement time also may be presented.

3.3.4 *measurement time, n*—the duration of a single measurement observed in real time. A measurement may comprise several individual readings or cycles.

3.3.5 *measurement value, n*—the readout of a lead concentration in mg/cm² obtained at the end of one cycle time (or several cycle times if multiple readings are averaged) or at the end of one measurement time.

3.3.6 *probe, n*—a hand-held device containing the radioactive source, x-ray detector, and associated mechanical and electrical components that is placed against the test sample to perform the measurement. The probe may constitute a part or all of the XRF instrument.

3.3.7 *radioactive source, n*—a radioactive material (for example, ⁵⁷Co, ¹⁰⁹Cd, and ²⁴¹Am) that emits X rays or gamma rays that serve to cause ionization of the lead atoms in the sample, and subsequently a cascade of higher energy electrons into the vacated lower energy shells. As these electrons fall into the lower energy orbitals, they emit energy in the form of X rays that are characteristic of lead.

3.3.8 *standard paint films, n*—free-standing, certified reference paint films, that is, certified reference materials (CRMs), that are acquired from the National Institute of Standards and Technology (NIST) or a commercial vendor. The lead levels in the standard paint films (CRMs) shall be based on "x" level for lead where "x" is equal to the appropriate local, state, or federal action level for lead in coatings (in mg/cm² of lead coating). The paint films shall be as follows:

- (Film 1) <0.2x
- (Film 2) 0.2x to 0.5x
- (Film 3) 0.7x to 1.3x
- (Film 4) 1.5x to 2x
- (Film 5) >2x

3.3.8.1 *Discussion*—An example CRM in use is the NIST Standard Reference Material (SRM) 2579, which consists of five films at <0.0001, 0.29, 1.02, 1.63 and 3.53 mg Pb/cm², respectively.

NOTE 1—The supply of NIST SRM 2579 standard paint films is now (©1998) exhausted, though it is likely that this SRM will be replaced with a new SRM. CRMs from NIST or commercial vendors may be used in place of NIST SRM 2579, provided the action level of concern, for example, HUD action level of 1.0 mg/cm², is represented by one of the new films.

3.3.9 *stray radiation, n*—the sum of leakage and scattered radiation as measured according to 7.1.8.

3.3.10 *useful beam, n*—radiation that passes through the window aperture, cone, or other collimating device of the source housing; sometimes called *primary beam*.

3.3.11 *X ray detector, n*—a device that generates an electronic signal as a result of the interception of an X ray. Examples include gas proportional counters, for example, Xe, solid scintillation counters, for example, CsI, and semiconductor devices of elemental, for example, Si or Ge, or compound, for example, HgI₂, CdTe, or CdZnTe, composition.

4. Summary of Practice

4.1 The x-ray instrument is evaluated with respect to a series of manufacturer's requirements for bias, precision, effects of environment, data display, battery operation, construction, markings, and documentation. The performance of the instrument is evaluated in the laboratory by measuring a series of standard lead-containing paint films placed on a wide variety of different substrate materials. Data from replicate measurements and comparison of measured and expected values are then used to determine bias, precision, limit of detection, linearity, interferences, substrate effects, and stability; radiation safety is evaluated, as well.

5. Significance and Use

5.1 The XRF instrument is used to measure the lead content in paint films in buildings and related structures in order to determine the potential lead hazard and the possible need for in-place control or abatement, or both.

5.2 This practice also is to be used for the laboratory evaluation of the performance of portable x-ray fluorescence instrumentation.

5.3 This practice is to be used as a guide for determining that the manufacturer of portable x-ray instrumentation has met certain requirements, most of which deal with instrument construction.

5.4 The evaluation may be performed by the manufacturer, or an independent party. The results may be presented to various government agencies and, upon request, potential purchasers and users of the instrumentation. All or parts of this practice also may be performed by an x-ray instrument owner/user to determine the acceptability of an instrument or whether the performance of an instrument continues to be acceptable, or both.

5.5 This practice may be used by field testers for quality control by performing selected activities described in the document on a regular and recurring basis in a manner similar to those protocols followed by users of laboratory instruments.

5.6 *Limitation*—Bias and precision, as determined in the laboratory by this practice, together provide only an estimate of the accuracy that may be achieved in the field. Accuracy in the field will depend upon the instrument calibration, the form and composition of the substrate, the structure of the paint film being analyzed, as well as other factors.

6. Requirements

6.1 Unless otherwise specified, the following requirements are to be met by the manufacturer of the x-ray instrument.

6.1.1 *Bias*—The manufacturer shall provide a value or values for the bias of the instrument model that has [have] been determined using the procedure presented in Section 7 of this practice. This value shall be made available to potential users, and also to interested parties, for example, the U. S. Environmental Protection Agency and the U. S. Department of Housing and Urban Development.

6.1.2 *Precision*—The manufacturer shall provide a value or values for the precision of the instrument model that has been determined using the procedure presented in Section 7 of this practice. This value shall be made available to potential users, and also to interested parties, for example, the U. S. Environmental Protection Agency and the U. S. Department of Housing and Urban Development.

6.1.3 *Limit of Detection*—The manufacturer shall provide a value or values for the limit of detection of the instrument that has been determined using the procedure presented in Section 7 of this practice. This value shall be made available to potential users, and also to interested parties, for example, the U. S. Environmental Protection Agency and the U. S. Department of Housing and Urban Development.

6.1.4 *Environment:*

6.1.4.1 *Operating Environment*—The instrument shall be capable of meeting the manufacturer's performance specifications for bias and precision when operating in an environment of 2 to 35°C (35 to 95°F) and a relative humidity of 15 to 95 %, noncondensing.

6.1.4.2 *Storage Environment*—The instrument shall be capable of meeting the manufacturer's performance specifications for bias and precision after imposing each of the following conditions: storage at 43°C (110°F) for up to one month; transportation for up to 12 h at a maximum of 50°C (120°F), with a relative humidity of 15 to 95 %, noncondensing; storage at 0°C (32°F) for up to one month; and transportation for up to 12 h at a maximum of -10°C (15°F). The manufacturer shall indicate the period of time necessary for the instrument's batteries to be charged and for the instrument to stabilize at the measurement temperature prior to a calibration check, or recalibration, if necessary.

6.1.4.3 *Manufacturer's Instructions*—The instruction manual shall include a statement that informs the user if the performance of the device may be degraded should one or more of the following occur:

(a) Operation outside the manufacturer's stated temperature and humidity range.

(b) Storage outside the manufacturer's stated temperature and humidity range.

(c) Mechanical shock equivalent to hitting the probe or display unit against a door frame or similar object, or dropping either more than a distance of 30 cm (1 ft).

NOTE 2—The critical velocity, V_c , which is the velocity at which product failure just begins to occur, may be determined using Test Methods D 3332. (Formal procedure for testing for failure due to rapid change in the velocity occurring from collision.)

6.1.5 *Data Display Resolution*—The digital data display shall have incremental steps not greater than 0.1 mg/cm²; that is, a resolution of at least ± 0.1 mg/cm².

6.1.6 *Battery Condition*—When the instrument is battery operated, the bias, precision, and other operational parameters such as calibration stability shall not be affected by battery condition, unless a continuous automatic indication of unreliable battery condition is provided. The indication of unreliable battery condition must be presented until the battery condition is corrected. When an instrument uses a rechargeable battery, some indication shall be provided by the instrument system to indicate that the battery is charging.

6.1.7 *Construction:*

6.1.7.1 *Electrical*—The instrument and accessories (such as battery chargers) shall meet the electrical safety requirements of UL 544 and UL 3101-1.

6.1.7.2 The surface of the instrument including the probe, control console, and accessories shall withstand physical cleaning using a damp cloth, HEPA vacuum, or manufacturer's recommended procedures without performance degradation.

6.1.7.3 The instrument shall withstand a free fall of 3 m (10 ft) onto a flat concrete surface at 25°C (77°F) without evidence of mechanical or electronic failure that could present a radiation, or electrical safety hazard, or both. The essential criteria for passing such a test are that there shall be no external dispersal of radioactive material and that the source capsule shall remain captive in its protective source housing. Dummy sources and x-ray detection devices may be used in these evaluations.

6.1.7.4 *Radiation*—The instrument shall meet the radiation safety features and requirements presented in Section 3 of ANSI N538-1979.

(a) *Source Integrity*—The radioactive material shall be in the form of a sealed source that shall be classified in accordance with ANSI N542-1977.

(b) *Gamma and X-Ray Beam Controls*—The probe shall include a manual excitation beam control designed so that when the source shutter is in the OFF condition, the measured radiation levels in the useful beam space are no greater than a dose rate of 50 Sv/h at 30 cm and 1000 Sv/h at 5 cm as measured in accordance with the procedures summarized in 7.1.8. A locking mechanism shall be provided to physically secure the radiation beam in the fully OFF condition. An easily checked indicator signal, which positively indicates when the excitation-useful beam control system is not in the fully OFF

condition, and when it is in the fully OFF condition, shall be located on or adjacent to the radiation source housing.

6.1.8 *Marking*—All markings for purposes of identification or instruction must be clear and legible. Deterioration of the markings on the instrument shall not occur when subjected to cleaning as described in 6.1.7.2 or according to manufacturer's procedures.

6.1.8.1 *Instrument Marking*—The instrument shall be marked with the manufacturer's or distributor's name, model designation, and serial number. It also shall be marked with the date of the most recent calibration of the instrument, as performed by the manufacturer. Alternatively, this latter information may be provided in a written document that is provided with the instrument.

6.1.8.2 *Measurement Probe*—The portion of the instrument containing the radioactive material shall be marked with a durable, permanent label indicating the type and amount of radioactive material, the measurement (radio assay) date, the standard radiation symbol, and a caution notice that shall read as follows, or similarly: **CAUTION—RADIOACTIVE MATERIAL.**

6.1.8.3 *Operating Instructions*—Operating instructions shall be provided with the instrument. Radiation safety warnings must be provided on the instrument.

6.1.8.4 *Care and Use Instructions*—Instructions for the care, use, and physical cleaning of the instrument shall be provided. Proper use and application of accessories, such as the battery charger shall be indicated.

6.1.8.5 *Health and Safety Hazard Marking*—Notices, as required by local, state, and federal regulations, shall be displayed on the instrument.

6.1.8.6 *Identification*—In order that purchasers may identify products conforming to all requirements of this specification, producers and distributors may include a statement of compliance in conjunction with their name and address on product labels, invoices, and sales literature. The following statement is suggested when sufficient space is available: This x-ray fluorescence instrument conforms to all of the requirements established in ASTM Standard E06.23.11. Full responsibility for conformance of this product to the specification is assumed by (name and address of producer or distributor).

6.1.9 *Documentation:*

6.1.9.1 *Manufacturer's Instructions*—Instructions for use shall be provided. These instructions shall contain sufficient detail to provide a means for training in the operation, application, care, and physical cleaning of the instrument and accessories.

6.1.9.2 *Service and Repair Manual*—A service manual shall be made available if user repair is permitted by the manufacturer. The service manual shall provide theory of operation, maintenance information, test procedures, test equipment requirements, detailed diagrams, parts list, and specifications.

6.1.9.3 *Bias and Precision Determinations*—The manufacturer shall make available specific instructions for tests to determine bias and precision of the instrument, and shall provide the results of the instrument performance evaluation.

6.1.9.4 *Recalibration*—The manufacturer shall recommend a periodic recalibration cycle, as necessary, to ensure continu-

ous performance to the requirements of 6.1.1 and 6.1.2. The manufacturer shall provide specific instructions for the adjustment of the instrument if user adjustment is permitted by the manufacturer. Test equipment or fixtures required for adjustment must either be described in sufficient detail to permit fabrication or purchase, or manufacturer's equipment or fixtures must be made available to users.

6.1.9.5 *Detailed Specifications*—The manufacturer shall provide specifications of the instrument's bias (see 6.1.1), precision (see 6.1.2), and environmental limitations (see 6.1.4).

7. Performance Tests

7.1 This section describes apparatus and procedures for verifying conformance to certain performance requirements of Section 6. These tests are not required of the manufacturer unless specified by the user. Verification procedures are not included for requirements that can be verified by observation or inspection, or where a standard procedure is not needed, such as the requirements of 6.1.4.1. The manufacturer shall certify that the product will comply with the requirements, if tested in accordance with this section. With the exception of the potentially destructive tests, any single portable x-ray fluorescence instrument shall be capable of undergoing the following tests in any sequence without impairment of performance.

NOTE 3—Bias, precision, and limit of detection, as determined by this practice, provide only estimates of the values that may be achieved in the field.

NOTE 4—It is expected that the manufacturer will evaluate only one unit of each model type produced and will reevaluate that model when any changes that could affect performance are made.

NOTE 5—The performance tests described below use a fixed total measurement time of nominally one (1) minute. Modification of these performance evaluation tests may be needed for instruments that are operated using variable measurement times, for example, instruments that read until the relative standard deviation of the number of X rays counted reaches a preset value.

7.1.1 Calibration:

7.1.1.1 The manufacturer shall provide a general description of the calibration procedure used, including the calibration range, a list of the standards used, and a list of the substrate(s) used or a description of the assumptions made regarding substrates; conditions under which the calibration is not applicable shall be presented.

7.1.1.2 The manufacturer shall provide a procedure and either the required standards or descriptions of needed standards to perform a check of the manufacturer's calibration. This procedure shall include instructions for performance of data treatment and criteria for deciding if the calibration is within the manufacturer's specifications.

7.1.2 *Accuracy*—Accuracy may be calculated from values of bias and precision using rigorous statistical procedures. These procedures can be found in the NIOSH Technical Report, "Guidelines for Air Sampling and Analytical Method Development and Evaluation."⁸ Such calculations are not required for this practice; rather, accuracy is to be represented by individual values of bias and precision.

7.1.2.1 *Bias*—This test is to be performed in an environment with a temperature of 19 to 24°C (66 to 75°F) and a relative humidity of 40 to 60 %. The excitation source shall