Designation: E2120 – 10 (Reapproved 2016)

Standard Practice for
Performance Evaluation of the Portable X-Ray Fluorescence Spectrometer for the Measurement of Lead in Paint Films[1]

This standard is issued under the fixed designation E2120; 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 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 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:*[2]

D3332 Test Methods for Mechanical-Shock Fragility of Products, Using Shock Machines
E344 Terminology Relating to Thermometry and Hydrometry
E456 Terminology Relating to Quality and Statistics
E1605 Terminology Relating to Lead in Buildings

2.2 *ANSI Standards:*[3]

ANSI N538–1979 Classification of Industrial Ionizing Radiation Gauging Devices
ANSI N323–1978 Radiation Protection Instrumentation Test and Calibration

2.3 *ISO Standards:*[3]

ISO 2919 Radiation Protection – Sealed Radioactive Sources – General Requirements and Classification

2.4 *UL Standards:*[4]

UL 544 Safety for Medical andDental Equipment
UL 3101–1 Chemical Analyzers

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.[5]

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 – β (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.[6]

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[1] This practice is under the jurisdiction of ASTM Committee E06 on Performance of Buildings and is the direct responsibility of Subcommittee E06.23 on Lead Hazards Associated with Buildings.

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

[4] Available from Underwriters Laboratories (UL), 333 Pfingsten Rd., Northbrook, IL 60062-2096, http://www.ul.com.

[5] 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.

[6] Currie, L.A., "Limits for Qualitative Detection and Quantitative Determination," *Analytical Chemistry*, Vol 40, No. 3, 1968, pp. 586–593.

3.1.4 *precision, n*—the closeness of agreement between repetitive test results obtained under prescribed conditions (see Terminology E456). 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 E344 and E1605 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/cm^2 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, ^{57}Co, ^{109}Cd, and ^{241}Am) 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/cm^2 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/cm^2, respectively.

NOTE 1—The supply of NIST SRM 2579 standard paint films is now (as of 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/cm^2, 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, HgI_2, 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:

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

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

(3) 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 D3332. (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/cm^2; that is, a resolution of at least ±0.1 mg/cm^2.

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.

(1) Source Integrity—The radioactive material shall be in the form of a sealed source that shall be classified in accordance with ISO 2919.

(2) 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 this practice. 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 continuous 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 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."[5] 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 have an isotopic strength within ±10 % of the isotopic strength of a nominally "fresh" or new source. To perform the test, use a wooden box having an inside width of 24 cm (9½ in.) and inside length of 37 cm (14½ in.) and a height of about 50 cm (20 in.) constructed from 10 mm (⅜ in.) plywood, or equivalent. The top of the box shall be open, and the box is to be filled with at least 35 cm (14 in.) of polyfoam beadboard (without foil facing). Center a 23–cm (9–in.) by 35 cm (14 in.) by 2.5 cm (1 in.) thick white pine board, which is clear of major imperfections such as knots, on the polyfoam in the box. Center a CRM at <0.2x on the top of the white pine board. Place the instrument on the film, and perform a total of ten measurements without moving the instrument. Each measurement is to consist of a total of nominally 1 min of measurement time. If the manufacturer's instrument automatically performs measurement cycles of less than one minute, perform the number of measurement cycles necessary to achieve at least 1 min of total measurement time and average the measurement values to obtain one "measurement" value. Using this same experimental system and test conditions, perform ten 1-min measurements with each of four additional CRMs as described in 3.3.8 (0.2x to 0.5x, 0.7x to 1.3x, 1.5x to 2x, and >2x). Next, perform ten 1-min measurements with each of these five CRM paint films (<0.2x, 0.2x to 0.5x, 0.7x to 1.3x, 1.5x to 2x, and >2x) on five different, additional substrates. Representative substrates and their order of testing are as follows:

(1) 0.76-mm (0.030-in.) T6, 6061 aluminum sheet, or 45-mm (1.75-in.) hollow core door, or 3.2-mm (0.125-in.) thick veneer paneling (cut to fit in the test box).

(2) 13-mm (0.5-in.) thick gypsum board, plasterboard, or 15- to 20-mm (0.625- to 0.75-in.) thick birch, fir, or oak boards cut to fit in the test box.

(3) 6-mm (0.25-in.) thick "836" steel plate cut to fit in the test box.

(4) Uncolored cinderblock slab or poured concrete slab 50-mm (2-in.) thick cut to fit in the test box.

(5) 40-mm (1.5-in.) thick red brick (place three-four bricks side by side in the test box).

7.1.2.2 Calculate and report the mean and standard deviation for each of these thirty measurement conditions. Report the differences between each mean value and expected CRM value as the bias of the measurement for each substrate and concentration. Include in the report a description of each substrate material and the method of substrate correction.

NOTE 6—Correction for substrate bias is to be performed according to the manufacturer's standard procedure.

NOTE 7—Some instruments may display a default value, for example, 1 mg/cm^2, if the measurement value is within some predetermined range of an action level or other level of concern. The default value cannot be used in this evaluation; the actual measurement value must be used. If the actual measurement value cannot be determined (displayed), perform this evaluation with a CRM that is removed from the action level or other level of concern such that it does not result in the display of a default value.

7.1.2.3 *Precision*—Using the data from the measurements performed under 7.1.2.1 and 7.1.2.2, calculate the slope, standard deviation of the slope, intercept, and standard deviation of the intercept of the measured versus expected values using a linear regression analysis.[7] Also calculate and report the correlation coefficient. Prepare a plot of standard deviations of the average of each of the ten measurement values versus the concentrations of the standard films. Include these results in the report of this evaluation.

NOTE 8—These results are not to be used as an indication of the instrument's accuracy. They are solely to characterize the repeatability of the readings.

NOTE 9—Bias and precision, as determined by this practice, provide only estimates of the bias and precision that may be achieved in the field. Bias and precision in the field will depend upon the instrument calibration, the form and composition of the substrate, and the structure of the paint film being analyzed.

7.1.2.4 *Linearity with Real-World Substrates*—The slope, standard deviation of the slope, and the correlation coefficient from 7.1.2.3 are taken as a measure of the linearity of response of the instrument.

7.1.3 *Estimate of Limit of Detection (LOD):*

7.1.3.1 The procedure presented here for estimating LOD has been developed especially for radiochemical applications. It has the advantage that it allows precision to vary with concentration, as predicted by counting statistics. This procedure is a simplification of the general method described by L.A. Currie.[6] The determination of the limit of detection is based on Hypothesis Testing, which is subject to two kinds of error: deciding that lead is present at or above a specified level, when it is not (error of the first kind), and conversely, deciding that lead is not present at or above a specified level, when in fact, it is (error of the second kind). In this approach, readings that fall above a particular threshold value, x_t, are classified as "containing lead," while readings that fall below the threshold are classified as "not containing lead." The analyst selects the threshold value, x_t, such that if the true concentration of lead on a surface is zero (0 mg/cm^2), 95 % of the readings on that zero-lead surface will fall below the threshold value, x_t, and therefore will be classified correctly as "not containing lead," while only 5 % of the readings on that zero-lead surface will be misclassified as "containing lead." The limit of detection, x_{LOD}, is the true concentration of a lead-bearing surface such that 95 % of the readings on that surface will fall above the threshold x_t and therefore will be correctly classified as "containing lead," while only 5 % of the readings on that lead-bearing surface will be misclassified as "not containing lead." Note that x_{LOD} is always higher than x_t, and is almost always at least twice x_t.

7.1.3.2 As noted, this procedure has the advantage that it allows precision to vary with concentration, as predicted by counting statistics, but it does not require intimate knowledge of the instrument calibration. Simplifying assumptions are that the variance of the population, σ_x^2, is linear with concentration ($\sigma_x^2 = A + Bx$), as is predicted by counting statistics, and that the distribution of readings is purely Gaussian, rather than the more exact Poisson (see Appendix X1 for additional information).

7.1.3.3 The procedure is as follows:

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[7] Hartley, T.F., *Computerized Quality Control: Programs for the Analytical Laboratory*, Chap. 1, John Wiley & Sons, 1987.

(1) Twenty replicate 1-min measurements are to be performed at a known concentration, x_0, below the expected detection limit. If an instrument provides for the display of negative results, a nondetectable-lead sample is to be used. If an instrument does not provide for the display of negative results, a lead standard of sufficient level to produce only positive results must be used. If the instrument provides for the display of negative results, use the same system and test conditions described in 7.1.2 and perform ten additional measurements with the CRM at <0.2x on the 1-in. white pine board (additional substrates may be used for this test). Using the data from 7.1.2 in combination with these data (for a total of 20 measurement values), calculate the standard deviation (σ_0) of the value for this CRM. As noted, if the instrument does not provide for the display of negative results, x_0 should be chosen large enough such that all readings are above zero. Assuming this higher level CRM is chosen, use the same system and test conditions described in 7.1.2, and perform ten additional measurements with this CRM on the 1-in. white pine board (additional substrates may be used for this test). Using the data from 7.1.2 in combination with these data (for a total of 20 measurement values), calculate the standard deviation (σ_0) of the value for this higher level CRM.

(2) Measure 20 replicates at a known concentration, x_1, above the expected detection limit. A CRM at 0.7x to 1.3x would be appropriate in most cases. Assuming this CRM is chosen, use the same system and test conditions described in 7.1.2, and perform ten additional measurements with this standard paint film on the 1-in. white pine board (additional substrates may be used for this test). Using the data from 7.1.2 in combination with these data (for a total of 20 measurement values), calculate the standard deviation (σ_1) of the value for this standard paint film.

(3) Calculate linear coefficients A and B as:

$$A = \frac{\sigma_0^2 x_1 - \sigma_1^2 x_0}{x_1 - x_0} \tag{1}$$

$$B = \frac{\sigma_1^2 - \sigma_0^2}{x_1 - x_0} \tag{2}$$

(4) If B is negative, reset it to zero. If A is negative, you have made a serious error.

(5) Calculate threshold level, x_t, as:

$$x_t = 1.645\sqrt{A} \tag{3}$$

NOTE 10—Currie calls the threshold level L_c.

(6) Calculate detection limit, x_{LOD}, as:

$$x_{LOD} = 3.29\sqrt{A} + 2.706\,B \tag{4}$$

NOTE 11—Currie calls detection limit L_D.

(7) If x_{LOD} falls below x_0, choose a lower concentration for x_0, and go back to Step 1.

(8) If x_{LOD} falls above x_1, choose a higher concentration for x_1 and go back to Step 2.

NOTE 12—Instruments set with a display resolution of 0.1 mg/cm^2 (that is, displays only one place to the right of the decimal point) may seem to have a precision better than can be displayed. If the instrument display resolution leads it to give identical readings for all replicates in a set, σ for that set should be changed from 0 to the display resolution divided by 3.46 before making calculations. (This calculation is based on the assumption that the probability distribution for the result will be flat over the interval [–resolution/2, +resolution/2] and zero outside the interval; then the root mean square width of a square bin is the bin width divided by the square root of 12.

NOTE 13—The x_{LOD} determined by this procedure is specific to the conditions of the test, which are the particular substrate and paint matrix and measurement time.

NOTE 14—The actual level of lead that can be measured is set by the combination of the precision and the bias of the instrument. This level may be substrate dependent.

(**Warning—**The x_{LOD} determined by this provisional practice provides only an estimate of the detection limit that may be achieved in the field.)

7.1.4 *Potential Sources of Error*—Potential sources of error include read-through, paint thickness, and paint layering. The following sections provide procedures to test for these potential sources of error.

7.1.4.1 *Effect of Read-Through*—Instruments have the potential for detecting lead behind the outermost substrate, for example, lead-containing paint on wood that has been covered by drywall. The following is performed to test for this potential source of error (bias).

7.1.4.2 Using the system and test conditions described in 7.1.2, perform ten one minute measurements with the 0.7x to 1.3x CRM on the ½-in. thick white pine board described in 7.1.6.1 having placed the >2x CRM underneath the pine board, with painted surfaces toward the XRF unit. Calculate the mean and the standard deviation for the ten measurements and report the difference between the mean value and expected value as a bias to the X-ray measurement due to read through.

7.1.4.3 *Effects of Paint Thickness*—Instruments have the potential for yielding a low response to lead within the innermost layers of very thick coatings of paint. The following is performed to test for this potential source of error (bias). Using the system and test conditions described in 7.1.2, perform ten 1-min measurements with the 1-in. white pine board and CRMs arranged in the following configurations: (1) the 1.5x to 2x CRM covered by the 0.7x to 1.3x CRM covered by the <0.2x CRM and; (2) the 0.7x to 1.3x CRM covered by the 0.2x to 0.5x CRM covered by the <0.2x CRM; all painted surfaces toward the XRF unit. Calculate the mean and the standard deviation for the ten measurements for both setups and report the difference between the mean values and the expected sum as bias to the instrument's measurements due to paint thickness.

NOTE 15—Real paint films are not usually as thick as the procedure prescribes. The purpose of the test is to show the potential impact, if any, of this particular condition, and also the ability of the instrument to compensate for this particular condition.

7.1.4.4 *Effects of Paint Layering*—Instruments have the potential for yielding a low response to lead within the innermost layers of multi-layered coatings of paint. The following is performed to test for this potential source of error (bias). Using the system and test conditions described in 7.1.2, perform ten 1-min measurements with the 1-in. white pine board and CRMs arranged in the following configurations: the 0.7x to 1.3x CRM covered by one, two, and three <0.2x CRMs, and the 1.5x to 2x CRM covered by one, two, and <0.2x CRMs; all painted surfaces toward the XRF units. Calculate the mean and the standard deviation for the ten measurements for all six of the setups and report the differences between the mean values and the expected values as bias to the instrument's measurements due to paint layering.

NOTE 16—Increasing thickness of paint and paint layering will result in fewer lead X-rays that are generated within the lowest layer(s) of paint from reaching the detector because of absorption of these X-rays by upper layers of paint, and decrease of radiation intensity with distance from the source (Inverse Square Law). Any instrument showing no effect from increasing thickness or layering should be considered suspect.

7.1.5 *Interferences:*

7.1.5.1 No standard test for interferences can be prescribed because of the lack of standard reference paint films with known levels of potential interferences. Experience has indicated, however, that the following potentially interfering elements may be found in paint, or substrates, or both:

K X-ray interferences–bismuth, mercury
L X-ray interferences–arsenic, bromine, copper, zinc

7.1.5.2 Bismuth and mercury are unlikely to be found in paint or substrates at levels high enough to cause problems; however, tests for interference from the copper, zinc, and arsenic may be developed using copper roof flashing, galvanized sheet metal, and arsenic-treated lumber, respectively. Measurement may be made on the materials directly or with placement of the <0.2x CRM standard over the substrate. Describe the substrate materials used and report the false lead signals measured, if any.

NOTE 17—The extent of interference is affected by the type of source and detector used.

7.1.6 *Substrate Effects:*

7.1.6.1 No two substrates are exactly the same. For example, gypsum board from one manufacturer will differ in composition, thickness and form from gypsum board from another manufacturer; therefore, in absence of any standard reference materials of this sort, the effects of various substrates can at best be estimated using substrate materials readily available to the tester. Using the same experimental system and test conditions described in 7.1.2, perform ten 1-min measurements with the 0.7x to 1.3x CRM on each of the following substrate materials:

(1) Cellulose-based material (for example, acoustical tile),
(2) ¼-in.-thick plywood (untreated),
(3) ½-in.-thick white pine board (untreated),
(4) ⅛-in.-thick steel sheet,
(5) Precast masonry; for example, patio block.

7.1.6.2 The tester is encouraged to include the following additional substrates in this test:

(1) Metal clad door section,
(2) Galvanized sheet metal,
(3) Plaster and metal lath wall section,
(4) Plaster and wood lath wall section,
(5) Bar-reinforced concrete section,
(6) Drywall over 2 × 4 studs,
(7) Drywall over metal electrical box.

7.1.6.3 Calculate the mean and standard deviation of the data for each substrate, and report the difference between the mean values and expected value as the bias of the measurement for each substrate. Thoroughly describe the substrate materials used in these tests.

NOTE 18—There are differences between the substrates listed in 7.1.2 and 7.1.6 in order to collect data with a maximum number of different substrates. Manufacturers are encouraged to provide similar data for other substrates.

NOTE 19—As noted, no two substrates are totally equivalent. Real world substrates may have cracks and contain nails and other foreign materials that may affect measurement results. Furthermore, it is not uncommon to find multiple layers of substrates that will affect the bias of the measurement; for example, metal sheet or veneer over a wooden core door, or wood, plaster, or gypsum board over some form of masonry.

7.1.7 *Stability:*

7.1.7.1 *Stability with Temperature*—Repeat the bias test described in 7.1.2.1 at 2°C (35°F) [cold], 21°C (70°F) [room temperature], and 35°C (95°F) [hot] using the 0.7x to 1.3x CRM on the 1-in. white pine board. Calculate the means and standard deviations and report these values as a function of temperature.

7.1.7.2 *Stabilization With Time*—Using the same experimental system and test conditions described in 7.1.2, turn on the instrument and perform 1-min measurements with a 0.7x to 1.3x CRM on the 1-in. white pine board consecutively until the values are within the limits of precision for this condition as determined in 7.1.2 for ten measurements in a row. Report the time to achieve this state of stability as the stabilization time.

7.1.7.3 *Stability With Time; One Day Test*—After the instrument has electronically and thermally stabilized, as determined in 7.1.7.2, take triplicate measurements of the 0.7 to 1.3x CRM on the 1-in. white pine board. Repeat these triplicate measurements after each ½ h; at the end of 4 h turn off the instrument for 1 h. Then, turn on the instrument, allow it to stabilize, and repeat the 4-h test. Calculate the means and standard deviations, and report these values as a function of time.

NOTE 20—If the instrument battery is not capable of operating for 8 h, replace the battery used for the first 4-h test with a freshly charged battery before the second 4-h test.

7.1.7.4 *Stability with Time; Five-Day Test*—Repeat 7.1.7.3 for a total of five consecutive days.

7.1.8 *Radiation Safety*—The radiation safety shall be evaluated as described in Section 7 of ANSI N538–1979. The procedures are summarized in 7.1.8.1 – 7.1.8.4.

7.1.8.1 *Test Conditions*—The instrument shall be tested for proper operation of its radiation safety features under high 60°C (140°F) and low 0°C (32°F) temperatures.

7.1.8.2 *Test Equipment*—The instrument for measurement of beta or electromagnetic radiation (gamma, X-ray, bremsstrahlung) shall be an air ionization chamber survey meter with an energy response flat within ±15 % over the energy range of 15 KeV to 1.3 MeV. The areal mass density of the meter window shall be no greater than 7 mg/cm^2. The test instrument shall be calibrated according to ANSI N323–1978.

NOTE 21—Alternate test instrumentation may be substituted provided its efficiency and sensitivity for the energies involved are equivalent. If the instrument's size precludes measurement at the 5-cm distance to the "center" of the active volume of the test meter, alternative dosimetry such as pen dosimeters or thermoluminescent devices may be used. Alternative test equipment or procedures that are used must reflect, however, methodologies widely accepted by the health physics community.

7.1.8.3 *Test Procedure*—Measurements shall be performed along two-dimensional, isodistance contours around the probe at 5 cm, 30 cm, and 100 cm from the nearest accessible surfaces. Distance shall be measured from the surface of the probe to the center of the active volume of the test meter. With the source in the OFF condition, measurements are made over all surfaces of the probe, including the location of the operator's hand while the operator is using the instrument and the region of the useful beam. The probe then is placed against a 0.7x to 1.3x CRM standard over a 0.25-in. steel plate (see 7.1.2) and the source is set to the ON condition. Isocontour measurements of stray radiation are made (excluding the region of the useful beam). The dose rate measurement shall be made under the condition "dose rate (300 mg/cm^2)." This condition applies to the dose rate to the "whole body, gonads, active blood-forming organs, head and trunk, or lens of the eye," and assumes that the lenses of the eyes are not protected by any type of eye shields. For this measurement, an external absorber shall be placed over the window of the survey meter as necessary; the total areal mass density of window and absorber shall be no greater than 300 mg/cm^2. For the external absorber, a material, such as polyethylene with a low effective atomic number (specific gravity approximately 1.0) shall be used.

7.1.8.4 The maximum stray radiation measured along the isocontours shall be as follows:

Stray radiation at 5 cm	1000 Sv/h
Stray radiation at 30 cm	50 Sv/h
Stray radiation at 100 cm	20 Sv/h

APPENDIXES

(Nonmandatory Information)

X1. JUSTIFICATION FOR THE FORMULA USED TO ESTIMATE LOD

X1.1 The assumption of variance being linear with concentration is approximate. Since fluctuations other than counting statistics contribute to variance, higher order terms are present. A better approximation would be $\sigma_x^2 = A + Bx + Cx^2$, but the quadratic term can be neglected at low concentrations, so $\sigma_x^2 \simeq A + Bx$.

X1.1.1 The Gaussian approximation becomes nearly exact as the total number of X-rays detected goes above 50 or so. The one-sided tail of a Gaussian for an error probability of 5 % begins at 1.645σ.

X1.1.2 Mathematically,

$$\sigma_x = \sqrt{A + Bx} \tag{X1.1}$$

$$x_t = 1.645\,\sigma_{zero} = 1.645\sqrt{A + B \cdot 0} = 1.645\sqrt{A} \tag{X1.2}$$

$$x_{LOD} = x_t + 1.645\,\sigma_{LOD} \tag{X1.3}$$

$$= 1.645\sqrt{A} + 1.645\sqrt{A + Bx_{LOD}} \tag{X1.4}$$

X1.1.3 Solving for x_{LOD}, we obtain:

$$x_{LOD} = 2(1.645)\sqrt{A} + (1.645)^2 B \tag{X1.5}$$

$$x_{LOD} = 3.29\sqrt{A} + 2.706B \tag{X1.6}$$

X1.1.4 Note that if precision is perfectly flat, that is, it does not vary with concentration, than $\sigma_1 = \sigma_0$, so $A = \sigma_0^2$ and $B = 0$, and therefore $x_{LOD} = 3.29\ \sigma_0$, which is remarkably close to the commonly used $x_{LOD} = 3\sigma_0$.

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