Designation: E 1828 – 01

An American National Standard

Standard Practice for Evaluating the Performance Characteristics of Qualitative Chemical Spot Test Kits for Lead in Paint[1]

This standard is issued under the fixed designation E 1828; 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 describes an evaluation procedure for the determination of performance characteristics of qualitative chemical spot test kits for lead, as applied to dry paint films, for a given dry paint film matrix on a given substrate.

1.2 This practice may be used to determine the performance characteristics of a given lead spot test kit for a given synthetic or field dry paint film matrix, independent of substrate effects.

1.3 To allow for comparisons of different spot test kits on an identical matrix, this practice covers the determination of test kit performance characteristics on a synthetically prepared standard white lead-containing paint film. The ingredients for the preparation of a standard synthetic leaded paint film based on basic lead carbonate are described.

1.4 This practice is not intended for the evaluation of performance parameters of chemical spot test kits on all types of paint or paint conditions that may be encountered during field use (as described in E 1753). Rather, this practice addresses the evaluation of the response of a chemical spot test kit for lead as a function of lead concentration for a limited set of paint films. Evaluating the performance of a chemical spot test kit for a given application requires tests on a representative sample of paints and substrates found at the location(s) to be tested.

1.5 *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 81 Specification for Basic Carbonate White Lead Pigment[2]

D 823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels[3]

D 1005 Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometres[3]

E 1605 Terminology Relating to Abatement of Hazards from Lead-Based Paint in Buildings and Related Structures[4]

E 1613 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[4]

E 1645 Practice for Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis[4]

E 1729 Practice for Field Collection of Dried Paint Samples for Lead Determination by Atomic Spectrometry Techniques[4]

E 1753 Practice for Use of Qualitative Chemical Spot Test Kits for Detection of Lead in Dry Paint Films[5]

2.2 *Federal Specifications:*

Federal Specification TTP 19 Paint, Latex (Acrylic Emulsion, Exterior Wood, and Masonry)[6]

Federal Specification TTP 102 Paint, Oil (Alkyd Modified, Exterior, White, and Tints)[6]

3. Terminology

3.1 For definitions of terms not listed here, see Terminology E 1605.

3.2 *identification limit, n*—for a qualitative chemical spot test kit, this is the lead content that yields a 50 % chance of either a positive or negative test result for a given sample matrix **(1)**.[7]

3.3 *performance curve, n*—for a qualitative chemical spot test kit, this is a plot of the test kit response (positive or negative) versus the lead content in a given sample matrix as determined by quantitative analysis **(2)**.

3.3.1 *Discussion*—The performance curve may be statistically modeled to yield qualitative test kit performance parameters for lead detection.

[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 Paint Abatement.

Current edition approved Dec. 10, 2001. Published March 2002. Originally published as E 1828–96. Last previous edition E 1828–96.

[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 04.11.

[5] *Annual Book of ASTM Standards*, Vol 04.07.

[6] Available from U.S. Government Printing Office, Washington, DC.

[7] The boldface numbers in parentheses refer to the list of references at the end of this practice.

3.4 *performance parameter*—for a particular spot test kit and a particular sample matrix, this is the lead content that yields a known degree of confidence in detecting lead.

3.4.1 *Discussion*—Examples of qualitative test kit performance parameters include the identification limit and the amounts of lead in a given sample matrix that yield a desired confidence (for example, 95 %) of a negative and positive test result, respectively.

4. Summary of Practice

4.1 Samples of paint are collected using an ASTM practice. Alternatively, samples may consist of referenced materials (or mixtures thereof) or may be prepared artificially in the laboratory **(3)**. Synthetic standard paint films may be prepared using basic white lead carbonate over an applicable range of lead concentrations.

NOTE 1—Other types of paint films may be prepared from other materials to yield data that are supplemental to this practice. For example, chromate paints of varied lead concentration could be fabricated to investigate performance curves of chemical spot test kits.

4.2 Paint samples are tested with a qualitative chemical spot test kit for lead according to Practice E 1753, and test kit results are recorded as positive or negative for lead detection.

4.3 Tested paint samples are prepared and analyzed for quantitative lead content using ASTM standards.

4.4 The quantitative lead content data from chemical analysis are compared to the qualitative lead spot test kit results.

4.5 The comparative data are statistically modeled in order to determine the performance parameters of a particular spot test kit for a particular paint matrix.

5. Significance and Use

5.1 This practice is to be used to evaluate the performance of commercially available or laboratory prepared qualitative chemical spot test kits for lead in dry paint films.

5.2 This practice is generic in the respect that the protocol is applicable to any desired combination of spot test kit and paint sample matrix.

5.3 Performance parameters of interest in the evaluation include the identification limit of a particular test kit for a particular paint matrix, and the lead content in paint that gives a known degree of confidence for a negative or positive test result.

6. Apparatus

6.1 *Qualitative Chemical Spot Test Kit*, for lead detection, either commercially available or prepared in the laboratory.

NOTE 2—Most spot test kits for lead are based on the use of rhodizonate or sulfide **(1,4)**. Spot test kits for lead based on each of these chemistries are commercially available. Laboratory procedures describing the preparation of lead spot test kits are outside the scope of this standard. However, these protocols are given in Refs **(1)** and **(4)**.

6.2 *Sealable Hard-Walled Sample Containers*, plastic or glass, for containment of dry paint samples following spot testing but prior to laboratory analysis.

6.3 *Laboratory and Field Supplies and Equipment*, as needed for sample collection, sample preparation, and analysis. Necessary laboratory and field supplies for particular sample matrices of concern are described in the pertinent ASTM standards listed in 2.1.

6.3.1 *Sample Collection*—Supplies and equipment for sample collection are described in Practice E 1729.

6.3.2 *Sample Preparation*—Supplies and equipment for sample preparation are described in Practice E 1645.

6.3.3 *Analysis*—Supplies and equipment for laboratory analysis of extracted lead samples are described in Test Method E 1613.

7. Procedure

7.1 *Sample Collection or Artificial Sample Preparation*—Field dry paint samples may be collected using Practice E 1729. Alternatively, samples may consist of Certified Reference Materials (CRMs) or mixtures thereof, or may be prepared artificially in the laboratory.

7.1.1 *Field Samples—Dry Paint Film Sample Collection*—Collect paint samples in accordance with Practice E 1729.

7.1.2 *Concentration Range of Field Samples*—It is necessary to collect or prepare samples of a particular matrix over a wide range of lead concentrations (at least two, preferably three or more orders of magnitude).

7.1.2.1 The lead content in the sample matrix of interest must extend from a concentration of at least one order of magnitude below the anticipated identification limit of the spot test kit to a lead content at least five times above the expected identification limit.

NOTE 3—In general, until samples are analyzed quantitatively (especially field samples), it will not be possible to know exactly the range of sample lead concentrations.

7.2 *Synthetic Samples*:

7.2.1 *Preparation of Standard Synthetic Dry Paint Films*:

7.2.1.1 *Leaded Paint Components*—The paste-in-oil form of basic white lead carbonate, meeting the requirements described in Specification D 81, shall be used in the preparation of standard leaded paint films for spot testing. This paste is to be dispersed and diluted in white alkyd paint that meets the requirements of Fed. Spec. TTP 102.

NOTE 4—Paint drying procedures are covered in Fed. Spec. TTP 102. General requirements are for the paint film to be dried for two weeks at room temperature, with the film being dry to the touch.

7.2.1.2 *Substrate*—Prepared synthetic paints shall be spread uniformly on an unleaded glass, poly(methylmethacrylate) (PMMA or lucite), or polyester (mylar) surface. The film may consist of one or more coats of lead-containing paint.

NOTE 5—For assistance in preparing films of uniform thickness, see Practices D 823.

7.2.1.3 *Overlayer*—A nonleaded overlayer consisting of two coats of latex paint, meeting the requirements of Fed. Spec. TTP 19, shall be spread atop the leaded paint layer(s).

7.2.1.4 *Thickness*—The thickness of each paint layer within the films shall be 35 to 80 µm.

NOTE 6—For assistance in measuring film thickness, see Test Method D 1005.

7.2.2 *Concentration Ranges of Synthetic Paint Samples*:

7.2.2.1 The range of lead concentrations in synthetically prepared leaded paint films shall extend from ≤0.001 % Pb to approximately 5 % Pb by mass, or ≤0.01 mg/cm^2 to approximately 2 mg/cm^2 by surface area.

7.3 *Number of Samples*—The number of samples required for the evaluation may differ for different matrices. However, as a general rule, hundreds of samples spanning the lead concentration range of interest must be obtained for each combination of spot test kit and sample matrix that is to be tested.

NOTE 7—In general, the greater the number of samples over a wide lead concentration range, the lesser the overlap between negative and positive test kit readings (in the modeled performance curve). To reduce the standard deviation about a given performance parameter by one-half, the sample size must be increased by a factor of four (see Appendix X1).

7.4 *Application of Spot Test Kit*:

7.4.1 *Commercial Kits*—Following instructions given in Practice E 1753, apply the spot test kit to each sample individually. Use only one spot test per sample.

7.4.1.1 If a positive control is provided by the manufacturer, apply the spot test kit to the positive control according to the manufacturer's instructions and record the result. If a negative result is found, the kit must be discarded. A negative result on a positive control indicates that the ingredients of the kit are no longer usable.

7.4.2 *Laboratory-Prepared Kits*—Following protocols outlined in Refs **(1)** and **(4)**, apply the spot test to each sample individually. Use only one spot test per sample.

7.4.3 Record results as either positive for lead detection or negative for no lead detection.

7.5 *Sample Containment after Spot Testing*—After testing with the chemical spot test kit, place the sample and all test kit components that contacted the sample during conduction of the spot test into a sealable hard-walled sample container and close the container.

7.6 *Sample Preparation for Subsequent Lead Determination*—Samples are to be prepared for subsequent lead determination in accordance with Practice E 1645.

7.7 *Determination of Lead in Extracted Samples*—Determine the lead content in each paint sample in accordance with Test Method E 1613.

7.8 *Modeling of the Spot Test Kit Performance Curve*—A general model to describe the performance of a generic qualitative spot test kit for lead can be developed from a binomial experiment (see Appendix X2). With knowledge of the lead content (7.7) and the test kit response (7.4) for each sample, a statistical model can be applied to the experimental data. In this fashion, the performance parameters of the spot test kit may be estimated from the modeled data. **(5)**

7.8.1 The experimental data (test kit response versus lead content) are modeled statistically using a sigmoidal curve (see Appendix X3). The applicable boundary conditions are 1) a probability of lead detection of zero for a negative response at zero lead content and 2) a probability of lead detection of unity at 100 % lead content.

7.8.1.1 *Discussion*—A preliminary screening model is used first to obtain knowledge concerning the expected test kit performance criteria **(6)**. Thereafter, the remainder of the experiment is designed so a targeted number of additional samples may be tested experimentally, and the data fitted using a statistical model that generates the performance parameters of interest.

7.8.1.2 For initial screening modeling based on few data points, use the model of X3.1. Use results from this model and the aid of a statistician, if necessary, to gain knowledge on 1) the final number of samples needed and 2) the needed range of lead contents, for more detailed statistical fitting of the experimental data.

7.8.1.3 Obtain additional experimental data as needed (7.4-7.6) and model the performance curve using an appropriate statistical model. Examples of suitable statistical models for data following a sigmoidal curve are given in X3.2 **(5)** and in Refs. **(10)** and **(11)**.

NOTE 8—Commercial software is available that can be used in statistical computations of the kind outlined here.

7.8.1.4 Performance parameters of interest include the identification limit of the test kit, and lead contents at which one may be 95 % confident (or other desired probability, for example, 90 %) of either a negative or a positive test kit reading (see Appendix X3) **(5)**.

8. Report

8.1 Report the following information concerning the sample matrix and analytical procedures used:

8.1.1 Type of sample matrix, for example, dry paint,

8.1.2 Form of sample matrix, for example, intact, ground, cut,

8.1.3 Color of dry paint sample,

8.1.4 Sample collection and preparation procedures used prior to testing with the spot test kit,

8.1.5 Type of spot test kit used (rhodizonate or sulfide) with information concerning manufacturer and model no. (if applicable); if a laboratory prepared kit is used, provide all details concerning its composition and use,

8.1.6 Sample preparation procedure(s) used following testing with the test kit and prior to laboratory analysis,

8.1.7 Laboratory analysis procedure(s) used,

8.1.8 Date sample collected,

8.1.9 Date sample tested with spot test kit,

8.1.10 Date sample prepared for laboratory analysis,

8.1.11 Date sample analyzed,

8.1.12 Personal identifiers,

8.1.13 Identifiers for all equipment used in sample preparation and analysis,

8.1.14 Test kit lot number and expiration date, and

8.1.15 Substrate type.

8.2 Report the following information concerning statistical modeling of the test kit performance curve:

8.2.1 Total number of data points,

8.2.2 Statistical model(s) used to fit the experimental data,

8.2.3 Document computer program(s) used in statistical modeling with information on commercial software used in statistical modeling (if applicable),

8.2.4 Input parameters used in statistical modeling, 8.2.5 Output parameters of interest (identification limit) and lead contents at which one may be 95 % (or other selected probability, for example, 90 %) certain of 1) negative and 2) positive test kit readings, and

8.2.6 Uncertainties in input and output parameters (standard deviations) or 95 % (or other, for example, 90 %) confidence intervals, or both.

9. Keywords

9.1 lead; paint; performance characteristics; performance criteria; spot test; statistical modeling

APPENDIXES

(Nonmandatory Information)

X1. SAMPLE SIZE FOR EVALUATING CHEMICAL SPOT TEST KITS

X1.1 Let θ be any one of the primary parameters or the performance parameters in the performance curve model and $\sigma_{\hat{\theta}}(k)$ be the standard deviation of the estimator $\hat{\theta}$ with samples of size *k*. Then, for large *n* and *m*:

$$\sigma_{\hat{\theta}}(n) \approx \sqrt{\frac{m}{n}}\,\sigma_{\hat{\theta}}(m) \quad \text{(X1.1)}$$

where:
m ≡ the experimental sample size and
n ≡ an arbitrary sample size.

X1.2 If $n = 4m$, $\sigma_{\hat{\theta}}(4m) \approx \frac{1}{2}\,\sigma_{\hat{\theta}}(m)$. To cut the standard deviation in half, increase the sample size by four.

X1.3 Consider, for example, $m = 370$ **(5)**:

$$\sigma_{\hat{a}}(n) \approx \sqrt{\frac{370}{n}}\,\sigma_{\hat{a}}(370) \approx 0.39\sqrt{\frac{370}{n}} \quad \text{(X1.2)}$$

$$\sigma_{\hat{b}}(n) \approx \sqrt{\frac{370}{n}}\,\sigma_{\hat{b}}(370) \approx 0.17\sqrt{\frac{370}{n}} \quad \text{(X1.3)}$$

and

$$\sigma_{\hat{x}_{0.05}}(n) \approx \sqrt{\frac{370}{n}}\,\sigma_{\hat{x}_{0.05}}(370) \approx 0.15\sqrt{\frac{370}{n}} \quad \text{(X1.4)}$$

$$\sigma_{\hat{x}_{0.50}}(n) \approx \sqrt{\frac{370}{n}}\,\sigma_{\hat{x}_{0.50}}(370) \approx 0.32\sqrt{\frac{370}{n}} \quad \text{(X1.5)}$$

$$\sigma_{\hat{x}_{0.95}}(n) \approx \sqrt{\frac{370}{n}}\,\sigma_{\hat{x}_{0.95}}(370) \approx 1.29\sqrt{\frac{370}{n}} \quad \text{(X1.6)}$$

X2. BINOMIAL DISTRIBUTION FUNCTION

X2.1 The probability of having a given number of one outcome from a given number of trials is given by the binomial distribution function:

$$P(X = x) \equiv b(x; n, p) = \{n!/[x!(n - x)!]\}\,p^x\,(1 - p)^{n-x} \quad \text{(X2.1)}$$

where:
P ≡ a probability,
X ≡ a binomial random variable,
x ≡ the number of successes,
b ≡ the binomial probability function,
n ≡ the number of trials, and
p ≡ the probability of success.

X2.2 The probability of having less than a given number of successes from a given number of trials is obtained from the cumulative binomial distribution function:

$$P(X \leq x) = \sum_{0}^{x} b(x; n, p) \quad \text{(X2.2)}$$

X2.3 The modeling of a qualitative spot test kit is a binomial experiment since each test is identical and independent, and the results are either positive or negative.

X2.4 The probability of detection is the value of *p* in the above Eq X2.1 and Eq X2.2.

X3. MODELING QUALITATIVE SPOT TEST KIT DATA

X3.1 *Model Selection:*

X3.1.1 *Performance Function, p(x; θ)*:

X3.1.1.1 The probability that the test kit obtains a positive response, $p(x; \theta)$ is when the lead concentration in the sample is *x*. Here, $\theta = (\theta_1, \ldots, \theta_k)$ is a *k*-dimensional parameter.

X3.1.1.2 Formula $p(x; \theta)$ should have the following properties:

(1) It is a continuous, increasing, and differentiable function and

(2) $p(0; \theta) \equiv 0$ and $p(x; \theta)$ approaches 1 as *x* approaches infinity.

X3.1.2 *Performance Parameters*:

X3.1.2.1 The γ100 % positive response point x_γ, $p(x_\gamma; \theta) \equiv \gamma$.

X3.1.2.2 Points with $\gamma = 0.05$, 0.50, and 0.95 are of interest.

(1) Identification limit, $x_{0.50}$ and

(2) Uncertainty interval, $(x_{0.05}, x_{0.95})$.

NOTE X3.1—$x_{0.05}$ is the lower limit of lead concentration with a 95 % confidence when a positive response is observed, and $x_{0.95}$ is the upper limit of lead concentration with a 95 % confidence when a negative response is observed.

X3.2 *Examples of Performance Functions:*

X3.2.1 *Sigmoidal Curves*:

X3.2.1.1 A sigmoidal curve of the form:

$$dP/dC \equiv RP(1 - P/K) \tag{X3.1}$$

where:

$P \equiv$ the probability,
$C \equiv$ the lead content or concentration,
$R \equiv$ a constant related to the data being modeled, and
$K \equiv$ a constant equal to the maximum value that P can attain (1 in this case).

X3.2.1.2 With $K = 1$, Eq X3.1 can be solved and rearranged to give the following:

$$P = B/(e^{-RC} + B) \tag{X3.2}$$

where:

$B \equiv$ the constant of integration.

X3.2.1.3 A value for B can be found by defining a lead content, C^*, as the value of the lead content where $P = 0.5$.

X3.2.1.4 Substituting the derived value for B into X3.2.1.3 yields:

$$P = e^{-RC^*}/(e^{-RC} + e^{-RC^*}) \tag{X3.3}$$

that is similar to a logistic model.

X3.2.2 *Weibull Distribution Function*:

$$p(x; a, b) = 1 - \exp(-(x/a)^b) \tag{X3.4}$$

X3.2.2.1 Let $x = f(p; a, b) = a[-\ln(1-p)]^{1/b}$ be the inverse function of $p(x; a, b)$.

X3.2.2.2 The $\gamma100$ % positive response point $x_\gamma = f(\gamma; a, b)$.

X3.2.3 *Data Collection*:

X3.2.3.1 Let (x_i, y_i), $i = 1, \ldots, n$, be the data to be obtained and $y_i = 0$ or 1.

X3.2.3.2 Then,

(1) Sample size, n,

(2) Concentration range, $\min\{x_i\} \leq 0.1x_{0.5}$, $\max\{x_i\} \geq 5x_{0.5}$, and

(3) Uniformity, x_is are uniformly distributed within the concentration range.

X3.2.4 *Parameter Estimation—Maximum Likelihood Method* **(7,8)**:

X3.2.4.1 *Point Estimation*:

(*1*) The maximum likelihood estimate of parameter θ can be obtained by maximizing the following log likelihood function:

$$L(\hat{\theta}; (x_i, y_i), i = 1, \ldots, n) = \max L(\theta; (x_i, y_i), i = 1, \ldots, n) \tag{X3.5}$$

where:

$L(\theta; (x_i, y_i), i = 1, \ldots, n) = \Sigma_i \ln(1 - |y_i - p(x_i; \theta)|)$.

(*2*) Estimates of performance parameters are given by:

$$\hat{x}_Y = f(\gamma; \hat{\theta}) \text{ for } \gamma = 0.05, 0.5, 0.95, \tag{X3.6}$$

where:

$f(p; \theta) \equiv$ the inverse function of $p(x; \theta)$.

X3.2.4.2 *Estimation with Confidence*:

(*1*) A $\gamma100$ % confidence region for θ is given by:

$$C_\gamma(\hat{\theta}) = \{\theta: (\theta - \hat{\theta})^t \hat{V}^{-1} (\theta - \hat{\theta}) \leq x_\kappa^2(\gamma)\} \tag{X3.7}$$

(*2*) For each parameter component θ_i, a 95 % confidence interval is given by:

$$(\hat{\theta}_i^{0.025}, \hat{\theta}_i^{0.975}) = (\hat{\theta}_i - 1.96\,\hat{\sigma}_{\hat{\theta}_i}, \hat{\theta}_i + 1.96\,\hat{\sigma}_{\hat{\theta}_i}) \tag{X3.8}$$

where:

$$\hat{V} = (\hat{\sigma}_{\hat{\theta}_i \hat{\theta}_j}) = \left(\frac{\partial^2 L}{\partial\theta_i \partial\theta_j}\right)^{-1}_{\hat{\theta}}$$

is the asymptotic variance and covariance matrix of $\hat{\theta}$, and $\chi_\kappa^2(\gamma)$ is the $\gamma100$ percentile of a χ^2-distribution with k degrees of freedom **(9)**.

(*3*) A two-sided 95 % confidence interval for $x_{0.5}$:

$$(\hat{x}_{0.5}^{0.025}, \hat{x}_{0.5}^{0.975}) = (\min\{f(0.5; \theta): \theta \in C_{0.95}(\hat{\theta})\}, \max\{f(0.5; \theta): \theta \in C_{0.95}(\hat{\theta})\})$$

(*4*) A one-sided 95 % confidence limit for $x_{0.05}$:

$$(\hat{x}_{0.05}^{0.05}, \infty) = (\min\{f(0.05; \theta): \theta \in C_{0.90}(\hat{\theta})\}, \infty)$$

(*5*) A one-sided 95 % confidence limit for $x_{0.95}$:

$$(0, \hat{x}_{0.95}^{0.95}) = (0, \max\{f(0.95; \theta): \theta \in C_{0.90}(\hat{\theta})\})$$

X3.2.4.3 The standard deviation of the estimate for x_γ may be estimated by:

$$\hat{\sigma}(\hat{x}_\gamma) = \sqrt{\sum_{i=1}^{\kappa} \sum_{j=1}^{\kappa} \hat{\sigma}_{\hat{\theta}_i \hat{\theta}_j} \frac{\partial f(\gamma; \hat{\theta})}{\partial\theta_i} \frac{\partial f(\gamma; \hat{\theta})}{\partial\theta_j}} \tag{X3.9}$$

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