Designation: D4752 – 10 (Reapproved 2015)

Standard Practice for Measuring MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub[1]

This standard is issued under the fixed designation D4752; 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 a solvent rub technique for assessing the MEK resistance of ethyl silicate (inorganic) zinc-rich primers. The MEK resistance of some two-component ethyl silicate zinc-rich primers has been shown to correlate well with the cure of the primer as determined by diffuse reflectance infrared spectroscopy.[2] The technique can be used in the laboratory, field, or in the fabricating shop. Practice D5402 is the preferred method for organic coatings.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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.* Specific hazard statements are given in Section 6. Consult supplier's Material Safety Data Sheet(s) for specific hazard information relating to the solvent used.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D740 Specification for Methyl Ethyl Ketone

D4138 Practices for Measurement of Dry Film Thickness of Protective Coating Systems by Destructive, Cross-Sectioning Means

D5402 Practice for Assessing the Solvent Resistance of Organic Coatings Using Solvent Rubs

D7091 Practice for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to Ferrous Metals and Nonmagnetic, Nonconductive Coatings Applied to Non-Ferrous Metals

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *double rub, n*—the act of rubbing a solvent saturated cloth in one complete forward and backward motion over a coated surface.

4. Significance and Use

4.1 Ethyl silicate zinc-rich primers cure by the reaction of the vehicle with moisture, thereby providing a binder. As relative humidity and temperature vary during the day, so does the rate of cure. A certain minimum degree of cure is necessary prior to topcoating. It has been shown that the degree of cure of ethyl silicate zinc-rich primers can be measured by the chemical changes occurring using diffuse reflectance infrared spectroscopy.[2] This solvent rub test has been shown to correlate well with the infrared spectroscopic results of some two-component ethyl silicate inorganic zinc systems.

4.2 The cure rating required for the application of specific topcoats must be agreed upon before the practice is used.

5. Reagents and Materials

5.1 *Methyl Ethyl Ketone* (MEK), in accordance with Specification D740.

5.2 *100 % Cotton, Shop Cloth,* approximately 300 by 300 mm (12 by 12 in.) contrasting in color to the coating to be evaluated.

5.3 *Squeeze Bottle.*

5.4 *Proper Safety Equipment,* as determined from the solvent MSDS, for example, solvent resistant gloves, respirator.

6. Procedure

6.1 Select areas on the primer surface at least 150 mm (6 in.) long on which to run the tests. Clean the surface with tap water or dry cloth to remove loose material.

NOTE 1—Tap water may influence the cure of the zinc-rich primer.

[1] This practice is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

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[2] Starr, T. L., Henton, L. E., Lewis, W. S., and Rideout, F. A., "Improved Field Reliability of High Performance Coating Systems: Phase II—Develop Procedures and Criteria in Critical Performance Areas," available from SSPC: The Society for Protective Coatings, 40 24th St., Sixth Floor, Pittsburgh, PA 15213, www.sspc.org.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

6.2 Measure the dry film thickness of the primer in the selected areas in accordance with Test Methods D7091 or D4138. Mark a 150- by 25-mm (6- by 1-in.) rectangular test area on the undamaged cleaned surface using a pencil or other suitable solvent resistant marker.

6.3 Fold the cloth into a pad of double thickness and saturate it to a dripping wet condition with the methyl ethyl ketone. Do not allow more than 10 s to elapse before proceeding to the next steps.

6.4 Place the properly protected index finger into the center of the pad while holding excess cloth with the thumb and remaining fingers of the same hand. With the index finger at a 45° angle to the test surface, rub the rectangular test area with moderate pressure first away from the operator and then back towards the operator. Complete each double rub (one forward and back motion) in about 1 s.

6.5 Continue rubbing the surface with the MEK saturated pad, wetting the pad as necessary without lifting it from the surface, until either the metal substrate is exposed or 50 double rubs have been completed. If the former, record the number of rubs when the substrate is exposed.

6.6 Select an adjacent area to be used as a control. Repeat 6.1 – 6.5, except use a dry cloth to establish the effect of burnishing without the influence of MEK. *Use this area as the control to visually show the appearance of No Effect.*

6.7 Inspect the test areas and the cloths. Rate the results in accordance with Table 1.

TABLE 1 Scale for Resistance Rating

Resistance Rating	Description
5	No effect on surface; no zinc on cloth after 50 double rubs
4	Burnished appearance in rubbed area; slight amount of zinc on cloth after 50 double rubs
3	Some marring and apparent depression of the film after 50 double rubs
2	Heavy marring; obvious depression in the film after 50 double rubs
1	Heavy depression in the film but no actual penetration to the substrate after 50 double rubs
0	Penetration to the substrate in 50 double rubs or less

7. Report

7.1 Report, as a minimum, the following information:

7.1.1 Dry film thickness of the primer.

7.1.2 Elapsed time between the application of the primer and the running of the tests.

7.1.3 Number of tests conducted.

7.1.4 Resulting ratings.

7.1.5 In the case of a zero rating, number of double rubs required to expose the substrate.

7.1.6 *Field and Fabricating Shop Tests*—Identification of the area or piece tested.

8. Precision and Bias

8.1 *Precision*—It is not practicable to specify the precision of this procedure since the rating scale is arbitrary and standard methods for conducting round-robin studies and analyzing data based on an arbitrary rating scale are not available. See Appendix X1 "Goodness."

8.2 *Bias*—Since there is no acceptable reference material, bias cannot be determined.

9. Keywords

9.1 curing characteristics; double rub method; drying or curing; ethyl silicate (inorganic) primer; methyl ethyl ketone; MEK (methyl ethyl ketone) resistance; primer; solvent rub method; visual examination; zinc-rich primer

APPENDIX

(Nonmandatory Information)

X1. GOODNESS

X1.1 An intra-lab round-robin evaluation was conducted in order to access the "goodness" of this practice. Using the procedure outlines in this practice, eight different operators were asked to rate, in triplicate, the methyl ethyl ketone (MEK) rub resistance of a particular inorganic zinc-rich primer. The primer was separately applied to multiple test panels at two different dry film thickness ranges; 2.7-4.5 mils and 6.8-8.0 mils. Separate test panels corresponding to each coating thickness category were scored after a 6-h cure period and a 24-h cure period. Performance ratings and general statistical information have been in Table X1.1 for each cure period/film thickness combination.

X1.2 An analysis of the means, (ANOM), was conducted on the performance data of Table X1.1. Figs. X1.1-X1.4 illustrate the raw scores and the mean score reported by each operator. The 95 % confidence limits of the overall mean score (combined average score of the eight operators) are represented on each of the figures by dashed lines. The following observations were made through this analysis:

X1.2.1 Rub results are cure time dependent, that is, higher rub scores are associated with longer cure periods, lower rub scores are associated with shorter cure periods.

X1.2.2 Rub results are dependent on coating thickness, that is, higher rub scores are associated with thick coatings films, lower rub scores are associated with thinner coating films.

X1.2.3 From the plots, the largest number of operators whose mean score deviated from the 95 % confidence limits of the overall mean score was found to occur when this practice is applied to thin film, 6-h cured primers. The second largest

TABLE X1.1 Round Robin Resistance Rating

Case 1: Thin Film, 6-hr cure

Operator ID	Rub 1	Rub 2	Rub 3	Range	Mean	Standard Deviation
A	0	3	3	3	2.0	1.4
B	3	3	3	0	3.0	0.0
C	0	0	1	1	0.3	0.5
D	3	3	3	0	3.0	0.0
E	4	2	3	2	3.0	0.8
F	0	1	0	1	0.3	0.5
G	3	3	3	0	3.0	0.0
H	1	0	1	1	0.7	0.5
			Overall Average	1	1.9	0.5

Case 2: Thin Film, 24-h cure

Operator ID	Rub 1	Rub 2	Rub 3	Range	Mean	Standard Deviation
A	5	4	4	1	4.3	0.5
B	5	4	4	1	4.3	0.5
D	4	4	4	0	4.0	0.0
F	4	5	5	1	4.7	0.5
G	5	5	5	0	5.0	0.0
H	4	4	5	1	4.3	0.5
I	4	4	4	0	4.0	0.0
J	4	4	4	0	4.0	0.0
			Overall Average	0.5	4.3	0.2

Case 3: Thick Film, 6-h cure

Operator ID	Rub1	Rub2	Rub3	Range	Mean	Standard Deviation
A	4	4	4	0	4.0	0.0
B	4	4	4	0	4.0	0.0
C	4	4	4	0	4.0	0.0
D	3	3	3	0	3.0	0.0
E	4	3	3	1	3.3	0.5
F	0	4	5	5	3.0	2.2
G	3	3	3	0	3.0	0.0
H	3	4	3	1	3.3	0.5
			Overall Average	0.9	3.5	0.4

Case 4: Thick Film, 24-h cure

Operator ID	Rub1	Rub 2	Rub 3	Range	Mean	Standard Deviation
A	4	4	5	1	4.3	0.5
B	5	4	4	1	4.3	0.5
D	4	3	4	1	3.7	0.5
F	4	5	4	1	4.3	0.5
G	5	5	5	0	5.0	0.0
H	4	5	5	1	4.7	0.5
I	5	3	4	2	4.0	0.8
J	4	4	4	0	4.0	0.0
			Overall Average	0.9	4.3	0.4

number of operators whose mean score deviated from the 95 % limits of the overall score was found to occur in the case of the thin film 24-h cured primers.

X1.2.4 All operators had mean scores within the 95 % confidence limits of the overall mean score when this practice was applied to either thick, 6-h cured primers or thick, 24-h cured primers.

X1.3 The findings of this practice's round-robin evaluation have been offered in this appendix to serve as a reference or guide for operators wishing to utilize this practice.

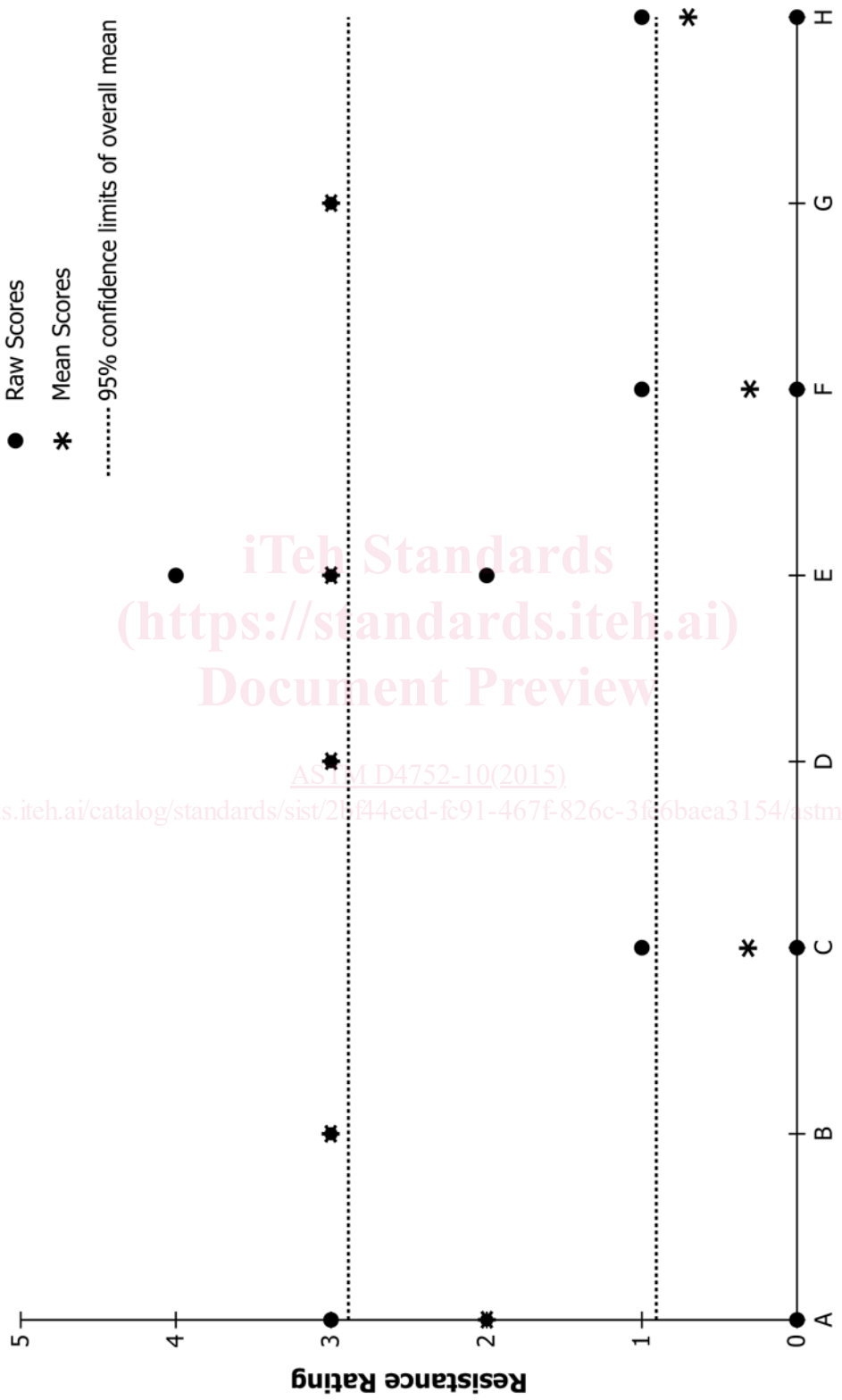

FIG. X1.1 Thin Film, 6-Hour Cure