



Designation: D 2674 – 72 (Reapproved 1998)

## Standard Methods of Analysis of Sulfochromate Etch Solution Used in Surface Preparation of Aluminum<sup>1</sup>

This standard is issued under the fixed designation D 2674; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 These methods offer a means for controlling the effectiveness of the etchant which is normally used for preparing the surface of aluminum alloys for subsequent adhesive bonding. As the etchant reacts with the aluminum, hexavalent chromium is converted to trivalent chromium; a measure of the two and the difference can be used to determine the amount of dichromate used.

1.2 The sulfochromate solution can be replenished by restoring the sodium dichromate and the sulfuric acid to the original formulation levels. The lower limit of usefulness will vary depending upon solution storage, adhesives used, critical nature of bond capability, variety of metals processed, etc. and should be determined. Replenishment will be limited to the number of times the chemical ingredients can be restored and maintained to the required levels and should be determined by the user. Sludge collecting in the bottom of a tank should be minimized by periodic removal of sludge. For some applications, the hexavalent chromium should not fall below the trivalent chromium content.

1.3 A typical aqueous sulfochromate acid etch solution formulation is as follows:

Sodium dichromate ( $\text{Na}_2\text{Cr}_2\text{O}_7$ )	4.5 oz/gal (33.7 g/litre)
Sulfuric acid (sp gr 1.84)	45 oz/gal (337.1 g/litre)

1.4 Maintenance of the aqueous concentrations is suggested as follows by appropriate additions of sodium dichromate or sulfuric acids.

Sodium dichromate	3.0 to 6.0 oz/gal (22.5 to 44.9 g/litre)
Sulfuric acid	40 to 50 oz/gal (299.6 to 374.5 g/litre)

1.5 Method A is intended for measuring the sulfuric acid content of a sulfochromate solution. Method B is intended for measuring the hexavalent and trivalent Chromium content of a sulfochromate solution. Method C is intended as an alternative method for measuring the hexavalent and trivalent Chromium content of a sulfochromate solution.

1.6 *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. Purity of Reagents and Water

2.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>2</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

2.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to ASTM Specification D 1193, for Reagent Water.<sup>3</sup>

### METHOD A

#### 3. Apparatus

3.1 *pH Meter.*

#### 4. Reagents

4.1 *Methyl Orange Indicator Solution*—Prepare in accordance with ASTM Practices E 50, for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals.<sup>4</sup>

4.2 *Sodium Hydroxide, Standard Solution (1.0 N)*—Dissolve 40 g of sodium hydroxide (NaOH) in 1 liter of water and standardize.

#### 5. Procedure

5.1 Pipet a 5-mL sample of sulfochromate etch solution into a 250-mL beaker. Add 100 mL of water. Titrate the sample with the NaOH solution to a pH of 3.5, using a pH meter.

5.2 An alternative method may be used whereby, instead of the pH meter, 1 drop of methyl orange indicator solution is

<sup>1</sup> These methods are under the jurisdiction of ASTM Committee D-14 on Adhesives and are the direct responsibility of Subcommittee D14.80 on Metal Bonding Adhesives.

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<sup>2</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N. Y., and the "United States Pharmacopeia."

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 03.05.