



## Standard Test Method for Solvent Tolerance of Amine Resins<sup>1</sup>

This standard is issued under the fixed designation D 1198; 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 approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of the quantity of hydrocarbon solvent that an amine resin will tolerate at 77°F (25°C).

1.2 *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 362 Specification for Industrial Grade Toluene<sup>2</sup>

D 891 Test Methods for Specific Gravity of Liquid Industrial Chemicals<sup>3</sup>

### 3. Summary of Test Method

3.1 A standard solvent prepared from three relatively pure hydrocarbons is employed to titrate a specimen of the resin to a defined degree of turbidity and the solvent tolerance is calculated from the volume of solvent added.

### 4. Significance and Use

4.1 This test method gives an indication of the solubility of an amine resin. This property is important in determining the utility of the resin in new paint formulations and in ensuring its compatibility with existing paint formulations. This test method is thus useful for evaluation and quality control of amine resins.

### 5. Apparatus

5.1 *Erlenmeyer Flask*, wide-mouth, 250-mL capacity.

5.2 *Buret*, 50-mL capacity, graduated in 0.1-mL divisions.

5.3 *Print Specimen*—A sheet of paper having on it printing in a black ink from 10-point, No. 31 old style type, including a double quotation mark (lower case letters approximately  $\frac{1}{16}$

in. (1.5 mm) high) with normal spacing, upper and lower case with no italicized or bold letters.

### 6. Reagents

6.1 *Decahydronaphthalene*, (decalin) having a boiling range between 188 and 195°C and a density, at 25°C, between 0.885 and 0.890 g/mL.

6.2 *Isooctane* (2,2,4-trimethylpentane), having a purity of at least 99 mol %.

6.3 *Toluene*, conforming to Specification D 362.

6.4 *Solvent Mixture*:

6.4.1 Blend the above three hydrocarbons in the proportions indicated below and determine the density at 25°C in accordance with Test Methods D 891.

	Parts by Weight
Isooctane	84
Decahydronaphthalene	8
Toluene	8

6.4.2 Standardize the solvent by titration against  $20 \pm 0.1$  g of mineral-spirits-standardized kauri-butanol solution. The volume of solvent necessary to reach the end point described in 7.3 should be between 31 and 32 mL. If the volume of solvent is not between 31 and 32 mL make an adjustment by varying the concentration of decahydronaphthalene. If the titrant volume is low add more decahydronaphthalene, if high add 84 + 8 isooctane-toluene mixture. Determine the density of the final mixture at 25°C.

NOTE 1—Although similar in certain respects to commercial products identified as mineral spirits, the solvent possesses an advantage for this determination in that it is a mixture of known constant composition.

### 7. Procedure

7.1 Adjust the temperature of the solvent and the sample to  $77 \pm 1^\circ\text{F}$  ( $25 \pm 0.5^\circ\text{C}$ ), and conduct the determination at this temperature.

7.2 Weigh the Erlenmeyer flask on a suitable balance to 10 mg. Transfer approximately 10 g of resin to the flask and again weigh to 10 mg to obtain the weight of the specimen.

7.3 Fill the buret with the solvent, and titrate the specimen while swirling the flask constantly. Add the solvent rapidly, but at a rate such that precipitation caused by local excess of solvent is kept to a minimum, until about 90 % of the required amount has been added. As the end point is approached, add the solvent in small increments. The end point is reached when

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<sup>2</sup> Discontinued; See 1989 Annual Book of ASTM Standards, Vol 06.03.

<sup>3</sup> Annual Book of ASTM Standards, Vol 15.05.

the 10-point print placed beneath the flask becomes illegible. Alternatively the end point may be taken as the point where a double quotation mark can no longer be resolved. Record the volume of solvent added.

## 8. Calculation

8.1 Calculate the solvent tolerance  $T$ , expressed as grams of solvent tolerated by 100 g of resin, as follows:

$$T = (VD/S) \times 100 \quad (1)$$

where:

$V$  = solvent used in the titration, mL,

$D$  = density of solvent, and

$S$  = specimen weight used, g.

NOTE 2—If preferred, instead of determining the density of the solvent the flask may be weighed again after completing the titration, thus obtaining directly the weight of solvent required.

## 9. Report

9.1 Report the grams of solvent tolerated by 100 g of sample to 1 g. Duplicate runs that agree within 2.7 % relative are acceptable for averaging (95 % confidence level).

## 10. Precision and Bias

10.1 The precision estimates are based on an interlaboratory study of this test method on one sample each of a modified

urea-formaldehyde resin and a butylated melamine-formaldehyde resin. Six laboratories analyzed each sample in duplicate and repeated the analysis on another day for a total of 48 determinations. The results do not include the component of variation due to difference in solvent since the same solvent was used by all participants. The within-laboratory coefficient of variation was found to be 0.65 % with 11 degrees of freedom and the between-laboratory coefficient of variation was found to be 1.67 % with 5 degrees of freedom. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence limit:

10.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 2.9 % relative.

10.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 6.1 % relative.

10.2 *Bias*—No bias can be determined since no standard amine resin is available.

## 11. Keywords

11.1 amine resin; decahydronaphthalene; hydrocarbon solvent; *isooctane*; solvent tolerance; toluene

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