

Method of Test for  
**VOLATILE CONTENT OF EPOXY RESIN SYSTEMS**

DOTD Designation: TR 701-85

**METHOD A**

(Volatile Content - Epoxy Resin Component)

DOTD TR 701-85

Adopted 10/85

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Method A

**Scope**

1. This method of test is intended to determine the volatile content of an epoxy resin for use in adjusting epoxide equivalent values.

**Apparatus**

2. (a) *Balance* - A Type I or II, Class A balance conforming to AASHTO M 231.

(b) *Oven* - Forced air oven capable of maintaining a uniform temperature of  $221 \pm 4$  °F.

(c) *Foil* - Aluminum foil sheets that measure 6 by 6 in. and 0.0015 to 0.0020 in. thickness.

(d) *Syringe* - Plastic disposable syringe, 10 ml capacity.

(e) *Glass Plates* - Two pieces approximately 3/16 in. thick. One piece should be 5 1/2 by 5 1/2 in. and the other piece should be 7 by 7 in.

(f) *Roller* - A steel or rubber roller with handle. The roller should be approximately 7 in. wide and 2 in. in diameter.

(g) *Mixing Tools* - Stainless steel spatulas or wooden tongue depressors.

(h) *Trays* - Trays measuring 6 1/2 by 6 1/2 in. constructed of No. 22 gauge aluminum sheet.

(i) *Desiccator* - A desiccator of sufficient size to hold the foil.

(j) *Thermometer* - A thermometer conforming generally to the requirements for ASTM 1C or 1F thermometers.

(k) *Timer* - A clock or watch capable of measuring minutes and seconds.

**Safety Precautions**

3. The following precautions should be observed when handling epoxy components and cleaning fluids:

(a) Persons handling these materials should use appropriate protective clothing, including rubber or plastic gloves, and appropriate eye protection such as safety glasses.

(b) If any epoxy or cleaning material should contact the skin, the material should be removed immediately with a dry cloth or paper towel, and the affected area should be washed thoroughly with soap and water.

(c) If any material should come in contact with the eyes, flush immediately with water and contact a physician.

(d) Adequate ventilation is necessary to prevent excessive inhalation of vapors.

(e) Observe all precautions as specified by the manufacturer before handling each material.

**Sample Preparation**

4. (a) Prior to mixing, condition the resin component and any equipment with which it will come in contact, to the test temperature of  $77 \pm 2$  °F by use of a water bath and/or laboratory temperature control.

(b) Thoroughly stir the sample of epoxy resin, for a minimum of one minute, immediately before testing.

**Procedure**

5. (a) Dry two foil sheets at  $221 \pm 4$  °F approximately 30 minutes or until a constant weight is obtained.

(b) Place the foil sheets in a desiccator and allow the sheets to cool sufficiently. Weigh the foil sheets to the nearest 0.0001 g and record weight as (DF).

(c) Place one of the dried foil sheets with the shiny side up on the 7 by 7 in. glass plate. Using the roller, roll the foil until the entire sheet is smooth and flat; repeat this step for the other sheet.

(d) Siphon approximately 5 ml of sample from the sample container into the disposable syringe.

(e) Weigh the syringe with resin to the nearest 0.0001 g and record weight as (SB).

(f) Inject 0.9000 to 1.1000 g of resin onto the center of the foil. Weigh the syringe after injection to ensure you have an adequate amount of sample; if not, repeat steps (e) and (f) until the proper amount is obtained, and record the weight as (SA).

(g) Place the other half of the foil over the sample.

(h) Center the small glass plate on top of the foil sheets and apply sufficient pressure to cause the sample to spread uniformly into a thin film, approximately 3 in. in diameter.

*NOTE: If a sample of low viscosity material should extend beyond the foil edge, repeat steps (a) through (h) allowing a few minutes for a portion of the solvent to evaporate from the weighed sample before covering and pressing it.*

(i) Remove the top glass plate and carefully separate the two foil sheets and place the foil in the tray.

(j) Place tray in oven for 2 hours at  $221 \pm 4$  °F.

(k) After the drying period, remove the tray from the oven and then carefully remove the foil sheets from the tray.

(l) Place the two foil sheets with the dried film surface in a face to face position and fold the edges together completely to prevent spillage.

(m) Place the foil sheets in the desiccator and allow the sheets to cool.

(n) Remove from the desiccator and weigh the foil sheets immediately to the nearest 0.0001 g and record weight as (DA).

#### Calculations

6. (a) Calculate the weight of the original sample according to the following formula:

$$SW = SB - SA$$

where:

SW = weight of original sample, g

SB = weight of syringe and sample before injection, g

SA = weight of syringe and sample after injection, g

(b) Calculate the weight of dry solids according to the following formula:

$$DS = DA - DF$$

where:

DS = weight of dry solids, g

DA = weight of oven dry sample and foil, g

DF = weight of foil, g

(c) Calculate the nonvolatile content according to the following formula:

$$NV = \frac{DS}{SW} \times 100$$

where:

NV = nonvolatile content, %

DS = weight of dry solids, g

SW = weight of original sample, g

(d) Calculate the volatile content according to the following formula:

$$VC = 100 - NV$$

where:

VC = volatile content, %

NV = nonvolatile content, %

#### Report

7. Report volatile content to the nearest 1%.

Normal testing time is one day.

Method of Test for  
**VOLATILE CONTENT OF EPOXY RESIN SYSTEMS**  
DOTD Designation: TR 701-85  
**METHOD B**  
(Volatile Content - Cured Epoxy Resin Systems)

DOTD TR 701-8  
Adopted 10/85  
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Method B

### Scope

1. This method of test is intended to determine the percent of volatile content in a cured epoxy resin system.

### Apparatus

2. (a) *Balance* - A Type I or II, Class A balance conforming to AASHTO M 231.

(b) *Beaker* - A 100 ml graduated disposable plastic beaker.

(c) *Oven* - Forced air oven capable of maintaining a uniform temperature of  $221 \pm 4$  °F.

(d) *Foil* - Aluminum foil sheets that measure 6 by 6 in. and 0.0015 to 0.0020 in. thickness.

(e) *Syringe* - Plastic disposable syringe, 10 ml capacity.

(f) *Glass Plates* - Two pieces approximately 3/16 in. thick. One piece should be 5 1/2 by 5 1/2 in. and the other piece should be 7 by 7 in.

(g) *Roller* - A steel or rubber roller with handle. The roller should be approximately 7 in. wide and 2 in. in diameter.

(h) *Mixing Tools* - Stainless steel spatulas or wooden tongue depressors.

(i) *Trays* - Trays measuring 6 1/2 by 6 1/2 in. constructed of No. 22 gauge aluminum sheet.

(j) *Desiccator* - A desiccator of sufficient size to hold the foil.

(k) *Thermometer* - A thermometer conforming generally to the requirements for ASTM 1C or 1F thermometers.

(l) *Timer* - A clock or watch capable of measuring minutes and seconds.

### Safety Precautions

3. The following precautions should be observed when handling epoxy components and cleaning fluids:

(a) Persons handling these materials should use appropriate protective clothing, including rubber or plastic gloves, and appropriate eye protection such as safety glasses.

(b) If any epoxy or cleaning material should contact the skin, the material should be removed immediately with a dry cloth or paper towel, and the affected area should be washed thoroughly with soap and water.

(c) If any material should come in contact with

the eyes, flush immediately with water and contact a physician.

(d) Adequate ventilation is necessary to prevent excessive inhalation of vapors.

(e) Observe all precautions as specified by the manufacturer before handling each material.

### Sample Preparation

4. (a) Condition and stir the individual components in accordance with paragraphs 4(a) and (b) of Method A.

(b) Combine and mix sufficient quantities of components A and B, in accordance with the manufacturer's recommendations such that a minimum sample quantity of 50 ml is obtained. If the manufacturer does not recommend a mixing time, mix the sample for at least 3 minutes. Use separate mixing tools when obtaining and mixing the desired quantities of each component to avoid contamination.

### Procedure

5. (a) Dry two foil sheets at  $221 \pm 4$  °F approximately 30 minutes or until a constant weight is obtained.

(b) Place the foil sheets in a desiccator and allow the sheets to cool sufficiently. Weigh the foil sheets to the nearest 0.0001 g and record weight as (WF).

(c) Place one of the dried foil sheets with the shiny side up on the 7 by 7 in. glass plate. Using the roller, roll the foil until the entire sheet is smooth and flat, repeat this step for the other sheet.

(d) Syphon into the disposable syringe approximately 5 ml of the mixed adhesive from the beaker.

(e) Weigh the syringe, with adhesive, to the nearest 0.0001 g and record weight as (SB).

(f) Inject 0.9000 to 1.1000 g of adhesive onto the center of the foil. Weigh the syringe after injection to ensure you have an adequate amount of sample; if not, repeat steps (e) and (f) until the proper amount is obtained. Record this weight as (SA).

(g) Place the other half of the foil over the sample.

(h) Center the small glass plate on top of the foil sheets and apply sufficient pressure to cause the sample to spread uniformly into a thin film approximately 3 in. in diameter.

*NOTE: If a sample of low viscosity material should ex-*

*tend beyond the foil edge, repeat steps (a) through (h), allowing a few minutes for a portion of the solvent to evaporate from the weighed sample before covering and pressing it.*

(i) Remove the top glass plate and carefully separate the two foil sheets to their full length and place foil in tray.

(j) Place the tray with foil in a desiccator and allow to cure for 7 days at  $77 \pm 2$  °F.

(k) After the sample cure period, remove the foil tray with sample from the desiccator and weigh the foil sheets and sample to the nearest 0.0001 g and record this weight as (WC).

(l) Place aluminum tray with foil sheets in the oven at  $221 \pm 4$  °F for 2 hours.

(m) After the drying period, remove the tray from the oven and then carefully remove the foil sheets from the tray.

(n) Place the two foil sheets with the dried film surface in a face to face position and fold edges together completely to prevent spillage. Place in desiccator and allow to cool. Weigh immediately to the nearest 0.0001 g and record weight as (DA).

#### Calculations

6. (a) Calculate the weight of the original sample according to the following formula:

$$SW = SB - SA$$

where:

SW = weight of original sample, g

SB = weight of syringe and sample before injection, g

SA = weight of syringe and sample after injection, g

(b) Calculate the weight of the cured sample according to the following formula:

$$WS = WC - WF$$

where:

WS = weight of cured sample, g

WC = weight of cured sample and foil, g

WF = weight of foil, g

(c) Calculate the weight of oven dry cured sample according to the following formula:

$$DS = DA - WF$$

where:

DS = weight of cured sample after drying, g

DA = weight of cured sample and foil after drying, g

WF = weight of foil, g

(d) Calculate the nonvolatile content according to the following formula:

$$NC = \frac{DS}{WS} \times 100$$

where:

NC = nonvolatile content, %

DS = weight of cured sample after drying, g

WS = weight of cured sample, g

(e) Calculate the volatile content according to the following formula:

$$VC = 100 - NC$$

where:

VC = volatile content, %

NC = nonvolatile content, %

#### Report

7. Report the volatile content to the nearest 1%.

Normal testing time is eight days.