

**STANDARD TEST PROCEDURE: 894-D**

**TITLE: Alcohol Extraction of Geon Resins**

**PRODUCT: Geon® Dispersion Resin**

**TYPE: Extraction**

**ASTM REFERENCE: None**

<b>Revision No.:</b>	<b>Date:</b>	<b>Changes:</b>
D	24 March 1998	General procedure update
C	10 June 1989	
B		

**I. SCOPE**

This procedure is suitable for determining the quantity of alcohol extractable materials in resins; especially when one or more of the extractable materials is volatile, such as is the case with Geon Dispersion type resins. Materials which will be extracted include soaps, moisture, residual monomers, etc., and the analysis is, therefore, not specific for soap content as is often assumed unless corrections are made for the other extractables.

**II. PRINCIPLE**

The resin sample is extracted for a given period of time in a recycling extraction apparatus. The percent extractables are calculated from the weight loss of the original sample.

**III. INTERFERENCES**

The drip screen must be properly placed inside the extraction thimbles (Step #5 of Procedure) to eliminate splashing which would result in resin loss; and consequently, high results.

**IV. PRECISION**

The  $S_t$  has been determined to be 0.106 % based on 35 samples tested at Pedricktown plant.

**V. SAFETY PRECAUTIONS**

- A. Methyl Alcohol or Ethyl Alcohol are flammable liquids, consult your appropriate MSDS, be careful of the hot surfaces associated with this test.
- B. The heating plates of the extraction bank, the Soxhlet extraction apparatus, the extraction thimbles and the extracting media will all be at an elevated temperature and should be handled accordingly.
- C. Allow the Heating [plate and the extraction flask cool completely before removing from the extraction unit.
- D. The extraction thimbles will be at either 80°C or 95°C when they are removed from the drying oven and should be handled accordingly.

## **VI. APPARATUS**

- A. Soxhlet Extraction Apparatus, Catalog No. J-1565, size C, Scientific Glass Co., Bloomfield, New Jersey.
- B. Extraction Thimbles, Alundum AN889, Medium porosity Catalog No. 09-660, Fisher-Scientific Co.
- C. Extraction Bank, Underwriter's Type or Bailey-Walker.
- D. Brass or stainless steel screening, 100 mesh, W.S. Tyler, Inc., Mentor, Ohio.
- E. Blue M, Class A Bench Model Oven with mechanical convection horizontal airflow, Model #DC-256A-FHP-1 capable of maintaining 80°C  $\pm$  1°C or 95°C  $\pm$  1°C.
- F. Analytical balance, capable of weighing to 0.1 mg.
- G. Desiccator
- H. Six (6) 250 or 300 ml flat bottomed boiling flasks.
- I. Small glass beads – boiling aids.
- J. Muffle Furnace capable of heating to 1000°C.

## **VII. REAGENTS**

Methyl Alcohol or Ethyl Alcohol (as required for the particular analysis being performed) – Reagent Grade.

## **VIII. PROCEDURE**

- A. Prepare the required number of glass extraction thimbles by drying them in a muffle furnace at approximately 500°C overnight. Let the furnace cool to room temperature before removing thimbles.
- B. Cool the thimbles to room temperature in a desiccator so as to avoid moisture pick-up from the atmosphere.
- C. The thimbles to be used are tared on an analytical balance which is accurate to 0.1 mg. Into each is weighed 4.0  $\pm$  0.5 grams of sample to the nearest 0.1 mg.
- D. To the required number of boiling flasks is added 150 ml +/- 10ml. of the alcohol to be used in the analysis. A few small glass beads are added to each flask. The boiling flasks are then set on the heating plates of the extraction bank.
- E. The extraction thimbles containing the samples are placed in the required number of Soxhlet extractors. A cap, made from 100 mesh brass screening, is placed on top of each extraction thimble. This drip screen diffuses the drops of alcohol coming from the condenser.
- F. The Soxhlet extractors are then inserted into the necks of the boiling flasks. The condensers from the Soxhlet extraction apparatus are properly positioned and the condenser tips are inserted into the extractors. All supporting clamps are tightened, with care being taken that the flat bottoms of the boiling flasks remain flush with the heater surfaces.
- G. The heaters are turned on and adjusted as necessary to initiate a moderate refluxing rate. Once equilibrium is established, this will require approximately thirty (30) minutes, refluxing is continued for six (6) hours +/- 5 min. The heaters are then turned off.

- H. The apparatus, heater and the sample flask is allowed to cool and the thimbles are removed from the siphoning chamber. They are allowed to drain, then placed in a Blue-M Class-A oven at 80°C +/- 2°C (for methanol) for 3 hours. If ethanol is used as the extractor, the temperature of the oven is to be set at 95°C +/- 2°C (see Note 1).
- I. Remove thimbles from oven and place in a desiccator until they come to room temperature.
- J. The thimbles are reweighed on the analytical balance and recorded to the nearest 0.1mg.
- K. To clean thimbles, gently remove resin with a lab spatula and then place in a muffle furnace overnight at approximately 500°C. Do not use water on these thimbles.
- L. Let thimbles cool and place in a desiccator.

**IX. CALCULATIONS**

$$\% \text{ Extractables} = \frac{(D + [S - S\{C/100\}]) - B}{S - S(C/100)} \times 100$$

- Where: D = Weight of thimble  
B = Weight of thimble and resin after extraction  
S = Original sample weight and  
C = Percent (%) moisture or percent (%) volatiles

**X. SPECIAL INSTRUCTIONS**

If preferred, an oven temperature of 95°C +/- 2°C (instead of 80°C) may be used when methanol is the extractor. A shorter drying time may be used accordingly.

**XI. REPORT**

Not applicable

**XII. REFERENCES**

None