

INTERNATIONAL TRUCK AND ENGINE CORPORATION

CEMS (CORPORATE ENGINEERING MATERIAL SPECIFICATION)		NUMBER: CEMS DT-3
TITLE: Methods of Determining Glass And PTFE Fillers In Thermoplastic Urethane		CURRENT ISSUE DATE: October 2001
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1.0 SCOPE

These are wet and thermogravimetric methods (Method A and B respectively) for determining the respective amounts of fiber glass and PTFE fillers in thermoplastic urethane.

METHOD A: Wet Method

2.0 APPARATUS

- 2.1 Flasks, Pyrex, 125 ml
- 2.2 Crucibles, 30 ml, Nickel or Platinum
- 2.3 Centrifuge Tubes, 100 ml
- 2.4 Balance 0.1 mg Sensitivity
- 2.5 Hot Plate
- 2.6 Oven, Gravity or Recirculating; Capable of Maintaining Temperature of $650 \pm 5^{\circ}\text{C}$
- 2.7 Centrifuge, Capable of 700 rcf
- 2.8 Fume Hood
- 2.9 Desiccator

3.0 MATERIAL

- 3.1 1 - Methyl, 2 - Pyrrolidinone, Commercial Grade
- 3.2 Acetone, ACS
- 3.3 Filter Paper, Ashless, Whatman 541 or Equivalent

4.0 PROCEDURE – WET METHOD

- 4.1 Dry an empty 125 ml flask 10 minutes at $105 \pm 5^{\circ}\text{C}$. Cool in a desiccator.
- 4.2 Weigh the dried flask to 0.1 mg, record this tare weight as (W1). Weigh into the tared flask approximately 2 grams of the material to be tested. Record weight of flask and sample as (W2).
- 4.3 Add 50 ml of 1-methyl, 2-pyrrolidinone and heat on hot plate to 200°C ; agitate the flask frequently until the urethane is completely dissolved.

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- 4.4** Weigh an empty dried 100 ml centrifuge tube, record this tare weight as (W3). Quantitatively transfer the contents of the flask to the tared tube. Rinse the flask into the tube with two 10 ml portions of 1-methyl, 2 pyrrolidinone. Centrifuge for 10 minutes at 700 rcf. (CAUTION: For balance, centrifuge tubes must be run in pairs and each tube must contain approximately the same amount of liquid.)
- 4.5** Carefully decant off the liquid and wash the solids twice with 1-methyl, 2-pyrrolidinone; then twice with acetone using 25 ml of fresh solvent each time; centrifuge and decant, as above, after each washing.
- 4.6** After the final wash, dry the tube and residue in a hot air oven for 45 minutes at $105 \pm 5^{\circ}\text{C}$; cool in a desiccator, and weigh; record the weight of the dried tube and contents as (W4).
- 4.7** Using either acetone or 1:1 acetone-distilled water mixture, quantitatively transfer and filter the residue. Discard the filtrate.
- 4.8** Weigh a dried empty 30 ml Pt or Ni crucible; record this tare weight as (W5). Transfer the filter paper and residue to the tared crucible. Ignite the filter paper and place in an oven at $650 \pm 5^{\circ}\text{C}$ for 2 hours. (CAUTION: Decomposition products of PTFE should not be inhaled; therefore, the ignition should be done in a fume hood and the oven should be located in a fume hood.)
- 4.9** Cool in a desiccator and weigh the crucible and contents; record as (W6).

5.0 CALCULATIONS

Calculate percent fillers as shown below:

$$\% \text{Glass} = \frac{W_6 - W_5}{W_2 - W_1} \times 100$$

$$\% \text{PTFE} = \frac{(W_4 - W_3) - (W_6 - W_5)}{W_2 - W_1} \times 100$$

W_1 = Tare Weight of Flask

W_2 = Weight of Flask and Sample

W_3 = Tare Weight of Centrifuge Tube

W_4 = Weight of Centrifuge Tube and Residue

W_5 = Tare Weight of Crucible

W_6 = Weight of Crucible and Residue after ignition

6.0 REPORT

Report filler content as % glass and % PTFE.

METHOD B: Thermogravimetric Method

7.0 THERMOGRAVIMETRIC ANALYSIS METHOD

This is an instrumental method for determining the respective amounts of fiberglass and PTFE fillers in thermoplastic urethane.

8.0 APPARATUS

Thermogravimetric Analysis (TGA) system capable of continuously recording temperature and sample weight as sample is heated in a controlled atmosphere. The system will be capable of uniformly increasing the temperature at a rate of 15°C per minute between 20 and 900°C . The integral balance will have a minimum capacity of 25 mg and 0.5 mg sensitivity.

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