

DETERMINATION OF INERT FILLERS IN POLYMERS**Application**

This method determines the concentration of inert fillers in polymer resins. Method A is primarily applicable to PTFE fillers but may also be used for other fillers if the hydrolysis products are soluble in the solvent. Method B is primarily applicable to graphite fillers but may also include diamond, silicon carbide, glass, bronze, and molybdenum disulfide (MoS₂).

Method 1**Apparatus**

Erlenmeyer flask, 500 mL
Reflux condenser to fit flask
Balance, analytical
Weighing paper
Graduated cylinders, 5 mL and 1000 mL
Hot plate
Filter flask with rubber adapter
Buchner funnel, medium fritted glass disk, 50 mL capacity
Wash bottle
Vacuum source
Vacuum oven

Reagents

50 % aqueous sodium hydroxide
Aqueous pyridine in volumetric ratio of water to pyridine of 4 to 1

Procedure

1. Weigh on glassine weighing paper approximately 2.0 grams of sample to the nearest 50 mg.
2. Carefully transfer sample to a 500 mL Erlenmeyer flask.
3. Reweigh paper.
4. Add 250 mL aqueous pyridine to sample.

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2000 11 09	Revised	Editorial – no technical change A. Cockman
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5. Add 4 mL of 50 % sodium hydroxide solution measured with a 5 mL graduated cylinder.
6. Assemble the flask and condenser on the hot plate.
7. Heat the mixture to a gentle boil, and maintain boiling for 30 minutes for margin resin. Extend the time for powder ground from part. Time will depend on particular size.
8. Swirl the mixture occasionally and wash the sides of the flask with a little aqueous solvent as needed to keep the sample down in the liquid.
9. Turn the hot plate off and let the solution cool slowly.
10. Weigh a Buchner funnel, previously dried 2 hours at 150 °C and cooled in a desiccator or dry box, to the nearest 0.1 mg.
11. When the flask is cool enough to be handled comfortably, filter the suspension, taking care to transfer the filler completely and to wash the filter cake well with water-pyridine solution.
12. Be sure no residue is left in the stem of the funnel due to evaporation; wash with pyridine if necessary to remove any deposit.
13. Dry the funnel and contents for at least 3 hours in a vacuum oven at 150 °C, 2.9 - 3.2 kPa vacuum, 0.03 m³/h N₂ gas purge.
14. Cool the funnel in dry box or desiccator and then weigh to the nearest 0.1 mg.
15. Clean the funnel with soap and water. Residual graphite may be removed with "Chromerge" cleaning solution.

Calculations

A. Total Filler

Funnel tare weight

Weight dry funnel + filler

Paper tare weight

Weight paper + sample

Weight % Filler = $\frac{(f-t) \times 100}{(s-p)}$

Where: f = weight of funnel from Step 14, mg

t = weight of funnel from Step 10, mg

s = weight of initial sample from Step 1, mg

p = weight of paper from Step 3, mg