

DETERMINATION OF FILLER CONTENT IN POLYTETRAFLUOROETHYLENE**Scope**

This test method outlines the procedure for determining the weight percent of fillers in filled and reinforced polytetrafluoroethylene (PTFE).

Application

Dry nitrogen or helium gas is used as an inert sweep gas to remove pyrolysis products of the polymer while preventing oxidation of the filler material at high temperature. This method is applicable to TFE polymers containing filler materials such as carbon, graphite and glass fiber. Duplicate samples are to be tested and results averaged.

When available, a thermogravimetric analyzer (TGA) should be used instead of this test method owing to the improved speed and precision obtainable with a TGA. (Refer to Ford Central Laboratory Services for the recommended TGA procedure.)

Note: This test method is not applicable to volatile inorganic fillers (such as CaCO_3 owing to loss of CO_2 during heating) or fillers which contain water of hydration.

Apparatus Required**Split-Tube Furnace**

Lindberg Model No. 55035 or equivalent with temperature range to 1100 °C.

Combustion Boats

Platinum.

Combustion Tube

Vycor glass with spring mounted gas restriction caps.

Glass Wool**Dry "Zero" Nitrogen or Helium Gas**

With regulator and flowmeter (1 ppm max moisture. Drier required if moisture > 1 ppm).

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2000 08 31	Revised	Editorial – no technical change A. Cockman
1992 07 13		

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Analytical Balance

Must have a precision of ± 0.05 mg.

Drier

VWR Catalog 26668-007 or equivalent. Required if gas with a moisture content in excess of 1 ppm is used.

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 ± 2 °C and 50 ± 5 % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Procedure

Note: This procedure must be carried out with furnace in a fume hood or other facility to assure adequate exhausting of toxic pyrolysis products.

The first part of the procedure (heating in a protective atmosphere) determines the total filler content. If it is desired to verify the level of fillers which can be totally oxidized to Co/CO, repeat steps 2 - 8 using oxygen or air in place of the nitrogen or helium. The weight of the resulting residue subtracted from the initial value obtained in step 8 will yield the oxidizable filler.

1. Weigh approximately 2 g of filled PTFE in clean, dry, tared, platinum combustion boat. Record weight of sample and boat to an accuracy of 0.1 mg. Weigh duplicate sample in second boat.
2. Carefully place boats in combustion tube. Insert loose wad of glass wool in inlet end of tube to diffuse incoming gas. Secure caps in place with springs.
3. Adjust nitrogen or helium gas flow to approximately 0.20 L/minute.

Note: The furnace should be completely purged before the furnace is turned on.

4. Set furnace control to approximately 7 1/2. Temperature should stabilize at 760 - 790 °C after about 45 minutes. (For faster heat-up, set control at "High" for approximately 10 minutes and then reduce to 7 1/2.)

Note: These settings are applicable only to the Lindberg Furnace. Other settings would have to be employed on other furnaces.

5. Maintain temperature at 760 - 790 °C with uniform gas flow for 1 - 1 1/4 h at heat.