TA Instruments

Thermal Analysis & Rheology

Thermal Analysis Application Brief

Determination of Moisture in Polyolefins

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SUMMARY

Moisture content in polyolefins is important because it affects the material's end-use properties. Moisture evolution analysis (MEA) provides a rapid method for determining moisture in polyolefins, normally requiring one gram of material to measure ppm levels of moisture.

INTRODUCTION

Moisture content of polyolefin resins is an important parameter for polymer manufacturers and fabricators, since water can affect both physical properties and product appearance (1). For example, polyethylene exposed to moisture can cause a decrease in tensile strength (2). Since polyolefins are molded or extruded at temperatures above the boiling point of water, excessive amounts of moisture present in the raw resin can cause bubbles and appearance differences in finished products.

Polyolefin insulation materials used in high voltage electrical cables prematurely fail due to the presence of moisture. This moisture can lead to the development of "water trees" or low resistance leakage paths, producing failure of the cable under actual use conditions (3).

The TA Instruments 903 Moisture Evolution Analyzer can be used to determine the amount of moisture in polyolefins, in both the resin and compounded states. Water content down to 25 µg/g can be determined routinely. With special care (such as dry box environments(levels down to 10 µg/g are possible.

EXPERIMENTAL

The experimental conditions used depend on the type of polyolefin and its moisture content. In the example given, polyethylene containing less than 25 µg/g moisture was analyzed.

Sample Weight : 1.0 g of pellets

Temperature : 120°C

Analysis Time : 20 minutes

1. The sample is placed in a tared boat, weighed, inserted into the instrument and analyzed under the conditions noted above.

2. The resulting "count" is compared to a blank count previously obtained by running the test with no sample in the instrument, but with otherwise identical conditions (including opening and closing of the sample chamber).

3. Using a calibration factor obtained by running a standard of known moisture content (e.g., sodium tungstate dihydrate), the water level in the sample is calculated from the equations given below.

CALCULATIONS

$$\text{Water level (mg/g)} = \frac{K(C_{sample} - C_{blank})}{Wt_{sample}}$$

$$K = \frac{F \times Wt_{std} \times 1000}{(C_{std} - C_{blank})}$$

Where:

C_{sample}	= count obtained for sample
C_{blank}	= count obtained for blank
C_{std}	= count obtained for standard material
Wt_{sample}	= weight of sample in grams
Wt_{st}	= weight of standard material in milligrams
F	= fraction of water in the standard material
K	= calibration factor in micrograms per count (should be near unity)

RESULTS

Using the procedure described above, an average deviation of 2.5% (relative) was obtained on a series of analysis for a sample determined to contain 24 µg/g water.

The sample of polyethylene analyzed at 120^0C did not adhere to the boat or cause any difficulty during analysis. Other samples, such as hygroscopic carbon-filled resins, may be placed directly into a dried sample boat and the weight of sample obtained after the analysis. A consensus standard test method for the determination of moisture in plastics has been issued by ASTM (4).

Other polymeric materials which have been analyzed for moisture content using the MEA include polycarbonate, ABS, nylon, and tire rubber (1).

The recommended calibration material is sodium tungstate dihydrate (F = 0.1092) which is stable to temperatures above 1000^oC. Water itself (F = 1.000) can also be used with the microcapillary technique described in the 903 Operator's Manual.

While analysis of total water present is most commonly the parameter of interest, Bair and co-workers have established that the concentration of clustered water (water molecule clusters trapped within the polymer molecule) in polyethylene is related to a loss of dielectric properties for polyethylene used in submarine cable cores (5,6). To determine the amount of water present in the clustered form versus total water, the clustered water is measured using Differential Scanning Calorimetry (DSC) while the total water is analyzed using the MEA technique described here.

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