TA Instruments

Thermal Analysis & Rheology

Thermal Analysis Application Brief

Determination of Moisture in Polyester Resins

Number TA-111

SUMMARY

Moisture content in polyester resins is important because it affects processing of the material. Moisture evolution analysis (MEA) provides a quick and easy method for determining ppm levels in these materials.

INTRODUCTION

Moisture content is an important quality parameter for polyester resins used in extruder processes. At the elevated temperatures of the extruder, any moisture that is present can hydrolyze the polyester polymer to free alcohol and acid. 1% water is stoichiometrically sufficient to destroy every twentieth polyester bond and completely destroy the polymer.

The TA Instruments 903 Moisture Evolution Analyzer can be used to quickly and accurately determine moisture content in polyester resins. Water content as low as 10 µg/g can be routinely determined. With special care (e.g., dry box environment) levels or accuracies down to 1 µg/g are possible.

EXPERIMENTAL

The experimental conditions used depend upon the type of polyester and its moisture content. In the example given, incoming polybutyleraphthalate (PBT) containing less than 0.2% moisture was analyzed.

Sample weight:	1.0 g of pellets
Temperature:	160°C
Time:	25 minutes

1. The sample is weighed into a tared sample boat and rapidly placed in the instrument.
2. The sample is analyzed under the conditions shown above and the "count" recorded.
3. The "count" thus obtained is compared with a blank count previously obtained with no sample in the chamber, but otherwise identical conditions (including opening and closing of the sample chamber).
4. 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 using the equations given below.

CALCULATIONS

$$\text{Water Level (ppm)} = \frac{K(C_{sample} - C_{blank})}{W_{sample}}$$

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

Where:

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

RESULTS

Using the procedure described, an average deviation of 1.3% (rel.) was obtained on a series of samples containing 0.13% (abs.) water.

This type of test method is applicable to many other polymers including polyolefins, polyamides, polycarbonates, ABS, and ionomers (1). A standard test method has been developed (2).

Sodium tartrate dihydrate (F = 0.1566), one commonly used moisture standard, should not be analyzed at temperatures greater than 220°C because the organic portion of the material begins to decompose and may deteriorate the detector. The recommended calibration material is sodium tungstate dihydrate (F = 0.1092) which is stable to temperatures above 1000°C. Water itself (F=1.000) can also be used in the microcapillary techniques described in the Operator's Manual.

REFERENCES

1. Goodyear Chemicals, Polyester Test Method R-1236 (1971).
2. ASTM Test D4019-81.

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