TA Instruments

Thermal Analysis & Rheology

Thermal Analysis Application Brief

Determination of Moisture in Epoxy Resin

Number TA-112

SUMMARY

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

INTRODUCTION

Reproducible flow properties for the thermoset molded resins are necessary to insure that the manufacturing conditions used, such as mold temperature, mold pressure and cure time, consistently yield a usable final product. Lot-to-lot variations in flow properties can result in material which does not mold properly and must be rejected. One quantity which can substantially influence thermoset flow, as well as cause other processing problems, such as blistering or decrease in hot strength, is moisture (1). Moisture content of 2% or less can often cause a several-fold change in moldability. Hence, rapid moisture evaluation and subsequent drying of thermoset resins before molding is vital.

The TA Instruments 903 Moisture Evolution Analyzer can be used to quickly and accurately measure the moisture content in thermoset resins. In MEA the sample is placed in an oven, and heated to volatilize the water which is swept to an electronic detector by a predried nitrogen carrier gas. In the electrolytic cell detector, the water is absorbed by phosphorus pentoxide coated on platinum electrodes, and electrolyzed to hydrogen and oxygen. The electrolysis current is integrated, scaled, and displayed as µg water on an LED display.

The electrolytic cell detector is remarkably selective for water. Water levels as low as 10 ppm can be routinely determined and with special care (e.g. dry box environment) levels or accuracies down to 1 ppm are possible.

EXPERIMENTAL

The experiment conditions used depend to some extent upon the moisture level and nature of the sample. The following conditions were used to analyze epichlorohydrin bisphenol A-type epoxy resins containing less than 1% moisture.

Sample weight:	0.3-0.5g
Temperature:	125°C
Time:	20 minutes
Flow Rate:	50 mL/min

1. The sample is placed in a tared sample boat and weighed, then placed in the instrument and analyzed under the conditions shown.

2. The "count" thus obtained is compared with a blank count previously obtained with no sample in the chamber, 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 by the equation given below.

CALCULATION

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

$$K = \frac{F \times W_{std} \times 100}{C_{std} - C_{blank}}$$

Where: C_{blank} = count obtained for blank

C_{sample} = count obtained for sample

C_{std} = count obtained for standard material

F = fraction of standard material attributable to water

K = calibration factor in milligram % water per count (should be near 0.1)

W_{sample} = weight of sample in mg

W_{std} = weight of standard in mg

RESULTS

Using the procedure described above, standard deviations of ±0.035% (abs) were obtained on epoxy resin samples analyzed to contain 0.45-0.70% (abs) water.

Observation of the moisture evolution as a function of temperature indicates that these powdered epoxy resins contain two types of moisture — loosely held surface moisture which is rapidly swept from the sample by the nitrogen purge gas at ambient temperature, and more tightly bound moisture which requires higher temperatures to free. Some of the deviation present in the epoxy resins here is due to differences in sample particle size which results in different surface moisture levels.

Epoxy resin moisture levels are also dependent on the nature of the resin and added processing agents. Moisture pickup is related to resin polarity with hydrophilic functional groups containing oxygen serving as absorption sites for water. Hence, for example, epoxides with low epoxy equivalent weights (smaller molecules) tend to show greater moisture pickup (2).

Finally, moisture pickup and retention is dependent on the resin form. Cured, laminated and otherwise reinforced epoxies have been shown (3) to exhibit substantial moisture affinity and deleterious effects on end-use properties. The moisture affinity of these materials also may be evaluated though the use of the MEA.

REFERENCES

1. J. E. Hauck, Modern Plastics, 48, 11 (1971)
2. E. Watanabe and R. Sezizawa, Tokyo Metropolitan Univ. Memoirs, 23, 2043 (1973)
3. E. L. McKogue, Jr., J. Appl. Poly. Sci., 22, 1643 (1978).

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