Kurze Mitteilungen

Estimation of the Curing Rates of Epoxy Resins; Improvements in Method *

Bestimmung des Härtingsgrades von Epoxyharzen;
Verbesserung der Methode

WALTER SELIG

Lawrence Radiation Laboratory, University of California,
Livermore, California

Received March 22, 1971

We have previously described a new method [3] for estimating the curing rates of epichlorohydrin/Bisphenol A type epoxy resins cured with complex amines and polyamides. Additional work has led to the following improvements:

The titrant, 0.1 N mercuric perchlorate, previously was standardized potentiometrically against potassium bromide. It may be more rapidly standardized as follows: Dilute an accurately measured amount of mercuric perchlorate to 30–40 ml with water. Add 1 ml of pyridine and 4 drops of 0.2% aqueous methylthymol blue [1]. Titrate with standard EDTA to a sharp color change from blue to yellow. The indicator deteriorates on standing and should be kept no longer than 4 weeks.

The tetraethylammonium bromide (Et_4NBr) solution previously was standardized against 0.1 N mercuric perchlorate in 70–75% p-dioxane/water. The results are more precise when this standardization is performed in p-dioxane only (no water), to which 3 ml of 1 N perchloric acid are added. Potential breaks near the endpoint are larger than in the partially aqueous medium, and the solvent is now the same as that used in the analytical procedure. Addition of the perchloric acid increases the dielectric constant of the titration medium.

Best results are obtained if the sample contains 0.7–1.4 mmoles of epoxide and is reacted with 2 mmoles of Et_4NBr (10.0 ml of 0.2 M reagent). This results in a 40–200% excess of Et_4NBr . Satisfactory results are obtained when the total volume prior to titration is 35–100 ml. These conditions were established with Epon 828 (Shell Chemical Co.), as shown in the Table. The mean epoxy equivalent for Epon 828 was 188.25, with a standard deviation of 1.97.

* Work performed under the auspices of the U.S. Atomic Energy Commission.

Table. Epoxy equivalent of Epon 828^a

Volume, ml	Epoxide taken, mmoles	Epoxy equivalent ^b	Excess Et_4NBr , %
35	0.95	190.1	112
50	0.41	182.8	75
50	0.72	186.7	178
50	0.89	188.8	123
50	0.91	189.6	117
50	0.95	188.0	111
50	1.12	188.8	80
50	1.15	188.2	75
50	1.36	189.9	47
50	1.53	191.9	32 ^c
50	1.72	212.4	17 ^c
65	1.15	188.9	75
80	1.02	187.8	98
100	0.95	189.4	112

^a Reacted with 10.0 ml 0.2 N Et_4NBr + 3 ml 1 N HClO_4 . Mean epoxy equivalent by the $\text{HClO}_4/\text{Et}_4\text{NBr}$ method [2] was 185.3.

^b Epoxy equivalent = sample weight (mg)/epoxide (mmoles).

^c Value excluded from calculation of mean and standard deviation.

References

1. Belcher, R., Wilson, C. L.: New methods of analytical chemistry, 2nd ed., p. 47. London: Chapman and Hall 1964.
2. Jay, R. R.: Anal. Chem. **36**, 667 (1964); cf. Z. Anal. Chem. **207**, 398 (1965).
3. Selig, W., Crossman, G. L.: Z. Anal. Chem. **253**, 279 (1971).

W. Selig
Lawrence Radiation Laboratory
University of California
Box 808
Livermore, Calif. 94550, U.S.A.

Anion Exchange Behaviour of Antimony(III) in Malonate Solution

Verhalten von Antimon(III) beim Anionenaustausch in Malonatlösung

S. B. AKKI and S. M. KHOPKAR

Department of Chemistry, Indian Institute of Technology,
Bombay-76 (India)

Received February 8, 1971

A systematic study has been carried out on the anion exchange behaviour of the antimony(III)-malonate complex and its use for the separation of antimony from various foreign ions. The method is extended to antimony based alloys. The negatively charged complex is formed in 5% malonate solution at pH 4.8.