Estimation of Diglycidyl Ether in Epoxide Resins

The estimation of diglycidyl ether (DGE) of bisphenol-A and also other components present in an epoxide resin enables the correct evaluation of it since its properties are influenced[1] by the amount of DGE initially present which is in turn dependent on the molar ratio of reactants.[1,2]

The present investigation deals with a column chromatographic method (CCM) for the quantitative isolation of DGE and with a preparative thin layer chromatographic (TLC) method for the determination of overall composition of the resin.

The resin was prepared, as usual, by condensing 10 moles of epichlorohydrin, 2 moles of bisphenol-A with 2 moles of NaOH in *sec*-butyl alcohol at 80–85°C. It had an epoxide equivalent of 191, hydroxyl value of 0.16 per molecule, and, on T.L.C. analysis according to Weatherhead,[3] indicated the presence of six components as represented in Figure 1.

The separation of DGE from other components of the resin was made by CCM, wherein activated silica gel (BDH) was used as adsorbent and chloroform (E. Merck) as eluting solvent in a column of 1 cm^2 cross section with an effective length of 32 cm loaded with 0.1869 g of the resin. Fractions (2 ml each) denoted by test tube numbers in Figure 2 were collected at an elution rate of 1 ml/min and weighed. The purity of the fractions was monitored with the help of TLC.[3] The sequence of separation is represented in Figure 2. The recovery of DGE was quantitative, amounting to 77.5% of the resin having an OH value of 0, epoxide equivalent of 170.5, and C/H = 10.5. Total recovery of the material from the column was 93.9%.

Since the separation of all the components besides DGE was not effective by CCM (*cf.* Fig. 2) further investigation on quantitative determination of individual component in the resin was made by a preparative TLC method using Silica Gel G, 0.8 mm in thickness, and chloroform as eluting solvent. Each plate was loaded in band form with 50 mg of 4% resin solution. The components thus separated in band form were located

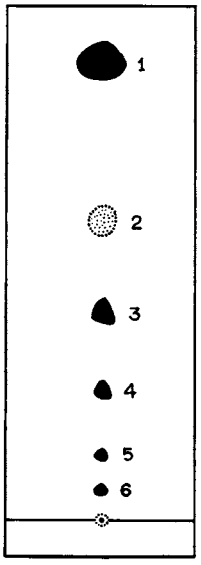

Fig. 1. Thin-layer chromatographic separation of the components of epoxide resin. The silica gel G plate was eluted once with chloroform and developed by spraying with 50% sulphuric acid, followed by heating at 150°C for 20 min. in an air oven, and reproduced by tracing the outlines of the spots.

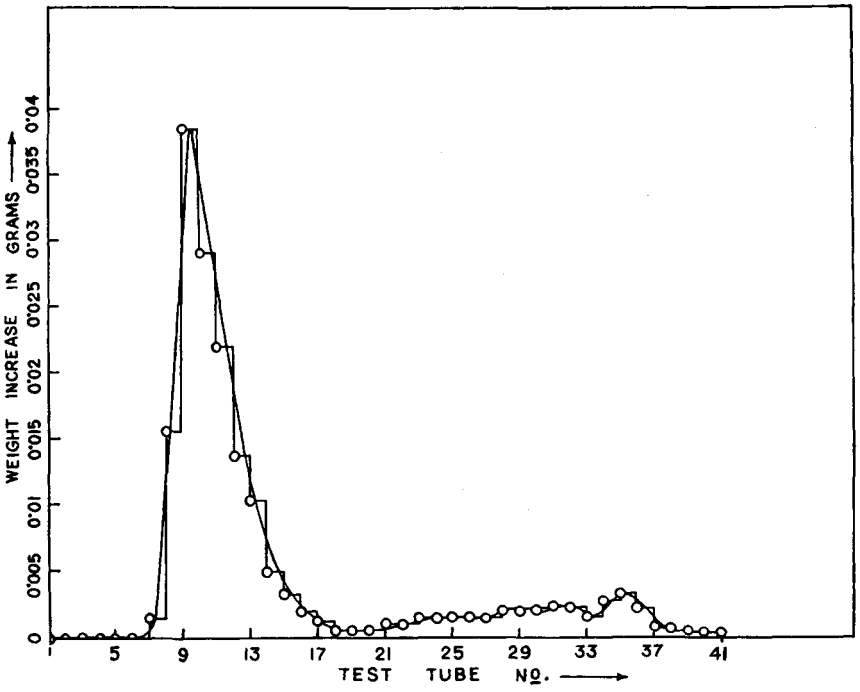

Fig. 2. Plot of test-tube number vs. amount of residue left in each test tube.

by using an indicator band of the sample along the edges of the plate. These components were detected by spraying with 0.005% aqueous Rhodamine B (visible under uv light) and finally estimated gravimetrically after extracting a separated fraction from each of three such plates with warm chloroform. The individual components present in the resin were thus found to be 75.5, 2.1, 12.4, 5.6, and 4.4% corresponding to spot number 1 (DGE), 2, 3, 4, and 5, 6 (combined) respectively (cf. Fig. 1).

References

1. P. K. Ghosh and A. N. Saha, *Indian J. Appl. Chem.*, in press.
2. H. Lee and K. Neville, *Epoxide Resins*, McGraw-Hill, New York, 1957, page 10.
3. R. G. Weatherhead, *Analyst*, **91**, 245 (1966).

P. K. Ghosh
C. Bandyopadhyay
A. N. Saha

Department of Applied Chemistry
University College of Science & Technology
Calcutta, India

Received March 5, 1968
Revised April 1, 1968