## The Dielectric Constant of Triethanolamine Borate

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The nearly spherical triptich or cage-type structure proposed for triethanolamine borate by Brown and Fletcher1) suggested the possibility of molecular rotation in the crystalline state. Although Westrum<sup>2)</sup> concluded, from his thermal measurements, that such rotation was unlikely, this paper will present a study of the dielectric properties in the crystalline state in an endeavor to shed further light on the nature of this transition.

The raw material synthesized from triethanolamine and boric acid was provided by the courtesy of the U. S. Borax Co.\*; it was vacuum-sublimed four successive times at 180°C and 10-3 mmHg. The melting point of the purified sample was 513°K, in good accord with the literature values of 508°K1) and 512°K2). The sample was put into the measuring electrode under streaming nitrogen gas. A bridge with a frequency range from  $3 \times 10^2$  to  $3 \times 10^4$  c. p. s., as described elsewhere,<sup>3)</sup> was used. Temperatures over the range from 10°C to 238°C were determined with a copperconstantan thermocouple attached firmly to the electrode cell. The dielectric constant versus temperature curves obtained are shown in Fig. 1.

No frequency dependency of the dielectric constant was noted over the available range studied. At lower temperatures the dielectric loss is approximately 10-2 to 10-3. However, the resistance of the sample decreased very rapidly to 105 ohm or less as the melting point was approached; hence, it was beyond the range of the bridge. Only a large and abrupt increase in the dielectric constant at the melting point could be observed; the dielectric constant of the liquid state was not obtained.

As is shown in Fig. 1, a small change in the di-

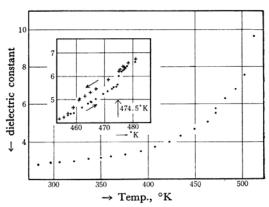


Fig. 1. The dielectric constant versus temperature curves of triethanolamine borate. The dotted and crossed points of the upper figure show the measured values in increasing and decreasing temperatures, respectively, near the phase transition.

electric constant may be noted at 474°K. The dielectric transition point presumably corresponds to the phase transition point, 466.54°K, found by Westrum.2) Since both the increment in the dielectric constant and the entropy of transition are relatively small, the change in chemical bonding from a triptich to a cage-type structure is probably not involved in the mechanism of the phase transition. Moreover, there is no evidence of a rotational transition of the molecular dipole at this temperature; presumably dipolar rotation commences only at the melting temperature.

These results thus confirm the conclusion by Westrum that this material is not plastically crystalline.

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<sup>1)</sup> H. C. Brown and E. A. Fletcher, J. Am. Chem. Soc., 73, 2808 (1951).

<sup>2)</sup> H. L. Clever, W. Wong, C. A. Wulff and E. F. Westrum,

Jr., J. Phys. Chem., 68, 1967 (1964).

\* This sample was obtained by the kind help of Professor Edgar F. Westrum, Jr., University of Michigan.
3) S. Kondo and M. Matsumoto, This Bulletin, 31, 319 (1958).