

a constant rate⁽⁶⁾ as the piston, K, is withdrawn in the precision bore of this cylinder, 5, by the constant-speed motor, M, and reduction gears, Q. A pressure seal for the piston was made with a washer of specially formulated rubber⁽⁷⁾ which was locked in position by a steel ring at the upper end of the piston. Washers of this material operated as pressure seals without leaking and were in good condition after 600 hours of use.

The camera, 6, is automatically operated by a synchronous clock motor so that a picture of the Bourdon gage and of a spring-driven watch is made every ten minutes. The watch provided a convenient way of assuring that the photograph was taken every ten minutes and that the electric current to the apparatus was uninterrupted. The maximum variation in a 10-minute interval was 3 seconds. Sufficient film was used for about 20 hours of continuous operation. Since the decompression rate and the thermostated bath were also automatically controlled, the apparatus could be run at night and over weekends without attention. About 4000 individual pictures from the pressure experiments on these acids were thus made. The pressure readings were plotted as a function of time. Phase transitions were represented as nearly horizontal portions of the curves. As the samples became more impure due to decomposition, these plateaus for the solid-liquid transitions became less distinct and exhibited increased curvature. These curves were analysed as described below to obtain the volume changes and sample purities.

3. EVALUATION OF SAMPLE PURITY

The time-pressure curve in the fusion region may be used to evaluate the impurity of the sample in procedures similar to those used for

(6) With cylinder 5 operating, the volume increase in the system at 4 was determined from calibration experiments to be $3.99 \times 10^{-3} \text{ cm}^3$ per minute. These calibration experiments with benzene in the sample container were performed prior to this research.

(7) The Rubber Section at the National Bureau of Standards supplied this rubber after a request to replace the polyvinylchloride [10] with a more elastic type of material. The greater elasticity was to overcome small imperfections (such as longitudinal scratches) in the bore of the decompression cylinder, which had become damaged on prior usage and leaked with polyvinyl chloride washers. The authors are indebted to Frank L. Roth for this special oil-resistant Neoprene rubber which was formulated in parts by weight as follows: 100, Neoprene; 20-30, carbon black; 5, zinc oxide; and 5, magnesium oxide.