

A Modified Capillary Dilatometer for Polymerization

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Synopsis

A relatively simple and inexpensive instrument giving good accuracy and easy operation has been developed for the measurement of volume change in monomer polymerizations. The instrument is based on a previously described capillary dilatometer intended for automatic operation.

The polymerization of monomers is a problem of considerable interest. In studies of polymerization of monomers change of volume is a very simple and fundamental polymeric experiment. An instrument which measures this volume change is valuable both in studying the mechanism of polymerization and in comparing the efficiency of catalyzers in promoting polymerization.

A simple device for this purpose, wherein a breaker-seal tube containing the sample was connected to the body of a dilatometer by means of a joint and the seal broken by various operations was described by Acres et al.¹ Acres et al.'s instrument (for measurement of volume change) in which the sample is held in the breaker-seal tube so that breaking by various operations does not contribute to the simple device has been somewhat modified to render it more versatile and convenient in use, the latest form being described below.

A reaction vessel for containing the sample was constructed. It has a two-tap device one on the top and the other at the end of the reaction vessel. The reaction vessel was marked with a scale. From this instrument a complete volume change-time curve of high accuracy can easily be constructed.

Construction and Procedure of Dilatometer

The sample is prepared in a reaction vessel 3 (Fig. 1), equipped with two taps 2, 4. One tap, 2, is at inlet I for sample and the other, 4, is connected to the capillary tube of dilatometer IO by means of capillary system 5 with mercury. When measuring the volume of sample the tap 4 is closed and the reaction vessel 3 is filled with sample. By changing the position of a mercury reservoir 9 and opening the tap 4, II, mercury is forced through a gum tube 8 into the capillary system 5, IO, and the reaction vessel 3. After the volume of the sample is measured, taps 2

and II are closed and the initial mercury level of the capillary tube 8 is measured by manual or automatic operation. The capillary tube of appropriate diameter IO, with an earthing connection sealed through it, was fitted into a socket 7 and is held by two short lengths of rubber tubing against a steel rod covered in polyethylene. The steel rod I2 has a small metal plate attached to one end for connection to the capacity bridge.

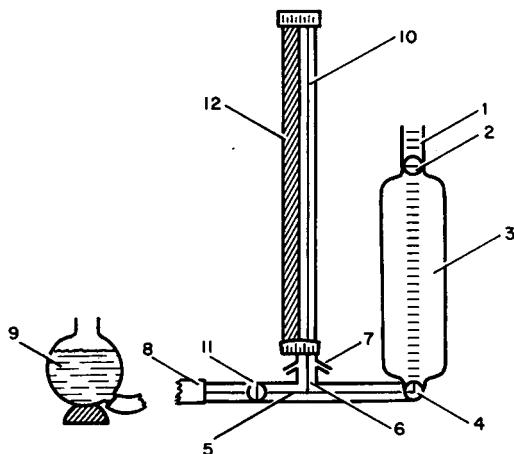


Fig. 1. Diagram of dilatometer.

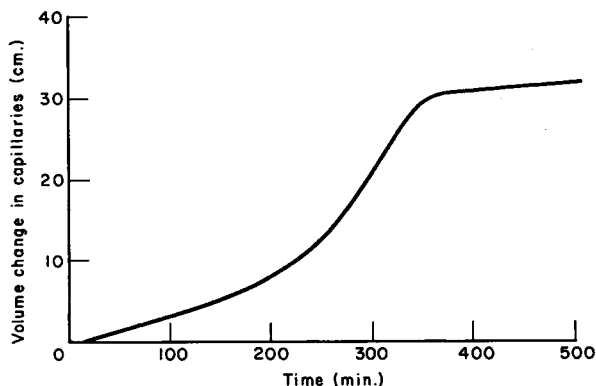


Fig. 2. Volume change-time curve.

The capillary tube 8 is connected by means of its socket 7 to a cone 6; vacuum grease is used to seal the joint.

The equipment has been used to measure the change of volume from 0.3–40 cm. in capillaries whose diameters ranged from 3 mm. to 0.5 mm. For changes of volume greater or less than these values capillaries of other diameters were used.

Very fine volume change-time curves have been obtained by use of the automatically and manually operated dilatometer (Fig. 2), and the in-

strument has the great advantage of being very easily very operable for the initial preparation and run.

Reference

1. G. J. K. Acnes and E. L. Dalton, *Nature*, **184**, 335 (1959).

Résumé

Un instrument relativement simple et peu coûteux donnant une bonne précision et facile à utiliser a été mis au point pour mesurer la variation de volume de monomère en cours de polymérisation. L'instrument est basé sur un dilatomètre capillaire précédemment décrit et mis au point en vue de permettre les opérations automatiques.

Zusammenfassung

Ein verhältnismässig einfaches und wohlfeiles Instrument mit guter Genauigkeit und leichter Handhabung wurde zur Messung der Volumsänderung bei der Polymerisation eines Monomeren entwickelt. Das Instrument hat ein für automatischen Betrieb ein gerichtetes, früher beschriebenes Kapillardilatometer zur Grundlage.

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