TABLE II

	$\Delta\beta$	$\Delta\beta_2/\Delta\beta_1$	K
Polyethylene	7×10^{-4}	0.57	0.49
Polypropylene	4×10^{-4}		

should be equal to the value of K (Table II); in this case there is only a fair agreement.

If transition temperatures are plotted against molar copolymer compositions, one can obtain a linear relationship, in agreement with the results of Natta and Crespi[11] obtained by minimum rebound temperatures measurements.

Because of the good reproducibility (Table I) of the dilatometric second-order transition temperature determinations, the propylene content of such a copolymer could be evaluated in this way.

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Received March 18, 1960

Low Temperature Bath

Several low temperature baths are described in the literature. Many designs call for elaborate and expensive refrigeration equipment. We have found none that meet the need for a bath requiring little capital outlay and capable of operating for several months at low cost. To meet these requirements, a bath has been designed and constructed from materials which should be available to any laboratory. The bath was used in crystallization studies described in this journal,[1] involving exposure of samples in dilatometers for periods of several weeks at various temperatures between 0 and −45°C. Cooling was accomplished by using metal conductors to transfer heat from the controlled bath to a cold source.

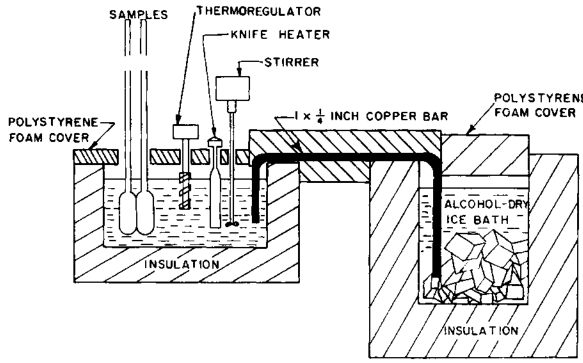

Fig. 1. Low temperature bath.

Referring to Figure 1, it can be seen that the controlled bath is cooled by conduction along 1 × 1/4-in. copper bars to an alcohol–Dry Ice mixture. This is similar in principle to the operation of a device called a cold bar described by Mooney and Wolstenholme.[2] The copper bars were bent to the shape indicated and their ends inserted in the controlled bath and cold source. Any well insulated container may be used for the controlled bath. In the work mentioned above, a 1-gal. Dewar flask was employed. Foamed polystyrene was found to be a convenient material for lids and for insulating the copper bars and other portions of the apparatus. The Dry Ice bath consisted of a 10-gal. bucket inside a 30-gal. can, the air space being filled with vermiculite insulation. A stirrer in the controlled bath was the only moving part. The number of copper bars needed to maintain a given temperature was determined experimentally. It was found, for example, that one copper bar conducted enough heat to maintain an equilibrium temperature of about 0± 1°C. Two bars resulted in an equilibrium temperature of about −20°C., three bars −35°C., and four bars −45°C. Equilibrium temperature was reached in about one day without any additional cooling.

More precise control was obtained by using a greater heat leak than necessary and opposing this with a controlled heater. This also permitted operation at intermediate temperatures. For example, to operate the bath at any temperature above −45°C., four copper bars were employed and temperature maintained with a 125-w. knife heater. The heater was controlled by a standard thermoregulator arrangement, which provided ±0.1°C. temperature control.

The bath seems ideally suited to long-term exposure of materials that impose only a small heat load on the apparatus. Thermal losses from the bath were found to be quite low, and Dry Ice consumption was minimal. For example, a 10-gal. Dry Ice–alcohol cold source was sufficient to maintain bath temperature over a Friday-to-Monday weekend.

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Received June 27, 1960