

thiocyanate ion must be more or less dissociated in solution, and we do not wish to deny that ions of the type postulated by Møller and by Bent and French play an important role in this test.

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### Dicyclopentadiene: Preparation from the Monomer; Dielectric Constants of Dimer at Several Temperatures

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The usual method of preparing dicyclopentadiene is to fractionally distill the crude, impure dimer, and allow it to polymerize at room temperature overnight. This procedure is repeated a number of times, depending upon the purity of the original material, until a fraction is obtained that distills over at 41.5°—the boiling point of pure cyclopentadiene. When this latter material polymerizes, it yields a white, crystalline solid. The accepted values for the melting point and refractive index of dicyclopentadiene are given as 31.5° and 1.5120 (20°), respectively.

In an attempt to shorten the preparation of the dimer, we have adopted a slightly different procedure. Dicyclopentadiene obtained through the courtesy of the Research Laboratory of the United Gas Improvement Co. of Philadelphia was a faint straw-colored liquid containing 98% dimer. It was fractionally distilled once, and the distillate which came over between 40 and 44° was collected. This was allowed to stand overnight at 15°. The incompletely polymerized liquid was then placed under vacuum to remove the last traces of monomer and any other volatile impurities. Crystallization took place suddenly. The melting point of this crystalline solid was 27.8°, corrected, but the refractive index of the melt was identical with that of the solid which melted at 31.5°. After resolidification, the melting point was 31.5°, the same as that of the dicyclopentadiene prepared by the previous method.

It was found that low temperature (15–20°) was essential to the formation of the above crystals which we shall call an isomer of dicyclopentadiene. This form of the dimer was readily reproducible, the melting point being 27.8° for each preparation. The melting point and freezing point after resolidification was always 31.5°. When polymerization was carried out at slightly

higher temperatures and when polymerization was not hastened by pumping off the more volatile monomer, the solid produced melted at 31.5° and possessed a strong, camphor-like odor. The isomer, on the other hand, had only a slight odor. Microscopic examination showed the crystalline forms to be quite different.

This evidence seems to indicate that a polymorph or an isomer of dicyclopentadiene has been obtained. From the accepted formula of this compound, *cis* and *trans* isomers are possible. It is reasonable to assume that these two forms exist as dynamic isomerides, since any operation which changes the form of the crystals melting at 27.8° leads, upon resolidification, to the more stable form having a melting and freezing point of 31.5°.

**Experimental.**—The dielectric constants were measured with a parallel resistance bridge previously described.<sup>1</sup> The beat frequency was set at 1000 cycles. A small multiple plate variable condenser inserted in a small Pyrex tube was used as the cell. About 25 ml. of liquid sample was required to completely immerse the plates of the condenser. The cell assembly was placed in a thermoregulated oven which was controlled to  $\pm 0.05^\circ$ . The capacitance of the cell in air did not change over the temperature range employed. The time allowed for the temperature of the cell to reach equilibrium with the oven was forty-five minutes for each 10°.

No difference in dielectric measurements was observed when a frequency of 21,000 cycles was used instead of 1000. The values of the dielectric constant of dicyclopentadiene thus obtained were

$t^\circ \text{C.}$	40	50	60	70	80	90	100
$\epsilon$	2.43	2.41	2.39	2.37	2.35	2.33	2.31

These data indicate that over this temperature range dicyclopentadiene does not possess a very high dipole moment.

(1) American Society for Testing Materials, Specification D150 36T.

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## NEW COMPOUNDS

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### Phenacyl, *p*-Phenyl- and *p*-Bromophenacyl, and *p*-Nitrobenzyl Esters of Certain $\alpha$ -Hydroxy Fatty Acids

The authors recently had occasion to synthesize a series of  $\alpha$ -hydroxy fatty acids, of which, among other derivatives, the following new esters were prepared. The procedures for their preparation and for the determination of