

of this method is not as good as the attainable accuracy of the optical diffraction or pulse method, particularly for materials the acoustic impedances of which differ appreciably from those for water or oil, but the low absorption at high temperatures at less than 1 Mc./s. is not a source of error. The velocity results at 350 and 750 kc./s. are not significantly different, and the transition temperature over a series of runs varied between 66° and 71° C. These results are similar to those obtained by Melchor² and by Krishnamurthi¹.

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Low-temperature Sintering of Titanium Dioxide for Ceramic Capacitors

THE investigations carried out by Berberich¹, Von Hippel² and Wainer³ and their associates on titanium dioxide point to certain optimum firing conditions. In particular, a temperature of 1,350° C. is found to be suitable for obtaining most of the desirable electrical characteristics. Among these characteristics may be specially mentioned low water absorption, high permittivity, high insulation resistance and low loss-angle. From the known values of the two principal permittivities of the single crystal of rutile, the permittivity for randomly oriented powdered rutile would be about 114. The values of the permittivity reported in the literature vary from 96 to 100, and water absorption is known to be about 0.1 per cent. The rutile form of titanium dioxide used in their experiments was prepared by calcining commercial quality titanium dioxide, probably of the anatase type, at 1,350° C.

In some large-scale experiments carried out at the National Physical Laboratory of India on a particular brand of commercial quality of titanium dioxide bearing the trade name "Rutiox CR" (Kronos), it has been possible to obtain ceramic bodies with excellent dielectric properties when fired at temperatures as low as 1,100° C. in electric furnaces. It was not found necessary to calcine the material first, and a straight firing schedule was adopted which gave well-sintered bodies. The neutral atmosphere of the electric furnace and the partially reducing atmosphere of a gas furnace gave identical results. It was also found that the sintering temperatures were not very critical, as reported by earlier investigators. Satisfactory bodies could be obtained when fired over a range of temperatures from 1,100° to 1,400° C.

X-ray diffraction studies and the density measurements showed that the raw material was of the rutile crystalline form. Measurements by microscope of particle-size indicated sizes generally in the range 1-5 microns, with crystal edges sharp, indicating that the titanium dioxide was perhaps manufactured in these particle sizes, by chemical processes and not by grinding calcined material.

Some typical values obtained with bodies prepared as stated above are: water absorption, 0.01 per cent; permittivity, 110.

It would appear that the low sintering temperatures, coupled with good electrical properties, are due to the rutile crystalline form and the finely divided active state of the titanium dioxide used.

Fuller details of the experiments will be reported elsewhere. We are indebted to Sir K. S. Krishnan for his advice and guidance.

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Polymerization Reactions occurring during Dinitrophenylation of α -, β - and ω -Amino-acids with Sanger's Reagent

NUMEROUS papers have been published concerning the identification and determination of amino-acids by the dinitrophenylation method introduced by Sanger¹. We are not aware, however, of any indications in the literature about the occurrence of polymerization reactions during dinitrophenylation. In an attempt to work out a procedure for the quantitative determination of minute amounts of ϵ -aminocaproic acid and its linear oligomers, we have found that such polymerization reactions do occur. Similar results have been found during dinitrophenylation of other ω -amino-acids and of glycine and β -alanine. Since these side-reactions may lead to complications in investigations on the separation and determination of dinitrophenyl amino-acids in general, it seems useful to communicate these facts briefly.

Dinitrophenylations were carried out by Sanger's method¹, using 1-fluoro-2,4-dinitrobenzene in aqueous ethanol in the presence of sodium bicarbonate. Isolated products as well as samples of reaction mixtures were analysed by chromatography on paper strips using a descending technique (see Table 1). Since pure ϵ -aminocaproic acid and the nearly pure synthetic dimer and trimer of the acid² were at our disposal, chromatographic spots of the corresponding dinitrophenyl derivatives could be easily identified. With the eluent used, R_F -values decrease with increasing molecular weight (Table 1). The purity of our samples of aminocaproic acid was checked by chromatography of aminocaproic acid as such (method worked out by van der Want², slightly modified).

From a mixture of initial composition 0.75 m.mol. ϵ -aminocaproic acid, about an equimolecular amount of fluorodinitrobenzene and 1.6 m.mol. sodium bicarbonate in 10 ml. aqueous ethanol (25 per cent of water), reacting at room temperature, samples were taken at successive intervals. Each sample served to place ten spots (of 10 μ l. each) on a chromatographic strip. Depending upon the duration of reaction, one or more additional spots appeared on the eluted chromatograms besides the expected spot of dinitrophenyl-aminocaproic acid. From their R_F -values relative to that of the latter, these spots could be identified as the dimer, trimer and so on.