A NEW METHOD FOR MEASURING THE INDUCTION PERIOD OF THE OXIDATION OF POLYMERS

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A simple method independent of oxygen absorption measurements was developed for estimating the induction period of the oxidation of polymers. The principle of this method is the estimation of the time in which a sample oxidized at constant temperature exhibits a temperature rise as a consequence of the exothermal effect of the oxidation reaction. The temperature rise is measured as the temperature difference between the sample and a reference material. Results of measurements of induction periods of the oxidation of polymeric substrates inhibited by various antioxidants, obtained both from oxygen absorption measurements and by the new method, are discussed.

The induction period of the oxidation of polymers is usually estimated from oxygen absorption measurements. In some cases these measurements are complicated, due to experimental difficulties or to the conditions of the oxidation test. A simple method independent of oxygen absorption measurements has been developed for these cases.

The principle of this method is the estimation of the time in which a sample oxidized at constant temperature exhibits a temperature rise as a consequence of the exothermal effect of the oxidation reaction. The temperature rise is measured as the temperature difference between the sample and a reference material. This principle was used for the first time by Rudin [1] for the evaluation of antioxidants for polyethylene. In spite of its advantages, the method is not widely used and there have been no further publications dealing with its application.

Apparatus

We tried to develop an apparatus to record curves from samples oxidized under the same conditions as used in oxygen absorption measurements [2], or to obtain both induction period values from one and the same oxidation test. This requirement and the principle of the method led to the apparatus illustrated in Fig. 1.

The sample is placed in an oxidation test tube, practically the same as for oxygen absorption measurements. A chromel–alumel thermocouple is inserted in the sample and connected counterwise to the reference thermocouple placed in a micro test tube close to the test tube with the sample. The reference thermo-
couple is placed in anhydrous alumina the weight of which is equal to that of the sample. Preliminary experiments showed that application of an inert reference material is not necessary. Compared to differential thermal analysis this technique is not a dynamic thermal process, and therefore the influence of the environ-

Fig. 1. Scheme of the apparatus. 1 — electronic relay, 2 — contact thermometer, 3 — auxiliary relay, 4 — autotransformer (2.5 A/220 V), 5 — rheostat, 6 — voltmeter (120 V), 7 — heating coil (350 W/220 V), 8 — thermocouples (chromel–alumel, 0.3 mm diameter), 9 — Dewar vessel with cold junctions, 10 — recorder (100 μV/280 mm), 11 — micro test tube with reference thermocouple, 12 — micro test tube with the sample, 13 — ground glass stopper, 14 — the part of glass stopper with thermocouples outlet, cast with silicone rubber, 15 — test tube for oxygen absorption measurements, 16 — side capillary, 17 — thermal insulation, 18 — hole for contact thermometer, 19 — cylindrical aluminium block with eight holes for oxidation test tubes

ment of the reference thermocouple is not significant. For better manipulation we worked without alumina except for testing the influence of certain factors. The constant temperature was maintained by an aluminium heating block with two heating coils. The temperature difference was recorded continuously

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