A Simple Attachment for the Interruption of Thermal Degradation of Polymers

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A complementary tube attachment to the equipment used for thermal destruction of polymers is described. This device enables to interrupt the thermal decomposition of polymers in any elect time by drawing the sample out of the heated part of the apparatus.

The equipment discussed previously [1] has been successfully used for the analysis of polymer mixtures of graft or block copolymers. This equipment is lacking, however, the possibility to terminate the pyrolysis process in exactly determined time. This purpose has been achieved introducing a simple device depicted in Fig. 1.

Experimental

Apparatus

One end of a glass tube *7* is necked-down for the inert gas inlet *1*. The other end is provided with a standard ground joint *8*. A metallic chain *3* and *6*, connecting two small hollow iron cylinders *2* and *5* and a platinum vessel *10*, is located inside of the tube. The vessel is attached to the chain with an eye from platinum wire and is further spatially stabilized with a piece of platinum wire *9* situated in the middle of the vessel, being traped to its lip. The glass tube is necked-down on the bending point. The narrowing *4* serves as the feed stop for the cylinders.

Procedure

Sample weighing 1 — 10 mg is placed into the platinum vessel *10*. The attachment is fitted to the quartz tube heated by an electric oven. The quartz tube, its heating and the temperature measurement has been described in our previous work [1]. After washing the apparatus with an inert gas, the sample is transferred into the heated part of the tube. This is performed by a strong magnet moving the iron cylinder *5* towards the ground joint *8* in the shortest possible time. The platinum vessel stops descending into the heated part of the tube as soon as the cylinder *2* reaches the glass tube narrowing *4*. The sample is thus situated in the space in which the pyrolysis takes place at the controlled temperature. The time interval of keeping the sample in this position depends first of all on the temperature as well as on the desired degree of the polymer destruction. The suitable selection of both time and temperature is particulary important when the described equipment is directly connected with a gas-liquid chromatographic column, in order to obtained a good peak resolution [2].

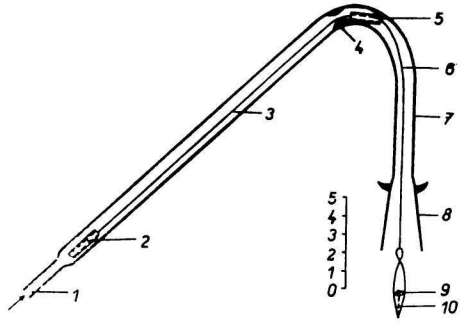

Fig. 1. A simple attachment to the equipment for thermal destruction of polymers. *1.* inert gas inlet; *2.* and *5.* hollow iron cylinders; *3.* and *6.* metallic chain; *4.* tube narrowing; *7.* glass tube; *8.* standard ground joint; *9.* platinum wire; *10.* platinum vessel.

The degradation of the sample can be suddenly stoped by magnetic actuation the iron cylinder *2* (traped at the neck *4*) towards the inert gas inlet *1*. The cylinder *2* stops when the other one *5* reaches the narrowing *4*. The platinum vessel *10* with the undegraded sample rises thus from the oven to its former position which is illustrated in Fig. 1.

References

1. Ďurďovič V., *Chem. Zvesti* **20**, 611 (1966).
2. Kyseľ O., Romanov A., Ďurďovič V., *Eur. Polym. J.*, in press.

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