

## NUCLEAR MAGNETIC RESONANCE IN POLYMERS IN THE AMORPHOUS AND CRYSTALLINE STATES\*

A. I. MAKLAKOV and G. G. PIMENOV

Kazan State University

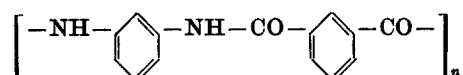
(Received 13 June 1964)

POLYMERS capable of crystallizing can be obtained in the amorphous state by rapid cooling of the melt to below glass temperature,  $T_g$ . Subsequent heating induces crystallization accompanied by increased rigidity of specimen and variation of macromolecular mobility, as confirmed by thermomechanical investigations [1].

Direct observation of the variation of molecular mobility is made possible by the use of a nuclear magnetic resonance technique (NMR) [2]. Therefore, NMR can be used to study the behaviour of macromolecules of amorphous polymers incapable of crystallization on being heated above  $T_g$ . This paper is devoted to a study of this problem.

### EXPERIMENTAL

Polyethyleneterephthalate (PETP), aromatic polyamide (APA)<sup>†</sup> of the following structure



and isotactic polystyrene (PS) were chosen as specimens for the investigation. These polymers were chosen because at room temperature, according to heat treatment, they can appear in amorphous or crystalline state.

The PETP used was a transparent amorphous non-oriented film which, when obtaining the first NMR spectra, became turbid, i.e. crystallized—which was verified by X-ray photographs. Subsequently, this specimen was treated as crystalline.

Initial by APA is an amorphous powder which similarly to PETP, on plotting NMR curves, crystallizes and is then used as such.

Isotactic PS is a powder with a degree of crystallization of  $\sim 90\%$ . To obtain an amorphous specimen, the powder was melted *in vacuo* at  $230^\circ$  [1] and rapidly cooled in water at  $15^\circ$ . Crystallization was induced by retention at  $160^\circ$  for 5 hours.

NMR spectra were obtained in the form of a derived absorption signal from protons at a frequency of 17 mc/s in the temperature range of  $50\text{--}350^\circ$ , in a laboratory apparatus. Before recording the spectrum, the specimen was held at a given temperature for 20–30 minutes.

\* Vysokomol. soyed. 7: No. 3, 536–538. 1965.

<sup>†</sup> Aromatic polyamide was kindly provided by L. B. Sokolov, G. A. Kuznetsov and V. A. Gerasimov, members of the staff of the Vladimir Scientific Research Institute of Synthetic Resins, for which the authors express their sincere gratitude.