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COMPUTER - AIDED SIMULTANEOUS TA-MS INVESTIGATIONS OF BEAT-RESISTANT POLYSTYRENE

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ABSTRACT

The TA-MS investigations described in this paper have been carried out in quasi-static air and dynamic argon atmosphere. The flow rate of the inert gas streaming has been adjusted to the heating rate of the quartz glass gas inlet system used as an interface between the thermal analyzer and the recipient of the mass spectrometer. The analogously recorded TA data are in good agreement with the computer-aided registered and evaluated MS results. The software package includes, among others, the routines for the acquisition, the storage and the graphic representation of the mass spectra. All programms have always been self designed with regard to the main application aspects, i.e. "easy learn and use".

INTRODUCTION

The chemistry of polymers especially that of polystyrene and related polymer blends has been described in detail as well as the application of various instrumental methods concerned with the degradation of these products (1-3). The applicapability of a simultaneous working TA-MS apparatus in the field of polymer degradation and identification of the decomposition products has been proved very successfully (4,5). In addition these experiments were subjected to matters like fire protection and flame retardance (6).

EXPERIMENTAL

The instrumental equipment consists of a Netzsch STA 429 Thermal Analyzer and a Balzers QMG Quadrupole Mass Spectrometer which is connected by an interface to a Digital Equipment Micro PDP 11/23 + computing system. The software operates on a RT 11 operating system using the Fortran IV programming language. The sample material has been beat-(impact)resistant polystyrene an industrial product.

All experiments have been performed applying the constand instrumental settings, i.E.: heating rate; 5 K/min. DTA measuring range; 0.05 mV, TG measuring range; 25 mg. The pressure in the

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recipient of the mass spectrometer has been 10^{-3} Pa constant.

RESULTS AND DISCUSSION

The TG-, DTG- and DTA-curves of polystyrene (20-25 mg weighed sample) have been recorded in quasi-static air atmosphere and argon streaming. The degradation of the product occurs in a one stage mass loss (100 %). The values of the characteristic temperatures are listed in Table 1.

TABLE 1

Characteristic temperatures of polystyrene (Py) in air and argon

Py(air),	T/K	Py(argon),	T/K
TG onset	580	TG onset	610
DTG,p	662	DTG,p	675
DTA,p,endo	670	DTA,p,endo	675
T fi	675	T fi	690

It can be drawn from the TA data that the protection of the inert gas against the decomposition, i.e. oxidation causes an increase of all characteristic temperature values. The slope of the DTA curves is endothermic in air as well as in argon atmosphere, whereas the results obtained in argon show more endothermic characteristics which are only due to the vaporization of the polymer avoiding oxidation phenomens. The mass spectrum of polystyrene in air at a temperature of 670 K exhibits Fig. 1.

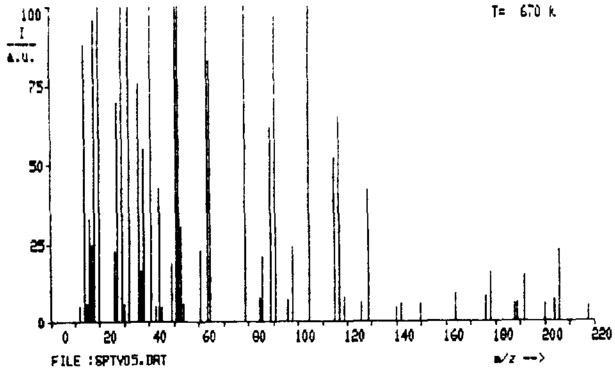

Fig. 1 Mass spectrum of polystyrene in argon atmosphere

The spectrum shows that great portions of benzene (m/z = 78 a.m.u.), protonated styrene (m/z ≈ 105 a.m.u.) naphtalene and tropylium-cation are formed during the vaporization process. The data have been recorded in the temperature range of the maximum reaction rate. No carbon monoxide and carbon dioxide molecules are produced from the sample material in contrast to the experiments carried out in air.

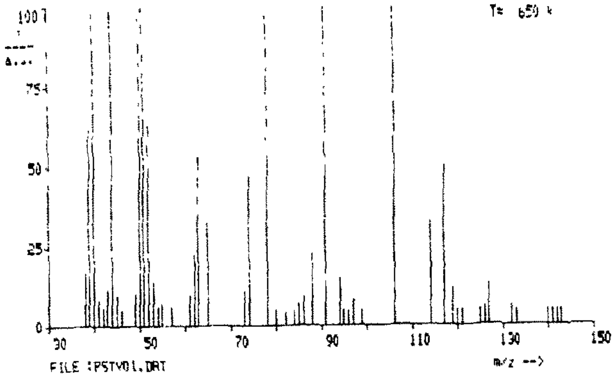

Fig. 2 Mass spectrum of polystyrene in air atmosphere

An important feature of the self designed graphic software applied is the possibility to plot any intensity or sum of intensities of detected ion currents in dependence of the temperature taken from the TA-MS runs. One example which can be compared with the overall recorded mass spectrum shown in Fig.1 is demonstrated in Fig.3.

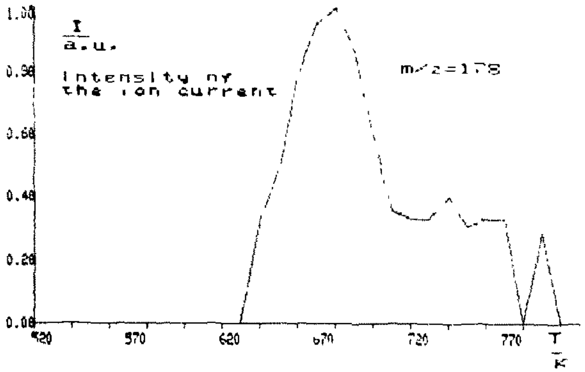

Fig. 3 Intensity of the ion current of the m/z = 178 a.m.u. (phenantrene, anthracene isomers)

In addition the polystyrene material has also been investigated by pyrolysis-gaschromatography which enabled us to identify the a.m. products and determine them quantitatively. The anthracene (phenanthrene) isomers are formed in the temperature range from 630 K to 790 K with a maximum formation (evolution) rate at 670 K.

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