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Short Communication

THERMAL ANALYSIS OF EUGENOL, ISOEUGENOL AND THEIR BENZOIC ACID ESTERS

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Eugenol and isoeugenol were investigated by means of thermal analysis. It was observed that eugenol and isoeugenol have one characteristic endothermic effect at 260° and 290°C, respectively, and two exothermic effects in the temperature intervals 270°–590°C. The DTA curves of the benzoyl esters of eugenol and isoeugenol begin with endothermic effects at 70° and 95°C, respectively, which coincide with the melting points of these compounds.

Introduction

The thermal analysis and more especially the thermogravimetry of organic compounds is widely used for their study [1]. Korshak [2] suggested that thermal stability should be characterized by the temperature at which thermochemical changes appear in the structure of the organic compound under study. These changes are normally reflected by changes in the properties of the compound.

During the past 10–15 years, thermal analysis has been more frequently and successfully used in the investigation of different types of drugs and pharmaceutical preparations. Dubler and Kamber [3] carried out DTA and TG studies of different biogenic compounds and their individual components (cholesterol, potassium salts of fatty acids etc.). Warne and Blazejowski [4] studied the thermal destruction of the hydrochlorides of some 2-aminohydroxy acids and their esters. Nakamura *et al.* [5] investigated

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the thermal behaviour of the diethyl ester of poly-*p*-phenylenediacrylic acid.

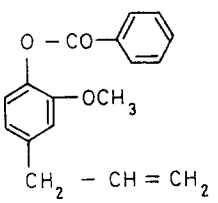
Experimental

The present investigations were carried out on eugenol and isoeugenol with the following constants: *b.p.* = 253.1° and 253.4°C, respectively, and refractive indices $n_D^{20} = 1.5410$ and 1.5726, respectively.

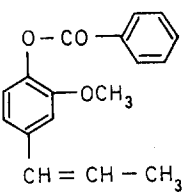
Esters of eugenol and isoeugenol were obtained through the acylation of benzoyl chloride according to the following scheme:

$$R'OH + RCOCl \xrightarrow{NaOH} RCOOR' + HCl$$

The benzoate of eugenol /I/ has *m.p.* = 67°–69°C and that of isoeugenol /II/ has *m.p.* = 90°–93°C.



I



II

These esters have the molecular formula $C_{17}H_{16}O_3$ and a molecular mass of 268. Their structures were analysed by means of IR, NMR and mass-spectroscopy.

An OD–102 derivatograph (MOM, Hungary) was used in the investigations. The sample mass was about 200 mg.

Differential thermal and thermogravimetric curves were recorded under the following conditions: heating rate: 9 deg/min; reference material: aluminium oxide; atmosphere: nitrogen. Figure 1 gives the DTA and TG curves obtained in the thermal analysis of eugenol and isoeugenol, and Fig. 2 the corresponding curves of their benzoyl esters.

A characteristic endothermic effect was observed for eugenol and isoeugenol at 260° and 290°C, respectively, followed by two exothermic effects with maxima at 270° or 275° and at 520° or 590°C, respectively. Significant changes in the mass of eugenol began after 50°C, and in that of isoeugenol immediately after 150°C. These changes continued up to 290°

and 300°C, leading to the characteristic endothermic effect mentioned earlier.

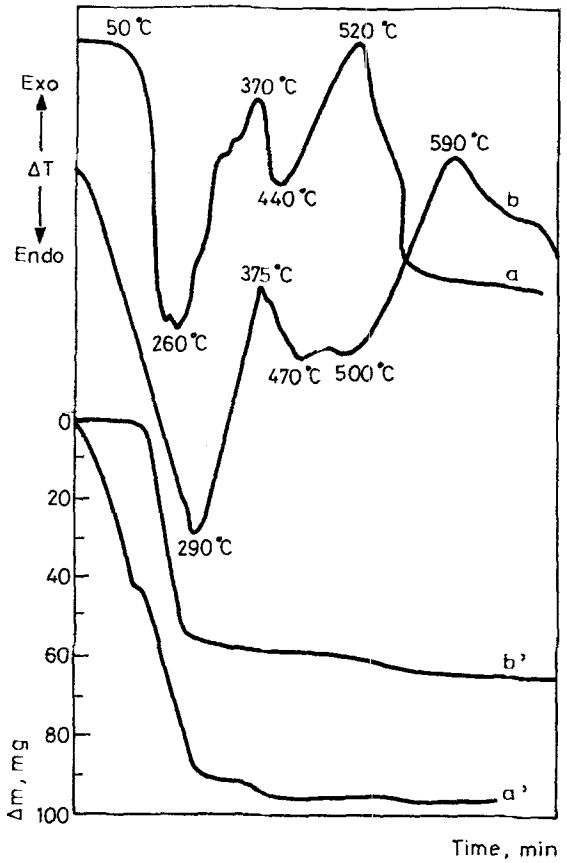


Fig. 1 DTA- and TG-curves of eugenol (a, a') and isoeugenol (b, b')

For the benzoyl esters of eugenol and isoeugenol, a characteristic endothermic effect was observed at 70° and 95°C, respectively (Fig. 2, curves *a* and *b*), which almost coincide with the temperature intervals in which these compounds melt (67°–69°C and 90°–93°C, respectively). Thermal destruction began after 140° and 170°C, respectively, with an endothermic effect at 370°C, associated with the liberation of about 60% of the molecular mass in the form of volatile products. Further endo- and exothermic effects were observed in the interval between 400° and 700°C.

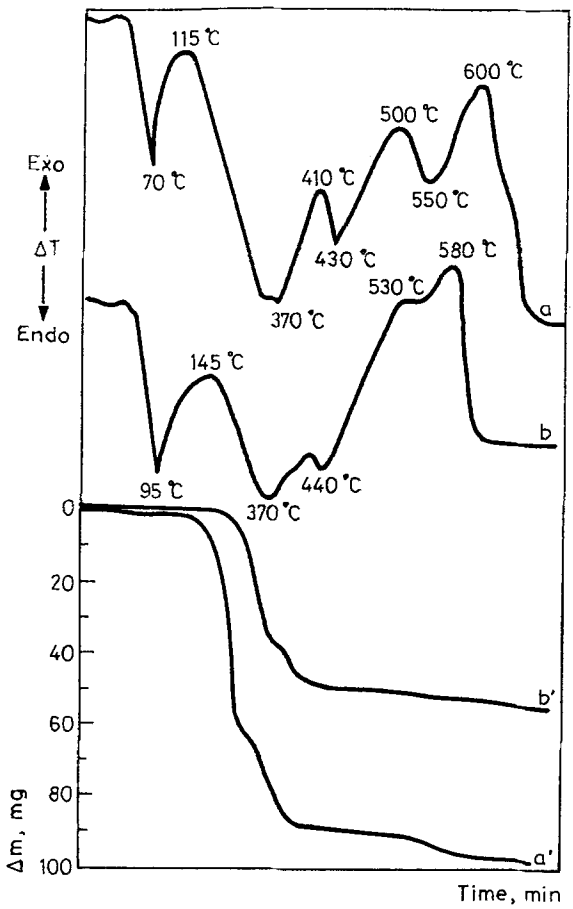


Fig. 2 DTA- and TG-curves of eugenol's (a, a') and isoeugenol's (b, b') bensoyl esters

Conclusions

1. Eugenol and isoeugenol have a characteristic endothermic effect at 260° and 290°C, respectively, and two exothermic effects in the temperature intervals 270°–275°C and 520°–590°C.

2. The DTA curves of the benzoyl esters of eugenol and isoeugenol begin with endothermic effects at 70° and 95°C, respectively, which coincide with the melting points of these compounds. Intensive thermal destruction of the esters starts at 140° and 170°C, respectively, and leads to significant endothermic effects at 370°C, at which 60% of the mass is liberated in the form of gaseous pyrolytic products.

References

1 S. S. Pavlova, I. V. Guravleva and U. I. Tolchinskii, Thermal analysis of organic compounds and highmolecular compounds, Moscow, Chemistry, 1983.
2 V. V. Korshak, Thermostable Polimers, Moscow, Science 1969.
3 E. Dubler and B. Kamber, Thermal Analyses, ICTA 80, Bd. 2, Birkhäuser, Stuttgart 1980, p. 531.
4 Z. Warne and J. Blazejowski, Thermochim. Acta, 93 (1985) 5.
5 S. Nakamura, K. Nakamura and Y. Seagusa, Thermochim. Acta, 93 (1985) 9.