

MCC-33 (Univ. Zagreb)

Master of Science Thesis

Croat. Chem. Acta 41 (1969)

Separation of High Boiling Isomeric Compounds by Preparative Gas Chromatography. Isolation and Identification of α -methylstyrene Dimerisation Products

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The dimerization of α -methylstyrene in sulphuric acid results in a mixt. of dimeric isomers, the composition of which is not well known. The gas chromatographic analysis of this mixture by Aitken gave four incompletely sepd. peaks, but it was not known which dimers gave rise to particular peaks.

In this work the sepn. as well as identification of α -methylstyrene dimers by gas chromatographic methods was achieved. The complete sepn. of the dimeric products is obtained on Apiezon L column (1/8" \times 6 ft, column temp. 200° C, carrier gas nitrogen). Six well sepd. peaks were obtained in the dimeric fraction. The isolation of each component was carried out on the preparative gas chromatographic column (3/8" \times 50 ft) with silicon rubber SE 30 on Chromosorb A and nitrogen as carrier gas at 240° C. The structure of the isolated compounds was detd. by means of KOVATS' retention indices. The indices increments ΔI and δI for investigated and model compds. were detd. on monopolar SE 30 and Apiezon L columns and polar Emulphor O column. On the basis of obtained results the compn. of α -methylstyrene dimerization products is as follows: (1) *cis*-2,4-diphenyl-4-methyl-2-pentene (?), (2) 1,1,3-trimethyl-3-phenylindane, (3) 2,4-diphenyl-4-methyl-1-pentene, (4) *trans* (?) 2,4-diphenyl-4-methyl-2-pentene, (5) ketone, aldehyde or ester, (6) ether.

The analysis of each component by N. M. R. spectrometry confirmed these results. The component (5) was identified by ir., uv., and org. microanalysis as 1,3-diphenyl-3-methyl-2-butanone. The further identification of component (6) failed due to the instability of this compound.

The thesis was reported in part at 1st Yugoslav Symposium on Gas Chromatography, Zagreb, April 10–12, 1968, and partly published: *J. Chromatog.* 38 (1968) 326, *Chimia* 22 (1968) 434.

Examiners: Dr. Dj. Deur-Siftar, Prof. S. Borčić, and Prof. S. Ašperger.

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1. Separation of High Boiling Isomeric Compounds by Preparative Gas Chromatography. Isolation and Identification of α -methylstyrene Dimerisation Products

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Gas chromatography, preparative scale
 α -Methylstyrene dimers, separation, identification
Retention indices

Y 2408

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