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Short Communications

Δ^3-Carene and (+)-Limonene Mixtures

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In a previous communication, Widmark and Blohm [1] have demonstrated the usefulness of an analytical micro-sorption method devised by Blohm [2], when studying terpene mixtures. In the above mentioned paper [1] it was shown that the refractive indices of Δ^3-carene and (+)-limonene, n_D^{25} = 1.4700 and 1.4703, respectively, were too close to allow good readings of the sorptograms of mixtures of these two pure terpenes.

This paper demonstrates that this limitation of the sorption method can be overcome by measuring in a microtube the optical rotations of 0.4 ml ethanolic solutions made up from the 5 μl fractions obtained from the sorption. This method is of course limited to compounds possessing marked differences in their optical activities, *e.g.* (+)-limonene, $[\alpha]_D^{25}$ = +123, and Δ-carene, $[\alpha]_D^{25}$ = +17, and is found to be less accurate and much more laborious than the ordinary way of reading the refractive indices.

Experimental part. The Δ^3-carene and (+)-limonene were purified to index homogeneity according to Widmark [3,4]. The mixtures were prepared by weighing *ca.* 0.4 g samples to form five solutions containing 5, 10, 50, 75 and 90 % (+)-limonene in Δ^3-carene. 40 μl lots of these solutions were pipetted into the top of a 250 mm high column (i Ø 1.4 mm) of activated silica gel [2]. The samples were displaced with ethanol at 1 atm. pressure in the ordinary way [2,1] and eight 5.0 μl fractions were collected in capillaries. The fractions were transferred to 0.40 ml quantities of ethanol in small test tubes. The capillaries were rinsed by sucking the solutions up and down several times. The optical rotations of the solutions were measured in a 5 cm microtube (Hilger Co., M 289, volume 0.35 ml) and the mean value of 10 readings was taken. The results are given in Fig. 1.

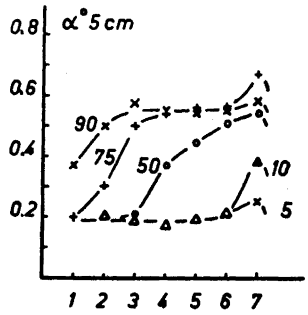

Fig. 1. Sorptograms of Δ^3-carene — (+)-limonene mixtures indicated by optical rotation. Percent(+)-limonene is given.

1. Widmark, G. and Blohm, S.-G. *Acta Chem. Scand.* **9** (1955) 1296.
2. Blohm, S.-G. *Arkiv Kemi* **6** (1954) 317.
3. Widmark, G. *Acta Chem. Scand.* **9** (1955) 925.
4. Widmark, G. *Acta Chem. Scand.* **9** (1955) 938.

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