Journal of Chromatography, 250 (1982) 141–142
Elsevier Scientific Publishing Company, Amsterdam — Printed in The Netherlands

CHROM. 15,228

Note

Preparative high-performance liquid chromatography of adamantane-2,4-diols

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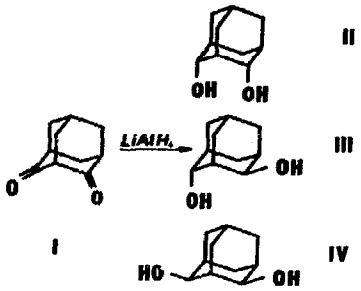
(Received July 26th, 1982)

A mixture of three stereoisomeric adamantane-2,4-diols, *i.e.*, adamantane-2a,4a-diol (II), adamantane-2e,4a-diol (III) and adamantane-2e,4e-diol (IV), was prepared previously[1] by reduction of adamantane-2,4-dione (I) with $LiAlH_4$. This note describes the separation of these three stereoisomers by preparative high-performance liquid chromatography (HPLC).



EXPERIMENTAL

The separation was performed on Chromatospac Prep 100 preparative chromatograph (Jobin Yvon, Longjumeau, France). A 100-g amount of octadecyl silica of irregular shape (particle size 10–20 μm) was packed into a column of 40 mm I.D.; the height of the bed was 170 mm. The silica was obtained by the method described previously[2]. The chemically bonded phase was prepared by the method of Halász and Sebastian[3] as modified by Hemetsberger *et al.*[4], using octadecyltrichlorosilane as a reagent and toluene with pyridine as the reaction medium. No further "capping" has been done.

Methanol–water (50:50, w/w) was used as the mobile phase; it was degassed by connecting its reservoir to a vacuum for about 15 min. The flow-rate of the mobile phase was 14 ml/min at a pressure of 800 kPa. A slurry of stationary phase and methanol was used for packing of the column. The sample (0.3 g in 2 ml of methanol) was introduced directly into the column by an injection syringe.

Detection was effected with a refractive index (RI) detector (Varian, Palo Alto, CA, U.S.A.).

0021-9673/82/0000–0000/$02.75

HPLC analysis of fractions obtained by preparative separation was carried out on a Varian 8500 instrument equipped with an RI detector. Column: Micropak CH-10, 250 × 2 mm I.D., packed with octadecyl silica, particle size 10 μm (Varian). The flow-rate of the mobile phase, methanol–water (20:80, w/w), was 10 ml/h.

RESULTS AND DISCUSSION

A preparative chromatogram of the separated stereoisomeric adamantane-2,4-diols, together with analytical chromatograms of fractions obtained, is presented in Fig. 1. The chromatogram in Fig. 2 shows the analytical separation of all three stereoisomers. The conditions for the preparative separation were chosen in accordance with the analytical results published previously[5].

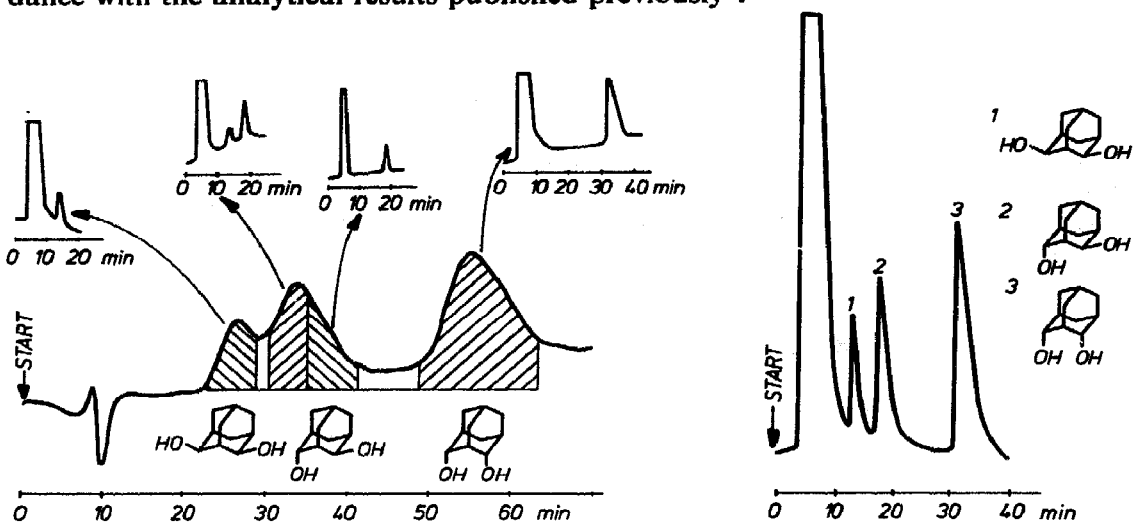


Fig. 1. Preparative separation of adamantane-2,4-diols together with analytical chromatograms of the separated fractions.

Fig. 2. Analytical HPLC separation of stereoisomeric adamantane-2,4-diols.

Owing to the small particle size and high viscosity of the mobile phase used, it was not possible to pack as long a column as when a mobile phase of lower viscosity was used[2]. The purity of all separated isomers was higher than 97%. The yield of the second eluted peak (*i.e.*, 2e,4a-diol) was lower. It had to be collected after the maximum of the peak.

When comparing the described method of separation of these compounds with classical column chromatography on silica, it is evident that this method is quicker and more efficient.

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