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Note

Chromatographic resolution

XXI*. Direct optical resolution of abscisic acid by high-performance liquid chromatography on cellulose tris(3,5-dimethylphenylcarbamate)

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Abscisic acid (ABA) is an important plant hormone which appears to be involved in plant growth and development[1]. Enantiomers of ABA are catabolized in different manners in plants, and naturally occurring ABA is the (+)-(*S*)-isomer. Therefore, the ready availability of this isomer is highly desirable for biological studies.

Optical resolution of commercially available racemic ABA and its methyl ester by liquid chromatography has been attempted by several groups[2–6], and efficient complete resolution of the ester[6] has recently been achieved by high-performance liquid chromatography (HPLC) using cellulose tris(3,5-dimethylphenylcarbamate) as a chiral stationary phase, which we developed[7].

Quite recently, we found that direct optical resolution of racemic carboxylic acids is possible on the above chiral column using hexane–2-propanol containing a small amount of a strong acid like formic acid, trichloroacetic acid or trifluoroacetic acid[8]. In this note, we report a very efficient direct optical resolution of ABA on cellulose tris(3,5-dimethylphenylcarbamate). Direct resolution is more valuable than the resolution of ABA esters in several respects.

EXPERIMENTAL

Cellulose tris(3,5-dimethylphenylcarbamate), prepared by the reaction of cellulose and 3,5-dimethylphenylisocyanate, was coated on the macroporous silica gel LiChrospher SI 4000 treated with 3-aminopropyltriethoxysilane[7]. This packing material was packed in a column (25 cm × 0.46 cm I.D.) by a slurry method. Chromatographic resolution was performed on a Jasco Trirotar-II chromatograph equipped with UV (240 nm) and polarimetric detection (mercury, full lamp).

* For Part XX, see Y. Okamoto, R. Aburatani, K. Hatano and K. Hatada, *J. Liq. Chromatogr.*, in press.

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RESULTS

Fig. 1 shows the optical resolution of (±)-ABA on a cellulose tris (3,5-dimethylphenylcarbamate) column. Complete direct optical resolution was achieved by using hexane–2-propanol containing 1% trifluoroacetic acid. The polarimetric detector showed that the (+)- and (−)-isomers were eluted at about 9 and 13 min, respectively. Without trifluoroacetic acid in the eluting system, ABA was not eluted from the column, like other racemic acids[8]. The elution order of ABA seems to be the same as that of ABA methyl ester[6]. The separation coefficient α, was 2.14, comparable to that of the methyl ester. Preparative separation was also possible. On the present analytical column, about 1 mg of ABA was completely resolved in one injection.

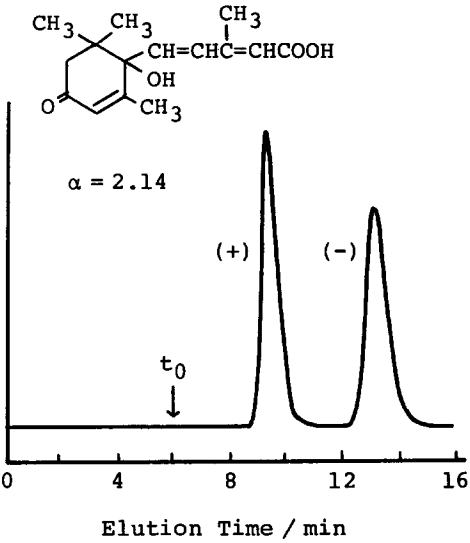

Fig. 1. Optical resolution of abscisic acid on a cellulose tris(3,5-dimethylphenylcarbamate) column with hexane–2-propanol–trifluoroacetic acid (80:20:1), at 0.5 ml min^{-1}.

By the use of the cellulose tris(3,5-dimethylphenylcarbamate) and hexane–2-propanol–trifluoroacetic acid (80:20:1), ABA was directly separated. This method may be useful for obtaining optical isomers of ABA as well as for determining the optical purity of ABA.

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