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Note

High-performance liquid chromatographic method for the analysis of alkene bromohydrins and iodohydrins

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A gas chromatographic method for the analysis of the halohydrins (chlorohydrin, bromohydrin and iodohydrin) of ethylene and propylene was previously described¹. Aside from that report, there seems to be no other reported chromatographic method for the analysis of this class of compounds.

We now wish to describe a convenient, rapid high-performance liquid chromatographic (HPLC) method for the analysis of the bromohydrins and iodohydrins of ethylene, propylene, butene and butadiene.

EXPERIMENTAL

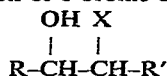
Reagents and standards

Bromohydrins and iodohydrins were purchased or prepared as described in Table I. All standards were 5 mg/ml (for each component) aqueous solutions. Methanol was HPLC grade obtained from Burdick and Jackson Labs., Muskegon, MI, U.S.A. Water was deionized and Milli-Q treated.

TABLE I

SOURCE OF BROMOHYDRINS AND IODOHYDRINS USED IN THIS STUDY

A = Purchased from Aldrich, Milwaukee, WI, U.S.A. B = A 20% impurity in 1-bromo-2-propanol. C = Synthesized by the reaction of 1-bromo-2-propanol and iodide². D = Co-produced in the reaction of 1-bromo-2-propanol (containing 20% 2-bromo-1-propanol) and iodide.



Compound name	R	R'	X	Source
2-Bromoethanol	H	H	Br	A
1-Bromo-2-propanol	CH ₃	H	Br	A
2-Bromo-1-propanol	H	CH ₃	Br	B
2,3-Dibromo-1-propanol	H	CH ₂ Br	Br	A
1-Bromo-3-butene-2-ol	CH ₂ =CH	H	Br	A
1,4-Dibromo-2-butanol	BrCH ₂ CH ₂	H	Br	A
2-Iodoethanol	H	H	I	A
1-Iodo-2-propanol	CH ₃	H	I	C
2-Iodo-1-propanol	H	CH ₃	I	D

High-performance liquid chromatography

A Spectra-Physics SP8000 high-performance liquid chromatograph equipped with a variable-wavelength ultraviolet (UV) detector was used. Detector attenuation was 0.1 a.u.f.s., set at wavelength 208 nm for bromohydrins and at wavelength 259 nm for iodohydrins. Standards (10 μ l) were injected onto a LiChrosorb RP-18 reversed-phase column (Brownlee Labs., Santa Clara, CA, U.S.A.; 10 μ m; 25 cm \times 4.6 mm). The mobile phase (water-methanol, 9:1) was set at a flow-rate of 2.0 ml/min. Column temperature was 25°C.

RESULTS AND DISCUSSION

Figs. 1 and 2 present the separations obtained with the described method. The anticipated order of elution was obtained: $C_2 > C_3 > C_4$, for a given halohydrin; C_n bromohydrin $>$ C_n iodohydrin.

Positional isomers exist for C_3 and larger halohydrins. Fig. 1 shows that the two isomers of propylene bromohydrin (1-bromo-2-propanol and 2-bromo-1-propanol

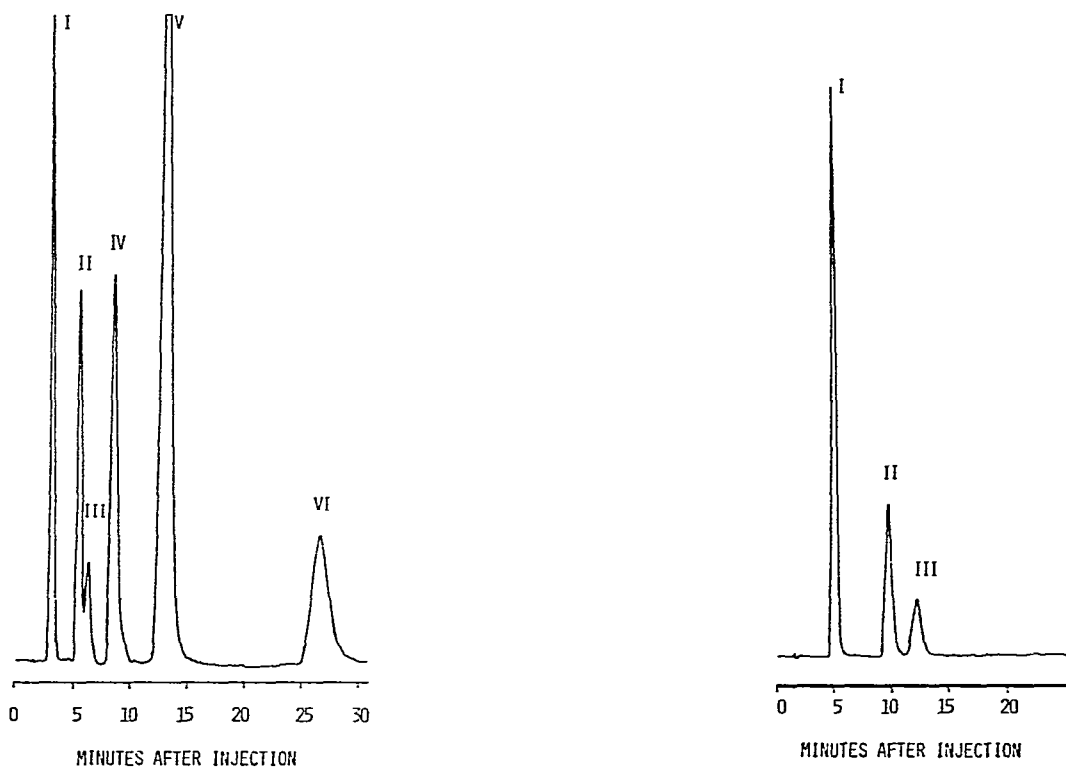


Fig. 1. HPLC separation of alkene bromohydrins, detected at wavelength 208 nm. Peaks: I = 2-bromoethanol; II = 1-bromo-2-propanol; III = 2-bromo-1-propanol; IV = 1-bromo-3-butene-2-ol; V = 2,3-dibromo-1-propanol; VI = 1,4-dibromo-2-butanol.

Fig. 2. HPLC separation of alkene iodohydrins, detected at wavelength 259 nm. Peaks: I = 2-iodoethanol; II = 1-iodo-2-propanol; III = 2-iodo-1-propanol.

noI) are partially resolved. Fig. 2 shows that the two isomers of propylene iodohydrin (1-iodo-2-propanol and 2-iodo-1-propanol) are baseline resolved. These isomers of propylene iodohydrin were poorly resolved on the previously reported gas chromatographic method¹.

By selecting the wavelength of detection, compounds containing organic-bound bromine (C-Br absorption, $\lambda_{\text{max.}} \approx 208$ nm) or organic-bound iodine (C-I, $\lambda_{\text{max.}} \approx 259$ nm) are readily detected. Organic-bound chlorine compounds (C-Cl, $\lambda_{\text{max.}} < 190$ nm) are not detectable with the UV detector.

REFERENCES

- 1 J. Geigert, B. M. Panschar and S. L. Neidleman, *J. Chromatogr.*, 205 (1981) 165.
- 2 C. A. Stewart and C. A. VanderWerf, *J. Amer. Chem. Soc.*, 76 (1954) 1259.