

CHROM. 11,255

Note

Simple and rapid determination of epichlorohydrin at the lower parts per billion* level by gas chromatography–mass fragmentography

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(Received May 26th, 1978)

It has recently been supposed¹ that epichlorohydrin (EPCH**; 1-chloro-2,3-epoxypropane) may be a mutagenic compound. A possible route by which EPCH may reach foodstuffs is via the packaging material. Epoxy resins are widely used as coatings for cans employed for packaging of food, and large containers for milk and juices are often coated with epoxy resins; one of the compounds used in the manufacture of the epoxy resins is EPCH. Residual epichlorohydrin may then be present in the can coating and migrate into the beer, juices, etc., contained in the can.

The method described here for the determination of EPCH is based on mass fragmentography, which provides the highest sensitivity of detection with high specificity. The determination of the fragment of *m/e* 49 in diethyl ether is possible at the level of 6 ppb.

EXPERIMENTAL AND RESULTS

A Finnigan 4000 gas chromatograph–mass spectrometer was equipped with a 1.5 m × 0.3 mm I.D. glass column, packed with 80–100-mesh Carbopack C (Supelco, Bellefonte, Pa., U.S.A.) loaded with 0.2% of Carbowax 1500. The carrier gas (helium) had a flow-rate of *ca.* 20 ml/min. The gas chromatograph was connected with the mass spectrometer through an all-glass separator. A vacuum diverter system was used to prevent the solvent contaminating the source of the mass spectrometer, which was used in the electron-impact mode with the multiple ion detection unit tuned at *m/e* 49.

Samples of 1 or 5 μ l were injected at 100° (isothermal) and the signals were measured on a Rikadeni recorder with a chart speed of 10 mm/min. Standard solutions of EPCH in diethyl ether were prepared.

Fig. 1 shows a mass fragmentogram of the blank diethyl ether and of standards containing 6, 12 and 30 ppb of EPCH. Fig. 2 is a calibration graph for these low contents of EPCH in diethyl ether, obtained using standard solutions with concentrations of 6, 12, 30, 72 and 96 ppb of EPCH.

* Throughout this article, the American billion (10⁹) is meant.

** It is common to use the abbreviation ECH for epichlorohydrin in the polymer industry. However this may lead to confusion, because ECH has also been used for chloroethanol in the literature². Therefore, epichlorohydrin is abbreviated to EPCH in this paper.

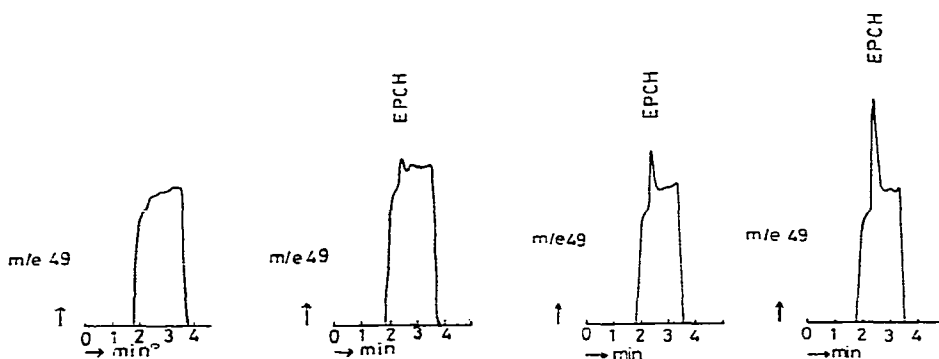


Fig. 1. Mass fragmentogram at m/e 49 of a blank of diethyl ether and of diethyl ether containing 6, 12 and 30 ppb of EPCH.

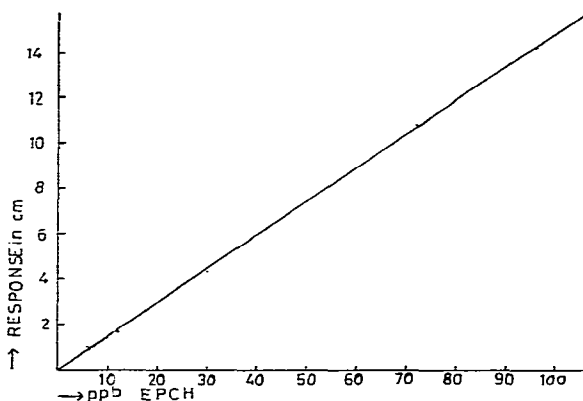


Fig. 2. Calibration graph for diethyl ether solution containing 6, 12, 30, 72 and 96 ppb of EPCH.

With standard solutions with EPCH concentrations of 10 and 20 ppb in water we established that after three extractions with diethyl ether after addition of sodium chloride the extraction of EPCH was quantitative.

Under standard conditions for migration³ of immersion at 40° for 10 days, three types of cans and four sheets coated with epoxy resins were investigated for the migration of EPCH into water. The resulting aqueous solutions were extracted with diethyl ether, using 50 ml of ether for 100 ml of solution, and the extracts examined by the method described. No EPCH was found in the extracts.

On the basis of these results it is possible to conclude that the maximum amount of EPCH that could have migrated is less than 3 ppb into the water.

REFERENCES

- 1 M. Kucerova, V. S. Zhurkov, Z. Polivkova and J. E. Ivanova, *Mutat. Res.*, 48 (1977) 355.
- 2 T. Stijve, R. Kalsbach and G. Eyring, *Trav. Chim. Aliment. Hyg.*, 67 (1976) 403.
- 3 *Ontwerp Verpakkingen en Verbruiksartikelenbesluit Warenwet, Bijlage Deel B, Methoden van Onderzoek*, Ministerie van Volksgezondheid en Milieuhygiëne, The Hague, 1977.