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Letter to the Editor

Applied headspace gas chromatography

Sir,

J. Novák's review of the book "*Applied Headspace Gas Chromatography*", edited by B. Kolb [*J. Chromatogr.*, 209 (1981) 494] included misleading remarks about a new technique for collecting headspace volatiles. This technique, which was developed by A. Rapp and W. Knipser, is discussed in Chapter 8 of the book under review.

Rapp and Knipser's method is new only in the trapping and concentration of headspace volatiles. The method of sweeping volatiles from a sample by gas extraction, as used traditionally in headspace methods, is unaltered. Thus the reviewer's question "One might ask why the authors did not extract the wine directly with Freon 11", suggests a fundamental lack of understanding of the difference in composition of volatiles obtained by headspace analysis and by simply solvent extracting a sample. Such differences, in the specific case of wine volatiles, have been discussed in the literature[1].

The method of Rapp and Knipser involves a three-phase partitioning of entrained headspace volatiles between the extracting gas, Freon 11, and an aqueous phase. Because of the limited solubility of ethanol in Freon 11, when partitioned against water[2], most of the unwanted ethanol goes into the aqueous phase. But most importantly, the volatile headspace components are efficiently trapped in the Freon F11 free from water and ethanol. Rapp and Knipser have elsewhere[3] discussed the precision and trapping efficiency of their technique.

The method has been used in our own laboratories for almost three years and in this time several hundred headspace analyses have been carried out. We have abandoned older methods of concentrating headspace volatiles on porous resins and this says much for the Rapp and Knipser technique, since we had earlier devoted considerable time to the development of a porous resin method for our research[4]. Reasons for adopting the new method include: (a) simplicity of both apparatus and technique, (b) several gas chromatographic analyses of the same headspace collection can be made, (c) there are no problems with selectivity of porous resin adsorbents distorting the composition of collected headspace volatiles[5], (d) there are no problems with artefacts from the decomposition of porous resins generated during thermal desorption of volatiles[6]. In our hands the technique has proved valuable not only for the analysis of volatiles from wines and other alcoholic beverages but also for fruit juices and plant tissue homogenates.

It is my hope in writing this to correct a wrong impression given of a new technique and to encourage all those interested in headspace analysis to look again at the Rapp and Knipser method. It is the first major advance in headspace trapping and concentration since Jennings *et al.*[7] introduced the porous resin technique in 1972.

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