Erroneous Peaks from Porapak-Q Traps

Temperatures of 170°C and above used to remove adsorbed volatiles from Porapak-Q traps can produce artifacts. Erroneous peaks were detected with high-sensitive gas chromatographic analysis when Porapak-Q alone was heated at 170°C; peaks were quite small or negligible when temperatures of 150°C and below were used.

The simple on-column trapping procedure for gas chromatographic analysis of flavor volatiles developed by Morgan and Day (1965) has found wide use among flavor chemists. Water often interferes when this technique is coupled with mass spectral analysis or when trapping is done in a capillary tube. Organic compounds have a long retention time compared to water on columns packed with porous polyaromatic polymers such as Porapak-Q (Ethylvinylbenzene–divinyl benzene copolymer; Waters Associates, Inc., Framingham, Mass.). Several workers have recently utilized the unique property of these polymers to eliminate water from samples [Andersson and von Sydow (1970); Dandoy and Delvaux (1971); Dravnieks and O'Donnell (1971); Schultz *et al.* (1971); Richard *et al.* (1970)].

Unloading Porapak-Q at 170°C and above may lead to erroneous peaks when the glc analysis is done at a sensitivity that will detect nanogram quantities and for a duration of time (15 min) used to elute trapped volatiles.

EXPERIMENTAL

An unused, acetone-washed Porapak-Q trap (1/4-in. o.d. × 4 in.) was conditioned by heating at 180°C for 24 hr and 80°C for 72 hr with a flow rate of 30 ml of nitrogen per minute. The conditioned trap was unloaded for 15 min into a 0.03-in. i.d. × 10-in. stainless steel capillary trap submerged in a Dry Ice–ethylene glycol bath. Unloading temperatures of 140, 150, 170, and 190°C were used with a flow rate of 15 ml per min of nitrogen. The trappings were transferred to a 500-ft × 0.03-in. i.d. strainless steel, Carbowax 20M capillary column for glc analysis according to the procedure reported by Scanlan *et al.* (1968). The glc conditions were: flow rate, 12 ml of nitrogen per min at 24°C; injector temperature, 200°C; flame ionization temperature, 210°C; column temperature, 70°C for 5 min, then programmed at 190°C at 1.1° per min.

RESULTS

Data in Figure 1 indicate the presence of erroneous peaks originating from the Porapak-Q trap when unloading temperatures were 170°C and above. These compounds were produced either by heat degradation of the Porapak-Q or inadequate conditioning procedures. The latter seemed less likely since repeated unloading of the same trap at high temperatures did not diminish the number or intensity of the peaks detected. The severity of this problem is directly related to the sensitivity of the glc analysis.

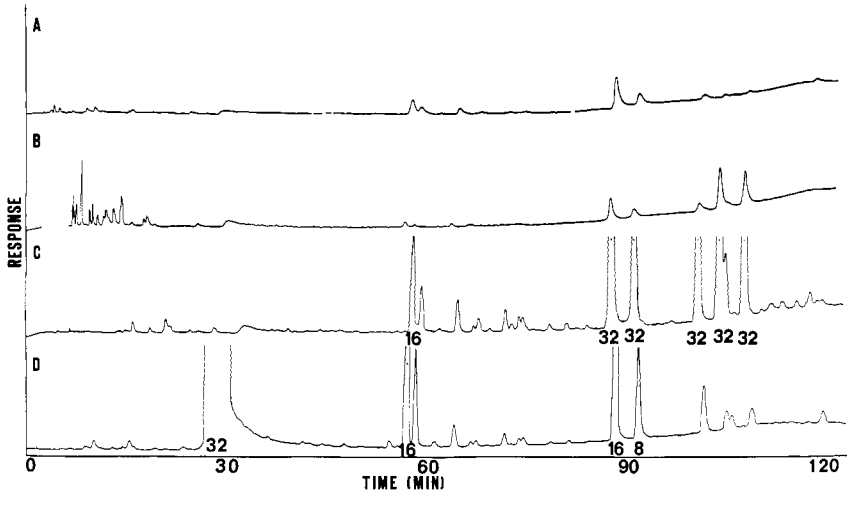

Figure 1. Chromatograms obtained from an unused, acetone-washed, conditioned Porapak-Q trap unloaded at 140 (A), 150 (B), 170 (C), and 190°C (D). Numbers below peaks indicate attenuation. Ten nanograms of dimethyl disulfide gave a peak responce equivalent to attenuation of 8

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