CHROM. 3778

Deposits on flame ionization detectors with silicone gum rubber columns

During the preparative gas chromatography of some organophosphorus esters using a silicone gum rubber column, a copious white deposit was formed on the collector electrode of the flame ionization detector over a period of 2 weeks. No similar deposits have been noticed on the detector when used with "Reoplex" (polypropylene glycol adipate) and poly-*m*-phenyl ether columns under similar conditions.

In addition a saucer-shaped depression was formed on the inner surface of each silicone rubber injection port septum used. White material in the depression crumbled very readily although the remainder of the septum retained its initial appearance and flexibility.

The gas chromatograph used was an F & M 776 Prepmaster Jr. with a flame ionization detector. The column was 80 in. of ¾ in. O.D. stainless steel packed with 20% W 98 on 60–80 Chromosorb P. Column temperatures used ranged from 100° to 140°; the injection port and detector block were maintained at 220°.

Although the deposits from both detector and injection port appeared similar to the naked eye, analysis by the Debye-Scherrer X-ray diffraction method and by optical emission spectroscopic techniques showed that the detector deposits were mainly silicon phosphate ($2\ SiO_2.P_2O_5$), and the injection port samples were alpha cristobalite (SiO_2).

Silicon phosphate formation could occur at the very high temperature of the hydrogen-air flame as phosphorus compounds elute in the presence of silicone vapour due to column bleed. Bleeding off of the stationary phase was confirmed by a marked decrease in retention times of the injected samples.

SPEAKMAN[1] in a paper on limitations set by column bleed using the flame ionization detector, mentioned the formation of white deposits on the collector electrode when he was using SE 30 columns but did not identify them. Since he was studying column bleed only and no samples were injected, the deposits were probably SiO_2.

The formation of cristobalite is probably due to the action of the hot (up to 220°) vapours, from volatilization of the phosphorus esters, on the silicone rubber septa. Two alternatives seem possible for the origin of the SiO_2; either liberation of the SiO_2 used as a filler in the preparation of the silicone rubber or oxidation of the silicone rubber itself. Temperature appears to be critical since a septum placed in refluxing triethyl phosphite (b.p. 160°) for six hours was unaffected.

Australian Defence Scientific Service,
Defence Standards Laboratories, Maribyrnong, Victoria 3032
(Australia)

B. R. LAKELAND
I. T. MCDERMOTT

1 F. P. SPEAKMAN, *Column*, Vol. 1, No. 3 (1966) 9.

Received September 2nd, 1968