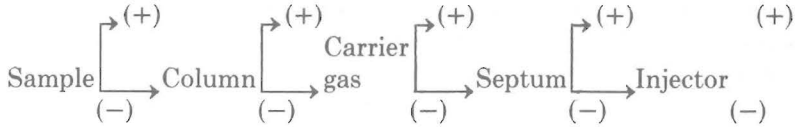
Research Note

GHOST PEAKS TROUBLE-SHOOTING GUIDE[1]

A stepwise procedure has been established and followed in our laboratory to determine the source of various ghost peaks which sometimes appear at the end of chromatograms. After each step, observations are made to determine whether, with the change introduced, the ghost peaks remain (−) or disappear (+). If the ghost peaks are still present, the following step in the scheme is followed:

Stepwise procedure



Three different aliquots of the same sample are analyzed under our laboratory conditions.[2] Before each injection the syringe is washed 20 times with distilled water, followed by heat and vacuum that vaporize and remove residues simultaneously. If identical results as well as same ghost peaks are obtained, distilled water is injected in the chromatograph. If ghost peaks appear with the same retention time as before, then air is injected. The appearance of ghost peaks indicates that they do not come from the sample being analyzed.

Different columns filled with the same packing are used to analyze distilled water. If ghost peaks appear, column packing is changed to Porapack Q, and distilled water is again analyzed. If ghost peaks persist a similar unpacked stainless steel tube is used as column to analyze distilled water. If the peaks appear, packing, as well as interaction between packing and solvent, is discarded as the source of the ghost peaks.[3]

Organic impurities present in the carrier gas will produce noise and peaks[4,5,6] and a hydrogen rich flame can detect the presence of inorganic

[1] Manuscript submitted to Editorial Board November 8, 1982.

[2] Gas chromatograph Hewlett-Packard Model No. 5750. A Carbowax 20M 5% on Chromosorb W AW 60–80 mesh column. Injector and detector temperature; 200° C. Helium at 25 ml/min. Flame ionization detector with air and hydrogen flows of 280 and 30 ml/min respectively. Initial column temperature 50° C. Final column temperature; 160° C. Temperature program rate; 15° C/min.

[3] Bayer, L. F., 1980. GC column conditioning, J. Chromatogr. Sci. 18 (12): 684–85.

[4] Scott specialty gases, 1982. Gas chromatography: detectors, gases, delivery systems, what are the variables?, Scott, Tech. Newsline 4: 4–6.

[5] Blades, A. T., 1976. The effect of the carrier gas on the flame ionization detector sensitivity, J. Chromatogr. Sci. 14 (1): 45–58.

[6] Schaefer, B. A., 1978. Thermal contributions to the response of the flame ionization detector, J. Chromatogr. Sci. 16 (5): 211–17.

gases too.[7,8] For checking whether the carrier gas is responsible for ghost peaks, another carrier gas such as nitrogen was used to analyze distilled water and air. If ghost peaks continue to show up in the chromatograms, their presence is independent of the carrier gas used.

Routine laboratory procedure requires that the septum be changed at the beginning of the day and used throughout that day. To determine whether the septum is related to the ghost peaks, a metal cap is prepared to block the entrance of the injection port, permitting the removal of the septum and preventing air and carrier gas leakages.

With the injection port sealed with this metal cap, blank runs without sample injection can be done. The presence of the peaks indicates that the septum is not responsible for the extra signal.

After eliminating sample impurities, column and carrier gases as possible sources of the problem, the only two alternatives are the septum

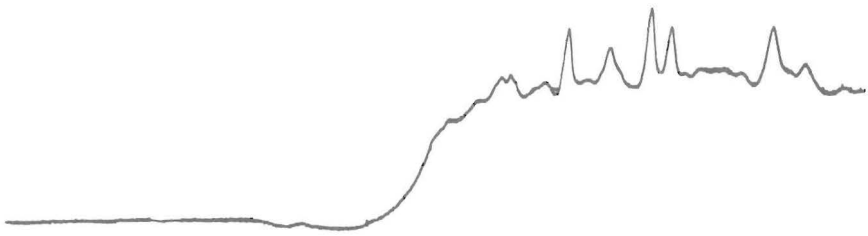


FIG. 1.—Gas chromatograph with the septum.

FIG. 2.—Gas chromatograph without the septum.

and the injector. A problem of this nature due to a faulty injector is very serious because of the high cost of replacing it.

Recently some extraneous peaks in the chromatographic analyses at the Rum Pilot Plant were noticed, and the procedure described clearly indicated that the septum was responsible (fig. 1). This was confirmed by the disappearance of the peaks when the metal cap replaced the septum (fig. 2).

The response obtained when the injection port temperature is varied suggests that some components of the septum are volatile or pyrolizable at temperatures above 160° C. Previous experience with these septa at

[7] Wiss, R. F., 1981. Determinations of carbon dioxide and methane by dual catalyst flame ionization chromatography and nitrous oxide by electron capture chromatography, J. Chromatogr. Sci. 19 (12): 611–16.

[8] Russev, P., Gough, T. A., and Woollam, C. J., 1976. Detection of inorganic gases using a flame ionization detector, J. Chromatogr. 119: 461–66.

temperatures near 200° C had been satisfactory, and no ghost peaks had been registered. The logical conclusion is that the problem arose from a low-quality lot of septa or that chemical changes occurred in them.

Several steps should be followed to avoid this source of ghost peaks:

The septum should be changed weekly; with the column disconnected from the detector, the injector temperature should be raised to 200° C and the column temperature to 20–30° C below its maximum. The following day the injector temperature should be decreased 20–50° C and a blank chromatograph run to detect any contamination before sample injection.

The injector temperature is then increased slightly in order to vaporize the sample, to avoid the appearance of the ghost peaks due to decomposition of the septum.

Guillermo Martínez Segura
Rum Pilot Plant