Gas Chromatography Problem Solving and Troubleshooting

Question:

Upon injection of a sample in carbon disulfide (CS_2), a large solvent front is visible when using a flame-ionization detector (FID). I thought FIDs did not respond to CS_2. I am using a 100:1 split ratio and a 1-µL injection volume, thus only a small amount of CS_2 should be entering the column. Is there something wrong with my FID or is my CS_2 contaminated with another solvent?

Answer:

There is nothing wrong with your FID or CS_2. It is commonly stated that an FID does not respond to CS_2. Actually, an FID does respond to CS_2, but the response is very poor when compared with other solvents. A 1-µL injection of CS_2 using a 100:1 split ratio still results in a typical and familiar-looking solvent front (Figure 1A). The CS_2 solvent front appears much like any other solvent front, and the FID response does not seem to be poor. Even though the FID response of CS_2 is poor, it is large enough to generate a large solvent front.

If the CS_2 response is directly compared with other solvents, a response difference becomes more apparent. In Figure 1B, each solvent is present at 1% in toluene. Now it is easy to see that the FID has a much greater response to these other solvents. The FID response to these solvents is about 75–200 times greater than CS_2. Although the CS_2 response is poor compared with most solvents, its response is high enough to generate a substantial peak at high concentrations.

FID response is related to the number of carbons in the compound. For saturated hydrocarbons, this behavior is

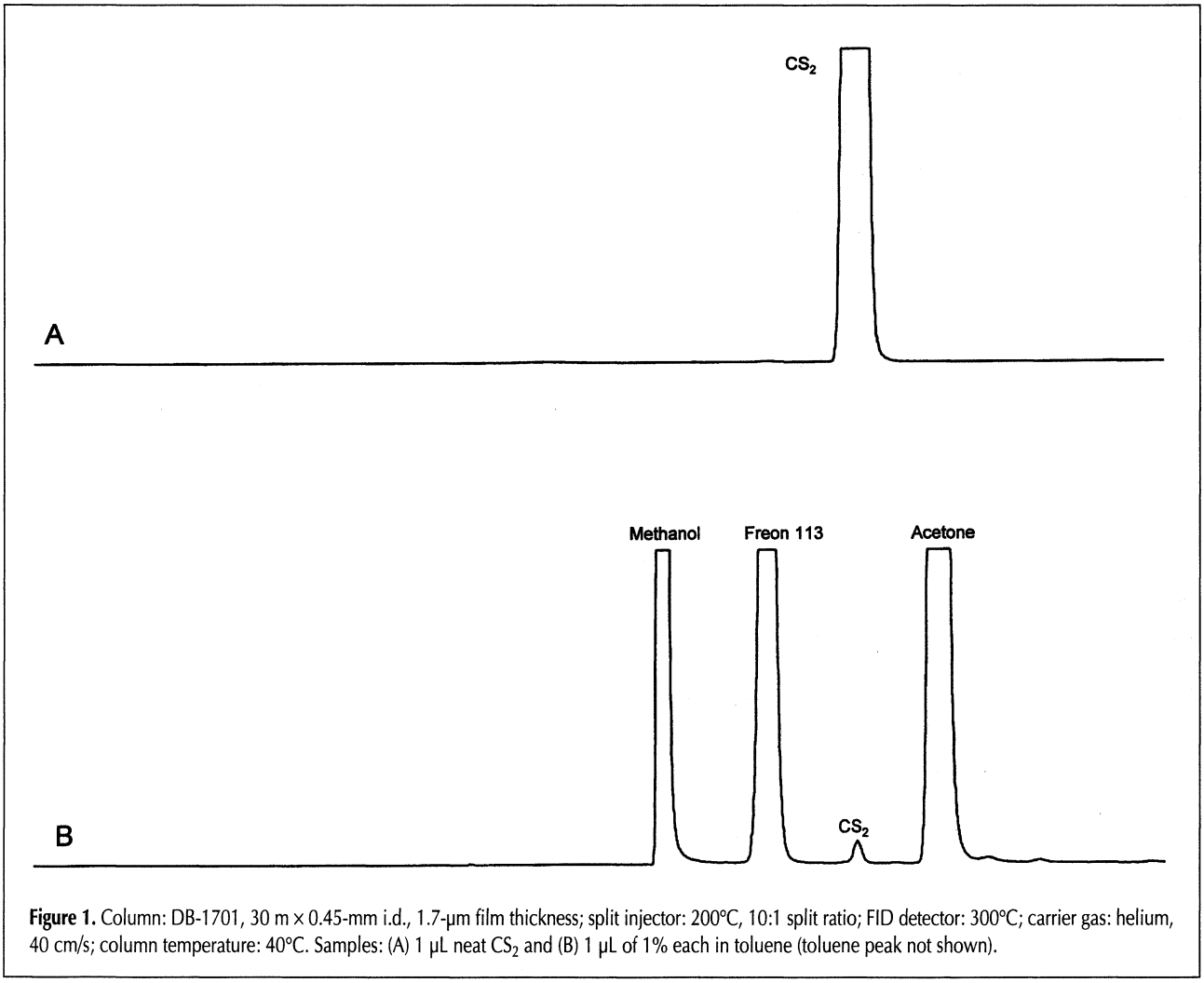

Figure 1. Column: DB-1701, 30 m x 0.45-mm i.d., 1.7-µm film thickness; split injector: 200°C, 10:1 split ratio; FID detector: 300°C; carrier gas: helium, 40 cm/s; column temperature: 40°C. Samples: (A) 1 µL neat CS_2 and (B) 1 µL of 1% each in toluene (toluene peak not shown).

Table I. Relative FID Response Factors

Group	Relative response
Aliphatic and aromatic C	1.00
Olefinic C	0.95
Acetylinic C	1.30
Carbonyl or carboxyl C	0.0
Ether O	–1.0
Alcohol O	–0.2 to –0.75
Halogens	variable

proportional to the number of carbons (e.g., FID response to *n*-octane is two times that of *n*-butane). The presence of other functionalities alters this relationship. The addition of oxygen, halogens, or another functional group usually decreases the response contributed by that carbon. In some cases, a non-carbon atom also decreases FID response. Table I lists the relative response factors for various types of carbons and other atoms (adapted from reference 1). A negative value indicates a decrease in FID response if that group or atom is present; a group with a zero value does not contribute to the FID response. Compounds such as formic acid and formaldehyde generate poor FID responses because only a single carbonyl carbon is present. Sometimes it is stated that a carbon–hydrogen bond is required for FID response. The chromatograms in Figure 1 show that this statement is false. Freon 113 contains no carbon–hydrogen bonds, and it has a good FID response. The lack of carbon–hydrogen bonds does reduce the FID response, but one is not required to obtain a reasonable response.

Reference

1. H.H. Hill and D.G. McMinn. *Detectors for Capillary Chromatography.* John Wiley & Sons, New York, NY, 1992.

The purpose of *Chromatography Problem Solving and Troubleshooting* is to have selected experts answer chromatographic questions in any of the various separation fields (GC, GC–MS, HPLC, TLC, SFC, HPTLC, open column, etc.). If you have questions or problems that you would like answered, please forward these to the *Journal* editorial office with all pertinent details: instrument operating conditions, temperatures, pressures, columns, support materials, liquid phases, carrier gas, mobile phases, detectors, example chromatograms, etc. In addition, if you would like to share your expertise or experience in the form of a particular question accompanied by the answer, please forward to JCS Associate Editor, *Chromatography Problem Solving and Troubleshooting,* P.O. Box 48312, Niles, IL 60714. All questions/answers are reviewed to ensure completeness. The *Journal* reserves the right not to publish submitted questions/answers.

Dean Rood
Associate Editor