



# Are GC techniques really optimized?

## Splitless injection as an example

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Users tend to think that basic GC techniques have been investigated in all details and sanctioned by a competent committee. In fact, why should one think about the design of an injector liner, after splitless injection has been used for more than 25 years? Modern companies invest 5-10% of their profits into research and development. Hence, bigger instrument manufacturers must have many labs with numerous people optimizing techniques. As splitless injection is probably the most widely used method of sample introduction in capillary GC, manufacturers must have tested their injector with all types of samples before releasing a new instrument. True or not? It would be difficult to find out. I have not seen behind the walls of all the instrument manufacturers, but I have

witnessed most of the development of splitless injection. I have come to the conclusion that the above views are awfully naive. There wasn't the idealist who invested many years to perfect splitless injection, nor an employer financing such a project. No instrument manufacturer had a single person working even just one year in extracting the knowledge from the literature available and checking all possible uses.

Splitless injection was shaped through a number of incidents and particular circumstances with only a few people involved. There were misunderstandings and errors; conditions were changed (such as carrier gas flow rates lowered or the injection process accelerated) without properly taking notice of the consequences. Some assumptions survived over decades without ever having been questioned. No one person took the responsibility for

providing the analyst with an optimized technique.

### Invention by Accident

Splitless injection was introduced by my father in 1968. He did not "invent" it by developing a concept in his mind and putting it into practice. He simply forgot one morning to open the split vent before performing what should have been a split injection. Peaks turned out to be very large (since all sample material entered the column). More surprisingly, all peaks were perfectly sharp. Everybody at that time was convinced that something like splitless injection would be impossible because the slow transfer of the components into the column created broad initial bands. Under other conditions, peaks were as broad as expected, and it took him about four years to determine the parameters required to produce sharp peaks, i.e. to understand the concepts of solvent effects and cold trapping.

Working in his spare time in the cellar of the school house (he was a teacher), my father had no means to modify the injector. Circumstances thus dictated that the new technique worked with the split injector available. It primarily had to solve his problems in trace analysis and was not developed with the interest of today's maybe 200,000 chromatographers in mind. For instance, he was not interested in highly accurate

quantitative data. His work was supported by a cigarette company to find out why smoke is harmful, not to develop an injection technique.

### Size of the vaporizing chamber

Because my father realized that a larger vaporizing chamber would be needed for storage of the sample vapors between their formation and transfer into the column, he had an injector made by a local mechanical shop. The design of this injector was described in *J. High Resolut. Chromatogr.* 1 (1978) 57. Since 1  $\mu$ l of liquid transforms into 100-400  $\mu$ l of vapor (further enlarged by mixing with carrier gas), an 80 x 4 mm i.d. chamber was selected with an internal volume of about 1 ml. There were long discussions concerning the geometry of the liner. A longer, more narrow chamber was preferable because it reduced mixing with the carrier gas and improved the transfer of the vapors into the column because of the higher gas velocity. However, this would require a very long syringe needle to allow the release of the sample near the bottom of the chamber. Because of its length, the syringe needle would be awkward and difficult to use.

This injector almost immediately became the standard for Carlo-Erba instruments. The other manufacturers continued to introduce injectors with chambers of merely 1-2

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