Why 5 cm syringe needles for capillary GC?

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GC is a complex technique. All too often the analyst stands in front of his instrument, surprised about a result, maybe annoyed about a problem, and at a loss.for an explanation for what he observes. Often, not even his colleague is able to explain. Another of these GC mysteries? Probably he would have the knack of it {f he knew the many details involved in the analyticalprocess. We make numerous choices without being aware of them, overlook variables clinging to the illusion that the)? had been thorough& investigated in the past and that an international committee has decided that this or that is the correct choice. The length of the syringe needle is one such frequently neglected detail and is an example of a parameter which has never received proper attention.

Many years ago, the manufacturers of GC syringes looked upon their customers and noticed that there was no agreement on how long syringe needles should be for conventional vaporizing (split or splitless) injection. Some said 1.5 inch (the needle protruding 37 mm from the glass barrel), others 3 inch (7 l mm), or even longer. So, father syringe producer decided to compromise and have it in between: 2 inch (51 mm). Whether or not he died in the mean time, that's how it still is. Some disagreed, but since it seems to be more important that GC is simple than that it is well optimized, the subject was commonly neglected. The subject of needle length seems not to be of sufficient scientific status to justify closer investigation.

As you can check by a few experiments, the length of the syringe needle and the depth by which a long needle is inserted into the injector often have an important impact on quantitative analysis. The reasons are explained below. It is concluded that they need to be adjusted to the situation. The length of the syringe needle determines from which point inside the liner the sample expands during the evaporation process. It may, however, also influence vaporization itself.

HEADSPACE ANALYSIS:

We start by looking at gas or headspace analysis, because the situation is particularly simple since no vaporization interferes. However the same principals will also apply to liquid samples. We refer to (manual or automated) injection with a gastight syringe of 0.5-l ml capacity.

Usually an amount of gas phase is injected that approaches the internal volume of the vaporizing chamber. For instance, a 4 mm ID liner of 80mm length has an internal volume of k ml. A 500u l sample mixes with carrier gas to form a vapor cloud of close to this volume (inlet pressure compresses the cloud, but increased temperature causes it to expand). Care must be taken to release the sample from the syringe needle in such a way that it ends being positioned inside the chamber.

Gas and headspace samples are usually injected in the split mode in order to achieve sharp initial bands. Depression of the plunger at normal speed introduces the sample at around 0.5-1 ml/s, i.e. 30- 60ml/ min. If the sum of the split and the (comparab1 ysmall) column flow rate corresponds to the rate of injection, expansion of the sample downwards replaces the gas flow from the rear. Gas supply is stopped; the gas phase running off originates from the syringe (assumption of a pressure-regulation/needle valve system, Fig.1). At higher split flow rates, the sample is diluted with additional carrier gas from the rear. Under these conditions, basically unlimited volumes of sample can be injected without overloading the injector. A short syringe needle merely entering the vaporizing chamber (2-3cm) serves the purpose, but longer needles are no drawback.

Since headspace analysis is mostly trace analysis, the split flow rate is usually substantially below the 30-60ml/min mentioned above. This leaves the choice of injecting at a correspondingly reduced rate or temporarily storing the vapor cloud inside the vaporizing chamber. The latter corresponds to common practice. If more sample is injected than gas runs off at the same time, carrier gas must be displaced within the injection system. Appropriately designed injectors with a pressure regulator at the rear and a needle valve in the split outlet have a relatively large internal volume in the gas supply and a small one in the split outlet, causing the sample to expand backwards (Fig.2). Long syringe needles are required such that the sample expands from a point near the column entrance towards the rear. If the liner is 80 mm long, the column enters by 5mm, and the injector head is some 12mm high, the syringe needle should be around 80mm

Figure 1 - Injection at a rate equal to the flow rate of the gas passing through the liner: the flow from the carrier gas supply is substituted by that leaving the syringe needle.