Use of a spinal needle as an injection device for solid substances in gas chromatography

A relatively inexpensive, readily available item, the B-D Yale Luer-Lok No. 462 LNR Spinal Needle ((Quincke) (Becton, Dickinson and Company, Rutherford, N. J.)) can serve, without alteration, as an injection device for solid substances into a gas chromatograph. Although several methods of introducing solids into gas chromatographs have been reported[1–3] this method for directly injecting up to a 6 mg sample without instrument modification nor disruption of gas flows is free from complexity. The injection of these sample sizes allows for detection with either thermistors or hot wires and also allows for sample collection for further analysis or investigation.

A spinal needle is shown in Fig. 1, item 1. It is made up of two units, a cannula, item 2, which is essentially a syringe needle, and a stilette, item 3, which is essentially a solid steel rod. The stilette fits into the cannula with a snug to sliding fit. Both the cannula and stilette have beveled tips. Although spinal needles with cannula outer diameters ranging from 0.025 to 0.050 in. have been used, a cannula outer diameter of about 0.035 in. and a stilette diameter of about 0.022 in. was found to be about optimum for injection of 3 to 6 mg samples. These cannula and stilette dimensions correspond to a 20 gauge spinal needle.

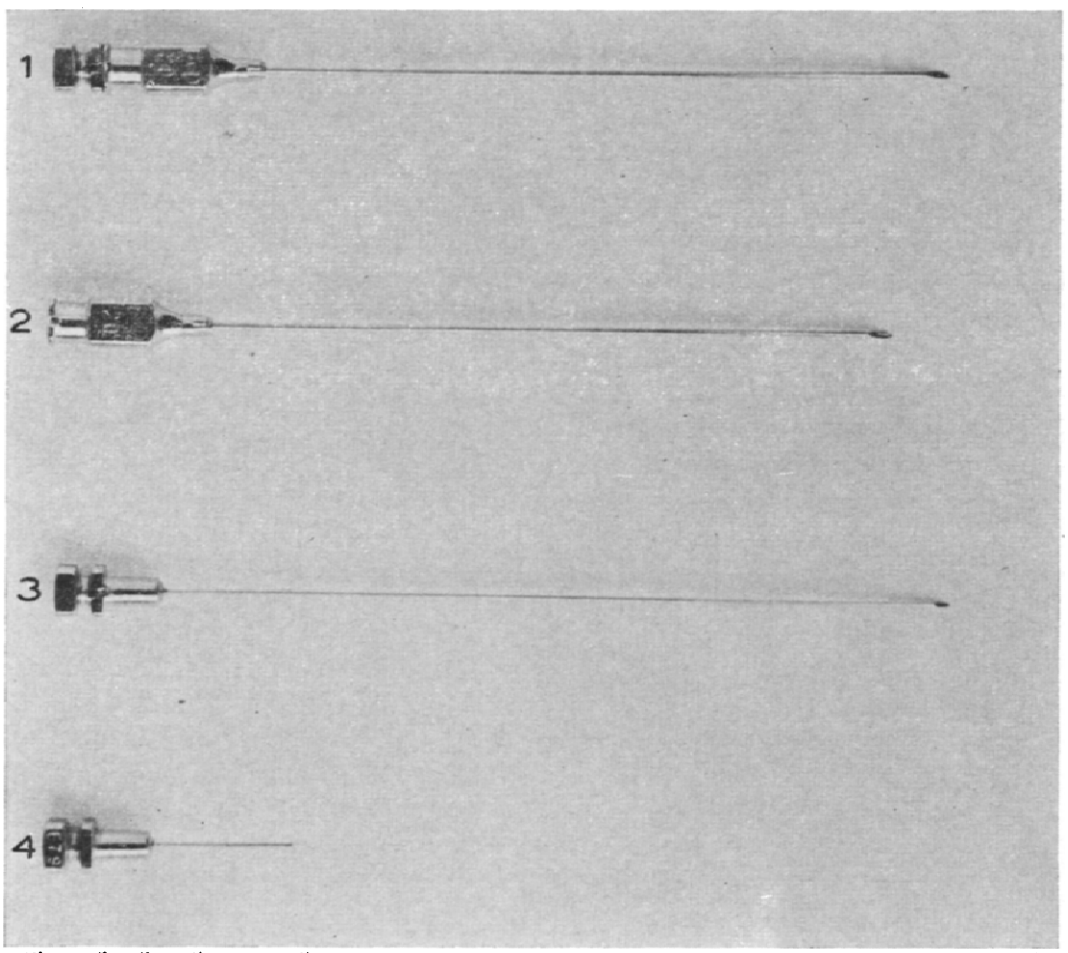

Fig. 1. Injection device (approximately actual size). 1: Spinal needle. 2: Cannula. 3: Stilette. 4: Packing rod.

In order to prevent leakage during use as a solids injector into a gas chromatograph, a little silicone gum rubber liquid phase column packing (F and M Scientific Corp., Avondale, Pa.) may be applied in the vicinity of the plunger end of the stilette and into the hub of the cannula. This, however, is not always necessary.

The device is filled by pulling the stilette into the cannula so as to form a cylindrical cavity at the end of the needle approximately $^1/_4$ to $^1/_2$ in. in depth and forcing the ground sample into this cavity. The ground sample is forced into the cavity by pushing the open end of the needle against a thin, packed layer of ground sample contained in a glass mortar. The packing rod, Fig. 1, item 4, having approximately the same diameter as the stilette but being somewhat shorter in length, is used to aid in the filling of the cavity.

Injection is accomplished in a similar manner as with a liquid or gas syringe, that is, the needle is inserted through an injection port silicone-rubber diaphragm or septum, and the sample is forced out of the cavity of the needle by application of pressure to the stilette. The needle is then immediately withdrawn from the injection port. About a dozen "passes" with a 20 gauge needle are possible before a silicone rubber septum would require replacement.

Quantitative relationships are obtainable by weighing before and after injection; however, "normalization", without sample weighing, is also quite frequently used.

This spinal needle injection technique has been used very successfully by our laboratories for some time where substances with melting points as high as 300° have been successfully chromatographed. This, of course, necessitates a chromatograph capable of providing high temperatures in the injection port and detector block systems. A F and M Model 500 Programmed High Temperature Gas Chromatograph was used for these studies, and the chromatography of these high melting point substances may be the subject of future papers. As an injection device for solid substances, the spinal needle has been found to be quite sturdy and fool-proof. After prolonged usage, the stilette may accumulate a carbon deposit and clogging may occur. The relative low cost of the unit, however, allows for discarding without concern.

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