A sampling device for viscous and solid materials for gas chromatography*

A number of devices have been reported for introducing samples of solids and difficult-to-handle materials into a gas chromatograph[1–4]. We have constructed a simple sampling device which can be used in conjunction with an independently heated injection port for sampling viscous materials and solids for gas chromatographic analysis. This device has a number of advantages. It is as easy to use as any

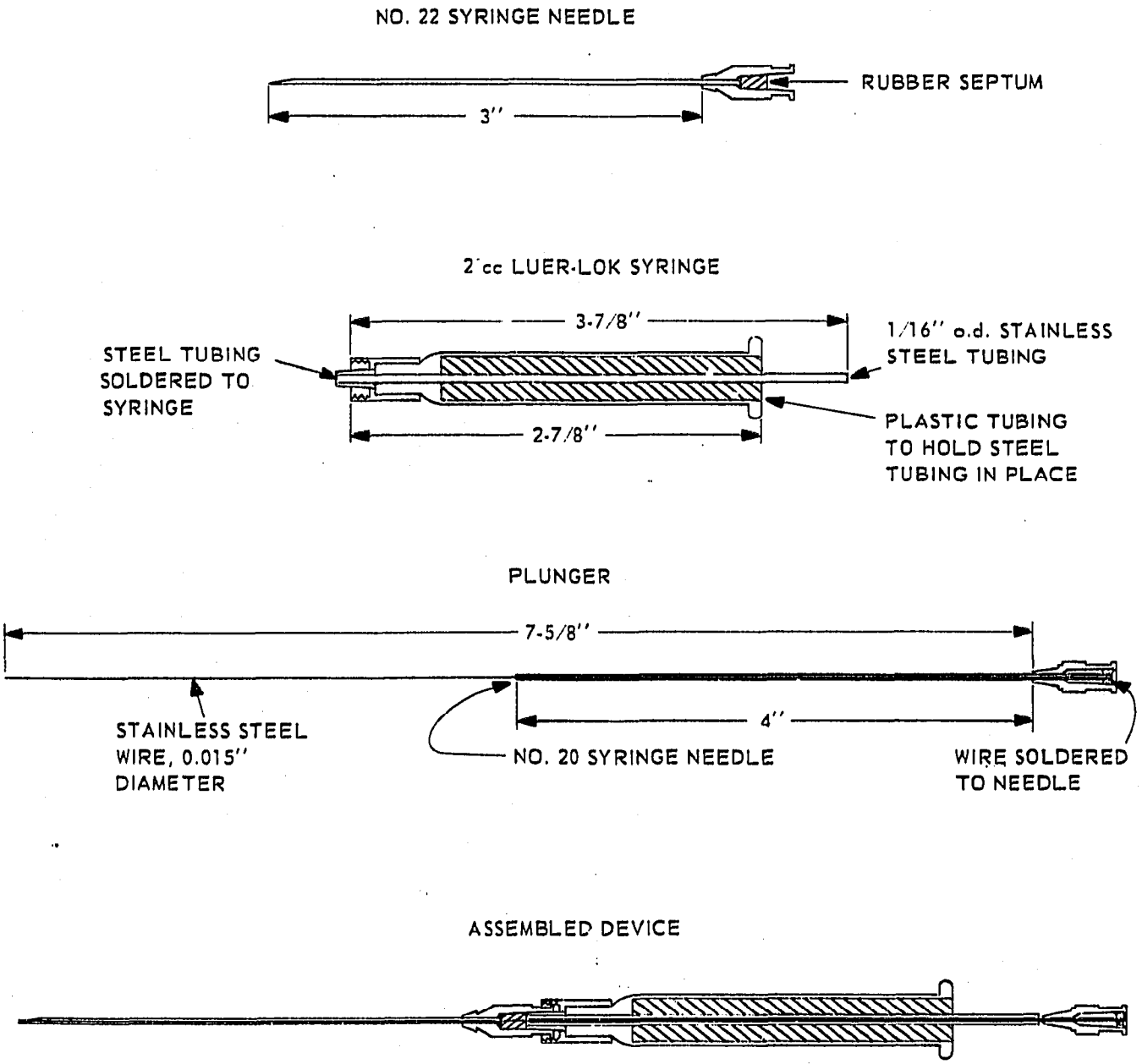

Fig. 1. Sampling device for viscous and solid materials.

* This work was supported in part by the U.S. Atomic Energy Commission, Contract No. AT(40-1)-2490.

other type syringe; it is not necessary to modify the gas chromatographic unit provided the injection port is heated independently; it is not necessary to dissolve the sample in a volatile solvent followed by evaporation of the solvent as the solution is applied to a plunger or trough[1,2]; the loss of volatile components can be minimized by taking samples through a rubber septum; and the flow of carrier gas is not interrupted. The device is shown in Fig. 1. It can be easily constructed from readily available materials with no instructions needed other than the details given in Fig. 1. Variations in the dimensions can be made to give a device with either a smaller or larger capacity. With a No. 22 syringe needle, samples up to about 10 λ can be taken. In practice, the sampling technique is to heat the sample so that it becomes less viscous or melts. After drawing a sample into the needle, the needle is immediately injected into the heated port. If necessary, a short period of time is allowed for the needle to heat up before the sample is ejected. In our work, samples of the molten material have been taken through a rubber septum so as to minimize any loss of the more volatile components present in small amounts. For quantitative work, internal standards have been used. A convenient manner for taking samples when only a small quantity of solid is available is to melt the sample in an ordinary melting point tube. The needle portion should be heated to approximately the same temperature before sampling.

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Received August 28th, 1964

A multi-purpose device for the collection of fractions separated by gas chromatography

The collection of fractions emerging from a gas chromatographic column has been the subject of several publications in the past few years. Effective trapping of these fractions is necessary for their further identification, which can be done by re-running them on other columns, by spectrometric analysis, etc. Generally the collecting devices described are designed in such a way as to meet the demands associated with only one of the above methods for further identification.

In this note we wish to describe a multi-purpose device for the collection of fractions separated by gas chromatography. It is suitable both for the re-running of fractions on other columns and for their further identification by I.R., U.V. or mass spectrometry. In Fig. 1 a photograph is given of this device. It consists of a socket joint ($^{12}/_{1}$) to which a capillary (1 mm I.D.) is attached. The capillary is bent to give it a zig-zag shape. It ends in a glass tube of 4 mm I.D., which makes an angle of