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Note

Universal sample inlet for gas chromatography

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A universal system for sample introduction in gas chromatography must allow sampling of gases, liquids and solids. There are only a few methods that fulfil this condition; most of them involve crushing of an ampoule and were reviewed previously¹. To overcome some of the difficulties associated with crushing devices, we developed an ampoule-tip breaker², but the apparatus was rather complicated and was unsuitable for direct injection.

Our new concept³ consists in a simple modification of commercially available external-injection inlets such as, *e.g.*, the Hamilton flush vaporizer, model No. 86800 (Micromesure B.V., The Hague, The Netherlands). The adapter (see Fig. 1) comprises a stainless-steel tube (1) that terminates in a plug (2) having one end inclined and drilled with an eccentric bore; four holes bored in the body (3) serve as inlet for carrier gas. A stainless-steel rod (4) with one end inclined to match the plug forms a movable plunger, which, when pushed, breaks off the ampoule tips (5) which have been previously notched with a file. A silicone-rubber ring (6) pressed to the adapter tube by a spring (Hamilton inlet) or by a capping nut ensures gas-tightness. The entire inlet (7), heated by means of a cartridge, is attached to the injection port of the gas chromatograph by a nut (8).

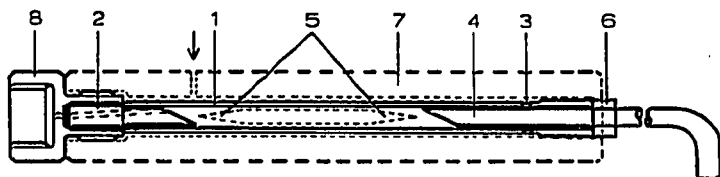


Fig. 1. Adapter for universal sample inlet. 1 = Stainless-steel tube, I.D. 5.5 mm and O.D. 7.5 or 8.3 mm; 2 = stainless-steel plug, O.D. 5.5 or 5.8 mm with one end inclined and eccentric hole of I.D. 1 mm; 3 = four holes 1 mm I.D. for carrier-gas entry; 4 = stainless-steel rod, O.D. 5.0 mm, with one end inclined; 5 = glass ampoule tips notched with a file; 6 = silicone-rubber sealing ring; 7 = external inlet; 8 = nut for connecting the inlet to the injection port of the gas chromatograph.

USE OF THE DEVICE

For the direct injection of liquids and gases, the movable rod with O-ring is replaced by a septum; this operation, like the removal of the ampoule from the adapter, requires only 1-2 min.

We tested the inlet system by successively introducing 0.2- μ l portions of standards in three ways: (A) by direct injection into the port of the gas chromatograph (Carlo Erba, Milan, Italy; model P-AICD-2f); (B) by injection through the adapter; and (C) by injection into glass ampoules and proceeding as already described¹. One standard consisted of a mixture of eight hydrocarbons (both aliphatic and aromatic), and the other of five strongly polar compounds. For their separation, we used an aluminium column (2 m \times 4 mm I.D.) packed with 3% of XE-60 on silanized Embacel (0.20–0.25 mm; May and Baker Ltd., Dagenham, Great Britain). The temperature of the column was kept initially at 60° for 5 min, then programmed to 180° at 10° per min. The results, expressed in the form of theoretical-plate numbers (n), separation factors (SF) and percentage peak separation (PS)⁴ are shown in Tables I and II for the non-polar and the polar mixture, respectively.

TABLE I
ANALYSIS OF NON-POLAR MIXTURE
For details of methods A, B and C, see text.

Compound	Method A			Method B			Method C		
	n	SF	PS	n	SF	PS	n	SF	PS
<i>n</i> -Pentane	1047			1047			748		
Benzene	892	1.73		1185	1.81		892	1.72	
Toluene	1086	1.47		1340	1.47		936	1.36	
<i>m</i> - + <i>p</i> -Xylene	1069	1.53		1186	1.57		981	1.53	
<i>o</i> -Xylene*	1601	1.18	97.8	1662	1.18	97.3	1966	1.17	94.9
Naphthalene	20,260	—		23,910	—		19,284	—	
Biphenyl**	22,910	1.16		29,520	1.16		24,000	1.17	
Diphenylmethane	18,820	1.04	91.6	15,220	1.04	93.4	13,140	1.03	90.2

* Program start.

** Program finish.

It can be seen that, for the non-polar mixture, there are no substantial differences between the three sample-introduction methods. However, for the polar mixture, the ampoule method seems to exert a slightly negative effect on the separation process. The results were improved when the quartz-wool plug in the ampoule was omitted, and when the plunger was moved back slightly after the ampoule tips had been broken. This led, perhaps, to a reduction in sorption forces and to improved purging of the adapter by the carrier gas, respectively. It is noteworthy that injection of samples through the external inlet is more effective than direct injection into the injection port of the gas chromatograph; this is attributed to better geometry of the former device.

TABLE II

ANALYSIS OF POLAR MIXTURE

For details of Methods A, B and C, see text.

Compound	Method A			Method B			Method C		
	<i>n</i>	<i>SF</i>	<i>PS</i>	<i>n</i>	<i>SF</i>	<i>PS</i>	<i>n</i>	<i>SF</i>	<i>PS</i>
Ethanol	936	1.12	50.9	958	1.13	61.9	864	1.10	45.0
Ethyl acetate	565	1.14	80.1	703	1.14	85.1	571	1.14	78.6
Ethyl methyl ketone	859	1.43		1086	1.43		864	1.42	
Dioxane	992	1.12	81.9	1208	1.13	90.3	986	1.10	85.2
<i>n</i> -Butanol	1097			1274			1074		

The ampoules are convenient also for the trapping of fractions separated by gas chromatography and for carrying out micro-scale chemical reactions, such as hydrogenolysis, hydrolysis, pyrolysis, and derivative formation. The procedure described is equivalent in efficiency to other sample-introduction methods used in gas chromatography; it also permits sampling of gases, liquids or solids.

REFERENCES

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- 3 J. Blatnický, Patent pending.
- 4 O. E. Schupp, III, *Gas Chromatography*, Interscience, New York, 1968.