A Simple Improved All-Glass System for the Microanalysis of Pesticides on the Dohrmann Model 100 Gas Chromatograph

by W. L. WINTERLIN, J. B. RIVERS and G. O. WALKER
Agricultural Toxicology and Residue Research Laboratory
University of California, Davis, California

The incorporation of an all-glass system into a gas chromatograph and the advantages of this system for the analysis of pesticides over a metal system have been investigated by Beckman and Bevenue (1, 2). The all-glass, one piece, injection block and chromatographic column as designed by Beckman and Bevenue (2) for the Dohrmann Model 100 Gas Chromatograph has several drawbacks, some of which are the inability to change columns, the difficulty in construction which requires a professional glassblower, the difficulty in reproducing results, and the inability to clean up the system, particularly the injection area, when desired.

Therefore, an all-glass system for the Dohrmann Model 100 Gas Chromatograph was designed to overcome the difficulties mentioned and can be constructed with ease by any glassblower using borosilicate glassware.

Construction

Fig. IA and IB illustrate the construction of the improved, all-glass system.

Fig. IA illustrates the construction of the all-glass injection unit. Special attention should be given to the constriction near the

Bulletin of Environmental Contamination & Toxicology,
Vol. 2, No. 1, 1967, published by Springer-Verlag New York Inc.

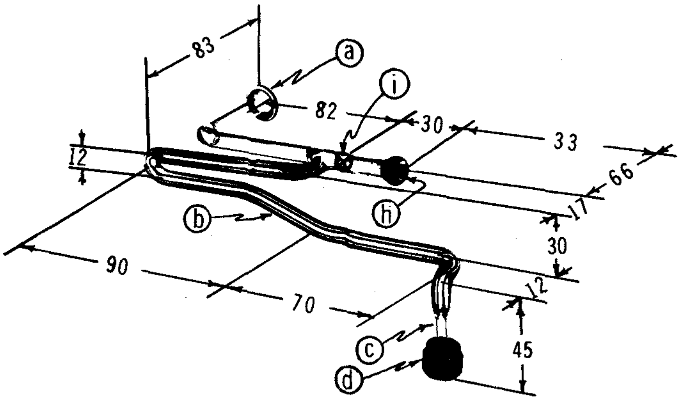

Figure IA. All-glass injection unit

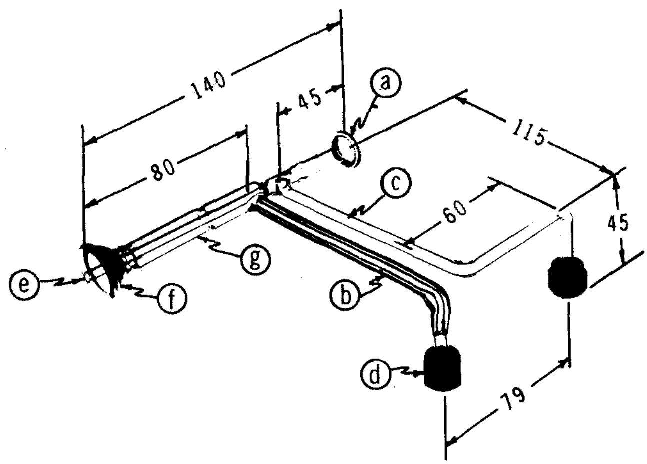

Figure IB. All-glass post column unit

ⓐ 12/5 standard ball joint, ⓑ 2 mm. I.D. thick wall glass tubing, ⓒ 6 mm. I.D. standard wall glass tubing, ⓓ 1/4" Swagelok, ⓔ 5 mm. I.D. standard wall tubing, ⓕ 18/9 standard ball joint, ⓖ 12 mm. I.D. standard wall glass tubing, ⓗ injection septum, 8 mm. long and 6 mm. O.D. hole halfway through, ⓘ constriction area 1 mm. I.D. where the needle is centrally directed upon injection. All dimensions shown are in mm.

septum injection area where the needle from the microliter syringe is centrally guided insuring uniform injections. When necessary, the injection unit may be removed for cleaning without disturbing the rest of the system. All connections are made of ball joints permitting flexibility in attaching the component parts, as well as assisting the interchanging of columns or parts with a minimum of delay. All joints are compressed together with metal, plain type clamps and Teflon washers made from Teflon sheets 0.01" thick and 3/4" in diameter with a 3/8" hole in the center. The Teflon washers are placed between the joints to prevent any possible gas leaks and also to allow for ease of assembly and disassembly of the component parts. The time required to cool the oven, change the column and reheat the oven is less than one hour. Flexibility in construction in both the horizontal and vertical measurements is permitted with the exception of the overall distance between ⓐ and ⓓ in Fig. IA.

Fig. IB illustrates the post-column unit. The construction of this unit is critical only in the overall distance from ⓐ to ⓓ and between the Swagelok fittings. This unit is designed to reduce the possibility of eddy currents when the carrier gas is moved into the combustion tube and is mixed with oxygen from the inlet tube ⓑ. Auxillary nitrogen, usually introduced into the gas system at the termination of the column, was found to be unnecessary and was eliminated. Column effluents not to be combusted are allowed to pass through the vent tube ⓒ. Fig. 2 illustrates the system as it would appear in the Dohrmann Model 100 Gas Chromatograph incorporating the three basic all-glass units, the injection unit, the pyrex column, and the unit between the column and the quartz combustion tube, best described as the post-column unit.

The injection area is separated from the column by a 1/4" thick asbestos-cement disc, cut in half, with two slots for the spacing of the column connections. The two halves are connected by metal strips and are readily separated. This disc permits a

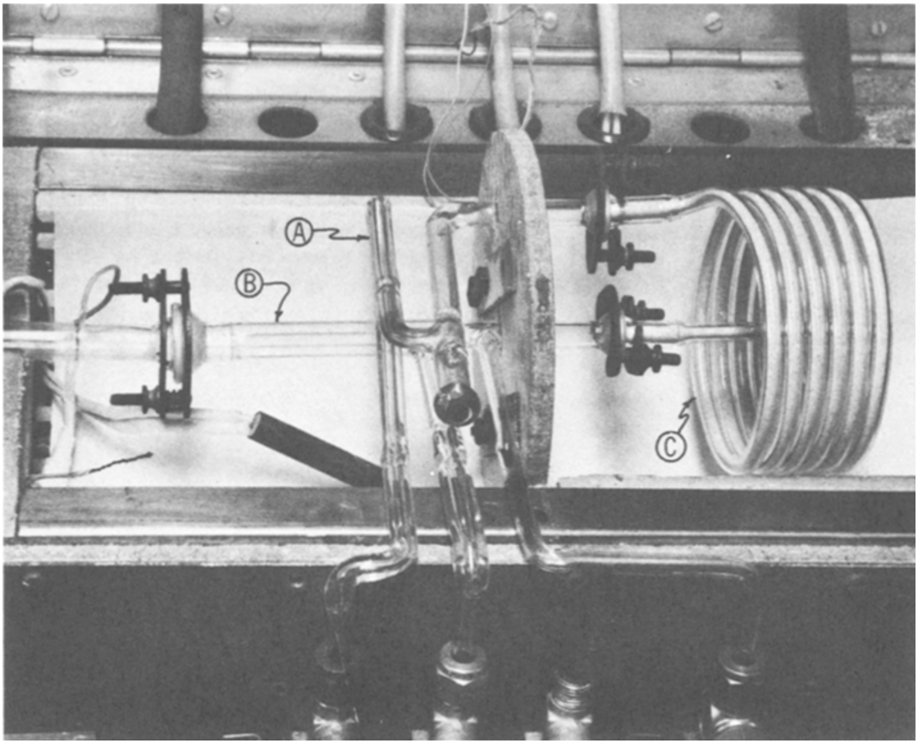

Fig. 2. An all-glass system for the Dohrmann Model 100 Gas Chromatograph: Ⓐ the injection unit, Ⓑ the chromatographic column, Ⓒ the post column unit.

differential in temperature between the injection area and the column.

An asbestos block (not illustrated) $2^1/_2$" x $2^1/_4$" and 1" thick is cut with appropriate slots to fit around the gas inlets and the septum injection area. The block is placed between the two sections of the oven to insure against heat loss and injury to the operator while injecting a sample.

Discussion

Table I shows the reproducibility of injection of the chlorinated pesticide 2, 4-D Me (2, 4-dichlorophenoxyacetic acid methyl ester) on a Dohrmann Microcoulometer C-200 with a chloride cell T-300 using a 6-foot, 1/4" O. D. column packed with a 10% SE 30 on acid-washed chromosorb W, HMDS treated. The reproducibility of the injections due to the all-glass system is well within the error of measurement.

Fig. 3 illustrates the difference in recovery of the all-glass system as compared to the metal injection block utilizing the same glass column and the same ohm setting on the coulometer. The optimum response is about 4.7 times greater with the all-glass system. The optimum conditions are based upon maximum response due to stirring rate within the cell, carrier gas flow and electrode positioning.

TABLE 1

The reproducibility of several injections for 2,4-D Me (2,4-dichlorophenoxyacetic acid methyl ester) using the all-glass system on the Dohrmann Microcoulometric Gas Chromatograph.

Added		Peak area[a/]		
Vol. µg.	µg 2,4-D Me	sq. in.		
6.0	0.12	1.49	1.49	1.48
5.0	0.10	1.24	1.24	1.23
4.0	0.08	0.97	0.98	0.99
3.0	0.06	0.72	0.74	0.74
2.0	0.04	0.50	0.50	0.51
1.0	0.02	0.25	0.25	0.26
		Std. Dev.		0.0066

a/ Peak area was measured with a Lietz Model 236 planimeter.

The theoretical recovery for the all-glass system was 344% as compared to the 74% theoretical recovery for the metal injection block-glass column system. This high theoretical recovery for the all-glass system is unexplained at this time; however, all data reported by our laboratory are based on standard curves run at the time of analysis, and the theoretical recovery does not pose a practical problem.

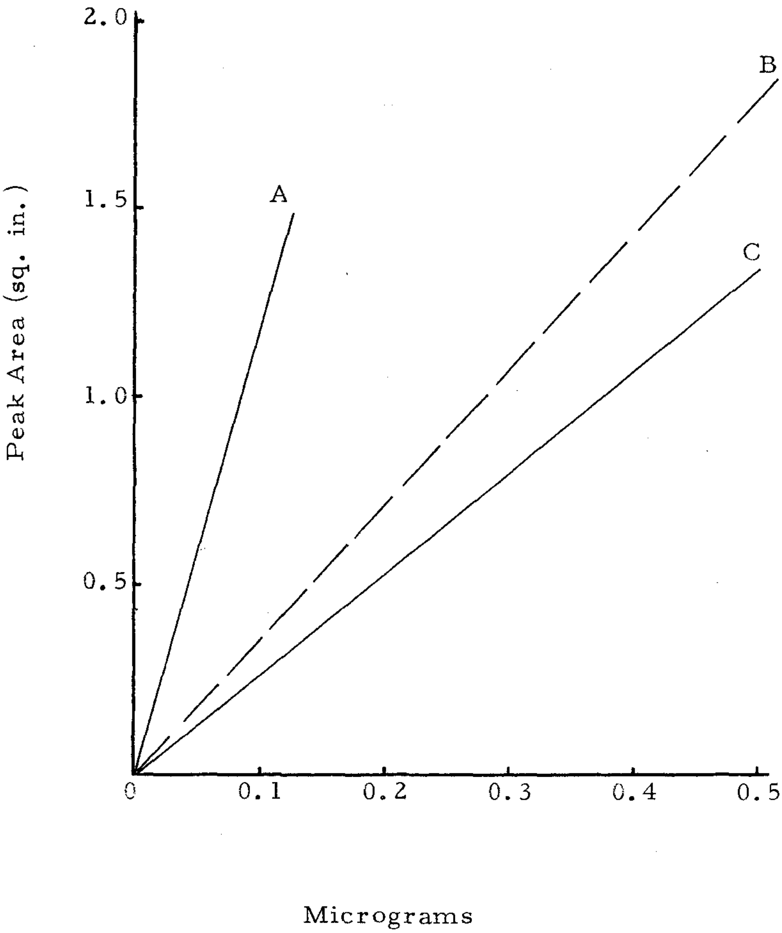

Figure 3. 2, 4-D Me Standard Curves. A. Using all-glass system; B. 100% theoretical recovery; C. Using metal injection block-glass column system.

Literature Cited

1. Beckman, H. and Bevenue A., J. Chromatog. 10, 231 (1963).
2. Ibid., 12, 109 (1963).