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Note

Pressurizable packer for gas chromatographic columns

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When a gas chromatographic column is packed by suction and vibration, the permeability of the bed decreases during its growth. Therefore, the gas flow-rate produced by the vacuum decreases and a long period is required to complete the packing.

To overcome this difficulty, the packing can be carried out under pressure, so keeping the gas flow-rate at reasonable levels. Commercially available appliances for pressurized filling usually inject the packing into the column almost instantaneously. Although the results are reproducible, the efficiency of the columns obtained was found to be less than that achieved with conventional hand-packing methods[1].

In this paper is described a device designed for pouring slowly and continuously the stationary phase under a gas pressure into the column, so assuring a homogeneous and tight packing.

EXPERIMENTAL

Pressurizable packer

A stainless-steel funnel (A) (Fig. 1) with a narrow-bore calibrated outlet (B) at the base is housed in a heavy-walled cylinder and held in position by the conical upper collar (C). A ring of perforations below the funnel cone (D) ensures equilibration of the gas pressure between the outer and inner space of the funnel. The outer, heavy walled, cylinder (E) is capped at the top by a screwed cover (F) with a lateral hole (G), closed by a pressure-proof screw. A central, leak-proof screwed needle (H) ensures the closure of the lower hole of the funnel as required. The base of the packer is connected by Swagelok fittings to the chromatographic column. The pressure is applied through a lateral valve connected to a gas cylinder with a pressure regulation gauge.

Packing procedure

The apparatus is assembled and the outlet of the funnel is closed by screwing down the needle. The column, held with the coiling plane in a horizontal position, is connected with Swagelok fittings and PTFE ferrules (Supelco, Bellefonte, Pa., U.S.A.) to the packer and through a rubber tube to a flow meter. Losses of stationary phase are avoided by stopping the powder at the end of the column with a nylon net held

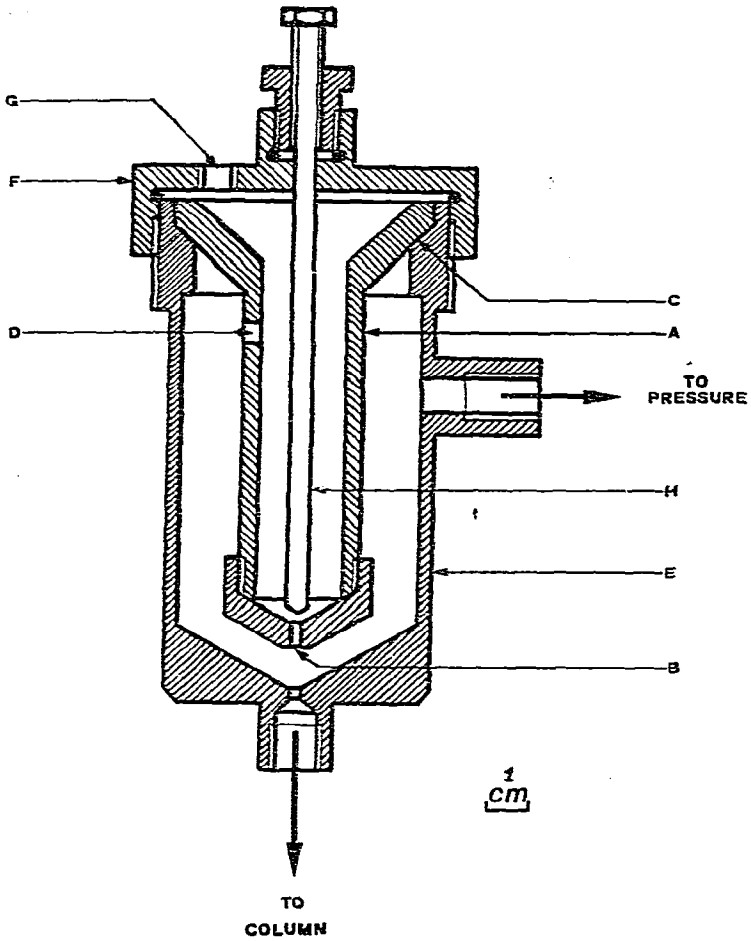

Fig. 1. Sectional drawing of the packer (the connections with the gas cylinder and column have been omitted).

in place by the rubber tubing. A weighed amount of stationary phase is introduced into the funnel through the hole (G), the hole is closed and the packer pressurized under the control of a pressure regulation gauge until the flow-rate at the end of the column is about 50–80 l/h.

The outlet of the inner funnel is opened by unscrewing the needle (H), then the packing drops by gravity and is carried into the column by the gas flow. The flow of the powder is aided by gently tapping the chromatographic tube and the body of the packer with a piece of thick-walled rubber tubing. As the chromatographic bed grows, the pressure is increased and the gas flow is held constant.

In a typical packing operation, with the calibrated outlet (B) of the funnel properly regulated for a suitable flow (usually 0.75–1 mm), the chromatographic bed grows at the rate of about 5 cm/min and the operation is completed in about 30 min. In operations of this kind, it is usual to adopt final pressures of about 5–6 atm.

At the end of the operation the gas flow is stopped and the pressure is allowed to decrease to ambient throughout the column.

Gas chromatography

All chromatograms were obtained on a Hewlett-Packard 5710 gas chromatograph with dual flame-ionization detectors, using the direct on-column injection technique. A coiled glass tube (6 ft. × 2 mm I.D.) (Hewlett-Packard, configuration 5) was used, packed with 3% OV-101 or OV-17 silicone oil on Chromosorb W HP (100–120 mesh) as the stationary phase.

The pressure at the top of the column was determined by a needle pressure gauge (Hamilton, Reno, Nev., U.S.A.); the carrier gas flow-rate was measured with a soap-bubble flow meter, and corrected for the oven temperature and pressure drop across the column[2]. The plate number was calculated from the peak width at half-height and the retention time; the linear gas velocity was calculated from the retention time of methane.

The performance of the columns was checked with a standard mixture of linear hydrocarbons and sometimes with a hydroxylated steroid (3α-hydroxy-5α-pregnane-11,20-dione). For gas chromatography the steroid was derivatized with bistrimethylsilylacetamide–trimethylchlorosilane (4:1)[3].

The columns were conditioned by heating at 50° for 32 min, then the temperature was increased at the rate of 1°/min to 280° and maintained at 280° overnight. All operations were performed under a flow of nitrogen (20 ml/min).

RESULTS AND DISCUSSION

Table I gives the pressure drops (ΔP) across a 3% OV-101 column at various linear flow-rates ($\bar{U}$). The data show good linearity and extrapolation to zero velocity gives a pressure of about 0.39 atm.

TABLE I

PRESSURE DROPS AT 120° ACROSS A 6 ft. × 2 mm I.D. OV-101 COLUMN

Carrier gas: nitrogen.

ΔP (atm)	$\bar{U}$ (cm/sec)
0.80	3.21
1.17	6.71
1.55	8.61
2.00	12.03

Table II shows the performance obtained with *n*-hexadecane run in the same column at 120° at various flow-rates. The performance is good at low flow-rates, while at higher flow-rates the mass transfer term becomes dominant and the HETP increases.

Table III gives the data obtained from three columns packed at different times with the same preparation of 3% OV-17 on Chromosorb W HP (100–120 mesh). In this instance the performance was checked at 255° with a standard mixture of linear hydrocarbons and a silylated steroid as previously described.

The better performance of the steroid compared with that measured with linear hydrocarbon is possibly correlated with the physico-chemical characteristics of the partition liquid. A run in a completely apolar phase (OV-101) shows about th

TABLE II

PERFORMANCE OF A 3% OV-101 COLUMN AT 120° AT VARIOUS FLOW-RATES

Sample: 0.5 μl of a 100 μg/ml solution of *n*-hexadecane in chloroform.

Parameter	$\bar{U}$ *(cm/sec)*			
	3.21	*6.71*	*8.61*	*12.03*
Plate number	4853	3745	3298	2790
HETP (mm)	0.38	0.49	0.55	0.65

TABLE III

GAS CHROMATOGRAPHIC BEHAVIOUR OF A STANDARD MIXTURE IN THREE DIFFERENT COLUMNS OF 3% OV-17 ON CHROMOSORB W HP

Oven temperature, 255°; injection temperature, 300°; detector temperature, 300°; sample, 0.5 μl of a 100 μg/ml solution of all components in chloroform. F and V_r are the flow-rate and the retention volume, both corrected for temperature and compressibility.

Column No.	$\bar{F}$ *(ml/min)*	$\bar{U}$ *(cm/sec)*	V_r *(ml)*	*Plate number*	*Component*
1	14.6	8.49	62	3544	*n*-Hexacosane
			109	3242	*n*-Octacosane
			333	3555	*n*-Dotriacontane
			243	4847	Steroid
2	15.2	9.13	60	3376	*n*-Hexacosane
			107	3059	*n*-Octacosane
			324	3927	*n*-Dotriacontane
			236	4567	Steroid
3	14.0	8.40	76	3399	*n*-Hexacosane
			135	3427	*n*-Octacosane
			375	3166	*n*-Dotriacontane
			266	4633	Steroid

same performance for *n*-dotriacontane and the steroid (plate numbers 4343 and 4352, respectively). The retention volumes and performances in all of the columns were close enough to permit them to be interchanged in routinary analytical applications, also at low concentrations.

In conclusion, the packing process is rapid, smooth and requires less care and time than the usual manual procedure. The need for vibrations to obtain uniform, tight packings is considerably reduced and the probability of fracturing the silica particles and creating new absorption surfaces in the grains is diminished. The performance of the columns obtained is satisfactory for routine analytical work.

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REFERENCES

1 W. R. Supina, *The Packed Column in Gas Chromatography*, Supelco, Bellefonte, Pa., 1974, p. 114.
2 E. S. Perry and A. Weissberger, *Technique of Organic Chemistry: Gas Chromatography*, Wiley-Interscience, New York, 1968, p. 15.
3 E. M. Chambaz and E. C. Horning, *Anal. Biochem.*, 30 (1969) 7.