

Notes

CHROM. 3906

Low pressure gas chromatography

Experimental

A Beckman Model GC-2 gas chromatograph was modified so that in addition to the normal mode of operation the instrument could be operated with the carrier gas at the inlet of the column being at atmospheric pressure, and the exit at the detector being held at a reduced pressure. A diagram of the instrument with the modifications is shown in Fig. 1.

The modification of the Beckman GC-2 consisted of placing two three-way Hoke valves in the normal flow pattern of the instrument. One of the three-way valves was placed in the instrument so that the carrier gas to the column could be supplied from either of two different sources. The other three-way valve was placed on the exit end of the detector so that the detector could be vented to the atmosphere during normal operation or connected to a vacuum source during low pressure gas chromatography.

During low pressure chromatography the carrier gas to the chromatograph is supplied from a helium reservoir which is a stainless steel cylinder in which helium is held at atmospheric pressure. Helium from a Linde 1A size cylinder is continuously supplied at the bottom of the reservoir and allowed to exit at the top of the reservoir by either of two outlets. One exit is vented to the atmosphere and the other is connected to the three-way valve at the inlet of the chromatographic column. The flow of helium to the reservoir is controlled by a needle valve in the line from the helium cylinder to the reservoir. The flow rate of carrier gas through the column during low pressure chromatography is determined by measuring (with a soap film flow meter) the flow rates of helium out of the reservoir at the atmospheric vent when the connection to the chromatograph is closed and opened.

The vacuum source used during low pressure chromatography is a Gast vacuum pump Model 0211-V201-G10X equipped with a vacuum gauge. The vacuum pump is connected to the top of a vacuum reservoir which is a stainless steel cylinder of approximately one liter capacity. The vacuum reservoir is used to minimize any fluctuations in vacuum due to the mechanical action of the vacuum pump. The exit of the detector of the chromatograph is connected to the vacuum reservoir with the three-way valve. A trap was placed in the line connecting the chromatograph and the vacuum reservoir to collect the compounds eluted from the column before they went into the vacuum pump.

When the two three-way valves are in the position shown in Fig. 1, the gas chromatograph can be operated in the normal mode with the flow rate of the carrier gas through the column being determined by the pressure that is applied to the inlet of the column. By rotation of the two three-way valves, the chromatograph is operated in the low pressure mode with the flow rate of the carrier gas through the column

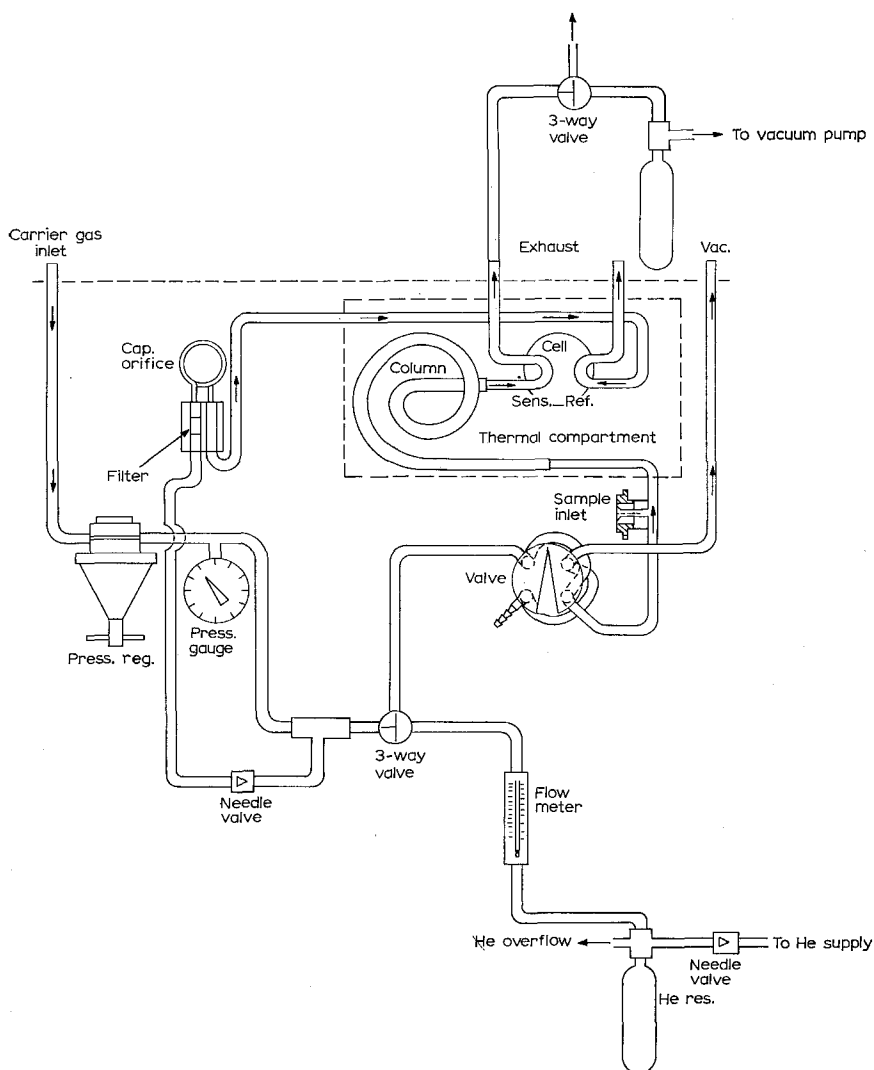


Fig. 1. A diagram of the modified gas chromatograph.

being determined by the amount of vacuum that is applied at the exit of the detector. The data collected were obtained with a vacuum of 26.5 in. of mercury at the pump.

The column used was stainless steel, 6 ft. by 2 mm I.D., packed with 10% SE-30 coated on 60-70 mesh HMDS treated Chromosorb W. The column temperature was held isothermal at 100° or 140° with a carrier gas flow rate of 24 ml per min for the normal mode and 20.3 ml per min for the low pressure mode. The detector current was 250 mA and the detector temperature was the same as the column, while the injection port was maintained at 208° for all samples. The sample size was 1 μ l for all compounds tested. The recorder used was a Hewlett Packard Autograf with Model 7101B input module. The input was 1 mV and the chart speed was 0.5 in. per min.

Results and discussion

The results presented in Table I show that the retention time of the compounds chromatographed under the low pressure mode was approximately half of what it was under the normal mode.

The gas chromatograms of the compounds obtained under normal and low pressure modes are shown in Fig. 2. It may be seen from this figure that the chromato-

TABLE I

RETENTION TIMES OF SOME COMPOUNDS RUN UNDER NORMAL AND LOW PRESSURE MODES

Compound	Column temperature (°C)	Retention time	
		Normal mode	Low pressure mode
Formic acid	100	0.52	0.38
Water	100	0.62	0.40
Acetic acid	100	0.90	0.42
2,2,4-Trimethyl pentane	100	1.00	0.58
Propionic acid	100	1.78	0.92
2-Octanol	100	5.80	3.18
Phenyl ethanol	140	4.38	2.19
<i>n</i> -Dodecane	140	6.82	3.81
Phenyl ethyl acetate	140	8.60	4.60

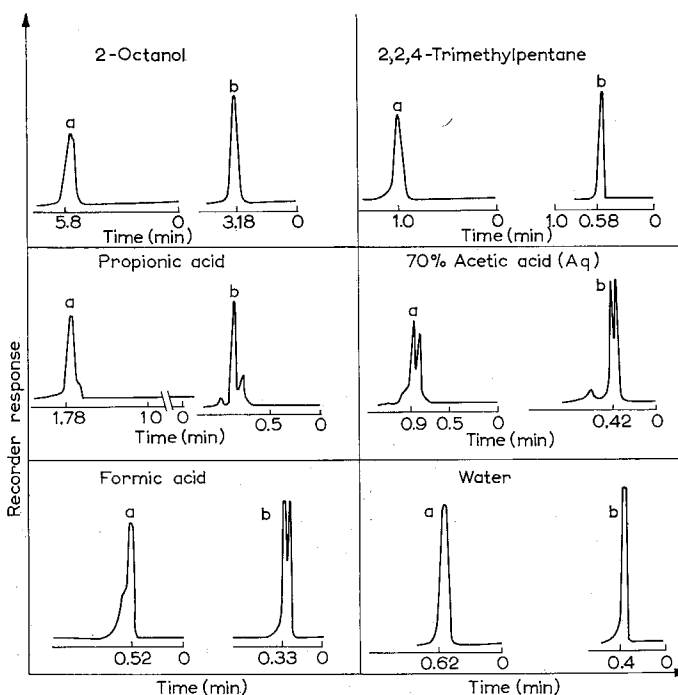


Fig. 2. Gas chromatogram of some compounds run under (a) normal mode, and (b) low pressure mode.

grams obtained under low pressure mode show sharper peaks than those obtained under normal mode. In the case of formic and propionic acids, the impurities which appeared as shoulders during the normal mode, gave sharp and well resolved peaks during the low pressure mode.

The method appears to hold out several advantages over the normal gas chromatographic analysis. Some of these would be the study of high boiling or low volatile compounds, and analysis of heat-labile compounds in addition to the faster elution and better resolution. A detailed work covering the study of these factors and others has been undertaken, and the results will be reported in subsequent communications.

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A new sampler for gas chromatographic analysis of liquids and solutions

According to LEIPNITZ AND STRUPPE¹, one of the most important prerequisites for exact analysis is that the amount of injected material and the method of introduction be as precisely reproducible as possible. The samplers presently available fulfil this condition only partially and also present cleaning difficulties, especially when viscous substances are used. Nor do they have a constant injection pressure, except when used with the "Reprojector"*. This reprojector, however, is even more difficult to clean than the other samplers. Thus we have tried to develop an improved sampler which eliminates these deficiencies and at the same time permits a high degree of reproducibility.

Fig. 1 shows the newly developed sampler** which consists of two parts: a syringe, which does not come into contact with the test mixture, and a calibrated component, namely an interchangeable cannula made of stainless steel. The syringe is a standard 2 ml syringe modified by the addition of a pressure relief valve. The method of operation is illustrated in Fig. 2. The 2 mm³ cannula containing the test sample (5) is attached to the cone of the air-filled syringe. Automatically the rear, wider end of the cannula (11) depresses the cylinder (8)—made airtight by a gasket (12)—of the pressure relief valve, thus effectively sealing the cannula. Next, the needle is inserted into the injector of the gas chromatograph and the plunger (2)

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