CHROM. 4991

An automatic device for injection of gas samples into a gas chromatograph

A study of the occurrence of gaseous hydrocarbon compounds in the soil atmosphere[1] has involved the analysis of a large number of samples by gas chromatography. To make more efficient use of the chromatograph, and reduce the time spent on manual operation, an automatic device for the injection of gas samples has been constructed.

Description of apparatus

The injection apparatus is shown in Fig. 1. Sixteen samples can be accommodated in glass syringes fitted with 22-gauge needles; the syringes are lubricated with a grease made from mannitol, glycerol and starch[2], which does not absorb hydrocarbon

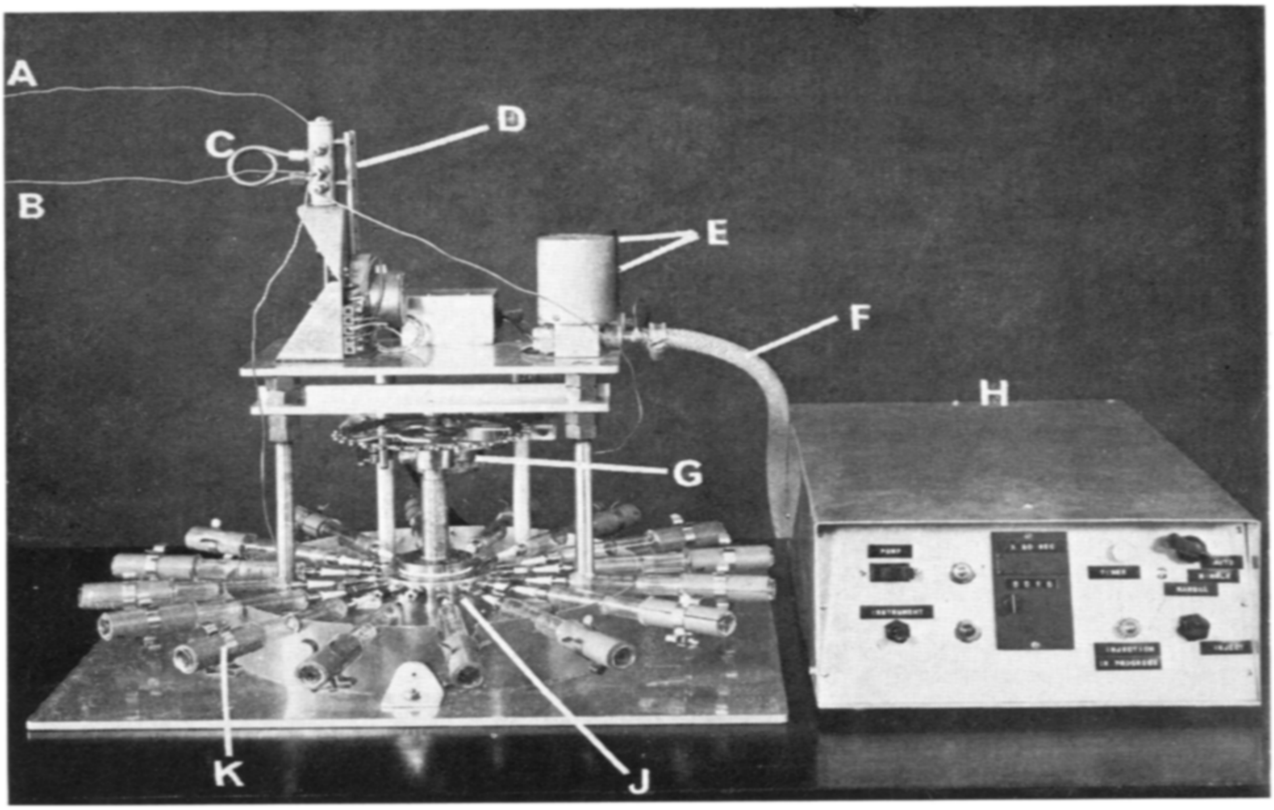


Fig. 1. Automatic gas-injection apparatus. Key: A = 1.6 mm (1/16 in.) O.D. carrier gas tube from chromatograph; B = tube carrying sample + carrier gas to chromatograph; C = sample loop; D = automatic sampling valve; E = solenoid valves; F = tube to vacuum pump; G = rotary valve drive motor; H = control unit; J = rotary valve; K = syringe collar.

gases to any significant extent. The needles are inserted through rubber septa in the inlet ports of a specially designed rotary valve (Fig. 2). The rotary valve is operated by an arm attached to the shaft of a synchronous motor turning at 1 r.p.m. The arm engages with a 32-tooth cycle sprocket attached to the valve rotor; thus each revolution of the arm turns the valve through 1/32 revolution. Each syringe, in turn, is connected

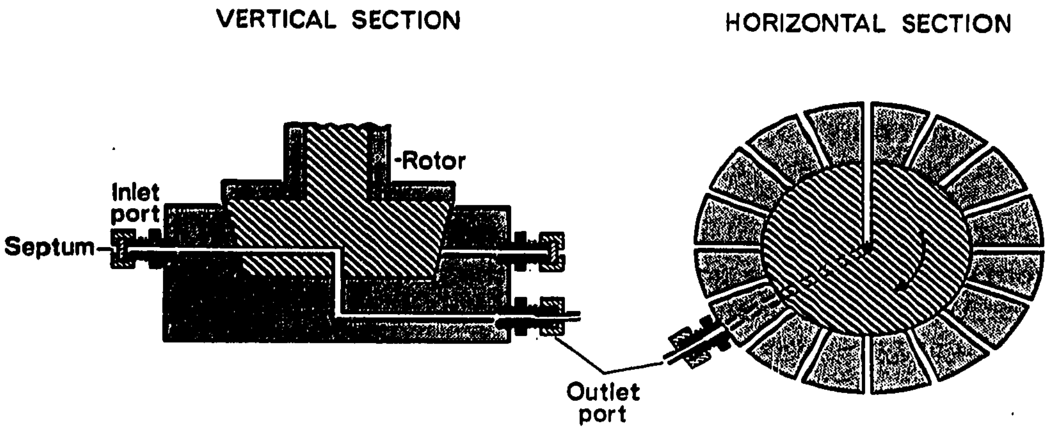


Fig. 2. Rotary valve of automatic gas-injection apparatus. The tapered end of the rotor is made of PTFE, and is fixed to a brass shaft; the body of the valve is brass. The sixteen inlet ports (not shown in the horizontal section) are made from Simplifix stainless steel couplings for 1.6 mm O.D. tubing, and fitted with latex rubber septa 1 mm thick.

by way of the outlet port of the rotary valve, and a 50-cm length of 1.6 mm O.D. stainless steel tubing, to the sample loop of a motor-operated gas sampling valve (Field Instrument Co., Richmond, Surrey, Great Britain), the loop having previously been evacuated and then sealed off from the pump by the closure of a solenoid-operated valve (Edwards High Vacuum Ltd., Crawley, Sussex, Great Britain, Type D11103). The gas connections are shown in Fig. 3.

Collars made from rigid PVC tubing are fitted to the syringe plungers to prevent them being forced in by atmospheric pressure when the syringes are connected to the evacuated sample loop, so that the total gas-filled volume, and the proportion of the sample which flows into the sample loop, remain constant. 5-ml syringes and a 2-ml loop have been used routinely, but any convenient combination of syringe and loop

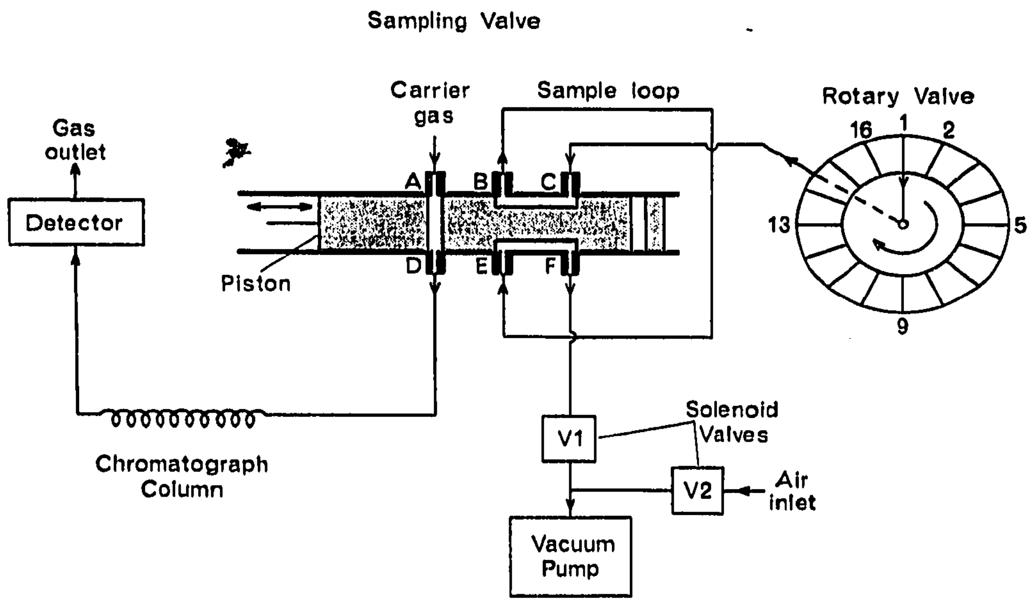


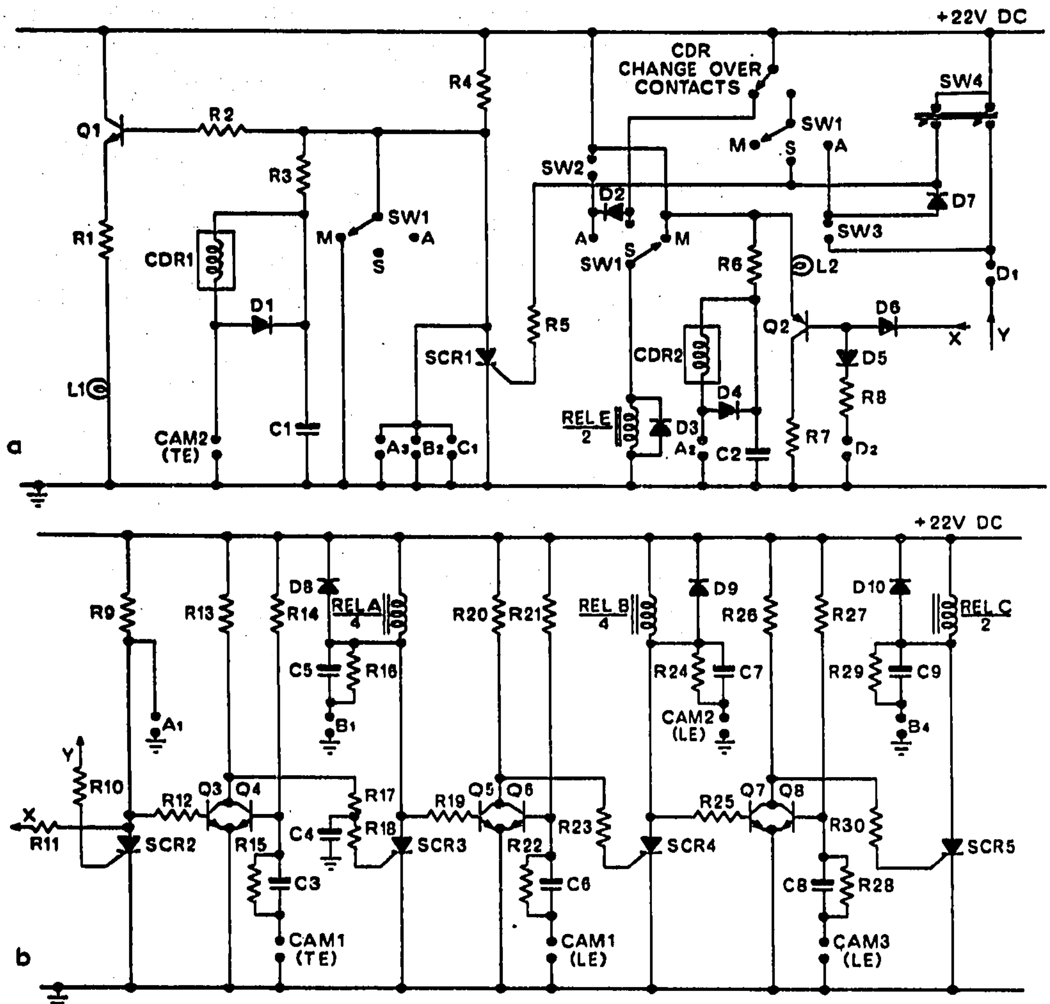
Fig. 3. Connections for the transfer of gas between components of the automatic injection apparatus and the gas chromatograph.

volumes may be employed. The gas in the sample loop is automatically injected into the chromatograph.

A 3-cam timing motor (2 r.p.m.) in the control unit produces a series of signals to initiate each operation, and to operate the countdown register of the timer which determines the interval between successive injections. The control circuits are shown in Fig. 4.

The sequence of operations is determined by the settings of the cams of the timing motor. All output signals are generated by the closure of the cam-operated microswitch contacts. When the instrument is in the "stand-by" condition, all relay contacts and microswitches are open, with the exception of B4, D1, SW2 and SW3, which are closed. After the "inject" switch SW4 has been operated the cam sequence is as follows: (1) Cam 1 trailing edge sets relay A; the closing of contacts A4 operates solenoid valve V1, which opens. (2) Cam 1 leading edge sets relay B, which, through contacts B3, closes the circuit to relay D. Contacts D3 start the motor of the rotary valve. Relay B (contacts B1) also resets relay A. (3) Cam 3 leading edge sets relay C (contacts C2) to start the motor of the sampling valve. (4) Cam 2 leading edge resets relays B and C. (5) Cam 2 trailing edge operates the count-down register.

Both the rotary and sampling valve motors continue to rotate until the micro-



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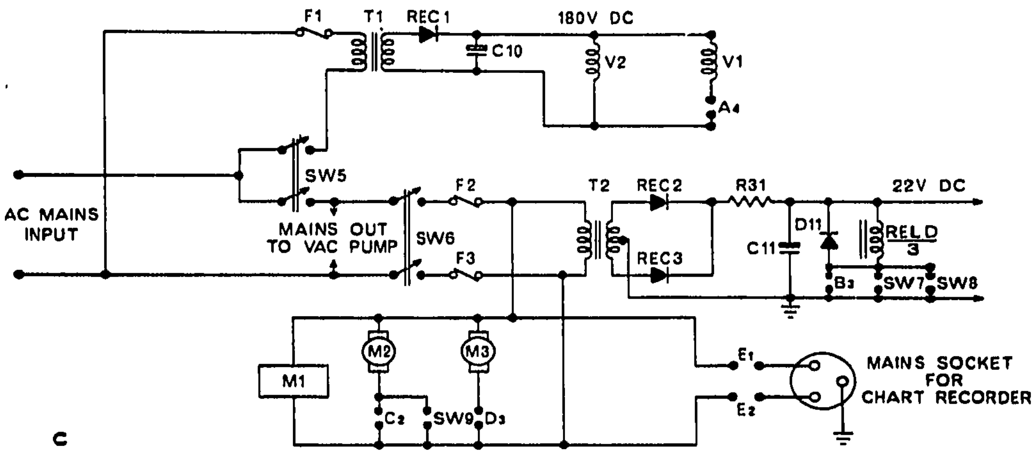

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Fig. 4. Circuit diagrams of the control unit. Key: SW1 = auto/manual mode switch; SW2, SW3 = "last sample" microswitches; SW4 = "inject" switch; SW5 = vacuum pump on/off switch; SW6 = control unit on/off switch; SW7 = "push to make–push to break" switch operated by rotary valve motor. M1 = 3-cam timing motor; M2 = rotary valve motor; M3 = sampling valve motor. CDR1 = count-down register coil; CDR2 = count-down register reset coil. LE = leading edge of cam; TE = trailing edge of cam. Components: R1, R7 = 330 Ω; R2, R17, R18, R23, R30 = 5.6 kΩ; R3, R15, R28 = 8.2 kΩ; R4, R9, R13, R20, R26 = 3.3 kΩ; R5, R8, R10, R11 = 6.8 kΩ; R6 = 2.7 kΩ; R12, R14, R19, R21, R25, R27 = 22 kΩ; R16 = 27 kΩ, R22 = 82 kΩ; R24, R29 = 100 kΩ; R31 = 10 Ω. C1, C11 = 500 μF; C2 = 1000 μF; C3, C6, C8 = 50 μF; C4 = 2 μF; C5 = 10 μF; C7, C9 = 1 μF; C10 = 60 μF. D1, D3, D4, D8, D9, D10, D11 = CV2290; D2, D5, D6, D7 = OA10. SCR 1, 2, 3, 4, 5 = TAG100 (Transistor AG). REC1 = 10D4 (Int. Rectifier); REC2, REC3 = 10D1 (Int. Rectifier). SW4 = DPST-loaded switch. CDR = Sodeco countdown register Type TCeF4PE. M1 = timing motor, Magnetic Devices Ltd. Model 8303B3/30S/3C6A/230-50; M2 = synchronous motor, Crouzet Type 395, 1 r.p.m.

switches operated by their shafts (SW8 and SW9, respectively), and switch SW7 on the rotary valve, have all returned to the "off" condition. The rotary valve motor thus makes two revolutions before coming to rest.

When the 3-position switch on the control unit (Fig. 1) is set to the "manual" position, a sample is injected only when the "inject" switch is operated, and the timer does not function; when set to "automatic", the apparatus will continue to operate until the sample in position 16 has been injected; if the unit is switched from "automatic" to "single sample" operation, only one further injection will be made.

The apparatus is switched off, after injection of the sixteenth sample, by microswitches operated by an arm attached to the rotating sprocket. This arm may be easily removed if it is desired to allow the rotary valve to operate for more than one complete revolution.

Sequence of operations

The rotary valve is at the intermediate position between inlet ports 16 and 1 (Fig. 3). The sampling valve is in the "fill" position (sample loop connected to ports C and F). The count-down register is set to give an appropriate time interval between injections. The solenoid valves V1 and V2 are closed, and the vacuum pump switched on.

The solenoid valve V1 is opened and the sample loop evacuated.

After 30 sec, V1 is closed, the rotary valve is turned to port 1, and the sample gas flows into the loop.

After a further 15 sec, the sampling valve is moved to the "inject" position (sample loop connected to ports A and D) and the sample in the loop is swept into the column by the carrier gas. The sampling valve is then returned to the "fill" position. The timer is reset at the beginning of the injection procedure.

The rotary valve is turned to the next intermediate position, between inlet ports 1 and 2.

When the count-down register of the timer reaches zero, valve V1 is opened, and the sequence is repeated.

After sixteen cycles (*i.e.* when the rotary valve has completed one revolution) the chart recorder is automatically switched off. When the control unit and the vacuum pump are switched off, or in the event of a mains power failure, valve V2 is opened to admit air to the pump.

Reproducibility of injections

In Table I the results are shown of two series of analyses, each involving sixteen successive injections of the same standard mixture of hydrocarbons in nitrogen. The standard deviations varied from 1.55 to 2.0% of the mean peak heights; these figures

TABLE I

REPRODUCIBILITY OF RESULTS OBTAINED BY AUTOMATIC INJECTION OF SIXTEEN SAMPLES OF A STANDARD MIXTURE OF GASES INTO A GAS CHROMATOGRAPH

Gas	*Concentration (p.p.m.)*	*Peak height (% full-scale deflection)*			
		Series No. 1 (12.5.70)		*Series No. 2 (14.5.70)*	
		Mean	*S.D.*	*Mean*	*S.D.*
Methane	6.1	46.34	0.77	46.45	0.78
Ethane	5.9	58.82	1.09	59.04	0.99
Ethylene	5.8	48.58	0.97	48.74	0.92
Propane	6.9	48.13	0.80	48.49	0.75
Propylene	7.9	31.45	0.55	31.53	0.51

indicate the upper limit of the errors associated with the injection process, because they also include any errors associated with sampling, and variations in chromatograph response and syringe volume.

The authors wish to thank Mr. P. M. LAY, Head of the Electronics Section of the Letcombe Laboratory, for his advice on the design of the apparatus, and Mr. F. W. HEPBURN for workshop assistance.

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Received August 10th, 1970