

CHROM. 4027

An automatic chromatography chamber

A number of devices have been described in the literature designed to initiate or terminate paper chromatography automatically or semiautomatically¹⁻³. BLAKE⁴ reported on a system which terminated the chromatographic process by detecting the mobile phase front and literally dumping the solvent from the chambers. The system is somewhat elaborate and cumbersome and as described was designed for ascending chromatography. The only mobile phase mentioned was water and no work had been reported with "low conductivity" mobile phases. At the time of preparation of this manuscript, HARDING⁵ published a note concerning a transistorized solvent front detector. This system, designed for electrically "conductive" mobile phases, triggers an alarm system indicating the necessity of intervention by the operator for termination.

This paper describes the design of a self-contained descending chromatographic system with circuitry able to detect the front of a variety of mobile phases and automatically terminate chromatography by removing the irrigating solvent. The system has been used successfully with mobile phases varying from highly conductive aqueous systems to organic mobile phases of relatively low conductivity.

Experimental

The electrical conductance of a number of chromatographic mobile phases used in this laboratory was determined by attaching a set of alligator clip electrodes to a sheet of Whatman No. 1 chromatographic paper in series with a model 269 Simpson ultra-sensitive volt, ohm, microammeter and a model-PS-4 Heath constant voltage power supply. The power supply was set at 150 V d.c. and mobile phases applied to the paper in a manner to allow slow diffusion to the electrodes. When the circuit was established, the current reading was recorded. The following table describes the composition of the various mobile phases used. Dielectric constants were determined using a 5 MHz Sargent Chemical Oscillometer.

TABLE I
MOBILE PHASE COMPOSITION

<i>Mobile Phase</i>	<i>Composition</i>	<i>Dielectric constant</i>
1	1-Butanol-water (84:16) + 0.25 % w/v PTSA	117 ^a
2	1-Butanol-water (84:16) + 2 % v/v piperidine	22.4
3	1-Butanol-glacial acetic acid-water (2:1:1)	44.2
4	1-Butanol-water (4:96)	75.8
5	Benzene-methanol-water (upper phase) (2:1:1)	2.35

^a This is an approximate value determined on the basis of a linear calibration between water and formamide.

Results and discussion

A block diagram of the automatic chromatography system is shown in Fig. 1. A basic feature of this system is the termination of chromatography by aspiration of the mobile phase from the stainless steel troughs. Since this is accomplished by activating a solenoid valve controlling a vacuum line, it was necessary to determine the

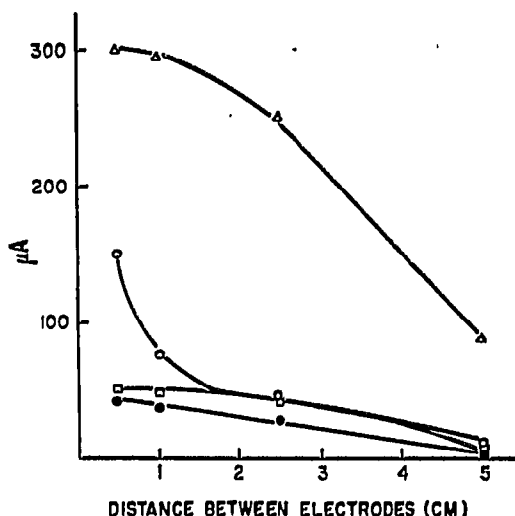
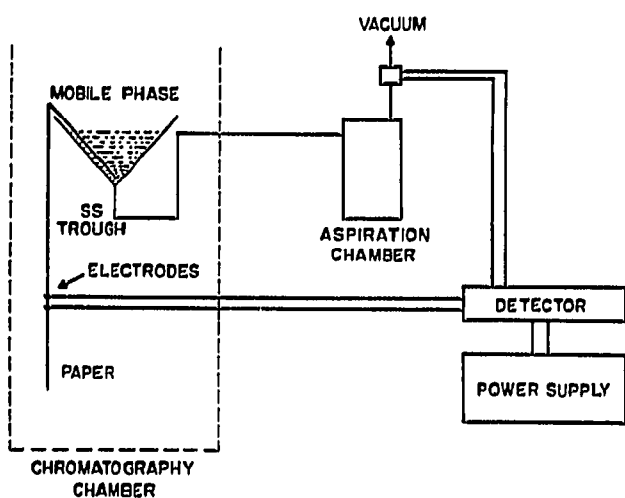


Fig. 1. Block diagram of the automatic chromatography system.

Fig. 2. Conductance of chromatographic mobile phases on Whatman No. 1 paper as a function of distance between detecting electrodes. (□) System No. 1; (●) System No. 2; (○) System No. 3; (Δ) System No. 4.

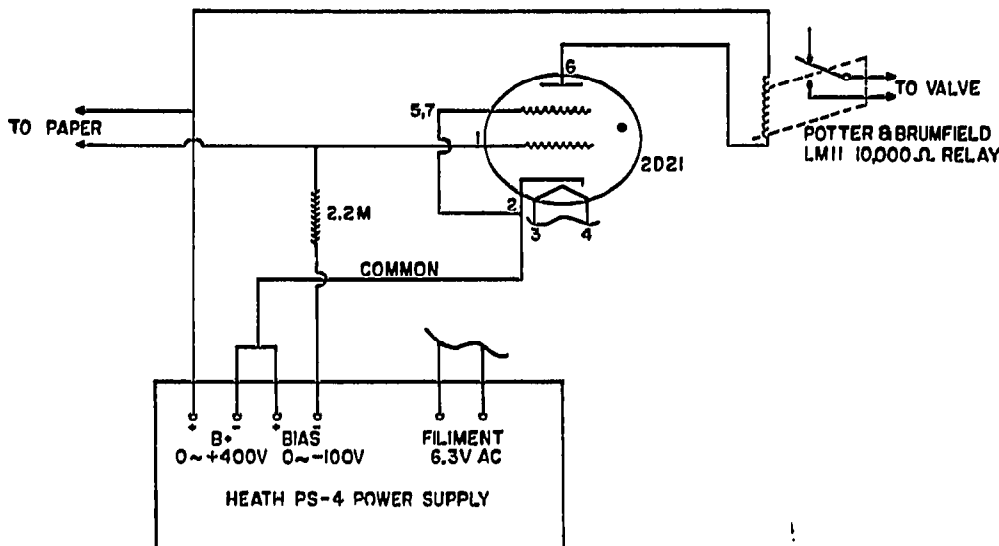


Fig. 3. Schematic diagram of the detecting circuit.

level of amplification required of the electronics to operate the valve. A plot of the current flow as a function of distance separating the detecting electrodes is shown in Fig. 2. These data were used as design parameters. The circuit shown in Fig. 3 is used to activate the valve whenever the solvent front reaches the electrodes attached to the paper. In the absence of solvent, the high resistance between the electrodes keeps the thyatron biased in its nonconductive state. Arrival of the mobile phase front decreases the resistance between the electrodes and results in a rising potential at the control grid of the 2D21 thyatron. When this potential reaches a value of about -2 V the thyatron fires, activating the relay. The contacts of the relay switch a solenoid valve, which controls the vacuum line, aspirating the mobile phase from the stainless

steel trough. Most design work and testing of the system was carried out using mobile phase 3 with electrode distances greater than 1 cm and under these conditions premature firing of the circuit was observed. This was traced to saturation of the chromatographic paper by vapors of the mobile phase. Normally the grid voltage (C-) in the thyratron circuit is held at -25 V, sufficient to prevent firing of the tube except by conductive mobile phases. However, the combined acetic acid and water vapor in system 3 produced sufficient resistance drop in the paper to establish a circuit. It was found that by decreasing the C-voltage, firing of the thyratron could be prevented until a liquid bridge existed between the electrodes.

Two features of this device make its operation especially suitable for solvent front detection and automatic control. First, since the bias supply is adjustable, solvents covering a wide range of conductivities may be used. The correct bias voltage for a particular solvent at any fixed electrode separation is determined experimentally, and it is found that the circuit will operate in aqueous systems as well as organic mobile phases of relatively low conductivity. Second, a thyratron will remain in the conductive state, once fired, until its plate supply voltage is removed or lowered to an extremely low value. Thus the vacuum control solenoid will remain in the open position after it has once been actuated regardless of any future changes in the resistance between the two conducting electrodes. This feature insures that the mobile phase will be aspirated completely once a solvent front initially triggers the device. In addition, since the solenoid remains in the open position, air is continuously pulled through the chamber drying the chromatogram before removal. Positive control of a wide variety of conditions is therefore assured without operator intervention.

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