An automatic fraction collector control and fraction counter

Numerous fraction collectors are commercially available for collection of chromatographic fractions. To the author's knowledge, however, none of these are completely automated to make the most efficient use of the limited number of tubes in a circular turntable. Furthermore, most of them make use of stepping relays which are subject to mechanical failure and possible overflow of inflammable solvent unless proper precautions are taken. An inexpensive (under $100.00) fraction collector control has been designed which eliminates the use of stepping relays and which has been in operation in the author's laboratory for over three years without a failure. This control will automatically (1) start the turntable after a predetermined number of forerun fractions have been syphoned into a waste container, (2) stop the turntable in the middle of a run and again empty a predetermined number of fractions into the waste container, (3) again start the turntable and collect the final fractions, and finally (4) shut off the flow of liquid from the chromatographic column, turn off all electric power to the equipment and record the elapsed time for the run. This control has made it possible to start a chromatographic separation at the close of a day's work and return the next morning to find it completed and the fractions ready for analysis. The fraction collector for which this control was designed has a large turntable with a single row of holes for tubes which is powered by a 0.6 A Bodine 1725 r.p.m. motor geared down to 24 r.p.m. Equal volume fractions are delivered from the column by means of a syphon and a stainless steel bellows pump is used to force solvent through a starch column[1]. Gravity alone is sufficient for a cellulose powder column[2].

Construction and operation of control

The wiring diagram for the control is shown in Fig. 1a. Fig. 1b shows the physical appearance of the control and Fig. 1c the upper and lower turntable control microswitches which are tripped by knobs attached to the edge of the turntable and extending above and below the table, respectively. All microswitches in the diagram are shown in their normal untripped positions. When the turntable is at rest the turntable microswitch is in the dotted position, actuated by a knob on the turntable motor shaft. When the turntable is in motion this switch assumes the normal position shown in the diagram. The power is 115 V a.c.

In operation the syphon contacts are closed by a salt solution or mercury and relay (3) closes. This activates counter (1) which counts down from the number of forerun fractions manually set on it each time the syphon empties until it reaches zero and activates its microswitch (shown immediately below it in the diagram). These fractions are emptied into a waste container placed below an empty hole in the turntable. The next signal from the syphon starts the turntable motor via the circuit: relay (3), upper turntable control microswitch, counter (1) microswitch, thermal delay relay (4) and turntable microswitch in dotted (rest) position. As soon as the turntable starts moving the turntable switch returns to its normal position and the thermal delay relay is by-passed. At approximately the mid-point of the turntable motion (1 sec) the thermal delay relay opens and when the knob on the turntable motor again trips the turntable switch it stops the turntable. The syphon then empties, the coils of the relays are inactivated and they both return to their normal positions for the next fraction. This operation continues until the upper turntable control microswitch is thrown by a knob extending above the turntable and attached at a point where a wide band of pure solvent occurs. The next signal from the syphon now activates counter (2) via the circuit: relay (3), counter (2) microswitch, upper turntable control microswitch, counter (1) microswitch, thermal delay relay (4) and turntable microswitch (dotted rest position). The tube at this stop of the turntable is omitted so that the number of fractions set on counter (2) will be emptied into the same waste container which collected the forerun fractions. This solvent can be re-used in another run without redistillation. When counter (2) counts to zero it trips its microswitch (immediately below it in the diagram) and the turntable again starts turning via the circuit: relay (3), counter (2) microswitch, upper turntable micro-switch, counter (1) microswitch, thermal delay relay (4) and turntable microswitch (in dotted position). With the turntable in motion the turntable switch again by-passes the delay relay and the upper turntable control switch returns to its normal position powering the turntable motor directly through counter (1) microswitch, by-passing counter (2) microswitch. Fractions are then collected until the lower turntable control microswitch is tripped by a knob attached to the edge of the turn-table at the end of the set of tubes and extending down from the table. This shuts off all power to the system thus stopping the pump and clock and at the same time ac-tivates the electrocock which stops flow of liquid from the column.

The various components of the circuit can be conveniently housed in a sloping front cabinet 7 in. square and 6.5 in. high. The outlets in the cabinet are essentially in the same order as in the wiring diagram with the excepton of the upper and lower

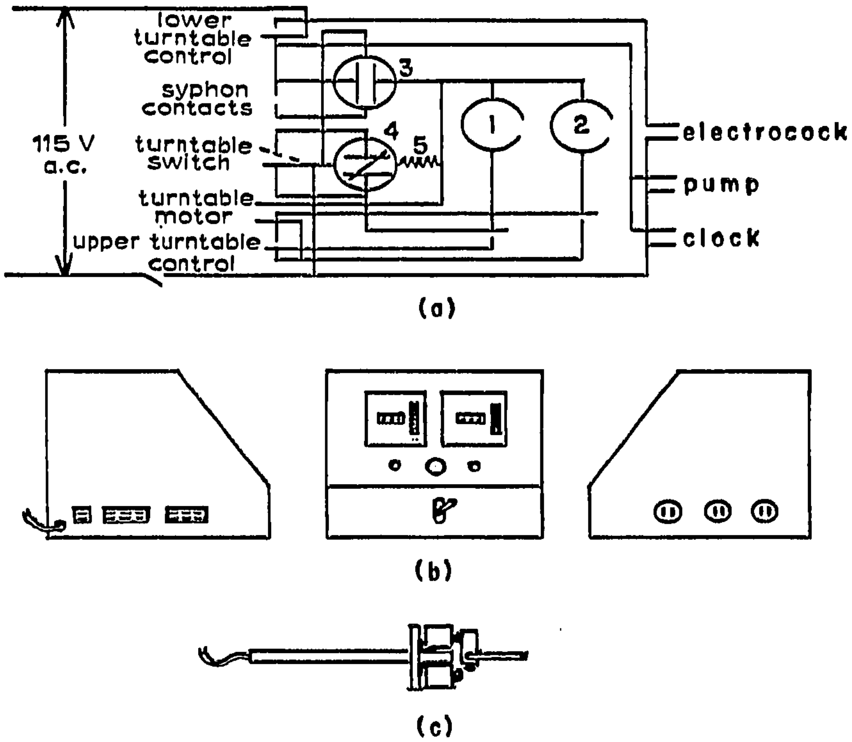

Fig. 1. (a) Wiring diagram for fraction collector control. (b) Physical layout of control. (c) Upper and lower turntable control microswitches.

turntable controls which are combined in one six-contact polarized outlet and six-wire cord to the turntable control microswitches. The turntable microswitch and turntable motor are fed by five wires in a second six-wire cord from a second six-contact polarized outlet. In Fig. 1b are shown two non-locking pushbutton DPDT switches, one immediately below each counter, and a pilot light in the center of the sloping panel. These are omitted from the wiring diagram for the sake of clarity. Each switch is wired so as to isolate the normally closed contacts of its counter microswitch and put them in a circuit with the 115 V pilot light so that the counter reset can be properly manipulated to put its microswitch into its normal position for the start of a run. When properly adjusted the pilot light goes on when either pushbutton switch is depressed.

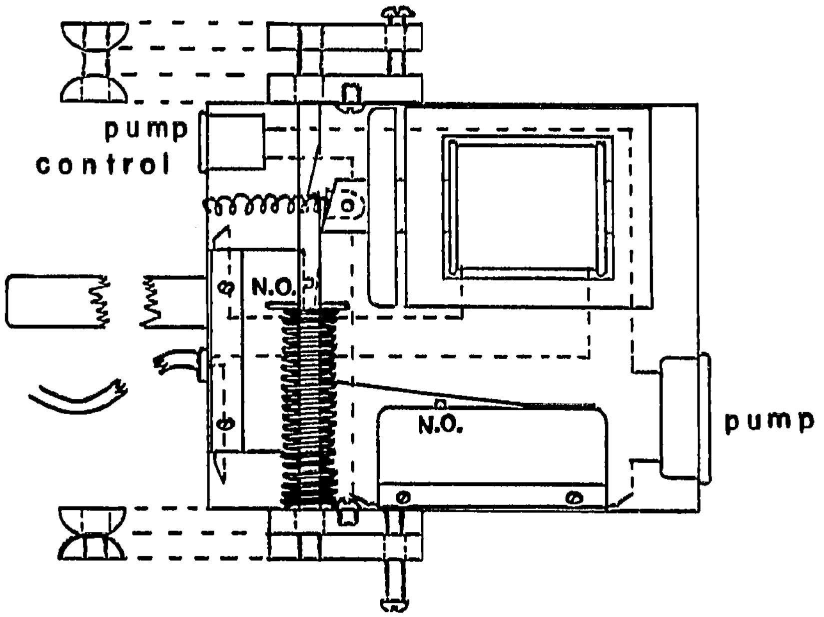

Fig. 2. Layout and wiring of electrocock.

Construction and operation of electrocock

For stopping flow of solvent from the column at the end of a run an electrocock was designed which would cut off its own power after closing. Fig. 2 shows the arrangement and wiring in a 3 × 4 × 4 in. box attached to a $^1/_2$ in. rod. The electrocock is shown in the closed position, the rubber tubing from the chromatographic column being pinched off between the lower rounded jaws. The electromagnet is an inexpensive 2 × 2 in. size with a 26 Ω coil for operation on 115 V a.c. The electrocock is opened by pressing on the upper jaw so as to compress the main spring and allow the core of the electromagnet to be drawn by its spring into the notch in the $^3/_{16}$ in. brass rod connecting the two jaws. The microswitches are closed by this action. Activation of the electromagnet draws its core in causing the jaws to snap shut and the microswitches to open. The microswitch in the pump circuit was included so that the pump would automatically start and stop when the electrocock was opened and closed without need for the rest of the fraction collector control power to be shut off. A single double pole microswitch would have served the same purpose. The upper jaws of the electrocock may be used to automatically start a flow by running a rubber tube through them and then pressing them closed. They are adjustable for different thickness of tubing by removing the screw and screwing the jaw out or in on the brass connecting rod.

Component list for collector control

2 counters, 115 V a.c., SPDT, manual reset, Veeder Root BX-150703;
1 relay, 115 V a.c., SPDT, 2–3 V.A., Advance Electric GHP/1C;
1 relay, 28 V, 1 sec thermal delay, G-V HM-O1;
1 wirewound adjustable resistance, 25 W, 750 Ω set for 600 Ω to supply delay relay coil from 115 V a.c. line;
2 non-locking pushbutton switches, DPDT, 3 A;
1 pilot light, 115 V a.c.;
2 AB sockets for chassis mounting, 6-contact, Cinch-Jones S-306;
2 CCT polarized plugs with cable clamps, 6-contact, Cinch-Jones P-306;
1 AB socket for chassis mounting, 2-contact, Cinch-Jones S-302;
1 CCT plug with cable clamp, 2-contact, Cinch-Jones P-302;
3 standard 2-pole female sockets with retaining rings, Amphenol.

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1 D. F. MOWERY, Jr., *Anal. Chem.*, 31 (1959) 1911.
2 D. F. MOWERY, Jr., *Anal. Chem.*, 29 (1957) 1451.

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Gas chromatography of hydroxyethyl derivatives

Hydroxyethyl derivatives are of diverse and widening interest in industry, agriculture, medicine and metabolic studies. The literature is fairly abundant with investigations concerning the role of C-hydroxylation, as well as N-hydroxylation, in the metabolic pathways for pesticidal carbamates[1–4] and 2-chlorotriazines[5–12]. N-(2-Hydroxyethyl) ethyleneimine has been proposed as a modifier for nitrogen plastics and other resins, and as an intermediate in the preparation of their polymers[13]. β-Hydroxyethyl hydrazine has been used as a growth regulator by pineapple growers to induce flowering and control the date of harvest[14,15]. A nitroimidazole derivative, 1-(2-hydroxyethyl)-2-methyl-5-nitroimidazole (metronidazole) is employed extensively as a specific agent against human trichomoniasis[16]. The technique of β-hydroxyethylation has been found useful for the characterization and enhancement of water solubility for complex molecules such as rutoside[17]. A number of *p*-acetylbenzenesulfonylureas utilized in diabetes therapy have been shown to be rapidly absorbed and metabolized in man by reduction to their corresponding *p*-α-hydroxyethyl derivatives[18].