MAGAZINE FED FRACTION COLLECTOR FOR MULTI-COLUMN LIQUID CHROMATOGRAPHY

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We have previously[1] described a fraction collector constructed specifically for use in multi-column liquid chromatography. The great simplicity in design with only one moving part besides the test tube carrier and an industrial type, heavy duty timer as the only electrical component has made this type of collector a highly reliable tool in multi-column work in our laboratory. One of these collectors has proved the soundness of the design by performing without breakdown in daily routine use over the last five years.

This type of collector has, however, been found to have one serious drawback that becomes particularly conspicuous in large scale multi-column work where a number of multi-column collectors may be needed simultaneously. It requires as much as 16 ft. of floor or bench space if it is to be left unattended for overnight use when loaded with 100 or more test tubes for each column in a multi-column set.

We have built a new type of multi-column collector to overcome this handicap. In this new model, test tube carriers pass from one magazine tower to another with the collection taking place from the columns placed in a thermostated water bath located between the magazines. A much more condensed and practical multi-column collector has been developed this way. The magazine fed collector fits conveniently on top of a laboratory bench and a 12-column collector of this type occupies only slightly more space than many conventional fraction collectors built for single column collections. Of necessity some of the simplicity in design of the earlier type multi-collector is lost in this new fraction collector, but care has been taken to avoid complex machinery and considerable efforts have been made to construct a fail-safe and simple piece of apparatus. The general design of a 12-column collector of this type is shown in Fig. 1. The test tube carriers, built essentially as described earlier[1] are loaded with empty clean tubes and stored in the magazine tower to the left in Fig. 1 with two of the carriers placed on the rails, one resting against the release–engage mechanism[1], the other right behind it on the rails. The eluants from the capillary columns[2] kept on a shelf in a thermostated waterbath located immediately over the latch are pumped through capillary teflon tubing into the front row of the test tubes in the carrier. The release–engage latch moves the carrier forward one row at a time on signal from the timer[1] when the appropriate fraction has been collected, and this continues until the tubes in the carrier are filled and the carrier is released. It then runs down the short distance until it hits the stop block at the end of the rail. The test tube carrier activates a microswitch here that starts the motor located at the bottom left in Fig. 1. This motor moves the filled test tube carrier to the right on the rail

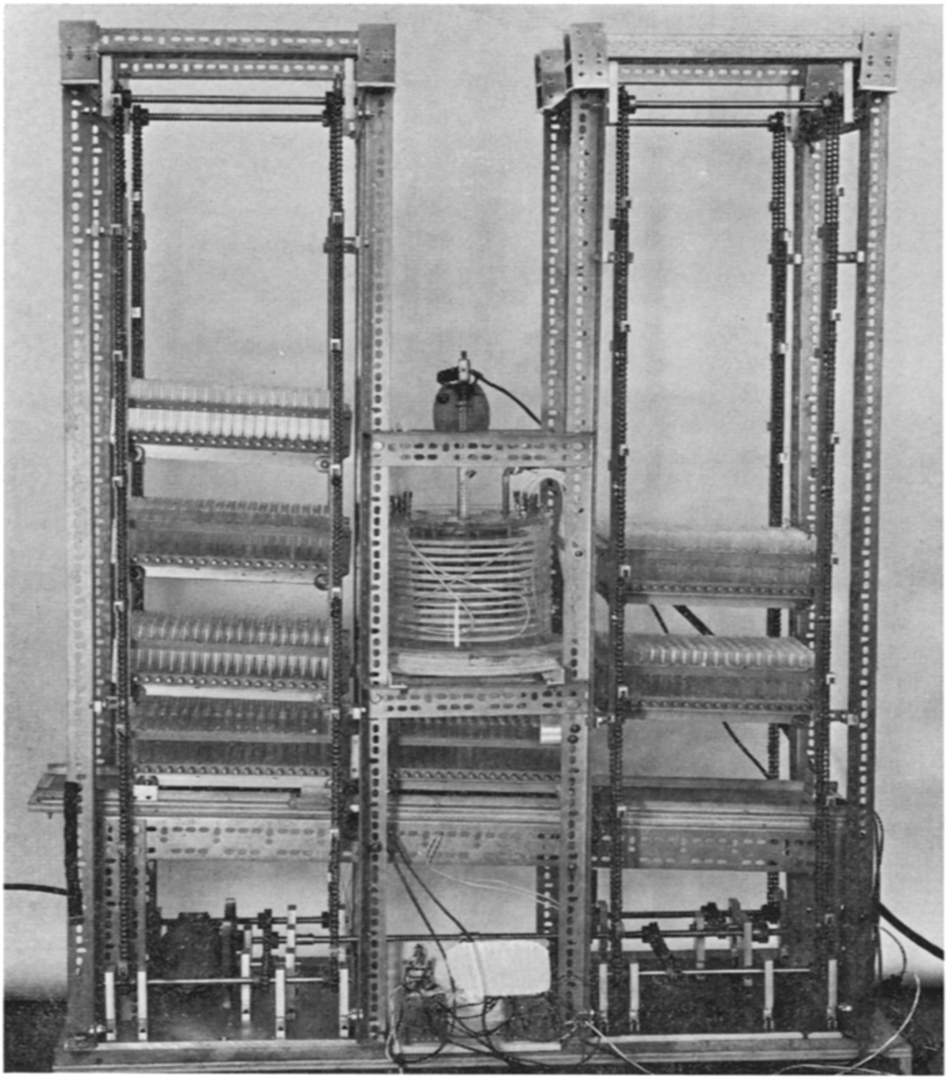


Fig. 1. The magazine fed multi-column collector. The magazine feeding the collector is to the left, the storage magazine for the filled tubes to the right. The capillary columns are placed in a thermostatted, heated and refrigerated waterbath located between the two magazines. The drive mechanism is located underneath the rails to the left, and the electrical circuitry is at the bottom in the center.

up into the right magazine tower and at the same time deposits a carrier with empty tubes to the left on the rails. This manouver is repeated, if necessary, until the storage magazine to the left is empty. This benchtop model holds 140 test tubes for each of the 12 columns or a total of 1680 test tubes, which in this type of collector, represents the maximum for unattended overnight runs. Floor type models can be built with larger storage capacity.

DETAILS OF CONSTRUCTION

Test tube carriers

These are somewhat modified from the original design[1] by the use of heavier material all through. We use now 1/8 in. thick aluminum plate fitted around a frame of 1/2 in. × 3/8 in. aluminum bar and 1/4 in. stainless steel rod for the pins.

The release–engage mechanism

This has consequently also had to be made heavier by the use of heavier material and a stronger solenoid.

The magazine towers

These towers (see Fig. 1) are constructed from 12 gauge 2¼ in. × 1½ in. slotted angle iron (Lyon, Aurora, Ill.). The test tube carriers are held in grooved holders, machined to fit the pins on the carriers. The holders for the carriers are attached—in each tower—to four vertically running roller chains (Morse, Ithaca, N.Y., No. 35 Riv., 3/8 pitch) that are stretched out between upper and lower sprockets (Morse, 3/8 pitch No. 35B12), mounted on half inch drill rods held in ½ in. × 1½ in. × 5 in. aluminum plate brackets attached to upper frame and bottom plate and running on ball bearings (Nice Ballbearing Co., Nicetown, Pa., No. 1616DS).

The drive mechanism

The power from a motor, an 80 r.p.m. Janette 1/12 h.p., 115 VAC Motor (Janette Electric Co., Boston, Mass.), is transferred through a 1:4 spur gear reduction (Boston Gear Co., Boston, Mass., No. NB 30B and No. NB120) to a central drive shaft from which a chain drive pulls vertical chains carrying the test tube carriers in such a way that the chains move in opposite directions, in the two magazine towers. The movement in the chain system is adjusted so that a test tube carrier is delivered to the rail on the left side and another simultaneously removed from the rails on the right side in approximately 5 sec after activation of the electrical circuit controlling the change of carriers (Fig. 2, upper left).

An important detail that makes it possible to deliver the test tube carriers accurately to the rails are a set of four wedges located on the rails opposite the wheels of the carriers hanging in the chain drives (Fig. 2, upper right.) These wedges serve to align the wheels to the rails.

Electrical circuit

A schematic presentation of the electrical circuit is given in Fig. 3. The arrangement of the two time delay relays and the mercury relay that supplies the power to the motor is shown in detail in Fig. 2, lower left. The positioning of the two microswitches is given in Fig. 2, lower right. Power to the motor that drives the chain drives is supplied when a test tube carrier is released from the latch and moves down and hits the stop block at the end of the rail. This closes the normally open microswitch (Acro, Columbus, Ohio, No. B2-2RL2) located here, and this contact closure activates the Agastat time delay relay (Elastic Stop Nut Corporation of America, Elizabeth, N.J., No. 2422 0.8–15 sec) set for approximately 2 sec and current is for this period of time supplied by the closed contact of this relay to the coil in the mercury relay (Ebert Electronics, Queens, N.Y., No. MR-10), which in its turn closes the contacts to the drive motor. Power to the coil in the mercury relay is, after the time delay period has expired, supplied *via* the second normally closed microswitch located on the left magazine tower in such a position that the switch contacts open when in contact with a carrier holder on the roller chain. The motor will, therefore, continue to move the carrier until the holder hits the second microswitch and opens this switch so that current no longer is supplied to the coil in the normally open mercury relay

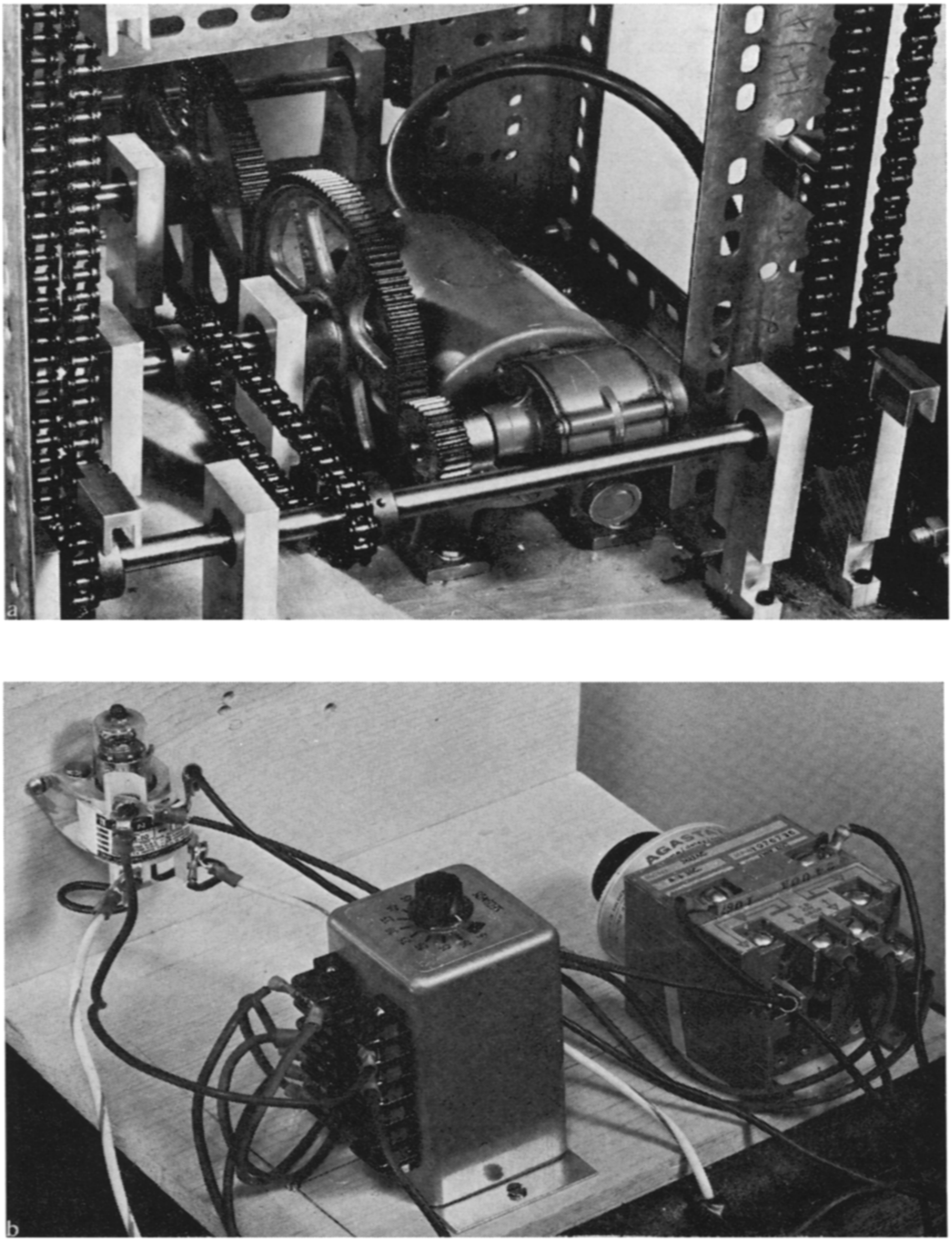
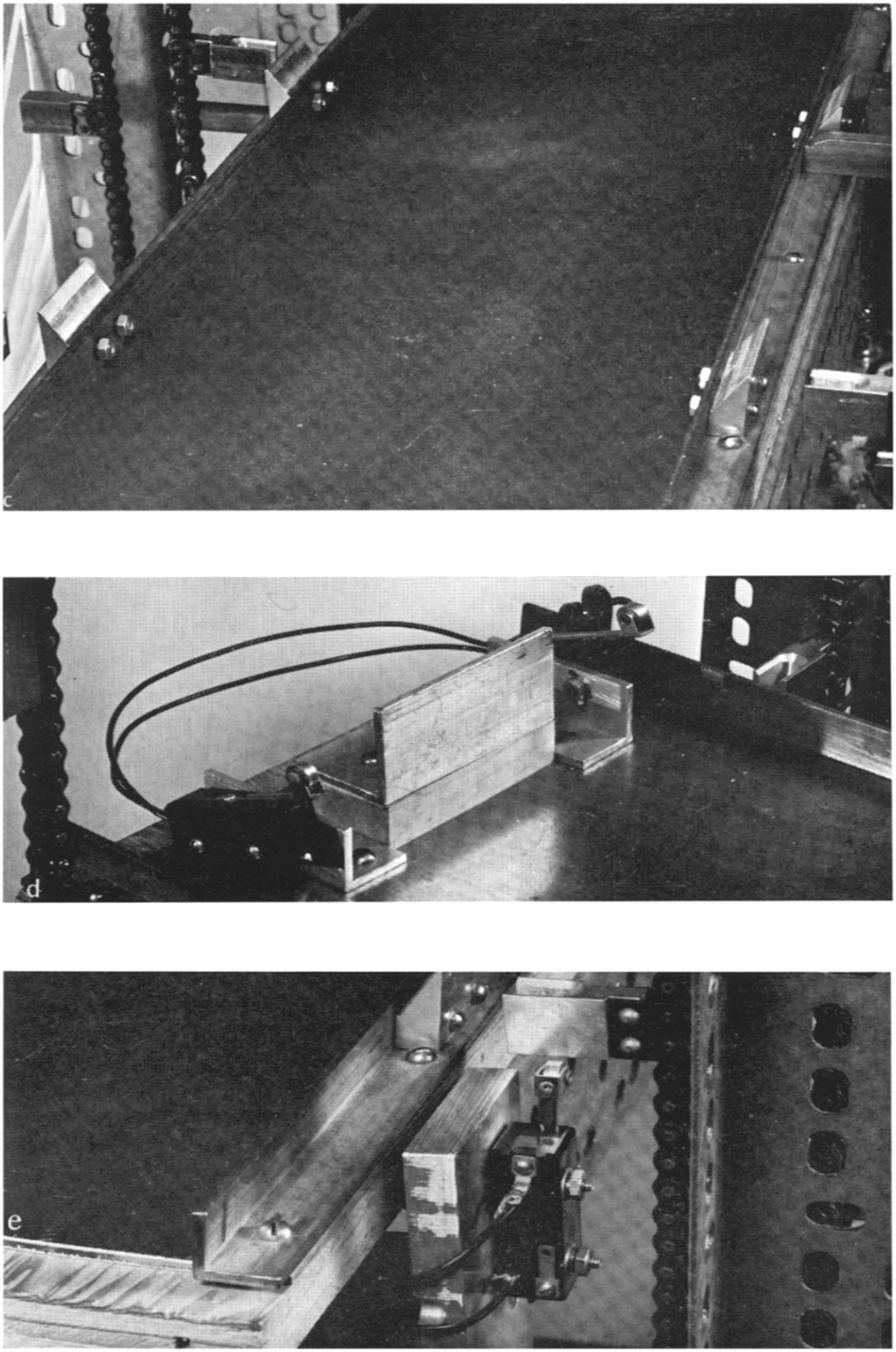


Fig. 2. Upper left shows the power transfer through reduction gears and chain drive to the vertical roller chains. Each of the larger gears drives a drive shaft in opposite directions, one for each magazine. Upper right illustrates the use of wedges bolted to the rails to force the wheels of the test tube carriers on to the rails. Lower left is a picture of the two time delay relays and the mercury relay controlling the motor. Lower right shows the positioning of the two micro switches, one is near the stop block at the end of the rails and the other is located on the frame of the left magazine so that it is activated by the holders on the vertical roller chains.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that supplies current to the motor. The current from this second microswitch is passed through the normally open contacts of the second time delay relay, a solid state Intermatic relay (International Register Co., Chicago, Ill., No. SS 11-D). This relay is activated simultaneously with the Agastat relay and is set so that it closes the set of contacts connected with the second microswitch and the coil in the mercury relay for a fraction of a second longer than it takes to deliver a test tube carrier to the rails. This second time delay is purely a safety feature, since this type of relay is accurate enough ($\pm$ 1 %) to ensure proper operation of the collector in the highly improbable event that the second microswitch, which is rated for several millions of operations and is used only 10–20 times a day, should fail. Failure, would however, lead to breakage of a number of the test tubes and we have felt it necessary to have the extra protection on this point.

Such protection can also be obtained by using two microswitches in series so that one of these switches will break the current if the other fails. It is similarly possible as a fail-safe measure to mount two microswitches in parallel for the initial activation of the time delay relay that starts the motor drive at the beginning of a carrier delivery cycle.

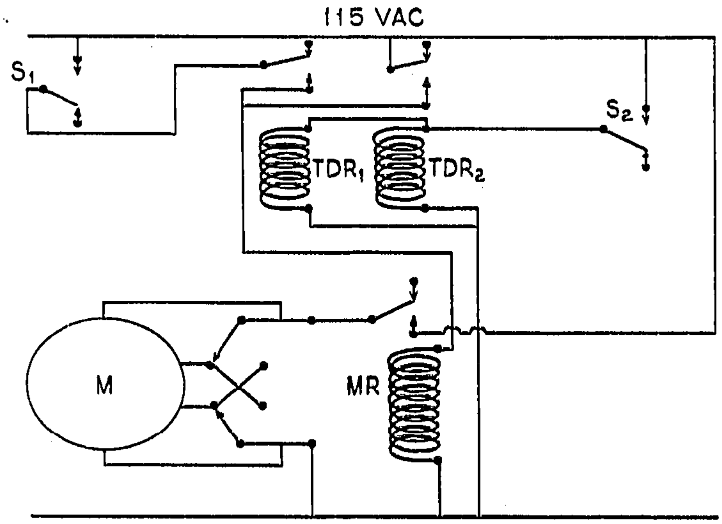

Fig. 3. This shows the electric circuitry. Current from the microswitch located to the right on the rails (S_2) passes through the two time delay relays (TDR_1 and TDR_2) on closure of the microswitch after release of a test tube carrier. The primary coil in the mercury relay (MR) is activated and this starts the chain pulling motor (M). This motor keeps running until the current is broken through the second microswitch (S_1) that is opened by the carrier holders on the chain in the left magazine tower.

The machinery is protected against test tube carriers running over the top by a microswitch mounted near the top of the frame. This normally open switch, upon closure of its contacts, activates a latching relay that breaks the current to the apparatus. We have as further protection on this point, used a heavy duty 55 A timer to supply current to the apparatus This timer has been set to cut the current off when the last carrier on the multicollector has been filled

The magazines in the collector can be filled and emptied at bench level by use of the reversal switch on the motor.

Positioning of the capillary columns

The development of teflon capillary columns for liquid chromatography[2] has made the condensed column arrangement illustrated in Figs. 1 and 4 possible. The columns are wound around a plastic rack as illustrated in Fig. 4 and the rack is placed in a cylindrical waterbath positioned on a shelf between the two magazine towers as

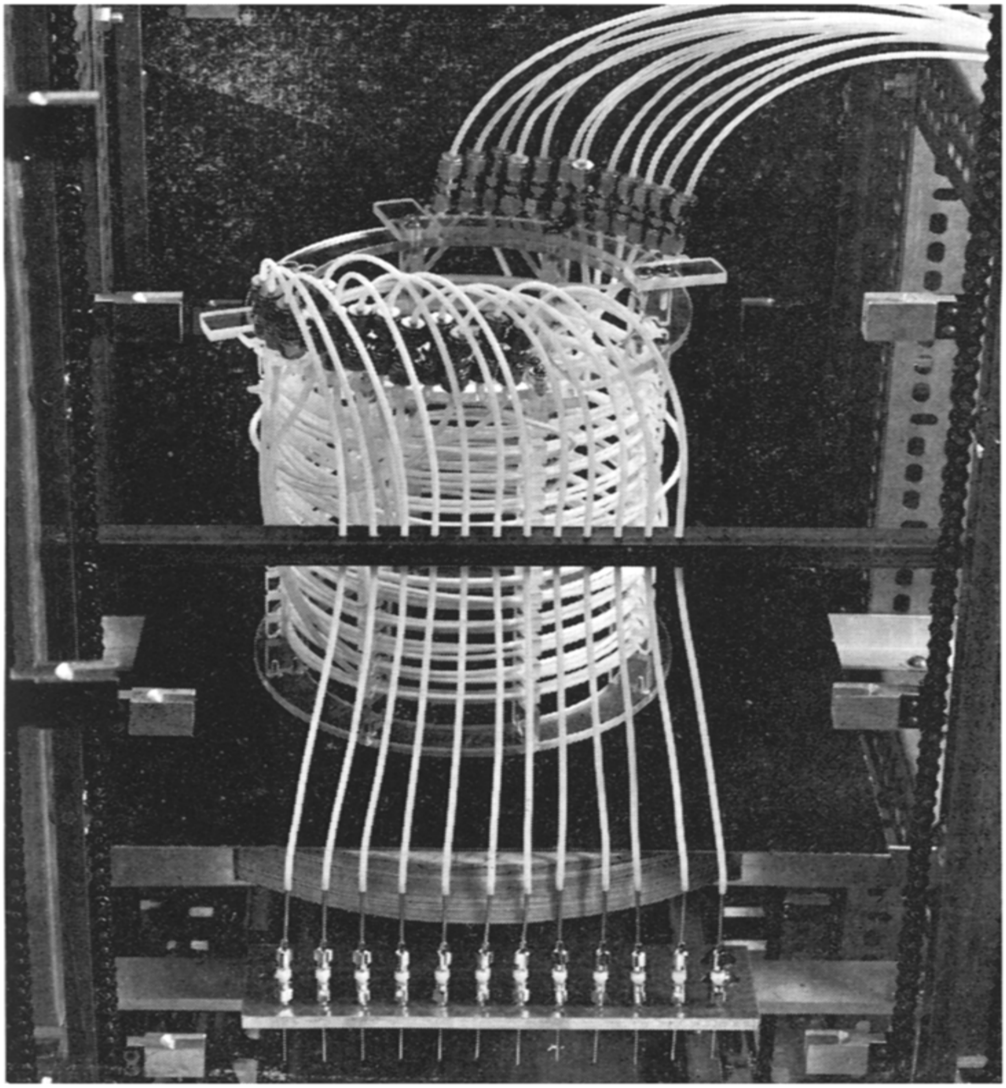

Fig. 4. Shows the arrangement of the capillary columns around a plastic rack with the connections to the pumps in the background and the short connections to the delivery tips in the foreground.

shown in Fig. 4. Both a refrigerated coil from a water bath cooler (Precision Porta-Cool) and a constant temperature circulator with heating element, thermostat and stirrer (Bronwill) fit in the center of the plastic rack holding the columns so that thermostatted operation of the columns is possible both below and above ambient temperature. An advantage of the condensed collector design is the small distance from column outlet to delivery tip. This keeps mixing in this section at a minimum.

DISCUSSION

The magazine fed multi-column collector in conjunction with the capillary columns opens the way for very large scale multi-column work. We have currently under construction, two 25-column collectors and we hope when they are built to be able to demonstrate that 100 or more liquid column chromatograms of, for example, steroid mixtures can be run within a 24 h period using the fast capillary column techniques described earlier[2]. An improved read-out system based on our earlier design[3] is under development and will make it possible to automatically record the chromatograms and computer-calculate values from that many column chromatograms in a day.

The potential of such systems both in medicine and industry, would appear to be considerable.

ACKNOWLEDGEMENTS

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SUMMARY

A redesign of a previously described fraction collector for multi-column liquid chromatography is presented. A more practical and compact design is obtained through the use of vertical magazines for the delivery of empty test tubes and for storage of filled tubes.

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