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Note

A rotating unit for preparing circular chromatographic plates at elevated temperatures

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The Harrison Chromatotron (Harrison Research, Palo Alto, CA, U.S.A.) is used to perform centrifugally accelerated preparative thin-layer chromatography. The instruction manual suggests that if the circular chromatography plates are rotated as they dry, a more uniform adsorbent layer will be produced which in turn will provide better separations[1]. A phonograph record player may be used to rotate these circular plates continuously as they dry at room temperature; the bands of separating compounds formed with such plates are regular and concentric to within ± 1 mm. We have found, however, that if layers of silica gel G are dried at room temperature while rotating and then developed on the Chromatotron with highly polar solvents, such as ethyl acetate–methanol–water (104:72:26, v/v/v), the plates may be used for only one separation. If a second run is attempted, we have repeatedly observed that the entire silica gel G layer separates from the plate, frequently as a single sheet. When the plates were, however, dried at 70°C with rotations through an arc of 120° at 5-min intervals, the layers did not separate from the glass plate, even after many uses with highly polar solvents [*e.g.*, methanol–water (3:1, v/v)]. Unfortunately, the layers produced in this way were manifestly less uniform than those obtained when the circular plates were dried with constant rotation at room temperature. Since commercial phonograph record players cannot be operated in 70°C ovens, we fabricated the unit described in this report to rotate the glass plates in an oven while they are drying. We have observed that plates dried at 70°C with constant rotation provide tighter bands of separating compounds than do plates dried at the same temperature with intermittent rotation. The unit can be fabricated and assembled for less than US $ 200.

MATERIALS AND METHODS

The unit is constructed from the following components: a Dayton right angle AC/DC gearmotor, Model number 2Z802A (Dayton Electric Manufacturing Co., Chicago, IL, U.S.A.); a 23 × 15 × 12 cm metal Bud box (Newark Electronics, Chicago, IL, U.S.A.); a 2-meter 18-3 power cord capable of tolerating 100°C for prolonged periods; an appropriately sized strain relief bushing (Heyco Molded Products, Kenilworth, NJ, U.S.A.); a standard 3-pronged plug; Plastilube No. 2 nonmelting grease (Warren Extruded Products, Cleveland, OH, U.S.A.); and a 24-cm diameter disc fabricated from a single piece of steel stock (Fig. 1).

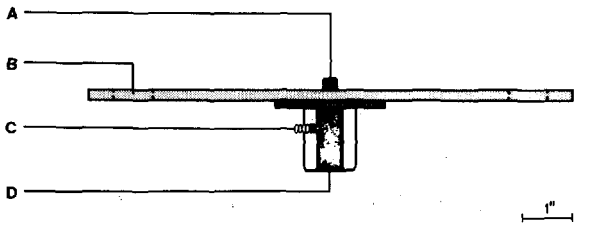

Fig. 1. Diagram of steel disc. (A) centering boss; (B) holes in disc; (C) 0.48-cm set screw; (D) collar.

The motor must be modified for operation at elevated temperatures. To do so, the motor is disassembled and the bearing and gear lubricant is replaced with a high temperature grease, such as Plastilube No. 2. The motor assembly is mounted in the

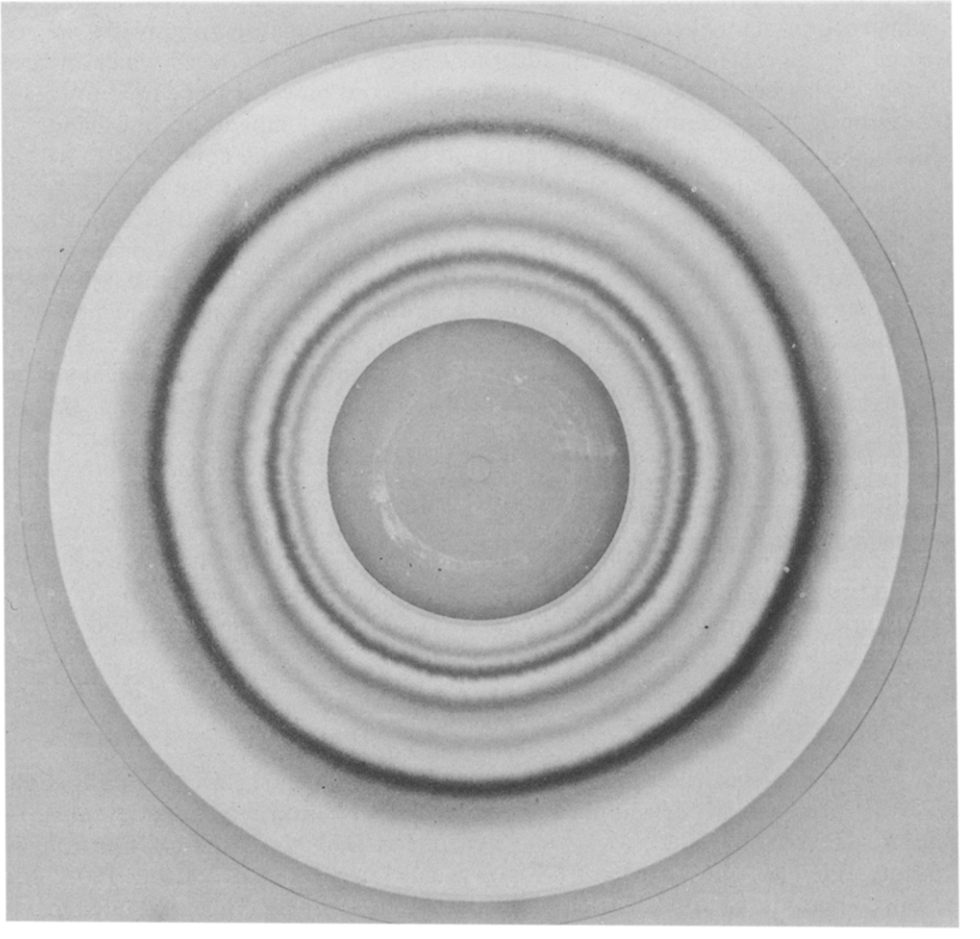

Fig. 2. Illustration of the separation of dyes obtained with the use of a circular disc dried with constant rotation at 70°C. Dye mixture number four (provided by Analtech, Newark, DE, U.S.A.; catalog number 30-04), was applied and the plate was developed with toluene as the solvent.

Bud box with three, 0.635-cm machine screws so that the shaft is positioned vertically. The ground wire is secured to the side of the box via a ring tongue and a 0.318-cm machine screw. The power cord is protected from accidental mechanical removal by securing it to the Bud box via a strain relief bushing. For convenience, a metal handle may be attached to the Bud box.

The steel disc is machined from a single piece of steel stock (Fig. 1) and has a centering boss (A) on the top to accept the hole in the glass Chromatotron plates. The collar (D) is secured to the shaft of the motor with a 0.476-cm set screw so that

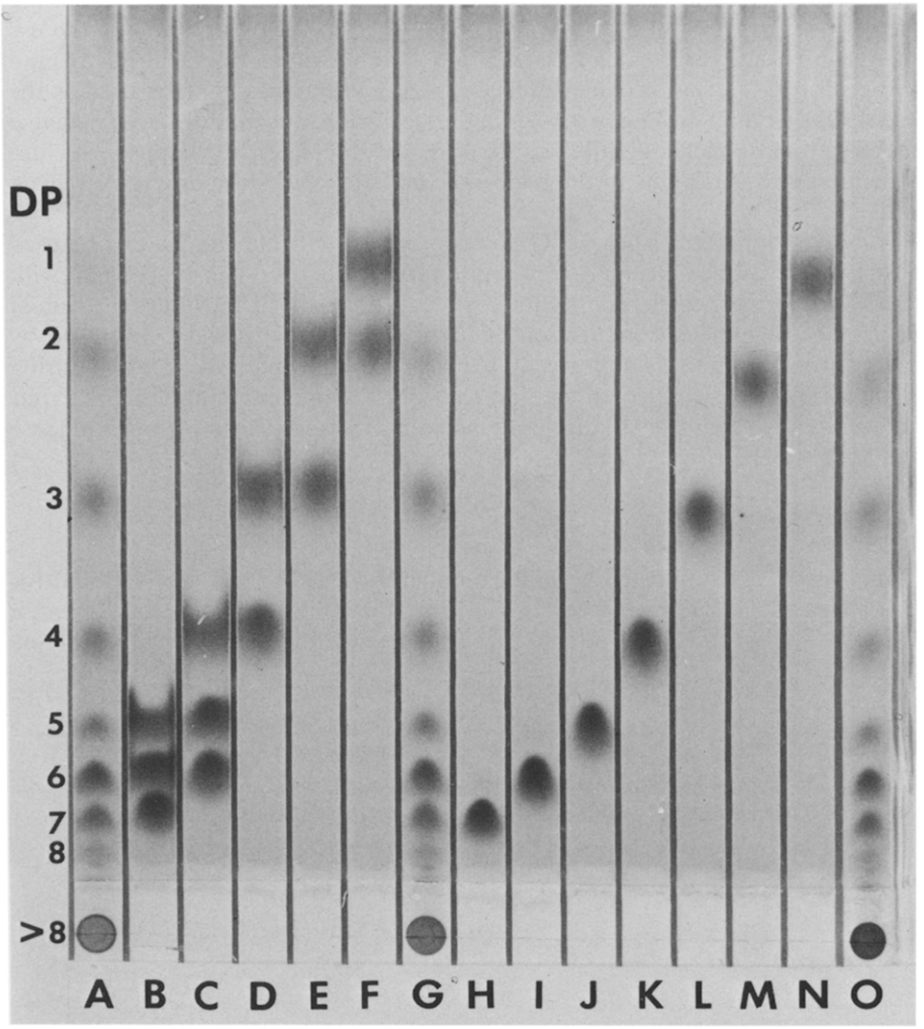

Fig. 3. Thin-layer chromatogram [solvent ethyl acetate–methanol–water (74:80:46, v/v/v)] of glucose oligomeric fractions obtained by centrifugally accelerated preparative thin-layer chromatography [solvent ethyl acetate–methanol–water (104:72:26, v/v/v)]. Lanes A, G and O contain a partial corn starch hydrolysate. Lanes B–F contain fractions isolated on a plate dried with periodic rotation at 70°C. Lanes H–N contain fractions isolated on a plate dried with constant rotation at 70°C. DP refers to degrees of polymerization, *i.e.*, the number of glucose units in the individual glucose oligomer.

the steel disc is positioned horizontally. To facilitate removal of the plate from the disc, two holes (2–2.5 cm in diameter) are drilled opposite each other near the outer edge of the plate (B). To prevent oxidation, the disc should either be chrome-plated or coated with a heat resistant paint. The assembled unit weighs 4.5 kg.

RESULTS AND DISCUSSION

There are several possible variations that may be employed in fabricating the unit. The use of flat cable between the motor and the plug would facilitate closing the oven door. The speed of rotation may be adjusted to the desired rate by the use of a speed control such as a standard laboratory variable transformer. We have found that a speed of 33–45 rpm provides excellent plates when silica gel G is used as the adsorbent. Rather than drilling two holes in the steel plate, one may prefer to machine the steel disc to a diameter slightly less than that of the plates. This approach eliminates temperature variations in the region of the holes, but still permits easy plate removal.

We have found that silica gel G layers prepared with this device can be used repeatedly with ethyl acetate–methanol–water (104:72:26, v/v/v) as the solvent; with such plates, a partial corn starch hydrolysate can be resolved into pure individual oligomers. The oligomeric composition of the various fractions was determined by thin-layer chromatography[2]. If, however, plates prepared by periodic rotation during the drying process are employed, we find that oligomers containing less than four glucose units elute from the circular plate as pairs, whereas those with 5–9 glucose units elute as triplets (Figs. 2 and 3).

ACKNOWLEDGEMENTS

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