The author expresses appreciation to Dr. W. E. GILSON for his valuable assistance in the mechanical perfection of the present apparatus.

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Received November 16th, 1959

J. Chromatog., 3 (1960) 488–491

Apparatus for collecting and drying organic acid fractions

Papers on the ion-exchange fractionation of organic acids have involved the use of commercial fraction collectors designed for collecting fractions in 18 × 150 mm test tubes[1–3]. These papers describe methods for evaporating the fractions in the test tubes in which they are collected. Evaporation is necessary in order to remove the volatile "swamping acids" used for elution before titrating the non-volatile residues. However, evaporating liquids in test tubes is difficult because of the low surface to volume ratio of the liquid, the long path the vapor must travel before leaving the tube, and the tendency of vapor to condense on the upper part of the tube. Test tubes are also inconvenient as titration vessels. The following describes a device which can be used on a Technicon Fraction Collector* for collecting 200 fractions in 25 (O.D.) × 50 mm shell vials and also simple methods and devices for evaporating and titrating the fractions.

Fraction collector attachment. Since the holes for inserting test tubes in the Collector Rack** were too close together for vials to be placed in the corresponding positions, a new pattern had to be made. It was found that to accommodate as many vials as there had been test tubes, the vials in the innermost circle would have to touch each other and those in the outermost circle be very close to the rim of the collector housing. This necessitated the vials being placed in four circles having radii of 20, 22.5, 25 and 27.5 cm instead of the normal 17.8, 19.7, 21.9 and 24.1 cm.

A disk 28.7 cm in radius was cut from 1.3 cm waterproof plywood and on it were drawn four circles having the above radii. Knobs were attached to act as handles,

* The mention of companies and commercial products in this paper does not imply that they are endorsed or recommended by the Department of Agriculture over others not mentioned.
** Capitalized names refer to the Technicon fraction collector or to parts of it as named in the Technicon instruction manual.

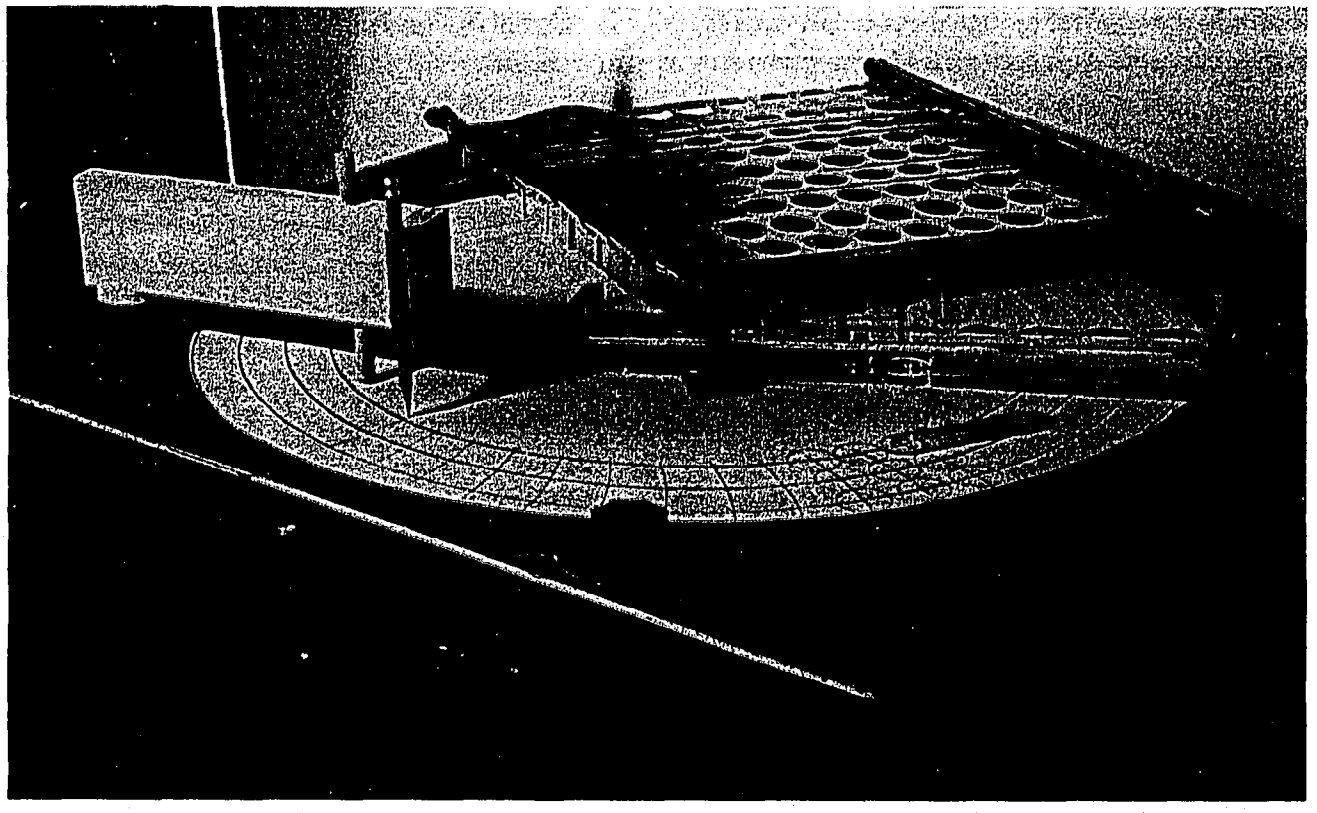

Fig. 1.

and the disk was attached to the Collector Rack by means of two screws. It was necessary to cut a narrow strip of metal from the back side of the Collector housing rim (heavy shears), because the Rack was not centered exactly under the housing opening. Dimensions are critical and should be checked against the individual Technicon before proceeding.

The Funnel Support Arm was replaced by a board drilled at one end to fit over the pins on the Funnel Arm Post and bearing on the other end a board attached at right angles to it and slotted to support a pencil in the vertical position (see Fig. 1). The latter board compensated for the greater radii of the circles of vials as compared with the circles on the Collector Rack. A dowel held in a hole drilled vertically through the board (friction fit) acted as a bearing to support the free end of the board. A thumb tack on the bottom end of the dowel allowed it to slide easily. The distance from the Post to the pencil (about 19.5 cm) should be such that at each position of the board (corresponding to positions of the Funnel Support Arm) the pencil is over one of the circles drawn on the disk. After ascertaining this, a wooden washer was placed under the board so that it could swing freely. Then, while operating the Technicon, the board and pencil attachment was used to draw arcs which intersected the concentric circles at the vial positions. The disk was removed from the Rack and 2.6 cm diameter, 1 cm deep, pits were drilled at these positions. The disk was then sprayed with an acid-resistant paint (thin coats!).

The Dropping Funnel was lengthened and bent so that its tip had the same

position as the pencil and was over the center of the pits at the various positions of the Collector Rack and the Funnel Support Arm.

The plywood disk worked satisfactorily, but some difficulty was presented by uneven thickness of the paint. There is also the possibility of future warping. A more durable, though more expensive, attachment for vials was made from 1.3 cm thick aluminum plate. This had as much of the center cut out as possible in order to reduce weight.

Evaporating and titrating fractions. Racks, each holding a row of ten vials, were made from two strips of aluminum sheet. The bottom strip was bent up at right angles at each end, and the top strip, containing holes for the vials and bent down along the sides for added strength, was attached to it by the tongue and slot method. The upright ends of the bottom strip were bent back to form slots through which rods could be passed holding the racks in groups of ten.

Drying chambers were made from four 48 × 41 cm pieces of plate glass (roof and floor of chambers) and three 43 × 8 × 4.5 cm blocks of wood (sides and central support). The chambers were set up in the front part of a fume hood, the hood window closed down to the chamber roof, and openings at the sides of the chambers were blocked off. This arrangement dried 200 1 ml-fractions in about 8 hours at room temperature. To increase evaporation rate a sheet-metal box containing two 500-W Chromalox air heaters was built. This, when placed in front of the drying chambers, raised the air temperature a few degrees and cut the drying time to 4 hours.

After the drying step the racks were separated by removing the connecting rods and the fractions titrated in groups of ten. One ml water and a micro stirring bar (Macalaster Bicknell Company, Millville, New Jersey) was placed in each vial. A strip of aluminum sharpened along one edge and painted white was slipped under the vials. The vials were stirred during titration by passing the rack over a magnetic stirrer. The stirring bars were easily transferred from one set of vials to the next by the use of a magnetized "pick-up" stick.

Acknowledgements. The author wishes to express his appreciation for help received from personnel of the shops and other divisions of the laboratory.

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Received October 19th, 1959

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