THE BRITISH RUBBER PRODUCERS' RESEARCH ASSOCIATION

Publication No. 73

A SIMPLE LABORATORY FALLING-FILM MOLECULAR STILL.

BY

E. HAROLD FARMER and D. A. SUTTON.

[*Reprinted from the Journal of the Society of Chemical Industry, June*, 1946, *Vol. LXV, pp.* 164—166.]

A SIMPLE LABORATORY FALLING-FILM MOLECULAR STILL

By E. HAROLD FARMER and D. A. SUTTON

A robust and easily operated falling-film molecular still in glass, the details of which had evolved over a period of 10 years' use, is described with all the necessary details regarding construction, accessories and lay-out. It is of non-cyclic type, gives a vacuum of 10^{-5}—10^{-6} mm. (as measured by an ionisation gauge) in the distillation vessel, and is suitable for laboratory research work in which moderate quantities of material are to be dealt with. It has a wide range of usefulness for the fractionation of mixtures of liquid of different *M*., and in straight high-vacuum distillation, but is unsuitable for use with viscous liquids which do not flow, or for mobile liquids which do not spread uniformly over the glass distilling surface, at the desired distillation temperature.

The falling-film molecular still is well-known in principle and has been the subject of numerous patents and publications. The basic designs, which provide for the accomplishment of distillation in high vacuum by evaporation from the surface of a fairly thin film of liquid, whilst the latter is heated for a short period to a relatively low temperature as it flows downwards under gravity, are due to Waterman and Oosterhof[1] and Carr and Jewell.[2] All subsequent falling-film stills are based on these patterns, but intensive study of the technique of molecular distillation in American laboratories has resulted in many striking advances, culminating in the production by Hickman and his co-workers of the rotating disc still,[3] which is undoubtedly superior to any other type, but which owing to its high cost is unfortunately out of reach of most workers in small laboratories. In spite of the simplicity of the falling-film still, this elegant research weapon has had only restricted use in the chemical laboratories of this country, partly it is true owing to wartime conditions and the difficulty of obtaining the necessary high vacuum equipment, but largely owing to the elaboration of detail and obvious expensiveness of published designs and the almost complete absence of reliable details of construction. No efficient small-scale falling-film still has as yet been marketed in this country. The present object is to describe in sufficient constructional detail a robust, easily worked still which can be cheaply made by a competent glassblower, together with the requisite evacuating equipment, and the layout of the parts. The details and arrangement here illustrated have resulted from ten years continuous experience in the construction and operation of high-vacuum stills, beginning in the senior author's laboratory in the Imperial College in 1935.* Although very numerous modifications in the detailed design of the still have been made since the beginning, no novelty of design is clai[m] those features which were novel at the time of their in[c] have for the most part now been described (at any rate i[n] co[n] by other workers. The still, unlike the commercial stills [a]e in the literature, does not provide for automatic re-dist[il] the fractions, since such provision adds considerably to [the] the apparatus, and it is not essential for dealing with t[he] quantities of material normally used in research laborat[ory].

Excellent readable reviews dealing with the theory a[nd] of molecular distillation are to be found in the literature, so that no detailed account is needed here. It is to be no[ted] ever, that stills of designs which ignore the fundamental p[rin] of short-path distillation appear in the journals from time

Very briefly the practical requirements of a good falling for laboratory (but not necessarily for commercial) use in[clude] following:

(1) The assembly should be made as completely as po[ssible] glass because it can then be left evacuated indefinitely leakage and because pin-hole leaks can readily be detec[ted] high-frequency discharge and sealed up.

(2) The pumping system should be capable of giving a high (10–4 mm. or less) with a high pumping speed, and should nected to the still by short wide tubing.

(3) A pressure gauge which registers pressure continuously manipulation—*e.g.*, of the Pirani or Ionisation type—should of the system and should be placed as near to the still as possible

(4) A cold trap should be provided between the pumping syst and the still to prevent diffusion of pumping fluid into the sti[ll] of distillate into the pump.

(5) The rate at which the distilland enters the system should capable of easy control.

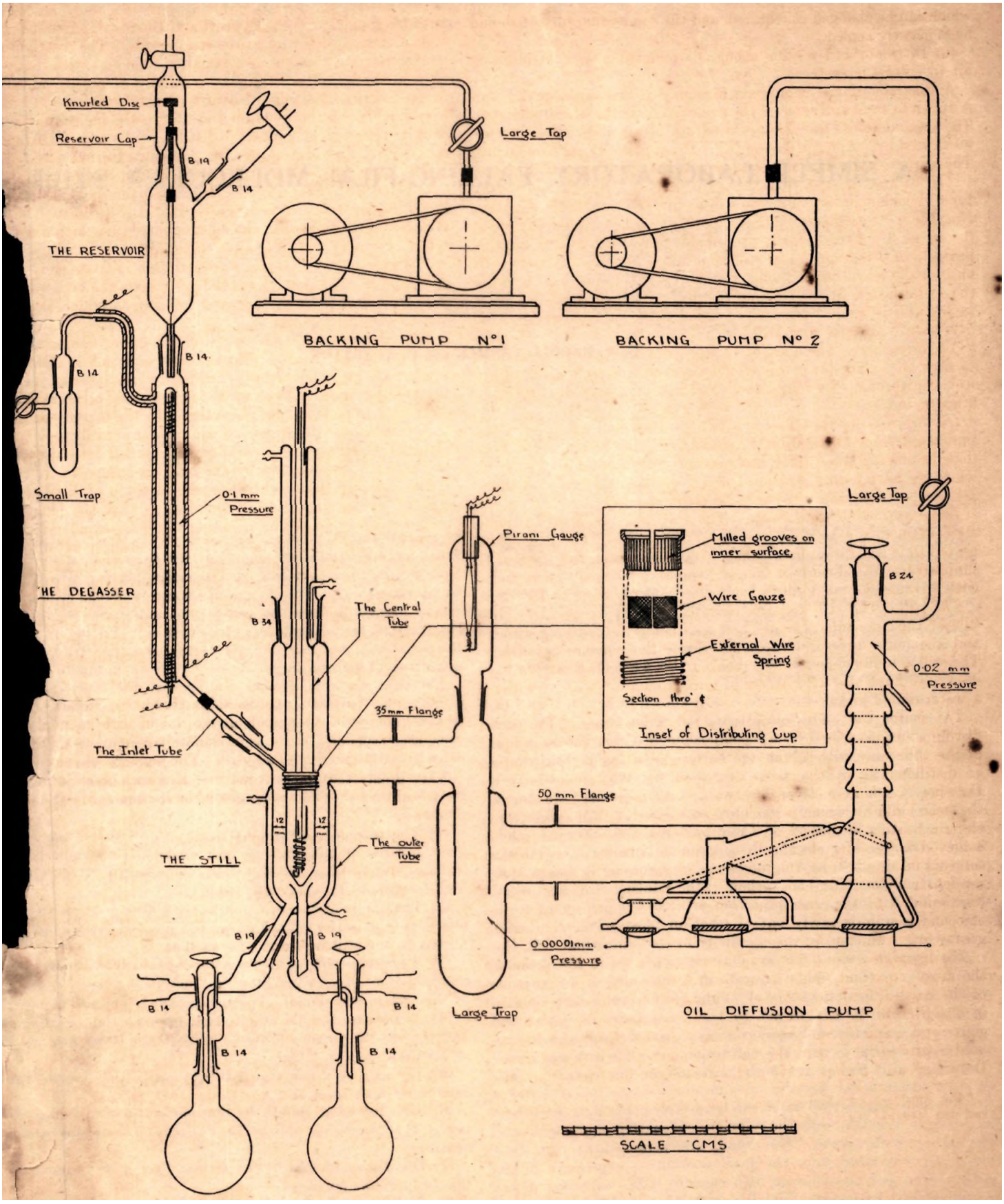
(6) The distilland should be efficiently degassed before distil tion; this degassing should be carried out without destruct heating.

(7) Distillation should be carried out from as thin and uniform film of liquid as possible.*

(8) The distilling surface should be kept at a uniform tempe[rature]

* The authors gratefully acknowledge the contributions and assistance of various of their collaborators and colleagues, and especially of Drs. F. Hilton, F. A. van den Heuvel, G. F. Bloomfield and Mr. A. W. Kenchington.

* Where the liquid is such that it obstinately resists distribution in a unif[orm] film over the heating surface (*i.e.*, it runs down in streams or "channels" is necessary to have recourse to Hickman's rotating disc still,[3] or to diss[olve] the distilland in a suitable liquid medium of low vapour pressure and ev[ap]it therefrom.[3]


Knurled Disc
Reservoir Cap
B 19
B 14
THE RESERVOIR
B 14
B 14
Small Trap
0·1 mm Pressure
THE DEGASSER
B 34
The Central Tube
35mm Flange
The Inlet Tube
12
12
THE STILL
The outer Tube
B 19
B 19
B 14
B 14
B 14
B 14
Large Tap
BACKING PUMP N°1
Large Tap
BACKING PUMP N° 2
Pirani Gauge
Milled grooves on inner surface
Wire Gauze
External Wire Spring
Section thro' ¢
Inset of Distributing Cup
50 mm Flange
0·00001mm Pressure
Large Trap
Large Tap
B 24
0·02 mm Pressure
OIL DIFFUSION PUMP
SCALE CMS

which can be changed as required, and the condensing surface should be efficiently cooled.

(9) Distillate and residue should be removable without allowing air to enter the system.

A diagram of the completed assembly is shown on p. 2; this is drawn to scale and some of the principal dimensions are indicated. The ground joints and flanges used are of standard sizes and are lubricated by "Apiezon Grease N." Pyrex glass is used throughout. The various parts of the assembly will now be discussed in detail.

The backing pumps.—Of these No. 1 is a "Speedivac" pump type 1 which maintains a pressure of around 0·1 mm. in the degasser. No. 2 is a "Speedivac" pump type 3 which has a pumping speed (listed at 3·7 litres/second, presumably measured at the limiting pressure of 0·005 mm.) and will maintain a pressure of about 0·02 mm. in the oil diffusion pump, trap and still. Connection between the metal nozzle of the backing pumps and the glass tubes leading to the still is made by means of bored thick rubber bungs waxed over with "Apiezon Wax W."

The large taps.—These are of 12 mm. bore and tend to be difficult to turn on a cold day; this trouble is overcome by winding a few feet of nichrome wire round them and applying a small current for a few minutes.

Oil diffusion pump.—This is a two-jet Hickman pump, all glass, purchased from Distillation Products Inc., Rochester, New York (listed pumping speed of 12 litres/second measured at 10^{-4} mm.) in which we have used "Apiezon Oil B" as pumping fluid. A three-jet pump of the same type is to be recommended.

Large trap and Pirani gauge.—The trap was immersed in liquid air contained in a large Dewar vessel during use. The gauge connected to it is a Pirani instrument of range 0·5 mm. to 0·0001 mm. supplied by W. Edwards & Co., of London. This may be replaced with advantage by a Philip's Ionisation gauge reading to 10^{-6} mm.

The still.—The still consists essentially of two concentric glass tubes; the inner tube is heated and constitutes the distilling surfaces and the outer tube is cooled and forms the condensing surface. The still actually shown in the diagram is the smallest one we have used; we have also used others of greater length,* with or without a water-cooled jacket.

The central tube.—This comprises a reflux condenser and a heated distilling surface joined in one piece. The distilling (outer) surface of the tube may with advantage be roughened to aid distribution of distilland as in the stills described by Waterman[1] and by Detwiler.[10] A boiling solvent is chosen as the source of heat to avoid the heat build-up which is usually experienced with a simple (dry) electric heating element; any required distillation-temperature is achieved by boiling electrically a suitable solvent or mixture of solvents inside the tube, the solvent being returned by means of the condenser. The B.34 cone forming the upper half of the ground joint connecting the central and outer tubes was incorporated into the condenser in order to keep it cool and so prevent the lubricating grease from running down into the still.

The distributing cup.—The even formation of a thin film is probably the most important single requisite of a molecular still and is one of the most difficult to achieve. Various distributors are recorded in the literature. Hickman[11] used two concentric strips of wire gauze and a spirally embossed column; Jewell, Mead and Phipps[7] used a wire spiral to turn the film continuously as it flowed down; Detwiler[10] used a glass sleeve and a mushroom head in conjunction with his rough surface; Fawcett and Burrows[12] surrounded tilling surface with wire gauze at intervals. The distributor in the diagram consists of a stainless steel cup with many (which can be either straight or spirally disposed) milled inner surface. The cup fits over a piece of stainless steel wi of a small mesh (between 50 and 150 to the inch) and is that it springs tightly over the distilling tube and gauze, an securely in place during use by a strong external wire spri

The outer tube.—The still shown in the diagram has a tube fitted with a water-cooled jacket, but this jacket is not if distillation temperatures above 125° are being used. stills we have found that cooling the outer tube with a wa spiral of copper tubing is often sufficient.

The inlet tube.—This has a constriction near its end, distilland flowing through it effectively seals the high va inside the still from the degasser interior, in which the approximately 0·1 mm.

The degasser and small trap.—The degasser consists tube, electrically heated, into the base of which is sea concentric tube, electrically heated by an internal l the annular space is kept at about 0·1 mm. pressure. distilland drops from the reservoir on to the top of the i and this assists the dissolved air and any easily volatil material which may be present to be expelled into the at or the small cooling trap by the backing-pump No. 1.

The reservoir.—Control of the rate of entry of distillan effected by turning the knurled disc so that the lengt tungsten wire partially obstructing the glass capillary is desired. Ingress of the distilland can be entirely cut off the milled disc until the ground glass surfaces fit together. the B.19 socket of the reservoir-cover over the B.19 co reservoir and evacuating through the B.14 socket of the the reservoir and its contents can be left out of conta atmosphere if necessary; alternatively by partial evac reservoir space an additional means of controlling t the distilland is provided.

The senior author expresses his thanks to Mes.ture, who at the earliest stage of his interest in high vacue no generously assisted him with information relating to the p of a practicable falling-film still. The authors' thanking to the British Rubber Producers' Research Association laboratories the later modifications have been made and t tested.

British Rubber Producers' Research Association,
48, Tewin Road,
Welwyn Garden City, Herts.

[1] H. I. Waterman and D. Oosterhof, Rec. trav. chim., 1933, **58**,
[2] F. H. Carr and W. Jewell, British Patent 415,088 (1933).
[3] K. C. D. Hickman, Chem. Rev., 1944, **34**, 51.
[4] C. R. Burch and W. J. D. van Dijck, J.S.C.I., 1939, **58**, 39.
[5] E. W. M. Fawcett, J.S.C.I., 1939, **58**, 43.
[6] G. Burrows, J.S.C.I., 1939, **58**, 50.
[7] W. Jewell, T. H. Mead and J. W. Phipps, J.S.C.I., 1939, **58**, 56.
[7] S. B. Detwiler, U.S. Dept. of Agriculture, Urbana, Illinois, "Abstr Articles and Patents on Molecular or Short-Path Distillation." Dec 1941.
[9] Farmer and van den Heuvel, J.S.C.I., 1938, **57**, 24.
[10] S. B. Detwiler, Ind. Eng. Chem. (Anal.), 1940, **12**, 348.
[11] K. C. D. Hickman, Ind. Eng. Chem., 1937, **29**, 968.
[12] E. W. M. Fawcett and G. Burrows, B.P., 480, 265 (1938).

* For consideration affecting area of the distilling surface *see* Farmer and van den Heuvel.[9]

Received December 20,

HARRISON AND SONS, LTD.,
PRINTERS,
44–47, ST. MARTIN'S LANE, W.C.2