A Multi-Purpose Low-Pressure Still*[1]

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SEVERAL new features have been added to a more or less conventional laboratory low-pressure still. These improvements are particularly useful when a vapor is purposely introduced into the charge either to aid in distillation or for some other reason.

Apparatus

Figure 1 shows a diagram of the still assembly. The system consists of the still, a cold trap, and a small mechanical pump as a source of vacuum.

The still is constructed of pyrex glass. This laboratory uses two sizes, the smaller for samples from about 10 ml. to 200 ml. in volume, the larger from about 100 ml. to 1,500 ml. Good vapor contact over this wide range in charge volume is due to the shape

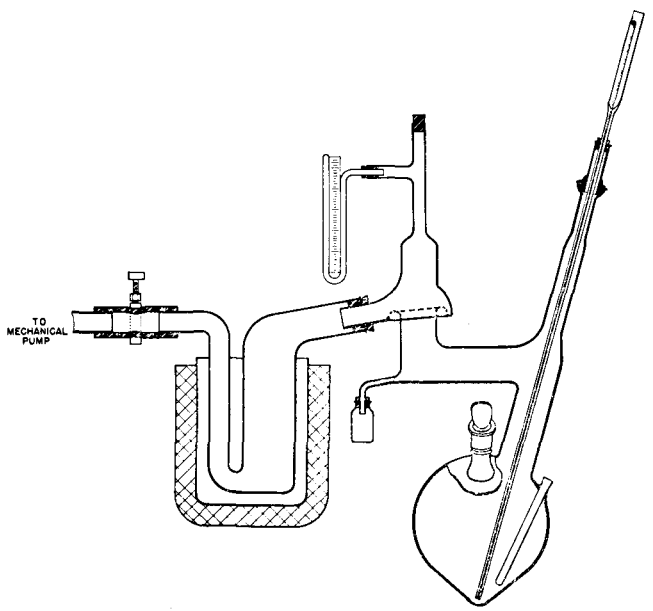

FIG. 1. Cross section of multi-purpose low-pressure still.

of the still pot. This effect is especially important when the volume of distillate coming off is relatively large, thus reducing the size of the charge. The shape of the pot, along with the location of the vapor outlet, permits violent agitation of the charge with a minimum of entrainment in the vapor stream. This is illustrated in Figure 2.

The still neck is slanted so that practically all of the splash drains back into the pot. This section can be insulated when removing a relatively large volume of distillate. Off of this is the "horizontal" section. The entire still can be tilted, even during operation, so that whatever splash reaches the horizontal section can be made to drain back into the pot, or into a separate receiver.

Several methods of introducing pure steam to a deodorizer have been reported (1, 2). In this still, the introduction of vapor, gas, or other material to the charge is accomplished by means of a uniform bore capillary tube as shown in Figure 1. The part outside the still is expanded in diameter to act as a reservoir for liquids or gases. It is graduated for convenience in following the rate of liquid flow. An enlarged view of this is shown in Figure 3. The rate of vapor, etc., introduced to the still can be readily controlled by the position of a wire that fits more or less snugly inside the capillary. The vapor rate can be from almost complete shut-off, when the wire is all the way into the capillary, to the rate obtainable when the wire has been removed. The slow rate of introduction of an inert gas at almost complete shut-off produces enough turbulence to prevent bumping. Liquids of reasonable viscosity, vapors, and gases can be handled in this way. When gas is used, it should be kept in mind that the cold trap does not function as a pump as when a condensable vapor is employed. Unless other steps are taken, a higher pressure results.

For most purposes one capillary with one or two wires of different diameter permits a wide range of flow rates. This can be extended still further by having available several capillaries of different bore. This laboratory uses the wire and capillary sizes as shown in Table I.

TABLE I

Diameters (mm.) of Wires and Their Corresponding Capillaries Used for Vapor Injection Control

Wire	(Avg.) Capillary
0.268	0.316
0.453	0.501
0.651	0.716
0.784	0.926

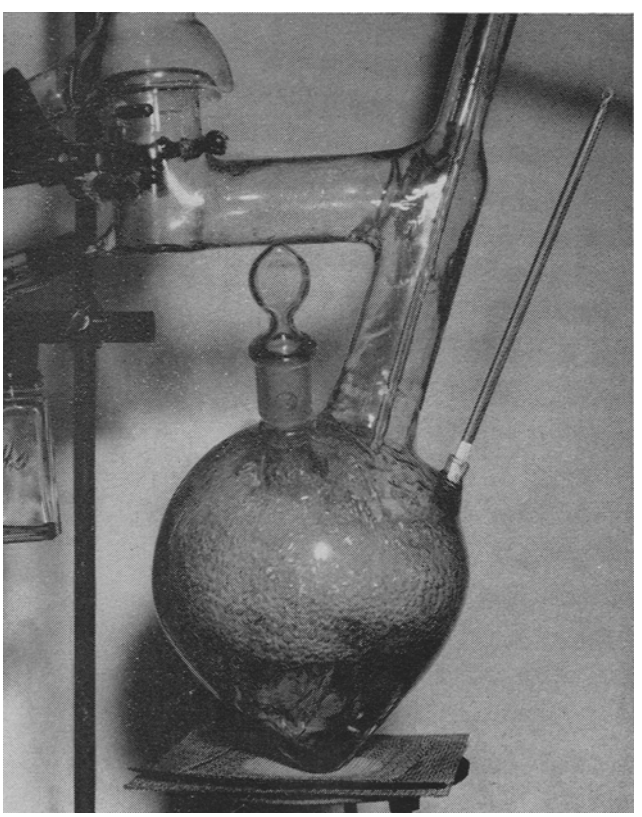

FIG. 2. Multi-purpose low-pressure still deodorizing soybean oil.

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The still conforms throughout to the best principles of vacuum still design (3, 4, 5). The cross-sectional area up to the trap outlet has been kept large to keep obstruction to vapor flow at a minimum. The vacuum measurement, by means of a closed arm mercury manometer, is taken reasonably close to the pot. A tee is located at this point for vapor temperature measurement. Connections are of glass except where rubber stopper and tubing can be safely used. A disc of aluminum foil located at the ball joint prevents drops of liquid from splashing upon the rubber stopper.

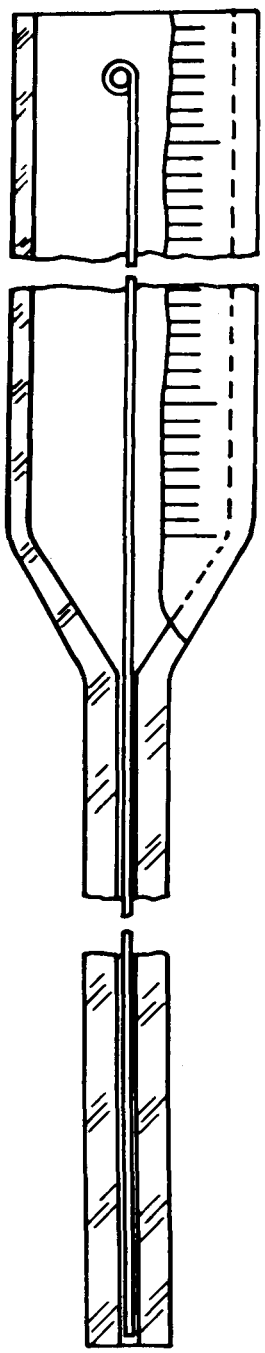

FIG. 3. Fluid and gas control.

In our laboratory the still pot rests on an asbestos-covered iron gauze, which is heated by an open gas flame. In this way, 1 kilo of oil can be raised to 200° C. in about 20 minutes. However, heat may be supplied by a hot oil bath or by some form of electrical heater.

The cold trap used here has been designed to handle large amounts of condensables and has proven very effective. The large arm is 65 mm. in diameter, the smaller is 25 mm., and the approximate over-all height is 240 mm. Despite the large amounts of water vapor used in some deodorizing experiments in this laboratory, no ice has reached the entrance to the smaller arm when dry ice was used for cooling. Under conditions where there is a large amount of distillate, a conventional condenser placed betweeen the still and the trap takes some condensing load off the latter.

All of the glass work on this apparatus was made by the Vacuum Equipment Division of Distillation Products inc.

Uses of the Still

We have mainly used this still for the vacuum-steam deodorization of fats and oils. For example:

In one experiment, 1,000 g. of alkali-refined soybean oil was charged to the larger apparatus and heated. While the temperature was rising to 40-50°C., the vessel surrounding the cold trap was filled with dry ice. Then as the vacuum pump was started, nitrogen was introduced into the capillary at a rate faster than it was sucked into the still. The temperature of the charge that was now being stirred by the nitrogen leak rose to 210°C. in 15 to 20 minutes. Then the nitrogen was removed and, at the same time, a measured amount of freshly distilled water was put into the capillary entrance. The wire was drawn out to the point where the desired rate of water flow was obtained. In this way the hot charge converted the water to steam at a point 10 to 20 cm. before it emerged from the capillary and into the charge. This insured a steady, even flow without bumping.

In this particular case, the removing of 30 cm. of the wire from the capillary gave a water vapor rate, based on the charge, of 2% per hour. After one-half hour at 210°C. heating was stopped, but the introduction of water vapor was continued. This, together with an air stream blown on the pot, lowered the temperature to 115°C. in 10 to 15 minutes. At this point, nitrogen replaced the water, for the charge has to be at a temperature above 110°C. to volatilize water while still in the capillary.

The line was then shut off between the trap and the pump and the still slowly rose to atmospheric pressure from the nitrogen. After 10 to 15 minutes the oil was at 40 to 50°C. when it was removed as a tasteless and odorless product.

This equipment has been used for other purposes in this laboratory which indicate that it can be advantageous in the following processes:

1. To strip off low volatile materials from a reaction which is otherwise retarded.
2. For the controlled introduction of reagents to a reaction that is being carried out under vacuum.
3. For the introduction of chemicals (e.g. antioxidants in edible oils) under anaerobic conditions.
4. For a wide variety of vacuum distillations.

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