An Inexpensive Automatic Fluoride Distillation Apparatus

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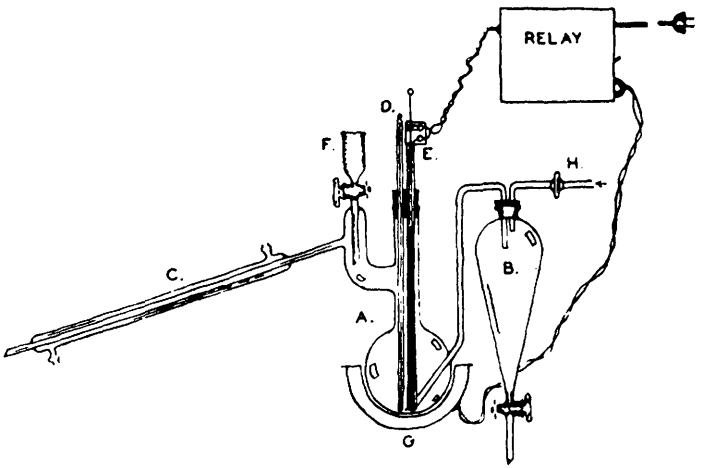
Since Willard and Winter (5) published their classical method for separation of fluoride from interfering substances by volatilization as hydrofluosilicic acid, many modifications of their procedure and distillation apparatus have been used. This method consists of heating a sample containing fluoride in a distillation apparatus containing a high boiling acid such as sulfuric, perchloric, or phosphoric. The temperature of the distillation mixture is usually controlled by manual adjustment of a heater, but automatic temperature control devices of varying complexity have been used. An electronically controlled apparatus was developed by Willard, Toribara, and Holland (6), and a custom-made distillation apparatus with automatic temperature control by means of a mercury switch and magnetic valve has been successfully used by White and Estill (4).

The ordinary chemistry laboratory, however, does not have the materials of construction for an automatic temperature control device as described above. The apparatus shown schematically in Figure 1 has automatic temperature control and also incorporates the self-emptying feature, as described by Parnas (2), and as used by these authors in a Kjeldahl apparatus (3), which makes it unnecessary to disassemble the apparatus for cleaning or for renewing the acid. The materials for construction of this apparatus are also readily available.



A CLAISSEN FLASK, 250ML.
B. SEPARATORY FUNNEL, 250ML.
C. CONDENSER
D. THERMOMETER
E. MERCURY SWITCH
F. ADDITION FUNNEL
G. ELECTRIC HEATER
H. STEAM INLET

FIGURE 1. Automatic Distillation Apparatus.

Construction and Operation of Apparatus Shown

A length of eight-mm. glass tubing is sealed through the side of a 250 ml. Claisen flask A (Figure 1). This steam inlet tube should reach very nearly

the bottom of the flask. Through a two-hole rubber stopper a thermometer, D, and tube containing mercury, E, are inserted into the center neck of the flask. This mercury tube should have a bulb at the lower end for greater sensitivity.

When the high boiling acid and sample are introduced through the addition funnel F and steam passed through the steam inlet tube H on through the separatory funnel B the line operated relay (e.g. Precision Temperature Regulator) is switched on and the electric heater G keeps the distillation mixture heated. The distillate is condensed in C and caught in a suitable container for subsequent determination. When the distillation mixture reaches the desired temperature, the mercury switch is adjusted to cut off the heater G by means of the relay. During the distillation the stopcock on F and B are closed but H is open. Steam which condenses in B may be drained during distillation. During continuous operation no further adjustments are necessary except the addition of samples through F.

When it is desired to dispose of the used acid in the flask, the steam is turned off and the stopcock on H is closed. The vacuum produced in B draws the used acid into B. The used acid may be drained from the separatory funnel and discarded. The flask may be further cleaned by rinsing and drawing the wash solution from the flask into B in the same manner.

An initial period of approximately 20 minutes is required for the system to reach thermal equilibruim, but in continuous operation the temperature has been observed to stay constant within three degrees of 147°C. using sulfuric acid and steam as recommended by the Association of Official Agricultural Chemists (1).

According to individual needs and preferences, the above apparatus may be modified without sacrificing the convenience of the automatic temperature control and self-emptying features. The separatory funnel B and Claisen flask A may be connected by Tygon or rubber tubing, for example, thus allowing the sample to be introduced through the steam inlet tube if the placement of the addition funnel F in the auxiliary neck of the Claisen flask is considered objectionable.

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