

By *H. E. Watson.*

In the process of distilling volatile oils by means of steam, the distillate usually consists of two liquid phases which it is necessary to separate. For work on a very small scale it is customary to collect the whole of the distillate and after allowing it to stand for some time, to separate the two layers which form by means of a separating funnel. On a somewhat larger scale a florentine flask is used which allows the water to run away but retains the oil. In some cases these flasks are fitted with an outlet through which the oil can be withdrawn at intervals. This arrangement is very convenient as long as the quantities of liquid are not too large to be dealt with in glass vessels, but for big stills it is necessary to make the separators of metal with cocks for the withdrawal of oil. The difficulty then arises that the bounding surface of oil and water cannot be seen and consequently when drawing off oil, a certain amount of water is usually obtained as well, involving a second separation. In addition, the cocks must be made of a material which will not discolour the oil, and unless carefully attended to, may leak, while with mixtures such as are obtained in the refining of oils by distilling with superheated steam, in which the quantity of oil is comparable with that of the water, a considerable amount of attention is required for drawing off the oil.

In order to overcome the defects of the florentine flask type of separator, the apparatus shown in the annexed figure has been designed. The metal of which it is made must be chosen to suit the oil to be distilled, but for most essential oils, well tinned copper or brass is the most satisfactory. It consists of a cylindrical vessel into which the mixture of oil and water from the still is admitted through the pipe A which is bent at the lower end so as to discharge the liquid horizontally. F is a float which is constructed so as just to float in hot water but to sink in the oil, or *vice versa* in the case of oils heavier than water. Underneath the float is a rod L carrying the valve T which closes the tube E leading out of the vessel. The shape of this valve is of some importance as it is essential that it should not stick or be sucked downwards by the rush of water. The form shown has been found quite efficient. The lower portion is conical and its widest

part is very slightly smaller than the hole in the seating, so that as the float moves up and down the rate of egress of the liquid is varied. The conical portion also serves as a guide which prevents the valve from being carried away from the seating. The upper portion of the valve is ground flat to fit the flat top of the seating so that when the float falls the outlet is completely closed. To the top of the float is attached a spindle S which passes through a hole in the lid of the vessel and keeps the float in position. A pin G through this spindle prevents the float from rising so high that the valve D can come away from its seating. The float is adjusted by means of a lead washer the correct weight of which is found by trial.

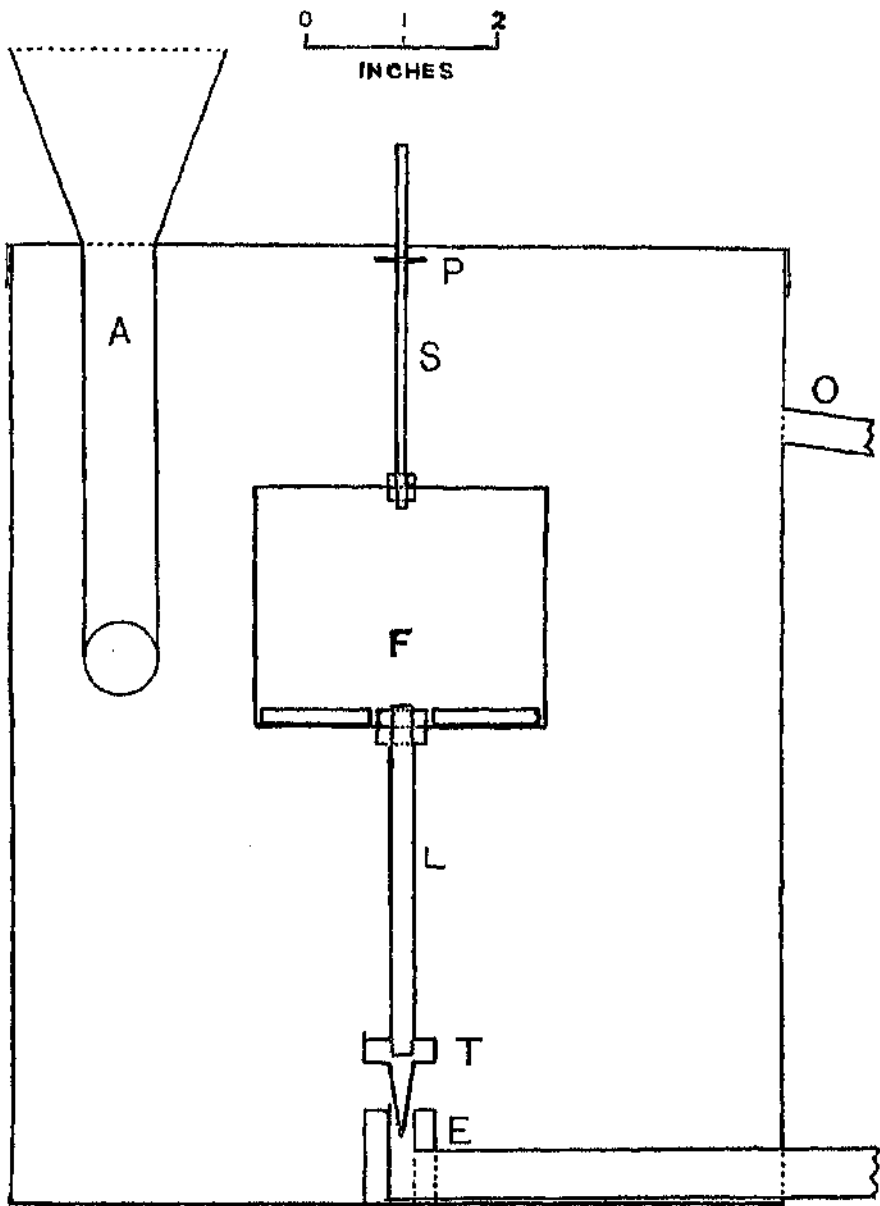
When a mixture of water and an oil lighter than water is introduced into the vessel the oil separates out on top of the water and the float takes up a position between the liquids. The valve controls the rate of outflow of the water in such a way that the bounding surface of the liquids remains at a constant level, and consequently, when sufficient oil collects, it runs out through the pipe O.

It is not essential for the valve to fit perfectly as only water escapes from it, and in fact, a small leak is rather an advantage as it tends to prevent sticking. If however it is necessary to stop the distillation for more than a short period the float should be depressed before shutting off steam so as to allow the vessel to fill with water which will displace all the oil.

The 8" diameter separator shown in the figure is of suitable dimensions for dealing with about 100 lbs. of distillate an hour, and separates practically all the oil. It is advisable, however, to run the water through a second separator to collect the remaining small quantity of oil and to guard against loss of oil in the event of the valve failing to close, a very unlikely occurrence.

For oils which are heavier than water the stem C should be made as short as possible and the valve seating should be flush with the bottom of the vessel so that all the oil can be withdrawn.

The lid of the vessel may be made to lock and the top of the spindle placed in a tube so that it cannot be raised by hand thus making it impossible to abstract any of the oil from the separator which may be connected directly to a locked receptacle.



Automatic Separator.

This separator has been used in these laboratories for distilling several hundred pounds of lemon grass oil and has been found to work very satisfactorily and to require no attention.

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