

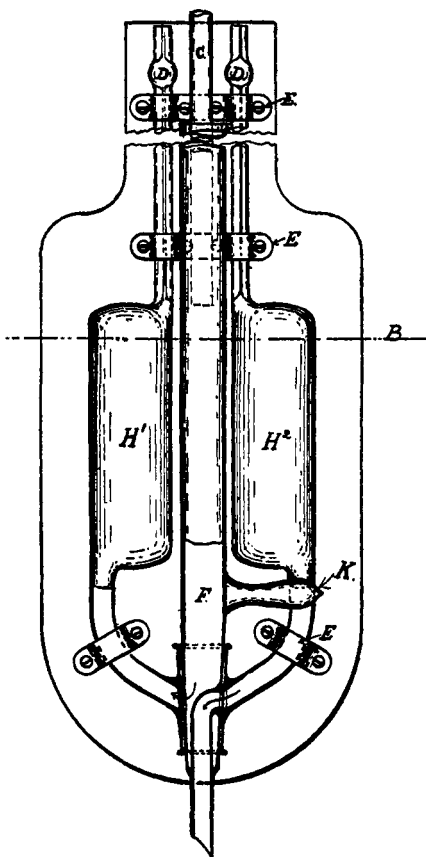
ger of the solution, which *C* siphons into it from the stock bottle, running over even when the latter is full. The small capillaries with the little safety bulbs *D, D* should be longer than *F* as at times drops of the solution from the last charge may collect in them and be forced upward by the outcoming air when *H* is again filling. *C* is a simple siphon having the leg in *F* a little shorter than the one in the solution reservoir, so that it may drain back rather than out to the pipette when lifted up to remove the stock bottle. The entire apparatus is fastened by lead strips *E*, to a board which is then secured to the shelf, on which stands the solution supply, at such a height that the tops of the bulbs *H*₁, *H*₂ are about on a level with the bottom of the reservoir.

The method of operating is obvious: with the little handle *K* turned to the right, the siphon is started by blowing into one hole of the two-hole rubber stopper which holds *C* in place in the stock bottle, and bulb *H*¹ fills until the solution stands up in the capillary level with that in the reservoir; a reversal of the stopcock then allows *H*¹ to discharge and *H*² to fill simultaneously.

This apparatus is not intended to deliver exact quantities of standard solutions but approximately a constant amount of some such reagent as the sulphuric acid for the Babcock milk test or Kjeldahl nitrogen digestion. The slight difference between the delivery capacity of the pipette when the reservoir is full and when it is empty, due of course to the variation in the height at which the solution stands in the capillaries, is very small. It need not be more than a few tenths of a cubic centimeter, as a capillary of that volume per foot of length is plenty large enough to allow the air to escape.

HERBERT S. BAILEY.

BUREAU OF CHEMISTRY, WASHINGTON, D. C.



Method for Determining Unsataponifiable Matter in Oils and Fats.—Two or 3 grams of the sample are weighed in a flask holding about 100 cc.

and an excess of strong alcoholic potash solution added and the whole boiled for not less than one hour under a reflux condenser. An equal amount of water is then added and the cooled solution poured into a 16 oz. separatory funnel and shaken with 75 cc. of petroleum ether. The solutions are allowed to separate and the soap solution drawn off into a second separatory flask. The petroleum ether solution is then washed three times with 15 cc. of 50 per cent. alcohol. The soap solution is extracted with a second lot of 75 cc. of petroleum ether, and this is repeated from three to four times. The various ether extracts are then combined in a suitable Erlenmeyer flask and the ether distilled off, down to about 10 cc. The flask is then immersed in water at from 60° to 70° and air blown in till all the ether is expelled. If any water shows, add a little alcohol and repeat the drying till constant weight is obtained. Calculate the per cent. from the original weight taken.

Details to be Carefully Observed.—If any petroleum oil is present, drying at 100° in a water bath will cause loss and incorrect results will be obtained, as all light petroleum oils are more or less volatile at that temperature.

In mixtures of heavy petroleum oils, and fatty oils where it is necessary to saponify the fats present, boiling for at least one hour is imperative.

While it is noted in standard works on fats that it is necessary in making up alcoholic potash solutions for the determination of saponification value, to use an alcohol that has stood for some days over potash, I have not seen this point brought out in making solution for determining unsaponifiable matter. It is, however, absolutely necessary, as otherwise high results will be obtained. A colorless or nearly colorless alcoholic potash solution must be used.

Denatured alcohol of following composition may be used, if it is first boiled under reflux condenser for 24 hours with caustic potash grain alcohol, 90 per cent.; methyl alcohol, 9.5 per cent.; and benzene, 0.5 per cent.

The necessity of three or more ether extractions of the soap solution, is shown by the following results:

1st ext.	2nd ext.	3rd ext.	4th ext.	Total ext.
8.45%	3.25%	1.85%	0.90%	14.45%

These extracts were then combined, resaponified and re-extracted.

1st ext.	2nd ext.	3rd ext.	4th ext.	Total ext.
5.75%	7.55%	1.00%	0.10%	14.40%

The same sample was saponified and extracted with ethyl ether.

1st ext.	2nd ext.	3rd ext.	4th ext.	Total ext.
10.10%	2.65%	1.05%	0.50%	14.30%

The above results show that the rate of extraction is not uniform, and that at least four extractions are required with a sample containing this amount.

A. G. STILLWELL.