APPARATUS AND METHOD USED IN SAPONIFYING COMPOUNDED OILS.

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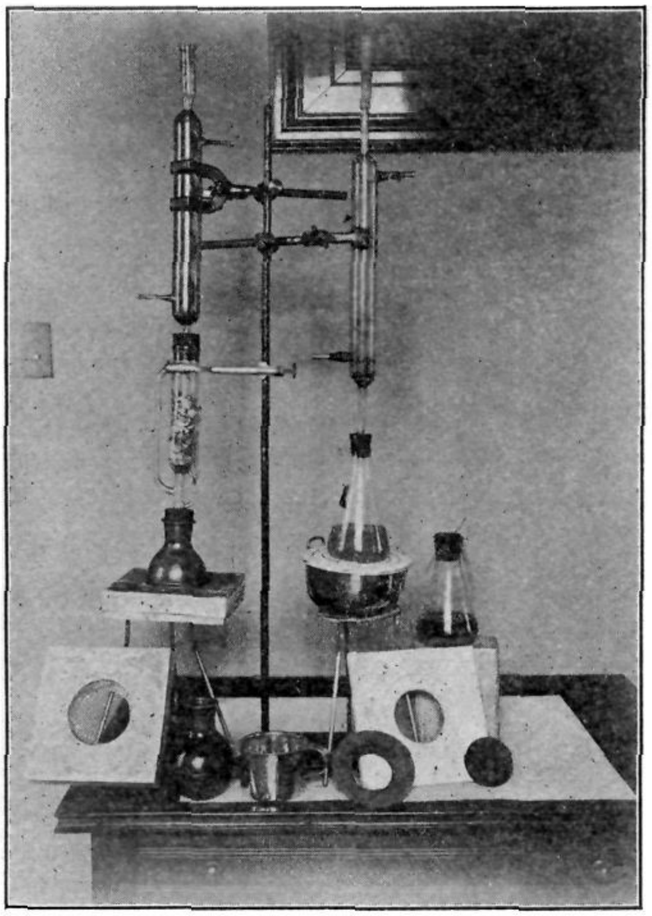
THE following method, with accompanying photograph showing a saponifying apparatus, I have found very useful and convenient in saponifying compounded lubricating oils, especially those containing heavy thick petroleum oils, such as valve and cylinder oils, and, of course, the apparatus is equally well adapted for any oil or fat.



APPARATUS.

(1) A 300 cc. solid, pure silver flask (made for me by Queen & Co., of Philadelphia) with wide and true ground neck, shape of the flask as shown in the photograph. (2) A medium Soxhlet's extraction tube filled with glass beads or broken marbles up to or a little above the upper bend of the siphon tube. (3) A 12-inch glass return condenser, according to Allihn. (4) 1, 2 and 3 properly connected with good corks, and placed on a tripod, on

which rests two asbestos blocks, as shown in the photograph, a thinner under one, and a thicker one with tapered holes cut out in the center to fit the silver flask: between the two asbestos blocks or plates is put an iron ring on which rests a small iron plate, fitting the somewhat concave shape of the bottom of the silver flask. All these parts are clearly indicated or shown on the photograph.

With an apparatus fitted up in this way there is no danger of the flask breaking; it can be left to itself, and a direct gas flame used for the boiling. Having a great deal of this kind of work to perform, I soon found glass flasks very unsatisfactory either from breaking, or the caustic potash solution used in saponifying soon begins to act on the glass in the flasks to such an extent as to seriously interfere with obtaining correct results. I therefore conceived the idea of having a couple of silver flasks made, as shown in the photograph, and they have given entire satisfaction; they have been in constant use for the last four or five years, and are now as good as new.

The Soxhlet's tube acts like an automatic agitator by the intermittent filling and emptying through the siphon tube, thus facilitating the saponifying process.

STANDARD ALCOHOLIC POTASH SOLUTION USED IN SAPONIFYING FATS AND OILS.

I use practically a half-normal alcoholic caustic potash solution made as follows: 50 grams pure caustic potash, dissolved in 680 cc. of pure 95 per cent. grain alcohol; allow the solution to stand for a while in a dark place to settle, then filter through an asbestos filter, and add to the filtrate 300 cc. of distilled water, shaking well, and allow the solution to stand for a day before standardizing. Twenty-five cc. of this solution requires about 50 cc. of fourth-normal hydrochloric acid. To standardize this alcoholic potash solution I proceed exactly in the same way as in saponifying oils. which will be described below, besides standardizing the same direct with fourth-normal hydrochloric acid, and fourth-normal caustic potash, using phenolphthaleïn as indicator.

SAPONIFICATION PROCESS FOR COMPOUNDED OILS.

The amount of oil taken depends upon the amount of saponifiable fat or oil expected to be present in the sample, varying from

2.5 to 10 grams. If the oil contains 35 to 65 per cent. saponifiable fat or oil, I use 5 grams or less, and if less than 20 per cent., 10 grams. Weigh out the oil in a small beaker, warm if necessary, transfer the oil from the beaker to the silver flask by means of 15 to 20 cc. of 88° gasoline, add 25 cc. of the standard alcoholic potash solution from a 25-cc. pipette (after drawing up the solution in the pipette to a little above the mark, wipe off the outside lower part of the pipette), allow the pipette to drain, have 80 cc. strong neutral alcohol in a 100 cc. measure, draw up alcohol into the pipette, about 20 to 25 cc. at a time, so as to rinse out the pipette and allow alcohol to run into the flask: in this way the pipette is entirely freed from the potash solution adhering to the inside walls and sides of the pipette. Connect the flask as shown in the photograph, and already described; boil quite briskly from five to eight hours.

Heavy, thick, compounded oils, such as cylinder and valve oils, containing heavy petroleum stocks, take a longer time to saponify than thin, light oils. After boiling a sufficient length of time, shut off the heat, remove the silver flask, and add to the flask any alcohol that may be in the Soxhlet tube. Then proceed to titrate to determine the amount of caustic potash used in saponifying the oil; to this end add a little phenolphthaleïn as indicator to the flask, and add gradually hydrochloric acid (fourth-normal and standardized against fourth-normal caustic potash) in small excess, or until the red color disappears on shaking (owing to the wide mouth and neck it is very easy to see when the red has gone, even with dark-colored oils), then transfer the contents in the silver flask to a 300-cc. glass flask, rinse out the former with a little neutral alcohol and a little 74° benzine, add to the silver flask 5 cc. hot water and 2 cc. of the fourth-normal hydrochloric acid, shake (this to dissolve any potash that might remain in the silver flask), add this to the contents in the glass flask, put on a steam- or water-bath and heat to boiling, boil for a few minutes (to drive off any carbonic acid that might be present), then neutralize the contents in the flask with fourth-normal caustic potash and calculate in the usual way how much caustic potash has been used from the number of cubic centimeters required, and from it the amount of saponifiable fats present.

For most fats or oils such as tallow oil, lard oil, cotton-seed oil, maize or corn oils, the same standard factor or equivalent can be used; if rape, colza or sperm oils are present, of course, it will be necessary to separate the total free fatty acids, and from this calculate the amount of saponifiable fats present.

FRANKLIN, PA.,
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[CONTRIBUTIONS FROM THE HAVEMEYER LABORATORIES OF COLUMBIA UNIVERSITY, No. 96.]

THE DETERMINATION OF MOLYBDENUM IN STEEL AND IN STEEL-MAKING ALLOYS.[1]

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INTRODUCTION.

As there is difficulty in obtaining an accurate and rapid estimation of molybdenum in steel and in steel-making alloys, the following work was undertaken in order to compare the best methods now in use. It was also the object of the investigators to point out the reasons for the discrepancies in the several methods, and, if possible, to devise a rapid and accurate method which will not be affected by the impurities in molybdenum steel, or by the other metals which are sometimes added, or may in future be added. The impurities are silicon, phosphorus, sulphur, manganese and copper; metals added are molybdenum, tungsten, chromium, and those which may in future be added are uranium and vanadium.

PART I.

In making a determination of molybdenum in steel and in steel-making alloys the main difficulty is the separation of molybdenum from iron.

There are two general methods for this separation. The first, and most commonly used, is to separate the iron from the molybdenum as ferric hydroxide, and the second is to separate the molybdenum from the iron as sulphide of molybdenum.

After the separation of the iron the molybdenum may be determined by three different methods. First, by reduction and titration with potassium permanganate; second, precipitation of

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[1] Read at the meeting of the New York Section of the American Chemical Society, January 8, 1904.