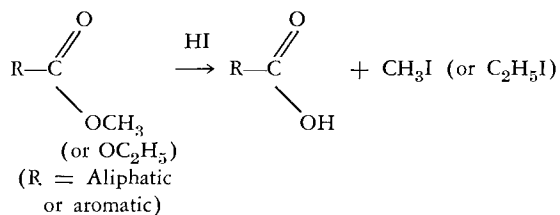


## Microdetermination of Alkoxy Groups (Methoxy and Ethoxy)

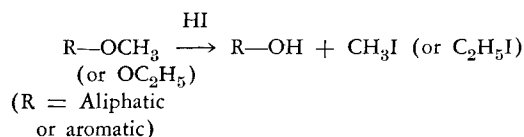
The determination of alkoxy groups (methoxy and ethoxy) in the form of either ethers or esters is accomplished by treatment of the organic compound with boiling hydriodic acid.<sup>12,13,28,33,44,45,50,51,82-84,86-89,97-100</sup> By this treatment the alkoxy is split off and converted to methyl or ethyl iodide. Where the alkoxy is present in the form of an ester, the splitting off is rather rapid and in fact often first yields the alcohol which then is converted to the iodide. Where the alkoxy is present in the form of an ether, the action takes place more slowly.

The alkyl iodide (methyl or ethyl) is then determined iodometrically or gravimetrically. For the former, a modification of the method of Vieböch and Brecher<sup>108</sup> is used, the alkyl iodide being oxidized with bromine to iodic acid. This is treated with an excess of potassium iodide in acid solution yielding iodine, which is determined by titrating with thiosulfate (compare standardization of thiosulfate, Chapter 5). For the gravimetric procedure, the alkyl iodide (methyl or ethyl) is reacted with alcoholic silver nitrate yielding a double salt,  $\text{AgI} \cdot \text{AgNO}_3$ ,<sup>50,51,86-89,100</sup> which in turn is split on the addition of water and nitric acid. The resulting silver iodide is then determined gravimetrically. The various reactions for both procedures are shown below.

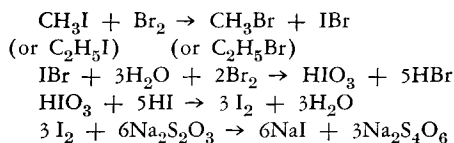
(a) For esters:



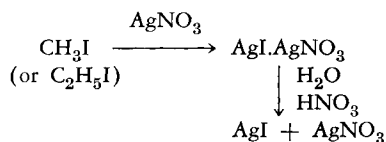
(b) For ethers:



(c) Then volumetric (iodometric) :



or gravimetric:



Two pieces of apparatus are described in the following pages, *both* of which may be used with a volumetric or gravimetric procedure, and with *either* give excellent results. The first method described is that employing the modified Clark<sup>99,101</sup> apparatus developed by the Committee on Microchemical Apparatus of the Division of Analytical Chemistry of the American Chemical Society and the method adopted by the Association of Official Agricultural Chemists following a collaborative study in which this apparatus was used. This adopted procedure is a volumetric (iodometric) one, but the same apparatus may be employed in the gravimetric procedure. Likewise, the procedure described using the apparatus developed by the author<sup>97,100</sup> is a gravimetric one, but the same apparatus may be used with the volumetric (iodometric) procedure. Each piece of apparatus has an advantage over the other. The Steyermark apparatus, due to its *two* reaction flasks, often gives better results with volatile substances but it is more fragile than the modified Clark apparatus. The latter is suitable also for semimicro-work or where relatively large amounts of material must be taken in order to analyze for small percentages of alkoxy groups.

Regardless of the method employed, there is no interference from fluorine.

## VOLUMETRIC (IODOMETRIC) METHOD

### Reagents

#### PHENOL<sup>33,50,51,82-84,86-89,97-100</sup>

Pure crystalline phenol is used as a solvent for the sample. (*Caution:* This must be handled with care.)

#### ACETIC ACID-POTASSIUM ACETATE-BROMINE SOLUTION<sup>99</sup>

Ten grams of potassium acetate is dissolved in glacial acetic acid and diluted with the acid to 100 ml., and 3 ml. of bromine is added to complete the reagent. Thus must be freshly prepared.

**SODIUM ACETATE SOLUTION**<sup>54,99</sup>

Twenty-five grams of sodium acetate,  $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$  is dissolved in distilled water and diluted to a volume of 100 ml. This is used in the scrubber<sup>54</sup> as well as in the titration mixture.

**STARCH INDICATOR**

Same as described in Chapter 5.

**STANDARD SODIUM THIOSULFATE, 0.01N**

This is prepared and standardized as described in Chapter 5.

**FORMIC ACID**

Reagent grade formic acid is used to destroy the excess bromine before titrating.

**POTASSIUM IODIDE CRYSTALS**

Reagent grade, stored in brown bottle.

**DILUTE SULFURIC ACID, 10%****HYDRIODIC ACID,**

**SP. GR., 1.7**<sup>50,51,82,86-89,97,99,100</sup>

The hydriodic acid generally purchased is unsuitable for use without purification, whether reagent grades or those designated as special for the microdetermination of methoxyl are used. They often contain hydrogen sulfide, phosphine, and possibly alkyl iodides formed from the reaction of the acid vapors on the material used to cover the bottle stoppers. These give large blanks, sometimes the amount is equal to or greater than that obtained in actual determinations.

**TREATMENT OF HYDRIODIC ACID.**<sup>84,97,99,100</sup> One pound of reagent grade hydriodic acid, sp. gr. 1.7, is placed in a round-bottomed flask which, in turn, is connected by means of a ground joint to an air condenser. The acid is heated to gentle boiling for about 2 hours, during which a slow stream of carbon dioxide or nitrogen is bubbled through by means of a glass tube extending to the bottom. At no time should the acid vapors be allowed to come in contact with organic material, which would cause recontamination. When heating is stopped the flow of gas likewise should be discontinued, as fuming acid is formed by passing the gas through at room temperature.<sup>76,97,100</sup> A blank methoxyl determination should then be carried out to determine the quality of the acid. If even a trace is obtained, the refluxing with carbon dioxide or nitrogen should be repeated, as it is possible to obtain a reagent which gives a perfect blank. The acid is stored in a brown glass-stoppered bottle at laboratory temperature and gives good results for a number of weeks. (The

color of the product is of no importance. In fact, the presence of dissolved iodine, causing the dark color, is advantageous since it converts organic bond sulfur to the elementary form, preventing its interference.<sup>33,97,100</sup>) Test samples should be run at frequent intervals to make certain that the reagent is still efficient.

## Apparatus

### CARBON DIOXIDE SOURCE

A small carbon dioxide cylinder, provided with a suitable reducing valve, is used as a source of carbon dioxide. Before entering the alkoxy apparatus,

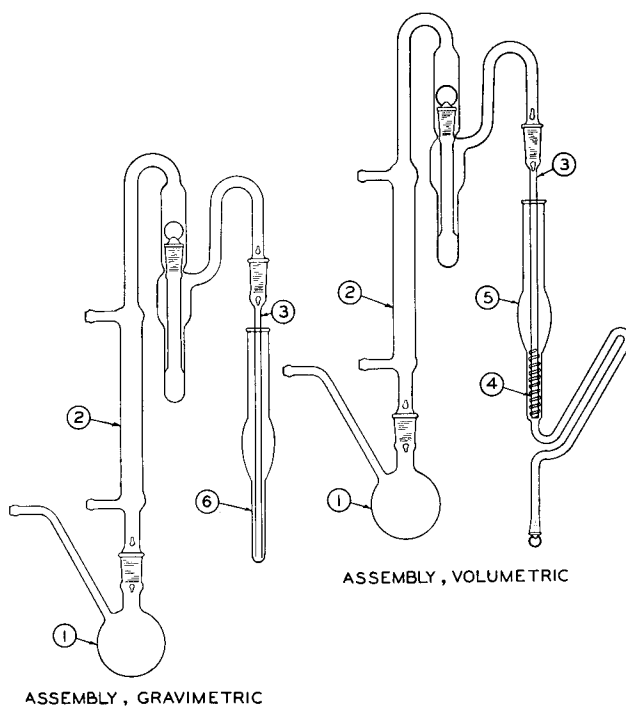


FIG. 179. Modified Clark alkoxy apparatus, assembly (gravimetric and volumetric).

the gas should be passed through some type of wash bottle or scrubbing tower which contains a concentrated solution of sodium carbonate to remove any acid vapors present. A bubble counter containing concentrated sulfuric acid may be used following the wash bottle or scrubbing tower as an aid to the subsequent regulation of the flow of carbon dioxide through the system. [Either the broken section of a bubble counter-U-tube (see Fig. 120, Chapter 9) may be used for this purpose or a complete unit without filling, except the  $\text{H}_2\text{SO}_4$ .]

MODIFIED CLARK ALKOXYL APPARATUS<sup>99,101</sup>

The apparatus used, shown in Fig. 179, is the modified Clark apparatus with which either volumetric or gravimetric procedures may be used depending upon the type of receiver. For the volumetric procedure, the apparatus consists of the reaction flask with side arm, (1), condenser with scrubber, (2), inlet tube, (3), spiral, (4), and volumetric receiver, (5), and is shown as-

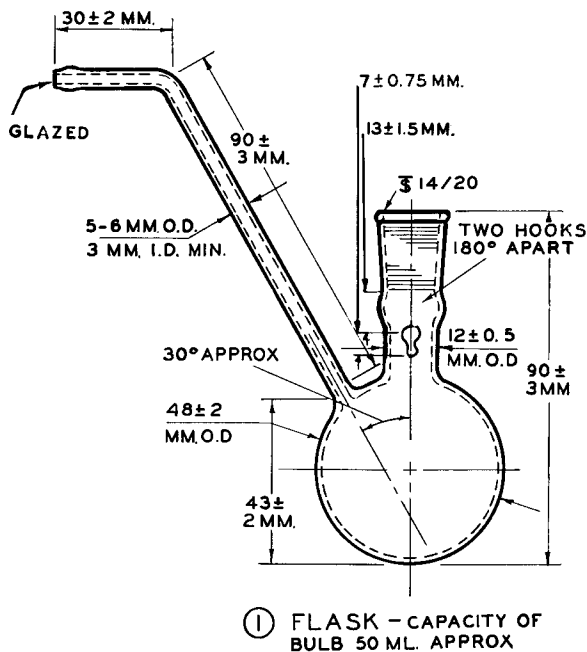


FIG. 179(1). Flask for modified Clark alkoxy apparatus—details of construction.

sembled. For the gravimetric procedure, the spiral, (4), and volumetric receiver, (5), are replaced by the gravimetric receiver, (6), and this is also shown assembled.

The dimensions for the side arm of the flask, (1), were arrived at after a number of experiments. Capillary tubes, with and without bulbs, were unsatisfactory because of condensation in the tube. The recommended length of the side arm is necessary to minimize contact of acid with the gas connection.

The condenser with scrubber, (2), has an enlarged section between the two parts to prevent suck back of liquid from scrubber into condenser at the end of a determination. (Several types of scrubbers were tested, including one constructed of two compartments connected by a capillary tube. The one selected operated more efficiently than all others tried.)

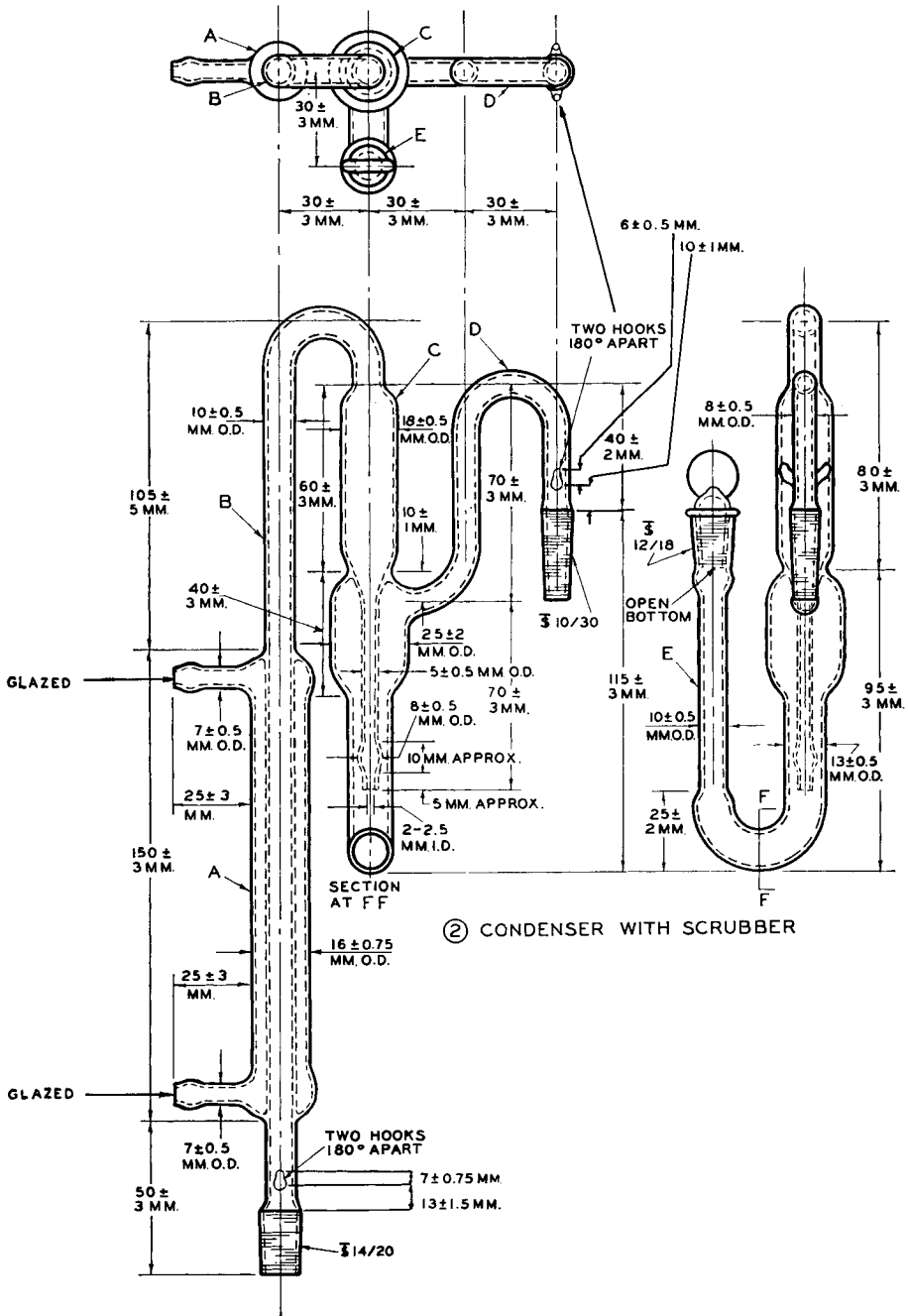


FIG. 179(2). Condenser with scrubber for modified Clark alkoxyl apparatus—details of construction.

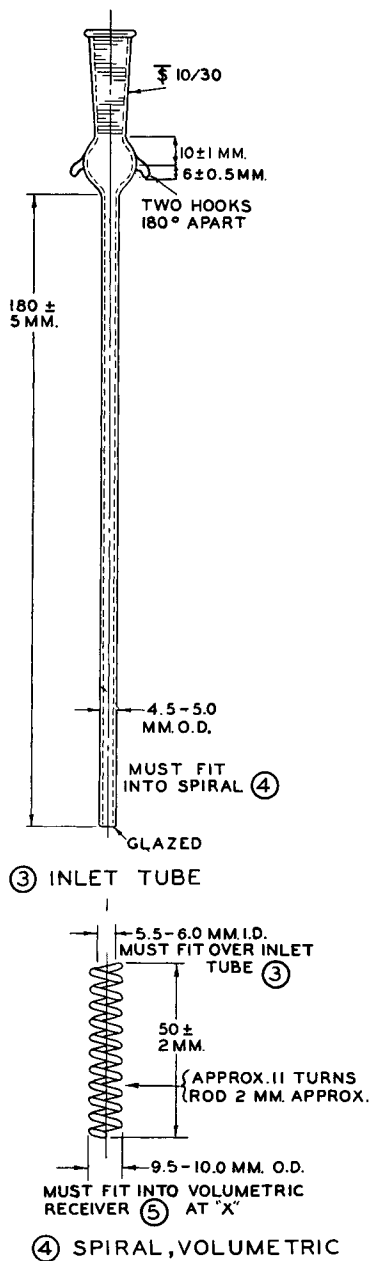


FIG. 179(3). Inlet tube for modified Clark alkoxy apparatus—details of construction.

FIG. 179(4). Spiral, volumetric, for modified Clark alkoxy apparatus—details of construction.

The section between the scrubber and the inlet tube, (3), was designed to prevent liquid being carried into the receiver.

Use of the spiral, (4), in the receiver, (5), is optional in the volumetric procedure. Extensive tests have shown that equally good results are obtained without the spiral.<sup>101</sup>

### BURETTE

An automatic burette of the type shown in Figs. 69 or 70 (Chapter 5) is used for the standard thiosulfate solution.

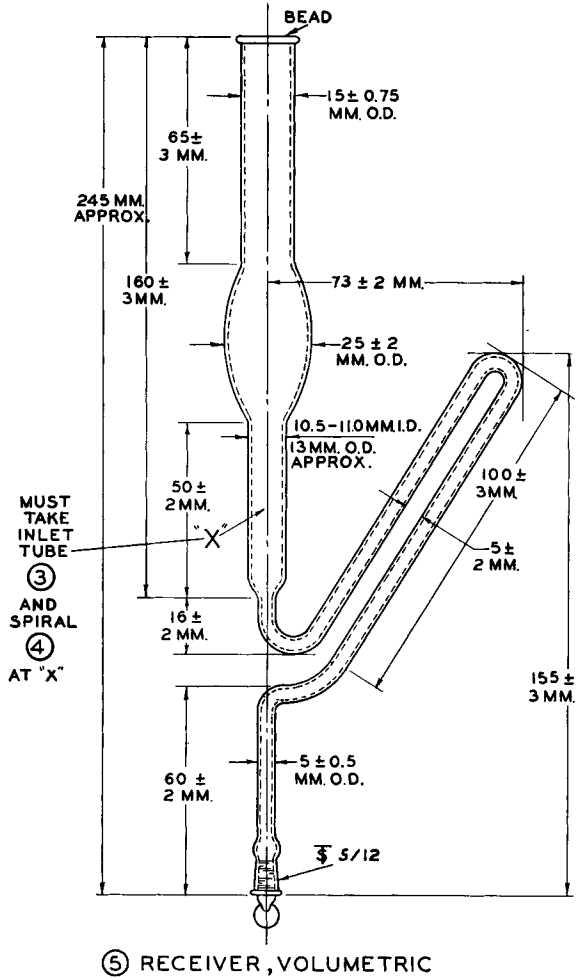
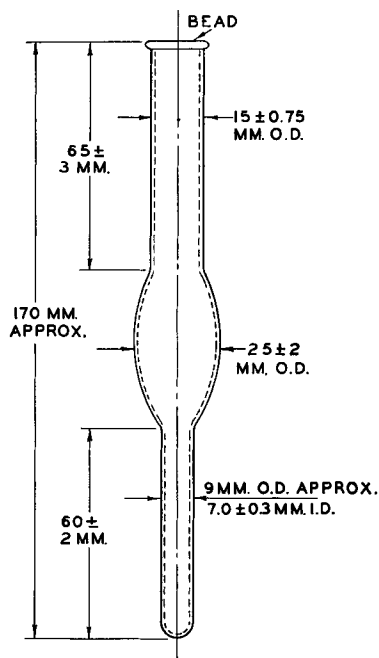


FIG. 179(5). Receiver, volumetric, for modified Clark alkoxyl apparatus—details of construction.



## ELECTRIC HEATER

A small electric heater<sup>53</sup> of the type used in the construction of the Kjeldahl digestion rack (Fig. 180) is particularly suitable for heating the contents of the reaction flask. Gas microburners of the type shown in Fig. 181 are also suitable, but the author prefers the use of the electric type since, with these, boiling is more easily controlled.



⑥ RECEIVER, GRAVIMETRIC.

FIG. 179(6). Receiver, gravimetric, for modified Clark alkoxyl apparatus—details of construction.

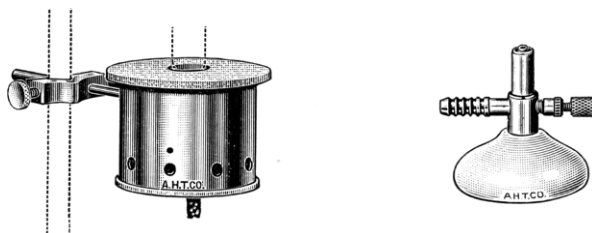


FIG. 180. (Left) Hanks heater.

FIG. 181. (Right) Gas microburner.

## Procedure

### USING THE MODIFIED CLARK

#### APPARATUS (FIG. 179)<sup>99,101</sup>

The scrubber, (2), is filled halfway with the sodium acetate solution and the volumetric receiver, (5), is filled two-thirds full with *freshly prepared* acetic acid-potassium acetate-bromine solution. (If desired, the spiral, (4), may be inserted into the receiver, but extensive tests have shown that equally good results are obtained without it.<sup>101</sup>) Enough sample is added to the reaction flask, (1), to require about 8 ml. of 0.01N sodium thiosulfate in the determination. Solid samples are weighed in a platinum boat (Fig. 24, Chapter 3), while volatile liquids are weighed in the customary capillaries (Fig. 54, Chapter 3), but, *in addition*, an empty platinum boat (or tetrahedra) is added as a means of preventing bumping. Two and one-half ml. of melted phenol is added to the flask, followed by 5 ml. of hydriodic acid. The reaction flask is *immediately* connected to the condenser with scrubber, (2). The source of carbon dioxide is attached to the side arm of the reaction flask and carbon dioxide passed through the apparatus at the rate of about 15 ml. per minute. Cold water is circulated through the condenser and the reaction mixture is allowed to remain at *room temperature for 30 minutes*. The mixture is then heated to boiling by means of the heater (or a microburner) and boiled at such a rate that the vapors of the boiling liquid rise into the condenser, but not more than halfway. Boiling is continued with *water circulating* in the condenser for *one-half hour*, after which the water is *drained* from the condenser and the *boiling continued* for an *additional one-half hour*.

The receiver and inlet tube are disconnected, the stopper removed from the end of the siphon, the unit tilted, and the contents siphoned into a 125-ml. ground glass-stoppered Erlenmeyer flask which contains 5 ml. of the sodium acetate solution. The receiver and inlet tube are washed with enough water so that the washings when added to the Erlenmeyer flask bring the total volume in it to about 50 ml. Formic acid is added, dropwise, with swirling of the flask, until the excess of bromine has been destroyed. Any remaining bromine vapors should be removed by blowing air over the liquid. To the contents of the flask are added 0.5 gram of potassium iodide and 5 ml. of the 10% sulfuric acid. The flask is stoppered and swirled to dissolve the potassium iodide crystals and to mix the contents. The liberated iodine is titrated with 0.01N sodium thiosulfate using starch as the indicator, as described under the standardization of thiosulfate (see Chapter 5). (Any blank value obtained by carrying out the determination in the absence of a sample should be subtracted from the volume of thiosulfate obtained with the sample. However, if the hydriodic acid has been properly treated and if all the reagents are of reagent grade, there should be absolutely no blank.)

*Calculations:*

*Factors:*

1 ml. of 0.01*N* sodium thiosulfate is equivalent to 0.05173 mg. of methoxyl ( $\text{OCH}_3$ ) or 0.07510 mg. of ethoxyl ( $\text{OC}_2\text{H}_5$ )

$$\therefore \frac{\text{ml. of } 0.01N \text{ Na}_2\text{S}_2\text{O}_3 \times 0.05173 \times 100}{\text{Wt. sample}} = \text{per cent } \text{OCH}_3$$

and

$$\frac{\text{ml. of } 0.01N \text{ Na}_2\text{S}_2\text{O}_3 \times 0.07510 \times 100}{\text{Wt. sample}} = \text{per cent } \text{OC}_2\text{H}_5$$

*Examples:*

*a.* 6.82 ml. of 0.01*N* thiosulfate is required to titrate the iodine liberated in the analysis of a 3.348-mg. sample, containing methoxyl groups

$$\therefore \frac{6.82 \times 0.05173 \times 100}{3.348} = 10.54\% \text{ OCH}_3$$

*b.* 6.03 ml. of 0.01*N* thiosulfate is required to titrate the iodine liberated in the analysis of a 4.012-mg. sample containing ethoxyl groups

$$\therefore \frac{6.03 \times 0.07510 \times 100}{4.012} = 11.29\% \text{ OC}_2\text{H}_5$$

The allowable error is  $\pm 0.3\%$ .

#### USING THE STEYERMARK APPARATUS<sup>97,100</sup>

The volumetric (iodometric) method may be performed with the Steyermark apparatus (Fig. 182) described below after substituting the test tube, *g*, with the volumetric receiver [Fig. 179, (5)]. All reagents are the same as used for the volumetric method described above (using the modified Clark alkoxy apparatus). With the Steyermark apparatus and volumetric procedure, the scrubber, *d*, is filled halfway with the sodium acetate solution, and the volumetric receiver [Fig. 179, (5)] is two-thirds filled with the acetic acid-potassium acetate-bromine solution. The rest of the manipulation is as described below and the reader is advised to refer to this, a *summary* of which is as follows. Water is run through the condenser (Fig. 182, *c*). The reaction mixture in the flask, *a*, is allowed to remain at *room temperature* for *one-half hour* while the hydriodic acid in the flask, *b*, is *boiled* and a stream of carbon dioxide is passed through the setup. The mixture in the reaction flask, *a*, is then brought to a boil and the contents of *both* flasks, *a* and *b*, boiled for an additional one-half hour with the water running through the condenser, *c*, and then for *at least* another one-half hour after the water has been drained from the condenser, *c*. The volumetric receiver is then removed, emptied, etc., as described above and the liberated iodine titrated. The calculations are the same as above.

## GRAVIMETRIC METHOD

This method gives good results for volatile substances as well as those containing more than one group although both of these types are known to present difficulties when analyzed by other procedures.<sup>33,44,45,97,100</sup>

During the course of the determination, a small amount of silver sulfide is formed resulting from the decomposition of the thiosulfate used in the scrubber. In addition, there is formed some reduced silver caused by allowing

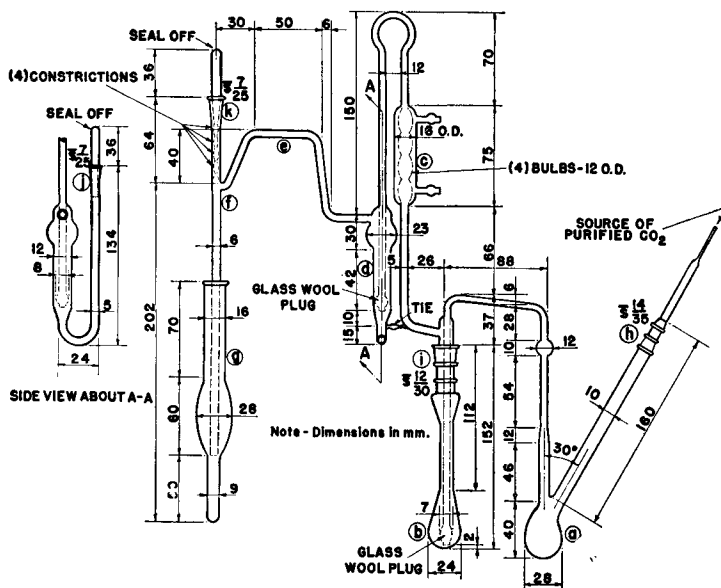


FIG. 182. Steyermark alkoxyl apparatus, gravimetric—details of construction.

the acid alcoholic silver nitrate solution to stand too long before filtration of the silver iodide. Both of these are removed by treating the precipitate with cold concentrated nitric acid<sup>97,100</sup> until the former is yellow in color.

## Reagents

**PHENOL**<sup>33,50,51,82-84,86-89,97-100</sup>

Pure crystalline phenol is used as a solvent for the sample. (*Caution:* This must be handled with care.)

**PROPIONIC ANHYDRIDE**<sup>33,83,97,100</sup>

Pure propionic anhydride is used also as a solvent for the sample.

**TIN FOIL**<sup>50,51,82,84,86-89,97,100</sup>

Thin, pure tin foil is cut into pieces about 2.5 sq. cm. in area and then rolled so that they will pass through the tube into the reaction flask.

**CADMIUM SULFATE, 5%**<sup>33,50,51,82-84,86-89,97,100</sup>

A 5% solution of cadmium sulfate in distilled water is prepared using reagent grade of material. This is used in the scrubber to remove hydrogen sulfide which might be formed in the determination.

**SODIUM THIOSULFATE, 5%**<sup>50,51,86-89,97,100</sup>

A 5% solution of sodium thiosulfate in distilled water is prepared using reagent grade of material. It is added to the scrubber to trap any iodine in the reaction flask.

**ALCOHOLIC SILVER NITRATE, 3.85%**<sup>50,51,82,86-89,97,100</sup>

Four grams of reagent grade of silver nitrate crystals is dissolved in 100 grams of 95% ethanol. The resulting solution is refluxed for 4 hours using a flask and condenser having ground glass joints. The flask is then disconnected from the condenser, closed with a ground glass stopper, and set aside in the dark for one week before using. The solution is then carefully decanted from the separated silver and stored in a brown glass-stoppered bottle. The solution may be used for as long as five to six months but not longer or low results will be obtained.

**HYDRIODIC ACID, SP. GR., 1.7**

Same as for the Volumetric (Iodometric) Method.

**DILUTE NITRIC ACID, 1:200**

This is prepared from pure concentrated nitric acid, sp. gr. 1.42, by diluting 1:200 with distilled water. It is used in transferring and in washing the precipitate.

**CONCENTRATED NITRIC ACID, SP. GR., 1.42**

Concentrated nitric acid, sp. gr. 1.42, is used for washing the precipitate to remove silver sulfide and reduced silver.<sup>97,100</sup>

**ETHANOL, 95%**

Ethanol, 95%, is used in transferring and washing the precipitate.

## Apparatus

### CARBON DIOXIDE SOURCE

This is identical to that described in connection with the volumetric procedure and includes: Carbon dioxide cylinder equipped with reducing valve, wash bottle or scrubbing tower containing sodium carbonate solution, and bubble counter containing concentrated sulfuric acid.

### STEYERMARK ALKOXYL APPARATUS<sup>97,100</sup>

Fig. 182 gives details of construction. It combines the advantageous features of two others which likewise proved satisfactory, namely, those of Elek<sup>33</sup> and of Furter.<sup>44,45</sup>

The apparatus consists of a reaction flask, *a*, which has an inlet tube for the passage of carbon dioxide and a distilling head connected to a tube passing to the bottom of a second reaction flask, *b*. Glass wool or sintered glass is used at the point shown to insure small bubbles. The flask, *b*, is connected to the reflux condenser, *c*, which in turn is connected to the scrubber, *d*. Glass wool or sintered glass again is used as shown for breaking up bubbles. The scrubber, *d*, is connected by means of a side arm, *e*, to the tube, *f*, which passes into the test tube (receiver), *g*, used for collecting the precipitate. Ground joints are present at the points, *b*, *i*, *j*, and *k*.

### ELECTRIC HEATERS<sup>53</sup> OR MICROBURNERS

Same as for the Volumetric (Iodometric) Method. Two are required, one for each reaction flask.

### FILTER TUBE

Same as used in Chapter 11 (Fig. 158).

### FILTRATION ASSEMBLY

Same as used in Chapter 11 (Fig. 160).

## Procedure<sup>97,100</sup>

### USING THE STEYERMARK APPARATUS<sup>97,100</sup>

The sample (5–9 mg.), if solid or a high-boiling liquid, is weighed in a platinum boat and placed in the reaction flask (Fig. 182, *a*). [Volatile samples are weighed in the customary capillaries (Chapter 3) and inserted along with a platinum boat, or tetraheda, for prevention of bumping.] Several crystals of phenol and five to six drops of propionic anhydride are added and the mixture warmed (unless the sample is volatile) to dissolve the sample. Hy-

driodic acid is added to the second reaction flask, *b*, so that the bulb portion is about one-half full and the flask is then put into place, using a drop of the acid to seal the joint. Equal parts of 5% solutions of cadmium sulfate and sodium thiosulfate are added to the scrubber, *d*, to fill it halfway. Water is run into the tube, *f*, through the ground joint, *k*, and the stopper is attached quickly so as to have the constrictions filled with water. The source of carbon dioxide is attached at the ground joint, *b*, and the gas is bubbled through the apparatus until the excess water in the tube, *f*, has been forced out (usually a minute or less). Two milliliters of 3.85% alcoholic silver nitrate is placed in the test tube (receiver), *g*, and this is put into place so that the delivery tube, *f*, goes to the very bottom. The ground joint, *b*, is opened and a piece of the tin foil (2.5 sq. cm. in area, then rolled to pass through the opening) is added to the flask, *a*, followed by enough hydriodic acid to fill the bulb part of the flask approximately half full. The source of carbon dioxide is *immediately* attached, using a drop of the acid to seal the joint.

The flow of gas is regulated so that the bubbles pass through the silver nitrate in the test tube (receiver), *g*, at the rate of about one bubble per second. Water is run through the condenser, *c*, and a heater, or microburner, is used to heat the acid in the flask, *b*, to gentle boiling. (*Caution:* Fuming acid is formed upon passing gas through at room temperature.<sup>76,97,100</sup>) In this condition, the apparatus is allowed to remain for one-half hour. The reaction mixture in the flask, *a*, is then heated to boiling by means of a heater, or microburner, and the boiling of the contents of the two flasks, *a* and *b*, is continued for one-half hour.

At the end of this period, the water is drained from the condenser, but the boiling of the contents of *both* flasks is *continued* for *at least* an additional one-half hour. The stopper of the ground joint, *k*, is then opened, the test tube (receiver), *g*, lowered, and the precipitate washed into the test tube, *g*, alternately and by means of 1:200 nitric acid and ethanol. Then 1:200 nitric acid is added to the test tube until it is about four-fifths full. Five drops of concentrated nitric acid, sp. gr. 1.42, is added and the contents of the tube are brought quickly to the boiling point on a steam bath and then *immediately* cooled and filtered into a filter tube (Fig. 158, Chapter 11) with the aid of the filtration assembly (Fig. 160, Chapter 11). The silver iodide precipitate is washed with 1:200 nitric acid and then with cold concentrated nitric acid, sp. gr. 1.42, by filling the filter tube with the acid, allowing it to soak through for several minutes and then sucking dry. (*Caution:* Silver iodide is insoluble in nitric acid, sp. gr. 1.42, but is quite soluble in fuming acid, sp. gr. 1.49–1.50.<sup>97,100</sup>) The washing should be repeated several times until the precipitate is *yellow*. The precipitate is then washed with 1:200 nitric acid followed by ethanol and dried in an oven at 120° C., after which it is weighed (refer to Chapter 11).

*Calculations:*

An empirical correction of 0.120 mg. of silver iodide (0.06 mg. per ml. of silver nitrate used) must be added to the weight of the precipitate.<sup>37-39,50,51,82,86,87,89,100</sup>

*Factors:*

For  $\text{OCH}_3$ , 0.1322  
 $\text{OC}_2\text{H}_5$ , 0.1919

$$\therefore \frac{\text{Wt. of precipitate (corrected)} \times \text{factor} \times 100}{\text{Wt. sample}} = \text{per cent } \text{OCH}_3, \text{OC}_2\text{H}_5$$

*Examples:*

a. 9.063 mg. of AgI, corrected (8.943 mg., actual, plus 0.120 mg. correction), is obtained from a 7.801-mg. sample containing methoxyl groups

$$\therefore \frac{9.063 \times 0.1322 \times 100}{7.801} = 15.36\% \text{ OCH}_3$$

b. 8.137 mg. of AgI, corrected (8.017 mg., actual, plus 0.120 mg. correction), is obtained from a 6.802-mg. sample containing ethoxyl groups

$$\therefore \frac{8.137 \times 0.1919 \times 100}{6.802} = 22.96\% \text{ OC}_2\text{H}_5$$

The allowable error is  $\pm 0.3\%$ .

**CLEANING THE APPARATUS**

The apparatus is best cleaned, after draining off the solutions from the flasks and scrubber, by immersing the delivery tip, *f*, in water and applying suction to the flask, *a*. After the apparatus is cleaned by the passage of water it may be dried either by placing in an oven or by passing acetone through it followed by a stream of warm air.

**USING THE MODIFIED CLARK APPARATUS<sup>99,101</sup>**

The gravimetric method may be performed with the modified Clark apparatus (Fig. 179 described above) using the gravimetric receiver, (6). All reagents are the same as used for the gravimetric method described above (using the Steyermark alkoxy apparatus). With the modified Clark apparatus and gravimetric procedure, cadmium sulfate and sodium thiosulfate are used in the scrubber and alcoholic silver nitrate is used in the gravimetric receiver. The silver iodide is treated as above, including washing and weighing.

*Calculation:*

Same as for Gravimetric Method above.



TABLE 28  
ADDITIONAL INFORMATION ON REFERENCES\* RELATED TO CHAPTER 16

In addition to the procedures described in the preceding pages of this chapter, the author wishes to call to the attention of the reader the material presented in the various references listed in Table 28. (See statement at top of Table 4 of Chapter 1, regarding completeness of this material.) This includes work on the determination of higher homologs, simultaneous determination of ethoxyl and methoxyl, and the determination of alkyl groups attached to sulfur instead of to oxygen. Repeated statements in the literature that the hydriodic acid must be fresh and colorless are in error.<sup>97,100</sup> The use of various scrubber solutions, particularly the thiosulfate has been attacked, chiefly by Heron and co-workers.<sup>54</sup> This led to the use of sodium acetate in the volumetric procedure which was adopted by the Association of Official Agricultural Chemists.<sup>99</sup>

**Books**

Association of Official Agricultural Chemists, 4  
Belcher and Godbert, 12, 13  
Clark, E. P., 28  
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**Simultaneous determination of ethoxyl and methoxyl**

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**Simultaneous determination of alkoxy and alkimide**

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