vinegar solids. The ash should constitute at least 6 per cent. of the solids. The alkalinity of 1 gram of ash should be equivalent to at least 65 cc. of tenth-normal acid. At least 50 per cent. of the phosphates in the ash should be soluble in water. The reducing sugars should be the same in amount after as before inversion, and should not exceed 25 per cent. of the solids. The polarization, expressed in terms of 200 mm. of undiluted vinegar, should lie between —0.1° and —4.0° Ventzke. Malic acid should be indicated by both the calcium chloride and lead acetate test.

A standard for malic acid is desirable, but before suggesting such, a larger number of determinations should be made than those hitherto recorded. The writers hope to cover this point more completely in the near future.

Routine Examination of Vinegar for Adulteration.—In determining whether or not an alleged vinegar is spurious, it is rarely necessary for the public analyst to make a complete analysis. Aside from the determination of acidity and total solids, by far the most important tests consist in the polarization and in the calcium chloride and lead acetate tests for malic acid. It is rare that spurious vinegar will fail of detection by at least one of these tests. Only in doubtful cases is it necessary to go farther. It is well, however, to be able in some cases to confirm one's judgment by added proof, and where litigation is involved a complete analysis may be helpful.

A PORTABLE OUTFIT FOR THE DETERMINATION OF CARBONIC ACID, DISSOLVED OXYGEN AND ALKALINITY IN DRINKING-WATER.

By Fred B. Forbes.

Received February 17, 1904.

Various forms of portable apparatus for different analytical purposes have been described and have proved their usefulness in practice. Having occasion to make many determinations of carbon dioxide, dissolved oxygen and alkalinity in the field, a portable outfit has been devised which has resulted in a considerable economy of time and labor. The following description is published as a suggestion to any one who may have similar determinations to make at present or in the future.

The methods employed for the different determinations are well known, but a brief review will serve to illustrate more clearly the uses for which the various parts of the outfit are intended.

DISSOLVED OXYGEN BY WINKLER'S METHOD.[1]

Reagents.—Saturated solution of manganous sulphate. Solution of potassium iodide in sodium hydroxide; 100 grams potassium iodide, 360 grams sodium hydroxide per liter. Sulphuric acid—1 part H_2SO_4 (1.84) and 1 part water. Starch paste indicator.

Standard Solution.—Sodium thiosulphate, 1 cc. equivalent to 0.0001 gram available oxygen. This solution is standardized against a solution of potassium permanganate (1 cc. $KMnO_4 =$ 0.0007875 gram oxalic acid, $C_2H_2O_42H_2O$).

Procedure.—The sample for analysis is collected in a glass-stoppered bottle of known capacity by means of a tube passing through a rubber stopper to the bottom of the bottle. A second perforation in the stopper allows the air to escape, and enough water is run through the bottle to make sure that the final sample has not been in contact with the air. The temperature of the water at the time of sampling is noted. Remove the stopper from the bottle, and, by means of a pipette, having a long capillary point, add first 1 cc. manganous sulphate, then 1 cc. potassium iodide solution, both solutions being delivered near the bottom of the bottle. Insert the glass stopper, leaving no air bubble, shake well, and allow to settle. When the precipitate has subsided enough to prevent loss, add 2 or 3 cc. of sulphuric acid, insert the stopper quickly, and shake until the precipitate is dissolved. Pour the contents of the bottle into a flask, add a few drops of starch solution, and titrate with sodium thiosulphate to the disappearance of the blue color, taking the first end-point, as the color frequently returns on standing. The actual amount of water acted upon is always 2 cc. less than the capacity of the bottle, due to the 2 cc. of reagents added, which expel an equivalent amount of the sample. The results are calculated in parts per 100,000 and as per cent. saturation at the observed temperature, the amount of oxygen which water will dissolve at different temperatures being obtained from a table. This table is based on that

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[1] *Ber. d. chem. Ges.*, 2843 (1888).

of Roscoe and Lunt,[1] but it is calculated in parts per 100,000 instead of cubic centimeters per liter.

FREE CARBONIC ACID BY SEYLER'S METHOD.[2]

Reagents.—Neutral alcoholic solution of phenolphthaleïn—(5 grams phenolphthaleïn in a liter of 50 per cent. alcohol).

Standard Solution.—N/50 sodium carbonate.

Procedure.—One hundred cc. of the sample are introduced into a tall glass cylinder (100 cc. nitrite tube) by means of a siphon, 5 or 6 drops of phenolphthaleïn added and N/50 sodium carbonate run in from a burette with careful stirring until a faint permanent pink color is obtained. If the water is high in carbon dioxide, it is better to take less than 100 cc., and too violent stirring is to be avoided; at the same time the titration should be quickly completed. The stirring rod used is made by coiling up a piece of glass rod in a circular disk and bending the long end at right angles to the disk, giving a sort of piston which can be moved up and down in the tube, thus thoroughly mixing the contents without introducing air. A second tube containing 100 cc. of the water under examination without the addition of indicator is held alongside of the determination to assist the eye in noting the end-point. The number of cubic centimeters of sodium carbonate required, multiplied by 0.44, gives the free carbon dioxide in parts per 100,000. If the water is neutral or alkaline to phenolphthaleïn, no free carbon dioxide is present.

ALKALINITY, OR FIXED CARBONIC ACID, BY HEHNER'S METHOD.[3]

Reagents.—Methyl orange indicator (1 gram in a liter of water).

Standard Solution.—N/50 sulphuric acid.

Procedure.—Measure 100 cc. of the water into a 6-inch white porcelain dish, add 2 or 3 drops methyl orange and titrate with sulphuric acid to the appearance of a pink color. The number of cubic centimeters of acid used gives the alkalinity in parts per 100,000. Multiplying this reading by 0.44 gives the fixed carbon dioxide.

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[1] Roscoe and Lunt: *J. Chem. Soc.*, **55**, 552 (1889).

[2] Seyler: *Chem. News*, **70**, 104 (1894); *Analyst* **22**, 312 (1897); Ellms and Beneker: This Journal, **23**, 405 (1901); Forbes and Pratt: *Ibid.*, **25**, 742 (1903).

[3] Ellms: This Journal, **21**, 359 (1899).

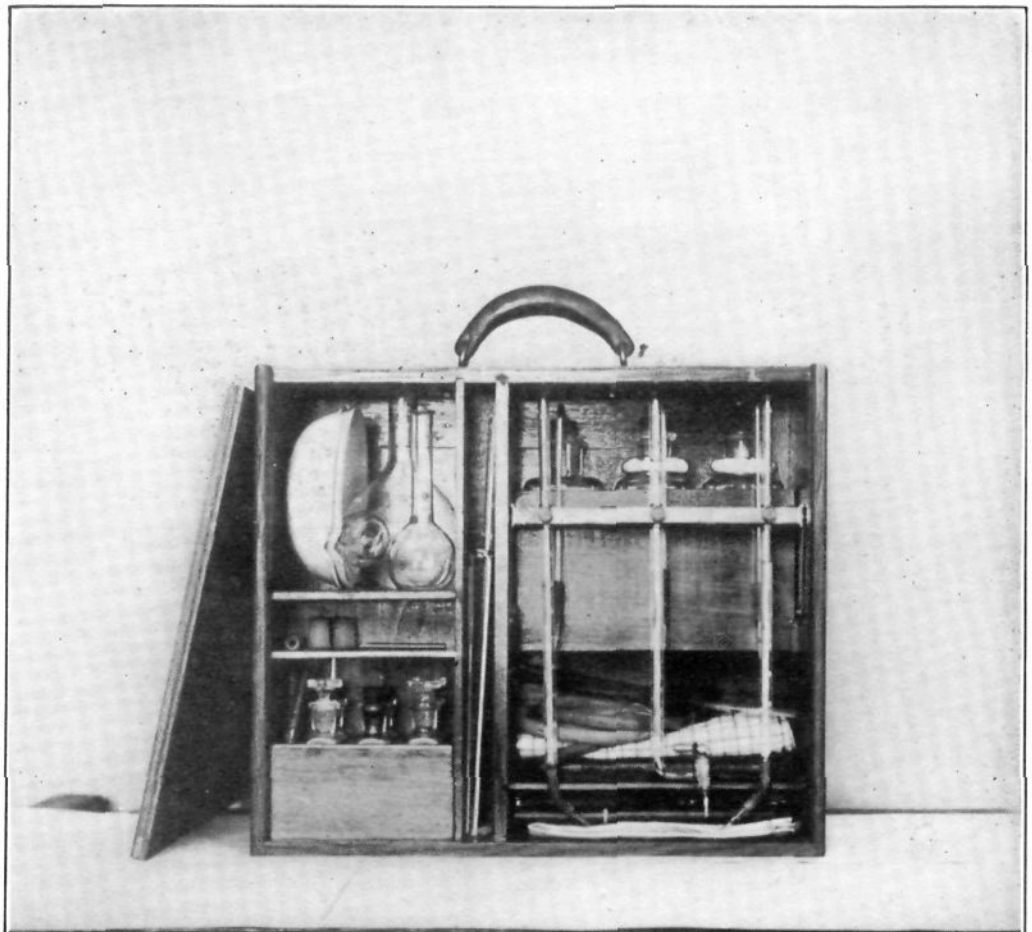

Fig. 1.

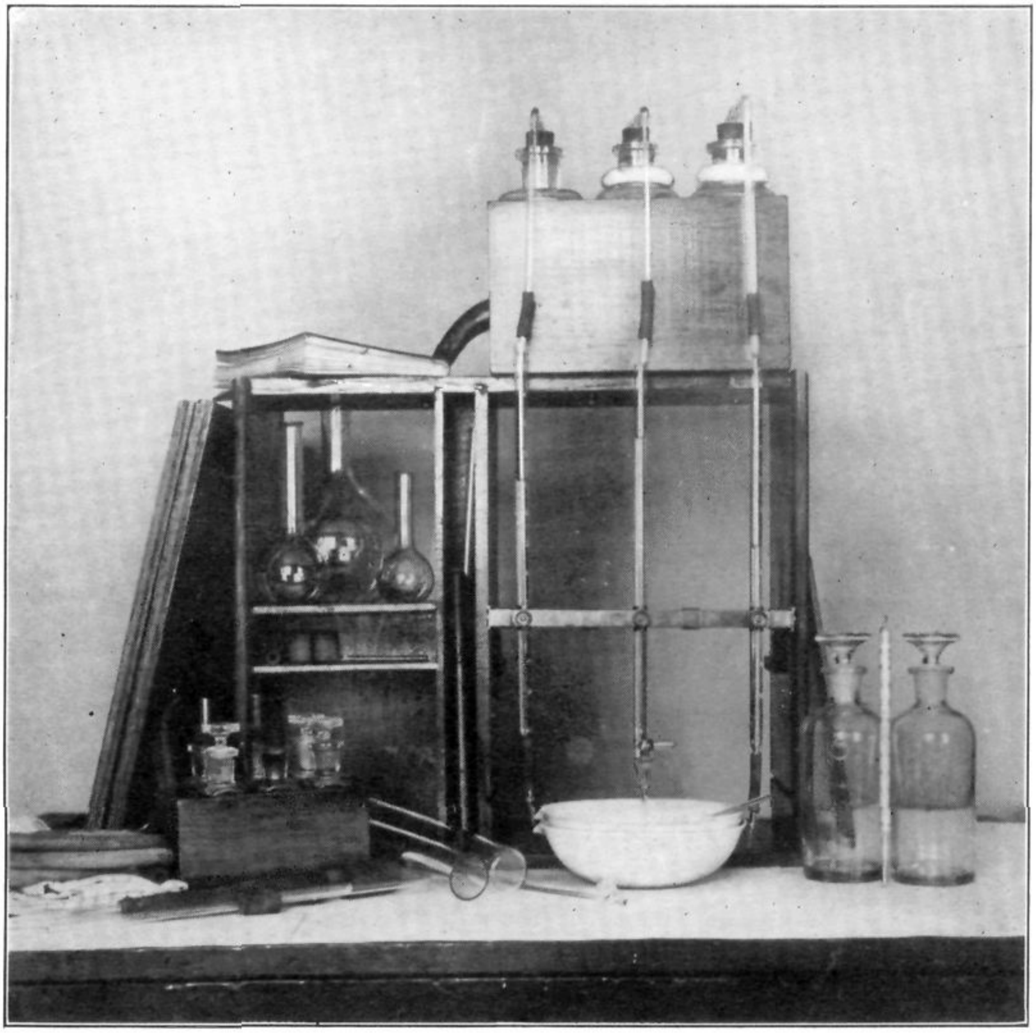

Fig. 2.

CALCULATION OF HALF-BOUND CARBON DIOXIDE.

When the water is acid to phenolphthaleïn, the alkalinity reading multiplied by 0.44 gives the half-bound carbon dioxide in parts per 100,000, as in this case it equals the fixed.

When the water is alkaline to phenolphthaleïn, titrate 100 cc. of the water with N/50 sulphuric acid with 5 or 6 drops phenolphthaleïn until the pink color disappears, observing all the precautions previously noted. Twice the number of cubic centimeters of acid required, subtracted from the alkalinity reading and the results multiplied by 0.44, gives the half-bound carbon dioxide in parts per 100,000.

The apparatus for the various determinations is contained in a box which is 17.5 inches long, 7.5 inches wide and 15 inches high, outside measurements, and is provided with a stout leather handle for carrying. The top and sides of the box are half-inch oak, strongly put together and oil-finished. The bottom is of half-inch, and the inside partitions of one-fourth inch and three-eighths inch whitewood, well shellaced. The sides of the box project a little beyond the top and are grooved to receive the front and back, which are panels of thin oak and slide in at the top. These panels are removed entirely when the box is in use, in order to allow a clear passage of light.

By reference to the following figures the disposition of the apparatus in the box will be seen:

Figure 1 shows the outfit packed ready for carrying, the front panel being removed to show the interior. Figure 2 shows the apparatus ready for use, both front and back panels being removed. Figure 3 shows the construction of the burette holder, which is the only particularly novel feature of the apparatus.

The three standard solutions are contained in square bottles fitting into three compartments in a deep tray. The sulphuric acid and sodium carbonate bottles are provided with U-tubes containing soda-lime to remove carbon dioxide from the air that is drawn into the bottles, and all three bottles are provided with siphons and pinch-cocks to deliver the solution into the burettes. The tray containing these bottles slides into the box at the back on guides, and a stop on either side holds it in place flush with the back panel. When the box is in use, this tray is placed on

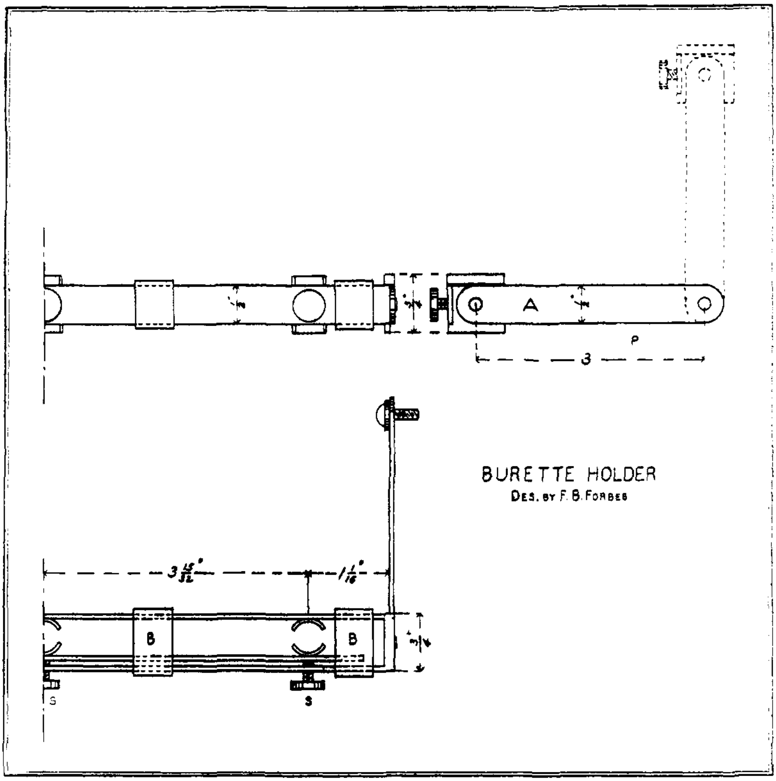

Fig. 3.

the top, when the siphon tubes will enter the burettes which swing out into place, as will be seen from Fig. 2.

In the lower part of this end of the box are three 100 cc. cylinders, two for use and one spare one, and a thermometer. A thin removable slide holds them in place during transportation. On the top of this slide are stored a note-book, towels, rubber tubing and connections, and a double perforated rubber stopper with glass tubes for taking samples.

The dissolved oxygen reagents and the indicators are contained in six small square bottles fitting into compartments in a small removable tray, which has also a narrow compartment in one end for the 1 cc. capillary pipettes. This tray takes up only about half the width of the box, and the other half is divided into two compartments for the storage of two 8-ounce bottles for the collection of dissolved oxygen samples. These are not shown in Fig. 1, as they are withdrawn from the back of the box. Above the tray of reagents is a small compartment for the storage of extra stoppers and any small articles that may prove of service. This space occurs on account of the dissolved oxygen bottles being higher than the reagent bottles.

Above this space is a large compartment, occupying the entire width of the box, in which are carried two 6-inch porcelain dishes, a 50 cc. and a 100 cc. graduated flask, and a short-necked 500 cc. flask for titrating the dissolved oxygen. These are wrapped in pieces of cloth when the case is closed for carrying.

The narrow space between the two vertical partitions was originally intended for a different sort of burette holder, and may be dispensed with, thus shortening the box an inch. It is utilized to carry an extra burette, stirring rods, etc.

Figure 3 shows plan and elevation of one-half of the burette holder, also a view of one end, the construction of the other half being identical. The material is **brass**, $^1/_2$ inch in width and $^1/_{16}$ inch in thickness.

To hold each 10 cc. burette are two curved pieces of metal projecting a little above and below the principal frame, the back one being soldered to the frame, while the front one is soldered to a strip of brass which slides back and forth between the strengthening bands B B, and is held firmly against the burettes by the thumbscrews S S. This sliding strip is sufficiently flexible so that each burette may be manipulated independently.

When the burettes are in use, the end bar A is in a horizontal position resting on a small pin P, which is driven into the sides of the compartment in which the whole arrangement fits closely. When the box is to be packed, the three burettes are lowered in their places, about 3 inches, by loosening the thumb screws, and the end bars swung through an angle of 90° to the position shown by the dotted lines, the burettes remaining vertical, as the frame which carries them is pivoted on the end bars. The burettes are now inside the box and the front panel may be put in place.

The advantage of this arrangement over any common form of clamp is that it takes up little room, does not add much to the weight of the outfit, and provides for three burettes which are always ready for use, it being unnecessary even to draw off the solutions. These features make it very convenient, especially if a number of determinations are to be made at points some distance apart.

If a table or bench is at hand, the box is placed in the position shown in Fig. 2, when the 6-inch porcelain dish can be set conveniently under the glass-stoppered acid burette. When using a 100 cc. tube or the large flask in which dissolved oxygen is titrated, the box is brought forward so that the burettes project over the edge of the table. The outfit can be used, however, under almost any conditions which one is liable to encounter in field work.

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SPRENGEL'S METHOD FOR COLORIMETRIC DETERMINATION OF NITRATES.

BY LAUNCELOT W. ANDREWS.

Received December 29, 1903.

THE colorimetric determination of small amounts of nitrates which is based on the use of phenol and concentrated sulphuric acid, appears to have been first devised and used by Sprengel.[1] It is probably the method now most frequently used for the determination of nitrates in water analysis and is the best of the rapid methods. Sprengel states that the yellow color obtained in the process is due to the formation of picric acid, a statement which has been repeated in text-book after text-book for forty years, seemingly without criticism or examination, until recently Montenari[2] has sought to show that not picric acid, but dinitrophenol is produced in this reaction.

Previous to the publication of Montenari, I had been impressed with the inherent improbability of the formation of trinitrophenol under the conditions of the experiment, that is, with the phenol in great excess as regards the nitric acid, and had also observed that the yellow coloring-matter formed does not give the characteristic reactions of picric acid. Since the conclusions of Montenari also appeared extremely improbable, I thought it worth

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[1] Pogg. *Ann.*, **121**, 188 in 1863.

[2] *Gazz. chim. ital.*, **32**-1, 87 (1902).