Summary

A micro method for the determination of surface tension and density has been described which requires not over one-tenth of a cubic centimeter of sample for both determinations, which uses the same apparatus (with the addition of a jacket) for the determination of density as for the determination of surface tension and has an accuracy of from one part in 100 to one part in 300, depending on the liquid.

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[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF WASHINGTON]

IMPROVED APPARATUS FOR THE REMOVAL OF DISSOLVED GASES FROM WATER[1]

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Many different forms of apparatus for the removal of dissolved gases have been described in the literature. In general they consist of three types: those employing vacuum only,[2] which include numerous modifications of the Van Slyke apparatus,[3] those employing heat only,[4] and those employing both heat and vacuum.[5]

Those of the latter type will remove gases from water more completely than the first two, and so an apparatus was designed which combines the good features of the Van Slyke type with those of the apparatus described by Treadwell-Hall. This improved apparatus has been used by the authors with excellent success for the removal of gases dissolved in natural waters. It is adaptable to any size of system or amount of gas, provides means of making a sharp separation of water from gas, employs the minimum number of joints consistent with ease of manipulation and also provides a seal for each joint.

[1] Read before the Chemical Section of the Pacific Coast Division of the American Association for the Advancement of Science.

[2] (a) Frankland, *J. Chem. Soc.*, **6**, 109 (1854); (b) Lothar Meyer, *Z. anal. Chem.*, **2**, 237 (1863); (c) Jones, Yant and Buxton, Bureau of Mines Reports of Investigations, No. 2553, December, 1923.

[3] (a) McClendon, *J. Biol. Chem.*, **30**, 259 (1917); (b) Van Slyke, *ibid.*, **30**, 347 (1917); (c) Van Slyke and Stadie, *ibid.*, **49**, 3, 44 (1921); (d) Austin and others, *ibid.*, **54**, 129 (1922); (e) Hall, *ibid.*, **55**, 751 (1923).

[4] (a) Bunsen, "Gasometrischen Methoden," 1st ed., **1857**, p. 18; (b) Reichardt, *Z. anal. Chem.*, 11, 271 (1872); (c) Jacobsen, *Ann.*, **167**, 12 (1873); (d) Buchanan and Dittmar, "Physics and Chemistry, Report on the Scientific Results of the Voyage of H. M. S. *Challenger*," Longmans and Co., or Macmillan Co., **1884**, I, p. 141; (e) Petterson, *Ber.*, **22**, 1434 (1889); (f) Treadwell-Hall, "Analytical Chemistry," John Wiley and Sons, Inc., New York, **1924**, 5th ed., Vol. II, p. 634, Fig. 128.

[5] (a) McLeod, *J. Chem. Soc.*, **7**, 313 (1855); (b) Hamberg, *J. prakt. Chem.*, **141**, 433 (1885); (c) Hoppe-Seyler, *Z. anal. Chem.*, **31**, 367 (1892); (d) Richardson, *J. Soc. Chem. Ind.*, **29**, 198T (1910); (e) *ibid.*, **38**, 32T (1919); (f) Ref. 4 f, p. 631, Fig. 127.

This apparatus consists of two separate pieces, a modified gas pipet and a special condenser. The latter consists of a $^{5}/_{16}$″ glass tube bent as shown in the diagram (Fig. 1) and surrounded by a water jacket, C. Adjoining this is a large bulb, B, to take care of the expansion of the water sample contained in the bottle when the latter is warmed. The tube, J, on the

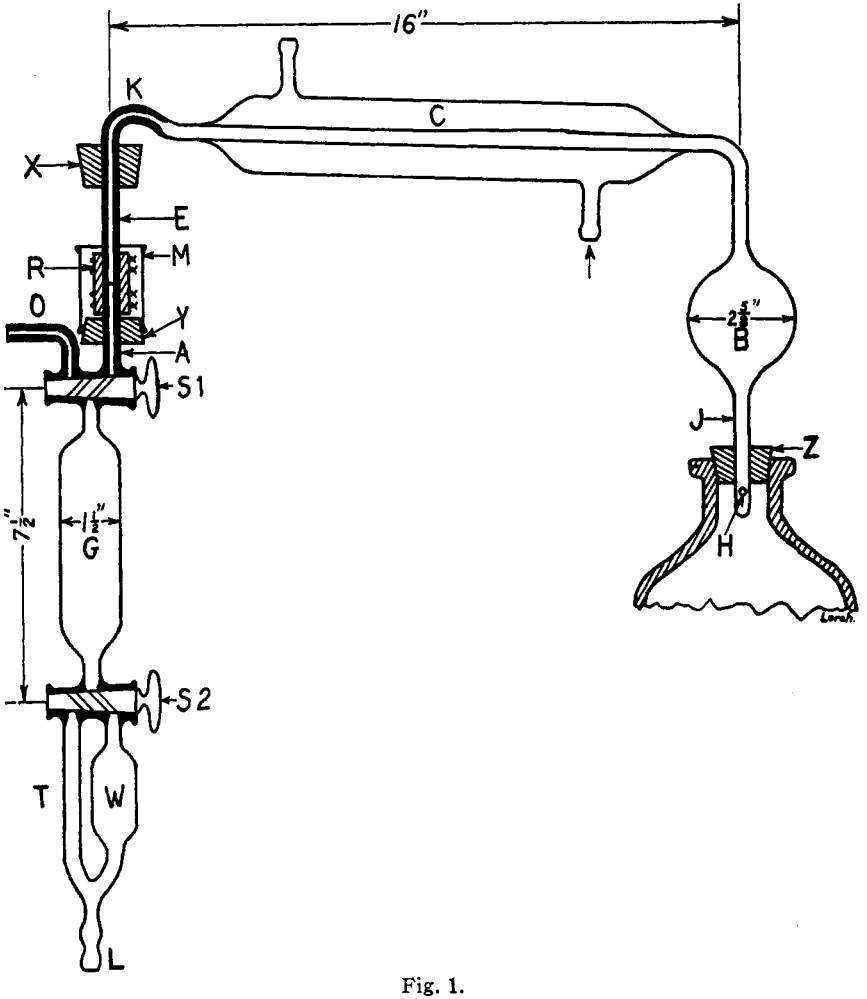

Fig. 1.

bulb, B, is sealed at the end and provided with a hole, H, about half an inch from the end. The opposite end, E, of the condenser tube is made of capillary tubing and is joined to the straight capillary tube, A, of the gas pipet by means of a short piece of heavy-walled rubber tubing, R. To provide a seal for this connection a large glass tube, M, is filled with mercury or water.

The gas pipet, G, is provided above and below with well-fitting stopcocks, preferably of the vacuum type. The upper stopcock, S1, leads to the condenser by means of the tube, A, and to a gas buret or storage pipet by means of the capillary outlet, O. The lower stopcock, S2, leads to the side tube, T, and to a small pipet, W, used for temporary storage of water. Both T and W open into L, to which a leveling bulb filled with mercury is attached. The entire apparatus is best made of Pyrex glass. The approximate dimensions are shown on the diagram, the capacity of B being about 200 cc., G, 100 cc. and W, 20 cc.

Since speed is essential in making the connections, the gas pipet and sample bottle of water must be arranged so that the ends of the condenser will fit with a minimum of readjustment. The condenser is sloped slightly so that the condensed water will run back into the bulb, B. The gas pipet is completely filled with mercury, including capillary tubes O and A.

The manipulation is as follows. Bulb, B, is filled two-thirds full with freshly boiled, distilled water by applying suction to the opposite end of the condenser tube and then stopper, Z, is moved down until it just covers hole, H. The condenser is held so that the water runs into the tube, J, and displaces all the air from it. The original stopper is now removed from the sample bottle and the neck of the latter carefully filled with freshly boiled, distilled water. This sample bottle, of course, has previously been placed in its correct position in the water-bath. Then stopper, Z, is inserted by pushing down on the bulb, B, with one hand and very slowly turning the stopper with the other, thus allowing a little water to be forced out of the bottle as the tube, J, is forced in. The latter is forced down until the top of the hole, H, just barely comes below the bottom of the stopper. As soon as the connection is made the height of the water in the water-bath is increased so that it forms a seal for this joint.

To remove all air from the condenser tube, bulb, B, is heated by a flame until almost all of the water has steamed away. During this operation no water is in the jacket, C, and the entire condenser must be turned horizontally to such an angle that no steam strikes the gas pipet. Then, with one person holding the condenser in position and continuing the heating of the bulb, B, with the flame, another person pushes the end, E, into the wetted rubber connection, R, while steam is still issuing from the end of the condenser tube. The rubber connection is immediately wired and the glass cylinder, M, lowered from stopper, X, to stopper, Y, and filled with water or mercury. While making connections the gas pipet should be protected from steam by means of toweling. Care should be taken to have the lower stopcock open to allow for possible expansion of mercury due to heating.

The sample of water is warmed to any desired temperature. If it is heated by applying external heat to the bottom of the bath it is best to

place blocks of wood under the bottom of the bottle to prevent superheating. A stirring device on the bath is very desirable.

Some water will expand from the sample bottle into the bulb, B. Any desired amount of vacuum may be produced by lowering the leveling bulb containing mercury attached to the bottom of the pipet at L. If any water accidentally enters the gas pipet, G, the mercury may be lowered until the water is drawn into the reservoir, W. Stopcock, S2, is then turned so that the by-pass, T, is open and the gas forced through, O, into a storage pipet or buret. Then the mercury is lowered until it reaches stopcock, S2, again, which is then turned so that as the mercury is raised again the water from W floats on top. It is carefully raised until the bend, K, is reached where the water flows back into the condenser tube. Before attempting this operation the collected gas already extracted must be removed as described.

After all gases have been removed from the sample, the water in bulb, B, is boiled for a couple of minutes to remove any gases present and then the stopper, Z, is loosened. By lowering the leveling bulb, the gas in the condenser tube and bulb is drawn over into the pipet, water entering the system through hole H. The bend, K, and the joint between the condenser tube and the capillary tube, E, enables one to make a clean separation of the gas and water. It was found that if a simple bend was used some gas always remained at the top of the bend.

Summary

A modification of the usual apparatus for removing dissolved gases in water has been described.

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[CONTRIBUTION FROM RESEARCH DEPARTMENT, NATURAL PRODUCTS REFINING COMPANY]

A METHOD FOR DETERMINING THE TENSILE STRENGTH OF GELATIN JELLIES

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Up to the present time the only scientific method for determining jelly strength has been by the use of the Sheppard torsion dynamometer.[1] This apparatus is used for testing the jelly strength of glues and gelatins by the pure shear of moulded cylindrical test pieces.

The following method, first suggested by A. Rosinger in 1923, depends on the static loading of jelly membranes, and is based on the fact that a circular elastic membrane supported rigidly at its circumference and subjected to air pressure on one side assumes a spherical form.

[1] (a) Sheppard and Sweet, THIS JOURNAL, **43**, 539 (1921); (b) Sheppard, Sweet and Scott, *J. Ind. Eng. Chem.*, **12**, 1007 (1920).