[CONTRIBUTION FROM THE LABORATORY OF THE UNITED GAS IMPROVEMENT CO.]

THE QUANTITATIVE ESTIMATION OF HYDROGEN SULPHIDE IN ILLUMINATING GAS.

BY C. C. TUTWILER.

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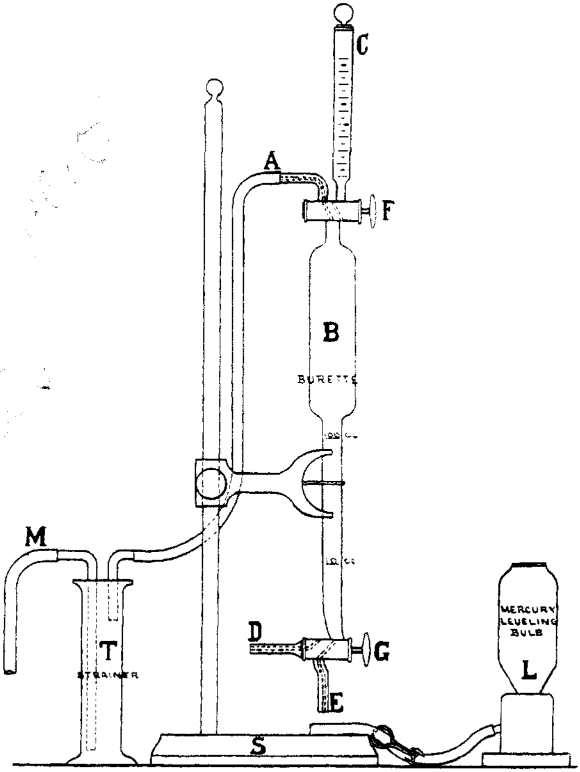
IN the manufacture of illuminating gas the importance of tracing the successive stages of purification of the crude gas from the time it leaves the retorts or water gas machine until it passes the purifiers is obvious, as by this means alone is it possible to determine the efficiency of the purifying plant and of the character of the raw material used, as well as the purity of the finished product. Tests on the purified gas are generally qualitative and are all that is necessary, as in most cities perfect freedom from obnoxious constituents is required by legislative enactment. This is particularly true of hydrogen sulphide on account of its exceedingly injurious products of combustion. The usual qualitative test for this impurity consists in allowing the gas to be tested, to impinge upon a piece of moist lead acetate paper for a few minutes, any discoloration showing the presence of hydrogen sulphide. It is, however, of great importance at times to know the exact hydrogen sulphide content of the unpurified or partly purified gas in order to ascertain the condition of the purifiers and the efficiency of the purifying material used. Where boxes are run in parallel it presents a ready means of ascertaining whether the work to be accomplished is equally distributed or is being done wholly or in part by one set of boxes. In order to ascertain this, it is necessary to make determinations at the inlet and outlet of the purifiers, and to be of value they must be made at practically the same time, hence the necessity for a rapid method of making the analysis.

The methods hitherto adopted in gas works have proved unsatisfactory on account of the slowness of the usual gravimetric estimations and the necessary manipulative skill required in making the determinations, besides a laboratory equipment not usually possessed by the smaller plants throughout the country. It was to overcome these difficulties that the apparatus about to be described was devised. Its action depends upon the well-known reaction between hydrogen sulphide and iodine according to the equation

$$H_2S + I_2 = 2HI + S,$$

hydrogen iodide and free sulphur being formed. Although the suitableness of iodine for estimating hydrogen sulphide in illuminating gas was suggested by Bunte many years ago, it seems never to have come into general use, due possibly to the great care necessary to obtain accurate results with a Bunte burette. The apparatus used in The United Gas Improvement Company's Works is shown in the accompanying sketch. It consists of a burette provided at top and bottom with three-way stop-cocks and communicating at the top through one of the outlets with a 10 cc. glass stoppered cylinder graduated into 0.1 cc. There are only two



graduations on the stem of the burette, one at the 100 cc. mark, the other, 50 mm. from the bottom stop-cock, dividing the remaining space into two divisions of about 5 and 10 cc. respectively. A mercury leveling bulb is attached to the lower stop-cock at E, and the burette mounted on a stand as indicated. When analyses are to be made on crude gas a tar-arrester consisting of a wash-bottle containing cotton is placed between the gas supply and the burette. The manner of using the burette is as follows:

Turn both stop-cocks so that A and E communicate with the interior of the burette. Connect gas supply to A, or to M, if testing crude gas, and let gas flow through the burette and out at E for a few minutes, thereby displacing the air by the gas to be tested for hydrogen sulphide. Close the bottom of the burette by turning G so that E communicates with D. Close F and disconnect from the gas supply at A. Allow the burette to stand for a few minutes until the gas reaches the temperature of the surrounding atmosphere, which should be noted. Attach leveling bulb tube to E and raise the mercury bulb until the mercury passes into the stop-cock, G, thus removing air from the rubber tube and E. Turn the cock so that E communicates with the interior of the burette, and bring the mercury up to the 100 cc. mark, thereby compressing the gas in the burette. Close the lower stop-cock and open B to A momentarily, in order to bring the gas in the burette to atmospheric pressure. Open B to E and draw the mercury back to G, closing the stop-cock by turning so that E communicates with D. We now have 100 cc. of gas measured at atmospheric temperature and pressure, under a negative pressure. Place the clip on the rubber tube and remove the same from E. Take the burette from the stand and by opening B to A allow the partial vacuum to draw into the burette about 5 cc. of starch solution from a portion contained in a small beaker. Close the cock and fill the graduate with a standard iodine solution, noting the reading. Admit the iodine solution into the burette, gradually shaking between each addition of three or four drops. Continue until the starch paste assumes a permanent blue color. Note the reading on the graduate which, subtracted from the previous reading, gives the amount of solution used. This, being multiplied by the number of grains 1 cc. iodine solution is equivalent to, gives directly grains H_2S per 100 cubic feet.

Suppose, therefore, that 5.2 cc. of iodine were required to give the blue color and 1 cc. = 100 grains, then the amount of hydrogen sulphide would be 5.2 × 100 = 520 grains per 100 cubic feet.

The strength of the iodine solution used is generally such that 1 cc. contains 0.0017076 gram of iodine per cubic centimeter. One cubic centimeter of this will then be equivalent to 100 grains H_2S per 100 cubic feet of gas.

Precautions.—The following precautions are necessary:

(1) For very accurate work, the starch solution should be previously treated with just enough iodine solution to color it faintly.

(2) The blue color must not be confused with the opalescent milky appearance given to the solution by the separation of free sulphur.

(3) For very accurate work, introduce correction for temperature and pressure, bringing the gas to 60° F. and thirty inches.

Extended use of the burette in our works has established its value beyond question as a quick and accurate substitute for the older methods. Very little skill is required in handling the apparatus, no more than can be acquired in a short time by any one of average intelligence. It will readily be seen that any variation in the strength of the iodine solution introduces no error in a determination of the percentage of impurity removed by a given purifier; for example, if it was found that single tests of the gas, before and after leaving a purifier, showed 500 and 400 grains of hydrogen sulphide per 100 feet respectively, assuming that 1 cc. of the iodine solution is equivalent to 100 grains, then the efficiency of the purifier is 20 per cent.; that is, it is removing 20 per cent. of the total hydrogen sulphide impurity. If a subsequent examination of the iodine solution showed that 1 cc. was equivalent to 110 grains of H_2S, the calculations on this basis would still show that the purifier was taking out 20 per cent. of the hydrogen sulphide in the gas, for instead of there having been 500 grains in the inlet gas, there would actually have been 550, and the outlet gas would have had 440, 110 grains having been removed or 20 per cent. of total content as before. Errors of manipulation common to both determinations are also eliminated.

Note.—Since the above article was admitted for publication, a paper has been read by Mr. Carroll Miller, of Newark, before the New England Association of Gas Engineers,[1] giving in detail results of experiments on purification conducted with this apparatus. The employment of starch paste solution instead of mercury in taking the sample, is perhaps an advantage, as it is easier to handle and does away with the necessity in very exact work for drying out the burette after each determination. In using starch paste solution the burette is previously filled with the same, and the sample of gas taken by allowing the liquid to run out down to the 100 cc. mark, having previously purged the connecting hose with the gas to be analyzed. A question as to the accuracy of the apparatus brought from Mr. Miller the opinion that 15 grains of hydrogen sulphide per hundred feet of gas could escape detection. Before the apparatus was put on the market it was tested and found to be susceptible to as small a quantity as 3 grains per one hundred feet.

ON TRITOLYLCHLORMETHANE.

BY M. GOMBERG AND O. W. VOEDISCH.

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ONE of us[2] published recently a method for the preparation of triphenylchlormethane. We have tried the same reaction on toluene and find that the corresponding tritolyl compound can be obtained in this way. As the action of aluminum chloride upon a mixture of carbon tetrachloride and toluene was rather energetic we added, as is customary, a large excess of carbon disulphide in order to lower as much as possible the temperature of the reaction and to prevent the splitting off of the methyl groups. The yield of the tritolylchloride was in this way increased, but is still far from satisfactory. The compound was purified similarly to the triphenyl derivative. Sixty-two grams of carbon tetrachloride, 100 grams toluene, 150 cc. carbon disulphide, and 85 grams aluminum chloride, give about 22 grams of the chloride derivative, as we found from ten trials of this reaction.

	Calculated for $(C_6H_4CH_3)_3CCl$.	Found. I.	II.
Chlorine	11.08	9.93	10.85

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[1] See *American Gas Light Journal*, **74**, No. 9, p. 325.

[2] This Journal, **22**, 752.