according to this method, with results tabulated below. Both salts employed were chemically pure crystals containing twelve molecules of water.

Per cent. known to be present.		Per cent. found.	
Na_3PO_4.	Na_2HPO_4.	Na_3PO_4.	Na_2HPO_4.
8.63	31.72	8.11	32.81
12.94	27.76	11.23	28.46
17.26	23.79	17.98	23.10
23.74	17.84	24.21	16.89
30.11	11.89	31.93	11.09
32.37	9.92	32.71	10.16
34.52	7.93	33.32	9.11
36.68	5.94	36.84	6.17

If alkaline carbonates are present in the original salts they must be determined by the Schrötter apparatus and the necessary correction applied to the titration.

LABORATORIES OF GEO. W. LORD COMPANY, PHILADELPHIA.

A DESCRIPTION OF IMPROVED APPARATUS AND OF A MODIFICATION OF DREHSCHMIDT'S METHOD FOR THE DETERMINATION OF TOTAL SULPHUR IN COAL GAS.[1]

BY EVERHART PERCY HARDING.

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OF ALL methods[2] discussed and used during the last half century for the determination of total sulphur in coal gas the one proposed and used by Drehschmidt appears[3] to be the most convenient and to give the most reliable results.

The method of Drehschmidt consists in the oxidation of the sulphur to sulphur dioxide in a supply of air freed from hydrogen sulphide, of the conversion of the sulphur dioxide into potassium sulphite by its absorption in a 5 per cent. solution of potassium carbonate, of the oxidation of the sulphite to potassium sulphate with bromine and of the subsequent precipitation of the sulphur with a solution of barium chloride, and the calculation of the amount of sulphur from the weight of the ignited barium sulphate.

[1] The writer is indebted to Mr. O. L. Bernhagen for the drawing of the apparatus used in this paper.

[2] Chem. News, 1861, p. 38; 1863, p. 73; 1868, p. 89; Z. anal. Chem. **15**, 175; **21**, 335; **22**, 171. Hempel: "Gas Analyse," Dritte Auflage,s. 304.

[3] Chem. Ztg. **11**, 1382 (1887.)

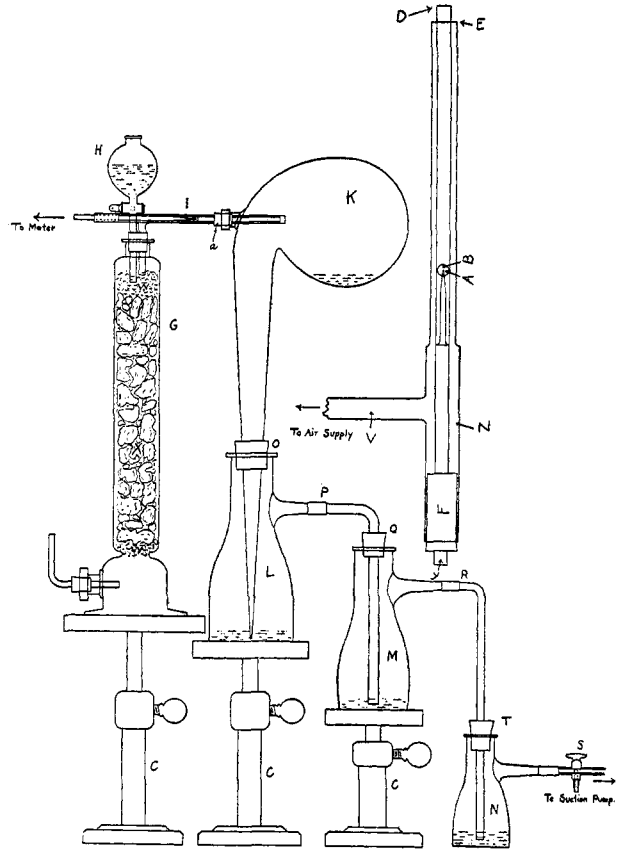
The apparatus used by Drehschmidt[1] consists of a gas meter for measuring the gas, of a drying tower containing pieces of pumice stone saturated with a solution of potassium hydroxide for removing hydrogen sulphide from the air, of a specially constructed Bunsen burner for burning the gas, of a cylindrical glass mantle which rests in a mercury seal and which encloses the burning gas and conducts the products of combustion to the absorption apparatus, of an absorption apparatus which is connected at one end by a ground glass joint to the upper end of the mantle which terminates in a glass tube bent downward and at the other end to an aspirator, and of an aspirator to produce a draft and to draw the products of combustion through the absorbing solution.

The writer of this paper has always experienced difficulties in manipulating Drehschmidt's apparatus. In the first place it was always difficult to regulate the gas supply and to control the aspiration so as to insure both a complete consumption and a continuous combustion of the gas. In the second place, the moisture of combustion produced during the first part of the determination would condense on the walls of the mantle holding some sulphur dioxide in solution, collect into large drops and flow down into the mercury seal, thus insuring loss of sulphur dioxide. Other objections to this apparatus are its delicately constructed absorption flasks and the non-flexible ground glass joint connecting the mantle with the absorption apparatus which, in the hands of inexperienced students, may be easily broken and which can be replaced only at an expense. Lastly, it is expensive.

The principle of the method in use by the writer consists in the combustion of the gas in a specially constructed hard glass burner within the body of a large retort in the presence of bromine vapors, the aspiration of the products of combustion together with some bromine vapors through a 5 per cent. solution of potassium carbonate, and the precipitation and weighing of the sulphur as barium sulphate. The apparatus with the exception of the burner is of the simplest construction and can be set up at once in any working laboratory.

A description of the apparatus is as follows: G is a purifying

tower 45 cm. high and 6.3 cm. in diameter filled with pumice stone contained between the glass wool layers WW. H is a dropping funnel for delivering continuously the potassium hydroxide solution. K is a retort 24 cm. in depth, 16 cm. in cross-section with a delivery tube 46 cm. in length and which is fitted into the



IMPROVED APPARATUS FOR THE DETERMINATION OF TOTAL SULPHUR IN COAL GAS

flask L by the rubber stopper O. L, M and N are heavy glass suction bottles with the respective diameters 10 cm., 8.9 cm., 6.3 cm., and are connected at P and R with closely fitting rubber tubing. C, C′ and C″ are adjustable wooden supports. S is a glass cock connecting the apparatus with the suction pump for

minutely adjusting the aspiration. I is a burner connected at *y* with the meter at V by means of a short piece of rubber tubing, with the purifying tower G, and which is fitted into the tubule of the retort with the rubber stopper *a*. The burner[1] which is represented in the drawing is made of hard glass tubing. E the air supply tube is 9 mm. in diameter at E and 12 mm. at Z. D the gas supply tube is 5.8 mm. in diameter with opposite holes B for the admission of air and drawn out at A to an opening sufficiently small to deliver, under aspiration, between 0.35 and 0.5 cubic foot of gas per hour.

To make a total sulphur determination the pumice stone is saturated with a 33⅓ per cent. solution of potassium hydroxide and the solution allowed to drop slowly and continuously from G during the process. The burner is connected with the meter and the tower, the gas turned on, ignited and allowed to burn for fifteen minutes. The retort and the absorption flasks in the meantime are cleaned and supplied with a 5 per cent. solution of potassium carbonate, 30 cc. being put into N, 30 cc. into M, 50 cc. into L, and 30 cc. together with 4 cc. of bromine into the retort K, and the flasks and retort connected as represented in the figure. The gas is then turned off, the burner inserted into the tubule of the retort and air aspirated for ten minutes through the apparatus. The aspiration is then momentarily discontinued, the burner removed from the retort, the gas turned on and ignited, the burner reinserted and the aspiration continued and regulated with the cock S.

After 1½ to 2 cubic feet of gas have been burned at the rate of 0.35 to 0.5 cubic foot per hour the gas is turned off and the aspiration continued until the apparatus cools down to room temperature. The burner is then withdrawn from the retort and rinsed with distilled water into a 500 cc. beaker. The retort and absorption flasks are disconnected, their contents poured into the beaker and then rinsed several times with small portions of distilled water. The solution is then acidified with hydrochloric acid, concentrated to a bulk of 100 cc., then transferred to a No. 3 beaker and the sulphur precipitated and weighed as barium sulphate by the usual method.

[1] This burner and others used with this apparatus were made by Francis C. Frary, Assistant in Chemistry at this University.

This apparatus has advantages over Drehschmidt's apparatus. It is inexpensive. The absorption apparatus and the retort combustion chamber are inexpensive and common apparatus of every laboratory. There are no non-flexible ground glass joints and no sulphur dioxide is lost by the condensation of moisture during the first part of the determination. The only joints the combustion gases come in contact with are the ones connecting the absorption flasks and in these the glass tubes are in close contact with each other. The burner is so small in diameter and has such a small opening at A and the gas is so well mixed with air at B that a complete consumption and a continuous combustion of the gas are assured. The flame withstands a strong aspiration and the opening A is so small as not to allow an excess of gas. The relative positions of the gas tube and air supply tube are adjustable so that if the openings A were too large for the opening B, D could be brought nearer to E thus insuring a greater supply of air for the combustion, but the flame would then be more easily extinguished and would not withstand such a strong aspiration. The aspiration is regulated to a nicety with the cock S.

The advantage of this method over that of Drehschmidt is the combustion of the gas in the presence of bromine vapors which immediately oxidize the sulphur dioxide in the presence of the moisture of combustion to sulphuric acid which is then converted into potassium sulphate, thus insuring no loss by an incomplete absorption of the sulphur dioxide. The heat of combustion vaporizes the bromine sufficiently rapidly to maintain a constant vapor in the retort. These vapors are sparingly carried through the apparatus so that no sulphur dioxide can possibly escape oxidation.

Hempel[1] has modified Drehschmidt's apparatus by burning the gas in a glass burner within a receiver flask but he uses the same expensive and delicately constructed absorption apparatus.

[1] Hempel: "Gas Analyze," Dritte Auflage, s. 304.

UNIVERSITY OF MINNESOTA.