ON THE ESTIMATION OF SULPHURETTED HYDROGEN IN GAS BY DIRECT WEIGHT.

By A. P. Hallock.

Last winter, while engaged in making some investigations in water gas, I made a few experiments in regard to the sulphur present, in order to see if I could not obtain some idea of in what form it was present. To do this I started to make some determinations of the total amount of sulphur in the gas. Using first the finished gas, which is the gas as it goes to the consumer, after being both enriched and purified, next the gas which had only been enriched but not purified, and lastly what is called the hydrogen. This is the water gas just as it comes from the gasogens, and before the addition of the naphtha vapor. It consists of a mixture of carbonic oxide, hydrogen, marsh gas, carbonic acid and sulphur compounds in varying proportions depending upon the kind of coal used.

With the finished gas I had but little trouble, for the total sulphur could be estimated in the same way as the sulphur in coal gas, by burning it in what is known as the Referee's apparatus. In this apparatus, the gas is burned very slowly at the lower end or mouth of a vertical trumpet-shaped chimney ; the upper end of the chimney having the small opening is connected with a condensing cylinder filled with glass beads in order to present the largest possible amount of surface. On a collar around the burner are placed some pieces of solid carbonate of ammonia, or a dish containing strong ammonic hydrate may be placed under the burner. Ammonia vapor is drawn up through the chimney by the current of air which supports combustion, and unites with the sulphurous acid to form a salt which condenses in the cylinder. After a little while the liquid condensed in the cylinder begins to trickle down the glass beads to the bottom, where it runs out through a small tube into a beaker or flask set underneath. After ten or more feet of gas have been burnt, the gas is turned off, the cylinder and beads washed, and the washings added to the contents of the beaker. After evaporating down to small bulk, the sulphur compounds are oxydized with bromine or chlorine to sulphuric acid, combined with barium, weighed as sulphate of barium, and from this the sulphur calculated. If the oxydation has been complete, the liquid dropping into the beaker will be colorless or very nearly so, but when the sulphur has not been completely oxydized the liquid will have a decidedly yellow color.

With the finished gas the solution was always colorless or very nearly so, but when I attempted to estimate the sulphur in the unpurified gas, either before or after enriching, I always obtained a deeply-colored liquid, and the total amount of sulphur but little more than the amount obtained from the purified gas, and much less than I had reason to suppose existed. Why this should be, I am as yet unable to explain, but as it was actually the case, after a few repeated attempts I gave it up and started to make estimations of the amount of sulphuretted hydrogen, thinking if I could accomplish this it might give me some idea of the sulphur.

I tried making some determinations in an absorption tube for gas analysis, using binoxide of manganese as recommended by Prof. Bunsen, but I soon found that in cases like this where the quantity of sulphur was comparatively small and the amount of gas which could be used was also very small, the liability to error was so great that no satisfactory results could be obtained. I then concluded to try it by absorption in a U-tube in order to employ a large volume of gas. At first liquids were used, such as solution of acetate of lead, sulphate of copper, etc., but none of these worked well. At the suggestion of Mr. Lees, I tried dehydrated sulphate of copper, this absorbed the sulphuretted hydrogen very nicely, forming sulphide of copper and sulphuric acid ; but the difficulty was that in order to completely dehydrate it, it was necessary to pulverize it, and in so doing, it generally became so finely powdered that it would pack in the U-tube and clog it up until the pressure would become sufficient to blow up through it, forming direct gas passages, through which the gas would pass and hardly be acted upon. About this time I was busily engaged with some other work and had no more time to devote to the sulphur determinations and left them for awhile. In the meantime, which was about the first of April, Mr. Lewis T. Wright, of England, published an article in the English *Gas Light Journal* on the estimation of sulphuretted hydrogen and carbonic acid in coal gas. In this article he mentioned the difficulty offered to the passage of the gas in the U-tube by the ordinary dehydrated sulphate of copper, and said he had overcome this by drying it first at about 100° C., then adding a little water to it and stirring it with a glass rod in order to slake it, which makes it granular, then drying it again at 100°C. and then at 155°C. At my first opportunity I prepared some in this manner and found it porous, and it worked very nicely, completely overcoming the previous difficulty.

In order to give a better idea of the method employed, I will describe the apparatus which was used. It consisted first of a drying cylinder containing fused chloride of calcium, next to this was attached a weighed six-inch glass stoppered U tube containing the dehydrated sulphate of copper, a bit of cotton was put on the top of the sulphate in the U-tube to prevent any of it being blown out. To this U-tube was attached another weighed U-tube containing soda-lime and chloride of calcium to catch any carbonic acid and thereby enable me to determine it at the same time, and after this was placed, a meter to measure the quantity of gas passed through the tubes, and from the meter was a tube leading to a small gas burner where the gas was burnt.

The object of the first chloride of calcium was to dry the gas, for any moisture would be caught in the sulphate of copper tube and weighed as sulphuretted hydrogen. In burning the gas in the Referee's apparatus, I had to pass it through a wet meter first, to measure it, and in order to avoid a loss by the sulphur being washed out by the water, the water was replaced by glycerine and gas allowed to pass through it for a long time before using it, but in this case I was able to place the meter after my absorption apparatus, and thus avoid any error from this cause.

The apparatus was first tried on the pipe leading from the hydrogen main; after letting a sufficient quantity blow out to ensure that it was fresh gas, for when the gas was first turned on it failed to produce any stain on acetate of lead paper showing that the sulphur was being removed from the gas by the iron rust in the pipe. Sometime after it began to produce a stain, the apparatus was attached by means of a T-shaped pipe so that the gas could be kept burning all the time and thus have the flow of gas up the iron pipe much more rapid, and thereby have it as little acted upon as possible.

The gas was allowed to pass very slowly through the apparatus, not over one foot in three hours; after one foot had passed, the U-tube was detached and weighed, and found to have increased 0.198 grammes in weight, which would give 315.51 grains of sulphuretted hydrogen in 100 cubic feet, or 0.47 per cent. by volume. After this, four more determinations were made, allowing only one-half foot to pass each time, and although in this case the results had to be multiplied by 200 they agreed very closely, as may be seen by the following table. The column marked A gives the increase in grammes of weight of the U-tube, B gives the number of grains of sulphuretted hydrogen in 100 cubic feet, and C the percentage :

	A.	B.	C.
No. 1............	.1085	344.91	0.51 per cent.
No. 2............	.1100	339.52	0.52 " "
No. 3............	.1110	342.59	0.52 " "
No. 4............	.1105	341.00	0.52 " "

These I consider as agreeing very closely.

A few days after I began the experiments upon the finished gas and was surprised to find that the U-tube gained more in weight than it did when using the unpurified, sometimes showing as much as 100 grains more in 100 cubic feet, while the Referee's apparatus, as used by Dr. Love and also by myself, did not give over 10 grains of total sulphur.

After a few attempts I found the sulphate of copper assumed a peculiar tint different from what I had seen while working on the unpurified gas, and, thinking that the sulphuric acid liberated might be in some way acting upon some of the hydrocarbons, I took some of the sulphate out of the tube and treated it with absolute alcohol and also with ether, and obtained a peculiar oily substance, apparently a mixture of hydrocarbons, but have not as yet been able to obtain a sufficient quantity of it to make any experiments upon it, but hope to be able to do so before long. This was sufficient to prove to me what caused the increase in weight of the U-tube and showed that it was not due to sulphuretted hydrogen.

I have not yet had an opportunity to try this method on unpurified coal gas, but according to Mr. Wright's statements it gives very satisfactory results.

ON THE FRACTIONAL DE-HYDRATION OF AMMONIUM ALUM, AND THE ATOMICITY OF ALUMINUM.

By J. W. Mallet.

In the course of experiments preparatory to my investigation of the atomic weight of aluminum,* I examined the behavior of crystallized ammonium-aluminum alum when dried under various conditions, among others when placed over sulphuric acid in a desiccator.

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* Philosophical Transactions, 1880, Part III.