

tity of molybdc solution is required, as, for example, in the determination of total phosphoric acid in ground bone, bone black, Thomas slag, etc., it may be added without fear that the larger quantity of ammonium nitrate contained in it will in any way interfere with the process. If, however, it is thought desirable, a special molybdc solution containing fifteen grams of added ammonium nitrate to every seventy-five or 100 cc. of the liquid may be prepared for such cases.

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ON THE ESTIMATION OF SULPHUR IN PYRITES.

BY THOMAS S. GLADDING.

Received March 30, 1896.

IN the October (1895) number of this Journal, Dr. Lunge continues his discussion of the estimation of sulphur in pyrites. He presents no new experimental support of his position. He indirectly accuses me of having published a "private" communication without the sanction of the writer, Prof. Richards. In reply I will say that the words of Prof. Richards were published by myself, not only with full permission, but after a careful revision of the same by their author. Had Dr. Lunge addressed a note to Prof. Richards or to myself, he would have been saved from making a most unkind and needless accusation.

While Lunge attempts no further support of his position by chemical experiment, he makes the claim that any occlusion of barium chloride that occurs is compensated for by the solubility of barium sulphate in the acid liquor of precipitation. In his own words "my claim has been that my method by compensation of unavoidable errors, gives *correct results*."

I have investigated this claim of Lunge's, as far as it applies to the occlusion of barium chloride in his method, in the following manner: I have had one of our assistants repeat the work with chemically pure sulphate of ammonia, using two grams in each case, making comparison precipitations with a ten per cent. solution of barium chloride, the first by adding the barium chloride solution drop by drop from a burette, the second by sudden addition from a small beaker in which it had been brought to the boiling point. The results were as follows:

	By slow addition. Gram.	By sudden addition Gram.
Sulphur	0.4840	0.4880
Sulphur	0.4820	0.4873

These experiments show an occlusion of about 0.040 gram barium chloride and a corresponding error of 0.50 to 0.55 per cent. in the estimation of the sulphur present.

I repeated personally the second series of precipitations obtaining:

	Barium sulphate obtained. Grams.	Sulphur. Gram.
1	3.5480	0.4878
2	3.5430	0.4871

These experiments show an error of about 0.55 per cent. in the estimation of sulphur present. The greatest care was taken in washing these precipitates. Three washings by decantation, using seventy-five cc. of hot water each time, were followed by five washings on the filter paper.

I now fused these precipitates with chemically pure sodium carbonate, dissolved the flux in water, filtered from barium carbonate, acidified the filtrate with nitric acid, added silver nitrate and from the silver chloride obtained, calculated the equivalent of barium chloride. I obtained in one case 0.0552 gram silver chloride, corresponding to 0.040 gram barium chloride, in the other 0.0500 gram silver chloride, equivalent to 0.0364 gram barium chloride. Deducting these figures, we have:

BaSO ₄	3.5480	3.5430
BaCl ₂	0.0400	0.0364
	<hr/>	<hr/>
Pure BaSO ₄ obtained.....	3.5080	3.5066

The sulphur obtained from these corrected results are 0.4823 gram and 0.4821 gram. These corrected results agree very closely with those obtained by our assistant when using the dropping method. I now fused one of the precipitates obtained by the dropping method with sodium carbonate in the same way and obtained 0.0027 gram silver chloride, equivalent to 0.002 gram barium chloride. This shows that the occlusion of barium chloride is practically avoided by adding the barium chloride drop by drop, as 0.002 gram barium chloride would cause an error of only 0.026 per cent. in sulphur.

I now took up the question of the solubility of barium sulphate in the acid liquor of precipitation, imitating the conditions under which the actual analysis of pyrites is made. To 400 cc. water were added fifteen cc. strong ammonia. Hydrochloric acid was now added to acidify. To two such preparations I added 0.020 gram and 0.040 gram chemically pure ammonium sulphate, respectively. The solutions were brought to the boiling point and ten cc. barium chloride added. The liquid was boiled till clear and allowed to settle. I obtained :

	Barium sulphate. Gram.	Sulphur obtained. Gram.	Sulphur present. Gram.
1.....	0.034	0.00468	0.00484
2.....	0.069	0.00949	0.00969

The sulphur lost by the solubility of the barium sulphate in the acid solution in the above experiments amounts to 0.016 and 0.020 per cent. respectively.

The above work demonstrates conclusively that the method employed by Lunge of adding the barium chloride all at once causes an error of about five-tenths per cent. in the percentage of sulphur, and that such error is *not* compensated for by the solubility of barium sulphate in the liquor of precipitation, as such solubility is very slight, causing an error of less than 0.03 per cent.

In his first reply (March, 1895), Dr. Lunge objected to my method of estimating the 0.20 to 0.40 per cent. of sulphur, (that may be left in the ferric hydroxide), by the simple process of dissolving the latter in dilute hot hydrochloric acid, adding ten cc. of barium sulphate solution and letting stand over night. This he declared was wrong, owing to the solubility of barium sulphate in such solution. I showed by a few simple and rigid experiments that I was entirely right. These experiments were very easily capable of repetition. But Lunge did not choose to repeat them. The above experiments showing that the sudden addition of barium chloride will cause an error of several tenths of a per cent. are likewise very easily repeated and the separation of the occluded barium chloride as described, is so positive as to the error caused by Lunge's method of procedure, that I hope for a further answer from Dr. Lunge. On account

of the commercial importance of an accurate estimation of sulphur in pyrites, I have no doubt that a prompt repetition of my work as described above will soon be published by some other analyst, and the main point at issue, namely, the necessity of a very slow addition of the barium chloride solution, be settled.

FURTHER NOTES UPON THE FATS CONTAINED IN THE TUBERCULOSIS BACILLI.

BY E. A. DE SCHWEINITZ AND MARION DORSET.

Received March 28, 1896.

IN the *Journal of the American Chemical Society*, for August, 1895, we published an article upon the composition of the tuberculosis and glanders bacilli, and noted the probable composition of the fats which are present in these germs in considerable proportion. The amount of crude fat in the tuberculosis bacilli is very large, having been found by us to be in round numbers thirty-seven per cent. of the weight of the dried germs. In the article referred to the amount of fat at our disposal was very small, and we could at that time determine only palmitic acid, and a high melting acid, which we stated appeared to be arachidic so far as the quantity at hand could be utilized. Recently we have made some further study upon these fats, and the results so far obtained seem to be of sufficient interest to warrant publication as a continuation of our previous work.

The quantity of crude fat available, which had been extracted from the germs was about three and five-tenths grams, and this was examined in the following way: It was first saponified in a closed flask with sodium hydroxide, in accordance with the method prescribed, for the determination of fats by the American Association of Official Agricultural Chemists, as this method seemed to give the most satisfactory results. The saponification yielded a hard soap which was difficultly soluble in water. The dissolved soap was acidified with sulphuric acid and submitted to distillation until 100 cc. of the distillate had been obtained, again in accordance with the usually prescribed method. The distillate had a pungent odor, something like that of sweet almonds, and when titrated with tenth normal hydroxide