

STABILITY OF BARIUM SULPHATE AT
HIGH TEMPERATURES

E. G. MAHIN and BRO. KILIAN, C. S. C., University of Notre Dame

Barium sulphate is almost universally utilized as a precipitate in both qualitative and quantitative analysis of barium salts and of sulphates. In qualitative testing the small solubility of the precipitate is the only important property, since simple recognition is sufficient for the purpose. But in quantitative determinations it becomes necessary to heat the barium sulphate in order to burn filter paper and possible traces of other organic matter, as well as to volatilize moisture and traces of volatile salts.

There has been a widespread belief among analytical chemists that it is necessary to use caution in this heating, on account of danger of decomposition of barium sulphate into barium oxide and sulphur trioxide:



This opinion seems logical, on account of the known decomposition, in a similar manner, of other sulphates, even of the alkaline earth metals. It has received support also from some published reports of investigations, although other researches indicate that only a negligible decomposition occurs, at any temperature that might be attained by use of ordinary laboratory burners. At all events, the caution has been repeated in a good many text books, including those by the senior author of this paper. In the last case this author has felt that as long as there is any doubt about the matter, the only safe course was to use care in the heating, employing a lower temperature for a somewhat longer time.

While it is probably true that many of the published statements and directions are little more than repetitions or expressions of opinion, the following two references to original papers will indicate opposite viewpoints, as a result of experimental work.

Hofman and Warjiekow* plotted decomposition curves for a number of metallic sulphates over a considerable range of temperatures. They showed that barium sulphate is decomposed at ignition temperatures, evolving a mixture of sulphur trioxide and dioxide. This would indicate the necessity for ignition at a temperature only high enough to complete the oxidation of carbon from the filter paper.

Mosttowitsch† investigated the behaviour of pure and impure barium sulphate and found that the pure salt does not appreciably decompose until 1500° is reached, this being only 80° below the melting point. He found that both silica and ferric oxide cause decomposition at lower temperatures, but that even with silica present the precipitate decomposed only at 1000° or above.

From the standpoint of quantitative analysis the question is important only as it concerns decomposition at temperatures obtainable by use of laboratory burners, which may include any of the various modifications of the Bunsen burner, the blast lamp or the Meker burner. In order to obtain some first hand information on the subject, the present investigation was undertaken.

*Bull. Am. Inst. Mining Eng., No. 69 (1907).

†Metallurgie, 6, 450 (1909).

Barium sulphate was prepared by precipitation from a solution of a high-grade barium chloride, sulphuric acid being used as the precipitant. The usual precautions were taken in precipitation, the barium sulphate being washed free from acid and then dried for five hours at 100°. Sufficient sulphate was prepared for several experiments, avoiding the necessity for burning the paper, since most of the precipitate could be removed without difficulty.

First method of procedure. Samples of about 0.2 gm of sample were heated to dull redness in a platinum crucible until the weight was constant. Small losses occurred during this preliminary heating, due to volatilization of traces of moisture, acid or volatile salts. The crucible was then heated over a Fisher burner (a patented modification of the original Meker burner) for successive periods of two hours. The temperature was probably 900-1000°.

Second method of procedure. This was identical with the first method, except that the barium sulphate sample was first moistened with sulphuric acid and then heated at dull redness to constant weight. This treatment would serve to convert any traces of occluded barium chloride into sulphate, and this to avoid slow volatilization or decomposition of the former during the subsequent heating at higher temperatures.

The results may be summarized as follows:

FIRST METHOD

Exp. No.	Sample weight gm.	Loss at dull redness	FURTHER LOSS OVER FISHER BURNER FOR ADDITIONAL 2-HOUR PERIODS		
			1	2	3
1	0.2204	0.0042	0.0003	0.0001	0.0000
2	0.2108	0.0038	0.0004	0.0000
3	0.2094	0.0039	0.0004	0.0001	0.0000

SECOND METHOD

Exp. No.	Sample weight gm.	Loss at dull redness	FURTHER LOSS OVER FISHER BURNER FOR ADDITIONAL 2-HOUR PERIODS		
			1	2	3
4	0.2235	0.0055	0.0000	0.0003	0.0000
5	0.2408	0.0048	0.0000	0.0002	0.0000
6	0.2154	0.0045	0.0001	0.0001	0.0000

Conclusions. So far as the results of this work are concerned, it may safely be concluded that it is unnecessary to keep the ignition temperature as low as "dull redness," and that, on the contrary, the small first losses on heating to higher temperatures indicate the desirability of volatilizing traces of other salts by such heating. When precipitation is carried out carefully, occlusion of barium chloride is not a serious source of error.