

AN EVOLUTION METHOD FOR THE DETERMINATION OF SULFUR IN SULFIDES AND SULFATES.

BY FRANK C. MATHERS.

The object of this research was to devise a rapid method for the estimation of sulfur in sulfates and sulfides, especially in the presence of such elements as molybdenum, which interfere with the precipitation of barium sulfate. The idea was to heat the material with some metal which would reduce all of the oxidized sulfur and then combine with it to form a sulfide. The rest of the method would coincide with the ordinary volumetric evolution method for sulfur in iron and steel.

Powdered potassium sulfate, containing 18.39 per cent. of sulfur, was used in these experiments. In each experiment, 0.2 gm. of the potassium sulfate was thoroughly mixed with the reducing metal and was placed in a crucible which was heated. Experiments with zinc dust as a reducing agent showed that it was impossible to reduce all of the sulfate at low temperatures and that at higher temperatures some sulfur was lost by volatilization along with the zinc.

Gms. of Zn Used.	Temperature.	Per cent. of S Evolved.	Per cent. of S in the Residue.
3.0	Bunsen burner, 5 minutes.	17.05	0.64
3.0	Blast, 5 minutes.	17.20	0.11

The use of a mixture of zinc and aluminum gave still poorer results. The addition of bases such as calcium oxide, calcium carbonate, sodium carbonate, or magnesium oxide were without beneficial effects. The use of some charcoal did not help.

Magnesium turnings gave the best results. To protect the porcelain crucibles, they were partly filled with 1.5 gms. of magnesium oxide and a cavity was made in this with the end of a test tube. The charge was placed in this cavity and was covered with 0.5 gm. of magnesium oxide. The porcelain crucibles, with well fitting lids, were heated for 5 minutes with a

Bunsen flame. A number of good results were obtained. Small amounts of sulfur remain in the residue in the evolution flask. The amount is apparently not constant and varies from 0.3 to 0.5 per cent.

These preliminary experiments have so far been just successful enough to encourage hope and call forth more work.