

when 100 cc. are taken for the determination the error is  $\pm 1$  part per million, so that the working errors may only be neglected when at least 200 cc. of solution are taken for the determination.

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## A RAPID METHOD FOR DETERMINING SULPHUR IN COAL AND COKE.

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THE necessity for a rapid method of determining sulphur in fuels, led to the investigation of the action of sodium peroxide on coal. This reaction was suggested by Professor S. W. Parr's paper on his calorimeter as given in this Journal, **22**, 646.

It was found that coal and coke, ignited with a liberal excess of the peroxide, left almost no residue insoluble in a faintly acid solution, and it became a question only of a suitable apparatus. Apparatus was required that would be cheap, in which the charge could be easily ignited, and out of which the melt could be quickly dissolved. For coke, a covered nickel crucible of about 30 cc. capacity answered every purpose; but coal had to be confined in a bomb, as it always burned with explosive violence.

### ANALYSIS OF COKE.

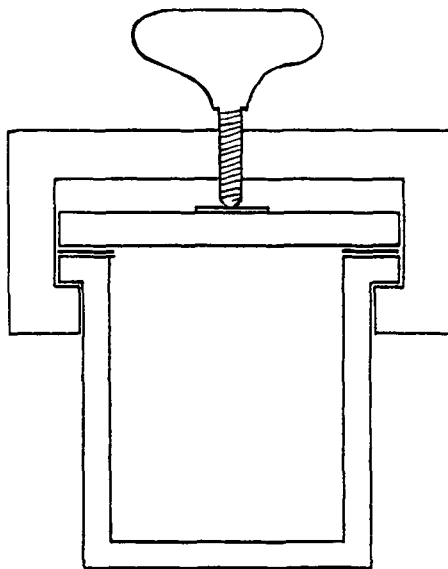
700 milligrams of finely powdered coke are intimately mixed with 13 grams of sodium peroxide, in a nickel crucible of about 30 cc. capacity. The peroxide must be finely powdered and dry, and should be weighed rapidly and brushed off the watch-glass with a glass brush. The crucible is covered and a 3-inch fuse is inserted under the edge of the cover and extended well into the mixture. The crucible is then supported on a triangle and placed in about  $\frac{3}{4}$  inch of water; the fuse is ignited, and in three or four minutes, when the mass has cooled sufficiently, the crucible and cover are placed in a small beaker and 30 cc. water added. The mass dissolves completely in about two minutes. After rinsing off the crucible and cover, the solution is made just acid with hydrochloric acid, and filtered through a small filter. The filtrate is boiled and precipitated with barium chloride in the usual way.

The fuses are made by nitrating cotton wicking with a mixture o. 1 part fuming nitric acid (sp. gr. 1.50) and 2 parts concentrated

sulphuric acid (sp. gr. 1.84) for twelve hours, at 15° C. The nitrated cotton is then washed in running water for twelve hours, to remove every trace of acid, and dried at laboratory temperature. When dry, it is treated with a cold, nearly saturated solution of potassium nitrate for an hour; then the excess of solution is pressed out, dried as before, and finally cut into lengths of 3 inches ready for use.

## ANALYSIS OF COAL.

The apparatus consists of a small, cylindrical, steel bomb, of the following dimensions: 1¼ inch internal diameter, 1½ inch internal depth, sides and bottom ⅛ inch thick, and a ⅛ inch flange



Sketch of bomb—full size.

around the top. A cover plate  $\frac{3}{16}$  inch thick and 1¾ inch in diameter, is clamped on by means of a small clamp, fitting under the bomb flange and pressing down the cover with one central screw. The bottom is hemispherical inside. The cover is insulated from the bomb by a thin mica gasket, and from the screw of the clamp by a piece of ordinary red fiber.

700 milligrams of finely powdered coal are mixed with 13 grams of sodium peroxide in the bomb, and compressed by means of a small press or vice. A No. 36 iron wire, 4 inches long, is now

inserted, one end under the mica gasket and touching the bomb, the other end above the gasket and in contact with the cover, the wire being looped so as to touch the mixture in the bomb. A current of 4 amperes is used to fuse the wire, one terminal in contact with the bomb, the other applied to the cover. After a minute or two, cool the bomb in a little water, unscrew the clamp and dissolve the mass, treating exactly as in the coke analysis.

This method compares very well with Eschka's method and requires much less time and cheaper apparatus while there is less liability of contamination with impure chemicals and exposure to gas flames. The peroxide used seldom contained any sulphates.

The following are some of the results obtained:

	Per cent. sulphur by Eschka's method.	Per cent. sulphur by peroxide method.
Coal and coke.		
River View coal . . . . .	1.69	1.64
" " " . . . . .	1.59	1.57
Hocking Valley, pea . . . . .	1.95	1.96
" " slack . . . . .	1.690	1.694
Coke (mixture) . . . . .	1.26	1.21
Pittsburg coke . . . . .	0.89	0.88
Connellsville coke . . . . .	1.67	1.73
Coke mixture . . . . .	2.027	2.031

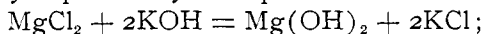
DETROIT, October 18, 1902.

## A STUDY OF MAGNESIUM AND MANGANOUS HYDROXIDES AND OF BARIUM SULPHATE WITH RESPECT TO THE PHENOMENA OF ADHESION AND OF SOLUTION.

BY HARRISON EASTMAN PATTEN.

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THE work on aluminum,<sup>1</sup> iron,<sup>2</sup> zinc,<sup>3</sup> and chromium<sup>4</sup> has shown that the action of their hydroxides in carrying down other substances into precipitation is chemical, and under certain conditions not proportional to the mass of precipitate. I have examined the action usually expressed by the equation



also

<sup>1</sup> "A Thermochemical Analysis of the Reaction between Alum and Potassium Hydroxide," *Am. Chem. J.*, 8, 23.

<sup>2</sup> "A Study of Ferric Hydroxide in Precipitation," *Ibid.*, 19, 512.

<sup>3</sup> Unpublished.

<sup>4</sup> "Chromic Hydroxide in Precipitation," *Ibid.*, 18, 608; also "Recherches sur le sulfate chromique, ses transformations et les acides complexes qui en derivent," *Ann. chim. phys.*, 7, 4 (1895).