

CLV.—*The Determination of Volatile Matter in Coke.*

By FREDERICK JOHN EATON and STUART PEXTON.

DURING certain investigations upon the reactivity of coke it was found necessary to determine accurately the content of volatile matter. The method of the American Society of Testing Materials (1924, D, 37, 1018) and a method adapted from the Bone and Silver method for coals (J., 1921, 119, 1145) were examined. In the former method the coke, in a platinum crucible, is heated in an electric furnace at 950° (Fieldner, U.S. Bureau of Mines, Tech. Paper No. 76), but no special precautions are taken to prevent losses in weight due to oxidation, although Fieldner (*ibid.*, p. 20) states that when the same method is used for coal, where oxidation is minimised by the presence of a reducing atmosphere in the crucible, oxidation losses amount to less than 0.3%. In the adapted Bone and Silver method, coke is heated in a silica tube filled with nitrogen until evolution of gas ceases; a disadvantage of this method is that, owing to the low conductivity of silica, the test is prolonged and the final temperature of the coke uncertain.

The following method has been found to combine the rapidity of the first method with the trustworthiness of the second.

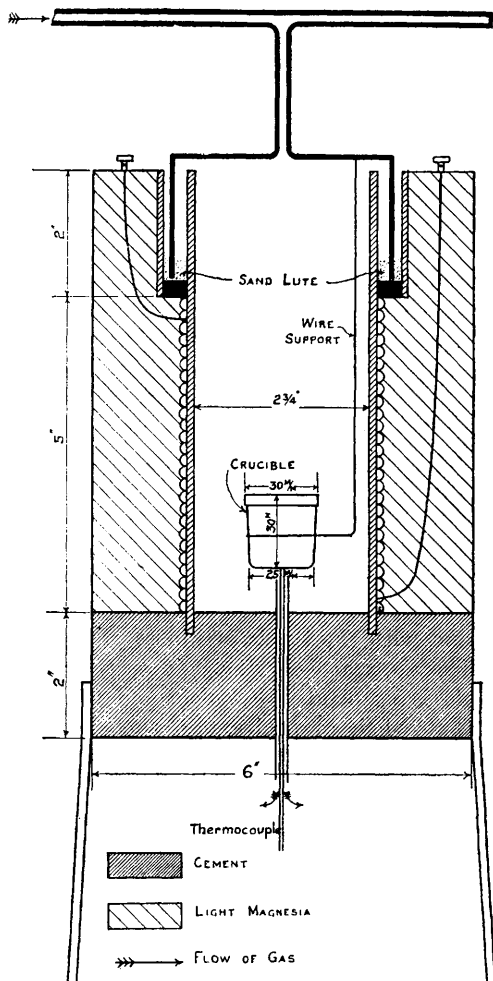
*Apparatus.*—The apparatus is shown in section in Fig. 1. The platinum crucible is suspended by nichrome wire from a monel metal cap, so that when in position in the electric furnace the thermocouple junction touches the base of the crucible. Nitrogen is fed at a steady rate (200 c.c. per minute) into the furnace through a cap sealed by the sand lute and escapes through the silica tube which carries the thermocouple.

*Procedure.*—1.1 G. of the sample, ground to pass a 70-mesh sieve (I.M.M. Standard), are weighed into a small test-tube and then dried for 1 hour at 120—130°. After cooling in a desiccator, the coke is weighed in a tared platinum crucible protected from moisture by a porcelain lid, which is used instead of the platinum lid because the weight of the latter, when in the furnace, is affected by particles of sand falling from the lute.

Air is displaced from the furnace tube by a current of nitrogen, and the platinum crucible, closed with a tightly fitting platinum

lid, is introduced into the furnace. The heating current is controlled by an external resistance so that the initial temperature of  $950^{\circ} \pm 10^{\circ}$  is regained after one minute. After 2 minutes the temperature should be stationary at  $950^{\circ} \pm 2^{\circ}$ . The temperature

FIG. 1.



is recorded every 10 seconds for the remaining 5 minutes. At the end of 7 minutes the crucible is withdrawn and placed on a slab of brass (to ensure sudden cooling) in a desiccator. When cold the crucible is reweighed with the porcelain lid. The loss in weight

expressed as a percentage by weight of the original coke represents the volatile matter.

If the determination is carried out as directed, the variations in the percentage obtained should not exceed  $\pm 0.05$ . Particular attention must be paid to the fineness of the sample, prevention of absorption of moisture during weighing, accurate temperature control, and the brightness of the crucible.

The new method possesses the following advantages: (1) Errors due to oxidation are avoided by heating in a current of nitrogen, and it is therefore possible to obtain comparable results with cokes of different reactivities towards oxygen. (2) The temperature of heating is accurately known, since the thermocouple junction is placed close to the base of the platinum crucible. (3) The method is rapid, since the coke is heated in a platinum crucible which has a high heat transmission and low heat capacity, and the coke rapidly attains a temperature of  $950^{\circ}$ .

The following table gives examples of the results obtained for the percentage of volatile matter.

Sample.	Adapted Bone and Silver method :		American method.	Authors' method.
	10 minutes' heating.	60 minutes' heating.		
A	1.37, 1.18, 1.30, 1.67	1.86, 1.71	3.02, 2.92, 2.76, 2.66, 2.58	1.52, 1.55, 1.58, 1.59
B*	1.86, 1.81	2.5, 3.12	7.84, 7.34, 7.52	2.56, 2.5, 2.61
C	1.74, 1.8	2.2, 2.36	3.06, 3.04	1.98, 1.92, 1.92, 1.92
D	0.92, 0.94	1.63, 1.73	4.4, 4.42	1.78, 1.76
E	0.75	0.94, 1.08	2.8, 2.76	1.03, 1.02, 1.06, 1.03

\* This sample was a specially reactive coke.

THE GAS LIGHT AND COKE Co.,  
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