Determination of the True Specific Gravity of Coke¹

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Some of the methods in use for the determination of the true specific gravity of coke vary in important respects. This paper is presented to show the urgent need of an official method for this determination.

Data are given to show the effect of the degree of pulverization of the sample, the nature of the liquid used, the time of boiling and standing, and the effect of partial vacuum.

When 60-mesh coke samples were pulverized to pass a 200-mesh sieve, the true specific gravity was found to increase by as much as 12.7 per cent. The greatest increase was noted in the case of cohes which had been made below 800° C.

When the effect caused by a change of liquids was examined, benzene was found to give much the lowest results. Figures obtained

by using benzene were as much as 8.1 per cent lower than those obtained by using water.

The true specific gravity of coke is usually determined for the purpose of calculating the porosity of the sample in question. The discrepancies mentioned above, when expressed in terms of porosity, amount to as much as 30 to 50 per cent of the total variation in porosity which will be found in different blast-furnace cohes.

Variations of the time of boiling and standing in the wetting liquid, and the use of partial vacuum during boiling, also produced discrepancies, although these were considerably smaller than those mentioned above.

THE TRUE specific gravity of coke is usually determined for the purpose of calculating its porosity. It is also of some value in judging the temperature at which the coke has been made. A number of methods for this determination have been published, but it appears that the problem is more complex than has been generally recognized.

According to methods published or in use, the determination may be made with pycnometer, volumeter, or by weighing coke pieces suspended from the beam of a balance. Some of these types of apparatus involve experimental errors and difficulties. The size of the particles of sample used in the different methods varies from a fine powder to full-sized coke pieces, and the exact fineness is not always specified. For the wetting liquid, water and organic liquids such as benzene, toluene, alcohol, and carbon bisulfide are variously used. Finally, boiling for long periods and the use of a partial vacuum may, or may not, be specified.

It was recently necessary in this laboratory to make a large number of determinations of the true specific gravity of coke with somewhat greater accuracy than that required for usual testing purposes. It was soon found that serious discrepancies were obtained when the kind of liquid was changed, or the degree of fineness of the sample was varied.

A survey of the scattered information available on the determination of the true specific gravity of coke soon indicated the desirability of making a brief compilation and comparison of a number of the methods that have been published or are in extended use. Unfortunately, space is not available for this résumé, and since methods in steel and coke plant use are for the most part unpublished, references cannot be given. It will be sufficient to state that such methods exist in considerable variety, and are in some cases obviously inaccurate.

EFFECT OF FINENESS OF SAMPLE

If the cellular nature of coke and its mode of formation be considered, it will be realized that a larger or smaller percentage of the total cell space may exist as small isolated cells. Unfortunately, this feature cannot be directly shown, as no comprehensive microscopic study of coke cell structure is available. Such cell spaces will not be penetrated by liquid in a true specific gravity determination, unless the cell walls have been broken by sufficiently fine grinding.

It is for this reason, presumably, that many methods require the use of finely powdered coke, the degree of fineness when specified ranging from 40 to 80 mesh. It has been found in this laboratory that a very distinct increase in the true specific gravity is obtained when the pulverization is carried to a still greater extent. No comparisons have been made with methods involving the use of large coke pieces, as this practice does not seem defensible from any standpoint.

EXPERIMENTAL DATA ON INFLUENCE OF FINENESS—Table I gives data concerning the effect of fineness of pulverization on the true specific gravity figure. The sample used consisted of a mixture of the middle thirds (the "cauliflower" and center of oven ends being rejected) of two full-sized pieces of by-product coke made from 100 per cent high volatile West Virginia coal.

The material was first crushed to 20-mesh size by means of steel rolls. 500 g. were then placed in an Abbé pebble mill—a porcelain jar 7 in. in inside diameter, and 7 in. deep, half filled with quartz pebbles. The pebble mill made 54 r. p. m. 100 g. portions were removed after the following intervals: 1 hr. 15 min., 2 hrs. 15 min., 5 hrs. 15 min. The remaining 200 g. were made into a thin paste with distilled water and were pebble-milled for another 24 hrs. Another sample was prepared by repeatedly passing a part of the original 20-mesh sample through steel rolls, and sieving. This process was continued until all had passed through a 60-mesh sieve.

A portion of each sample was dry-sieved in order to obtain an approximate idea of its fineness. Ash determinations were also made in duplicate, to find out whether or not mineral impurity was introduced during the prolonged grinding.

The determinations in Table I, and all others reported in this paper, were made in the following manner, except where otherwise noted.

METHOD USED—A 10-g. sample of coke which had been dried at 105° C. was introduced into a 50-cc. volumetric flask provided with a ground-glass stopper. The flask was filled about one-third full with C. P. benzene, and the mixture gently boiled on an electric hotplate for one-half hour. The flask was cooled, filled approximately to the mark with benzene, and placed for at least 15 min. in a water bath kept at exactly 25° C. The meniscus was adjusted to the mark at that temperature, and the flask was stoppered, wiped dry, and weighed.

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The calculations were made as follows:

Let S = weight of coke sample

B = weight of flask filled with benzene

D = weight of flask, coke, and benzene (after boiling).

Then

$$\text{True specific gravity} = \frac{S}{B + S - D} \times \text{sp. gr. benzene}$$

All results given in this paper have been calculated to true specific gravity at 25° C., referred to water at 15.5° (15.5° C. is the usual coke plant standard and approximates 60° F.)

TABLE I

SAMPLE NO.	METHOD OF PREPARATION	SIEVE TEST PER CENT	ASH CONTENT PER CENT	TRUE SPECIFIC GRAVITY
A-1	Steel-rolled	Through 60 mesh On 100 mesh 38.0 On 200 mesh 34.8 Through 200 mesh 27.2	7.01	1.699 1.698 Av. 1.699 ¹
A-2	Pebble-milled 1 hr. 15 min.	Through 60 mesh On 100 mesh 25.2 On 200 mesh 46.8 Through 200 mesh 28.0	7.06	1.678 1.671 Av. 1.675
A-3	Pebble-milled 2 hrs. 15 min.	Through 60 mesh On 100 mesh 1.2 On 200 mesh 34.8 Through 200 mesh 64.0	7.10	1.710 1.700 Av. 1.705
A-4	Pebble-milled 5 hrs. 15 min.	Through 200 mesh 100.0	7.09	1.739 1.731 1.734 Av. 1.735
A-5	Pebble-milled 5 hrs. 15 min. dry, plus 24 hrs. wet	Through 200 mesh 100.0	9.02	1.753 1.753 Av. 1.753

¹ A possible explanation of the fact that this value is higher than the succeeding one is that metallic iron may have been introduced during repeated passage through the steel rolls.

NOTE: Portions of the same sample of benzene were used throughout, and under identical conditions, so that the results above are strictly comparable.

COMMENTS ON TABLE I: (1) Since the wet-pebble milling of coke for 24 hrs. resulted in a 2 per cent increase in ash content, this treatment would not be permissible. The rise in the true specific gravity figure after wet grinding is 0.018. The calculated effect of the introduction of 1.93 per cent quartz happens to be exactly 0.018. Thus, in this instance, there appears to be no real gain in the true specific gravity when 200-mesh coke is pulverized still finer.

(2) Dry-pebble milling of a large sample for as long as 5 hrs. did not introduce more than a trace of mineral impurity.

(3) The maximum and minimum acceptable figures obtained above show a variation of 0.060, which amounts to 3.4 per cent.

From an examination of the results of other tests made at the same time, it was soon found that the effect of fineness of pulverization was still greater in the case of cokes made at comparatively low temperatures (below 800° C.). To secure information on this point, several series of cokes were made in the laboratory at various temperatures. In making these cokes a standard practice was followed, which proved to yield coke most nearly comparable to that obtained in full-scale operation.

The cokes obtained were crushed to 20-mesh size and divided into two portions. One portion was crushed in a diamond steel mortar to just pass 60 mesh. The other half

TABLE II

SAMPLE NO.	DESCRIPTION	Temp. of Coking ° C.	True Sp. Gr. of Coke 60 Mesh	True Sp. Gr. 200 Mesh	Increase of Sp. Gr. Due to Pulverization
B	Laboratory coke made from 100 per cent Pittsburgh Seam, high volatile coal	700 750 850 950	1.558 1.633 1.742 ...	1.702 1.798 1.817 1.823	0.144 0.165 0.075 ...
C	Same coke as B, except that when made the heating was continued for 2 hrs. longer	700 750 850 950	1.639 1.693 1.815 1.842	1.723 1.800 1.863 1.886	0.084 0.107 0.048 0.044
D	Laboratory coke made from a mixture of 80 per cent Pittsburgh Seam coal and 20 per cent low volatile material	750 850 950	1.547 1.677 1.840	1.743 1.735 1.846	0.196 0.058 0.006

was pulverized for about 3 hrs. in an Abbé pebble mill, until it readily passed through a 200-mesh screen.

Table II gives the results of tests on these cokes. The determinations were carried out in the same way as described for Table I.

COMMENTS ON TABLE II: (1) The effect of fineness of pulverization is greatest in the case of cokes made in the laboratory at 700° and 750° C., the discrepancies at these temperatures averaging 8.6 per cent, and reaching a maximum of 12.7 per cent.

(2) When calculated to porosity

$$\left(\text{Porosity} = 100 - \frac{100 \times \text{apparent sp. gr.}}{\text{true sp. gr.}} \right)$$

assuming for convenience an apparent specific gravity of 0.900, these discrepancies average 4.5 per cent with a maximum of 6.6 per cent.

(3) Since the actual range of porosity of different blast furnace cokes is only about 15 per cent (from 42 to 57 when determined by a particular method), as small a matter as the degree of fineness of pulverization of a particular sample may make a difference 40 to 50 per cent as great as the variation to be expected between the densest and most porous cokes in use. It is clear that the degree of pulverization must be carefully regulated.

VARIATIONS DUE TO THE LIQUIDS USED

A series of determinations was made, using water, benzene, carbon tetrachloride, and carbon bisulfide. The organic liquids had been obtained from standard chemical supply houses, and were labeled "pure" or "C. P." The specific gravity of these liquids was determined by pycnometer, under the same conditions as were used for the tests with coke. Except for the variation in liquids used, the determinations of Table III were made according to the method immediately preceding Table I.

TABLE III

SAMPLE NO.	Water	Benzene	Carbon Tetrachloride	Carbon Bisulfide
A-5 (See Table I)	1.838 1.865	1.750 1.750	1.851 1.823	1.865 1.887
Average....	1.852	1.750	1.837	1.876

The low figure for true specific gravity obtained by using benzene will at once be noted. While the agreement between individuals of duplicate determinations is not especially good in some of these cases, the low results obtained with benzene are much too large to be ascribed to experimental error. The variation between results obtained by benzene and water in the instance above is 0.102. Several more determinations were made to further compare the results obtained by using benzene and water.

TABLE IV

SAMPLE NO.	DESCRIPTION	Water	Benzene	Lowering of True Specific Gravity Due to Benzene
A-1	(See Table I)	1.870 1.828	1.699 1.698	
	Average.....	1.849 ¹	1.699	0.150
A-3	(See Table I)	1.876 1.869	1.753 1.753	
	Average.....	1.873 ¹	1.753	0.120
E	200-mesh sample "Cauliflower" 1/3 of two pieces of coke made from 100 per cent high vol. W. Va. coal	1.922 1.901	1.809 1.804	
	Average.....	1.912 ¹	1.807	0.105
F	By-product coke from mixture of three Colorado coals	1.965 1.924	1.907 1.934	
	Average.....	1.945	1.920	0.025

¹ These determinations were made by boiling for 2 1/2 hrs. under moderate vacuum.

The largest lowering of specific gravity due to the use of benzene that is recorded in Table IV, is 0.150, or 8.1 per cent.

When calculated to "porosity," as explained in the comments on Table II, this discrepancy will be about 30 per cent as great as the total variation in porosity of the most widely different blast-furnace cokes.