Assessing Coke on the Basis of the Yield of Volatiles


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Abstract—Existing methods of assessing coke in production conditions are analyzed. Significant deficiencies are noted in the gravimetric method of determining the yield of volatiles and in methods of determining the temperature of the coke leaving the coking chamber. Assessment of coke on the basis of a modified method of determining the yield of volatiles (by volume) at 1150 °C is proposed. In the coke shops at PAO Zaporozhkok, samples of industrial coke are assessed by the proposed method. A close correlation is found between the yield of volatiles (by volume) and two important coking characteristics: the final temperature and the coking rate. Determination of the yield of volatiles by volume is currently used in the central laboratory at PAO Zaporozhkok in continually assessing the coke produced and correcting the coking temperature.

Keywords: coke assessment, yield of volatiles, final coking temperature, coking rate

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In practice, the condition of coke is assessed in terms of the final temperature along the axis of the coke cake for the last 15 min before its removal from the chamber [1] or the residual yield of volatiles, as determined in accordance with State Standard GOST 6382–2000 (ISO 562–1998).

According to the operating specifications, the weighted mean temperature along the axis of the coke cake at the end of coking should be 1050 ± 10 °C, in order to ensure satisfactory coke characteristics [2].

As we know, the final temperature along the axis of the coke cake is monitored by measuring the coke temperature at different heights in the coking chamber, by means of Chromel–Alumel thermocouples inserted through the charge holes in three coke chambers of different series.

The measurements are complicated by the need to use metallic sheathes, ceramic beads, and Chromel–Alumel thermocouples. In addition, the measurements are physically laborious. Because most coke batteries are old and the conditions in the lining on the chamber walls vary, it is difficult to establish the true mean temperature for all the coke produced by the battery on the basis measurements only in three chambers of different series.

The appearance of optical pyrometers with built-in memory permits ongoing determination of the coke temperature over the width, height, and length of the cake at discharge from the coking chambers.

However, the installation of dust-free discharge systems has greatly complicated the visual assessment of the coke temperature, on account of the dust-trapping hood.

Therefore, in modern batteries, the coke temperature is mainly monitored by direct measurement, by means of an optical pyrometer, at discharge to a rail car and over the length and width of the charge within the cars. Obviously, the pyrometer readings are distorted by external factors (by day or by night) and will be below the actual coke temperature in the coking chamber. However, such ongoing measurement permits temperature assessment of the coke discharged from a large number of ovens, and comparison of the results gives an idea of the temperature uniformity of the coke cake over the length and height. Measurement of the coke temperature by an optical pyrometer at discharge to a rail car has been successfully employed at PAO Zaporozhkok.

Note that such methods of coke assessment are not always objective or reliable.

An alternative is to assess the coke produced in terms of the yield of volatiles by volume. In Technical Specifications TU U 322-00190443-114–96, the limiting yield of volatiles from metallurgical coke above which the coke quality is regarded as unsatisfactory is 1.2%. This value is somewhat arbitrary and derives from the production data available at the time (1995 and 1996) regarding the correlation between the yield of volatiles from the coke and the coking temperature.
When the standard method from State Standard GOST 6382–2001 (ISO 562:1998) is employed, it is difficult to obtain reliable and reproducible results [3]. The problem here is that the temperature of the coke cake is 1000–1150°C, while the yield of volatiles is determined in laboratory conditions at 900°C. Accordingly, the mass loss from the coke sample at 900°C is slight. In addition, partial combustion of the coke occurs during the determination of the yield of volatiles, because insufficient volatiles are released from the coke to maintain the required gas pressure in the crucible until the end of the experiment. That distorts the results and impairs their reproducibility.

In the standard method of determining the yield of volatiles, either quartz or porcelain crucibles are employed. Practical experience shows, however, that the difference in the results obtained in quartz and porcelain crucibles exceeds the tolerances of the GOST standard. Table 1 presents illustrative experimental data.

Analysis indicates that, in porcelain crucibles, the yield of volatiles in experiments 1, 4, and 5 exceeds the specified limit (1.2%). In other words, the coking temperature employed does not ensure coke of satisfactory quality. This result may be attributed to the free access of atmospheric oxygen to the coke in porcelain crucibles, on account of the loosely fitting lids. In quartz crucibles, that is not the case, and the measurements are consistent with the limit.

State Standard GOST 7303–77 outlined a method of determining the yield of volatiles from anthracite by volume [4]. In this method, an anthracite sample is heated to 900 ± 10°C for 15 min, in the absence of air, and the volume of gases liberated is determined from the quantity of liquid displaced from an aspirator. We now consider the applicability of this method to coke.

To improve the method, we manufacture a new tubular furnace, with a Fechral alloy heating element capable of raising the temperature of the coke sample to 1150 ± 10°C. That should permit objective assessment of the residual yield of volatiles for coke produced at 1000–1150°C and assessment of the coke characteristics.

We have developed a draft DSTU Ukrainian State Standard regarding a method of determining the yield of volatiles. In this method, the coke sample is heated in a vertical tubular electrofurnace, with a regulator maintaining constant temperature 1150 ± 10°C within the working zone (Fig. 1). This temperature is attained in the furnace 3–4 min after the introduction of the test tube with the coke sample.

The apparatus is shown in Figs. 2 and 3. The quartz test tube is attached by means of a system of pipes and valves to an aspirator filled with sodium-chloride solution and to a U-tube manometer. After testing that the system is gas-tight, the furnace is heated to 1150 ± 10°C and the correction for the air volume liberated from the empty test tube on heating is determined. This air volume, measured in terms of the volume of liquid displaced from the aspirator and reduced to normal conditions, is constant for the given test tube. A coke sample (mass 1 ± 0.01 g) is placed in the quartz test tube. The test tube with the coke sample is connected to the instrument and lowered into the furnace, to a fixed distance from the heating surface; valves 7, 9, and 14 must be open in this process (Fig. 2).

On heating the test tube, the overflow of NaCl solution from the aspirator is regulated so that the pressure in the equipment is constant, as confirmed by the manometer. The test is conducted for 15 min, with recording of the temperature in the aspirator at 3-min