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UDC 662.33 Experimental Determination of the Sulfur Content in the Shubarkol Coal

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Abstract: The sulfur content of the Shubarkol coal of the Karaganda coal basin was determined experimentally. The methods for determining the content of total sulfur, inorganic (sulfate) sulfur, inorganic (pyrite) sulfur are given.

Keywords: Shubarkol coal, Pyrite sulfur, Sulfate

Introduction

The combustion of coal is accompanied by evolution of sulfur compounds which corrode equipment as well as pollute the environment [1-4]. The coke sulfur worsens its quality as a metallurgical fuel, since in the blast furnace it goes into cast iron, giving it fragility and lowering the quality of the steel obtained from it.

According to the content of sulfur (S,%), coal is classified into: low sulfur –from 0,5 to 1,5; mid-sulfur –from 1,6 to 2,5; sulphurous from –2.6 to 4.0; high-sulfur coal more than 4,0

Determination of the total sulfur content.
The method is based on burning of the fuel sample with a mixture of magnesium oxide and sodium carbonate (Eshka mixture), by further dissolving the formed sulfates, on precipitating sulfate ions with barium chloride in a hydrochloric acid medium in the form of barium sulphate and on determination its weight.

Chemicals and Instruments

Muffle furnace with heating temperature up to 900 ° C.
The ceramic plate or is made of heat-resistant metal, length and width in accordance with the muffle working space the thick is 5 mm.
Porcelain crucibles in accordance with Standard 9147-59.
Glasses for weighing (boxes) in accordance with Standard 7148-70.
Hydrochloric acid in accordance with Standard 3118-67, diluted 1: 1.

Mixture Eshka according to the Standard 5144-69.
Barium chloride according to the Standard 4108-65, a solution of 100 g / l.
Silver nitate according to the Standard 1277-63, 3% solution, acidified with nitric acid.
Methyl orange (para-dimethylaminoazobenzenesulfonic acid sodium) according to the Standard 10816-64, 0.1% solution.

Materials and Methods

A sample of 0.5 g of coal (with a sulfur content up to 15% in fuel) is placed to a porcelain crucible in which 2 g of Eshka mixture is weighed with high accuracy, then are thoroughly mixed and another 1 g of Eshka mixture is added. The crucible with the fuel sample is placed in a preheated to 850 ± 25 ° C muffle furnace, pushing the plate into the zone of maximum heat for 3 minutes, and calcined at this temperature for 30 minutes.

The crucible with a sample of Shubarkol coal is placed in a cold muffle furnace, which is heated gradually, for at least 1 hour, to 850 ± 25 ° C. The calcination is continued at this temperature for 30-40 minutes.

Further, the mixture in the crucible are cooled, loosened and transferred to a 150 ml beaker, then 100 ml of hot water is added by washing the inner walls of the crucible and is heated to boiling. Provided that unburned coal particles are found that float to the surface, the burning of a new sample is repeated.

The solution above the precipitate is decanted through a filter into a 600 ml beaker.
The precipitate is washed three times with hot water, decanting the settled liquid in the same beaker. The precipitate is then transferred to a filter and washed thoroughly with a hot water jet from the wash-bottle. The total volume of the solution should not be more than 300 ml.

To the solution, is added 2-3 drops of methyl orange indicator and diluted 1: 1 of hydrochloric acid until the color of the indicator changes to pink. The solution is heated to boiling and 10 ml of a barium chloride solution is added in small portions with stirring. The solution with the precipitate of barium sulphate is aged for at least 30 minutes in a boiling water bath or on a sand bath at a temperature close to the boiling point.

The liquid that has settled in the beaker is filtered through a dense ashless filter, the precipitate is transferred to the same filter and washed with hot water until the chlorine ions are completely removed (sample with silver nitrate).
The filter with the precipitate is placed in the pre-calcined to constant weight crucible, slightly compacted, charred, without allowing the filter to ignite, and calcined in a muffle furnace at a temperature of 800 ± 25 ° C for 15-20 minutes. Then the crucible with the contents is cooled in air and weighed.
The content of total sulfur in the analytical sample of coal (S_{tot}) in percent is calculated by the formula:

$$\mathbf{S_{об}^{a}=\frac{(G1-G2)*0.1373*100}{G}},$$

where, G1 is the mass of barium sulfate obtained by analyzing the test coal, g;
G2 is the mass of barium sulfate obtained in the control experiment, which should not exceed 0.005 g, g;
G - weight of coal, g;
0.1373 - coefficient of conversion of barium sulfate to sulfur.
Substituting the obtained values, we have:

$$\mathbf{S_{tot}^{a}=\frac{(0,024-0,005)\cdot 0,1373\cdot 100}{0,5}=0,5}$$

Determination of the Content of Inorganic (Sulfate) Sulfur

The method is based on dissolving the sulfates contained in the fuel in dilute hydrochloric acid, precipitating the sulfate ions with barium chloride in the form of barium sulphate and by weighing of the latter.

Chemicals and instruments

The apparatus and reagents specified at first experiment are used during this analyze, with the exception of the Eshka mixture, and in addition:
acid hydrochloric in accordance with Standard 3118-67, diluted 1: 7 and 1:20;
Ammonia water according to Standard 3760-64, diluted 1: 1;
water bromine, 3% solution.

Analysis Methods

A sample of 1 g coal is placed in a beaker, 100 ml of diluted 1: 7 hydrochloric acid is added, the solution is boiled for 10 minutes and filtered through a uncompacted filter. The precipitate on the filter is washed 5-6 times with a dilute 1:20 hydrochloric acid solution.

1 ml of the bromine water is added to a filtrate (200-250 ml) and is boiled for 5 minutes, then is added dilute ammonia until precipitation formation. The solution is filtered through a filter. The precipitate on the filter is washed with hot water until disappearing of chloride ion (sample with silver nitrate). The filtrate is evaporated to a volume of 200-250 ml, 2-3 drops of a methyl orange indicator are added, then diluted 1: 1 with hydrochloric acid until the color of the indicator changes to pink, and then continues as indicated in 2.2.1.

Processing of Results

The content of sulfate sulfur in the analytical sample of coal (Sca) in percent is calculated by the formula:

$$S_c^a = \frac{(G1-G2)*0.1373*100}{G},$$

where, G1 - mass of barium sulphate, obtained in the analysis of the test fuel, g;
G2 - is the mass of barium sulfate obtained in the control experiment, which should not exceed 0.005 g, g;
G - weight of coal, g;
0.1373 - coefficient of conversion of barium sulfate to sulfur.
The content of sulfate sulfur in the analytical sample of Shubarkol coal (Sca) is:

$$S_c^a = \frac{0,02-0,005\cdot 0,1373\cdot 100}{1} = 0,2$$

Determination of the Content of Inorganic (Pyrite) Sulfur

The method is based on the oxidation of pyrite sulfur with nitric acid to soluble sulfates and its determination by titrimetry on the pyrite iron.

Chemicals and Instruments

The apparatus and reagents specified at first experiment are used during this analyze, with the exception of the Eshka mixture, and in addition:
shaker;
acid hydrochloric in accordance with standard 3118-67, diluted 1: 7;
acid nitric according to the standard 4461-67, solution with a density of 1.2g / cm^3;
hydrogen peroxide (perhydrol) according to standard 10929-64;
ammonia aqueous according to standard 3760-64;
potassium iodide in accordance with standard 4232-65, a solution of 200g / l;
sodium sulfate (sodium thiosulfate) according to standard 4215-66, 0.05 n solution;
starch, soluble according to standard 10163-62, freshly prepared solution of 10g / l.

Analysis Methods

A sample of Shubarkol coal with a mass of 1 g is placed in a beaker with a capacity of 300-500 ml, 100 ml of dilute hydrochloric acid are poured, covered with a watch glass, the contents are boiled for 10 minutes and filtered through a dense filter. The filter precipitate is washed 4-5 times with hot water. The filter with the precipitate is placed in a beaker in which the sample is treated with hydrochloric acid, 80 ml of nitric acid are added, mixed thoroughly and left for 24 hours.

For accelerated oxidation, the precipitate is placed in a flask with a capacity of 200-300 ml then 80 ml of nitric acid is added, the flask is closed with a well-fitted stopper and agitated on the shaker for 2 hours.

The insoluble residue is filtered onto a medium-density filter and is washe 7-8 times with hot water. The filtrate and washings are collected in a single beaker. If the filtrate is colored in a brown color, 3 ml of hydrogen peroxide is added and boiled to discoloration and termination of the release of bubbles. Further, to a boiling light filtrate, ammonia is poured into a stable weak odor and another 5 ml into excess. The precipitate of the iron oxide hydrate is filtered onto a medium-density filter and washed with hot water with a few drops of ammonia.

The precipitate on the filter is dissolved by hot dilute 1: 7 with hydrochloric acid and washed 5-6 times with hot water. The filtrate and washings are collected in a conical flask. The solution in the flask is neutralized with ammonia, adding it dropwise until a precipitate appears. Then 10 ml of diluted 1: 7 hydrochloric acid, 10 ml of potassium iodide solution are added to the solution, cover the flask with a watch glass and put in a dark place for 5 minutes. The formed out iodine is titrated with sodium thiosulfate before the color transition into a straw-yellow color, the starch solution is added and the titration is finished after the discoloration of the solution.

Processing of results.
The content of pyrite sulfur in the analytical coal sample (Ska) in percent is calculated by the formula:

$$\mathbf{S_к^{\,a} = \frac{n*(V-V1)*0.064*100}{G}},$$

where, n is the normality of the sodium thiosulfate solution;
V is the volume of sodium thiosulfate solution used for titration, ml;
V1 is the volume of sodium thiosulfate solution used for titration in the control experiment, ml;
G - fuel sample, g;
0.064 - the amount of sulfur corresponding to 1 ml of a 1N solution of sodium thiosulfate, g.

The content of pyrite sulfur in the analytical sample of Shubarkol coal is:

$$\mathbf{S_к^{\,a} = \frac{0{,}004\cdot(5-2{,}5)\cdot 0{,}064\cdot 100}{1} = 0{,}06}$$

The content of organic sulfur in the analytical sample of coal (Sora) in percent is calculated by the formula:
$S_{ор}^{\,a} = S_{об}^{\,a} - (S_с^{\,a} + S_к^{\,a})$,
where, Soba - the total sulfur content in the analytical fuel sample,%;
Sca - content of inorganic (sulfate) sulfur in the analytical sample of coal,%;
Ska - content of inorganic (pyrite) sulfur in the analytical sample of coal,%;
$\mathbf{S_{ор}^{\,a} = S_{об}^{\,a} - (S_с^{\,a} + S_к^{\,a})}$
The content of organic sulfur in the analytical sample of Shubarkol coal (Sora) is:
0,5-(0,2+0,06) =0,26

High sulfur content in coals worsens their quality, causes a significant increase its cost and the risk of pollution of the environment with toxic sulfur oxides during energy use, and coking deterioration in the quality of coke and an increase in its consumption in the smelting of pig iron. For example, an increase in the sulfur content of coal by 0.1% leads to a decrease in the productivity of the blast furnace and an increase in the consumption of coke (by 1.8%). On the other hand, coals represent one of the main potential sources of raw sulfur. Desulfurization of coals is considered as a way of solving these two problems.

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