

UDC 547.211:622.411.33:552.57

Laboratory-Scale Methods for Determining the Gas Content of Coal Seams

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(Received June 16, 2013; revised July 15, 2013)

Abstract

Direct and indirect methods for studying the gas content of coal seams are considered. Advantages and disadvantages of volumetric, gravimetric and dynamic methods are demonstrated with respect to the estimation of potential methane content.

Key words: coal, gas content, maximum methane content, methane, coal seam, adsorption, desorption

INTRODUCTION

Coal produced in Russia *via* the mining method exhibits a high methane content ranging within 12–19 m³/t of coal. The coal bed methane represents one of the alternative sources of energy, whose reserves only in the Kuznetsk coal basin are estimated to amount to 13 trillion m³. On the other hand, methane represents a hazardous satellite of coal bed that must be extracted in order to provide safe working conditions in coal mines and in order to reduce greenhouse gas emissions into the atmosphere. To date, it remains an urgent problem to estimating coal-bed methane content for the purpose of production and utilization of methane.

The coalbed methane is contained therein in several states:

- 1) free gaseous state in the system of pores, cracks communicating with the outer surface;
- 2) adsorbed state on the surface of coal;

- 3) located in closed pores;

- 4) as a solution in the organic bulk of coal [1].

The amount of methane absorbed by carbon under certain conditions could be connected with the physical structure of the carbon material as a highly porous naturally occurring sorbent. In order to quantitatively determine the amount of methane adsorbed, researchers apply the methods of direct and indirect determination [2].

DIRECT METHODS FOR DETERMINING THE GAS CONTENT

The method is based on using special coring shells (core gas samplers) those allow one to take the samples of coal, rocks and gas in the natural ratio thereof and in order to determine the gas content in the core close to the natural one (Fig. 1). Coal samples taken from the well into sealed metal containers or core gas samplers (CGS) are delivered to the laboratory. The

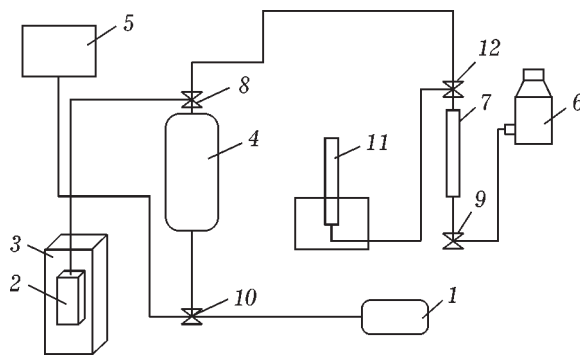


Fig. 1. Schematic diagram of a thermal vacuum degassing setup: 1 – vacuum pump; 2 – core gas sampler; 3 – thermostat; 4 – gas collector; 5 – elevated tank; 6 – equalizing vial; 7 – measuring burette; 8, 10, 12 – three-way valve switches; 9 – two-way valve switch; 11 – part of the gas sample taken for determining the componential composition.

degassing of coal cores is begun from the collection of free gas released from coal at an external pressure of 1 atm and at a room temperature. Further, the CGS is connected to the gas collection vessel is previously evacuated to a residual pressure of 10–15 mmHg, then thermostated at 80 °C for 8 h. The degassing of the coal core is considered to be complete, when under the conditions of heating and vacuum the gas evolution rate becomes equal to 10–15 cm³/h. From the gas collection container the gas is transferred to a burette for measuring the volume of gas released [3, 4]. The analysis of the gas extracted from coal core is performed by means of gas chromatography [5, 6].

The international standard [7] establishes a technique for determining the gas content in coal based on a direct determination. A carbon core after rising to the surface is placed in a sealed canister. The volume of the gas evolved is determined by means of a volumetric method every 10–15 min in the course of the first several hours, further the determination is performed every hour, and finally the measurements are carried out every 24 h, until the evolution of the gas ceases or becomes insignificant. The desorption the gas from the coal samples is performed at 62 °C or at the seam temperature, depending on the fact what the data are required for further calculations. The desorption process under these conditions could take several months, so to reduce the analysis time

it is recommended to use an accelerated method. After several steps of measuring the amount of the gas released from coal core, the sample of coal is ground in a ball grinder and degassed until reaching a complete extraction of the residual gas. The gas loss in the course of lifting the core to the surface and placing the sample in a sealed canister is estimated via extrapolating the data obtained in the course of measuring the desorption.

METHODS FOR INDIRECT DETERMINING THE GAS CONTENT

The method of indirect determination is based on establishing the methane content in coal or in rocks according the gas capacity thereof (the ability of coal to absorb carbon dioxide under certain thermodynamic conditions) obtained in a laboratory way, for the conditions of gas pressure and gas temperature measured within the wells in a coal or rock layer.

In order to determine the maximum methane content, researchers use methods based on of on a quantitative determination of free and adsorbed methane based in the pores and microcracks of coal under equilibrium conditions. The amount of methane is measured by means of volumetric, gravimetric and dynamic methods those are used in gas chromatography. These methods for determining the methane content are considered as indirect methods.

Volumetric method

The method is based on measuring the volume of gas adsorbed by carbon after adsorption equilibrium being established [8–11]. A schematic diagram of the setup for testing by means of this method is demonstrated in Fig. 2.

According to the authors of [8], the coal previously ground into the fraction of 0.2–0.25 mm and dried at 100 °C (the adsorption equilibrium is reached sooner for the fraction with the smallest grain size and lower moisture content) is placed in a flask (4), whereafter the flask is evacuated. The free space volume of the sorption flask is determined via helium puffing with a subsequent pumping thereof out. After measuring the free space, methane is fed into the flask at a pressure of

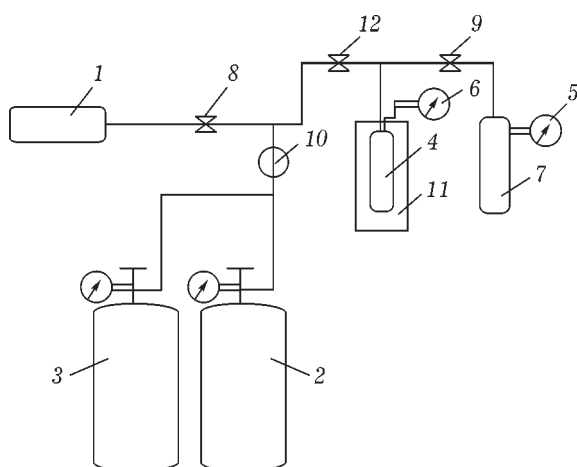


Fig. 2. Schematic diagram of the setup for determining the maximum methane content in coal with the use of volumetric method: 1 - vacuum pump; 2, 3 - cylinders with He and CH₄; 4 - absorber flask with the sample; 5, 6 - pressure sensors; 7 - a vessel of known volume (gas collector); 8, 9, 12 - valve switches; 10 - gas flow controller; 11 - thermostat.

30–50 atm, and then the flask is placed in a thermostat (11). After establishing the equilibrium sorption, which is registered according to the pressure constancy with the use of a manometer (6), the flask is connected to a previously evacuated gas collector (7), and the gas is slowly released from the flask into the gas collector. When the gas pressure in the gas collector reaches an atmospheric pressure value, valve (9) should be closed. Further, the gas pressure and temperature in the gas collector is registered. The next release of the gas is carried out in 4–5 h.

In order to construct the sorption isotherm, the gas is charged 8–9 times, until it is completely removed from the coal. The amount of the gas desorbed is summed, with the subtraction amount of free space volume in the flask. The disadvantage of this method consists in an incomplete desorption of methane from the coal. One could eliminate the error of measuring the amount of methane absorbed by carbon under reaching the sorption equilibrium, if before the flask with the coal a gas flow regulator with a control unit and display is connected. The accuracy of the method depends on the accuracy of measuring the pressure, volume, free space and thermostating conditions.

The authors of [9, 11] improved the volumetric method of determining the amount of methane in free and adsorbed states in open pores and cracks of coal under equilibrium conditions. The method is implemented in three stages. At the first stage, the methane amount ($Q_{o/p}^f$) in open pores is calculated:

$$Q_{o/p}^f = P_s V_{o/p} m_c / P_{atm}$$

where $V_{o/p}$ is the specific volume of open pores and cracks, which is determined *via* pycnometry; P_s is the methane pressure in the saturation chamber; m_c is the mass of coal; P_{atm} is atmospheric pressure.

At the second stage, the amount of methane adsorbed on the surfaces of coal ($Q_{o/p}^{ads}$) is determined from the difference between volumes of gases desorbed from the container with the coal after the saturation thereof with helium, and then after the saturation with methane at a pressure of 30 atm. The amount of methane adsorbed onto the exposed surfaces of coal is almost two times higher than the amount of methane in the free state.

At the third stage, the amount of the gas in closed pores ($Q_{c/p}$) is determined using a volumetric method. In the course of discharging the pressurized gas from the container with the coal, there occurs an evacuation of free and adsorbed methane from open pores and cracks. Then there occurs a slow and long process of methane desorption from the closed pores of coal. The disadvantage of the volumetric method consists in an incomplete extraction of sorbed gas, which could be easily eliminated via cooling the storage vessel by liquid nitrogen. The vapour pressure of methane at -196°C is equal to about 10 mmHg, which results in maintaining a low pressure in the vessel, and the process of methane desorption occurs in a more complete manner. Upon completing the desorption the storage vessel should be closed and thermostated at a room temperature to register the pressure established therein. The total amount of methane released from the coal after the saturation thereof with the methane compressed to a pressure of 3 MPa is equal to

$$\Sigma Q_{CH_4} = Q_{o/p}^f + Q_{o/p}^{ads} + Q_{c/p}$$

Gravimetric method

The gravimetric method is based on weighing the coal previously prepared (just so as for the volumetric method) before the saturation with methane and after establishing the adsorption equilibrium [12, 13]. An increase in the mass quantifies the adsorption. The disadvantage of this method consists in the accuracy of weighing the flask with coal before and after establishing the adsorption equilibrium. For this reason, the volumetric method is implemented in the laboratory practice much more often than the gravimetric one.

Thermal desorption method

The widespread method of thermal desorption represents a dynamic method used in gas chromatography [14]. The main metering instrument is presented by a detector that registers the concentration of an adsorbate in the carrier gas. Thermal conductivity detectors are relatively simple and widespread.

Through a column with a preliminary prepared adsorbent (carbon) is passed a flow of helium-adsorbate (methane) mixture at a constant flow rate. When the column temperature is high enough so that methane almost is not adsorbed onto the coal, the composition of the mixture before and after the column is the same and the chromatographic profile exhibits a zero line. In the case of further cooling the column down to low temperature values, for example, via placing the column in a Dewar vessel filled with liquid nitrogen, the mixture composition at the outlet should change due to the gas adsorption onto the adsorbent, to exhibit a deviation from the zero line in the chromatographic profile. After reaching the equilibrium, the composition of the mixture at the inlet and the outlet should become equal again. Heating the column up to the initial temperature (for example, simply *via* removing the Dewar vessel) should result in the adsorbate desorption. The signal of the chromatograph detector in this case should have an opposite sign. The value of adsorption or desorption level can be determined from the area of corresponding peaks with the use of calibration data. In practice, researchers consider the peak of desorption,

inasmuch as it is less diffused due to rapid releasing the adsorbate.

Express method for estimating the gas content in coal

In order to determine the gas content of coal in a critical area of formation the authors of [15–17] proposed a new rapid method. The method includes sampling the drilling culm into sealed devices, simultaneous measuring the amount of methane desorbed by the drilling culm at different moments of time (V_t), and the value of coal temperature decrease of at the time moment t (ΔT_t). From the results of measuring the ΔT_t value one can determine the amount of gas desorbed from coal at different time points (V_t^T):

$$V_t^T = [0.01((100 - W)C + WC_w)\Delta T_t]/q$$

where W is the humidity of coal, %; C is the specific heat capacity of dry coal, $J/(g \cdot ^\circ C)$; C_w is the specific heat capacity of water, $J/(g \cdot ^\circ C)$; ΔT_t is the decrease of coal temperature due to desorption of gas therefrom just at the moments of time t , when the amount of desorbed gas is measured, $^\circ C$; q is the integral heat of gas desorption from coal, J/cm^3 . To calculate the gas content the highest value among them is used.

CONCLUSION

Basic laboratory methods those are used in order to study the gas content of coal seams are considered. The most common is direct method, however, the reliability of the analysis depends on many factors such as the time from the moment of lifting the coal core to the moment of sealing thereof, the quality of sealing the core gas sampler, timely delivering the samples to the laboratory. The objective evaluation of coalbed gas content by means of indirect methods is possible only the in the course of performing the investigations under thermodynamic conditions those correspond to naturally occurring conditions inherent in the coal seam. The advantage of indirect methods consists in the fact that a series of tests can be performed with a single sample taken from the coal seam.

At the same time, in the course of studying the methane content of coal using a laboratory-scale method, an analyst faces a lack of regulations those meet modern analysis requirements. So, according to [3, 4] (direct method for determining the gas content of coals), the componential composition of the gas should be determined according to the normative document [5], that is no longer valid now. A new regulatory document is imposed such as State Standard GOST 31371–2008 [6], but it does not take into account a specific composition of the gas extracted from the coal core, which composition varies within a wide range of concentration values. At the same time, the indirect volumetric method outlined in [8], does not take into account the most contemporary achievements in the laboratory equipment.

Taking into account the data obtained and the experience of the authors concerning this subject, one can conclude that in order to improve the quality of laboratory tests and to achieve the significance of objective evidence concerning the methane content in coal it is necessary to develop new measurement methods with the use of modern hardware support, as well as regulations for the determination of natural gas content in coals.

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