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NATIONAL STANDARD OF THE
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ICS 13.100

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GB/T 23249-2009

**Coalbed Gas Content Measurement Methods in
Geological Exploration Period**

地勘时期煤层瓦斯含量测定方法

Issued on: March 11, 2009

Implemented on: November 1, 2009

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine;**

**Standardization Administration of the People's Republic of
China.**

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Coalbed Gas Content Measurement Methods in Geological Exploration Period

1 Scope

This Standard stipulates the terms and definitions, measurement methods, records and sample delivery, coal sample degassing and gas analysis, determination result calculation and evaluation of coalbed gas content in geological exploration period.

This Standard is applicable to the determination of coalbed gas content and gas composition through coal core during the geological exploration period.

2 Normative References

Through the reference in this Standard, clauses of the following documents become clauses of this Standard. In terms of references with a specific date, all the subsequent modification sheets (excluding the corrected content) or the revised editions are not applicable to this Standard. However, all parties that reach an agreement in accordance with this Standard are encouraged to explore the possibility of adopting the latest version of these documents. In terms of references without a specific date, the latest version is applicable to this Standard.

GB/T 212 *Proximate Analysis of Coal* (GB/T 212-2008, ISO 11722:1999, NEQ)

GB 474 *Method for Preparation of Coal Sample* (GB 474-2008, ISO 18283:2006, MOD)

GB/T 3715 *Terms Relating to Properties and Analysis of Coal* (GB/T 3715-2007, ISO 1213-2:1992, NEQ)

GB/T 13610 *Analysis of Natural Gas Composition - Gas Chromatography*

GB/T 15663.8 *Terms Relating to Coal Mining - Part 8: Mine Safety*

3 Terms and Definitions

What is defined in GB/T 15663.8 and GB/T 3715, and the following terms and definitions are applicable to this Standard.

3.1 Coalbed Gas Content

Coalbed gas content refers to methane and heavy hydrocarbon gas content that unit mass or unit volume of coal body contains under the original state.

- b) Before using the desorption instrument, use suction balloon to raise the liquid level in the measuring tube to the zero point; close the screw clamp (see Figure 2). Then, place it still for 10 min, if the liquid level in the measuring tube remains still, then, it shall be qualified.

4.1.3 Sampling of coal sample

Sampling and requirements are as follows:

- a) Use common coal core tube to drill-take coal core. The length of coal core taken in one time shall be not less than 0.4 m;
- b) After the coal-drilling is completed, during the process of elevating the drilling tool, back-fill flushing fluid into the drill hole; maintain the drill hole often filled with flushing fluid (if this requirement cannot be satisfied because the drill hole severely leaks water, this shall be indicated in the sampling record). The elevating process shall be as continuous as possible. If due to mechanical failure, this process is suspended in the midway, in area where the hole depth is less than 200 m, the suspension time shall not exceed 5 min; in area where the hole depth is 200 m ~ 500 m, the suspension time shall not exceed 10 min; in area where the hole depth is 500 m ~ 1,000 m, the suspension time shall not exceed 20 min; in area where the hole depth is more than 1,000 m, the suspension time shall not exceed 30 min (conditions of suspension time: under 1/2 hole depth; the medium of well drilling is water or mud);
- c) In terms of sampling time, relevant items shall be filled out in details in accordance with the format of Table A.1 in Appendix A. The stipulations of the timing (accurate to min) of several operations are as follows:

T_1 ---drill-elevating time, expressed in (min);

T_2 ---time of elevating drilling tool to the well head (calculated by the finished elevation of the last drilling tool), expressed in (min);

T_3 ---time of starting desorption and determination (calculated by the time of opening the spring clip after the desorption instrument's exhaust tube is connected with the puncture needle), expressed in (min);

- d) After coal core is pulled out of the drill hole, disassemble the coal core tube as soon as possible. Fill the adopted coal sample into the sealing tank. The time that the coal core is exposed in the air shall not exceed 8 min;
- e) After taking out the coal core, in terms of columnar coal core, adopt an intact part from the middle which contains little gangue; in terms of powdered and blocky coal core, the gangue, mudstone and grinding burnt parts shall be eliminated. Do not use water to clean the coal sample; maintain its natural state, load it into the sealing tank. Do not compact it. The tank mouth shall

retain around 10 mm of space;

- f) Firstly, insert the puncture needle into the sealing rubber pad, which is above the tank cover, then, use a wrench to screw on the tank cover. Then, connect the desorption instrument's exhaust tube with the puncture needle; immediately open the spring clip 8 (see Figure 2); record this time as T_3 .

4.2 Determination of Gas Desorption Speed

4.2.1 Gas desorption speed determination device is shown in Figure 2. After the sealing tank connects with the desorption instrument through exhaust tube 9, open the spring clip 8. Soon after that, gas, which leaks out from coal sample, enters the measuring tube. Open the sink's water discharge tube 7; use water drainage and gas collection method to collect gas in the measuring tube.

4.2.2 Within a certain interval, record the reading of the measuring tube and the determination time; maintain consecutive observation for 120 min. The stipulations of the reading interval time are as follows: within 60 min before starting the observation, 1 min ~ 2 min of interval from the 1st point to the 3rd point; after that, at each interval of 2 min ~ 5 min, read once; within the second hour, at each interval of 10 min ~ 20 min, read once.

4.2.3 If the volume of the measuring tube is not sufficient to contain all the gas, which leaks out from coal sample within 120 min, use spring clip 8 to firmly clamp the exhaust tube in the midway, re-elevate the liquid level to the measuring tube's zero point, meanwhile, supply sufficient clean water into the sink. Then, open the spring clip, continue the observation.

4.2.4 The above-mentioned observation shall be conducted in places with relatively stable temperature. The sealing tank shall receive thermal insulation and anti-freezing treatment.

4.2.5 If under the observation with the desorption instrument, no gas is discharged, examine whether the puncture needle, the exhaust tube and the upper gas vent of the sealing tank are blocked. If they are not blocked, then, it is caused by an excessively small content of gas. At this moment, the observation may be terminated; submit it to the laboratory for determination.

4.2.6 After the above-mentioned observation is completed, pull out the puncture needle, slightly screw the compression screw (apply moderate force; avoid becoming too tight, which will make the rubber pad lose its elasticity). Fill out the observation result in the table of record (see Appendix A.2); record the temperature of the gas and water, and atmospheric pressure.

4.3 Calculation of Gas Loss

4.3.1 Desorption gas volume correction

5 Record and Sample Delivery

5.1 Attached Documents of Coal Sample

Attached documents are as follows:

- a) Sampling record table (see Table A.1 in Appendix A);
- b) Coal sample gas desorption speed determination record table (see Table A.2 in Appendix A);
- c) Gas loss calculation chart and calculation result;
- d) Gas coal sample delivery note (see Table A.3 in Appendix A).

The above-mentioned documents shall be in duplicate: after the sample delivery organization's review, one copy shall be provided to the test organization, the other copy shall be kept by the sample delivery organization.

5.2 Sampling Record

Sampling record shall simply describe the geological condition (structure, igneous rock, lithology of seam roof and floor) of the sampling location, and coal quality (grain size, degree of contamination and physical properties, etc.); indicate unforeseen circumstances in sampling and tests.

5.3 Coal Sample Tank Marking and Coal Sample Preservation

Use steel seal to mark the number and the organization. The sample delivery organization shall uniformly number it and load it into a specially-made sample box for delivery. The box shall be filled with thermal insulation materials. Preserve it at room temperature (20 °C). Relevant documents of samples and tests shall be delivered to the test organization within 2 d ~ 3 d.

6 Coal Sample Degassing and Gas Analysis

6.1 Instruments, Equipment and Materials

Instruments, equipment and materials are as follows:

- a) Vacuum degassing device: degasser, as it is shown in Figure 4.

- a) After coal sample is delivered to the laboratory, firstly, inspect the leakage. Immerse the sealing tank in clean water, then, meticulously observe it for 5 min; check whether there are bubbles. If bubbles are found, after certain treatment (screw down the tank cover or the compression screw), if the test can be continued, it shall be considered as reference sample, otherwise, it shall be considered as rejected. The inspection result shall be indicated in the report;
- b) Check whether the gas coal sample delivery note is consistent with the tank No.; whether the test documents are complete. After confirming there are no mistakes, uniformly register and number it. Then, proceed to the next step of determination as soon as possible.

6.3 Preparation before Degassing

Preparation is as follows:

- a) Before the assembly of various glass components of the vacuum degassing device, clean and dry them. After the assembly, fill gas aspiration bottle 11, vacuum bottle 12, measuring tube 13 and 14 (see Figure 5) with a proper amount of acid saturated saline as the limiting fluid. The various connecting parts of the vacuum system shall be sealed by vacuum sealing compound. After rinsing the vacuum piston, put on a coating of vacuum sealing grease. When rinsing the piston, prevent organic solvent's contamination of the instrument;
- b) Before using the vacuum degassing device, conduct rigorous leakage test. It is requested that the vacuum system shall be placed for 240 min under the instrument's maximum vacuum degree, the elevation of the vacuum gauge's mercury level shall not exceed 5 mm. Under the circumstance of lowering the leveling bottle, the various measuring tubes shall manifest no gas leakage; the liquid level shall remain still;
- c) After overhauling, the instrument shall receive a re-test of leakage.

6.3.1 Coal sample degassing before smashing

6.3.1.1 Pre-evacuation

Before coal sample is connected with the degasser, evacuate the left-side vacuum system of the instrument. When it reaches the maximum vacuum degree 10^{-2} Pa, stop the pump; observe the vacuum gauge's mercury level. If it maintains still within 10 min, then, it shall be determined as qualified.

Connect coal sample tank with the degasser. Firstly, close the degasser's vacuum gauge. Through the puncture needle and the vacuum rubber tube, connect the sealing tank with the degasser (if the upper rubber pad of the sealing tank is so tight that the

inhalation of air. The storage time (count from the finish of degassing) of gas sample in the bottle shall not exceed 120 min.

After the sampling of gas sample is completed, use limiting liquid to completely discharge the residual gas in the gas storage bottle and the tomb-shaped tube, so that the next test result will not be affected.

6.3.1.5 Gas analysis

Conduct gas analysis in accordance with GB/T 13610. Determine the volume fraction of various components in the degassed gas.

6.3.2 Coal sample smashing

The requirements of coal sample smashing are as follows:

- a) Before using the ball grinding tank, in accordance with 4.1.2, conduct air tightness test;
- b) When loading coal sample into the tank, if the coal sample is in a large block, beforehand, mash the coal sample in the tank to the grain size of below 25 mm. Then, screw down the tank cover, seal it up;
- c) When the mass of coal sample that is smashed to the grain size of below 0.25 mm exceeds 80%, it shall be determined as qualified.

6.3.3 Degassing and weighing of coal sample after smashing

After smashing, coal sample degassing shall be conducted in accordance with 6.3.1 in this Standard. In this stage, degassing shall be maintained, till the mercury column of the vacuum gauge becomes stable. Then, close the vacuum gauge, take down the ball grinding tank. Wait till the tank body cools down to the room temperature, then, open the tank body; weigh the mass of the coal sample (accurate to 1 g). In accordance with the method in GB 474, shrink it into analytical coal sample. The analysis of M_{ad} , A_{ad} and V_{daf} shall comply with the method in GB/T 212. The remaining coal sample shall be retained for 1 month, then, be processed.

7 Calculation and Evaluation of Determination Result

7.1 Calculation of Coalbed Natural Gas Composition

Coalbed natural gas composition is calculated in accordance with the gas composition which is obtained through coal sample degassing before smashing.

Set the respective concentration of various gas components obtained through the gas chromatography of gas, which is mixed with air and obtained in the first stage of degassing to be: $c(O_2)$, $c(N_2)$, $c(CH_4)$, $c(CO_2)$, $c(\text{heavy hydrocarbon})$, %. In

Except from methane, combustible gas in coalbed also contains heavy hydrocarbon and hydrogen, etc. Firstly, in accordance with Formula (13) and Formula (14), calculate the content of each combustible gas. Then, add up the content of the various combustible gases. Thus, the total combustible gas content (mL/g) can be obtained.

7.3 Processing of Calculated Numerical Value

After processing the above-mentioned calculated numerical value in accordance with the rounding-off rule of numerical value, the digits stipulated below shall merely be retained:

- a) The calculation result of gas volume shall retain 1 decimal place;
- b) The analysis result of gas shall retain 2 decimal places;
- c) The mass of coal sample shall retain 1 decimal place;
- d) The calculation of gas content in coal shall retain 2 decimal places;
- e) When the calculated value is less than 2 decimal places, it shall merely be indicated as “micro” in test report.

7.4 Test Report

7.4.1 Laboratory's degassing determination result, and gas analysis result shall be filled out in the record table (see Table A.4, Table A.5 and Table A.6 in Appendix A). In addition, various circumstances (such as air leakage of sealing tank, gas loss during the degassing process, dirt band mixed in coal sample, improper smashing of coal sample, etc.) that happen during the degassing process shall be filled out in the NOTE in details.

7.4.2 The test organization shall collect desorption determination result and submit a final test report (see Table A.7 in Appendix A). The test report shall be in duplicate. One copy shall be mailed to the sample delivery organization; one copy shall be retained.

7.4.3 Original data formed during the test process, and test report shall be archived by the test organization and retained as long-term data. Archived files include:

- a) Original data:
 - 1) Sampling record (see Table A.1 in Appendix A);
 - 2) Coal sample gas desorption speed determination record (see Table A.2 in Appendix A);
 - 3) Gas loss calculation chart;
 - 4) Gas coal sample delivery note (see Table A.3 in Appendix A);

b) Test report:

- 1) Degassing record (see Table A.4 in Appendix A);
- 2) Coalbed gas content determination result summary table (see Table A.5 in Appendix A);
- 3) Gas analysis test report (see Table A.6 in Appendix A);
- 4) Coal quality analysis test report;
- 5) Coalbed gas content test report (see Table A.7 in Appendix A).

7.5 Evaluation of Determination Result

7.5.1 Qualified samples

The requirements of qualified samples are as follows:

- a) Below 1/2 hole depth, and when the medium of well drilling is water or mud, during the drill elevation process, in area where the hole depth is less than 200 m, the suspension time shall not exceed 5 min; in area where the hole depth is 200 m ~ 500 m, the suspension time shall not exceed 10 min; in area where the hole depth is 500 m ~ 1,000 m, the suspension time shall not exceed 20 min; in area where the hole depth is more than 1,000 m, the suspension time shall not exceed 30 min;
- b) The length of drilled-taken coal core shall be more than 0.4 m; the mass of coal sample shall be more than 250 g;
- c) Coal sample ash content (A_{ad}) shall be less than 40%. In mining areas with complicated coalbed structure and high ash content, geological exploration corporation shall make its own standards in accordance with the local conditions;
- d) Desorption instrument's measuring tube shall not leak air; air channels (including sealing tank's exhaust vent, needle and rubber tube, etc.) shall not be blocked;
- e) Desorption measurement points shall be regularly distributed. In the initial stage of desorption, linear relationship shall exist. However, in terms of sample which has no desorption or sample whose desorption is less than 50 mL, this requirement is not applicable;
- f) Coal sample's exposure time in surface air (t_2) shall be less than 8 min. The time from finishing loading the tank with coal sample to starting desorption ($T_4 - T_3$) shall be less than 2 min. The degassing process shall have no gas loss;

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