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Method of determining coalbed methane content

National foreword

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Foreword

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The committee responsible for this document is ISO/TC 263, *Coalbed methane (CBM)*.

Method of determining coalbed methane content

1 Scope

This International standard provides methodology for measuring coalbed methane content of coal core samples obtained by coring or sidewall coring during well drilling. This standard has to be also applied to drill cuttings samples, if the equipment for the determination of the coalbed methane content according to a respective national standard is not available. The selection of the most appropriate method shall consider the purpose of the test and the possibilities of sampling.

This International standard is applicable for the direct method of measuring coalbed methane content. It includes sample preparation, experimental procedures and calculation methods. Indirect methods of measuring gas content of coal (not included in this International standard) are generally based on either the gas sorption characteristics of coal under defined/specified pressure and temperature conditions.

This International standard includes three types of direct measuring methods: conventional desorption (slow desorption) of core samples, fast desorption of core samples, fast desorption of cuttings or lump samples. The difference among them lies in the time allowed for gas to desorb before final crushing and in sample size and shape.

This International standard is applicable for the determination of the methane content of coal during coal and coalbed methane exploration for the determination of free gas content of low rank coals is included.

Procedure of free gas content determination of low rank coals need to be defined.

All units used and referred to in this International standard are international standard units. Reference temperature is 0 °C (273,15 K) if not stated differently. Reference pressure is 0,1 MPa (1 bar) if not stated differently.

This International standard does not point out all the potential safety hazards associated with its use. The users are responsible for establishing appropriate safety measures and health practices when applying the procedures defined in this standard.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
coalbed gas content
under natural conditions, quantity of gas contained in the unit mass of coal

Note 1 to entry: It is the sum of lost gas, measurable gas and residual gas.

2.2
coalbed methane content
methane content, which is commonly the major component of coalbed gas

Note 1 to entry: The terms “coalbed methane”, “coalbed gas” and “coal seam gas” are frequently used synonymously.

Note 2 to entry: If a component other than methane is the predominant component of coalbed gas this should be stated explicitly.

2.3
Q1 (lost gas)
gas lost from the samples subsequent to its removal from its *in situ* position and prior to its containment in the canister, expressed as volume (at standard conditions) per unit mass of coal

2.4
Q2 (measurable gas)
measurable gas desorbed at atmospheric pressure from the non-pulverized coal sample, expressed as the quantity per unit mass of coal

2.5
Q3 (residual gas)
gas still contained in the coal sample before its pulverisation, expressed as the quantity per unit mass of coal

2.6
conventional desorption (slow desorption)
method of gas determination in which volumetric readings of canister gas content are taken frequently (every 5 min) during the first few (2 to 6) h, followed by hourly measurements for several hours, and then measurements on 24-h intervals until no or very little gas is being desorbed for an extended period of time

2.7
fast desorption
method of gas content determination in which a trend of gas desorbing from the coal sample is not established

Note 1 to entry: Measurements are taken over a period of time typically less than 1 day

2.8
sampling
activity that take some representative part of coal from coalbed

2.9
core sample
cylindrical section of rock (coal) that is usually 5 cm to 10 cm in diameter taken as part of the interval penetrated by a core bit and brought to the surface for geological examination, representative sampling, and laboratory analysis

2.10
cuttings sample
rock (coal) fragments, chips, particles with a size of typically ≤5 mm that break off because of the action of the drill bit and are transported to the surface by the drilling circulation system

2.11
coal lump samples
coal fragments or pieces of coal fragments that break off in the extraction or development process of a coal mine operation, from coal transport or storage or by manual removal from the coal seam or an intact core sample. The size is larger than that of cuttings and smaller than the inner diameter of the canister used for transportation and desorption

2.12
low-rank coal
the coal and rock thermal evolution degree is low, vitrinite of oil-immersed maximum reflectance $R_{\mathrm{v,max}} \leq 0{,}65\%$, including lignite and long flame coal

[SOURCE: ISO 11760:2005]

2.13
high-rank coals
coal and rock thermal evolution degree is high, vitrinite of oil-immersed average random reflectance $\overline{R}_r \geq 2{,}0\%$, including anthracite coal

3 Apparatus

3.1 Desorption canister

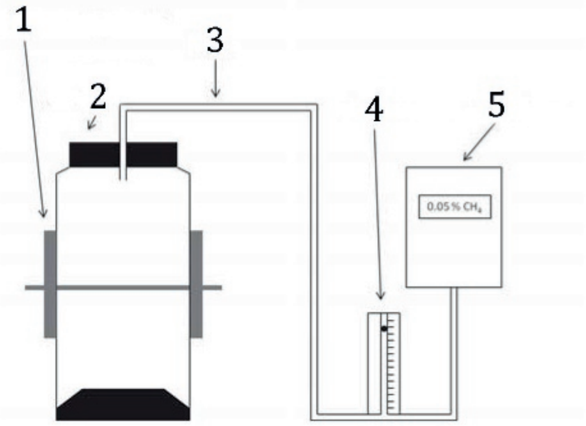
The desorption canister is a sealable container into which the coal samples are placed to determine their gas content. The canister should be designed such that the coal can be transferred into a pressure-tight status as quickly as possible.

Canisters should be easy to handle and fill and should close rapidly to form a gas sealed space.

The material of the canister shall not absorb coalbed gas, or react with drilling fluid and coalbed gas. Therefore, it is recommended to use aluminium alloy, coated aluminium, PVC and/or stainless steel materials.

When using core samples, the canister volume shall be more than 1 000cm^3 with a suitable inner diameter. It should maintain gas tightness up to a pressure of 0,3 MPa.

When using cuttings samples or coal lump samples, the canister volume shall be ≥ 500 cm^3. The ratio of canister volume and sample volume shall be between 70 and 150. The desorption canister shall be equipped with a shut-off valve and a rubber hose to connect it to a gas metering device and gas analyser (Figure 1).



Key

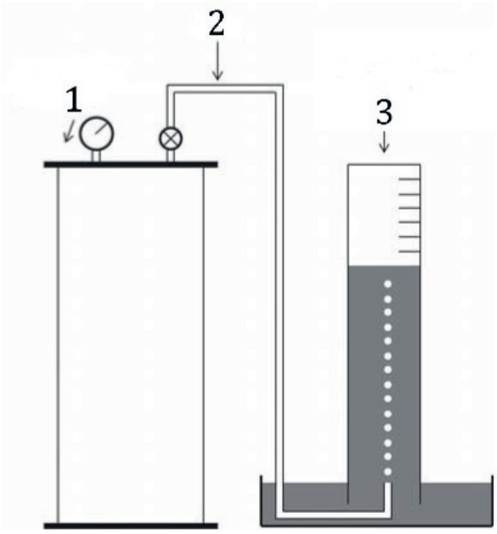
1 squeezing device
2 desorption canister
3 hose
4 flowmeter
5 gas analyser

Figure 1 — Desorption canister with connection to gas analyser

3.2 Metering device

The measurement of desorbed gas volumes shall be executed either by the displacement of water (displacement method) or by flushing the crusher head with a defined volume of air and subsequent analysis of the gas composition (flushing method).

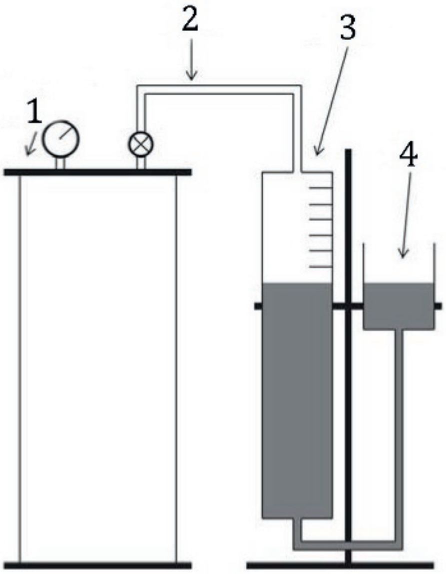
For the displacement method, a graduated measuring cylinder (burette) is used. The volume and scale of the measuring cylinder shall be appropriate to the desorption volume. The measuring cylinder and storage reservoir are preferably of glass. Readings of the gas volume are taken at atmospheric pressure by adjusting the liquid levels in the tube and the storage cup. The minimum graduation on the metering scale shall be no more than 10 cm^3. The measuring cylinder is connected at the bottom to a storage reservoir and at the top to the desorption canister head. The connections shall be made of flexible and gas-tight plastic or rubber.



Key

1 desorption canister
2 hose
3 graduated cylinder

Figure 2 — Desorption canister with graduated cylinder for collection of desorbed gas



Key

1 desorption canister
2 hose
3 burette
4 storage reservoir

Figure 3 — Desorption canister with graduated burette and storage reservoir for pressure equilibration

For the flushing method, a defined volume of air is passed through the crusher headspace and collected in a vessel, preferably of glass. The collecting vessel shall be connected to the gas analyser [see 3.13] by a flexible hose. The connections shall be flexible and gas tight plastic or rubber.

3.3 Thermostat

A water, oil or air thermostat shall be used with a temperature stability and controlling accuracy of ± 1°C.

3.4 Temperature measuring devices

Temperature should be measured with an accuracy of 1°C or better. The temperature range of the measuring devices (thermometer, thermocouple, resistance thermometer) should cover both the ambient temperature and the temperature in the thermostat holding the desorption canisters. Typically this should correspond to a range from 10 °C to 80 °C.

3.5 Barometer

The measurement range of the barometer for metering the ambient pressure should cover the local range of ambient pressure. Scale intervals should be 0,1 kPa.

3.6 Electronic balance (top load)

A precision (accuracy ± 1 %) electric, top-load balance with appropriate capacity is used to weigh empty and filled canisters.

3.7 Standard sieve

The sieve shall be 60 mesh (maximum grain size 2 mm)

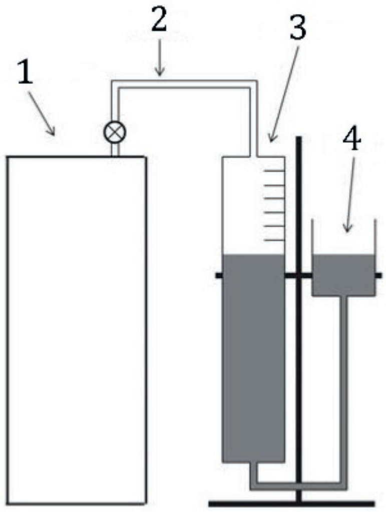
3.8 Coal crusher

A coal crusher is required for pulverization of the coal in order to release all remaining gas rapidly.

For conventional desorption test, samples are completely broken during the residual gas test in the measurement process.

In case of using the displacement method, the crusher shall be gas tight and equipped with an adapter for connecting a rubber or plastic hose to transfer gas to the metering device (Figure 4). The crusher headspace shall be minimized.

In case of using the flushing method, the tightness of the crusher shall be adjusted to the flushing process. The gas being released during crushing shall be mixed with inflowing air completely with all mixed gas being captured in the collecting vessel.



Key

1 coal crusher

2 hose

3 burette

4 storage reservoir

Figure 4 — Coal crusher with graduated burette and storage reservoir for pressure equilibration

3.9 Hose

The hose material shall not adsorb coalbed methane and not react with it.

3.10 Gas-sample bottles

Gas-sample bottles are used for collecting samples for subsequent analysis. The volume of the gas-sample bottle shall not be less than 100 cm^3

3.11 Padding

Cylinders, glass spheres and hollow tubes are used to reduce the void volume of the desorption canister. These should be made of materials not adsorbing coalbed methane and not reacting with coalbed methane.

3.12 Quick connector

Quick connector is a pipe fitting designed for easy and rapid connection and disconnection.

3.13 Gas analyser

The gas analyser shall be equipped with sensors for the main components of the adsorbed gas, at least with a sensor for methane, preferably an infrared sensor. The accuracy of the analyser shall be ± 5 % of the measured value or better.

4 Sample collection

4.1 Preparation of sampling

4.1.1 General

The container [3.1] volume shall be above 1 000 cm^3, and its diameter shall be suitable for coal cores, and maintain gas tightness under pressure of 0,3 MPa. The material of the container shall not absorb coalbed methane, or react with drilling fluid and coalbed methane. Therefore, it is recommended to use aluminium alloy, coated aluminium, PVC and stainless steel materials.

4.1.2 Desorption canister

All desorption canisters (3.1) for measuring coalbed methane content shall pass an air tightness test before use. For this test the desorption canister shall be pressurized with air to a pressure of 0,3 MPa. The canister is usable if the pressure does not drop by more than 0,001 MPa within 12 h.

4.1.3 Metering device

Before use, the metering device shall be filled with water and initialized to check its air-tightness and performance.

4.1.4 Thermostat

Before filling the coal samples into the canister for the desorption test, the thermostat shall be adjusted to the reservoir temperature. Sufficient time shall be allowed for the thermostat to stabilize at this temperature.

4.2 Sampling principle

4.2.1 Sample quality

4.2.1.1 Sample quality of core samples

The mass of coal sample placed into a canister shall not be less than 800 g.

If coal core recovery is not sufficient but the measurement is still required, the minimum sample mass shall not be less than 300 g and only desorption gas could be measured in this situation. This fact shall be explicitly stated in the report.

4.2.1.2 Requirements for cuttings samples and coal lump samples

For samples from in-seam drilling, cuttings with a defined grain size, preferably ≥ 2mm shall be taken from the outbye end of the borehole without mixing coal from different positions within the borehole.

Cuttings and lump samples shall have a mass of about 10 g. The actual mass of the coal sample shall be appropriate to the desorption canister.

The sample can also be a lump of coal taken from the core. The weight shall be about 10 g.

In both cases, the sample shall be dry. Otherwise, a suitable drying agent shall be added to the desorption canister.

Sampling of cuttings shall be preferable by rapid sucking through a hollow drill string. Samples shall be verifiable taken from outbye the degassed zone.

4.2.2 Sampling time

Sampling time is the actual time between encountering the virgin coal with the sampling device and sealing of the coal sample in the desorption container.

For core samples, the time of coal samples being pulled to wellhead is stipulated as:

Pulling core time should be less than 2 min in every 100 m.

After reaching the surface, the samples should be sealed in the container within 10mins.

4.3 Sampling procedure

4.3.1 General

The equipment should be installed and adjusted before the drill bit encounters the coal seam, the sampling technician shall arrive at the site

4.3.2 Core samples

After the core being pulled out of wellhead, open the core barrel as quickly as possible. With the assistance from sampling people, geologist shall quickly take pictures, make brief descriptions, remove non-coal material and drilling mud, quickly sample the core into the desorption container according to coal seam orientation and seal the core without compressing.

Samples used for CBM content determination should be filled to up to 1cm from the container top. If the sample quantity is not enough to fill the container, inert non adsorbing spacer should be used to reduce the void volume of the container. The maximum void volume in the container should not exceed 1/4 of the container volume.

4.3.3 Cutting samples

Before sampling, all desorption canisters shall be cleaned, checked for possible leakage and labelled. Desorption canisters shall be flushed with ventilation air immediately before sampling.

After the cuttings have been recovered from the borehole, cuttings shall be separated using an appropriate sieve. The fraction used as sample shall be of a defined grain size (preferable of ≥ 2 mm). The sample shall be sealed in the desorption canister as quick as possible. For in-seam drilling, the sampling time shall be no more than 3 min.

4.4 Coal lump samples

4.4.1 General

Before sampling, all desorption canisters shall be cleaned, checked for possible leakage and labelled. In case of underground sampling, desorption canisters shall be flushed with ventilation air immediately before sampling. After the core has been recovered from the borehole, a lump of coal shall be separated from the core. The sample shall be sealed in the desorption canister as quick as possible.

4.4.2 Parameters to record

The following relevant parameters should be collected during sampling, some of parameters depend on coal samples used or cutting samples or coal lump samples:

a) geographical information: well/ borehole name, well/ borehole coordinate, name of sampling location (coal mine, mine working),

b) geological information: coal seam name or number, the stratum age, burial depth, reservoir temperature;

c) time parameters: time of encountering the virgin coal with the sampling device, time of pulling core, time of pulling core at surface, time of sealing the coal sample in the desorption container, and sampling date;

d) sample parameters: container No, sample ID, container mass, sample mass and the sample state; for cutting samples or coal lump samples, record container number and sampling location within the borehole.

e) parameter of atmosphere at drilling site: methane concentration, temperature and barometric pressure,

f) record form: see Annex A –Original data of gas natural desorption; Annex F-Sampling form for in-seam drilling; or Annex G- Sampling form for core drilling

5 Measuring method and process

5.1 Use of core samples

5.1.1 Natural desorption

5.1.1.1 Desorption steps

After filling and sealing the container, the container should be transferred into the thermostat device which has already reached at reservoir temperature.

Connect the container and metering devices with a hose (3.9), adjust the measuring device fluid level, let the desorbed gas go into the graduated cylinder, adjust the metering device and the water cup so that the water pipe is aligned with reading, record the gas volume, ambient temperature and atmospheric pressure, at the same time, and fill them in the table in Annex A respectively.

In the next measurement, note the volume readings, subtract desorbed gas volume obtained from last reading. Later repeat measuring and record data according to above mentioned requirement.

If the reading of gas volume is over the maximum reading, discharge the gas from the burette adjust the metering device to the original state and then close the valve and repeat the steps above.

During the whole measurement process, especially during the initial desorption period when desorption speed is relatively fast, metering device should be adjusted to keep volume pipe fluid level and water cup level relatively close in order to keep natural desorption pressure.

5.1.1.2 Time intervals of volume readings

The time intervals of volume readings depend on the pressure build up in the canister. Recommended intervals: measure every 5 min the first four times then every 10 min after the samples are put in the container, together for 1 h, then test every 15 min for 1 h, test every 30 min for 1 h, test every 60 min for 1 h, test every 120 min for 4 h, until ultimately tested for 8 h.

After the first 8 h of desorption measurement, the appropriate time interval should be determined according to pressure gauge but not longer than 24 h.

5.1.1.3 Criteria for termination of desorption measurement

Termination of the desorption measurement depends on whether fast desorption method, the conventional desorption method or both are applied. The selection of the method depends on how urgent the data are needed.

In the conventional desorption method, the test terminates when desorption volume per day is less than 10cm^3 for consecutive 7 days.

In the fast desorption method, when adequate data are measured to calculate the lost gas (according to the schedule of 5.1.1.2 for the first 8 h), the desorption container could be opened and the coal can be transferred to a crusher.

In the combination of these two methods, the desorption rate shall reach at a relatively low rate even if the time is required timely, the gas lost because of samples removed from desorption container to crusher could be minimized.

5.1.1.4 Sampling for weighing, crushing and proximate analysis

Open the container when desorption terminates and conduct description of coal including fracture and cleat density, spacing, orientation partings and macroscopic, petrographic description etc. Take photos of the samples with well No, depth, seam No, etc.

Crush the sample into 2 cm to 3 cm blocks and put 300g to 500g of the blocks into ball milling pot for residual gas measurement. Alternatively, samples could also be divided into three parts (50g-100g for each part), then placed separately into ball milling pot for residual gas measurement. Take the rest of the samples for as- received moisture test, proximate analysis .

5.1.2 Measurement of residual gas

5.1.2.1 Measurement methods

One can heat the samples in the desorption container after 8 h of measurement and the samples in the ball milling pot in residual gas measurement process, so that the gas desorption rate is accelerated.

Note that the heating temperature should not exceed the maturity temperature.

Crush for 2 h to 4 h in the ball mill. Then, place it in thermostat till reaching reservoir temperature. Record the gas volume, ambient temperature, ambient pressure and desorption time in Annex B. Note that the reading interval should be intense at early desorption stage and should not exceed 24 h in the late stage.

The crushing time can be reduced when using multiple small samples.

5.1.2.2 Termination restriction for residual gas measurement

See 5.1.1.3

5.1.2.3 Weighing and calculating

When the measurement of residual gas is complete, open the mill pot and sieve the samples with a 60-mesh sieve. Weigh the sample and calculate the residual gas content.

5.1.3 Gas sampling, composition analysis and isotope analysis

5.1.3.1 Gas capture

During the desorption measurement, representative gas samples shall be collected for gas compositions analysis. Typical equipment includes hose (3.9), gas-sample bottles (3.10) (minimum 100 cm^3 of glass bottles or gas bags) and water trough for gas capture.

The principle of gas sampling is that the gas is captured when a large volume of gas is desorbed.

For natural desorption, capture 3 gas samples, at 1st, 3rd and 5th day respectively.

For samples with low gas content, gas can be captured at early stage.

Water displacement method should be used for gas sampling when the gas volume is over the capacity of the burette:

a) Set the gas-sample bottle upside down in water and fill it fully with water.

b) Close the connection between the burette and desorption canister.

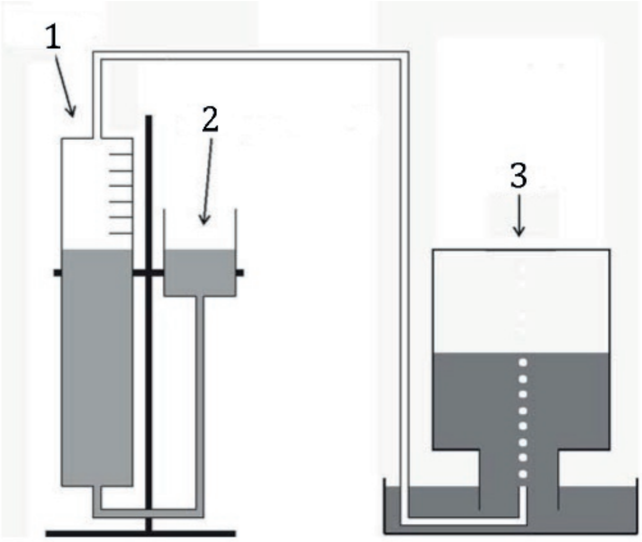
c) Lift the storage reservoir and remove air in the hose.

d) Open the connection to the gas-sample bottle and make gas flow into it.

e) Remove the hose and insert the stopper into the bottle when the volume of gas in the bottle is sufficient for applied tests.

f) Open the connection between the burette and desorption canister.

Set the gas-sample bottle upside down in box and send it to laboratories for analysis.



Key

1 burette

2 storage reservoir

3 bottle

Figure 5 — Water displacement method for gas sampling

5.1.3.2 Number of gas samples and frequency of sampling

See 5.1.3.1.

5.1.3.3 Gas composition analysis and isotope analysis

The sampled gas should be transferred to the laboratory as quickly as possible. The gas composition analysis is conducted according to that of natural gas.

5.2 Use of cuttings samples or coal lump samples

5.2.1 Q2 measurement

The gas volume released into the desorption canister (Q2) shall be measured, immediately after the desorption canister has arrived in the laboratory, but not more than 72 h after sampling (see Figure 1).

a) The desorption canister is connected via the hose to the gas analyser.

b) By squeezing the desorption canister, the gas shall be pressed into the gas analyser at a volume flow according to the gas analyzers specification.

c) The gas composition and the time of measurement shall be recorded.

d) After this, the desorption canister shall be opened and the coal sample shall be weighted with an accuracy of ± 0.2 g. The sample mass shall be recorded.

e) The calculation of the Q2 volume shall consider the volume of the desorption canister, the replacement of gas by the sample and drying agent mass and the pressure resulting from gas release into the sealed desorption canister.

f) Temperature and barometric pressure in the laboratory shall be measured and recorded.

5.2.2 Q3 measurement

The measurement of the gas volume being released during milling (Q3) shall be carried out immediately after the Q2 measurement.

a) The sample shall be placed into the crusher (3.8) without delay.

b) The crusher shall be closed immediately, and connected to the metering device via flexible hose.

c) In case of using the water displacement method, the Q3 volume shall be determined after crushing by direct readings from the measuring cylinder. While doing the reading, the water level within the measuring cylinder shall be equal to the water level in the storage reservoir.

In case of using the flushing method, the gas composition within the gas collecting vessel shall be measured by the analyser.

It shall be ensured that gas release has stopped before completing the crushing process.

d) Temperature and barometric pressure in the laboratory shall be measured.

6 Data processing

6.1 Conversion of the desorbed gas volume

The volume of measured desorbed gas and residual gas shall be converted into standard state which shall be at 0°C (273, 15 K) and 100 000 Pa. The formula of gas volume conversion is as Formula (1):

$$V_{\mathrm{STP}} = \frac{273{,}15 \times P_{\mathrm{m}} \times V_{\mathrm{m}}}{100\,000 \times (273{,}15 + T_{\mathrm{m}})} \quad (1)$$

where

V_{STP}	is the gas volume under standard state;
P_{m}	is the atmospheric pressure, expressed in Pa;
T_{m}	is the temperature, expressed in °C;
V_{m}	is the gas volume, expressed in Pa.

6.2 Reporting of gas content

The gas content may be reported in three ways:

a) raw coal basis;

b) air-dried basis;

c) dry ash-free basis.

By the measurement of total moisture and proximate analysis of the samples, the gas content of the samples may be converted into the expressions of raw coal basis, air-dried basis and dry ash-free basis as required.

6.3 Calculation of gas composition

The concentration of various gas components shall be calculated according to the natural gas industry.

The gas analyser shall be equipped with sensors for the main components of desorbed gas, at least with a sensor for methane, preferable an infrared sensor. The accuracy of the analyser shall be ± 5 % of the measured value or better.

6.4 Calculation of gas content

6.4.1 General

According to the measurement procedure, gas content comprises of the lost gas (2.3), measurable gas (2.4) and residual gas (2.5).

6.4.2 Calculation of lost gas content (Q1)

6.4.2.1 Use of core samples

If the drilling fluid is water and mud, time zero is the time at which the core is halfway to the surface; if the drilling fluid is foam or air, time zero is the time at which the drill reaches the coal seam.

The lost time is the time from zero-time to the time of sealing.

Its formula is as follows:

For the situation that the circulatory mediator of drilling is water and mud, the formula is as Formula (2):

$$t_{\mathrm{LOST}} = \frac{t_3 - t_2}{2} + (t_4 - t_3) \tag{2}$$

For the situation that the circulatory mediator of drilling is foam or air, the formula is as Formula (3):

$$t_{\mathrm{LOST}} = (t_4 - t_1) \tag{3}$$

where

t_{LOST}	is the lost time, expressed in s;
t_1	is the time of drill reaching coal seam, expressed in s;
t_2	is the time of pulling core, expressed in s;
t_3	is the time of core arriving wellhead, expressed in s;
t_4	is the time of sealing container, expressed in s;

The direct method is used for Calculation of lost gas volume. At the beginning of the desorption, desorption volume is proportional to the square root of time. Plot the cumulative readings of the initial desorption volume against the square root of elapsed time. Select the first linear protion of the curve and extrapolate it to zero-time. The intercept is taken as the volume of lost gas. See Annex C (direct method).

Lost gas content is calculated as given by Formula (4):

$$G_{\mathrm{CL}} = V_{\mathrm{LOST}} / m_{\mathrm{T}} \tag{4}$$

where

G_{CL}	is the lost gas content, expressed in cm^3/g;
$\mathrm{V}_{\mathrm{LOST}}$	is the volume of lost gas, expressed in cm^3;
m_{T}	is the dry weight (or as received weight) of the sample received, expressed in g.

6.4.2.2 Use of cutting samples or coal lump samples

The gas volume lost during sampling shall be determined by a traceable procedure. This calculation, shall consider the desorption rate being associated with the specific grain size of the sample and the sampling time. The procedure of the Q1 determination shall be part of the documentation.

The calculation may be executed by following Formula (5):

$$Q_1 = Q_2 \cdot \frac{t_a^{(1-k_t)}}{t_b^{(1-k_t)} - t_a^{(1-k_t)}} \tag{5}$$

where

Q_1 is the volume of gas released during sampling (lost gas) in cm^3;

Q_2 volume of gas released between sealing the desorption canister and executing Q2 measurement in cm^3;

t_a is the sampling time in min;

t_b is the time between sealing the desorption canister and executing Q2 measurement in min;

k_t is the measure of the change in desorption rate of a coal sample;

$$k_t = \frac{\ln \dot{V}_1 - \ln \dot{V}_2}{\ln t_1 - \ln t_2} \tag{6}$$

where

$\dot{V}_1$ is the volume of gas desorbed at time t_1;

$\dot{V}_2$ is the volume of gas desorbed at time t_2.

k_t can be set at 0,65 in case of normal coal with a grain size of > 2mm coal.

6.4.3 Calculation of gas desorption content (Q2)

Q2 is calculated as follows:

a) Calculated the open space volume of the desorption canister (consider the replacement of gas by the sample and drying agent mass and the pressure resulting from gas release into the sealed desorption canister).

b) Calculate Q2 volume based on open space volume of the desorption canister and the methane concentration reading.

c) Convert the Q2 volume to standard conditions (0°C and 0.1MPa).

d) Divide Q2 volume by the sample mass.

Gas desorption content is calculated as Formula (7):

$$G_{\mathrm{CD}} = V_{\mathrm{D}} / m_{\mathrm{T}} \tag{7}$$

where

G_{CD} is the content of natural gas desorption, expressed in cm^3/g;

V_{D} is the volume of natural gas desorption, expressed in cm^3;

m_{T} is the total weight of the sample, expressed in g.

6.4.4 Calculation of residual gas content (Q3)

Convert the Q3 volume to standard conditions (0°C and 0,1MPa) and divide it by the sample mass.

Residual gas content is calculated as Formula (8):

$$G_{\mathrm{CR}} = V_{\mathrm{R}} / m_{\mathrm{T}} \tag{8}$$

Where

G_{CD}	is the content of residual gas, expressed in cm^3/g;
V_{D}	is the volume of residual gas, expressed in cm^3;
m_{T}	is the total weight of the sample, expressed in g.

6.4.5 Calculation of free gas content

The porosity and the water saturation shall be measured to calculate the free gas content for the low rank coal samples because of the high content of free gas. The calculation of low rank samples shall add the measurement of free gas, shown as Formula (9) and Formula (10):

$$Q_{\mathrm{FG}} = \frac{\phi \times (1 - S_{\mathrm{w}})}{\rho \times B} \tag{9}$$

$$B = 3{,}458 \times 10^{-4} \times Z \times \frac{273{,}15 + T}{p} \tag{10}$$

Where

Q_{FG}	is the free gas content at reference temperature (T_{std}) and pressure P_{std}, the unit is cubic meters of gas per kilogram of dry coal;
ϕ	is the core porosity, the unit is percentage;
S_{w}	is the water saturation,the unit is percentage;
ρ	is the apparent relative density of dry coal, the unit is kilogram per cubic meter;
p	is the reservoir pressure, the unit is MPa;
B	is the volume factor V/V_{std}, the unit is $\mathrm{m}^3/\mathrm{m}^3$;
Z	is the gas compressibility factor, dimensionless;
T	is the reservoir temperature, the unit is degrees Celsius (°C).

Total porosity, water saturation determination of rock according to API RP40:1998 " Recommended Practices for Core Analysis", the apparent relative density of coal is measured according to ISO 5072:2013 Brown coals and lignites — Determination of true relative density and apparent relative density.

6.4.6 Calculation of gas content

The coalbed methane gas content (G_{C}) is equal to the sum of lost gas (G_{CL}, Q1), the measurable gas (G_{CD}, Q2) and the residual gas (G_{CR}, Q3). The coalbed methane content of low rank coal should include the free gas.

$$G_{\mathrm{C}} = G_{\mathrm{CL}} + G_{\mathrm{CD}} + G_{\mathrm{CR}} \tag{11}$$

6.4.7 Calculation of adsorption time

The adsorption time (T) refers to the time that used to desorb 63,2 % of the gas in the sample. Generally, use day as the unit of count. The adsorption time should be calculated in graphic method or calculation method.

First, calculate the gas volume that accounts 63,2 % of the total volume of the gas on the basis of the Formula (12).

$$V_{63,2\,\%} = V_{\mathrm{T}} \times 63,2\,\% \tag{12}$$

where

$V_{63,2\,\%}$ is the volume that accounts 63,2 % of the total volume of the gas, expressed in cm^3;

V_{T} is the sum of lost gas (2.3), measurable gas (2.4) and residual gas (2.5) expressed in cm^3.

Second, calculate the time based on the measured data points and the cumulative gas volume of each data point (at reference temperature and pressure).

Third, find out the range of $\mathrm{V}_{63,2\,\%}$ from the cumulative gas volume data.

At last, calculate the time (T) that $\mathrm{V}_{63,2\,\%}$ corresponding in the method of linear interpolation.

The time on the way should be deducted from the cumulative time when the environmental temperature is lower than the reservoir temperature during the sample transportation. Also the adsorption time does not include shipping time, otherwise the time is depending on the desorption curve.

6.5 Numerical accuracy requirements

The gas content database of coalbed methane rounds to two decimal places.

7 Measurement report

Measurement report of coalbed methane content is as follows:

Original data of gas natural desorption (Annex A) and original data of residual gas desorption (Annex B), lost gas volume calculation (Annex C), cumulative gas desorption curve (Annex D) and measurement results of coal core gas content (Annex E) as well as the gas composition results and proximate analysis results. Sampling data (Annex F or G), original data of gas desorption (Annex H) and results of gas content test (Annex I)”

8 Quality commentaries

Detailed records on sampling, measurement and equipment failure should be made in implementation process, which is a basis of the quality evaluation of measurement results; the quality evaluation includes the followings:

8.1 Quality commentary of sample collection

8.1.1 Situations should be noted on the record

The following two situations should be noted on the record:

a) the core cannot be raised in time

b) the sample has not been sealed within 10 min after reaching the surface

When coal core recovery rate is too low or the sample is relatively small, the actual sample weight should be recorded. The relevant reasons why the corresponding measurement cannot be done should be contained in the quality commentary.

8.2 Quality commentary on measuring operation

The following two situations should be noted in the record:

a) The sample is too fragmented,

b) Blockage occurred with resulted in delay of the measurement process.

The results could be used as reference only.

In measuring process, mistakes, omission or inaccurate results due to equipment leakage should be indicated.

8.3 Conclusion of quality commentary

8.3.1 Qualified samples

If problems mentioned in 8.1 and 8.2 do not occur, the samples shall be defined as qualified samples and the measurements shall be applied directly.

8.3.2 Reference samples

If problems mentioned in 8.1 and 8.2 do not occur, the samples shall be defined as qualified samples and the measurements shall be applied directly.

8.3.3 Discard

If any serious mistakes occur, the sample should be discarded and the measurements are invalid.

Annex A
(normative)

Raw data of gas natural desorption

area: well name:

coal seam number: sample number:

depth/m reservoir temperature/°C

time of drilling to coal bed upgrade core time

up to ground time sealing time

lost time during up/min lost time during sampling/min

No. of container date

weight of container/g weight of sample and container/g

Date [yy.mm.dd]	**Time** [hh:mm]	**Interval** [min]	**Measure** [cm^3]		**Gas volume** [cm^3]	**Ambient temperature** [°C]	**Atm** [kPa]	**Remark**
			begin	**end**				
Sampling people:		Experimental people:			Reviewer:		Page/Total Page	

Annex B
(normative)

Lost gas original records table

number of sample number of well number of coalbed

reservoir temperature/°C weight of sample date

Date [yy.mm.dd]	**Time** [hh:mm]	**Interval** [min]	**Measure** [cm^3]		**Gas volume** [cm^3]	**Ambient temperature** [°C]	**Atm** [kPa]	**Remark**
			begin	**end**				
Experimental people:			Reviewer:				Page/Total Page	

Annex C
(normative)

Lost gas volume calculation of Sample XX (direct method)

Lost gas volume calculation figure (direct method) is shown in figure c.1.

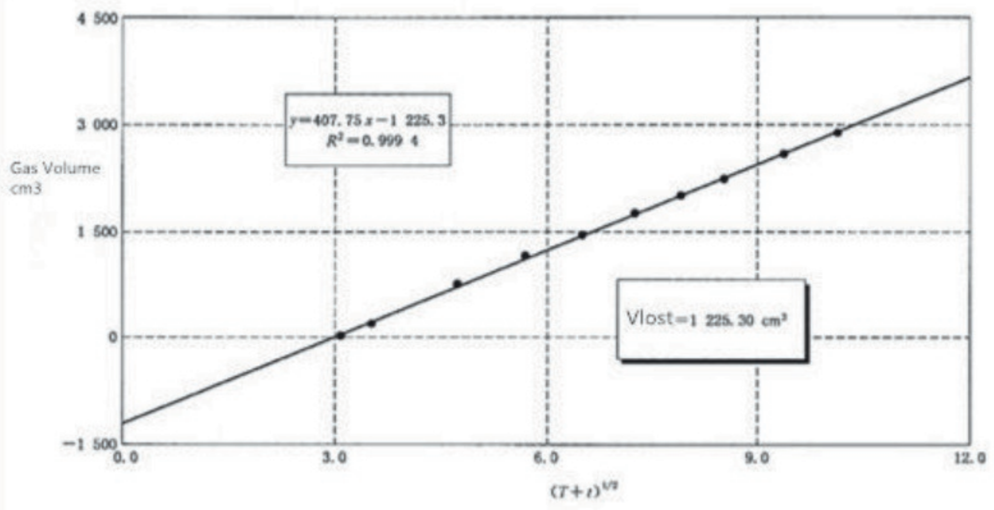


Figure C.1 — Well XX seam XX Lost gas volume calculation figure

Annex D
(normative)

Cumulative gas desorption curve of sample XX

Cumulative gas desorption curve of sample XX is shown in Figure D.1.

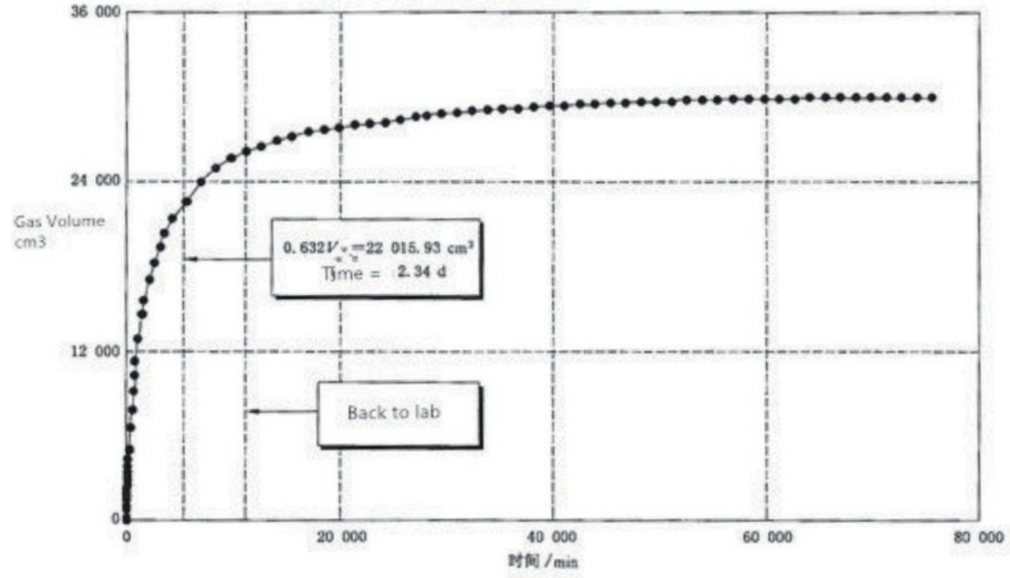


Figure D.1 — Cumulative gas desorption curve of sample XX

Annex E
(normative)

Measurement results table of CBM content

Table E.1 — Measurement results table of CBM content

Coal seam No	Sample ID	Sample weight [g]	Desorbed gas	Lost gas		Residual gas		Total gas content/ ($cm^3 \cdot g^{-1}$)		Moisture [%]	Ash content of air-dried basis [%]
			Gas volume [cm^3]	Lost time [min]	Lost volume [cm^3]	Residual sample mass [g]	Residual gas volume [cm]	Raw coal basis [$cm^3 \cdot g^{-1}$]	Dry ash-free basis [$cm^3 \cdot g^{-1}$]		
	Experimented by:			Reviewed by:						Page/Total Pages	

Annex F
(normative)

Sampling form for in-seam drilling

Mine:

Coal seam:

Sampling location:

Borehole:

Coordinates: x-value y-value

Altitude: m above sea level

Date: Sampler:

Air temperature: °C Barometric pressure: kPa

CH_4 concentration of ventilation air: %

Date and time of initial exposure of coal face: Date Time

Sample no.	**Borehole depth (m)**	**Sample canister no.**	**Time of encountering sample**	**Time of sealing sample in canister**
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				

Annex G
(normative)

Sampling form for core drilling

Sample container No.:

Borehole (well name):

Sampling location:

Coordinates: x-value y-value

Altitude: m above sea level

Date: Sampler:

Air temperature: °C Barometric pressure: kPa

Core No.: Coal seam:

Depth of core sample: from m to m

Distance of sample from top of core:

Length of core sample: cm

Drilling time of core: from to

Closing time of canister:

Annex H
(normative)

Laboratory test form

Q_1

Sample no.	Date	Time of drilling the sample	Time of sealing the bottle	Pressure at sampling location P_V	Temperature at sampling location T_V	Methane concentration at sampling location c_V	Grain size
	TT.MM.JJ	hh:mm	hh:mm	[kPa]	[°C]	[%]	[mm]
1							
2							
3							
4							
5							

Q_2

Sample no	Date	Time	Pressure at laboratory P_V	Temperature at laboratory T_V	Methane concentration in canister c_{Fl}
	TT.MM.JJ	HH:MM	[kPa]	[°C]	[%]
1					
2					
3					
4					
5					

Q_3

Sample no.	Pressure at laboratory P_V	Temperature at laboratory T_V	Coal mass m	Gas volume released while crushing Q_3
	[kPa]	[°C]	[g]	[cm^3]
1				
2				
3				
4				
5				

Annex I
(normative)

Short report

Mine:	**Coordinates:**	X	
Seam:		Y	
Sampling location:		Z	[m a.s.l.]
	Volatile matter:		% waf
		Conditions:	101,325 kPa; 0°C

Canister no.	Borehole depth	Grain size	Raw coal mass	Ash content	Mass ash free	q_1 fraction	q_2 fraction	q_3 fraction	Total gas content
	[m]	[mm]	[g]	[%]	[g]	[m^3/t]	[m^3/t]	[m^3/t]	[m^3/t]
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									

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