



Determination of gas content of coal and carbonaceous material—Direct desorption method



This Australian Standard® was prepared by Committee MN-001, Coal and Coke. It was approved on behalf of the Council of Standards Australia on 4 November 2016. This Standard was published on 28 November 2016.

The following are represented on Committee MN-001:

- Australasian Institute of Mining and Metallurgy
 - Australian Coal Industry Reference Samples
 - Australian Coal Preparation Society
 - Australian Energy Council
 - Coalfield Geology Council of NSW
 - CSIRO
 - Department of Natural Resources and Mines, Qld
 - Minerals Council of Australia
 - National Association of Testing Authorities Australia
 - University of Newcastle
 - University of New South Wales
 - University of Queensland
-

This Standard was issued in draft form for comment as DR AS 3980:2016.

Standards Australia wishes to acknowledge the participation of the expert individuals that contributed to the development of this Standard through their representation on the Committee and through the public comment period.

Keeping Standards up-to-date

Australian Standards® are living documents that reflect progress in science, technology and systems. To maintain their currency, all Standards are periodically reviewed, and new editions are published. Between editions, amendments may be issued.

Standards may also be withdrawn. It is important that readers assure themselves they are using a current Standard, which should include any amendments that may have been published since the Standard was published.

Detailed information about Australian Standards, drafts, amendments and new projects can be found by visiting **www.standards.org.au**

Standards Australia welcomes suggestions for improvements, and encourages readers to notify us immediately of any apparent inaccuracies or ambiguities. Contact us via email at **mail@standards.org.au**, or write to Standards Australia, GPO Box 476, Sydney, NSW 2001.

Australian Standard[®]

Determination of gas content of coal and carbonaceous material—Direct desorption method

Originated as AS 3980—1991.
Previous edition 1999.
Third edition AS 3980:2016.

COPYRIGHT

© Standards Australia Limited

All rights are reserved. No part of this work may be reproduced or copied in any form or by any means, electronic or mechanical, including photocopying, without the written permission of the publisher, unless otherwise permitted under the Copyright Act 1968.

Published by SAI Global Limited under licence from Standards Australia Limited, GPO Box 476, Sydney, NSW 2001, Australia

ISBN 978 1 76035 604 0

PREFACE

This Standard was prepared by the Standards Australia Committee MN-001, Coal and Coke, to supersede AS 3980—1999, *Guide to the determination of gas content of coal seams—Direct desorption method*.

The objective of this Standard is to provide procedures for the determination of the desorbed gas content of coal and carbonaceous material by directly measuring the volume of gas, using the water displacement method, from material obtained by coring or collection of lump samples.

The major changes in this edition are to—

- (a) change from a guidance document to a Standard;
- (b) refine reporting requirements;
- (c) addition of remaining gas, Q_3' , and change the Q_3 method to extend the measurement time to reach equilibrium; and
- (d) inclusion of generic gas content uncertainty calculation.

The terms ‘normative’ and ‘informative’ have been used in this Standard to define the application of the appendix to which they apply. A ‘normative’ appendix is an integral part of a Standard, whereas an ‘informative’ appendix is only for information and guidance.

CONTENTS

	<i>Page</i>
1 SCOPE	4
2 REFERENCED DOCUMENTS	4
3 DEFINITIONS	5
4 PRINCIPLE	6
5 APPARATUS	7
6 PREPARATION FOR FIELD TESTING.....	9
7 SAMPLING	10
8 MEASURING GAS QUANTITIES—SLOW DESORPTION METHOD.....	11
9 MEASURING GAS QUANTITIES—FAST DESORPTION METHOD	14
10 MEASURING GAS COMPOSITION.....	14
11 CALCULATIONS	14
12 DATA RETENTION.....	16
13 TEST REPORT	16
APPENDICES	
A FACTORS AFFECTING PRECISION AND ACCURACY	18
B CALCULATION OF DESORBED GAS VOLUME (Q_2 AND Q_3 AT STANDARD PRESSURE AND TEMPERATURE CONDITIONS).....	21
C DETERMINATION OF Q_3'	25
D ESTIMATION OF LOST GAS.....	27
E CALCULATION OF 'AS-RECEIVED' GAS COMPOSITION ON AN AIR-FREE BASIS.....	33

STANDARDS AUSTRALIA

Australian Standard**Determination of gas content of coal and carbonaceous material—Direct desorption method****1 SCOPE**

This Standard covers the following two direct methods, which vary only in the time allowed for the gas to desorb from the intact sample prior to final crushing:

- (a) The slow desorption method.
- (b) The fast desorption method.

The Standard is confined to the direct method using lump or core samples.

Desorption limitations to the method are specified in Appendix A.

This Standard does not include the gas content determination using cuttings from drilling and interpretation of the results do not fall within the scope of this document.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard.

AS

- 1038 Coal and coke—Analysis and testing
- 1038.3 Part 3: Proximate analysis of higher rank coal
- 1038.21.2 Part 21.2: Higher rank coal and coke—Relative density—Lump sample
- 2163 Laboratory glassware—Measuring cylinders
- 2418 Coal and coke—Glossary of terms

ISO/IEC

- GUIDE 98-3 Uncertainty of measurement—Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)

Eurolab 1/2007

Technical Report No. 1/2007 March 2007, Measurement uncertainty revisited: Alternative approaches to uncertainty evaluation

Nordtest TR537

Handbook for calculation of measurement uncertainty in environmental laboratories (NT TR 537—Edition 3.1)

NATA Technical Note 33

Guidelines for estimating and reporting measurement uncertainty of chemical test results, February 2016

3 DEFINITIONS

For the purpose of this Standard, the definitions given in AS 2418 and those below apply.

3.1 Desorption methods

3.1.1 Fast desorption method

A method of gas content determination in which the trend of the gas desorbing from the sample is not fully defined. This method only differs from the slow desorption method in the length of time Q_2 is measured and will result in differing values for Q_2 and Q_3 dependant on the length of Q_2 time in comparison to the slow desorption method.

3.1.2 Slow desorption method

The method of gas content determination in which the trend of the gas desorbing from the sample is defined by measurements taken over a period of time. Until the rate of gas evolution approaches an asymptotic value, <1% of total gas make achieved in the test over 5 consecutive days.

3.2 Direct method

Determination of the gas content of coal and carbonaceous material by directly measuring the volume of gas desorbed, using the water displacement method.

3.3 Measured gas content (Q_m)

The sum of Q_1 , Q_2 and Q_3 .

3.4 Quantity of gas

Gas, expressed as volumes of gas per unit mass of sample, as defined in Clauses 3.4.1 to 3.4.4 and illustrated in Figure 3.1, and determined at a standard temperature of 20°C and a pressure of one atmosphere (1 atmosphere = 101.325 kPa).

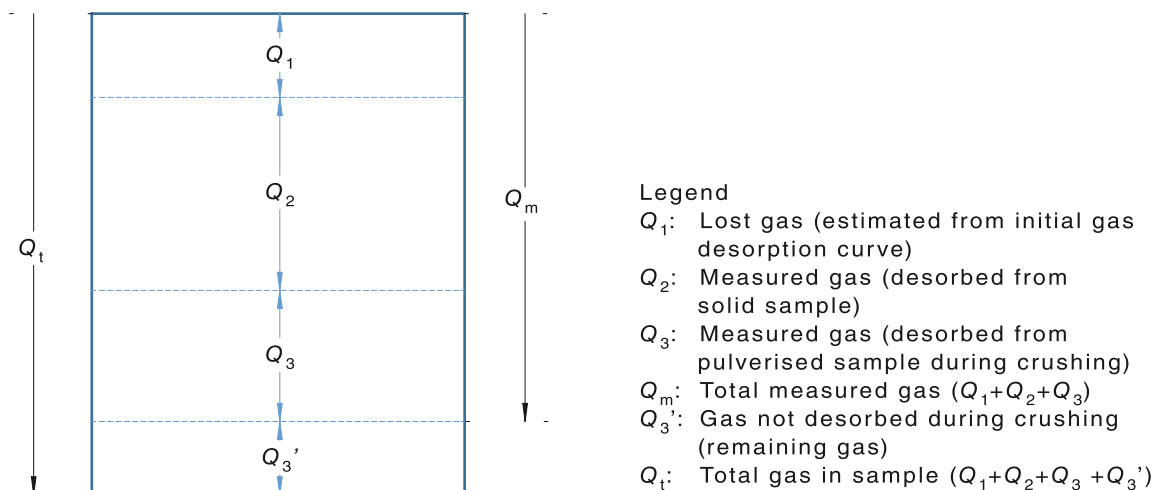


FIGURE 3.1 GAS RELEASED FROM COAL AT DIFFERENT STAGES OF GAS CONTENT TESTING (NOT TO SCALE)

3.4.1 Q_1 (lost gas)

Gas lost from the sample subsequent to its being removed from its in situ position and prior to the start of volume measurement; expressed as the volume of gas per unit mass of sample.

NOTE: Q_1 is estimated from initial gas desorption curve.

3.4.2 Q_2 (measured desorbed gas from solid sample)

Gas desorbed at near atmospheric pressure from the non-pulverized coal sample; expressed as the volume of gas per unit mass of sample.

3.4.3 Q_3 (measured desorbed gas from pulverized sample)

Gas released by crushing sample to a fine powder, with gas desorption ceasing under the equilibrium conditions in the crushing vessel; expressed as the volume of gas per unit mass of sample.

3.4.4 Q_3' (remaining gas)

Gas not released from sample during crushing; expressed as the volume of gas per unit mass of sample, and measured at zero partial pressure of the seam gas in contact with the sample.

3.5 Sample

Coal or carbonaceous material in the form of core or lump sample.

3.6 Times**3.6.1 t_e (elapsed time)**

Duration, in minutes, between zero time and time on test, i.e. t_0 to t_1 .

3.6.2 t_1 (time on test)

That point in time at which the canister is connected to the measuring apparatus.

3.6.3 t_0 (zero time)

Zero time (t_0) is defined as the point in time when the sample starts desorbing its gas.

For surface drilled core, the midway point in time between pulling the core from the bottom of the borehole to when it reaches the surface.

For in-seam drilled core, the midway point in time between the beginning of coring and the end of coring.

3.7 Total gas content (Q_t)

The sum of Q_m and Q_3' .

4 PRINCIPLE

The strata is sampled by coring or lump sampling, the sample is then placed in a canister and put on test with minimum delay. The initial desorption rate is measured and used for the calculation of Q_1 .

The total quantity of gas evolved from the canister is measured volumetrically to determine Q_2 .

Subsamples are then taken from the canister and crushed. The quantity of gas evolved by crushing is measured to determine Q_3 .

The measured gas content (Q_m) is then calculated.

Q_3' may be determined by calculation or direct measurement.

Total adsorbed gas content (Q_t) is then calculated.

5 APPARATUS

NOTE: This apparatus is applicable for either core or lump samples.

5.1 Canister

A canister to contain the sample during transport and desorption is required.

The canister shall be designed to withstand potential pressures created by the desorbing gas.

The length and diameter of the canister shall be sufficient to accept the bore core or lump samples, with minimal air space to reduce the effect of changes in atmospheric conditions on the gas volumes measured. Splits and unreactive packing material may be used for ease of handling, and to protect the bore core from damage during transport.

The canister shall be sealed at one end and the other end shall have a removable gastight cap fitted with bi-directional, gastight valve. The cap is sealed by an O-ring or proprietary snap-lock coupling.

5.2 Splits

Splits are separators used to preserve the coal core in its drilled condition until the end of the test.

Where using splits, care shall be taken to ensure that securing is minimal and done in a way that does not allow the sample to become jammed in the canister at the time of removal, and so that it is unlikely that the outlet valve can be blocked by tape. Splits shall be secured at both ends and the middle to prevent expansion inside the canister.

The use of splits is not mandatory but, use is recommended where it is required to keep the core intact.

5.3 Volume measurement apparatus

5.3.1 General

The measurement of desorbed gas volumes is carried out by the water displacement method in the field and subsequently in the laboratory. The field measurement is directed towards determining the lost gas and the first portion of Q_2 in desorption is used.

5.3.2 Measurement system

The following items of equipment are required:

- (a) *Measuring cylinder* Graduated, glass or transparent plastics, of capacity appropriate to the desorption rate.

NOTE: Cylinders should be calibrated. AS 2163 provides tolerances for cylinders.

- (b) *Water reservoir* Size appropriate to the cylinder used.

NOTE: The pan may need an over-flow at a sufficient height above the outlet level and the water level to be measured to correct for hydrostatic head unless a method/device is installed to provide a constant water level.

- (c) *Tubing* Of plastic or rubber (preferably clear), flexible, durable, gastight and free of restricting bends and water traps. The tube is introduced into the measuring cylinders from the top (see Figure 5.1).

NOTE: Clear silicone rubber is favoured to allow easy detection of water traps and for minimization of maintenance. The volume of the tubing should be minimized.

5.3.3 Assembling

Assembly shall be as follows:

- (a) The cylinder, secured to a stand, is filled with water (see Figure 5.1) by drawing water into it by applying suction to the tube passing through the top of the cylinder.
- (b) The pan shall be of such a size and shall contain sufficient water so that any drawback of water that may occur as a result of a fall in temperature or loss of water due to evaporation will not expose the lower end of the cylinder.
- (c) Lead the desorbed gas to the cylinder through the tubing. A gas sampling tube may be located in the delivery line (see Note 1).

NOTES:

- 1 The gas over a suspended liquid column has a lower pressure than the surrounding atmosphere and thus a greater volume. The pressure due to water column height will be included in the calculation of the system pressure. Appendix B describes the procedure for correcting the measured gas volume to 20°C at 101.325 kPa.
- 2 Alternatively, a sealable opening at the top of a specially made cylinder will assist in gas sampling and in refilling the cylinder by vacuum, as shown in Figure 5.1.
- 3 This apparatus can be used for prolonged periods of degassing without supervision by connecting a number of cylinders in parallel but preventative measures for CO₂ dissolution should be in place to use this method.

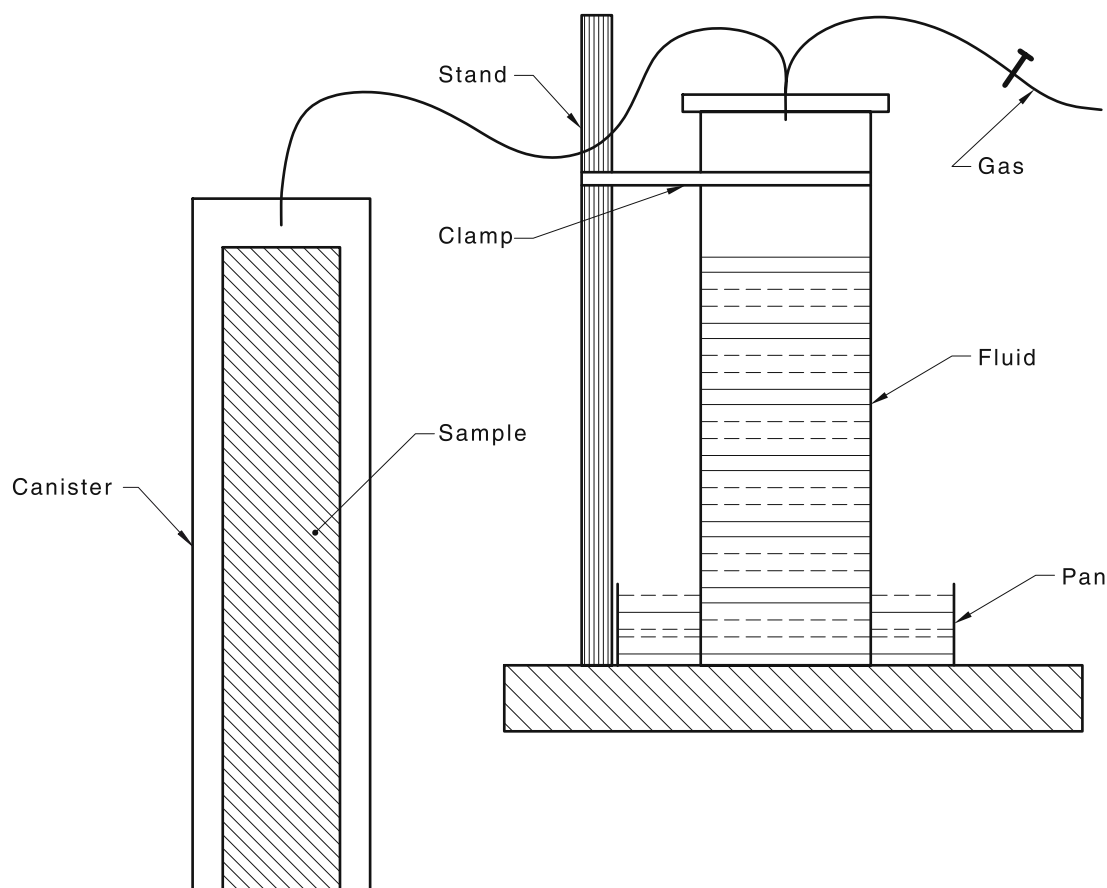


FIGURE 5.1 VOLUME MEASUREMENT APPARATUS—TUBE ENTERING AT TOP

5.4 Gas sampling apparatus

Both gas-barrier plastics bags and in-line gas sampling tubes are suitable. In-line sampling tubes placed between the canister and the measuring apparatus minimize the risk of composition change in the measured gas caused by solution in the measuring fluid.

NOTE: The sample size is determined by the method of gas analysis.

5.5 Temperature control of canisters

Canisters shall be managed in such a way to avoid exposure to excessive temperature swings caused by direct exposure to sun light and cold morning temperatures.

If specified by the end user of the data, a temperature controlled water bath may be used (or any other suitable temperature control method) to maintain the canisters at a consistent temperature. This method is acceptable if the desorption curve shape at reservoir temperature is required for reservoir modelling.

5.6 Coal crusher

A crusher is required to pulverize the coal sub-sample into fine powder to release the remaining gas rapidly.

The crusher shall be capable of pulverizing the sample to 95% of material passing a 212 μm mesh.

Typical operating laboratories use a sample mass-to-crusher volume ratio within the range 1:3 to 1:7. The ratio shall be kept constant within each laboratory.

The crusher shall have the capability to release the gas either during or after crushing.

5.7 Weighing device

A weighing device capable of measuring the sample mass to an accuracy better than 1% is required.

5.8 Miscellaneous equipment

A calibrated timer, thermometer and barometer are required.

6 PREPARATION FOR FIELD TESTING

Prior to taking samples, the field equipment shall be on site and set up for immediate use. The canisters shall be cleaned, tested for leaks at positive and/or negative pressure sufficient to guarantee an effective seal but below the design pressure of the canisters. Care shall be taken to ensure that the testing pressure does not exceed the design capability. The canisters shall be weighed (including cap and splits) and labelled prior to use.

At the field site, the canisters and system shall be re-tested by positive and/or negative for leaks immediately prior to use.

NOTE: The majority of leaks in the field are caused by particles from the sample contaminating the canister seal or where fittings on canister lid have been dislodged during transport.

For surface drilling, the equipment shall be located in an area free from temperature extremes and in a safe location near the drilling site. It is essential that the drillers are advised of the testing; understand their involvement in the testing process and that Q_1 time is kept to a minimum.

For underground work, the test apparatus shall be set up in a safe location near the sampling site, away from the general traffic flow, in accordance with site safety regulations. It is essential that the drillers/machine operators be advised of the testing, and understand their involvement in the testing process.

The following conditions shall be adhered to:

- (a) *For surface drilling:*
 - (i) The driller shall notify the sampler when coring of the test seam starts and stops, and when retrieval of the core commences.
 - (ii) The core shall be recovered to the surface in minimal time and initial desorption measurements are to commence without delay.
 - (iii) The core barrel shall be pumped out or opened before further drilling operation is commenced.
 - (iv) Geological logging and photography shall be avoided or kept to a minimum to avoid increasing uncertainty in the Q_1 estimation.
- (b) *For underground in-seam coring:*
 - (i) The driller shall notify the sampler when coring of the sample run starts and finishes. These times shall be recorded.
 - (ii) The core shall be retrieved as quickly as possible and initial desorption measurements shall commence without delay.
- (c) *For underground working face:*
 - (i) Samples shall be collected in a safe manner at the discharge of continuous miner or fresh from the longwall face. Ensure sample does not contain fines. Sample solid, intact material only.
 - (ii) All samples (core and lump) shall be retrieved as quickly as possible and initial desorption measurements shall commence without delay.

7 SAMPLING

7.1 General

The source of the sample is beyond the scope of this Standard; hence, this Clause is restricted to the preparation of the sample for testing. To minimize canister void space, it is important that the quantity of sample be suitable for the size of the canister or the void is filled with inert fillers if required. It is essential that sampling and containment of the sample be done quickly to reduce the lost gas quantity.

7.2 Surface bore cores

The sampling shall be carried out as follows:

- (a) Note the times when the bore core is pulled off the bottom of the hole and when it reaches the surface. The time to retrieve and place into a desorption canister shall be as quick as possible to minimize the uncertainty associated with the estimation of Q_1 .
- (b) When the core barrel is at the surface, immediately remove the upper split. Quickly measure the core recovery, if necessary, wash down the exposed core. Note the proportions of coal and non-coal material, and note the state of the core as well as any abnormalities that have occurred during the drilling to retrieval time.
- (c) Either—
 - (i) select the entire coal seam core in the driller's splits; or
 - (ii) select particular core sections, label the core log with the canister identification and transfer the sections to individual splits or directly to the canisters. Label the canister with the core identification code or number.

NOTE: If a significant proportion of the sample is highly fractured during drilling, or contains coal fines, erroneous readings may be obtained.

- (d) If splits are used, tape them together at the ends and in the middle of the splits to retain the bore core.
- (e) Insert the sample into the canister, ensure that the seal is clean and that the valve is open, close the cap, connect the valve outlet to the measuring cylinder, and record the time.

NOTES:

- 1 If the valve is closed before the canister is placed on test, pressure will build up within the canister. This will need to be taken into account in Q_1 calculations. Core samples should not be left sealed in canisters with valve shut for any extended period of time before Q_1 readings are commenced.
- 2 Canister purging is generally adopted in the field for removal of head space air present in the canister after sealing the core sample inside the canister. Various methods are practiced, such as filling with reservoir fluid, flushing with helium or argon etc., to minimize the head space air contamination as well as oxidation of coal during gas desorption measurements. The purging process adopted should be described in the report for comprehending the gas composition results. Refer to Figure 5.1 where two valves are required in the canister cap to allow for purging.
- 3 It is recommended that the orientation of the core is maintained within the canister by marking the top and bottom of the splits and using the convention (usually top of core to the canister cap) during transfer.

7.3 Underground bore cores

The procedure is similar to that for surface bore cores. In-seam borehole samples shall be numbered with respect to distance from the collar. Zero time (t_0) is defined as the point in time mid-way between the commencement and completion of coring.

7.4 Lump sampling

Samples shall be collected from the face immediately after coal cutting. Manual sampling at the working face is not recommended for safety reasons. The sample shall be sieved quickly to +50 mm nominal size, and obvious non-carbonaceous material removed before placing the sample into the canister. Close the cap with the valve open.

8 MEASURING GAS QUANTITIES—SLOW DESORPTION METHOD

8.1 Determination of Q_1

The following procedure for the determination of lost gas in bore core samples shall be carried out at the sampling site:

NOTE: Significant errors will occur in the determination of lost gas if the initial gas desorption is not measured within about 40 min of zero time. Include the elapsed time in the final report.

- (a) Immediately prior to sealing the sample in the canister, record the temperature and barometric pressure; at the moment of connection to the apparatus. Note the time (t_1) and the initial reading.
- (b) Measure the gas evolved at regular intervals for a sufficient time to allow accurate estimation of lost gas, typically over a 20 min to 60 min period. This period may be longer if required. However, the period shall be 20 min as a minimum. Readings may be taken on either a time basis or a volume basis. Time shall be recorded to the nearest second. A minimum of ten readings shall be taken in this period.
- (c) Do not refill the measuring cylinder or close the canister or cylinder valve during initial desorption measurement. If further Q_2 measurements are required close the valve each time the measuring cylinder needs to be refilled with liquid. The valve shall be closed for as short a time as possible.