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Standard Practice for
Determination of Gas Content of Coal—Direct Desorption Method[1]

This standard is issued under the fixed designation D7569/D7569M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε^1 NOTE—Designation was corrected editorially in February 2015.

1. Scope

1.1 This practice describes methods for the direct determination of the gas content of coal by desorption using samples obtained by drill coring methods from the surface. It sets out guidelines for the equipment construction, sample preparation and testing procedure, and method of calculation.

1.2 Indirect methods for the determination of the gas content of coal (not covered in this practice) are based on either the gas absorption characteristics of coal under a given pressure and temperature condition or other empirical data that relate the gas content of coal to such other parameters as coal rank, depth of cover, or gas emission rate.

1.3 This practice covers the following two direct methods, which vary only in the time allowed for the gas to desorb from the core, or sidewall core, before final crushing:

1.3.1 The slow desorption method in which volumetric readings of gas content are taken frequently (for example, every 10 to 15 min) during the first few hours, 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.

1.3.2 The fast desorption method in which after initial desorbed gas measurements to obtain data for lost gas calculations are taken, the canister is opened and the sample is transferred to the coal crusher. The remaining gas volume is measured on a crushed sample.

1.4 This practice is confined to the direct method using core, or sidewall core obtained from drilling. The practice can be applied to drill cuttings samples; however, the use of cuttings is not recommended because the results may be misleading and are difficult to compare to the results obtained from core desorption. The interpretation of the results does not fall within the scope of the practice.

1.5 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as the standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D121 Terminology of Coal and Coke
D167 Test Method for Apparent and True Specific Gravity and Porosity of Lump Coke
D1412 Test Method for Equilibrium Moisture of Coal at 96 to 97 Percent Relative Humidity and 30°C
D2799 Test Method for Microscopical Determination of the Maceral Composition of Coal
D3172 Practice for Proximate Analysis of Coal and Coke
D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke
D3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal
D3176 Practice for Ultimate Analysis of Coal and Coke
D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases
D3302 Test Method for Total Moisture in Coal
D5192 Practice for Collection of Coal Samples from Core
E1272 Specification for Laboratory Glass Graduated Cylinders

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[1] This practice is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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2.2 *Australian Standard:*

AS 3980 Guide to the determination of gas content of coal—Direct desorption method[3]

2.3 *ISO Standard:*

ISO 6706 Plastics laboratory ware—Graduated measuring cylinders[4]

2.4 *DIN Standard:*

DIN 12681 Plastics laboratory ware—Graduated measuring cylinders[5]

3. Terminology

3.1 *Definitions:*

3.1.1 For additional definitions of terms used in this practice, refer to Terminology D121.

3.1.2 *absolute permeability, n*—permeability of a rock to a particular fluid when the rock is 100 % saturated with the flowing fluid. **(1)**[6]

3.1.3 *absorbed gas, n*—gas that is molecularly dissolved within a liquid phase or has penetrated into the lattice structure of a solid. **(1)**

3.1.4 *actual lost gas time, n*—lost gas time determined from the time at which the sample being recovered reaches a depth where the hydrostatic pressure of the drilling fluid column equals the original (immediately before sampling) reservoir pressure in the sample to the time at which the sample is sealed in a desorption canister.

3.1.4.1 *Discussion*—Essentially, the actual lost gas time is the amount of time between when the core starts its trip to the surface and when it is sealed in the canister. **(1)**

3.1.5 *adsorption, n*—attachment, through physical or chemical bonding, of fluid phase molecules to an interfacial surface.

3.1.5.1 *Discussion*—The adsorbed phase molecules are sequestered at the interfacial surface in a metastable equilibrium state, the stability of which is strongly affected by changes in temperature and pressure. **(1)**

3.1.6 *adsorption isotherm, n*—quantitative relationship, at constant temperature, describing how the concentration of adsorbed phase molecules at an interfacial surface varies as a function of increasing system pressure. **(1)**

3.1.7 *as-received basis, n*—analytical data calculated to the moisture condition of the sample as it arrived at the laboratory and before any processing or conditioning.

3.1.7.1 *Discussion*—If the sample has been maintained in a sealed state so that there has been no gain or loss, the as-received basis is equivalent to the moisture basis as sampled. **(D3180, D5192, D1412, D3302)**

3.1.8 *canister, n*—container that can be sealed into which a coal sample is placed to allow desorption to occur.

3.1.8.1 *Discussion*—The reduction in pressure to atmospheric pressure (at surface) causes the sample to release gas into the canister. By measuring the amount of gas released and the weight of the sample, the gas content can be determined. Gas content is conventionally reported in units of cubic centimetres/gram (cm^3/g), cubic metres/tonne (m^3/ton), or standard cubic feet/ton (scf/ton). **(1)**

3.1.9 *continuous coring, v*—refers to continuous coring with wireline recovery. **(1)**

3.1.10 *conventional core, n*—"large" diameter core (8.9 cm [3.5 in.] diameter or larger) in which the core barrel is recovered to the surface after drilling a fixed interval by pulling the drill string. **(1)**

3.1.11 *core, n—in drilling*, a cylindrical section of rock (coal) that is usually 5 to 10 cm in diameter taken as part of the interval penetrated by a core bit and brought to the surface for geologic examination, representative sampling, and laboratory analyses. **(D121, D5192)**

3.1.12 *cuttings, n—in drilling*, rock fragments that break away because of the action of the drill bit and are transported to the surface by the drilling circulation system (mud or air).

3.1.12.1 *Discussion*—Cuttings may be screened and collected from the circulation medium for lithologic characterization or analytical tests. **(2)**

3.1.13 *delivery tube, n*—flexible tube connecting a desorption canister to a volumetric displacement apparatus. **(1)**

3.1.14 *desorption, n*—detachment of adsorbed molecules from an interfacial surface (see *adsorption*). **(1)**

3.1.15 *desorption data analysis software, n*—software used for analyzing desorption data. **(1)**

3.1.16 *desorption rate, n*—volumetric rate at which gas desorbs from a sample. **(1)**

3.1.17 *diffusion, n*—process whereby particles of liquids or gases move from a region of higher to lower concentration independent of the pressure gradient. **(1)**

3.1.18 *diffusivity, n*—ratio of the diffusion coefficient to the square of a typical diffusion distance. **(1)**

3.1.19 *direct desorption method, n*—method for representing desorption of gas from coal or other materials in which gas storage as a result of adsorption is significant.

3.1.19.1 *Discussion*—It mathematically presumes constant temperature diffusion from a sphere initially at uniform gas concentration. The solution of the basic equation adopted suggests that the measured desorbed gas volume is proportional to the square root of time since the start of desorption (time zero). The direct method is the most widely used method for estimating lost gas volume. **(1)**

3.1.20 *dry, ash-free basis, n*—data calculated to a theoretical base of no moisture or ash associated with the sample.

3.1.20.1 *Discussion*—Numerical values as established by Test Methods D3173 and D3174 are used for converting the as-determined data to a moisture- and ash-free basis. **(D3180)**

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[3] Available from Standards Australia Limited, 286 Sussex St., Sydney, NSW, 2000, Australia, GPO Box 476, Sydney, NSW, 2001 Australia or via the website: www.standards.org.au.

[4] Available from the International Organization for Standardization (ISO), 1, rue de Varembé, Case Postale 56, CH-1211 Geneva 20, Switzerland or via the website: http://www.iso.org/.

[5] Available from Deutsches Institut für Normung e. V., 10772 Berlin or via the website: http://www2.din.de.

[6] The boldface numbers in parentheses refer to the list of references at the end of this standard.

3.1.21 *fast desorption method, n*—after initial measurements to obtain the basis for lost gas calculations, the canister is opened and the sample is transferred to a coal-crushing device that is modified so that the remaining desorbed gas volume from the crushed coal sample can be measured. **(AS 3980)**

3.1.22 *free gas, n*—unabsorbed gas within the pores and natural fractures. **(1)**

3.1.23 *gas-in-place, n*—amount of gas present in a seam or an interval of discrete thickness or in multiple seams or intervals determined from the gas content, bulk density, thickness, and drainage area.

3.1.23.1 *Discussion*—Estimates of gas-in-place usually reflect total gas content, which in addition to methane, may include other gases such as carbon dioxide or nitrogen. **(1)**

3.1.24 *gas saturated, adv*—state in which the gas content (determined from direct or indirect desorption measurements) is equal to the gas storage capacity (determined from isotherms). **(1)**

3.1.25 *gas storage capacity, n*—maximum amount of gas or gas mixtures (normalized according to the relevant basis) that can be held by a sample at various reservoir pressures, reservoir temperature, and a specific moisture (water) content. **(1)**

3.1.26 *head space volume, n*—void space in a canister containing a sample.

3.1.26.1 *Discussion*—Canister desorption measurements are corrected for the effect of expansion or contraction of gases in the canister head space in response to temperature and pressure fluctuations. **(1)**

3.1.27 *indirect method for the determination of the gas content of coal, n*—method based on either the gas absorption characteristics of coal under a given pressure and temperature condition or other empirical data that relate the gas content of coal to such other parameters as coal rank, depth of cover, or gas emission rate.

3.1.28 *in-situ basis, adj*—a basis in which gas content is determined from a plot of gas content versus bulk density (determined from open-hole high-resolution bulk density log data). **(1)**

3.1.29 *isotherm (sorption isotherm), n*—quantitative relationship, at constant temperature, that describes how the concentration of adsorbed phase molecules at an interfacial surface varies as a function of system pressure. **(1)**

3.1.30 *lost gas time, n*—time between when the sample gas pressure falls below the reservoir pressure during sample recovery (time zero) and the time when the sample is sealed in a desorption canister. **(1)**

3.1.31 *lost gas volume, n*—volume of gas that is released from a sample (generally under conditions of decreasing temperature and pressure) before it can be placed in a canister and measured (between the time when the sample gas pressure falls below the reservoir pressure during sample recovery and the time when the sample is sealed in a desorption canister). **(1)**

3.1.32 *macropore, n*—pores in the coal larger than 50 nm. **(3)**

3.1.33 *manometer, n*—see *volumetric displacement apparatus*.

3.1.34 *measured gas volume, n*—measured volume of gas that is released from a sample into a desorption canister, reported at standard temperature and pressure conditions. **(1)**

3.1.35 *mesopores, n*—pores in the coal larger than 2 nm and less than 50 nm. **(3)**

3.1.36 *micropores, n*—pores with a width of less than 2 nm. **(3)**

3.1.37 *modified direct method, n*—modification of the direct method by the U.S. Bureau of Mines according to Diamond and Levine **(4)** and Diamond and Schatzel **(5)** to account precisely for changes in the concentration of gaseous species during desorption, with particular applications to situations in which small amounts of gas are evolved. **(1)**

3.1.38 *quick connect fittings, n*—pipe fittings designed for easy and rapid connection and disconnection.

3.1.39 *raw basis, n*—basis for gas content calculation whereby the gas volume is divided by the actual sample weight regardless of the moisture content or the presence of non-coal in the canister sample. **(1)**

3.1.40 *residual gas volume, n*—volume of the total sorbed gas that remains in the sample after desorption into a canister has effectively ceased (after termination of canister desorption).

3.1.40.1 *Discussion*—Residual gas volume, as defined and reported, can be very different for slow desorption and fast desorption methods. Early termination of desorption followed by sample crushing will obviously lower desorbed quantities of gas and increase the residual values. Maceral composition, lithotype composition, and the coal bench being sampled may all affect permeability on small-scale samples. **(1)**

3.1.41 *sidewall core, n*—small diameter core taken downhole by wireline methods using percussion or mechanical methods to drill into the side of the borehole.

3.1.41.1 *Discussion*—The percussion method cores by explosively firing hollow core barrels into a coal seam and then retrieving the coal plug to the surface. The mechanical method uses hollow rotary drills to core into the coal seam, pull the core plugs back into the tool, and then they are retrieved.

3.1.42 *slow desorption method, n*—volumetric readings of canister gas content are taken frequently (for example, every 10 to 15 min) during the first few hours, 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.

3.1.42.1 *Discussion*—Some coals can desorb in excess of one year and a desorption base line may be established with measured gas volumes consistently below 10 cm^3 per reading. At this slow desorption rate, no gas is expected to be lost when transferring the sample from the desorption canister to the residual gas canister.

3.1.42.2 *Discussion*—If the measured gas volume of a canister falls at or below 10 cm^3 per reading (where measurement error becomes too great), then that canister may be elevated to the next time measurement period. This procedure is continued until measured gas volumes are consistently below 10 cm^3 per reading. **(AS 3980, (6))**

3.1.43 *sorbed gas, n*—coalbed gas retained by adsorption or absorption processes or both. **(1)**

3.1.44 *sorption time, n*—time required for 63.2 % of the total sorbed gas (including residual gas) to be released.

3.1.44.1 *Discussion*—It is reported in either hours or days (since time zero) depending on the relative rate at which gas is released from a desorption sample. **(1)**

3.1.45 *sorption standard temperature and pressure conditions (STP), n*—various standards exist.

3.1.45.1 *Discussion*—Historically, the petroleum industry almost universally has used Imperial units of 60°F [15.56°C or 288.6K] as the reference temperature and 14.7 psia [101.3 kPa] as the reference pressure. See Ref **7**. SI systems have used 0, 20, and 25°C [32, 68, and 77°F] most commonly, depending on the data and the area of specialty. The American Petroleum Institute (API, see Ref **8**) has opted for 15°C [59°F] because it is close to 60°F [15.56°C]. The Society of Petroleum Engineers (Refs **9** and **10**) suggests that the choice between 0 and 15°C [32 and 59°F] is arbitrary. Government gas reserve reporting regulations may mandate which system to use, Imperial or SI (metric) (Ref **10**). For coal gas desorption purposes, a standard of 15°C has been adopted simply to conform to API standards. It may be desirable to have a flexible temperature standard for various applications. **(1)**

3.1.46 *time zero, n*—time at which a sample falls below the reservoir or desorption pressure during sample recovery.

3.1.46.1 *Discussion*—Time zero is generally marked when the sample lifts off the bottom of the hole. However, more study of the sample retrieval process is required to determine better the depth that the sample desorption process actually begins. **(1)**

3.1.47 *total gas volume, n*—sum of lost gas, measured gas, and residual gas volumes (all measured on the same sample volume). **(1)**

3.1.48 *U.S. Bureau of Mines (USBM) lost gas time, n*—lost gas time determined from time zero, where time zero is defined as the time when the sample reaches a depth halfway to the surface. **(1)**

3.1.49 *volumetric displacement apparatus (manometer), n*—device, maintained at ambient conditions, for measuring the amount of gas desorbed into a canister. **(1)**

4. Summary of Practice

4.1 This practice describes standardized guidelines for the determination of the gas content of coal by desorption using samples obtained by drill coring methods.

4.2 Immediately after the coal core sample reaches the surface and after the depth of the sample, state of the core, and proportion of coal to non-coal material are recorded, the sample is transferred into a canister and the canister is sealed. Multiple samples from a coal bed should be collected to obtain a gas content representative of the whole coal bed.

4.3 Desorbed gas content (in cubic centimetres) can be measured using a volume displacement apparatus by the slow or fast desorption method, or a combination of the two methods, depending primarily upon the urgency of having gas content data. In the slow desorption method, gas measurements are continued until measured gas volumes are consistently below 10 cm^3 per reading, which for some coals may take more than a year's time to desorb to this level. In the fast desorption method, when sufficient measurements are made to obtain data for lost gas calculations (usually more than 4 h of frequent measurements), the canister is opened and the sample is transferred to the coal crusher. The remaining gas volume is measured on a crushed sample. For gas composition or gas isotope analyses or both, the gas is sampled during desorption.

4.4 All data are entered and maintained on predesigned data forms and spreadsheets. Lost gas, desorbed gas, and residual gas contents are added to obtain total gas content that, after recalculation on sample weigh basis, yields total gas content expressed in cm^3/g, m^3/ton (SI units) [scf/ton (Imperial units)].

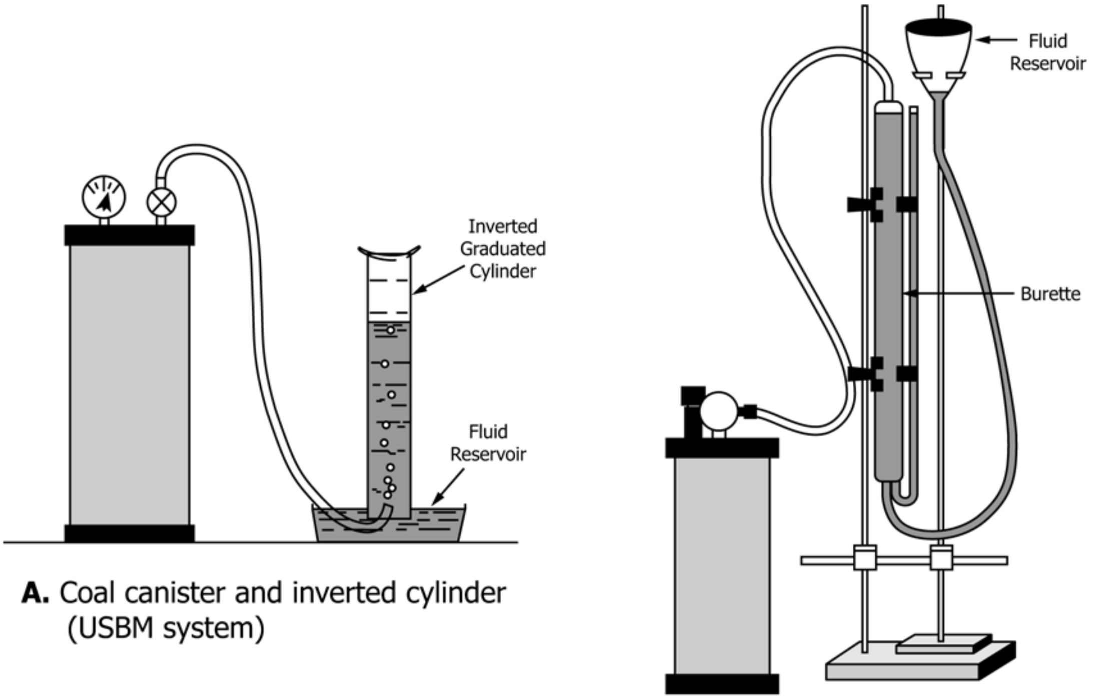
5. Significance and Use

5.1 Canister desorption is a widely used technique to measure the gas content of coal. The gas content data when normalized to volume/weight and multiplied by coal mass is used to estimate the gas in place in an area around the cored well.

6. Apparatus

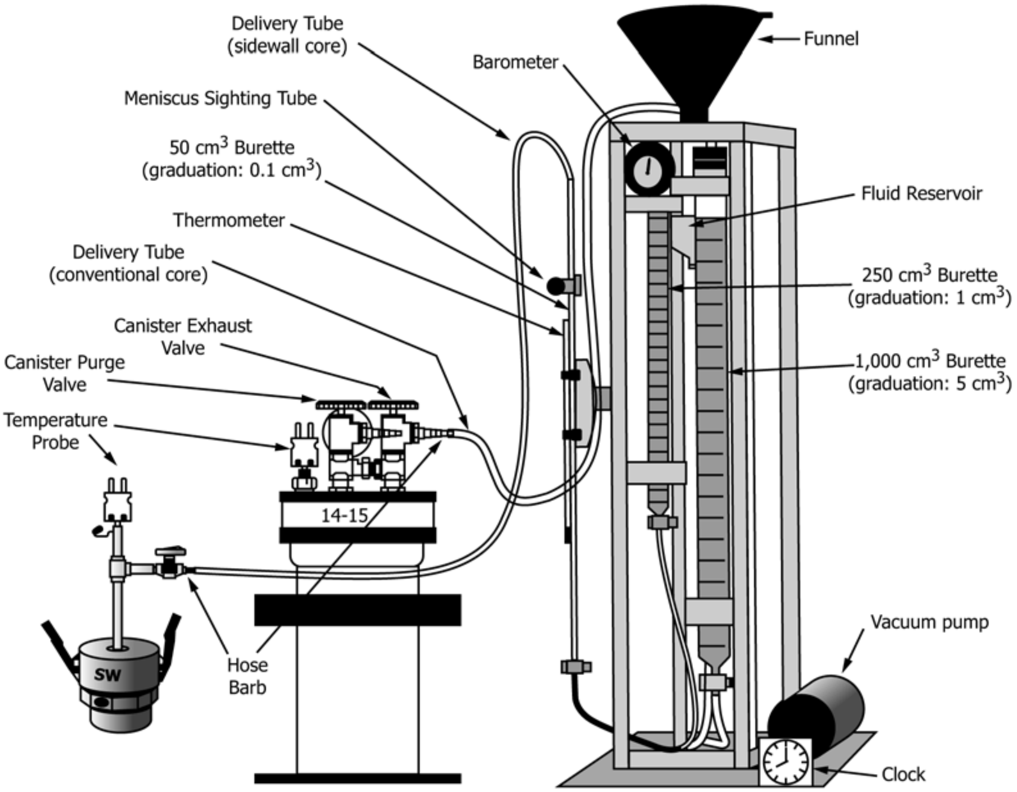
6.1 *Background*—In desorption studies of methane content in coal beds, the goal is to capture quickly the coal sample in a pressure-tight container purged of the air-bearing headspace gas using an inert gas or water to stabilize the sample. Towards this goal, a container (canister) shall be designed and constructed/fabricated for core that would be easy to handle, fill, and close rapidly forming a reliably gastight seal, and facilitate rapid desorbing gas measurements. The primary coal core desorption equipment consists of desorption canisters made of sealed aluminum or plastic and a volumetric displacement apparatus or manometer; these items can be purchased from suppliers that use custom designs **(1, 11)** (Fig. 1) or locally constructed with off-the-shelf materials and parts **(12-14)** (Fig. 2).

6.2 *Materials and Construction of Desorption Canisters*—Canister materials in widespread use today are aluminum, plastic-coated aluminum, and plastic, usually polyvinyl chloride (PVC) materials. The use of stainless steel, although advantageous because of its inert qualities, is not in widespread use because of the high cost of materials and labor. The use of canisters made of unsealed aluminum is not recommended in this practice because of the potential for significant reaction with coal gases and related formation or drilling fluids after the canister is closed. In general, all components of the desorption canister should be made of material that is, or treated to be, nonreactive with regard to the coal or the normally low pH fluids associated with the coal and hydrogen sulfide (H_2S) or other corrosive gases that may evolve during desorption. The canisters should be made leak-proof by using a sealing device or cap such as a neoprene and plastic plug held in place by a wing nut or clamp, a threaded PVC plug sealed by an O-ring, or a coated aluminum cap sealed with a neoprene gasket. One



A. Coal canister and inverted cylinder (USBM system)

B. Improved design for volumetric measurement system



C. Typical industry design for sidewall and core canisters, and volumetric displacement apparatus

FIG. 1 Progressive Development of Canister and Volumetric Displacement Apparatus Design (Courtesy of Gas Technology Institute [see Refs 1, 4, and 10])

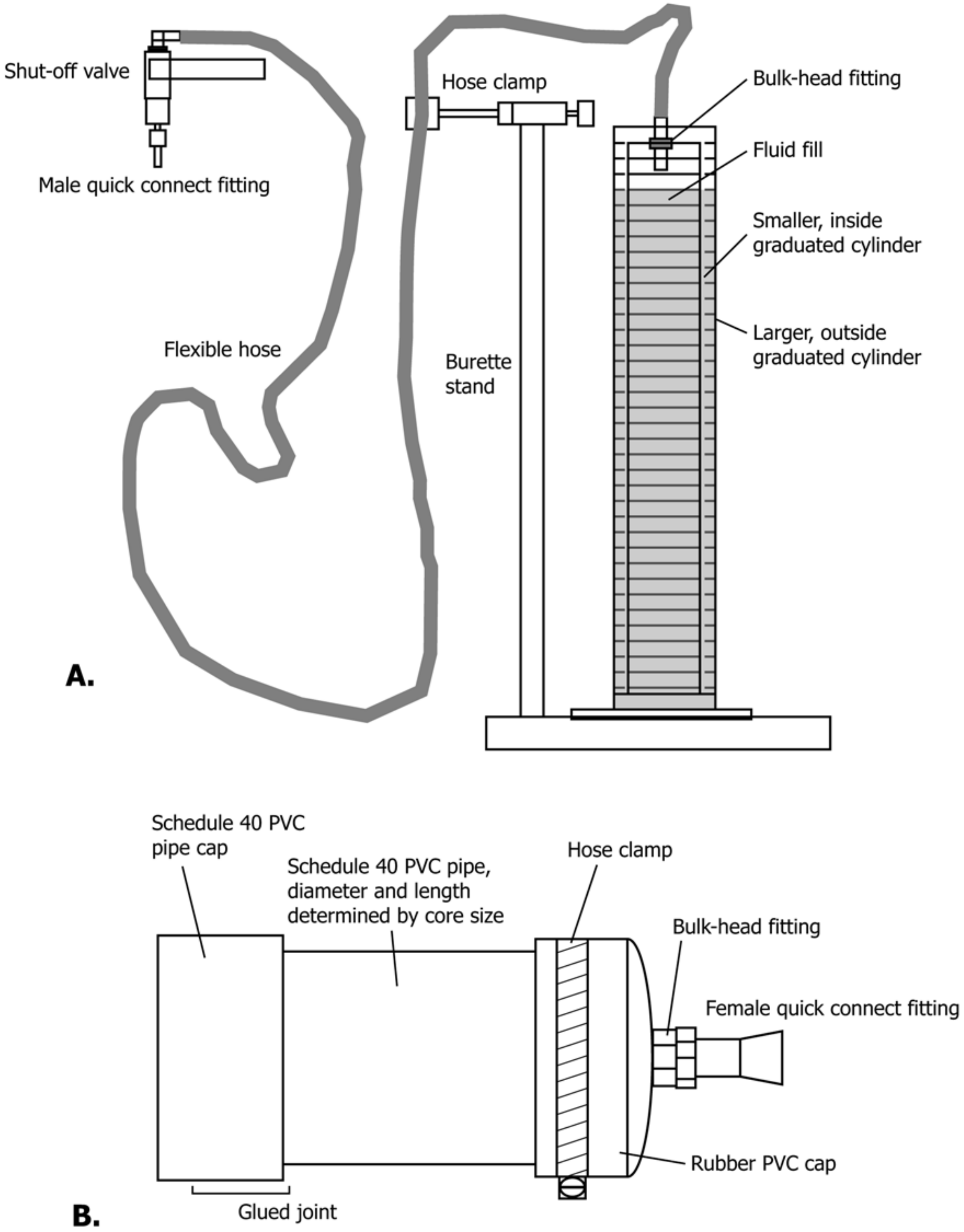


FIG. 2 Custom-Made (A) Volumetric Displacement Apparatus and (B) Canister Constructed from Off-the Shelf Materials (see Refs 12 and 13)

end of the canister should have a permanent cap glued in place (Fig. 2). To prevent leaks, the removable cap area shall be cleaned of coal particles after the coal sample is placed in the canister and before the canister is closed. The use of PVC for gas desorption canisters was first done in the 1980s by the USBM (**5**, **15**).

6.3 *Equipment for Making Measurements*—During desorption, the closed canister is periodically connected via a hose and quick-connect system to a volumetric displacement apparatus (manometer) to measure the desorbed volume of gas. At the time of measurement, the barometric pressure (*P*) and ambient temperature (*T*) in the volumetric displacement apparatus are recorded. National Institute of Standards and Technology (NIST) traceable calibrated digital barometers and thermometers are recommended for these *P* and *T* measurements. If the canister headspace is not filled with water to make its volume zero, then it is necessary to measure headspace gas temperature to correct for expansion or contraction of the headspace gas. This is called the headspace correction. The data-entry forms and calculations for making this correction are discussed in Refs **1**, **13**, and **14**.

6.4 *Materials and Construction of Volumetric Displacement Apparatus (Manometer)*—Most desorption systems are designed to work with desorbed gas volume data collected at ambient temperature and atmospheric pressure, and consequently, a manometer is required to make zero-head-pressure measurements. A zero-head measurement is facilitated using a sliding reservoir tank, a hand-held reservoir, or a hand-held graduated cylinder (**1, 4, 5, 12-16**). The recommended manometer design is based on nested polymethylpentene plastic graduated cylinders reportedly developed by River Gas Corporation (Fig. 2); design and its use are described in Barker et al (**13**) and Barker and Dallegge (**14**). In this design, zero-head measurements are made by manually lifting the measuring graduated cylinder until the water levels in the reservoir and the measuring graduated cylinder are equal in height and, therefore, at zero head. Multiple manometer volumes (50, 100, 250, 500, 1000 cm^3) are required to measure accurately decreasing gas volumes produced from the canisters as the desorption process proceeds.

6.5 The plastic graduated cylinders used should conform to at least Class B accuracy requirements set out in Specification E1272 and ISO 6706. The volume measurement tolerance needs to exceed the requirements of DIN 12681. The accuracy of graduated cylinders also increases as the volume capacity of the graduated cylinder decreases. To maintain adequate accuracy, it is recommended that the size of the inside graduated cylinder of the nested pair should be scaled to be about two times the volume of each measurement from the desorption canister.

6.6 *Materials and Construction of Canister Water Bath*—The desorption canisters should be maintained at a constant temperature (either reservoir or drilling mud temperature) for the duration of the desorption process. Mavor et al (Ref **17**) have shown that estimates of lost gas using desorption measurements made at ambient surface temperatures may be significantly less than estimates obtained from canisters maintained at higher reservoir temperatures. A constant temperature can be achieved by submerging the filled canisters into a water bath heated by submersible electric water heaters or coolers if the ambient temperature is higher than the desired desorption temperature. Large storage tanks can be purchased from local hardware stores and are adequate for low-temperature desorption jobs; however, heat-resistant tanks are required for higher desorption temperatures. Check the heat tolerance of the tanks before use. Submersible electric water heaters and coolers are available from most scientific supply dealers. "Dry" heaters for individual canisters can also be used to maintain a constant canister desorption temperature.

6.7 *Materials and Construction of Residual Gas Measurement Equipment*—Residual gas content of the coal can be estimated at any time during the desorption process after the initial measurements have been made to obtain the basis for lost gas calculations. Following the slow desorption method, residual gas is measured after the samples have completely desorbed. The fast desorption method allows the samples to be removed from the canisters for residual gas determination soon after the measurements for lost gas calculations are completed. It is recommended that the samples be allowed to desorb as long as practical considering time and budgetary constraints. To measure residual gas, a crusher is required to pulverize the coal core to release and measure the remaining gas. The crusher should be capable of pulverizing the sample to 95 % of the material passing a 212-μm mesh. Typical representative coal-mass-to-crusher volume ratios used range from 1:1 to 1:7. The ratio should be kept constant. The crusher should allow the released gas to be bled off and volume measured either during or after crushing. See Guide AS 3980. Gas volumes can be measured using the volumetric displacement apparatus discussed in 6.4.

6.8 *Gas-Sampling Apparatus*—Gas-barrier plastics bags and in-line gas sampling tubes are suitable. In-line sampling tubes placed between the canister and the measuring apparatus eliminate the risk of composition change in the measured gas caused by solution in the measuring fluid. Another gas collection method by gas displacement of water in glass bottles may also be used to collect desorbed gas directly from the manometer hose. The sample size is determined by the method of gas analysis. (See Ref **1** and Guide AS 3980.)

6.9 *Weighing Device*—A scale with accuracy better than 1 % is required to weigh the canisters, coal-filled canisters, and coal- and water-filled canisters.

6.10 *Potential Problems Encountered*—Major problems that can lead to spurious gas content measurements are: *(1)* sample recovery too long; *(2)* canister leaks; *(3)* incorrect desorption temperature; *(4)* excessive pressure buildup in canister; *(5)* reaction of canister materials with coal, gas, or fluids; and *(6)* biogenesis in the canister.

6.10.1 *Sample Recovery Time*—It is critical to minimize the time required for sample collection, retrieval, and placement into the canister. If too much time elapses between coal sampling and placing the sample in the canister, much of the gas may be lost and an accurate lost gas estimate will not be possible. To mitigate this problem, try to use a fast core-retrieval system such as the wireline method. Make all preparations for sampling well in advance of the core reaching the surface to minimize the time required to get the samples into the canister and sealed. For best results, follow the coal-sampling techniques described by Luppens et al (**18**).

6.10.2 *Leaks*—Canisters should be pressure tested before use to find and eliminate any leaks. Pressure testing involves pumping gas into the canister and confirming that it can maintain a 70-Kpa [10-psia] internal pressure for at least 12 h at a constant room temperature. After filling with coal at the drilling site, canisters should be tested for leaks with a gas-leak-detector fluid solution (soap solution). Also, after the canisters are completely submerged into the water bath, their airtightness should again be checked visually for bubbling gas just before being attached to gas-measuring apparatus. After filling with coal and closure, canister leak detection is done by visually detecting bubbles in the desorption tank water or bubbles growing on the canister or the canister top or in fluid placed in the open end of the quick connect. Canisters should also be checked for leaks after one or more near-zero or zero volume measurements.

6.10.3 *Desorption Temperature*—During coal core retrieval, gas is lost as the core travels to the surface. This temperature at which the lost gas occurs is usually the borehole mud temperature, rather than reservoir temperature (**13**, **14**). Circulating drilling fluid during the coring process brings the core temperature approximately to that of the mud temperature. To maintain a stable desorption temperature, the desorption canister should remain submerged up to the neck of the quick connect during desorbed gas measurements. The water bath, or other constant temperature technology, is kept at a stable temperature of ±2°C equal either to the sampled coal seam in-situ temperature or the drilling mud temperature depending on how the data will be used.

6.10.4 *Pressure Buildup in Canister*—Gas pressure inside the sample container will inhibit the desorption process. Typically, gas desorption is rapid initially and declines significantly through time. Initially, measure gas volume every few minutes. As gas content and pressure declines, the measurements can be reduced to an hourly and then, eventually, a daily schedule. Usually, the initial pressure inside the cylinder is less than 35 Kpa [<5 psi] above ambient atmospheric pressure. The point of the closely spaced measurements during the first 4 h, commonly termed the lost gas period, is to keep pressure from building up in the canister and inhibit desorption. After the lost gas period, the desorbed gas volume measurements can go on for days to months depending on the physical diffusion/desorption character of the coal sample.

6.10.5 *Reaction of Canister Materials with Coal, Gas, or Fluids*—Any material (in particular, bare aluminum) that can react with coal gases and related fluids should be avoided in the construction of desorption canisters. An example of this problem is documented by Faraj and Hatch (**19**).

6.10.6 *Biogenesis in the Canister*—Biogenesis in the canister has been conjectured to be the cause of a secondary sharp increase in gas flows during some canister desorption tests (**20**). The secondary gas flows are often associated with a sucking of gas back into the canister as may occur late in the desorption test when outside pressure can exceed the pressure in the canister. Therefore, it is critical to try to increase the time between desorption measurements to assure adequate pressure has built up inside the canister. If a backflow does occur, quickly close the valve on a canister when backflow occurs and wait some time before measuring the canister again. Chances of backflows can also be reduced by waiting for low-pressure weather systems to arrive or slightly increasing the temperature at which the canisters are being held. This practice discourages the use of biocides in the fluids used to fill the canister head space.

7. Equipment and Sample Preparation at the Well Site

7.1 *Project Preparation*—The key to improving the accuracy of canister desorption measurements is to minimize the lost gas period. Preparations should be made before coring to get the core into the canisters as soon as possible after retrieval off the well bottom commences.

7.1.1 Have the desorption measurement equipment and canisters ready to receive the core samples. Previously pressure test canisters and have their dry weight and can number already scribed or affixed to them. Place the canisters near where the core will be laid out. All measurement equipment should be pretested and made ready.

7.1.2 A table with all of the measuring equipment arranged on it for sequential reading should be placed in a position where the manometer hose can conveniently reach the canisters in the constant temperature bath.

7.1.3 At the drill rig and before coring, set a washing tray on sawhorses and prepare it to receive core. Set up a water hose with a spray attachment to rinse the drilling mud off of the core. Have a hammer and broad blade chisel available to break core into pieces about 2 to 3 cm [1 in.] shorter than the canister cylinder length. Also, it helps to tape a red and a black felt-tip pen together with their felt tips aligned so that the core can be marked with red right—with right being your right when looking towards the start or top of the core run. Also, have a tape ready so that the depth along the core can be quickly marked.

7.1.4 Use a digital camera to photograph the core quickly in the wash tray after marking red right and depth on the core but before it is broken up. The core cannot be viewed again until desorption is terminated, which in some cases can be months.

7.1.5 Prepare the desorption data forms by recording canister numbers, canister dry weight, well information, the time the core was lifted off bottom, the time the core reached the surface, after the canister is filled, and the time that the canister was closed. Use a 24-h clock to reduce the chance of confusion of an AM or PM designation inherent in the 12-h clock. Focus on rapid retrieval as part of the choice of drill rig (a wireline method is preferred), and then coach the drill rig crew on the importance of rapid core retrieval at the well site at predrilling meetings and finally insist upon it during coring.

7.2 *Core Selection for Desorption*—There are two sampling methods in widespread practice. The first is to sample all of the coal and, sometimes, the related carbonaceous mud rocks in a coal zone. The other practice is to take one 30-cm piece out of each metre of core spaced across the coal seam. A recent study, however, has shown that the minimum samples needed to estimate accurately coal gas resources for a given level of uncertainty may vary between coals (see Ref **21**). In any case, the widest range of rock types should be included in the sample from the purest coal to a faintly carbonaceous mud rock in the coal zone. This practice leads to a resource analysis by the construction of gas content versus density plot that is used to assess the gas content in portions of the coal zone either not sampled or not recovered in the core run.

7.3 *Use of Drill Cuttings for Desorption*—This practice does not recommend using desorption results obtained from cuttings for coal gas resource estimates. Because of the small particle size of the cuttings, gas desorbs much more quickly than from solid coal core. Despite these disadvantages, a drill cuttings sample may indicate the presence of coalbed gas.

7.4 *Filling and Closing the Canister*—The canister is designed to be a pressure-tight container that also allows measurement of the amount of gas evolving from the porous matrix of the coal. It is intended to be a simple device but can easily be compromised by improper sealing—usually related to a poorly seated or dirty sealing device surface.

7.5 *Purging the Canister Headspace*—There are two recommended options for purging canister head space. Option 1: the canister should be purged of oxygen with an inert gas to avoid potential oxidation of the coal. This is especially important for lignite and subbituminous coals in which oxidation may increase pore space and alter desorption results. An inert gas such as helium can be used for this purpose. Ideally, the head space can be eliminated by Option 2, which consists of filling the canister with formation water or distilled water.

7.5.1 *Helium Purge*—Set the helium regulator to 30 to 40 Kpa [4 to 6 psi]. Insert the male quick connector from the end of the helium hose to the female quick connect on the canister. Pressurize the canister for a few seconds, then purge with helium by removing the helium hose and venting the canister with the open-ended quick connect. Repeat this procedure at least five times. After the last fill, again insert the open-ended quick connect and purge the excess helium pressure. Remove the quick connect as the last helium escapes the can. In this manner, there is no extra pressure in the can and air exposure to the coal has been reduced.

7.5.2 *Water Fill to Purge Headspace*—Formation water from a nearby well that produces from the interval of investigation or distilled water, if formation water is not available, may be used to purge the canisters of ambient air. Preheat the formation or distilled water to the water-bath temperature. This is done by simply placing the jugs of water in the heated tanks well ahead of use. The advantages of using water to fill the canister headspace are that it keeps the sample moist and it purges nonabsorbed air in the headspace that may react with the sample. However, some problems can occur from using water to fill the canister headspace. The added water may introduce microbes that are capable of generating gas in the canister during the desorption process. Further, water is an oxide and may be capable of supplying oxygen species to the core. Also, hydrogen exchange may occur between coal and water and affect isotopic analyses of the desorbed gas. Chemical reactions between an aluminum canister and the coal may be enhanced by the presence of water as a solvent or possible galvanic action that conductive fluid-like water makes possible or both. Water pressure in the canister may inhibit desorption—especially when internal canister gas pressure approaches atmospheric pressure near the end of desorption. Another problem is that because desorbed gas slowly dissolves into the water when the canister is closed and exsolves when it is open, the manometer response can be very sluggish. Consequently, the endpoint of each volume measurement on a water-filled canister is a subjective judgment by the geologist. Also, keeping the headspace completely filled proves difficult in practice because the desorbed gas dissolves in the water and, when the canister valve is opened to the manometer, the exsolution of gas bubbles can cause the water to move up into the manometer tubing and be lost. So, as volume measurements proceed, zero headspace evolves to a small headspace. Most workers assume that the small headspace that evolves is negligible. Lastly, according to Ryan and Dawson (**16**), adding water to fill the headspace may make it necessary to correct the desorbed gas volumes for a continuing contribution of water vapor to the desorbed gas stream.

7.5.3 *Use of Biocides in Headspace Filling Fluid*—The use of biocides, such as zephiran chloride, in the fluid used to fill the canister headspace has been proposed, but this method is not well tested and it may cause unintended consequences. This practice discourages the use of biocides.

7.5.4 *Recording Time that the Canister Is Closed*—Note the time when the valve was closed after helium purging or when the cap was closed after adding water fill. Write the canister closure time and the date on the desorption form for each canister.

8. Procedure for Measuring Gas Content

8.1 *Measuring Gas Volume*—This is the key measurement in the canister desorption method and it requires several values to be recorded simultaneously for an accurate measurement. First of all, as described in the following, the measuring device shall be capable of measuring volume at zero head pressure. Secondly, because the measurement will be corrected to standard temperature and pressure conditions (STP), the ambient barometric pressure and temperature need to be recorded. In canisters with unfilled headspace volume, the headspace temperature should also be measured inside the canister. Finally, keep in mind that released gas may be from other sources besides desorption, for example, biogenesis in the canister, gas emitting inorganic chemical reactions, or, early in the desorption process, free gases flowing out of coal or rock pores. These nondesorption sources are usually detected by changes in the slope of the cumulative desorbed volume-versus-time plot.

8.2 *Water Vapor Correction*—Gas volumes from canister desorption of coal may need to be corrected for water vapor as a result of the inherent moisture content of the coal and if water was used to fill the canister headspace. The method and calculation are discussed in Ryan and Dawson (**16**).

8.3 *Determining Volume of Released Gas by Manometer*—The manometer system should be tested before every reading by inflating it to approximately 80 % of a full-water column. The volume of the gas in the manometer, under constant temperature conditions, should not change. Proper reading of graduated cylinders is a key to accurate volumes. When reading the volume, make sure your eyes are at the level of the liquid meniscus by moving your eyes up and down until you are assured that you are looking across the flat liquid surface in the plastic graduated cylinder. It may improve accuracy to use colored water in the cylinder and place a white paper card with a line scribed across it on the back side of the cylinder during readings.

8.4 *Gas Sample Collection for Analysis*—Gas samples should be collected throughout the desorption process to determine gas composition and for optional isotopic analyses. If only one or a few samples can be collected during the desorption process, the time of collection should be carefully planned to get as representative a sample as possible (gas composition and isotopic signature change during desorption; see Ref **22**). Several techniques can be used for gas sample collection. These include stainless steel cylinders, evacuated, hematological, glass vials with resealable stoppers, inert sampling bags, and glass bottles.

8.4.1 *Evacuated Stainless Steel Gas Sample Collection Cylinder Method*—The objective is to collect a sample of desorbed gases that is representative of the coal gases being emitted at the sample time, while eliminating or minimizing contamination by air. The sample is collected in evacuated stainless steel gas sample collection cylinders closed by stainless steel valves and connected to the canister by quick-connect connectors. This assembly is sealed by polytetrafluoroethylene tape at all threaded connections. The gas sample should be collected as dry as possible to avoid post-sample alteration in the sample cylinder or else a biocide should be added to the fluid. The gas sample cylinders should be kept under vacuum to: *(1)* minimize the possibility of contamination while not in use; *(2)* assure that residual gases from previous samples are not mixed with the new sample; and *(3)* allow samples of unpressurized, or low-pressure gases, to be rapidly collected while reducing the chances of air contamination from the gas sample cylinder. The gas sample is taken after the gas sample volume is allowed to build in the sample cylinder by skipping measurements from the selected desorption canister. After the gas sample is built up in the canister, the gas sample cylinder is connected to the desorption canister by quick-connect connectors. The sample is taken. Then the canister is connected to the manometer and a normal reading is taken. All measurement data is taken in the routine manner except that the measured volume is equal to the sample cylinder assembly volume plus the manometer reading. After the sample is collected, it is recommended that the gas sample cylinder be doubly sealed by placing polytetrafluorethylene tape on the valve threads and screwing on stainless steel caps. This reduces the chances of inadvertent sample loss before analysis.

8.4.2 *Water-Filled Glass Bottle Method*—This method uses a clean rubber and ceramic-stoppered bottle with a new silicone or red rubber gasket (such as used on some beer bottles). This method is included because it allows sampling gases with equipment that is widely available and can be quickly acquired in many field areas. In most cases, it is recommended to use the dry stainless steel cylinder method (8.4.1). To sample gases with the water-filled bottle method, a water bath is filled to two thirds full with distilled water that has benzalkonium chloride (also marketed as zephiran chloride) disinfectant added to make a 1 % volume-to-volume (v/v) solution. The bottle is immersed in the water bath and all air bubbles removed by slightly tilting, tapping, or shaking the bottle while still under water. After a gas measurement using the standard manometer has been made, place a hose clamp on the manometer hose near the tip of hose. Place the sample hose and hose clamp from the manometer in the bucket of distilled water treated with zephiran chloride. While holding the water-filled bottle upside down in the bucket, open the lid and place the sample hose with the closed hose clamp near the tip up into the bottle. Open the hose clamp. If there is a lot of gas, it should replace the water by bubbling up into the bottle and displacing the water with methane. If there is a small quantity of gas, then it may need help to exit the manometer by pushing on the top of the inside graduated cylinder and possibly slightly tilting the collection bottle. It is important that the collection bottle not get above the surface of the water and possibly contaminating the sample with air.

8.5 *Residual Gas Analysis*—The residual gas volume can be determined by crushing the sample in an airtight container and measuring the volume of gas released by the same method as that used for the desorbed gas. The volume of residual gas measured in the laboratory for samples subjected to elevated temperatures to approximate actual reservoir conditions will probably be less than would have been measured if the sample had equilibrated to ambient laboratory temperature during desorption monitoring. See Refs (**1**)and (**5**). Some studies recommend crushing duplicate samples and taking the mean of the two samples as the residual gas content (**6**).

9. Report

9.1 A spreadsheet print or digital version including well header data, sample depth, mud weights, and sample timing (off bottom, at surface, and closed in canister), along with desorption readings should be included in the report. The spreadsheet should also show the calculated gas content for the sample on an "as-analyzed" or "raw" basis (including moisture) and a dry basis. Coal gas content for each sample should be reported in cubic centimetres per gram (cm^3/g) and standard cubic feet per ton [scf/t]. Several predesigned spreadsheets are available for use; these include those found in McLennan et al (**1**), Barker et al (**13**), and Barker and Dallegge (**14**).

9.2 The report should include a desorption cumulative volume-versus-time plot with the addition of sample history points (time moved to laboratory from the well and so forth), gas sample points, and the sorption time plotted. Also, the plot should show canister number, depth interval, and desorption temperature. Examples of such plots can be found in McLennan et al (**1**), Barker et al (**13**), and Barker and Dallegge (**14**).

9.3 A plot of lost gas should also be included in the desorption cumulative volume-versus-time plot. Depending on the spreadsheet that is used, the lost gas plot can be either by a calculated best-fit line generated by the spreadsheet or a "visual" best-fit line manually drawn on the desorption cumulative volume-versus-time plot. These methods are discussed in McLennan et al (**1**), Barker et al (**13**), and Barker and Dallegge (**14**).

9.4 The sample report should include proximate analysis of each coal sample (Practice D3172) and sample specific gravity (Test Method D167). Other data such as ultimate analyses (Practice D3176), maceral composition (Test Method D2799), adsorption isotherm data, and so forth are optional.

9.5 A plot of coal specific gravity versus gas content of the various samples collected from the drill project should be included with the report. As discussed in McLennan et al (**1**), such a plot is useful to determine the gas content of similar coal samples that have not been tested for their gas content using the canister desorption method.

9.6 Gas sample compositional and isotope data are optional for the desorption report but should be included if analyses are available at the time of report writing.

9.7 When appropriate, a correction of truncated, short-term desorption data to a long-term basis should be included with the final report.

10. Precision and Bias

10.1 *Accuracy of the Measuring Devices*—In the volumetric displacement apparatus, burettes with volumes of 50, 250, and 1000 cm^3 (depending on the volume of the gas in coal) are used, with a graduation of $1 cm^3$. Their precision is ± 1 cm^3. A precision (±1-g) top-load balance is used to weight empty and filled canisters.

10.2 *Repeatability and Reproducibility of Gas Determinations*—It is not possible to specify the precision of this practice because gas determinations involve numerous steps of both direct determinations and calculations. Also, because of great difficulty to an impossibility to perform the whole procedure on two identical samples, repeatability and reproducibility have not been rigorously tested and determined. Differences are expected between gas volumes obtained by a slow versus fast desorption method. Also, temperature and pressure variations during desorption, empty space volume in the canister, and frequency of taking measurements from the canisters influence the final numbers. Additional factors include imprecision and differences in lost gas calculations. Special care needs to be taken to ensure that all these factors are considered and standardized as much as possible.

10.3 *Bias*—No information can be presented on the bias of the procedure in this practice because no material having an accepted reference value is available.

11. Keywords

11.1 coal bed; coal gas; core; desorption; gas content; gas sampling; gas volume measurement; methane; resource assessment

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Table 1 Links

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