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GEOLOGICAL SURVEY

A Field and Laboratory Procedure for Desorbing Coal Gases

by

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INTRODUCTION

Coal bed methane is assuming a major role in gas exploration and now constitutes important reserves in this country (Diamond et al., 1986). Many geologists, however, have had little experience in desorbing methane from coals. We have described the procedure we used and include a discussion to help the novice in coalbed methane studies by providing an outline of a basic coal core desorption procedure. The best discussion of coal desorption procedure we have found is Close and Erwin (1989) whose report we have used as a starting point. The main difference is that we present more details of the procedure and some suggestions on improving the equipment and equipment sources.

INSTRUMENTATION

Coals were desorbed using the canisters and burette type manometer illustrated in Close and Erwin (1989). A burette type manometer was constructed from commonly available laboratory parts (Fig. 1). It is also possible to make one from a graduated cylinder inverted into a pan of water (Diamond and others, 1986, Fig. 2). The automatic rezeroing type of burette (Fig. 1) seems more practical when many canisters have to be read quickly. However, the graduated cylinder type (Fig. 2) is better for sampling gas. All hose couplers for connecting the manometer and canister must be of the same type (and there are many different types) so it is best to buy these as male/female coupler sets to assure compatibility and that they are gas tight. The manometer fluid was a colored propylene glycol and water mix (generally available as the potable water system antifreeze used in recreational vehicles to prevent freezing if the trailer heater failed). The canister to manometer gas line length was held at a minimum to reduce the chance of error due to temperature differences.

SSD Inc. (manufacturers addresses listed below) plastic desorption canisters with aluminum caps were used. The canister caps were modified so the coal core temperature could be measured by inserting a thermocouple probe into the closed tube that is in turn in direct contact with the coal core. This modification was accomplished by removing the pressure gauge from the canister cap and inserting a 6 inch long 1/4 inch diameter copper tube (Fig. 3). The tube is plugged on one end by solder and left open on the other end. The open end is soldered into a 1/4 to 1/8 inch brass pipe-thread reduction-bushing. The 1/4 inch outside diameter threaded bushing screws into the threaded hole for the pressure gauge (Fig. 3). Omega Engineering also makes a thermocouple well adaptable to this same purpose.

The canisters and manometer were kept inside an insulated box to minimize temperature differences between the canisters and the manometer and to allow the canisters to be heated to reservoir temperature. During our desorption measurements, the samples were held at ambient room temperature (usually 70-75°F) and not heated to reservoir temperature because at shallow core depths, these temperatures are nearly the same. We note that when desorbing in the laboratory (or indoors) the samples should possibly be cooled from ambient room temperature to reservoir temperature. In the Wind River Basin, for example, the mean annual surface temperature at the drill site is 43°F and the temperature gradient is about 1.2°F/100 ft suggesting reservoir temperatures of about 50°F for a 600 ft well. In deeper (and presumably hotter) wells, heating the canisters to reservoir temperatures is critical for proper desorption measurement. Some workers use a water bath for heating the

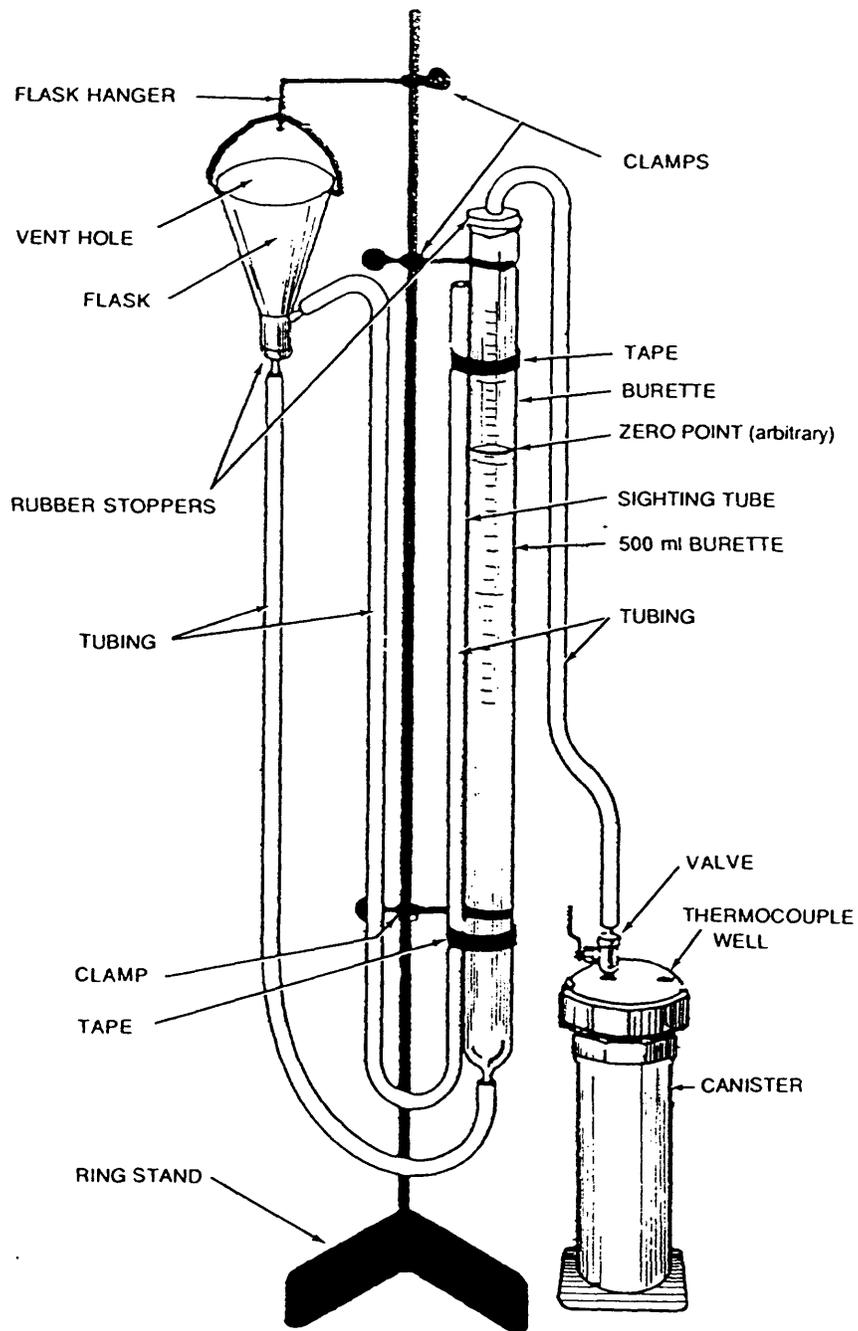


Figure 1.--A manometer constructed of a standard glass burette modified for coal desorption volume measurements. Figure modified from Close and Erwin (1989).

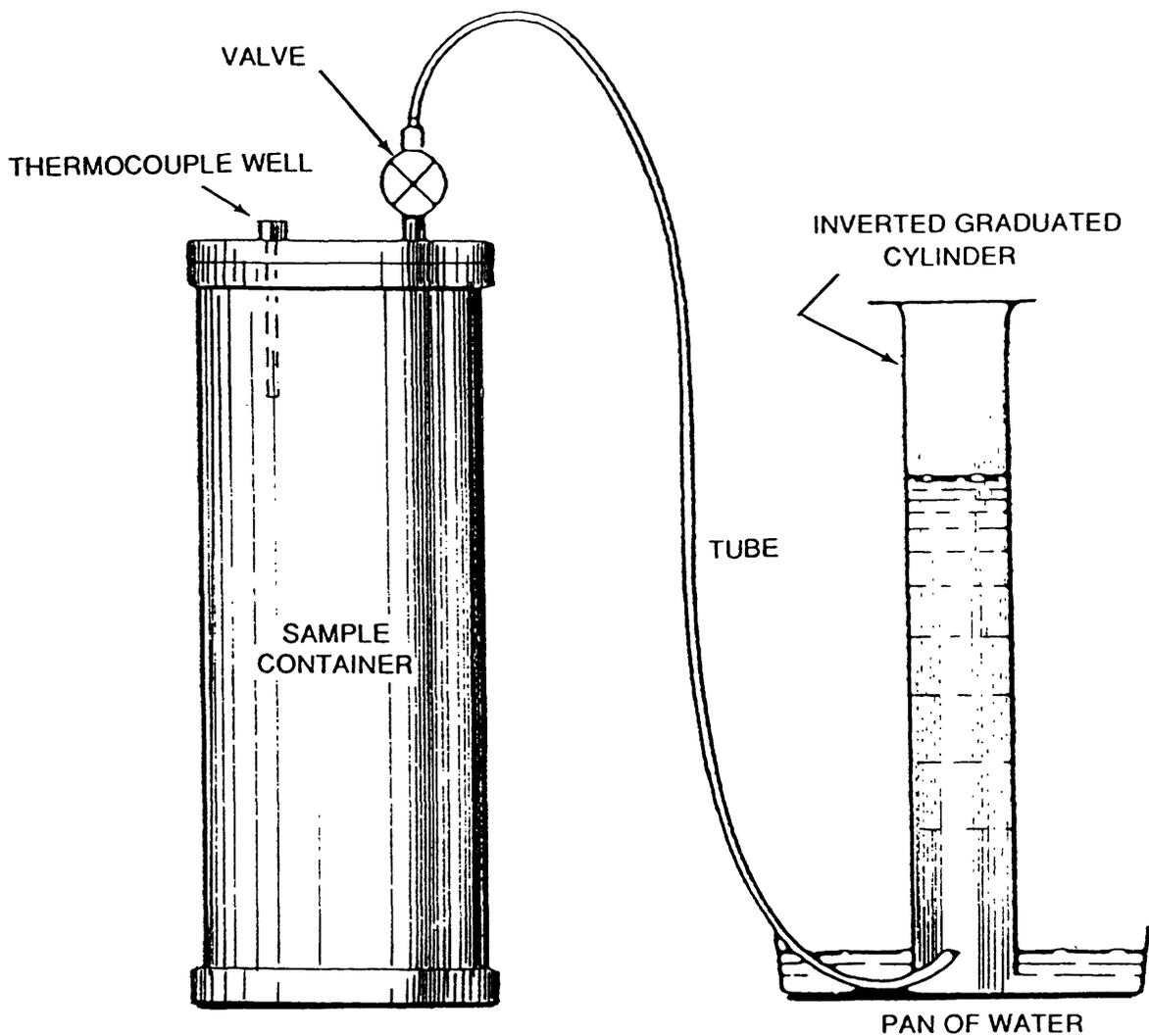


Figure 2.--A manometer constructed of a standard laboratory graduated cylinder for measuring gas volumes and sampling the gas for analysis. Figure modified from Diamond et al. (1986).

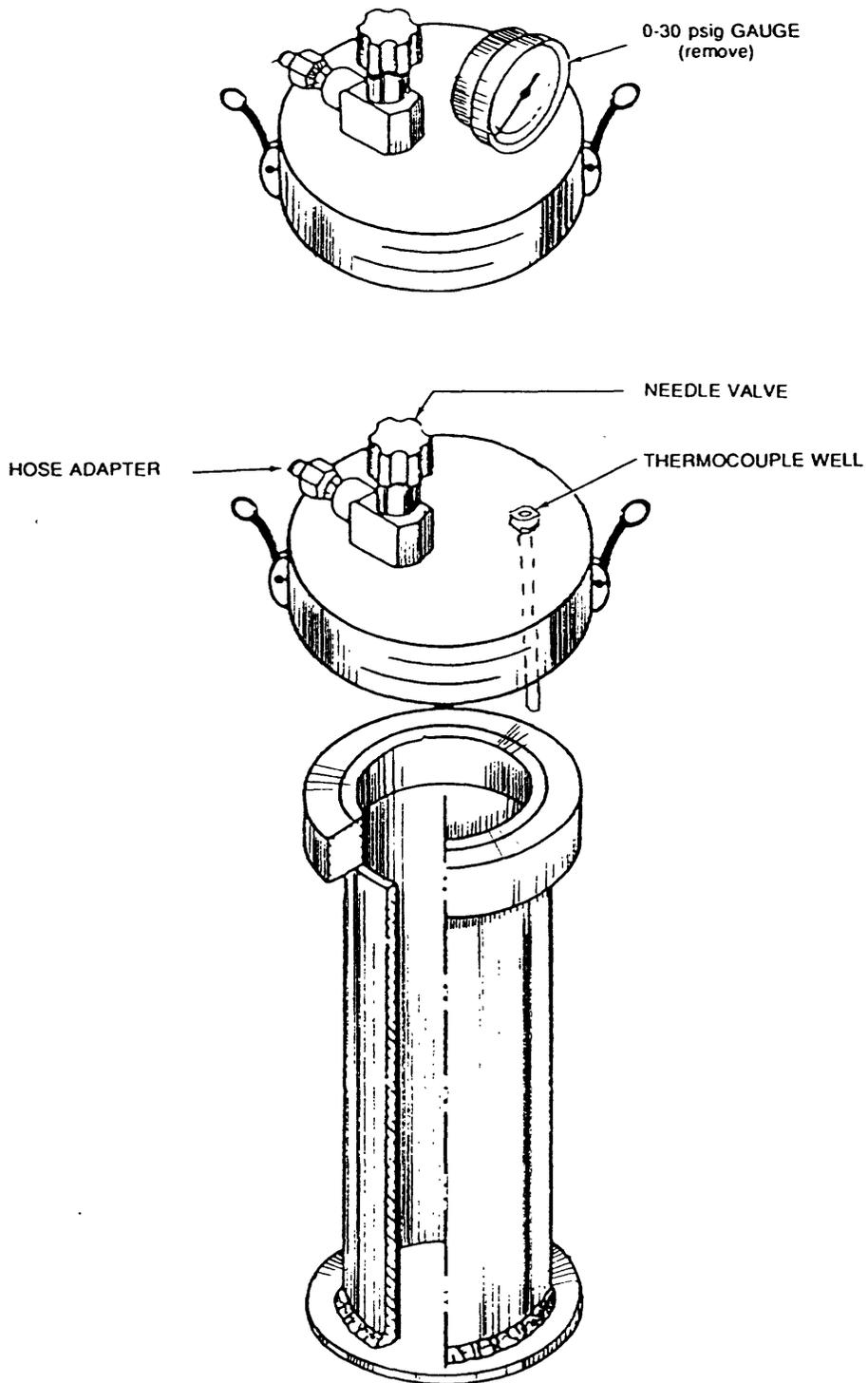


Figure 3.--A modified canister for coal desorption that allows temperature measurement by direct contact with the coal core. Figure modified from Close and Erwin (1989).

canisters, but we think it impractical to move a water-filled tank around in the field. We used an insulated box with air as the heat transfer media. For desorption at elevated temperatures, plastic canisters made of polyvinyl chloride (PVC) can only be used to about 175°F without deforming. Aluminum canisters must be used if desorption is performed at high temperature.

For correction of desorbed gas volume measurements, a digital reading of ambient pressure was obtained using a Solomat model 500E digital meter equipped with a 515BPX barometric pressure probe. The pressure probe was calibrated both before and after each field session with a Princo model 543 Fortin type mercury barometer. Internal canister temperature at the core surface was made by a digital thermometer constructed of a 6-inch long, 0.05-inch diameter, Omega Engineering type K miniature thermocouple plugged into the Solomat 500E meter.

Correction of the desorption measurements used the equation given in Close and Erwin (1989) which is based on measurements taken in English units. The Close and Erwin formula is based on the General Gas Law (See Schaum and Rosenberg (1966, chapter 7 for review) and can be recomputed to any unit system. English (1984) has formulated an alternate procedure.

MANUFACTURERS ADDRESSES

Omega Engineering, Box 2721, 1 Omega Dr., Stamford CT. (203-359-7807)

Princo Instruments, 1020 Industrial Highway., Southampton, PA 18966
(215-355-1500)

Solomat Instrumentation, 652 Glenbrook Rd., Stamford, CT 06906 (203-348-9700)
SSD Inc., 361 27&1/2 Rd, Grand Junction, CO 81501 (303-245-5889)

A USGS COAL GAS DESORPTION PROCEDURE

Prior to going out in the field

- A. Well ahead of core recovery time, clean the canisters, inspect and replace damaged gaskets or defective gauges, if present. Clean the canister cap seal and if necessary, recement it in place with silicone cement. Replace the canister cap and tighten it. Pump up cylinder to 10-15 PSI using a bicycle pump fitted with a coupler that can be mated to the canister fitting. Mark pressure gauge needle position on the gauge plastic cover with grease pencil; Immerse in water bath. If leaks are detected, repair and retest. In some cases it may be advantageous to lightly apply silicone vacuum grease to promote a good seal, but the disadvantage of this procedure is that the grease may attract dirt, degrading the seal.

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Important: use both hands and tighten both screws simultaneously to evenly seat canister cap. Tighten the screws until you have to strain your fingers to turn them. Use no tools at this point as this may deform the cap or seal.
=====

In some cases, it may be necessary to fit washers and hex nuts to the canister screws and torque them down if it is too difficult to attain proper seal of the cap by hand.

Be prepared to fix the equipment by putting together a box of extra parts, valves, fittings, and so forth, because leaks can easily develop and require immediate repair often in a remote area.

Equipment-- Bicycle air pump or equivalent; water bath. **Supplies**-- Vacuum grease is best for O-rings and gaskets; Teflon paste is best for sealing valves and gauges; felt-tip pens; grease pencils; as needed, a torque wrench, washers, and hex nuts.

- B. Sequentially number canisters.
- C. Weigh canisters and write mass on a label placed on cylinder before desorption measurements. We have found it necessary to place the canister on the same spot on the scale surface to increase reproducibility of the measurements. The required scale needs an accuracy of +/-1 gram and a capacity of 6-10 kg depending on the length and material used in constructing the canister (see expected results section following).

Equipment-- 10+/-0.001 kg capacity scale; 4 to 6 kg mass for calibration checks.

AT THE DRILL SITE

- D. Fill the burette (SSD Inc., Grand Junction, CO; Fig. 1) with fluid before coring to be ready to make desorption readings every 15 minutes for the first two hours. It is useful to blow a small amount of air into the manometer to displace the liquid and see how long it takes to refill to the zero point. Note initial manometer zero point volume (V_0) by marking it with a grease pencil on the graduated cylinder. We found it handy to have the zero point down some 50-100 ml from the top of the manometer because, if the zero point is higher and the canister pressure below atmospheric, the manometer fluid would be sucked into the chamber. In any case, if sucking occurs the canister valve should be immediately closed (see the critical measurements section below). Also we note the amount of gas sucked back into the canister and subtracted it from our cumulative volume readings.

During desorption, the next measurement should not be taken until the manometer refills to the zero point. With the antifreeze fluid in the manometer at low temperatures, the viscosity increase made refilling too slow. This problem was corrected by using larger diameter tubing and fittings on later burette manometer designs. Also, note that some canister to hose couplers seal automatically when opened; these types must have a valve to open the manometer to atmosphere for rezeroing.

Equipment-- Burette manometer filled with lightly dyed water (to aid discrimination of the meniscus level) and(or) colored antifreeze for recreational vehicle potable water system (a propylene glycol mixture:

necessary only if freezing temperatures at drill site are expected).
Low pressure air hose. Appropriate hose and fittings.

- E. At the drill site and before coring, set up washing tray on saw horses and prepare it to receive the core. The tray is not necessary if a split core barrel is used because the core can lie in the opened barrel until it is ready to go into the canister. Set up a water hose (for rinsing drilling mud off core). In wells drilled using air, the core may be clean enough to use without washing. Have a hammer and chisel or other cutting tools ready to break core into 12 inch pieces. Have a felt tip pen ready ready to mark footage and orientation of core. Photographing the core in the tray before breaking it up could be useful because the canister cannot be opened again until desorption is complete which in some cases can be several months.

Equipment-- Core tray; flowing water source; water hose; hammer and chisel; camera; film; and water.

- F. Attach burette to canister hose; set up the barometer, thermometer and clock in the trailer; arrange canisters; write in well information on desorption forms. These preparations and any others deemed necessary should be made before coring in order to get the cleaned core into the canisters as soon as possible (ASAP) after coring.

DURING DRILLING

1. Record crucial times, dates, initial burette volume and weights on desorption chart.

Use 24 hour clock to facilitate cumulative time calculations.

Equipment-- clock; desorption form (Fig. 4).

2. As soon as possible (ASAP) after coring, remove the rock cylinder from the core barrel, place it in the tray or open the split core barrel. If necessary, quickly wash the core with fresh water. Mark core with black and red felt tip pens from top to bottom (put the red pen to the right) and break core into about 12-inch long pieces. Photograph core.

Before opening the canister, check to see if it still has a pressure reading near the grease pencil mark (indicating the canister has not leaked; if it has leaked set it aside and repair later), then open the valve and release the pressure before unscrewing the cap. However, remember that a decrease in temperature after sealing the canister can decrease pressure. Therefore, if the needle is slightly displaced from grease pencil mark several days after closure, the canister may not be leaking. Pressure decreases of up to 5 psi due to weather changes have been noted in non-leaking canisters (Donna L. Boreck, U.S. Bureau of Mines, personal communication, 1991).

Equipment-- core wash tray; water spray; saw or hammer/chisel; compressed air to dry core; camera; and felt tip pens.

USGS COAL METHANE DESORPTION RECORD

Canister number: _____ Canister Core interval: _____ Page _____ of _____

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Well name: _____ Field: _____ Project: _____

Core formation/member/bed: _____

State: _____ County: _____ Location(T,R,S,1/4): _____

Basin/Structure: _____ Elevation(s): _____

Well/Geologic Notes: _____ Mud Type: _____

Spud date: _____ Completion date: _____ Core Start Date: _____

Geologists: _____ Drillers: _____

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Crucial Times-Weights-Volumes

(USE 24 HOUR CLOCK)

Time of coal bed penetration: _____ Canister + Core mass: _____ (g)

Time at end of coring: _____ minus Canister mass: _____ (g)

Time core reached surface: _____ equals Raw Coal mass: _____ (g)

Time core sealed in canister: _____ minus mass of Ash (dry): _____ (g)

Burette zero point volume: _____ (cc) equals mass DAF Coal: _____ (g)

Reading no.	Baromet. (in.)	Temperature (°F)	Reading Time (date)	Elapsed Time (hours)	Burette (V _f -V _i) (cc)	Vol. @ STP (cc)	Cumulative vol. @STP(cc)	Cum. Vol. @STP,cc/g raw coal	Cum. Vol. @STP,cc/g DAF coal	Reading Notes
1										
2										
3										
4										
5										
6										
7										
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NOTES:

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Figure 4.--An example of a desorption form.

3. ASAP place about 12 inches of core (or chips, if indicated) in a 12-16 inch canister. Note the precise footage of core being placed in each canister. Replace the cap and tighten screws to close the canister. Make sure valve on canister is closed. **NOTE EXACT CLOSURE TIME on desorption form (Fig. 4).**

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Tighten both screws simultaneously to as tight as can be attained using your fingers only.
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4. ASAP place the canister in a water tank to check that it is not leaking. If it is leaking, further tighten screws and recheck by immersing canister. If it is still leaking, open the cap and check that no dirt has contaminated the seal. If necessary, clean or replace damaged gaskets and lightly grease seal with silicone vacuum grease. **EACH TIME THE CANISTER IS RECLOSED THE EXACT TIME SHOULD BE NOTED.** After a good leak test, enter the final closing time on desorption form (Fig. 4).

Desorption

5. It is recommended that the desorbed gas volume be read every fifteen minutes for the first two hours. The reason for such closely spaced measurements is that rapid initial pressure build up in the cylinder can inhibit desorption of gas, and closely spaced measurements are needed to constrain the lost-gas value.

The critical measurements

1) Attach canister to tubing leading to burette; 2) open canister valve; 3) after the fluid in the burette stops descending, note final volume (V_f); 4) write the change in burette volume on desorption form. For each measurement, desorbed gas equals the absolute value of $V_o - V_f$.

Also note on the desorption form at the time of volume measurement: 1) barometric pressure; 2) canister temperature which can be read by inserting the thermocouple into its well in the canister top (be sure to let the reading settle down); and 3) measurement time and date. Note: Use actual barometric reading to calculate STP volume. Do not correct the pressure reading for elevation. It is not important to correct to STP immediately. This can be done later.

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CRITICAL: IF THE CANISTER BEGINS TO SUCK FLUID OR GAS FROM THE BURETTE CLOSE THE VALVE IMMEDIATELY.
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Sucking oxygen into the canister can initiate biological activity (fermentation?) that produces gas on its own and invalidates further desorption measurements. The canister can suck in gas and fluid in the later stages of desorption because barometric and temperature changes can cause the pressure in the canister to be less than atmospheric. **THE DESORBED GAS VOLUME CAN BE MEASURED ANOTHER DAY WHEN CONDITIONS HAVE CHANGED AND(OR) CANISTER PRESSURE INCREASES.**

6. Close canister valve. Disconnect from burette hose. Let burette rezero. measure next cylinder starting at step 5.

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This is the last of the time critical measurements. The measurements indicated below can be done at any time.
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7. Once the initial need for closely spaced burette readings abates, use the scale to measure core+canister mass: enter on desorption form. Subtract canister weight (previously measured and noted on canister label and desorption form). Start to calculate and plot cumulative desorption curve (cumulative cc gas/g raw coal versus elapsed time) on linear-linear graph paper. Only after desorption can the mass of dry ash-free (DAF) coal be calculated so don't worry about that now.

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A canister that has sucked air can be detected by plotting the cumulative gas volume/gram raw coal versus time curve. Usually the curve becomes asymptotic to some value representing final gas desorption volume. In the case of air entering the cylinder the resulting biologic activity can cause the curve to break with this asymptotic approach (e.g. break in slope) and diverge from its normal path.
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After return to the lab

8. Continue desorption until less than 10 cc of gas are recovered each day for a week (Close and Erwin, 1989). This may take several months!
9. Correct the volume measurements to STP (Close and Erwin, 1989; Schaum and Rosenberg, 1966; English, 1984). After correction to STP compute the cumulative gas volume as a function of the square root of cumulative time (decimal hours or minutes) on linear-linear scale graph paper. This plot produces an initially straight line that is used to extrapolate to the Y-axis. The amount of gas lost on the way out of the well is estimated by the Y-axis intercept value which is then added to the final cumulative gas volume.

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For wells drilled with mud, the start of the elapsed time for the loss gas correction is the time the core is lifted off bottom. This zero point for elapsed time is used because it is assumed that desorption starts when the core begins to depressurize. For a well drilled with air and the well is not making water, the zero point is the time of coal bed penetration.
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Residual Gas measurement

10. After desorption is completed, some workers make a measurement of the residual gas left in the coal. This measurement is made by removing the core from the canister and sawing it into quarters. Take one quarter of the core, and place it in gas-tight blender container or ring mill.

Pulverize it and measure the volume of gas given off in the manometer (English, 1984). This residual gas is added to the total of the desorbed and lost gas volumes (all corrected to STP).

Equipment-- core saw; gas-tight pulverizer; desorption manometer

GAS SAMPLING

11. We recommend that the desorbed gases be analyzed for hydrocarbon composition by gas chromatography (GC) and isotopic composition by mass spectrometry (MS). GC identifies what gases are present in the desorbed coal gas, and MS determination of the hydrogen and carbon isotopes can indicate the origin of the gas, e.g. biogenic or thermogenic (see Johnson and Rice, 1990, and the references therein). When collecting gas for chemical or isotopic analysis, we used a hand-held graduated cylinder inverted in a water tank about 18 inches deep (in a configuration similar to Fig. 2). This system is used instead of a burette (Fig. 1) because it is easier to purge the lines and containers of air and minimize cross contamination between canisters. The water should be treated with Zephirin Chloride to prevent biologic activity altering gas composition.

When collecting gas samples use the shortest hose possible to reach from the canister underwater to the inverted graduated cylinder (Fig. 2). This apparatus is similar to that shown in figure 1 of Diamond and others (1986) except we use a much larger water container (an insulated water cooler) and the graduated cylinder is hand-held under water. Also placed in the water is a clean, rubber-stopped Grolsch beer bottle. Fill the bottle with water by immersing it in the tank. Let the filled bottle sink to the bottom. The canister valve is opened and air in the hose is purged into the graduated cylinder that has been filled and submerged in the water (Fig. 2). The water temperature should be close to the ambient canister temperature, so gas volume can be measured under similar circumstances. The canister valve is closed quickly, so that some gas remains in the canister. This volume of gas is recorded and the cylinder emptied of the air and coalbed gas mixture. The time, pressure, and canister temperature during measurement should be noted as discussed above so as to not interrupt the cumulative measurement sequence. The graduated cylinder is refilled with water and submerged again, with the hose from the canister in it. The canister valve is reopened and the remaining gas collected. The volume of gas is noted and a funnel is inserted into the Grolsch bottle. The gas from the graduated cylinder is bubbled up into the bottle through the funnel. Close the rubber stopper on the Grolsch bottle.

This collection procedure can be repeated into the same Grolsch bottle during the next measurement time if more gas is needed for analysis and care is taken not to lose or contaminate the gas already in the Grolsch bottle.

Equipment--Water tank (we use an insulated plastic water cooler), graduated cylinder, short hose, gas sample bottles, funnel.

CORRECTING PROBLEMS AND ERRORS

Missed desorption volume reading: If the volume measured on the manometer is missed (i.e. not written down) it can be estimated from the reading made at the same time on adjacent canisters. This suggestion is based on our experience which shows that readings from adjacent canisters seem to be very similar in volume as long as the core material in them has a similar coal content.

Barometer breakdown: use the barometric pressure reported from a local weather station, weather map in the newspaper and so forth. However, the pressure readings published by the weather service have an elevation correction which must be removed to obtain the raw barometric pressure reading.

Core temperature in canister: After canisters have been stored in a room for many hours and the temperature in that storage room remains stable, then the ambient room temperature can be used to approximate the core temperature. Care should be used to be sure that heater vents, window drafts, and so forth are not affecting the canisters. We have measured temperature differences of over 5°F between canisters in the same box placed under a heater vent, so there can be a significant effect. Also if the packing around the canisters becomes wet, it can greatly influence the temperature because of evaporative cooling.

Emergency desorption containers: Military ammunition containers widely available in surplus stores are pressure tight if the gasket is in good condition. However, a valve must be inserted by drilling and tapping a 1/4 inch pipe thread hole. We also would consider using 4 inch diameter PVC plastic pipe with two end caps threaded on after the core is inserted. As above, one cap would have to be drilled, tapped and a valve inserted for measurement.

EXPECTED RESULTS

Mass of canisters

- The 16-inch plastic canister (SSD Inc.) without coal weighs about 2.7 kg (6.0 lb.).
- The 16 inch plastic canister (SSD Inc.) filled with about 12 inches of nearly pure coal core weighs about 4.8 kg (10.6 lb.). Raw coal mass is about 2.1 kg (4.6 lb.). The same canister design but only 12 inches long holds about 1.7 kg (3.7 lb.) of coal.
- The 36-inch aluminum canister made by the USGS weighs 8.5 kg (18.7 lb.) when filled with a typical coal core. Other 36 inch aluminum canisters weighed about 10.6 kg (23.4 lb.) when filled with coal chips.

Total gas desorbed

- The Anderson coal deposit (subbituminous B rank) in the Powder River Basin has a cumulative desorbed gas content of about 2 cc/g (about 60 scf/ton) (Boreck and Weaver, 1984). This is about 2000-2500 cc cumulative volume of gas. A good general reference of the expected total gas desorbed from United States coal is Diamond et al. (1986).

Rate of desorption

The Anderson coal deposit (subbituminous B rank) in the Powder River Basin desorbed from 26 to 54 percent of total cumulative gas in the first day. By the fifth day, 54 to 86 percent of the total cumulative gas had been desorbed (Boreck and Weaver, 1984).

Volume of gas first day of desorption

Using the rate of desorption measurements from the Anderson deposit, about 500-1000 cc of gas may be desorbed the first day (Boreck and Weaver, 1984) in low rank coal. Higher rank coal and(or) coal from deeper levels can be expected to produce much greater volumes the first day.

SUGGESTIONS FOR FUTURE WORK

1. Use 1-5 PSI pressure gauges on the canisters-- our canisters had 0-15 PSI pressure gauges which showed no response during desorption runs. However, note that pressure measurements from the canister are only useful in a qualitative way and for testing the seal of the canister before desorption.
2. Make large wooden screens and catch cuttings from drilling of a pilot hole and desorb them in a canister to find out if gas is expected in core taken out of the later twin hole.
3. Be sure to make or buy canisters more than 12-inches long. Close and Erwin (1989) suggest a 16-inch long canister chamber for 12 inch portions of core, because if the core is fragmented it can fill a larger volume.
4. When cuttings or core only partially fill up the canister, make a plastic plug that fits snugly into canister chamber to close up dead space. Some workers fill the dead space with water but we feel this could lead to later complications.

ACKNOWLEDGMENTS

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