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Abouna Saghafi
University of Wollongong

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DETERMINATION OF THE GAS CONTENT OF COAL

Abouna Saghafi^{1,2}

ABSTRACT: In coal mining the gas content of coal is required primarily to quantify the gassiness of coal for safe mining, but also to quantify potential greenhouse gas emissions from mining. In Australia the gas content of coal is determined using a direct method, whereby the gas desorbed from solid or crushed coal is collected and the volume and composition of the desorbed gas are measured. The determination of gas content is associated with errors of measurement of the volume and composition of the gas. It is undertaken at several stages of gas desorption. Relative errors and resulting uncertainties of determination are more significant for the estimation of lost gas during drilling and gas remaining in coal following the completion of the standard stages of measurements, whence the rate of gas desorption is significantly reduced. This paper discusses the current Australian method and potential errors and uncertainty associated with this method. A new method of measurement for measurement of remaining gas in coal following the completion of standard gas content testing is also suggested. The new method should allow the release of almost all remaining gas in powdered coal following the last stage of standard gas content testing.

INTRODUCTION

Gas in coal occurs in both adsorbed and free phases. The adsorbed gas, which constitutes almost all desorbed gas from coal, is in a liquid-like state and covers mostly the internal meso- and micro-pores surfaces. The free phase fills the void volume of all pores in coal. The free and adsorbed phases are in equilibrium which is expressed mathematically through relationships between the amount of gas adsorbed and the pressure of the free phase adjacent to the adsorbed layer. These relationships are either derived from an equilibrium kinetic approach to gas adsorption, assuming a monolayer formation mechanism (e.g. Langmuir 1918) or are derived from a potential theory, assuming a pore filling mechanism (e.g. Dubinin 1968).

The gas content of coal refers to the volume of the adsorbed phase in coal as the released gas from coal during gas content testing consists mainly of desorbed gas from the adsorbed phase. The contribution of the free gas phase to the total gas volume in coal is generally small (<10%) for shallow coals (<500 m), however it increases with depth and should be considered at great. Methods of measurement of gas content of coal used in coal producing countries are generally variants of the methods older methods developed in European countries (see Firedamp Drainage, Handbook for the Coalmining Industry 1980). In most methods gas is initially allowed to desorb 'slowly' from solid coal and the volume of the desorbed gas with time is measured. Such curves can be useful to estimate desorption parameters of coal such that a 'characteristic desorption time', otherwise known as τ parameter, is obtained. In early methods the slow desorption is monitored until no 'measurable' desorption takes place (see Kissell *et al.*, 1973; Diamond and Levine 1981 and Diamond and Schatzel 1998). In subsequent methods the slow desorption period is shortened and coal sample is crushed and pulverised (generally to less than 200 μm) to desorb its gas rapidly. Variants of later methods have been used as early as 1960's in Europe (Bertard *et al.*, 1970). Although various methods are used in coal producing countries today, at present there are only limited number of agreed standards across the world for the determination of gas content of coal. In Australia, since the early 1990's work has been conducted to produce standard 'guides' for the determination of gas content. The most recent document, which was developed under the auspice of the Australian Standards, is AS 3980-1999 (1999), which is an update and improvement of a previous document (AS 3980-1991 - 1991). More recently the US ASTM also published a document (D7569-10 -

¹ Visiting Professor, School of Civil, Mining & Environmental Engineering, University of Wollongong, NSW, Australia. E-mail: abouna@uow.edu.au.

² Adjunct Professor, School of Petroleum Engineering, University of New South Wales, NSW, Australia, Email: a.saghafi@unsw.edu.au.

2010) on the determination of gas content with heavy reference to the Australian AS 3980 document. Currently a new standard is being developed under the auspice of the Australian Standards, expected to be available in the course of the current year.

In the next sections of this paper first the current Australian method of gas content testing is briefly described and aspects of uncertainties in gas content determination and generic approaches to estimate these uncertainties are presented. A method of direct measurement of gas volume (remaining gas content) in powdered coal is also presented.

Determination of the gas content of coal – Australian Method

A variant of the fast desorption method, otherwise known as quick crash method, was developed in Australia and used in the early 1990's (for example see Williams *et al.*, 1992). The method was then extensively used and is the basis of the current guide (AS-3980 1999). It has been validated through repeatability and reproducibility experiments and through inter-laboratory round robins exercises (Saghafi *et al.*, 1998; Danell *et al.*, 2003 and Saghafi 2012). This current Australian gas content guide (Standards Australia AS-3980, 1999) describes three main stages for the determination of the gas content of coal. Each stage of gas content testing provides a volume of gas corresponding to that stage. These volumes are represented by Q_1 , Q_2 and Q_3 parameters and their sum is presented by Q_m , or the 'measured gas content' (AS-3980, 1999):

$$Q_m = Q_1 + Q_2 + Q_3 \quad (1)$$

The first component of measured gas content is Q_1 , which is the amount of gas lost from a coal sample during drilling, during its retrieval from the borehole, and before sealing in a desorption canister. Lost gas is not directly measured but estimated from the measured gas desorption rate once coal is sealed in the desorption canister.

The second component of measured gas content is Q_2 , which is the amount of 'slow' desorbed gas from the intact (non-pulverised) coal sample sealed in the desorption canister during its transport to the laboratory and prior to its pulverising at the third stage of gas content testing. In the traditional method of testing called the 'slow desorption method', desorption measurement is conducted until the rate of gas desorption is so small that is not sensed by the measuring device. In the standard method, or 'fast desorption method', however, this stage can be very short and is independent of the rate of desorption at completion of this stage.

The third and last component of measured gas content is Q_3 , which is the amount of 'fast' desorbed gas from the pulverised coal. This component is measured by taking sub-samples from the main coal sample and crushing them to a fine powder using a crusher. Note that gas desorption measurements should be carried out at near atmospheric gas pressures (partial pressure of gas in the crusher bowl should be about atmospheric).

Gas may remain in coal beyond the three stages of gas content testing. This 'remaining' gas, represented by Q_3' , can be determined directly by measurement or indirectly using the adsorption properties of coal. However, its determination is not yet considered by the standard method for gas content testing in Standards Australia AS-3980 guide.

If Q_3' is measured then the total gas content, or Q_t can be calculated. It is the sum of the 'measured' and 'remaining' gas contents:

$$Q_t = Q_m + Q_3' \quad (2)$$

Figure 1 shows the relationship between the various gas content components and coal gas adsorption properties (adsorption isotherm). The free partial gas pressure in pores should reduce to a certain level to allow the release of the adsorbed gas from coal.

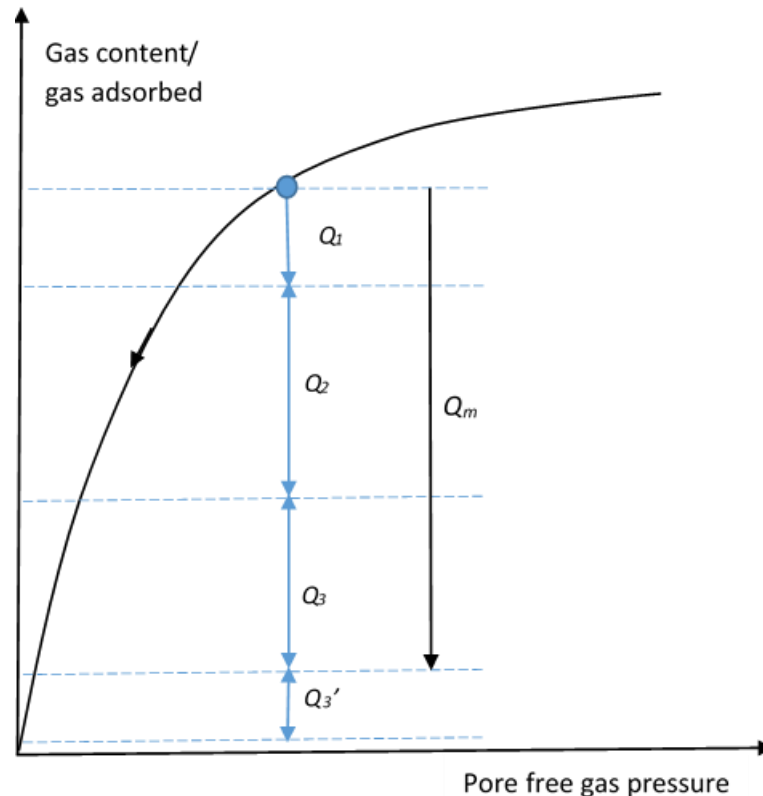


Figure 1: Relationship between the gas adsorption isotherm and the components of gas content, not to scale

Note that all components of gas content are expressed in terms of gas volume per unit mass of coal (m^3 per tonne), where volumes are presented for standard temperature and pressure (STP) conditions. For the Australian standard (Australian Standards AS-3980, 1999), the STP conditions are 101.325 kPa and 20°C. Note that ASTM D7569-10 (2010) recommends using 101.3 kPa and 15°C for gas volume calculation so to conform to API standards.

Measurement of the desorbed gas volume from coal (during all three stages) is carried out using water displacement in an inversed cylinder held above a water basin (Figure 2). Prior to measurement a level of suction (vacuum) is kept in the head space of the cylinder so that a water column in the cylinder is formed above water level in the basin. Desorbed gas is allowed to flow into the head space, which displaces water down and reduces the water column height, so that the volume of desorbed gas can be measured. To reduce the dissolution of desorbed gas (particularly CO_2) in water, acidified water is used and desorbed gas enters the measuring cylinder from the top (Figure 2).

Uncertainties associated with the determination of gas content

Only limited studies have been undertaken to evaluate the uncertainty of this method for the determination of gas content. Saghafi et al (1998) studied the variation (reproducibility) of measuring the gas content of coal by comparing results from three Australian laboratories, and found that there was $\pm 15\%$ variation in the values of gas content for a suites of similar coal samples measured concurrently by these laboratories. The samples were produced from the same cores obtained from in-seam drilling. Coal core pieces were mixed to produce similar sub-samples for participating laboratories.

The uncertainty of gas content determination within a single laboratory depends upon field and laboratory equipment and measuring tools used but also on operators' skills and judgement. For example the type of graduation of the measuring cylinder and the material it is made of, i.e. whether it is made of glass or plastic, can all affect the measurement error and the level of uncertainty of gas content determination. Operator judgement influences the final uncertainty. For example, for the determination of Q_1 it is required that the operator evaluates the rate of initial desorption from measurements of the desorption curve in the field and time zero, that is a point in time when the coal started to release its gas in the exploration borehole. Different operators may have different judgement on the value of parameters of desorption and hence end up estimating different values for Q_1 . Similar concerns are also raised for Q_2 and Q_3 and when one should decide that Q_3 is completed and how much gas may be still in coal.

In the next section some of the uncertainties associated with estimation of Q_1 and measurement of Q_2 and Q_3 are discussed.

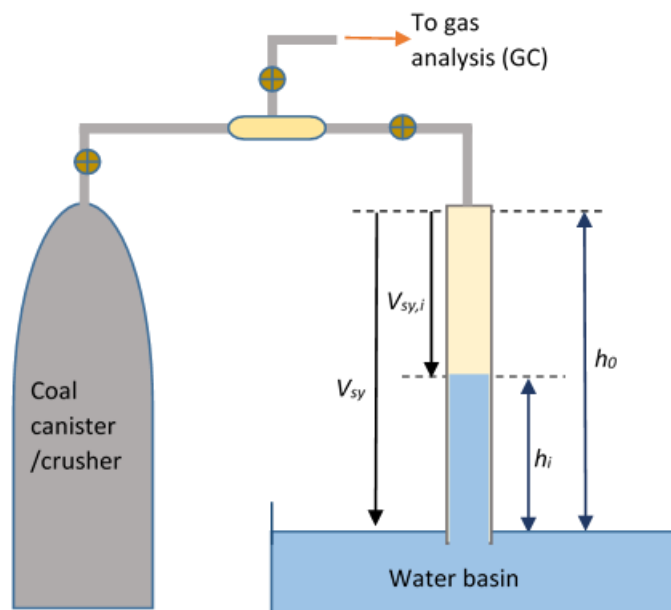


Figure 2: Schematic of the method of measurement of desorbed gas volume by water displacement, not to scale

ESTIMATION OF LOST GAS (Q_1) AND ASSOCIATED UNCERTAINTIES

Gas is released from coal as soon as the water pressure falls to below the combination of capillary and free gas pressures in the coal pores. Lost gas is the gas which is released in the borehole and following the retrieval of coal at the surface prior to sealing the sample in a desorption canister. This gas is lost because as it can not be measured. However, the amount of lost gas may be estimated assuming that desorption follows a certain kinetic which can be established from an initial gas desorption curve from coal sample in the canister (accumulated volume of desorbed gas is measured against time).

In estimating the lost gas, one important input parameter is time zero or the time when coal starts to release its gas in the borehole. In AS-3980 (1999) time zero is defined as the time mid-way between the start and completion of coring for the section to be sampled. However, this can be subject to discussion as the length of the water column and piezometric pressure can significantly influence the onset of desorption. The other parameter in question is the temperature, which changes as the sample is retrieved from the borehole. The elapsed time since time zero, can be called 'lost time' and in this paper it is expressed by the variable t_e . The temperature of drilling fluid may be used as the desorption

temperature. This aspect has not been commented on AS-3890, and to opinion of the author should not be considered a major source of error.

Initial rate of desorption as basis for estimation of Q_1

Experimental data suggest that gas desorption from coal follows a first order diffusion equation and hence the evolution of gas desorption in a canister can be explained in terms of diffusion physics. In this regard, the cumulated volume of gas released from coal (q) over a period of time is proportional to the square root of the product of effective diffusivity (D_e) and the elapsed time (t) since the start of desorption or the start of the measurement of desorption:

$$q(t, D_e) = \alpha \sqrt{D_e t} \quad (3)$$

If the diffusivity of gas in coal remain constant for the period of measurement then the cumulative volume of gas will be proportion to square root of time only and the volume q is a linear function of square root of time. This is what is observed during field measurement of gas desorption which show that for short period of time the hypothesis of the linearity of the volume of gas against the square root of time is held. Assuming that the effective diffusivity of gas in coal has not changed since the start of desorption in the borehole and the desorption measurement in the canister then the lost gas can be estimated by back extrapolation of this line. If after a time t_e (lost time) coal is sealed in the desorption canister and the measurement of the volume of desorbed gas (q) is plotted against square root of time t , according to Eq. 3, the shape of curve should follow the following equation:

$$q(t) = k\sqrt{t} - Q_1 = k(\sqrt{t} - \sqrt{t_e}) \quad (4)$$

Note that the origin for the time axis in Eq. 4 is the time when coal started to release its gas in the borehole (not the time when the coal is sealed in the canister and measurement starts). The slope of the regression line, or coefficient of proportionality is $k = \alpha D_e$. This latter is determined from the data from the initial desorption curve measured in the field. Coefficient k depends on environmental conditions, mainly temperature of coal, but also on the initial gas content and diffusion properties of coal.

Uncertainty of determination of Q_1

Besides the error associated with measurement equipment, two main parameters affecting the results are the value of lost time t_e , and the slope of initial desorption line k in Eq. 4.

The assumption of linearity of the cumulated volume of desorbed gas with the square root of time and the back extrapolation of the regression line to estimate the lost gas is only valid for short values of t_e and error of estimation increases with larger value of t_e . Hence, the lost time shall be kept as short as the operation would allow. Evaluation of the true t_e is difficult and can only be based on assumption. The regression line (k) can vary because of environmental conditions; with coal drying at the surface k increases. Similarly an increase (or decrease) in coal temperature at the surface can increase (or decrease) the value of k . This often happens in the field after coal is placed in the canister. It takes some time for coal to reach the assumed in-situ (pre-set) temperature of the water bath in which the coal canister is placed.

Figure 3 shows an example of the measurement of initial rate for the determination of Q_1 . In this example we assume that the value of the lost time ($t_e = 20$ minutes) is reasonably correct. However, for the slope of gas desorption curve, we have two values ($k_1 = 0.22$ and $k_2 = 0.50 \text{ m}^3/\text{t per min}^{0.5}$). It could be asked how the operator should evaluate the data and then work out a most appropriate value for k to determine lost gas volume? One reasoning could be that gas desorption has a lower rate at the start of desorption because coal has not reached the pre-set temperature (in-situ temperature) in the water

bath. Once coal reaches its pre-set temperature then desorption should take place at in-situ temperature. Operator may then choose the slope (k_2) for calculation of Q_1 , which yields an estimate of $Q_1 = 2.2 \text{ m}^3/\text{t}$ for lost gas.

Note that t_e and k are two independent variables for the estimation of Q_1 and the choice for value of t_e does not affect the choice for value of k . Therefore the calculation of Q_1 is only affected by different rates of desorption (slopes of desorption lines).

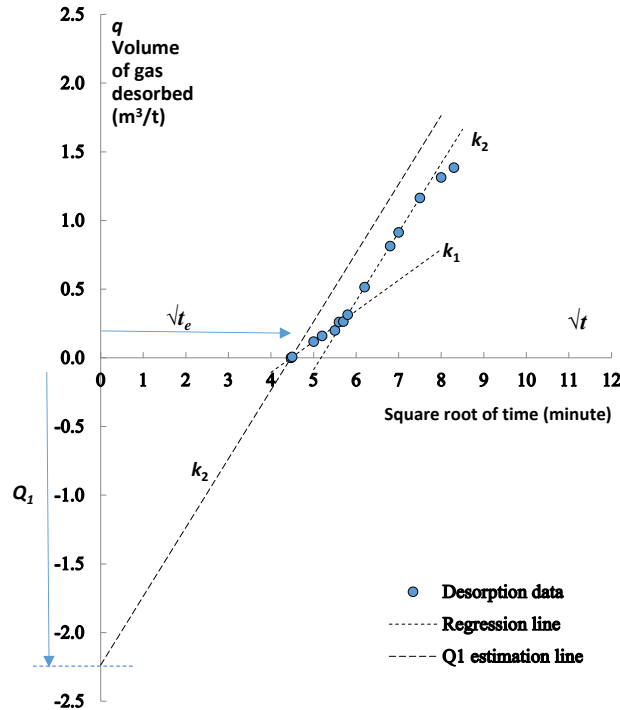


Figure 3 Determination of lost gas Q_1 and changes in the initial canister gas desorption rate

A generic method for the calculation of the uncertainty of Q_1

Various estimation error affect the level of uncertainty of Q_1 . Besides the errors associated with equipment and measurement tools (such as the thermometer for measuring the temperature or the measurement of the volume of gas using a measuring cylinder) there are larger uncertainties associated with with accurate evaluation of the lost time and the rate of initial desorption (t_e and k in Eq. 4). If the error of evaluation of these variables is expressed in terms of partial derivatives, the variation in the value of Q_1 due to the variation in evaluation of values of t_e and k is:

$$\delta Q_1 = \frac{\partial Q_1}{\partial t_e} \delta t_e + \frac{\partial Q_1}{\partial k} \delta k \tag{5}$$

Using partial derivation of Eq. 4 and assumption of quadratic additions of individual uncertainties (see for example JCGM, 2008), the uncertainty of Q_1 associated with uncertainties of lost time and the rate of desorption is:

$$\delta Q_1 = \pm \sqrt{\frac{k^2}{4t_e} (\delta t_e)^2 + t_e (\delta k)^2} \tag{6}$$

For example for the estimation of Q_1 in Figure 3, assuming that the lost time and desorption rates are expressed as $t_e \pm \delta t_e = 20 \pm 4$ minutes, and $k \pm \delta k = 0.5 \pm 0.1 \text{ m}^3/\text{t per min}^{0.5}$, respectively, then the uncertainty of estimation of lost gas is $0.5 \text{ m}^3/\text{t}$ and the lost gas is $Q_1 = 2.2 \pm 0.5 \text{ m}^3/\text{t}$.

MEASUREMENT OF SLOW (Q_2) AND FAST DESORBED GAS (Q_3) AND ASSOCIATED UNCERTAINTIES

Once coal is sealed in a desorption canister after its retrieval from borehole any gas desorbed from coal is part of the Q_2 stage of gas content testing including field gas desorption for estimation of Q_1 . Slow desorption may be left to continue until the operator decide that the sample is ready to go through the Q_3 stage of measurement and fast desorption by crushing the sample either in the same canister or in a separate crusher.

Measurement of Q_2 is generally straight forward and if the system is gas tight and properly sealed then, the uncertainty of measurement would be mainly related to the uncertainty in evaluating the coal mass, the void volume in the system and the measurement of pressure and temperature. Measurement of the volume of gas released for both Q_2 and Q_3 stages of gas content testing are generally measured using the same technique (water displacement as shown in Figure 2). The total desorbed gas in stage Q_2 is determined by adding successive volume increments (water displacements) over the period of measurement:

$$Q_2 = \frac{1}{m} \sum_{i=1}^n \Delta V_i \quad (7)$$

where m is the mass of sample, and

$$\Delta V_i = V_i - V_{i-1} \quad (8)$$

Volumes V_i and V_{i-1} are the volume of gas occupying the system void at two consecutive steps of $i-1$ and i of the measurement. The volume of gas at STP conditions at step i is:

$$V_i = \frac{T_a}{P_a} \left(\frac{V_{void}}{T_{void,i}} + \frac{V_{cy,i}}{T_{cy,i}} \right) p_i \quad (9)$$

where P_a and T_a are STP absolute pressure and temperature ($^{\circ}\text{K}$) and $T_{void,i}$ and $T_{sy,i}$ are absolute temperatures in the system (void volume in the desorption canister/crusher and tubing) and in measuring cylinder at step i of measurement of gas volume. V_{void} is the void volume in the system (void volume in coal canister and tubing and fittings) and V_{cy} and $V_{cy,i}$ are the full volume capacity of measuring cylinder and empty volume of this cylinder at step i of measurement. Pressure p_i is the absolute pressure of gas in the system. Note that this pressure is smaller than atmospheric pressure due to the suction exerted by the water column (Figure 2). For instance if at step i the height of water column is h_i , then pressure p_i is:

$$p_i = P_a - ah_i \quad (10)$$

Note that if pressure is measured in kPa and height in metre then $a = 9.8$. The height of the water column h_i can be measured directly or estimated from the following equation if the graduation of the cylinder are in unit of volume (Figure 2):

$$h_i = \left(1 - \frac{V_{cy,i}}{V_{cy}}\right) h_0 \quad (11)$$

where h_0 is the height of the water column at the start of measurement (when measuring cylinder is fully filled with water) and h_i is the water column height at current step i of measurement of water displacement in the cylinder. The two volumes V_{cy} and $V_{cy,i}$ are total volume of the measuring cylinder and its void volume at step i of measurement, respectively (Figure 2).

For the Q_3 stage of gas content testing, the same calculation can be carried out as the same method of measurement for the volume of the desorbed gas is used. However, the results are also affected by the

partial pressure of the seam gas in the system as well as temperature of the coal (which can become quite high during crushing).

Another factor is the final particle size distribution of powdered coal. It is recommended to keep temperature and pressure at near STP conditions if possible and that the particle size be below 200 μm . Smaller size have larger exposed surface and most desorbable gas would be released during crushing. The partial pressure of gas near atmospheric pressure would produce the 'measured gas content' value near or equal to the desorbable gas content value of coal.

Uncertainty of measurement of Q_2 and Q_3 : effect of equipment used and void volume

Many factors affect the volume of gas desorbed during Q_2 and Q_3 measurements. Some factors have effect on the physics of desorption and increases or decreases the amount of gas that can be released from coal; factors such as the moisture content of coal and excess water vapour in the system or the composition of air and gas in the void space and partial pressure of seam gas (whether the canister/crusher is rinsed and filled with an inert gas before measurement) can have significant effects on desorbable gas from coal. Another aspect of uncertainty is related to the equipment used such as the type and accuracy of the measuring cylinder or the remaining volume of void in the system once the coal sample has been placed into the desorption canister. In this section a simple method is presented for the calculation of the uncertainty of gas content due to the uncertainty of the measured gas volume, which involves the void volume in the system and in the measuring cylinder.

For the sake of simplicity assume that the temperature is kept the same across the measurement system (i.e. $T_{\text{void},i} = T_{\text{cy},i} = T_i$), so that Eq. 9 could be simplified to:

$$V_i = \frac{T_a}{P_a T_i} (V_{\text{void}} + V_{\text{cy},i}) P_i \quad (12)$$

The uncertainty of the volume of desorbed gas using Eq 12 depends on the uncertainties associated with values of temperature (T) and p_i which change in each measurement step. It also depends on the uncertainty of the void volume in the system, and the uncertainty of the volume of empty space in the measuring cylinder ($V_{\text{cy},i}$).

Note that p_i is not an independent variable but is dependent on $V_{\text{cy},i}$, hence for the uncertainty of determination of the volume of desorbed gas associated with $V_{\text{cy},i}$ and p_i the effect of the latter can be evaluated in terms of the uncertainty in the measurement of $V_{\text{cy},i}$.

If we use a partial derivation technique and assumption of quadratic additions of individual uncertainties, then the variation in the calculated volume of desorbed gas in the system would be a function of the uncertainties in data on the void volume in the system (δV_{void}) and the void volume in the measuring cylinder ($\delta V_{\text{cy},i}$) as follows:

$$\delta V_i = \pm \frac{T_a P_i}{P_a T_i} \sqrt{(\delta V_{\text{void}})^2 + (\delta V_{\text{cy},i})^2} \quad (13)$$

Note that the uncertainty due to volume of the change in pressure (δp_i) is ignored in developing the above equation (Eq. 13). The uncertainty in volume of desorbed gas calculated in Eq. 13 is associated solely with the uncertainty of evaluating the void volume in the system and the uncertainty in reading the amount of water displacement in the measuring cylinder.

DETERMINATION OF THE REMAINING GAS IN COAL (Q_3')

Once Q_3 measurement is completed there is still a chance that volumes of gas remain in coal. Depending on the purpose of gas content testing, the determination of the remaining gas volume may be required (e.g. for the estimation of greenhouse gas emissions from mining). The amount of Q_3' (remaining gas) left in coal depends on a multitude of factors such as seam gas partial pressure in the system, moisture and diffusivity properties of the coal. Large volumes of gas can also be retained because of wettability properties of coal (contact angle of gas and water interface with coal surface). It is often suggested to determine Q_3' indirectly using the adsorption isotherm (Figure 1) and the final partial pressure of the seam gas in the Q_3 measuring apparatus. This implies that gas partial pressure in the Q_3 apparatus be determined and an isotherm of powdered coal be measured following the Q_3 measurement. However, this indirect method for determining Q_3' only relies on the partial pressure and the isotherm. The measurement of these parameters would introduce additional uncertainties.

Direct method of measurement of Q_3'

Over the last few years we have developed a direct method for measuring Q_3' (Saghafi, 2011; Saghafi, 2012). In this direct method the crushed coal in the Q_3 apparatus is vacuumed and then flushed with an inert gas (e.g. N_2 , He or Ar depending on the GC carrier gas). Coal is then left to desorb its gas over a period of time. For most coals gas desorption can be more complete and enhanced if the apparatus is pressurized above atmospheric pressure. Once the pressure and temperature in the apparatus reach their equilibrium, gas pressure and temperature are recorded. Gas samples are also collected from the apparatus and measured for the desorbed gas composition (Figure 4). Using data on gas concentration of seam gas in the system (c , vol/vol), total pressure (p) and temperature of the gas in the apparatus Q_3' can be calculated as:

$$Q_3' = \frac{1}{m} \frac{T_a}{P_a} c V_{void} \frac{p}{T} \quad (14)$$

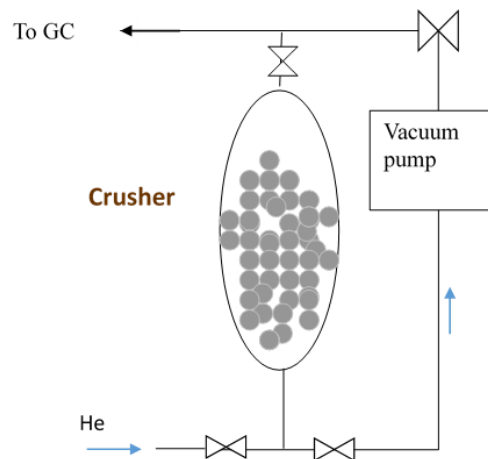


Figure 4: Measurement of the remaining gas (Q_3') using a direct method; gas is allowed to release from pulverised coal over a period of time into an inert gas atmosphere (modified from Saghafi, 2011)

CONCLUSIONS

Gas content is the most important parameter for characterising a coal seam in relation to gas emissions from mining. In this regard the current Australian method for the determination of gas content requires revision to reduce the uncertainty and increase the accuracy of the method. In light of the new understanding of the interaction between gas and coal and the adsorption and desorption mechanisms,

enhancement can be made to the current method and new approaches are required to increase the accuracy and measurability of gas content. Methods for the determination of uncertainties of gas content using the current method should also be part of the new methods for determination. Inventories on gas emissions require total gas in coal to be determined. Based on these new requirements, particularly in relation to greenhouse gas emissions, current methods for the direct measurement of remaining gas in coal should be further developed.

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