

# Accuracy of Measurement of Gas Content of Coal Using Rapid Crushing Techniques

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## SUMMARY

The rapid crushing technique for the measurement of gas content of coal consists of accelerating the rate of gas desorption from coal by crushing. This technique allows the determination of gas content in the space of hours rather than days or weeks as required for the traditional slow desorption technique. The crushing technique is used particularly when gas content determinations are urgently required for mine safety purposes. The method is now routinely used in underground coal mines of NSW and to a lesser extent in QLD. Despite the rapidity and efficiency of this method, concerns have been raised upon the reliability of the method to deliver accurate results. Experience in Australia has shown that there is in fact some discrepancy in results between gas laboratories using the rapid crush technique. The development of rapid crushing techniques for seam gas content measurements in Australia and the main causes of variability in the gas content test results were investigated. The gas laboratories participating in the gas content testing were CSIRO Division of Coal and Energy, BHP Technical Services and GeoGas Pty. Ltd.

## INTRODUCTION

Gas content is probably the most important parameter to be quantified in order to characterise a coal seam both from mine safety and gas recovery viewpoints. Worldwide, numerous direct and indirect methods of determining the gas content of coal are practiced. The direct method is based on the direct measurement of gas volume evolved from coal whereas the indirect method consists of measuring other coal properties and using the established relationships between these parameters and gas content to evaluate the latter. The two principal variants of the direct method are the slow desorption technique and the quick crush method.

In Australia the slow desorption method was widely used for the last 20 years. More recently the quick crush method was introduced and used initially for residual gas content determination followed by its current use for full gas content determination. While this method is fully operational, some concerns were raised by industry on variability of gas content results between gas laboratories using the quick crushing method. This paper addresses and quantifies some of the factors, which are identified by the authors to have influence on the gas content results using this method.

## DEVELOPMENT OF QUANTITATIVE MEASURE OF SEAM GAS CONTENT

In Australia, initial rudimentary estimates of coal seam gassiness were made in the 1960s and 1970s during exploration drilling by coal geologists in the field using the Hargraves test. This consisted of quickly enclosing a coal core still in the inner tube of the core barrel in a sealed vessel for 24 hours and then sampling the gas released. On the basis of the gas analysis the level of gassiness of the coal bed was qualitatively determined by examination of the ratio between air and gas in the sample. In the early 1980s increased coal production rates and deeper mining conditions demanded greater interest in the gas content of coals ahead of the working face. A modified version of the USBM Direct Method (McCulloch and

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Diamond, 1976) was adapted by operating gas engineers and geologists in the industry to quantify the seam gas content ahead of mining (Battino and Doyle, 1983).

The need to standardise and document guidelines to provide industry with an acceptable, reliable and reproducible method of gas content determination led to the formation of a professional working group of industry personnel in March 1984. In 1986, an initial draft document reflecting the views of committee members was prepared and reviewed. This resulted in a document entitled " Guide to the determination of desorbable gas content of coal seams - Direct Method " which was circulated for public comments and recommendations. In 1991, the Australian Standard document AS3980 was issued as an acceptable working guide for the determination of seam gas content using the Direct Method. This was then used as the operating standard for Australian industry (Standards Association of Australia, 1991).

In 1995, the SAA working group was reconvened to discuss and review recommended changes to the existing document with particular emphasis on the following issues,

1. precision and reporting of the test results;
2. sampling and sub-sampling of materials tested;
3. the inclusion of the Quick Crush method; and
4. the factors affecting the accuracy of seam gas content determination.

Over the next 18 month period, regular committee meetings were held to examine and debate the above issues and this has led to the current Pre Publication Review Draft of a new AS 3980 Guide which is in its final completion stage. The factors affecting the precision and accuracy of seam gas content determination are included in order of importance (as deemed by the working group) in Appendix A of the new document. While a test result accuracy of 10% is indicated in the Guide, this has yet to be proven. To this end, an ACARP funded project aiming to study and quantify the effects of these factors was operated by the CSIRO Division of Coal & Energy, with support from the BHP Technical Services and GeoGas laboratories.

## **DIRECT METHODS OF SEAM GAS CONTENT MEASUREMENT**

### **Slow desorption method**

In Australia the slow desorption method of gas content testing has been used for almost 20 years. This modified USBM method (McCulloch and Diamond, 1976) consists of enclosing a coal sample in a sealed container and measuring the volume of the gas evolved. Gas desorbs from coal naturally and the rate of desorption depends uniquely upon the coal gas desorption characteristics but a single gas content determination may take up to a few months to complete. This time span can introduce various sources of error, which Lama (1995) examined and quantitatively assessed in detail. There are three main sources of error uniquely related to the slow desorption method.

#### **1. *System leakage***

Clearly, the longer the period of time required to determine the seam gas content in the laboratory, the more chances of error due to system leakage, repetitive visual errors in taking desorption readings and mistakes in opening or closing valves during testing.

#### **2. *Solubility of CO<sub>2</sub> in water***

Significant volumes of CO<sub>2</sub> gas can be dissolved by the water of the gas collection system (even when using acidified brine) particularly if the gas is bubbled up through the water from the bottom of the test cylinder. This depends on the size of the bubbles influenced by the exit nozzle diameter and the height of the water column. The residence time of

the desorbing gas contained by the water also plays a role as the dissolved CO<sub>2</sub> diffuses through the water column to the atmosphere. The slow desorption method is thus more prone to this error.

### 3. *Effect of changes in temperature*

During long periods of testing (such as weeks or months), there are likely to be substantial variations in ambient temperatures which can have a pronounced effect on the final gas content value obtained. It is generally recommended that tests be carried out in temperature-controlled laboratories at 20 °C. Tests carried out by R.D. Lama at temperatures in the range of 20 to 40 °C indicated errors of 0.135cc/g/°C for CO<sub>2</sub> and of 0.07cc/g/ °C for CH<sub>4</sub>.

### **Quick crushing method**

The continuing increase in the rate of heading development and longwall face advance has forced the mine gas laboratories in Australia to seek faster ways of determining the gas content of coal. Some researchers suggested to estimate gas content of coal based on the initial gas desorption rate and using a database for correlating the initial gas release to the total desorbable gas content (Williams, 1997). Lama (personal communication, 1993) has suggested to use gas release rate from wet and dry drill cuttings to estimate gas content of coal. However in both of these procedures gas content is not directly measured and intensive site specific correlation between gas content and other parameters are required to be established before using the method.

The most reliable way of rapid and direct gas content determination is to crush coal, which significantly increases its gas, desorption rate. The crushing method has been widely used in most European countries with deep coal mining. The method was first developed and applied in Western Europe (Bertard et al., 1970). The rapid desorption or quick crush method has been effectively used in Australia more recently (Williams et al., 1992). The method consists of accelerating the process of gas desorption by crushing coal in a sealed container. Using this method, the total seam gas content can be determined in a space of 2 to 3 hours and this is now extensively used by the most active gas laboratories in NSW and is also rapidly progressing in Qld.

There are six major steps of this method.

1. The coal sample or core obtained from the selected underground site is first used to estimate lost gas during drilling (Q<sub>1</sub>). This is achieved by measuring the gas desorbed for a period of 20 to 30 minutes. The volume of gas released underground (Q<sub>2U</sub>) must be recorded for determination of total desorbable gas content.
2. At the completion of the underground measurement, the canister is sealed and transported to the gas laboratory where the volume of gas evolved since sealing at the underground site is now measured (Q<sub>2L</sub>).
3. Some laboratories need to subsample coal (200 to 400 g) for crushing by transferring coal from the transport canister to the crusher. To enable the calculation of the gas lost in the period between opening the transport canister for sub-sampling and sealing in the coal crusher, the rate of gas release should be determined. This is achieved by measuring the gas evolved over a period of time. The gas released during this time and transfer time is Q<sub>2T</sub>.
4. Coal sample is then crushed and gas released during crushing, Q<sub>3F</sub>, is determined.
5. The total desorbable gas content (Q<sub>TD</sub>) is then determined by adding all the various gas components:

$$Q_{TD} = Q_1 + Q_{2U} + Q_{2L} + Q_{2T} + Q_{3F}$$

6. In some instances gas may continue to be released from crushed coal for a longer period. This gas (Q<sub>3F'</sub>) can be measured if total gas content, Q<sub>T</sub>, is required.

The gas content values are generally reported in normal conditions (20 °C temperature and 1 atm. pressure).

### *Advantages of the quick crushing method*

The significant advantages of this method include:

1. Very fast evaluation of the total gas content of the coal at a particular test site in the mine, essential for mining authorisation from the Mining Inspectorate and for gas predrainage purposes.
2. Significant reduction in the chances of leakage, dissolution of CO<sub>2</sub> in water and oxidation of the coal during testing.
3. Quick turnaround in freeing up the available field and laboratory equipment which can then be reused for other tests.
4. Reduction in laboratory cost because of shorter time for monitoring, less frequent gas volume and composition measurements and better resources utilisation.

## **INTER-LABORATORY COMPARISON OF GAS CONTENT MEASUREMENTS USING THE QUICK CRUSH METHOD**

### **Field measurements**

In order to achieve the desired level of comparison of gas content test results from the different operating laboratories, drilling and coring were conducted at a number of underground test sites in various NSW collieries. A total of 38 bore cores were collected from 8 mine sites situated in 5 collieries of the Illawarra and Hunter Valley coalfields.

At each site, the work consisted of drilling a borehole from a development heading into a virgin coal area and then coring end to end at regular spacing of 1.5 to 2 m. Most cores were obtained at a depth of 10 to 20 m from the rib, with the exception of one case where 5 cores were taken at the depth of 50 to 60 m. From each borecore, coal samples were apportioned to the three participating laboratories so that the samples were uniform and representative across laboratories. For each core section obtained, underground readings for  $Q_1$  were undertaken using one of the canister samples while the other two canisters were left open. After the completion of  $Q_1$  readings, all coal sample canisters for that particular core section were sealed, transported and delivered for testing to each of the participating gas laboratories as soon as possible (generally 2 to 12 hours after underground testing).

### **Laboratory measurements**

Once the coal samples were received by each laboratory, the coal canisters were tested for leakage and the gas volumes released during transport and in the laboratory were measured. This quantity defined as  $Q_L$  is similar to  $Q_{2L}$  of quick crushing method described in previous sections. The coal samples were then placed in the crusher and the gas volume released during crushing was measured. This quantity defined as  $Q_C$  is similar to  $Q_{3F}$  in quick crush method described in previous sections.

The three operating laboratories use three different sets of coal canister and crushing equipment. The volumes of the canisters and crushing systems are also different. BHP and GeoGAS laboratories use larger coal transport canisters and crusher and must open the coal transport canister to subsample a part of the coal for crushing. With the CSIRO method, the coal transport canister can be directly mounted on the crusher and coal is crushed without being removed from the underground canister. GeoGAS uses a ring crusher whereas BHP and CSIRO use ball mill crushers.

### **Variability in measured gas content**

The gas content results across participating laboratories were compared on the basis on the value of  $Q_L+Q_C$ . This is the total volume of gas desorbed from the time the coal sample was sealed underground in the transport canister until the

crushed coal was removed from coal crusher. A comparison of the  $Q_c$  or  $Q_L$  values alone would give rise to errors as the timing to start coal crushing was not strictly the same across the three participating laboratories.

In Fig. 1, the results of gas content measurements undertaken for the 38 borecore samples are presented. The gas contents are given in terms of  $Q_L + Q_c$ . The mean gas content for a given borecore is the arithmetic average of gas content values obtained by participating laboratories for that bore core. The bisector corresponds to the mean gas content. A measure of the variability in gas content measurements can be presented by the absolute or relative distance of the gas content measured by each laboratory from this line. This approach was adopted in this study and all individual values are compared to the mean values of gas content.

To estimate the variability in gas content results two terms: 'absolute variability' and 'relative variability' were defined. The absolute variability for a borecore is equivalent to the standard deviation of gas content results from the mean gas content for that borecore. The relative variability of results for a borecore corresponds to the standard deviation normalised over the mean gas content of the borecore. In Fig. 2 the absolute variability against mean gas content is shown. As can be seen, there is no strong trend in the data. However there is a weak trend in the scattered data, suggesting that a larger seam gas content value would produce a larger absolute variation between laboratories. The graph also shows that the maximum variation for all measurements except for one was under 1.2 m<sup>3</sup>/t. In Figure 3, the graph of relative variability versus mean gas content is shown. This indicates that the relative variability in gas content across participating laboratories has no correlation with gas content and in all cases except two remains under or near 15%.

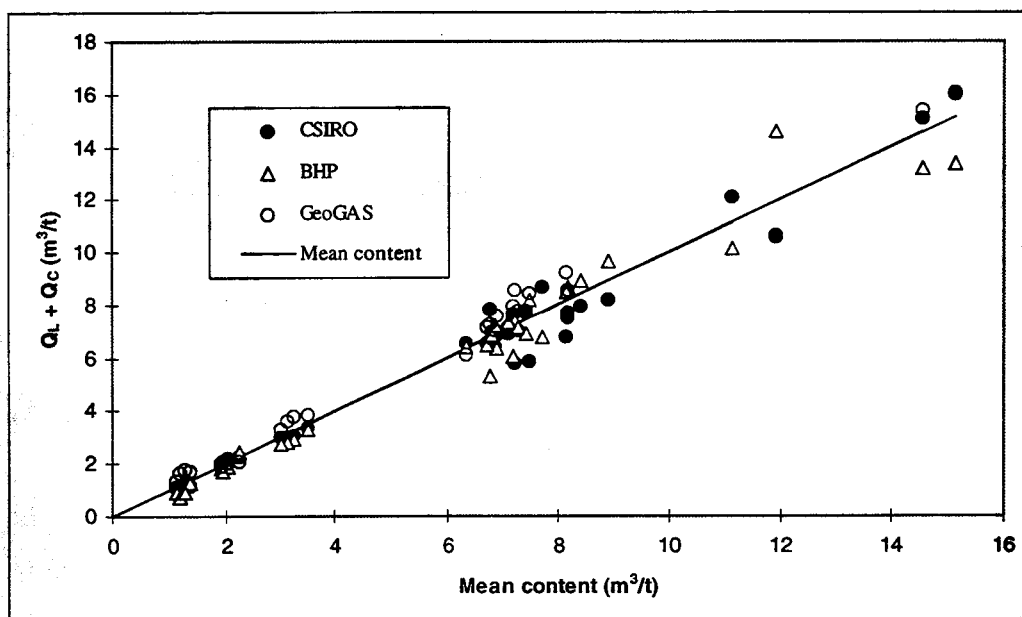


Fig. 1 - Borecore gas content measured across participating laboratories

#### Discussion on causes of variability in gas content measurements

Gas content of borecore samples measured by the three laboratories showed a relative variability of 15%. The calculation of variability was based on the standard deviations from mean gas contents, consequently in terms of relative variability between two individual labs the upper limit was as high as 30%. This amount of variation is rather high and in the course of the study, some causes of the variability were removed. While tests were conducted by the various laboratories to identify leakage prior to commencing desorption, no leaks were reported. Three sources of variation in gas content results across the participating laboratories were identified.

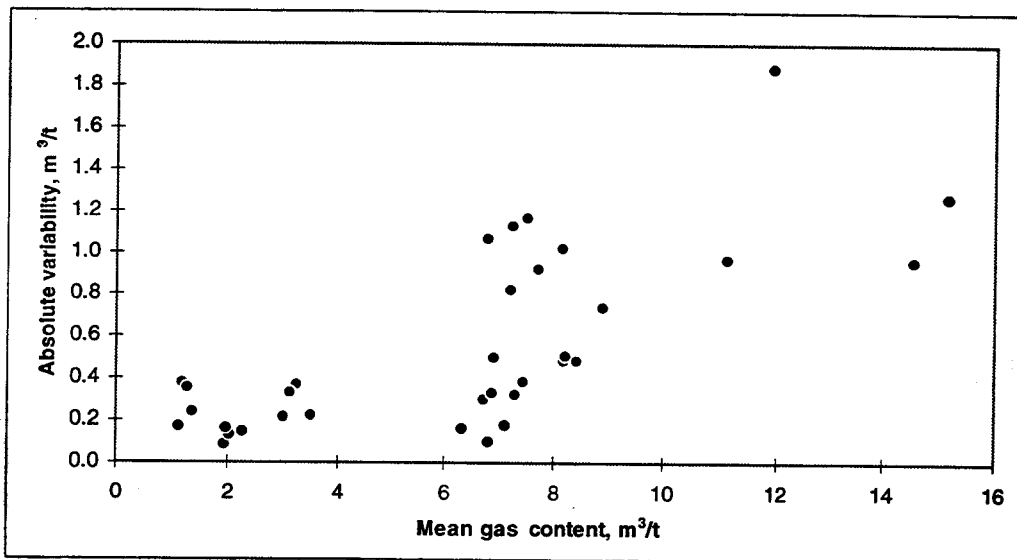


Fig. 2 - Absolute variability in gas content measured across laboratories

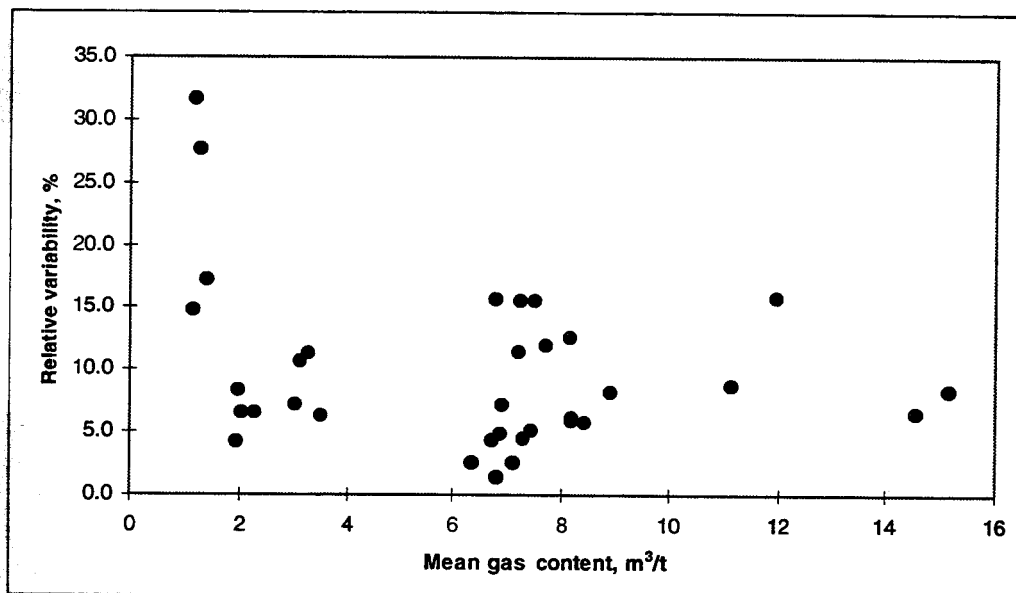


Fig. 3 - Relative variability in gas content measured across laboratories

*Effect of high solubility of CO<sub>2</sub> in water*

Substantial error can occur when high proportions of carbon dioxide (CO<sub>2</sub>) are present. CO<sub>2</sub> gas is highly soluble in water and current methods of measuring the gas volumes by water displacement in an inverted cylinder can only increase the variability in gas content results across laboratories. The CO<sub>2</sub> loss is especially high when gas is fed from the base of the test cylinder and has to travel through the water column. CO<sub>2</sub> is also lost at the interface between water column and gas. The factors, which determine the quantity of gas loss, are the residence time, the area of water-gas interface in the measuring cylinder and the area of the water-air interface in the water tray.

The traditional method to prevent CO<sub>2</sub> dissolution is to use an acidified water solution instead of pure water. This however merely inhibits the formation of carbonate ion, which is very soluble in the acidified brine. In an experiment at the CSIRO gas laboratory, 500 mL of pure CO<sub>2</sub> gas was injected from the bottom into an inverted 2L measuring cylinder filled with 1 liter of either distilled water or acidified brine. The height of the water column was 20 cm and bubbling of the gas

through the water column took approximately 60 seconds. The gas volume lost traveling in pure water was 95 mL or 19 % and in acid brine dissolution was 73 mL or 15%.

The effect of gas standing over a long period of time over acidified water in an inverted gas collection cylinder was also studied. Gas containing CO<sub>2</sub> was injected from the top of the cylinder and gas samples were collected and measured at regular intervals. It was found that the gas composition changed with time as the CO<sub>2</sub> dissolved. Gas initially containing 40% CO<sub>2</sub> and 60% CH<sub>4</sub> only had 27% CO<sub>2</sub> after 20 hours while for gas starting at 89% CO<sub>2</sub> and 11% CH<sub>4</sub>, the level fell to 68% CO<sub>2</sub>.

In an another experiment, the effect of separating gas and liquid phases on gas loss was investigated. A layer of raw linseed oil of 4mm thickness was placed on top of the acidified brine and then gas containing 90.6% CO<sub>2</sub> and 11.4% CH<sub>4</sub> was injected from the top of cylinder. After 24 hours, the concentration fell to 90.2% CO<sub>2</sub> and after 336 hours, it fell to 80.2% CO<sub>2</sub>.

All the laboratories involved in this project now feed gas from the top of the measuring cylinder. In addition CSIRO uses an oil barrier between water and gas to minimise contact between the two phases.

From the above discussion it can be seen that an error of up to 15% in gas content can be expected when seam gas is very rich in CO<sub>2</sub> and gas is fed from the base of measuring cylinder. It should be noted that methane (CH<sub>4</sub>) gas is also soluble in water but in much lower quantity compared to CO<sub>2</sub>. The error relating to CH<sub>4</sub> loss in water is estimated to be about 1%. The effect of residence time, that is the time of gas standing over the water column, can be neglected as in the crushing method this time is in order of only minutes.

#### *Effect of gas partial pressure*

The partial pressure of seam gases can alter the overall volume and the rate of gas desorption from coal. The void volumes in the crusher and transport canister varied among the participating labs. The volume of void space in the crushing container depends on the volume of the container and the mass of coal. The ratio of crushing container volume (mL) to coal mass (g) is used to express the void volume. This ratio varied from 2.1 to 6.1 across the participating laboratories. The variation in this ratio has an effect on the rate of desorption when the coal gas content is low or the coal contains mixed gases.

In order to quantify the effect of gas partial pressure, after each completion of a crushing and measurement of gas content some 10% of crushed coal was left in the crusher and gas released from coal was monitored for a day or longer. In most case gas continued to desorb from crushed coal under the new low partial pressure. In Fig. 4, the measured 'residual' gas contents under very low gas pressure conditions, of 38 borecore samples, are shown. From Fig. 4 it can be seen that no correlation exists between gas released after crushing ( $Q'_c$ ) and gas released before and during crushing ( $Q_l + Q_c$ ). The majority of coal samples showed a  $Q'_c$  below 0.8 m<sup>3</sup>/t. The same data are shown in Fig. 5 where  $Q'_c$  is normalised by  $Q_l + Q_c$  values. The graph shows that gas released under low partial pressure for the borecores measured is in most cases under 12%, but is more significant for the lower gas contents and occasionally can reach as high as 19% of measured  $Q_l + Q_c$ . Consequently it can be concluded that variability due to variation in gas pressure is limited to 12% in most cases.

#### *Effect of temperature*

Coal crushing generates heat and temperature rise changes the gas desorption rate. The effect of temperature rise on gas desorption rate is under study and is not yet quantified. Crushing time varied among laboratories from 7 to 90 minutes. At this stage all participating laboratories try to avoid temperature rise by using a multi-cycle crushing and cooling procedure or they use correction factors to compensate for the increase in temperature. During the measurements the ambient temperatures in participating laboratories were in the range of 20 to 22 °C.

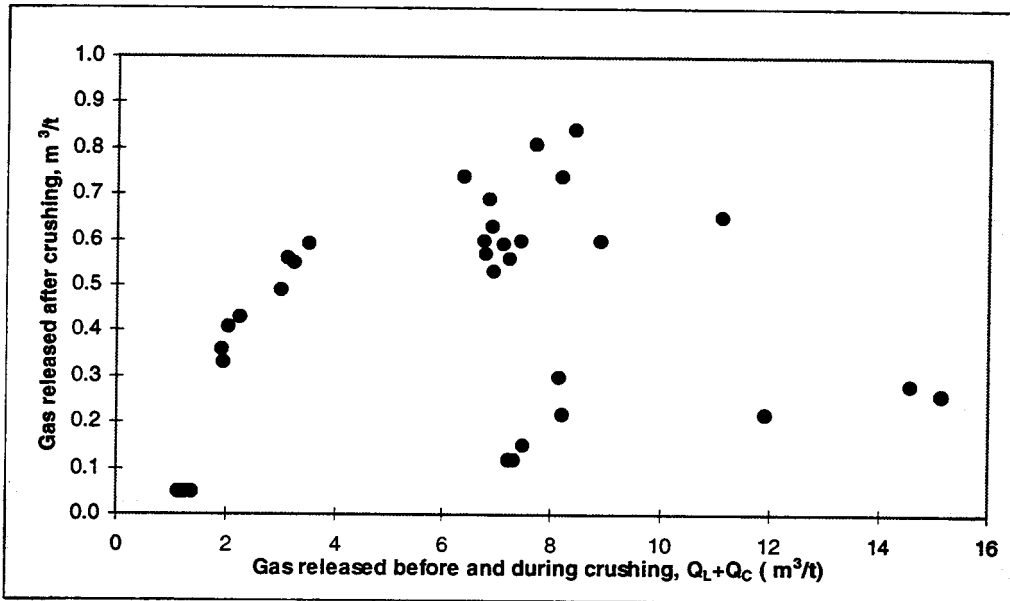


Fig. 4 - Gas released from crushed coal under low gas partial pressure

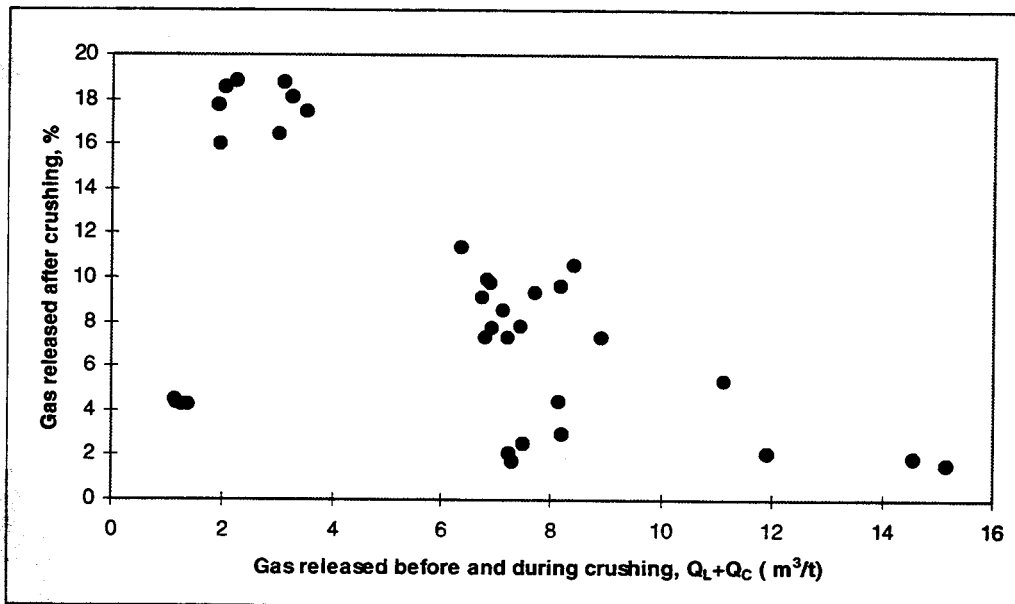


Fig. 5 - Ratio of gas released after crushing under low gas partial pressure

## CONCLUSIONS AND RECOMMENDATIONS

Analysis of seam gas content and composition figures determined by the participating laboratories indicates that there are some variations in both gas content and gas composition results. The causes of variability in gas content can be the dissolution of CO<sub>2</sub> in water, different void ratio or gas partial pressure in coal containers and temperature changes during crushing. The causes of variability in gas composition can be the dissolution of CO<sub>2</sub> in water and oxidation of coal particularly for low gas content coal samples. The problem of gas dissolution in acidified water was reduced dramatically by feeding gas from the top of cylinder. All laboratories involved in this study now practice this method. Using a linseed oil barrier to separate gas from water in the measuring cylinder further reduced this dissolution. Effect of temperature



increase on gas volume and rate of desorption is minimised by crushing coal in a sealed container and then allowing a cooling period before opening the valve and measuring the gas volume desorbed.

For the 38 bore cores tested for gas content, the absolute variability from the mean gas content in all cases except one was found to be less than 1.2 m<sup>3</sup>/t. The relative variability in terms of mean gas content was in all cases except three below 15%. However variability in results between two individual labs can be higher and in some cases be as high as 30%. Further research is essential if close positive correlation to the suggested level of 10% is to be achieved.

The necessity to achieve accurate, reliable and reproducible gas content and composition test results at all operating laboratories remains paramount to ensure the safety of underground mine personnel as well as the required development rates. While it is accepted that the accurate measurement of seam gas content of coal is no easy task, it is imperative that continuing research be maintained to overcome all significant variability and create an Australian Standard which ensures that comparable and consistent data are obtained and reported at all times.

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