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IMPACT OF CORE SAMPLE RECOVERY TIME ON ACCURACY OF GAS CONTENT MEASUREMENT

Dennis Black

ABSTRACT: Gas content measurement has a significant impact on mine safety and operational efficiency at many Australian underground coal mines. There is an expectation that reported gas content results are accurate and correctly represent the gas content of the coal seam in the area where the coal samples were collected. The practice of some coal sample testing service providers to add ‘correction’ factors to the measured gas content of coal samples increases the reported gas content to account for assumed loss of gas content due to extended recovery time. The methodology used by one gas testing service provider to establish a gas content correction factor to apply to coal samples with extended recovery time has been investigated and discussed.

A new approach to testing the impact of extended core sample recovery time on the accuracy of gas content measurement has been developed and is presented. Initial results from testing at three Australian underground coal mines indicates that, for core sample recovery times extending to 180 minutes, there is no discernible loss of gas content that would warrant the addition of a ‘correction factor’.

INTRODUCTION

Gas content measurement of coal seams in advance of mining is a significant tool in the assessment and management of outburst risk within the Australian underground coal industry. All Australian underground coal mines refer to a defined gas content threshold in the permit to mine process which determines whether mining is permitted to take place. It is therefore of great importance for the safety and productivity of mine operations that the measurement of gas content is accurate and correctly reports the seam gas conditions in the area from which the coal sample was collected.

Standards Australia have developed and published guidelines for measuring the gas content of coal samples which includes the fast desorption method typically used to measure the gas content of core samples collected from Australian underground coal mines (AS3980, 2016). The fast desorption method is widely used among gas content testing laboratories and it is generally accepted that measurement accuracy is within ±10%. While AS3980 recommends reporting gas content in either an as-received (AR) and/or dry-ash-free (DAF) basis, some laboratory service providers do apply in-house determined correction factors for variables, such as relative density (RD) of the coal sample and core sample recovery time, that increases the value of the reported gas content.

Coal samples used for gas content testing are typically sourced using one of the following three methods: (a) drilling from surface, (b) drilling from underground, or (c) collection of fresh coal samples from the working face area. This paper focuses on the gas content testing of coal samples sourced from underground-to-inseam (UIS) drilling.

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If the time to recover a core sample from a UIS borehole exceeds a nominal threshold time period of say 40 minutes – is there an increasing loss of gas from the coal sample that warrants the addition of a ‘correction factor’? Current practice assumes gas loss does occur and the measured gas content is less than the in situ content of the seam which supports the addition of correction factors. This study investigates the potential impact that core sample recovery time has on the accuracy of gas content measurement.

GAS CONTENT TESTING IN AUSTRALIAN UNDERGROUND MINES

The method used to determine the gas content of coal samples collected from Australian underground coal mines is described in Australian Standard AS3980:2016 – Determination of gas content of coal and carbonaceous material – direct desorption method.

The total measured gas content of a coal sample (QM) is comprised of three main components: lost gas during coal sample collection (Q1), desorbed gas content (Q2), and residual gas content (Q3), as illustrated in Figure 1. Further explanation of the three gas content components is provided below.

Figure 1: Gas released from coal at different stages of gas content testing (after AS3980:2016)

Lost Gas Content (Q1)

During the time taken to drill a core sample, recover the sample from the borehole, transfer the sample from the core barrel and seal it into a desorption canister, gas will be lost from the coal sample. The volume of gas lost from the sample cannot be measured and it is accepted practice to estimate the lost gas content (Q1). For gas content testing of underground bore cores, gas is assumed to start escaping from the core when the surrounding fluid pressure is equal to or less than the gas pressure in coal (at time \( t_0 \)), which is assumed to start from midway through the coring cycle (AS3980, 2016).

As soon as practical after recovering the core sample from the UIS borehole and sealing the core sample into a gas canister, the canister is connected to a gas desorption measurement apparatus at the drilling site, the valve opened, and volume of gas desorbed is measured incrementally over a period of typically 20 minutes. The gas volume desorbed from the core sample during this initial field measurement period is referred to as Q2field. Figure 2 provides an example of the initial Q2field desorbed gas volume measurements recorded during the initial 20 minute period, 34.5 to 54.5 minutes, after sealing the core sample into the gas canister (t1), 34.5 minutes (t0) after the assumed start of gas loss from the core sample (t0).
Plotting the cumulative readings of the initial desorption volume against the square root of elapsed time, selecting the linear portion of the curve, and extrapolating that line to the Y-intercept at \( t_0 \) is taken as the volume of lost gas (AS3980, 2016). The lost gas content \( Q_1 \) is determined by dividing the volume of lost gas (in millilitres) by the ‘as-received’ mass (in grams) of the sample in the canister.

**Figure 2: Q2field initial desorbed gas volume measurements 34.5 to 54.4 minutes after \( t_0 \)**

Figure 3 provides an example using the Q2field desorbed gas volume measurements presented in Figure 2 to estimate the gas loss from the core sample while being recovered from the borehole, 1541.8 mL at the Y-intercept corresponding to \( t_0 \). Dividing the estimated lost gas volume (mL) by the known sample mass (g) gives the lost gas content \( Q_1 \) (m³/t).

**Figure 3: Using initial Q2field gas desorption data to estimate the lost gas content \( Q_1 \) (m³/t)**

The volume of gas lost prior to sealing a coal sample into a desorption canister generally depends on sample retrieval time, physical character of the sample, the type of drilling fluid, and water saturation/relative amount of free gas (Diamond et al., 2001). Virtually all methods
for estimating lost gas benefit from minimizing the lost-gas time over which measured desorption data must be extrapolated.

The assumption of linearity of desorbed gas volume relative to square root of time is only valid for short values of core sample recovery time and the error of Q1 estimation increases with increasing recovery time (AS3980, 2016). It is therefore recommended that core samples are retrieved as quickly as possible, and initial desorption measurements completed without delay. AS3980 also warns that significant errors will occur in the determination of lost gas if the initial gas desorption is not measured within about 40 minutes of zero time ($t_0$).

Kissell et al. (1973) and McCulloch et al. (1975) demonstrated that the physical character of the retrieved coal sample can influence the desorption rate and hence the volume of lost gas that must be estimated. Blocky coals which remain intact during coring and subsequent retrieval emit their adsorbed gas at a relatively slow rate in comparison to friable coals which tend to break apart into smaller fragments releasing their adsorbed gas at a faster rate due to the shorter diffusion distances.

**Desorbed Gas Content (Q2)**

The desorbed gas content of a coal sample (Q2) is a measure of the gas desorbed at near atmospheric pressure from the non-pulverized coal sample; expressed as the volume of gas per unit mass of sample (AS3980, 2016).

The total quantity of gas evolved from the coal core sample contained within the gas desorption canister is measured volumetrically by the water displacement method in the field (Q2field) and subsequently in the laboratory (Q2lab) using apparatus similar to that illustrated in Figure 4 (AS3980, 2016).

![Figure 4: Volumetric desorbed gas measurement apparatus (AS3980:2016)](image)

**Residual Gas Content (Q3)**

Coal will naturally retain gas at atmospheric pressure and to measure the volume of gas retained within the coal sample, known as residual gas content (Q3), representative sub-samples of the coal core are crushed to a fine powder. The crusher shall have the capability to release the gas either during or after crushing and the volume of gas released during crushing is recorded and divided by the sub-sample mass to determine Q3 (m$^3$/t).
AS3980 (2016) states the crusher shall be capable of pulverizing the sample to 95% of material passing through a 212 μm mesh. Each sub-sample is to be crushed separately and for no longer than the time taken to ensure that at least 95% of the material passes 212 μm mesh. This will vary from seconds to minutes depending on the quantity and quality of the sample and the mill configuration.

Figure 5 shows a photo of four ring mill crushers used to crush coal sub-samples of approximately 200-gram mass for measurement of Q3. This figure also shows an example of the result of crushing one coal sub-sample for seven (7) minutes which indicates over-crushing and wafer-like agglomeration of fine coal particles.

Factors that Affect the Precision and Accuracy of Gas content Testing

AS3980 (2016) lists and explains the factors considered to potentially affect the accuracy of gas content measurements that include:

1. System leakage resulting in loss of gas during transport and testing;
2. Origin and integrity of the coal sample;
3. Solubility of carbon dioxide in water;
4. Partial pressure effects on the equilibrium end point for desorption;
5. Desorption rate (effect on lost gas calculation);
6. Temperature (effect on magnitude of measured desorbed gas);
7. Barometric pressure (effect on magnitude of measured desorbed gas);
8. Borehole back pressure (effect on assumed start time of gas desorption during sample recovery);
9. Sample moisture content; and
10. Accuracy of the desorbed gas measuring apparatus

GAS CONTENT ERROR DUE TO EXTENDED CORE RECOVERY TIME

AS3980 (2016) states that significant errors will occur in the determination of lost gas if the initial gas desorption is not measured within about 40 minutes of zero time (t0). Whist it is ideal to recover core samples in the shortest time possible, there are many circumstances and examples in operating underground mines where the time to recover core samples from UIS boreholes cannot be achieved within the recommended 40 minute timeframe. In such cases, some coal mine operations and their gas content testing provider will either deem the sample to be ‘invalid’ or add an additional gas content ‘error’ factor to the measured gas content and
report a higher gas content to account for the uncertainty and assumed gas loss during the extended core sample recovery time.

An assessment of the methodology used by one gas content testing service provider to determine the error in gas content measurement resulting from extended core sample recovery time has been undertaken and summarised below. Once established, an additional gas content ‘correction’ is added to measured gas content of core samples with recovery time exceeding the nominated threshold values that is typically 40 minutes, to account for the corresponding ‘error’.

The methodology is based on recovering coal core samples from UIS boreholes within the threshold time period and then conducting Q2field gas desorption measurements at the drill site for an extended period, recording the desorbed gas volume at short time increments, approximately 2 minute intervals, extending to approximately 200 minutes following initial coring (t0). Figure 6 provides an example of the extended Q2field desorbed gas volume measurements from one UIS core sample, recorded at 2-minute intervals for a period of 202 minutes following initial coring.

![Figure 6: Extended Q2field desorbed gas volume and corresponding regression line](image)

Using the data collected during the extended Q2field gas desorption, extended core sample recovery time (t_e) is simulated and Q1 is estimated based on t1 at increasing time periods. Figure 7 shows nine 20 minute Q2field gas desorption intervals that have been selected to represent extended core sample recovery time, i.e. indicating t1 commencing 20, 40, 60, 80, 100, 120, 140, 160 and 180 minutes following initial coring (t0).

![Figure 7: Assumed desorbed gas volume at increasing sample recovery time](image)
The recorded desorbed gas volume for the 20 minute period following each selected extended core samples recovery time (t1) is plotted relative to square root of time to determine the corresponding lost gas content estimate for each extended core sample recovery time. Figure 8 shows the Q1 estimation curves, desorbed gas volume relative to square root of time, for the nine 20 minute time intervals using the desorbed gas volume data recorded during the extended Q2 field gas desorption period. From the data presented in this example, it is shown that Q1 increases, reaching a maximum at approximately 120-130 minutes after which Q1 decreases.

![Figure 8: Desorbed gas volume at increasing core sample recovery time (t1) based on extended Q2 field desorbed gas volume measurement](image)

The results reported by the gas testing service provider for testing on this core sample are summarised in Table 1A and 1B. The results indicate the total measured gas content (QM) was 6.41 m³/s, residual gas content (Q3) was 4.18 m³/t and the desorbed gas content recorded in the laboratory (Q2 lab) was 1.26 m³/t. This methodology assumes that Q2 lab and Q3 remain constant and extending core sample recovery time only affects Q2 field, which in turn affects Q1.

Table 1A: Reported gas content at simulated core retrieval times

<table>
<thead>
<tr>
<th>Simulated Core Retrieval Time (minutes)</th>
<th>Lost Gas (m³/t)</th>
<th>Lab Q2 (m³/t)</th>
<th>Field Q2 (m³/t)</th>
<th>Q3 (m³/t)</th>
<th>Measured Gas Content (m³/t)</th>
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<tbody>
<tr>
<td>20</td>
<td>0.25</td>
<td>1.26</td>
<td>0.63</td>
<td>4.18</td>
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<td>0.48</td>
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<td>1.26</td>
<td>0.44</td>
<td>4.18</td>
<td>6.43</td>
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<td>0.39</td>
<td>4.18</td>
<td>6.44</td>
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<td>0.35</td>
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<td>0.03</td>
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</table>

Table 1B: Reported gas content error at simulated core retrieval times

<table>
<thead>
<tr>
<th>Simulated Core Retrieval Time (minutes)</th>
<th>Calculated Qm (m³/t)</th>
<th>Original Qm (m³/t)</th>
<th>% Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>6.52</td>
<td>6.41</td>
<td>1.41</td>
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<tr>
<td>30</td>
<td>6.36</td>
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<td>0.84</td>
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<tr>
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<td>6.39</td>
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<td>0.37</td>
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<td>60</td>
<td>6.43</td>
<td>6.41</td>
<td>-0.26</td>
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<td>70</td>
<td>6.44</td>
<td>6.41</td>
<td>-0.42</td>
</tr>
<tr>
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<td>6.41</td>
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<tr>
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<td>-0.44</td>
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<tr>
<td>190</td>
<td>6.09</td>
<td>6.41</td>
<td>5.05</td>
</tr>
</tbody>
</table>
Table 1B summarises the %Error associated with extended recovery time which is the difference between the Original QM and the Calculated QM values. The reported methodology does not provide any detail as to how and when the original QM value is determined. Given there is no reference to duplicate core samples being used in this testing methodology, one would assume that at the conclusion of the extended Q2field desorption period, the core canister would be sealed and transported to the laboratory where Q2lab and Q3 would be determined. Q1 would be based on the Q2field gas desorption during the initial 20 minute period after sealing the core into the gas canister. The results presented in Table 1A and 1B do not support this assumption.

The %Error in gas content measurement reported against increasing core retrieval time is presented in Figure 9. The results indicate that relative to the reference ‘Original QM’ value, gas content values calculated for recovery times less than 50 minutes and greater than 110 minutes will be understated and the gas content values calculated for core retrieval times between 50 and 110 minutes will be overstated.

**Figure 9: Reported gas content error from extended Q2field comparative analysis**

The gas testing service provider then presents the results obtained from similar testing on multiple core samples which are used to determine the gas content correction factor recommended for the mine site. Figure 10 shows the correction factor recommended to be added to the measured gas content for core samples with extended recovery time, which is the average of the results obtained from extended Q2field gas desorption testing on multiple core samples plus three (3) standard deviations.

**Figure 10: Reported gas content error from extended Q2field comparative analysis**
Given the many operational scenarios in an underground coal mine that would warrant the collection of core samples for gas content testing that take greater than 40 minutes to recover from the borehole, it is important that the impact of extended core sample recovery time on the accuracy of gas content measurement is fully understood and appropriately dealt with in gas content measurement and reporting.

**NEW APPROACH TO ASSESS IMPACT OF CORE RECOVERY TIME ON GAS CONTENT MEASUREMENT ACCURACY**

While some may consider the methodology reported above to be appropriate for estimating ‘error’ in gas content measurement due to extended core sample recovery time, this section describes an alternate approach. This new approach involves the collection of five core samples from the same location in the coal seam and testing each core, in accordance with the AS3980 guideline, to measure and report the gas content.

As illustrated in Figure 11, a 3.0 metre core barrel is used to collect a representative section of core from the target coal seam using UIS drilling. A core sample collection and testing procedure has been developed to maintain consistency in testing procedure at each mine site. UIS drillers collecting the core samples are to complete a roof touch to confirm hole position in the seam approximately 20 metres prior to reaching the target coring depth. The drillers are instructed to maintain the borehole parallel to the coal seam through coring to collect the 3.0 metre length of core from the same coal ply.

At each test location, the objective is to recover the core from the borehole in less than 40 minutes. Upon recovering the core barrel from the UIS borehole, a 600 mm length of core is removed from the core barrel and sealed into a prepared gas canister and Q2field desorption testing is completed for the recommended 20 minute period. The measured gas content of the first core sample recovered from the UIS borehole provides a baseline gas content for the test location and the measured gas content of the remaining core samples are compared against the baseline to assess the potential impact of simulated extended core sample recovery time.

The remaining 2.4 metre of core remains contained within the core barrel, placed in a 3.0 metre length of 6” pipe filled with water, to simulate extended core sample recovery time. For the purpose of this investigation, five 600mm sections of core sample are removed from the core barrel at time intervals of 60, 90, 120 and 180 minutes following initial coring (t0). As each section of core is removed from the core barrel following the simulated period of extended recovery time, it is sealed into a prepared gas canister and Q2field desorption testing completed prior to transporting to the nominated gas testing service provider to complete the gas content testing in accordance with AS3980.

![Figure 11: UIS core sample in a 3.0m core barrel for extended Q1 gas content testing](image-url)

Figure 12 illustrates the testing sequence and timeframes to complete the core sample collection and gas content testing at each nominated test location. The objective of this testing program is to collect gas content data from test locations covering a broad range of Australian coal seam conditions and to analyse the results to determine if increasing the recovery time of
coal core samples from UIS boreholes has a material impact on the accuracy of gas content measurement. This study will also consider whether the addition of a ‘correction factor’ to the measured gas content of core samples with extended Q1 time to account for ‘error’ due to gas loss is warranted.

Results of testing conducted in three, methane rich, Australian coal seams, has shown minimal difference in the measured gas content of core samples with extended recovery time compared against the initial baseline QM measurement for each test location. In all but one coal sample tested to date, the variability in measured gas content values of the five cores from each test location is within 10% of the baseline reference and is below the 10% target maximum variability considered acceptable by the Australian Standard (Danell et al., 2003 and Saghafi and Williams, 1998).

Summary of Results from comparative extended Q1 gas content testing

Figures 13, 14 and 15 show the summary results obtained from comparative testing at three mines, each in different coal seams, to assess the impact of delayed core sample recovery (extended Q1) on the accuracy of gas content measurement. Each of the five graphs includes ±10% error bands based on the first core sample recovered that was tested without delay as recommended by AS3980 (2016). In addition to the measured Q1, Q2 and Q3 gas content components, each graph also shows the gas content value reported by the testing laboratory for each core sample. Figure 13 shows results from Mine A operating in Seam 1. At this mine, an increasing gas content error correction factor is applied to the measured gas content of samples with recovery times between 40 and 120 minutes. Any core samples taking longer than 120 minutes to recover from UIS boreholes are deemed invalid and discarded. It is interesting to note that in the case of Mine A, with an outburst threshold gas content value of 7.0 m³/t, the measured gas content of the core sample with 99 minute recovery time was 6.93 m³/t and with the addition of the correction for extended sample recovery time, the reported gas content was increased to 8.76 m³/t.

Figure 14 shows results from Mine B operating in Seam 2. The results obtained from testing at Mine 2 do not indicate a loss of gas content from samples with longer recovery time. While both
graphs indicate the addition of gas content correction factor for samples with recovery time exceeding 70 minutes, the reported gas content from the 33 minute (benchmark) and 68 minute sample at location 1 (left graph) are greater than the measured gas content which is the result of an RD correction factor applied by the gas testing service provider.

Figure 13: Extended Q1 Gas Content Results – Mine A, Seam 1

Figure 14: Extended Q1 Gas Content Results – Mine B, Seam 2

Figure 15 shows results from Mine C operating in Seam 3. While only three of the planned five core samples were recovered from this test location, the results are consistent with those obtained from Mine A and Mine B. Overall the results do not indicate gas content error, outside of the bounds of accepted test repeatability, from core samples that take longer than the preferred 40 minute time period to recover from the UIS boreholes.

Figure 15: Extended Q1 Gas Content Results – Mine C, Seam 3
Trending of Gas Content Component Values

Figures 16, 17 and 18 show results of the measured gas content component values, Q1, Q2 and Q3, obtained from the extended core sample recovery time testing at each of the three test mines.

Contrary to the simulated Q1 values obtained during the extended Q2 field desorption testing discussed in the section above, the results obtained from Mine A, B and C all show a consistent increase in Q1 value relative to increasing sample recovery time.

The results of testing at location 1 at Mine A with QM ≈ 3.0 m³/t, shown in the left graph of Figure 16, has no recorded Q1 gas content, very low Q2 gas content and minor variability in Q3 gas content. The measured variability in Q3 gas content from samples collected at the same test location in a low gas content area, highlights the variability in repeatability of gas content measurement among laboratory services providers, which is acknowledged within the Australian Standard.

In addition to increasing Q1 in response to extended sample recovery time, the results from each test location also indicate decreasing Q2 and relatively constant Q3 gas content component values. These results are consistent with accepted gas desorption theory, with a greater percentage of the total gas contained within the core sample being desorbed and lost from the coal sample with increasing time period between initial coring and sealing inside the gas canister. The non-desorbing, residual gas content Q3 component, is not expected to change significantly over the 180 minute extended desorption period as this time period is less than the standard Q2 lab desorption time period.
CONCLUSIONS AND RECOMMENDATIONS

Given the significant impact that gas content measurement has on mine safety and operational efficiency among Australian underground coal mines there is an expectation that reported gas content results are accurate and correctly represent the gas content of the coal seam in the area from which the coal sample was collected. There is a practice among some coal sample testing service providers to add ‘correction’ factors to the measured gas content of coal samples. These ‘correction factors’ increase the reported gas content to account for assumed loss of gas content due to extended recovery time and even for variability in relative density of the coal sample.

The methodology used by one gas testing service provider to establish a gas content correction factor to apply to coal samples with extended recovery time has been investigated and discussed. A new approach to testing the impact of extended core sample recovery time on the accuracy of gas content measurement has been presented. The initial results obtained from testing at three Australian underground coal mines indicates that for core sample recovery times extending to 180 minutes, there is no discernible loss of gas content that would warrant the addition of a ‘correction factor’. Figure 19 shows the percentage variance in the measured gas content of core samples at increasing recovery times relative to the initial benchmark gas content recorded at each test location.

![Figure 18: Extended Q1 Gas Content Component Values – Mine C, Seam 3](image)

![Figure 19: Variance in Gas Content during Extended Q1 Testing Relative to Benchmark](image)
Further testing is planned to increase the size of the reference dataset covering a broad range of Australian coal seam conditions to establish if the results found from this initial investigation apply to all Australian coal seams. Australian underground coal mines interested in participating in this study are invited to contribute.

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