Core: Coal Sampling and Core Analysis

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• The importance of coal properties
• Their variability
• The importance of sampling
• Determination of some of the more commonly used
  • Different reporting bases
  • Core
  • Gas
To a piece of coal

Were you once alive?
I touch you
You shimmer like a fern leaf
I cover you with my lens
What are you, banded one?

Clarence A. Seyler
The Big Picture and the Four Grand Processes of Coal Utilization
Coal Type
Coal Rank
Coal Grade
Variability of Chemical Properties as a Function of Rank

- Reflectance (% $R_{m_{oil}}$)
- Volatile matter (% daf)
- Moisture (%)
- Calorific value (kcal/kg daf)
- Carbon (% daf)
- Hydrogen (% daf)
Variability of Physical Properties as a Function of Rank
<table>
<thead>
<tr>
<th>Rank stages</th>
<th>Coal class</th>
<th>Description</th>
<th>Australian classification (AS 1086)</th>
<th>Rank variables</th>
<th>Overseas classifications</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>$R_e$ max percent Vitrinite/Huminite</td>
<td>$F_1$ (See Note)</td>
<td>Moisture$^*$ (See Note)</td>
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<td>percent Vitrinite/Huminite</td>
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</table>

*Note: $R_e$, $F_1$, $C$, $S_F$, and Sporinite fluorescence max mm are variables used in the classification process. $R_e$ is the ratio of the ash-free dry weight of the coal to the ash-free dry weight of the sample. $F_1$ represents the percent of the sample that is volatile matter. $C$ indicates the carbon content of the coal. $S_F$ is the sulfur content of the ash-free dry weight of the sample. Sporinite fluorescence max mm indicates the maximum fluorescence of the coal sample.
### A few of the more common hard coal tests

<table>
<thead>
<tr>
<th>Hard Coal Test</th>
<th>Sub-test (Abbreviation)</th>
<th>Standard /Reference</th>
<th>Test Description</th>
<th>Reference</th>
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<td>Ash Yield (A)</td>
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<td>Volatile Matter (VM)</td>
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<td>Fixed Carbon (FC)</td>
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<td>Nitrogen (N)</td>
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<td>Hydrogen (H)</td>
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<td>Sulphur (S)</td>
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<td>Ash Fusibility</td>
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<td>Adsorption Isotherm</td>
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<td>Vitrinite Reflectance</td>
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<td>Drop Shatter Test</td>
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<td>Tumbler Test</td>
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<td>Roga Test</td>
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<td>plus lots and lots of other even more exotic tests as well as many variations on the above themes....</td>
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</table>
Proximate Analysis

- moisture + ash + volatile matter + fixed carbon = 100
- M, A, VM are determined; FC = 100 – M – A – VM
- Moisture = weight loss when heated to 110°C in inert atmosphere
- Ash = weight loss when heated to high temperature in air
  (AS/BS 500°C/815°C; ASTM 750°C or 500°C/750°C)
- VM = weight loss when heated to high temperature in inert atmosphere
  (AS/BS 900°C; ASTM 950°C)
Coal Density

• Bulk density
  • fill a box of known volume with coal and weigh it
  • variability due to packing density and moisture content

• True density
  • known mass with volume being determined using a fluid that completely
    fills all pore spaces e.g. Helium

• Relative density is the density compared to water and is therefore
  dimensionless

• Relative density – air dried coal, crushed to -0.212mm; placed in
  pycnometer with a wetting solution commonly methylated spirits

• Apparent relative density – air dried lump coal; weigh in air and then weigh
  in water – includes large pore spaces so the bigger volume means the ARD is
  less than the RD; gives more variable results than RD
Coal Density

- insitu coal density depends on the inorganics, the organics, coal rank degree of open porosity

- you CANNOT use the unadjusted RD values to determine coal reserves and you cannot use ARD values as these are too unreliable

- RD must be adjusted:

\[
\text{RD}_{\text{insitu}} = \frac{\text{RD}_{\text{lab}} (100 - M_{\text{ad}})}{100 + \text{RD}_{\text{lab}} (M_{\text{insitu}} - M_{\text{ad}}) - M_{\text{insitu}}}
\]

GIP = area x thickness x density x gas content
Reporting Bases for Coal Analyses

- **as received (ar)**
- **as analysed (aa)**
- **dry basis (db)**
- **air dried (ad)**
- **dry, mineral matter free (dmmf)**
- **ash yield**
- **organic VM**
- **inorganic VM**
- **volatile matter**

**Organics**
- **fixed carbon**
- **dry, mineral matter free (dmmf)**
- **dry, ash free (daf)**

**Minerals**
- **moisture**
- **ash yield**
- **organic VM**
- **inorganic VM**
- **volatile matter**

**Bases**
- **as received (ar)**
- **as analysed (aa)**
- **dry basis (db)**
- **air dried (ad)**

**Coal Components**
- **water of hydration of minerals + organically bound water (part of VM)**
- **ash free (af)**
- **mineral matter containing basis (mmcb)**
- **moist basis (mb)**
- **moist containing basis (mcb)**

**Moisture Types**
- **bed moisture**
- **total moisture**
- **equilibrium moisture**
- **air dried moisture (inherent moisture)**

**Coal Analyses**
- **fixed carbon**
- **volatile matter**
- **organic VM**
- **inorganic VM**
- **ash yield**
- **moisture**
- **mineral matter containing basis (mmcb)**

**Coal Types**
- **dry, mineral matter free (dmmf)**
- **dry, ash free (daf)**
- **moisture**
- **water of hydration of minerals + organically bound water (part of VM)**
- **ash free (af)**
- **mineral matter containing basis (mmcb)**
- **moist basis (mb)**
- **moist containing basis (mcb)**
Mineral Matter vs Ash

• Minerals present within coals: clays, quartz, carbonates, sulphides etc
• Minerals are transformed during ashing by loss of volatiles, oxidation, phase changes
• Importantly water of hydration is lost; carbonates and sulphides decompose
• Volatile trace elements are lost (e.g. mercury)
• MM can be determined by low temp. ashing; plasma ashing; chemical oxidation
• Parr’s formula: \[ MM = 1.08A + 0.55S_{\text{total}} \]
• KMC formula \[ MM = 1.09A + 0.5S_{\text{py}} + 0.5CO_2 - 1.1SO_4^{\text{ash}} + SO_4^{\text{coal}} + 0.5Cl \]
Core
The Description of Core

- Lithotypes
- Fractures (geotechnical logging)
Lithotypes
## ICCP Lithotype Nomenclature (under discussion)

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<th>Unbanded coal</th>
<th>Bright coal</th>
<th>Vitrain</th>
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<td>Semi-bright coal</td>
<td>Clarain</td>
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<td>Dull coal</td>
<td>Durain</td>
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<tr>
<td>Banded coal</td>
<td>Banded bright coal</td>
<td>(Duroclarain)</td>
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<td></td>
<td>Banded semi-bright coal</td>
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<td></td>
<td>Banded dull coal</td>
<td>(Clarodurain)</td>
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<td>Fibrous</td>
<td>Fibrous coal</td>
<td>Fusain</td>
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## Australian Standard Brightness (AS2916)

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<th>&gt; 90% bright</th>
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<td>Bright Banded</td>
<td>BB</td>
<td>&gt; 60% bright ≤90%</td>
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<tr>
<td>Banded</td>
<td>BD</td>
<td>&gt; 40% bright ≤60%</td>
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<tr>
<td>Dull Banded</td>
<td>DB</td>
<td>&gt; 10% bright ≤40%</td>
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<tr>
<td>Dull, Minor bright</td>
<td>DM</td>
<td>&gt; 1% bright ≤10%</td>
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<tr>
<td>Dull</td>
<td>DD</td>
<td>≤1% bright</td>
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Coal Sampling

• The key issue is trying to obtain a representative sample of an extremely heterogeneous material

• different sets of problems arise for drill core samples versus mined coals versus outcrops

• for broken / crushed coals, different particle sizes will have different chemistries and properties at all size scales

• during any kind of movement, particle size segregation occurs; if the particles are in ‘dry’ contact with each other, then the coarse particles move to the surface of lowest energy (e.g. the top) to create reverse grading effects

• Manual sampling – e.g. diamond saw slabbing; crushing; cone + quarter; rotary sample divide

• Augur sampling (e.g. for stockpiles)

• Mechanical systems (e.g. for conveyor belts)

• Continuous monitoring systems (e.g. for ash on conveyor belts)
One of a suite of extremely poor samples received at ERC in early 2010 from a CSG exploration programme. A finer fraction obtained after crushing part of the core has been supplemented by a few random lumps of core to make up the weight requirements for testing. Some of these samples tested at >40% ash when proximate analysis data from the same core indicated <15% ash.
Gas
pores in coal

- micro - pores : < 20 Å
- meso - pores : 20 - 50 Å
- macro - pores : > 50 Å
- coals are extremely microporous with most pores < 12 Å in size
coal structure
• Origin of gases
• Storage mechanisms
• Determination of gas contents – desorption and adsorption
• The adsorption isotherm – its uses and constraints
• Gas Saturation
Origin of the Gases

Gases present – CH₄, C₂H₆, CO₂, N₂, H₂S

Thermogenic

Biogenic
### Thermogenic Hydrocarbons

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<tr>
<th>Depth (km)</th>
<th>Hydrocarbon Maturity</th>
<th>Maximum Palaeo-temperature (°C)</th>
<th>Maximum Vitritine Reflectance, ( R_o ) (%)</th>
<th>Spore Coloration</th>
<th>Level of Organic Metamorphism LOM</th>
<th>Hydrocarbon Product</th>
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<td>initial maturity (zone of oil generation)</td>
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<td>0.5</td>
<td>orange</td>
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<td>oil</td>
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<td>heavy hydrocarbons</td>
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<td>0.7</td>
<td>brown</td>
<td>10</td>
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<td>3</td>
<td>light hydrocarbons</td>
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<td>1.0</td>
<td>brown/black</td>
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<td>condensate/wet gas</td>
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<td>mature and post-mature (high temperature methane)</td>
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<td>1.3</td>
<td>black</td>
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<td>high temperature methane</td>
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<td>180</td>
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thermogenic

Estimated gas generation potential of two different coal types as a function of coal rank (after Higgs, 1986).
Coal

CO₂ reduction

CO₂ + 4H₂ → CH₄ + 2H₂O

Acetate fermentation

CH₃COOH → CH₄ + CO₂

Smith, Pallaser and Rigby, 1992

Gases from coal pyrolysis experiments

Coal

Acetate fermentation

CH₃COOH → CH₄ + CO₂

Gases from coal pyrolysis experiments

Smith, Pallaser and Rigby, 1992
Gas Storage Mechanisms

most of the gas is physically adsorbed on the coal’s surface

• large storage capacity of coal
• equilibrium between solid and gas is reversible
• relatively rapid sorption rate when temperature or pressure are changed
• small heat of adsorption
• involves van der Waals forces
large storage capacity of coal

Methane Storage Capacity (20°C; 1 atm)

- Coal
- Sandstone (20% porosity)
Diagrammatic representation of molecules adsorbed on a surface freely exposed to the gas phase. (b). Diagrammatic representation of molecules adsorbed on the walls of a micropore.
coal structure
Factors Affecting Gas Sorption

- coal type
- coal rank
- moisture content
- particle size
- gas / hydrostatic pressure
- temperature
- mineralisation
- pore structure
- stress
Methane Adsorption Isotherm (20 °C, 101.1 kPa)
Analysis Conditions: T = 30.3 °C; Moist. = 4.6 (%); Ash = 12.4 (%)

- zero moisture
- 1.0 (%) Moisture
- 2.0 (%) Moisture
- 3.0 (%) Moisture
- daf
- As analysed
temperature
Measuring Gas Content of Coal

a) Total Desorbable Gas Content

b) Adsorption Capacity
a) Total Desorbable Gas Content

Q1 = lost gas
Q2 = desorbed gas
Q3 = residual gas
Q1 + Q2 + Q3 = total desorbable gas content
Q2 – desorbed gas: drilling and core recovery
Q2 – desorbed gas: core logging
Q2 – desorbed gas: field desorption
Q2 – desorbed gas: laboratory desorption
Q2 – desorbed gas

**FIGURE 1** VOLUME MEASUREMENT APPARATUS—TUBE ENTERING AT TOP

**FIGURE 2** VOLUME MEASUREMENT APPARATUS—TUBE ENTERING AT BOTTOM
Q2 – desorbed gas

Trend line extrapolated to asymptote gives $Q_2$ of 7.68 m$^3$/t

NOTE: Desorption is not quite complete.

FIGURE D2 SLOW DESORPTION CURVE
Q1 – lost gas

\[ y = 421.03x - 1447.9 \]

Lost gas = 1447.9 mL
Q3 – residual gas
## Slow desorption versus quick crush

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<th>quick</th>
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<tbody>
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<td>Time</td>
<td>months</td>
<td>hours to days</td>
</tr>
<tr>
<td>Sample size</td>
<td>whole core (kg)</td>
<td>subsample (10’s to 100’s g)</td>
</tr>
<tr>
<td>Tau</td>
<td>yes</td>
<td>problematic</td>
</tr>
<tr>
<td>Gas Composition</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>Changes with time</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>CO2</td>
<td>?problematic</td>
<td>fewer problems</td>
</tr>
</tbody>
</table>
The time constant $\tau$

\[
\frac{Q}{Q_m} = 1 - e^{-\frac{t}{\tau}}
\]

$Q = \text{total gas desorbed at time } t$

$Q_m = \text{total desorbable gas}$

$\tau = \text{time constant related to diffusion}$

$= \text{time taken for 63\% of the gas to desorb}$
## Variability in Gas Content

<table>
<thead>
<tr>
<th>Sample #4950</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Depth (m)</td>
<td>764.15</td>
<td>764.90</td>
</tr>
<tr>
<td>Thickness (m)</td>
<td></td>
<td>1.19</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.20</td>
</tr>
<tr>
<td>Moisture (%)</td>
<td></td>
<td>4.54</td>
</tr>
<tr>
<td>Ash (%)</td>
<td>25.22</td>
<td>25.2</td>
</tr>
<tr>
<td>VM (%)</td>
<td>38.63</td>
<td>31.7</td>
</tr>
<tr>
<td>FC (%)</td>
<td>31.83</td>
<td>38.4</td>
</tr>
<tr>
<td>Q1 (cc/g, aa)</td>
<td>0.12</td>
<td>0.26</td>
</tr>
<tr>
<td>Q2 (cc/g, aa)</td>
<td>2.74</td>
<td>0.98</td>
</tr>
<tr>
<td>Q3 (cc/g, aa)</td>
<td>1.09</td>
<td>0.58</td>
</tr>
<tr>
<td>Q1 + Q2 + Q3 (aa)</td>
<td></td>
<td>3.95</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.82</td>
</tr>
<tr>
<td>Q1 + Q2 + Q3 (daf)</td>
<td></td>
<td>5.62</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.60</td>
</tr>
</tbody>
</table>
b) Gas Adsorption Isotherm

Methane Adsorption Isotherm (20 °C, 101.3 kPa)
Analysis Temperature 30.3 °C

\[ V = \frac{24.86 \, P}{P + 2.87} \quad \text{(As analysed)} \]

\[ V = \frac{29.95 \, P}{P + 2.87} \quad \text{(daf)} \]
• two basic methods – volumetric and gravimetric
• Sample preparation: grain size; moisture
• know the empty volumes of sample and reference cells by prior determination
• measure the free space in the sample cell using He after the coal has been added
• use either the change in weight or change in pressure to calculate both the free gas and adsorbed gas
• PV = znRT
volumetric

gravimetric
• PV = znRT
• amount of adsorbed gas is calculated in moles
• the moles of gas are converted to a volume of gas at any conditions you care to nominate (usually 1 atm and 20°C / 60°F and 14.7 psia)
• calculation to daf basis also made

\[ V = \frac{24.86\ P}{(P + 2.87)} \quad \text{(As analysed)} \]
\[ V = \frac{29.95\ P}{(P + 2.87)} \quad \text{(daf)} \]

Analysis Temperature 30.3 °C
The five types of adsorption isotherm in the classification of Brunauer, Deming, Deming and Teller (also called the BET classification)
some isotherm models

\[ V = \frac{V_L P}{(P + P_L)} \quad \text{Langmuir} \]

\[ V = cP^n \quad \text{Freundlich} \]

\[ \frac{1}{V \left( \frac{P^0}{P} \right) - 1} = \frac{1}{V_mC} + \frac{(C-1)P}{V_mC \ p^0} \quad \text{BET} \]

\[ V = K \ln A_o P \quad \text{Temkin} \]

\[ \log V = \log V_0 - D \left[ \log \left( \frac{p^0}{P} \right) \right]^2 \quad \text{Dubinin – Radushkevich (D-R)} \]

\[ \log V = \log V_0 - D \left[ \log \left( \frac{p^0}{P} \right) \right]^n \quad \text{Dubinin – Astakhov (D-A)} \]
some isotherm models

- Data
- Yellow Line: Langmuir
- Red Line: BET
- Blue Line: D-R
- Pink Line: D-A
- Green Line: Log fit

Graph showing the relationship between volume (cc/g) and pressure (MPa abs) for different isotherm models.
gas flow and recoverable gas

Methane Adsorption Isotherm (20 °C, 101.3 kPa)
Analysis Temperature 30.3 °C

\[ V = \frac{24.86 \, P}{P + 2.87} \] (As analysed)
\[ V = \frac{29.95 \, P}{P + 2.87} \] (daf)
Methane Adsorption Isotherm (20 °C, 101.1 kPa)
Analysis Conditions: T = 30.3 °C; Moist. = 4.6 (%); Ash = 12.4 (%)

oversaturation
We Burn
Marie Carmichael Stopes

We speak of fire
When oxygen leaps swift
In fierce embrace to carbon,
Then the lift
Of heat flicks red-hot tongues
So fierce they heavenward aspire.
Eyes that perceive the smoke,
The glow, the cinder,
Of swift embrace divalent,
Yet are blind
When the same force plays on a lower scale
Whose ranges lend to man his lissom life
His power, his love, and all his leaping strife.

Man burns!
In every fibre and revolving cell
The seeing eye can tell
Man burns to live,
Man burning lives—
No, not in hell, but here
On the sweet-girdled earth.
His burning is so cool
That like a fool
He feels it not himself
Nor knows the flame he is,
Nor feels the ember in his breath.
Blindly he turns to death
For fiery torments,
Though he burns in life.

His very breath
A cascade of those swift divalent loves
Hot fire creates.
He reinstates
His soul’s vitality with pulsing warmth
From the slow furnace of his vibrant cells.
A gazing child, eyes sun-lit asks
“Does any live in God’s great bonfire there?”
Arrogant wise-heads shake their gloomy ‘No’s’
“Impossible! For ’tis all fire,” they say.
The child pretends great dragons there once fell
And, breathing fire, in that great heat, still dwell.
With incandescent breath upon Earth’s plains
Had silicon, not carbon, made your chains
It might be true
Of you.

Cool Truth proclaims
Life lives by burning.
Tuned to our slow-scaled speed in ceaseless fire we dwell
Breathing a smoke so cool
We in the plangent rhythm of life’s heart
Know not the pain of burning, but apart
Live, love, serenely in each rose-flecked cell.

(1939)
COAL PETROLOGIST — TO HIS LOVE

BLACK LUMP OF COAL
BEHIND YOUR SENSUAL SHINING FACE
LIE MYSTERIES
THE MIND OF MAN
CAN SCARCE IMAGINE.

COME —
LET ME HOLD YOU NOW
AND LIKE A WOMAN
BY DESIRE AROUSED

ONE KISS OF FLAME
AND YOU WILL BE CONSUMED

AND I WILL BE ALONE
— BLACK LUMP OF COAL
Coal Petrography
| Macerals of the vitrinite group (ICCP) |  |
| Telovitrinite                  | Telinite  |
|                                | Collotelinite  |
| Detrovitrinite                 | Vitrodetritinite  |
|                                | Collodetritinite  |
| Gelovitrinite                  | Corpogelinite  |
|                                | Gelinite  |
Macerals of the inertinite group (ICCP)

Macerals with plant cell structures:  
- Fusinite
- Semifusinite
- Funginite

Macerals lacking plant cell structures:  
- Secretinite
- Macrinite
- Micrinite

Fragmented inertinite:  
- Inertodetrinite
Macerals of the liptinite group (ICCP)

Sporinite
Resinite
Cutinite
Fluorinite
Suberinite
Alginate
Bituminite
Exsudatinite
Liptodetrinite
<table>
<thead>
<tr>
<th>Maceral subgroup</th>
<th>Maceral</th>
<th>Maceral type</th>
<th>Maceral variety</th>
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<tr>
<td>Telohuminite</td>
<td>Textinite</td>
<td>A (dark)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>B (light)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ulminite</td>
<td>A (dark)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>B (light)</td>
<td></td>
</tr>
<tr>
<td>Detrohuminite</td>
<td>Attrinite</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Densinete</td>
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<tr>
<td>Gelohuminite</td>
<td>Corpohuminite</td>
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<tr>
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<td>Phlobaphinite</td>
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<td>Texto-ulminite</td>
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<tr>
<td></td>
<td></td>
<td>Eu-ulminite</td>
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<td></td>
<td>Telocollinite</td>
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<tr>
<td></td>
<td>Detrovitrinite</td>
<td>Attrinite</td>
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<td></td>
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<td>Densinite</td>
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<td>Desmocollinite</td>
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<td>Fusinite</td>
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<td>Sclerotinite</td>
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<td>Detro-inertinite</td>
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</tr>
<tr>
<td></td>
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<td>Micrinite</td>
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<tr>
<td></td>
<td>Gelo-inertinite</td>
<td>Macrinite</td>
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</tr>
</tbody>
</table>

Minerals
Brown Coal – Telohuminitite (Ro = 0.25%)
Brown Coal – Detrohuminite (Ro = 0.25%)
Brown Coal (Ro = 0.25%)
Subbituminous Coal (Ro = 0.50%)
High Volatile Bituminous Coal (Ro = 0.70%)
High Volatile Bituminous Coal (Ro = 0.95%)
Low Volatile Bituminous Coal (Ro = 1.70%)
Anthracite (Ro = 2.50%)
Detrital mineral matter in subbituminous coal
Banding in Coal
Influence of Maceral Composition on Gas Content

Well #1

% Difference from Mean

Sample Number

Gas Content (m3/t)  Liptinite (%), mmf