Errors in Sampling

Fundamental Error

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Errors in Sampling - Contents

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Introduction

• If every coal particle in lot was identical ➔ only need to sample 1 x particle

• Coal has degree of variation (heterogeneity)

• The mass of sample required is a factor of how heterogeneous the coal is
Heterogeneity

There are two types of Heterogeneity:

- **Constitutional Heterogeneity (CH)** or Random Variation or Intrinsic Heterogeneity
  - Difference between each particle making up lot
  - Raw Coal has greater CH than washed product

- **Distributional Heterogeneity (DH)** or Segregation Heterogeneity
  - Scatter or spread of particles within lot
  - Grouping of particles will increase DH
  - Lumps rolling to outside of stockpile etc.
Heterogeneity - Errors

• **Constitutional Heterogeneity (CH)**
  - Error = Fundamental Error (FE)
  - Can be estimated before sampling
    » Pierre Gy
    » Dr Geoff Lyman
  - Use FE to calculate:
    » Mass of sample required
    » Sampling errors

• **Distributional Heterogeneity (DH)**
  - Error = Grouping & Segregation Error
  - Cannot be estimated before sampling
  - Determine per AS4264.1 or ISO13909-2
Distribution Heterogeneity

• Grouping & segregation - the “enemy” of representative sampling

True value red circles = 17.3%

Grab Sample
Weighted average red circles = 4.2%

• Sampling error should improve with more increments
• AS4264.1 & ISO13909-2 calculate number of increments required
• But error could still be large
Distribution Heterogeneity

- Segregation can give a consistent bias in samples
- Distribution Heterogeneity must be considered

True value
red circles = 17.3%

Beam of radiation
weighted average
red circles = 9.5%
Fundamental Error – Pierre Gy

Based on Constitutional Heterogeneity

\[
M_S = \left( \frac{c \ell f g d^3}{\theta^2_{FE}} \right)
\]

- \( M_S \) = Mass of Sample Required
- \( c \) = Mineralogical Factor (heterogeneity of material)
- \( \ell \) = Liberation Factor
- \( f \) = Shape Factor (how close particles are to cube)
- \( g \) = Particle Distribution Factor (size variation of particles)
- \( d \) = Nominal Top-size
- \( \theta^2_{FE} \) = Required Sampling Error
Fundamental Error – Mass of Sample

\[ M_S = \left( \frac{c \ell f g d^3}{\Theta_{FE}^2} \right) \]

- \( c \) = Mineralogical Factor - Calc. for 10% Ash
- \( \ell \) = Liberation Factor - Assume 0.1mm
- \( f \) = Shape Factor = 0.5
- \( g \) = Distribution Factor = 0.25
- \( d \) = Nominal Top-size = 31.5mm
- \( \Theta_{FE}^2 \) = Required Sampling Error = 0.1% Ash Absolute

\[ M_S = \text{Mass of Sample Required} = 52.3 \text{ kg} \]
CH Factor – Dr Geoff Lyman

- Size into multiple size fractions
- Washability on each size fraction

\[ K_S = \sum_{i=1}^{N_s} X_i V_i \sum_{j=1}^{N_d} Y_{ij} \frac{\rho_j}{a_T^2} \left( a_{ij} - a_T \right)^2 \]

\[ K_S = \text{Constitutional Heterogeneity Factor} \]
\[ N_s = \text{Number of Size Fractions} \]
\[ X_i = \text{Proportion of the Size Fractions in the Sample} \]
\[ V_i = \text{Average Volume of the Particles in the } i^{\text{th}} \text{ Size Fraction} \]
\[ Y_{ij} = \text{Proportion of a Density Fraction within a Size} \]
\[ \rho_i = \text{Average Density of Density Fraction} \]
\[ a_{ij} = \text{Ash of Density Fraction within a Size} \]
\[ a_{ij} = \text{Ash of Total Raw Sample} \]
### CH Factor – Dr Geoff Lyman

**Summary for each Size Fraction**

<table>
<thead>
<tr>
<th>Size Fraction (mm)</th>
<th>Mass (Xi)</th>
<th>Average Volume (Vi)</th>
<th>Wash Hetero Factor</th>
<th>Total Heterogeneity Factor (Ks)</th>
</tr>
</thead>
<tbody>
<tr>
<td>di</td>
<td>di+1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>26.4 +</td>
<td>13.2</td>
<td>0.231</td>
<td>5.1749</td>
<td>4.46</td>
</tr>
<tr>
<td>13.2 +</td>
<td>5.6</td>
<td>0.294</td>
<td>0.6189</td>
<td>0.182</td>
</tr>
<tr>
<td>5.6 +</td>
<td>2.8</td>
<td>0.139</td>
<td>0.0494</td>
<td>0.007</td>
</tr>
<tr>
<td>2.8 +</td>
<td>0.5</td>
<td>0.189</td>
<td>0.0055</td>
<td>0.001</td>
</tr>
<tr>
<td>0.5 +</td>
<td>0</td>
<td>0.147</td>
<td>0.0000</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.000</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Calculate the mass of material required to give the required Ash variance (0.1%)**

\[
M_s = \frac{K_s a_T^2}{\theta^2}
\]

= 16921 grams

= 17 kg
Fundamental Error – Uses

- Calculate mass of sample required
- Also to calculate sampling errors of sampling schemes
- “Sampling Error” does not mean a mistake has been made
- Precision of sampling caused by heterogeneity

\[ O^2 (FE) = \frac{fgcl \cdot d^3}{M_s} \]

- Bore-core programs
- General analysis samples
- Standard reference materials
- Estimate CH error in auto-sampler
## FE Worked Example – Bore-Core

![Diagram showing the process of extracting coal sample from bore core](image)

<table>
<thead>
<tr>
<th>Stage</th>
<th>Weight of Sample</th>
<th>Initial Size</th>
<th>Crush @</th>
<th>Constitution Heterogeneity</th>
<th>Fundamental Error Variance</th>
<th>Relative Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>grams</td>
<td>mm</td>
<td>mm</td>
<td>grams</td>
<td>RSD</td>
<td>%</td>
</tr>
<tr>
<td>Halving</td>
<td>2000</td>
<td>63</td>
<td>63</td>
<td>32.603</td>
<td>0.00815</td>
<td>9.03</td>
</tr>
<tr>
<td>Crush 25mm</td>
<td>2000</td>
<td>63</td>
<td>25</td>
<td>3.234</td>
<td>0.00000</td>
<td>0.00</td>
</tr>
<tr>
<td>Halving</td>
<td>1000</td>
<td>25</td>
<td>25</td>
<td>3.234</td>
<td>0.00162</td>
<td>4.02</td>
</tr>
<tr>
<td>Crush 4mm</td>
<td>1000</td>
<td>25</td>
<td>4</td>
<td>0.033</td>
<td>0.00000</td>
<td>0.00</td>
</tr>
<tr>
<td>RSD 300g</td>
<td>300</td>
<td>4</td>
<td>4</td>
<td>0.033</td>
<td>0.000008</td>
<td>0.88</td>
</tr>
<tr>
<td>Milling</td>
<td>300</td>
<td>4</td>
<td>0.212</td>
<td>0.000</td>
<td>0.000000</td>
<td>0.00</td>
</tr>
<tr>
<td>Ash result</td>
<td>1</td>
<td>0.212</td>
<td>0.212</td>
<td>0.00</td>
<td>0.000002</td>
<td>0.46</td>
</tr>
</tbody>
</table>

Total Variance (FE) = 0.00987

Total Fundamental Error = 9.93 %
## Sampling Error – Limits

### Pierre Gy's Recommended Limits for Total Fundamental Error

<table>
<thead>
<tr>
<th>Standard Reference Materials</th>
<th>&lt;0.5</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Superintending or bias testing</td>
<td>0.5</td>
<td>%</td>
</tr>
<tr>
<td>Process control or mining evaluation</td>
<td>1.5 - 5.0</td>
<td>%</td>
</tr>
<tr>
<td>Environmental or exploration</td>
<td>5 - 8</td>
<td>%</td>
</tr>
<tr>
<td>Early exploration</td>
<td>8 - 15</td>
<td>%</td>
</tr>
</tbody>
</table>

### Relative Standard Deviation

<table>
<thead>
<tr>
<th>Stage</th>
<th>Weight of Sample</th>
<th>Initial Size</th>
<th>Crush @</th>
<th>Constitution Heterogeneity</th>
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<th>Relative Standard Deviation</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>grams</td>
<td>mm</td>
<td>mm</td>
<td>grams</td>
<td>RSD</td>
<td>%</td>
</tr>
<tr>
<td>Halving</td>
<td>2000</td>
<td>63</td>
<td>63</td>
<td>32.603</td>
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<td>9.03</td>
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<tr>
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<td>25</td>
<td>3.234</td>
<td>0.00000</td>
<td>0.00</td>
</tr>
<tr>
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<td>1000</td>
<td>25</td>
<td>25</td>
<td>3.234</td>
<td>0.00162</td>
<td>4.02</td>
</tr>
<tr>
<td>Crush 4mm</td>
<td>1000</td>
<td>25</td>
<td>4</td>
<td>0.033</td>
<td>0.00000</td>
<td>0.00</td>
</tr>
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<td>RSD 300g</td>
<td>300</td>
<td>4</td>
<td>4</td>
<td>0.033</td>
<td>0.00008</td>
<td>0.88</td>
</tr>
<tr>
<td>Milling</td>
<td>300</td>
<td>4</td>
<td>0.212</td>
<td>0.000</td>
<td>0.00000</td>
<td>0.00</td>
</tr>
<tr>
<td>Ash result</td>
<td>1</td>
<td>0.212</td>
<td>0.212</td>
<td>0.000</td>
<td>0.00002</td>
<td>0.46</td>
</tr>
</tbody>
</table>

**Total Variance (FE) = 0.00987**

**Total Fundamental Error = 9.93 %**
FE Worked Example – Bore-Core

- 63mm Core 1 meter ~4kg
- Take 1/2 ~2kg
- Crush @25mm
- Take 1/4 ~1kg
- Crush @4mm
- Sample 300 g Pulverise
- Analyse Ash (1 gram)

<table>
<thead>
<tr>
<th>Stage</th>
<th>Weight of Sample</th>
<th>Initial Size</th>
<th>Crush @</th>
<th>Fundamental Error Variance</th>
<th>Relative Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>grams</td>
<td>mm</td>
<td>mm</td>
<td>$\sigma^2_{FE}$</td>
<td>%</td>
</tr>
<tr>
<td>Crush 25mm</td>
<td>4000</td>
<td>63</td>
<td>25</td>
<td>0.000000</td>
<td>0.00</td>
</tr>
<tr>
<td>Take 1/4</td>
<td>1000</td>
<td>25</td>
<td>25</td>
<td>0.00243</td>
<td>4.93</td>
</tr>
<tr>
<td>Crush 4mm</td>
<td>1000</td>
<td>25</td>
<td>4</td>
<td>0.000000</td>
<td>0.00</td>
</tr>
<tr>
<td>RSD 300g</td>
<td>300</td>
<td>4</td>
<td>4</td>
<td>0.00008</td>
<td>0.88</td>
</tr>
<tr>
<td>Pulverise</td>
<td>300</td>
<td>4</td>
<td>0.212</td>
<td>0.000000</td>
<td>0.00</td>
</tr>
<tr>
<td>Ash result</td>
<td>1</td>
<td>0.212</td>
<td>0.212</td>
<td>0.000002</td>
<td>0.46</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.00252</td>
<td></td>
</tr>
</tbody>
</table>

Total Fundamental Error = 5.02 %
## FE – Automatic Sample Plants

### Based on 60,000T Cargo

#### 2 Stage

<table>
<thead>
<tr>
<th>Stage</th>
<th>Number of Cuts</th>
<th>Mass of Increment</th>
<th>Total Mass</th>
<th>Initial Size</th>
<th>Crush @</th>
<th>Relative Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary Cut (every 118 Tonnes)</td>
<td>507</td>
<td>33</td>
<td>16,720</td>
<td>50</td>
<td>50</td>
<td>0.10</td>
</tr>
<tr>
<td>Crusher</td>
<td></td>
<td></td>
<td>16,720</td>
<td>50</td>
<td>11.2</td>
<td>0.00</td>
</tr>
<tr>
<td>Secondary Cut (6/primary)</td>
<td>3040</td>
<td>0.04</td>
<td>122</td>
<td>11.2</td>
<td>11.2</td>
<td>0.19</td>
</tr>
</tbody>
</table>

Total Fundamental Error = 0.22 %

#### 3 Stage

<table>
<thead>
<tr>
<th>Stage</th>
<th>Number of Cuts</th>
<th>Mass of Increment</th>
<th>Total Mass</th>
<th>Initial Size</th>
<th>Crush @</th>
<th>Relative Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary Cut (every 125 Tonnes)</td>
<td>480</td>
<td>575</td>
<td>276,000</td>
<td>50</td>
<td>50</td>
<td>0.03</td>
</tr>
<tr>
<td>Secondary Cut (6/primary)</td>
<td>2880</td>
<td>4</td>
<td>11,520</td>
<td>50</td>
<td>50</td>
<td>0.12</td>
</tr>
<tr>
<td>Crusher</td>
<td></td>
<td></td>
<td>11,520</td>
<td>50</td>
<td>11.2</td>
<td>0.00</td>
</tr>
<tr>
<td>Tertiary Cut (12/primary)</td>
<td>5760</td>
<td>0.053</td>
<td>305</td>
<td>11.2</td>
<td>11.2</td>
<td>0.12</td>
</tr>
</tbody>
</table>

Total Fundamental Error = 0.17 %
Other Sampling Errors

• FE = min error (if sampling is “perfect”)
• Many other errors occur in real life:
  • Delimitation Error
    • Cutters not moving @ constant speed
  • Extraction Error
    • Cutters not correct width
    • Ladle overflows during sampling
    • Not all particles accessible
  • Preparation Error
    • Errors from crushing, drying etc.
    • Losses, contamination, mistakes
Minimizing Sampling Errors

- Sample plants must be inspected
  - Regular visual audits
  - 12 monthly – full compliance audit
  - 3 years – full bias test

- Precision Testing on “routine” preparation
- Routine calibrations of equipment
- Correct sample labelling
- Training -- Training -- Training
Conclusion

“Typical examples are given by companies that are spending tremendous amounts of money trying to measure the effect of bad sampling instead of spending this money in finding the causes and correcting them”

Pierre Gy
Thank-You TO ALS Coal