Analysis of QAQC Data: How Good is Good Enough?

Or

How to Make a Talk on QAQC Interesting

Dennis Arne, PGeo (BC), RPGeo (AIG), Principal Consultant - Geochemistry, CSA Global
Outline

• What do the reporting codes have to say about QAQC?
• Sampling – getting it right from the start
• The importance of the correct digestion method
• QAQC train wrecks
• What’s it really all about?
• **Item 11: Sample Preparation, Analyses, and Security** – Describe
  
  – (a) sample preparation methods and quality control measures employed before dispatch of samples to an analytical or testing laboratory, the method or process of sample splitting and reduction, and the security measures taken to ensure the validity and integrity of samples taken;
  
  – (b) relevant information regarding sample preparation, assaying and analytical procedures used, the name and location of the analytical or testing laboratories, the relationship of the laboratory to the issuer, and whether the laboratories are certified by any standards association and the particulars of any certification;
  
  – (c) a summary of the nature, extent, and results of quality control procedures employed and quality assurance actions taken or recommended to provide adequate confidence in the data collection and processing; and
  
  – (d) the author's opinion on the adequacy of sample preparation, security, and analytical procedures.

But that’s not always the QP’s job!
• “The nature, quality and appropriateness of the assaying and laboratory procedures used and whether the technique is considered partial or total.”

• “For geophysical tools, spectrometers, handheld XRF instruments, etc, the parameters used in determining the analysis including instrument make and model, reading times, calibrations factors applied and their derivation, etc.”

• “Nature of quality control procedures adopted (standards, blanks, duplicates, external laboratory checks) and whether acceptable levels of accuracy (ie lack of bias) and precision have been established.”
• “The QAQC protocols meet current industry best practice.”

• “Paired field duplicate data suggest that the gold grades are difficult to reproduce by fire assay….However this trend is not uncommon in gold deposits with highly variable grades.”

• “In the opinion (of the QP), the data are sufficiently reliable for the purposes of resource estimation.”
Sampling & Analysis

- Sample size, grain size and analytical methods generally default to convention or what the labs recommend
- Should be optimised for individual projects (i.e. what are the data quality expectations for the project?)
- Objective is to obtain representative samples
- The digestion should be relevant for proposed mineral processing
- Have we maximised precision (i.e. minimised uncertainty)?
  - note that errors are additive; how are these uncertainties carried through to resource estimates?
  - potential to add metal value to a project through representative sampling
• Non-representative sampling does not reflect the underlying real \textit{in situ} distribution
• Samples at the low end of the false distribution report as waste
• Samples at the high end of the false distribution report as high-grade and are usually trimmed
• Results in an overall lowering of grade; value has been lost

From Sketchley, 1998; after Pitard, 1994
Sampling for Coarse Au - I

- ¼ core duplicate analyses compared to ½ core original samples
- Analyzed by 30 g fire assay
- Data show poor reproducibility
- Also show a bias toward higher grades in original ½ core samples
- These uncertainties carry through to the resource estimate

Data from George NI43-101 Technical report dated 2010
# Effect of Sample Size on Precision

## Effect of Sample Size on Precision

<table>
<thead>
<tr>
<th></th>
<th>25 g FA</th>
<th>RSD %</th>
<th>LW + tails</th>
<th>SFA</th>
<th>Bulk RSD (SFA/LW)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average grade</td>
<td>13.23</td>
<td></td>
<td>13.73</td>
<td>13.11</td>
<td></td>
</tr>
<tr>
<td>Average (all)</td>
<td>28</td>
<td></td>
<td></td>
<td></td>
<td>9</td>
</tr>
<tr>
<td>Average (area 1)</td>
<td>25</td>
<td></td>
<td></td>
<td></td>
<td>11</td>
</tr>
<tr>
<td>Average (area 2)</td>
<td>33</td>
<td></td>
<td></td>
<td></td>
<td>8</td>
</tr>
<tr>
<td>Average (&gt; 10 ppm)</td>
<td>30</td>
<td></td>
<td></td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>Average (&gt; 5 ppm)</td>
<td>30</td>
<td></td>
<td></td>
<td>10</td>
<td></td>
</tr>
</tbody>
</table>

RSD = relative standard deviation; LW + tails = 200 g Leachwell accelerated CN + tails fire assay; SFA = 200 g screened metallic fire assay

- Average grades and relative standard deviations calculated for 25 duplicate pairs
- Grade estimates are similar, but the bulk relative standard deviation is ~1/3 of that obtained for the 25 g fire assays using 200 g samples
Sampling for Coarse Au - II

Gold grades estimated over a 23 m interval at the Portia coarse gold deposit, South Australia

<table>
<thead>
<tr>
<th>Gold grade (g/t)</th>
<th>Method Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.7</td>
<td>2 kg samples pulverised for 50 g fire assay</td>
</tr>
<tr>
<td>0.8</td>
<td>2 kg screened metallic fire assay using 75 micron screen</td>
</tr>
<tr>
<td>6.6</td>
<td>10-12 kg of sample weighed, pulverised, tabled to produce a weighed heavy mineral concentrate which is then analysed by fire assay and the grade back-calculated based on the original weight</td>
</tr>
</tbody>
</table>

Data from Havilah Resources press release dated November 4, 2016
Sampling for Coarse Au - III

- Beatons Creek, Western Australia
- Pulverisation of 9 kg sample; split of 3 kg analyzed by Leachwell accelerated CN analyses

Data from van Heerdan NI43-101 Technical report dated October 1, 2015
Are Fire Assays Sufficient?

<table>
<thead>
<tr>
<th>CL Test</th>
<th>Oxidation</th>
<th>Grain Size (µm)</th>
<th>Grade</th>
<th>Au Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxide</td>
<td>100 %</td>
<td>96</td>
<td>7.0</td>
<td>96.0</td>
</tr>
<tr>
<td>Oxide</td>
<td>100 %</td>
<td>99</td>
<td>7.0</td>
<td>96.5</td>
</tr>
<tr>
<td>Transitional</td>
<td>95 %</td>
<td>108</td>
<td>3.4</td>
<td>90.7</td>
</tr>
<tr>
<td>Transitional</td>
<td>95 %</td>
<td>114</td>
<td>3.4</td>
<td>91.5</td>
</tr>
<tr>
<td>Fresh</td>
<td>0 %</td>
<td>90</td>
<td>4.4</td>
<td>2.0</td>
</tr>
<tr>
<td>Fresh</td>
<td>0 %</td>
<td>35</td>
<td>4.4</td>
<td>2.8</td>
</tr>
<tr>
<td>Fresh</td>
<td>0 %</td>
<td>20</td>
<td>4.4</td>
<td>5.3</td>
</tr>
</tbody>
</table>

- ~50 % of original reported resource is from transitional material
- Primary sulphide mineralisation strongly refractory
- Recoveries in the transition zone are directly proportional to oxidation
- Cyanide Au recoveries important for a proposed heap leach operation
A fire assay gives a total Au value for this sample.
Cyanide digestion will only liberate the free Au component.
A component of Au is refractory and requires floatation.
Metallurgical testing was done on a single sample based on underground access.
Complex mineralogy = complex recoveries.

**High fineness Au (supergene?)**

**Sb-Ag-Au sulphosalt**

**Aurostibite (AuSb₂)**

**Au with ~20% Ag**
### Appropriate Assay Methods

- **Leachwell (accelerated CN digestion)** with a tails fire assay of provides:
  - an estimate of the CN-extractable Au in the sample
  - an estimate of the refractory component
  - the two values combined provide a more precise bulk analysis
  - ratio of free to refractory Au in each sample

<table>
<thead>
<tr>
<th>Sample</th>
<th>25 g Fire Assay (ppm)</th>
<th>25 g Fire Assay Repeat (ppm)</th>
<th>200 g Leachwell (ppm)</th>
<th>25 g Leachwell Tails Fire Assay (ppm)</th>
<th>Total Au from BLEG + Fire Assay (ppm)</th>
<th>% Cyanide soluble to total Au</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample008</td>
<td>13.16</td>
<td>n/a</td>
<td>1.16</td>
<td>12.73</td>
<td>13.89</td>
<td>8.8</td>
</tr>
<tr>
<td>Sample009</td>
<td>17.23</td>
<td>13.03</td>
<td>5.00</td>
<td>12.32</td>
<td>17.32</td>
<td>33.0</td>
</tr>
<tr>
<td>Sample010</td>
<td>5.23</td>
<td>n/a</td>
<td>0.43</td>
<td>5.35</td>
<td>5.78</td>
<td>8.2</td>
</tr>
<tr>
<td>Sample011</td>
<td>13.12</td>
<td>12.33</td>
<td>4.72</td>
<td>5.50</td>
<td>10.22</td>
<td>37.0</td>
</tr>
<tr>
<td>Sample012</td>
<td>30.24</td>
<td>30.60</td>
<td>19.95</td>
<td>12.38</td>
<td>32.33</td>
<td>65.6</td>
</tr>
<tr>
<td><strong>Sample013</strong></td>
<td><strong>38.70</strong></td>
<td><strong>27.98</strong></td>
<td><strong>47.85</strong></td>
<td><strong>0.75</strong></td>
<td><strong>48.6</strong></td>
<td><strong>98</strong></td>
</tr>
</tbody>
</table>

Note that refractory Au analyses are repeatable; precision decreases with increasing free Au.
Either quantitative or semi-quantitative information on metal recoveries can be built into block models.

### Upgrade in Gold Assay Results

- Example is from a Au-rich epithermal system in Turkey
- Samples were re-assayed using 500 g screened metallics fire assay
- A 12 m interval shows an average upgrade in Au values of 17.5%
- Potential to add metal value to this project

<table>
<thead>
<tr>
<th>From (m)</th>
<th>To (m)</th>
<th>Interval (m)</th>
<th>Au (g/t) original fire assay</th>
<th>Au (g/t) screened metallics</th>
<th>Change %</th>
</tr>
</thead>
<tbody>
<tr>
<td>116.0</td>
<td>117.5</td>
<td>1.5</td>
<td>2.26</td>
<td>2.35</td>
<td>4.1</td>
</tr>
<tr>
<td>117.5</td>
<td>119.0</td>
<td>1.5</td>
<td>47.9</td>
<td>44.7</td>
<td>-6.6</td>
</tr>
<tr>
<td>119.0</td>
<td>120.5</td>
<td>1.5</td>
<td>12.8</td>
<td>13.5</td>
<td>5.4</td>
</tr>
<tr>
<td>120.5</td>
<td>122.0</td>
<td>1.5</td>
<td>338</td>
<td>382</td>
<td>13.1</td>
</tr>
<tr>
<td>122.0</td>
<td>123.5</td>
<td>1.5</td>
<td>236</td>
<td>231</td>
<td>-2.3</td>
</tr>
<tr>
<td>123.5</td>
<td>125.0</td>
<td>1.5</td>
<td>73.0</td>
<td>92.8</td>
<td>27.1</td>
</tr>
<tr>
<td>125.0</td>
<td>126.5</td>
<td>1.5</td>
<td>681</td>
<td>880</td>
<td>29.3</td>
</tr>
<tr>
<td>126.5</td>
<td>128.0</td>
<td>1.5</td>
<td>17.2</td>
<td>16.2</td>
<td>-5.6</td>
</tr>
<tr>
<td>128.0</td>
<td>129.5</td>
<td>1.5</td>
<td>140</td>
<td>157</td>
<td>11.8</td>
</tr>
<tr>
<td>129.5</td>
<td>131.0</td>
<td>1.5</td>
<td>3.89</td>
<td>3.63</td>
<td>-6.6</td>
</tr>
</tbody>
</table>

**Total: 12.0 metres (117.5m-129.5m) averaging:**

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th>Au (g/t) original fire assay</th>
<th>Au (g/t) screened metallics</th>
<th>Change %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>193 g/t</td>
<td>227 g/t</td>
<td>17.50%</td>
</tr>
</tbody>
</table>

Data from Pilot Gold press release 22/01/2013
An Exploration Example

- Original 10g aqua regia soil results not reproducible
- 500 g splits trialled using cyanide extraction
- Significantly more anomalous samples @ >10 ppb Au in bulk samples
- 20% more highly anomalous samples @ 100 ppb Au in bulk samples

High probability of false negatives (missed opportunities)
Grain Size Matters

Steam Sediment Orientation Data Set – Golden Saddle/Coffee Deposits

- Field duplicate orientation stream sediment samples analysed by:
  - Standard -177 micron (#80) and aqua regia digest
  - 1 kg -177 micron conventional BLEG
  - <2 micron clay fraction and aqua regia digest
- Only the clay fraction separates gave good field reproducibility (ie. no nugget effect)

Data from Arne & MacFarlane, 2014
The QAQC Iceberg

- What level of control is required?
- Where is the major source of variation?
- Are you using coarse blanks to assess cross contamination?
- Are the certified reference materials (CRMs) appropriate?
- Do the CRMs cover the range of expected grades & anticipated cut-off?
- Are suitable procedures documented?
- Can you access internal laboratory QAQC data?
- What constitutes a QAQC failure?

Too many QAQC failures reduce confidence in the data.
### PERFORMANCE GATES FOR OREAS 42P - MINOR ELEMENTS

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Recommended value</th>
<th>1 Standard Deviation</th>
<th>Performance Gates</th>
<th>Performance Gates</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>2σ</td>
<td>3σ</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>4 Acid arsenic, As (ppm)</td>
<td>113</td>
<td>16.6</td>
<td>80</td>
<td>147</td>
</tr>
<tr>
<td>Perchloric arsenic, As (ppm)</td>
<td>107</td>
<td>8.36</td>
<td>90</td>
<td>124</td>
</tr>
<tr>
<td>AR arsenic, As (ppm)</td>
<td>100</td>
<td>10.4</td>
<td>79</td>
<td>121</td>
</tr>
<tr>
<td>3A arsenic, As (ppm)</td>
<td>104</td>
<td>20.8</td>
<td>62</td>
<td>146</td>
</tr>
<tr>
<td>PF arsenic, As (ppm)</td>
<td>131</td>
<td>16.6</td>
<td>98</td>
<td>165</td>
</tr>
<tr>
<td>PPPXRF arsenic, As (ppm)</td>
<td>106</td>
<td>8.3</td>
<td>90</td>
<td>123</td>
</tr>
</tbody>
</table>

- Use the standard deviation from the clean round robin data
- **Not** the confidence interval on the mean determination
- Make sure digestion method is appropriate

CRM – certified reference material
The CRM Should be Matrix Appropriate!

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Au (ppb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soil</td>
<td>1.5</td>
</tr>
<tr>
<td>Soil</td>
<td>2</td>
</tr>
<tr>
<td>Fresh sulphide CRM</td>
<td>2720</td>
</tr>
<tr>
<td>Soil (memory effect)</td>
<td>32.1</td>
</tr>
<tr>
<td>Soil (memory effect)</td>
<td>7.5</td>
</tr>
<tr>
<td>Soil</td>
<td>3.4</td>
</tr>
<tr>
<td>Soil</td>
<td>2.5</td>
</tr>
<tr>
<td>Soil</td>
<td>2.3</td>
</tr>
<tr>
<td>Soil</td>
<td>2.2</td>
</tr>
<tr>
<td>Soil</td>
<td>1.5</td>
</tr>
<tr>
<td>Soil</td>
<td>1.3</td>
</tr>
</tbody>
</table>

- Targeting soil anomalies >5 ppb Au
- Inappropriate CRM matrix
- Inappropriate grade
- Carry-over of Au in the ICP-MS (memory effect)
- Data needed to be cleaned before use; if not caught it would have led to many false anomalies
Old bulk CRM (blue) compared to fresh CRM (red).

- Client and the lab were using the same CRM
- Client CRM results were good
- Laboratory CRM results were bad
- Lab refused to release the data
- Client became very frustrated
- Problem was resolved when the lab purchased new CRM
- Reason for the discrepancy is unclear (gravity settling during transport? oxidation of Ni sulphides?)

Vancouver MEG, December 15, 2016
### What Constitutes a CRM Failure?

<table>
<thead>
<tr>
<th>Rule</th>
<th>Description</th>
<th>Chart Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rule 1</td>
<td>Any single data point falls outside the $3\sigma$ limit from the centerline (i.e., any point that falls outside Zone A, beyond either the upper or lower control limit)</td>
<td><img src="chart1.png" alt="Rule 1: Any point beyond Zone A chart example" /></td>
</tr>
<tr>
<td>Rule 2</td>
<td>Two out of three consecutive points fall beyond the $2\sigma$ limit (in Zone A or beyond), on the same side of the centerline</td>
<td><img src="chart2.png" alt="Rule 2: Two out of three consecutive points fall Zone A or beyond chart example" /></td>
</tr>
</tbody>
</table>

- Codified in 1956 by the Western Electric Company for white goods coming off the assembly line
- Allows for a simple pass/fail decision to be made at batch level
- Can be made more complicated, but then becomes harder to implement

(Modified from Wikipedia, accessed Sept. 13, 2013)
There Are Statistical “Failures”

11 of 225 > 2 SD = ~5%, which is statistically expected

1 of 225 > 3 SD = ~0.4%, which is also statistically expected

The Au CRM results show the natural statistical spread in the data.
All Labs Have a Bias

- Assessment of client CRMs showed a positive bias.
- Internal laboratory QAQC data shows instrumental drift over the period of concern
- Re-assisys performed after instrumental correction
- Intervention was timely – overall data quality >98 % pass rate
What’s an Acceptable Bias?

- The 5% average negative bias for this CRM is similar to the uncertainty in the certified mean of the CRM
- What are the data to be used for?
  - Exploration?
  - Grade control?
  - Resource estimation?
  - PEA, PF, BFS?
- The bias will come through into the resource model if the data are not adjusted
Induced Bias

- A negative bias can be introduced into a dataset by selectively reassaying only high values and averaging the results.
- High assays have a chance to decrease but low assays don’t have a chance to increase.
- Check assays should be random, but focused on mineralised intervals.

(Figure modified from Clifton et al., 1969, Sample size and meaningful gold analysis, USGS Prof. Paper 625C)
Is Your Blank Contaminated?

- Locally sourced sand used as blanks but not analysed to demonstrate blankness
- Blank contains nuggetty Au (best anomalies!)
- Other bloopers include using:
  - Nipissing diabase for PGE project (high background Pd)
  - Local laterite for oxidised Au project (supergene Au?)
Cross Contamination

<table>
<thead>
<tr>
<th>Sample</th>
<th>Au_ppm</th>
<th>Ag_ppm</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>992308</td>
<td>202.4</td>
<td>198.3</td>
<td>Core</td>
</tr>
<tr>
<td>992309</td>
<td>13.9</td>
<td>10.6</td>
<td>Blank</td>
</tr>
</tbody>
</table>

- Samples were contaminated at the crushing stage
- Re-assay of blank coarse rejects also indicates high values
- Core had to be re-sampled
- Note that including a blank right after a high-grade sample will not always be effective if dual pulverisers are used, as pictured

* Note that the prep lab involved in the cross contamination is not the one pictured

(photo courtesy of Chris Lee)
Quality Expectations

- Quality expectations will depend on project stage
- Data need to be fit for purpose
- The greater the uncertainties at the sampling and analytical stage, the greater the uncertainties propagated through to mineral resources and mineral reserves
- Uncertainties can be included in final grade and tonnage estimates (i.e. +/- X)

Increasing assay data accuracy, precision and relevance

Figure 1 from JORC 2012
Conclusions

- Errors are additive - it starts with sampling
- Set corporate data quality objectives early
- Data should be fit for purpose
- Quality expectations should evolve with the project
- Focus on the desired result (JORC), not the process (NI43-101)
- Don’t forget the quality assurance (follow up failures promptly)
- Mitigate risk by propagating sampling and analytical uncertainties through to resource estimates
- Representative sampling has the potential to add value