LBMA Proficiency Testing

Jonathan J. Jodry
Metalor Technologies SA, Switzerland

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What is a Proficiency Testing?

Sample composition, Participation, Z-scores

Detailed results, Issues

Statistics, Corrective actions

Conclusion

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What is a Proficiency Testing?

- Also called *Interlaboratory testing* or *Round robin*
- Homogeneous material analysed by multiple laboratories
- Comparison of results obtained on an anonymous basis

Z-scores definition: Reference = mean of all valid results

- $Z = 1$ for $0.049\%$ (fixed by LBMA)
- $Z = 2$
- $Z = 3$
- $Z = -1$
- $Z = -2$
- $Z = -3$

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Why Join a Proficiency Testing

- detection of (systematic) errors
- validating new analytical methods
- discovery of method sensitivity
- analysing different samples
- comparing performance with competitors
- assessing performance and capabilities of staff
- planning for equipment and training
- requirement of ISO 17025 standard

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6 years of Au PT

- From 995 to 999.5
- Classic impurities: Ag, Cu, Pt, Pd
- In 2017: Zr added

First year for Ag PT

- Pd-Au-Pt

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Participation & Results

Number of laboratories joining the PT

<table>
<thead>
<tr>
<th>Year</th>
<th>2012</th>
<th>2013</th>
<th>2014</th>
<th>2015</th>
<th>2016</th>
<th>2017</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total</td>
<td>40</td>
<td>40</td>
<td>41</td>
<td>49</td>
<td>48</td>
<td>48</td>
</tr>
<tr>
<td>Non-GDL</td>
<td>10</td>
<td>14</td>
<td>12</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2017 Ag: 38
Non-GDL: 11

Z-score

-2 < Z < 2
2 < |Z| < 3
|Z| > 3

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2017 Ag Detailed Results

- ISO accredited (GD)
- ISO accredited (non-GD)
- non accredited (GD)
- non accredited (non-GD)

---Reference

- 10 labs out of 49 (20%) would have failed GDL application (+/- 0.15‰)
- 5 labs did not submit average value!
- Non-Spectroscopy methods (titration, fire assay, gravimetry) are not robust enough
17 labs out of 60 (28%) would have failed GDL application (+/-0.15‰)

Significant and abnormal bias toward higher titles
- More than 50% of issues are with spectroscopy!
- 6 labs did not identify the presence of Zr in the sample
- 7 GD labs would have qualified this material as 995+…
Comment from lab 003:

- We decide to perform an additional ICP-OES analysis because the fire assay result was < 995. ICP-OES analysis is perfectly aligned with the fire assay and (...) we found 518 ppm of Zr.
• Good overall performance
2016 Au Detailed Results

- Good overall performance

Fire Assay
Fire Assay (non-GD)
Spectroscopy (difference)
Spectroscopy (difference, non-GD)
Reference

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Fire Assay vs ICP-OES

Results normalised to 1000

After reprocessing (2016 removing NG labs; 2017 removing “Zr issue” labs):

FA

ICP

Results normalised to 1000

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Uncertainty of ICP-OES is proportional to element concentration...

<table>
<thead>
<tr>
<th></th>
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</thead>
<tbody>
<tr>
<td>Mean</td>
<td>1054</td>
<td>2033</td>
<td>3263</td>
<td>3988</td>
</tr>
<tr>
<td>St. Dev.</td>
<td>39</td>
<td>42</td>
<td>81</td>
<td>144</td>
</tr>
<tr>
<td>St. Dev. %</td>
<td>3.7%</td>
<td>2.1%</td>
<td>2.5%</td>
<td>3.6%</td>
</tr>
</tbody>
</table>

St. Dev. [mg/kg] vs Impurity [mg/kg]
How to react to a bad Z-score

So you received your lab performance… Z-score out of the -2 to +2 area?

PT is about learning and improving, not “passing” or “failing”

Don’t

• Hide the result to your staff / auditors / quality staff / management
• Assume the issue is not important or will be taken care of by “someone”
• Limit investigation at the first problem identified

Do

• Study results in detail
  - mean value
  - standard deviation
  - false positive for impurities
  - error on specific impurities
How to react to a bad Z-score

So you received your lab performance… Z-score out of the -2 to +2 area?

PT is about learning and improving, not “passing” or “failing”

Don’t

• Hide the result to your staff / auditors / quality staff / management
• Assume the issue is not important or will be taken care of by “someone”
• Limit investigation at the first problem identified

Do

• Study results in detail
• Identify all possible sources of problems: Preparation, Method, Equipment, Calculation, Reporting
• Root cause investigation: Investigate, Implement corrective actions, Execute analysis, Control effectiveness
• Adapt your methods or procedures, inform and train staff
• Participate next year and see how much you improved!

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Danger of Synthetic Samples

PT supervised by Referee Committee (composition, preparation)

Goal is:
- Cover the whole 995 to 999.9‰ range
- Composition not too simple (e.g. Au-Ag binary), not too complex
- No “exotic” element
- Samples are specifically prepared for PT

Those samples are however NOT real-life materials…

Risk is:
- Not having impurities which can be found in the refineries
- Oversimplifying the compositions
- Creating a false sense of “comfort”

Usually, real factory samples lead to worse results than PT samples!!
Two years ago…

Future of PT

- Au: Different compositions 995-999.9
- Improvement of spectroscopic methods for Au 995
- Increase participations of non-GD laboratories
- New methods (MP-AES, ICP-MS, …)
- Ag PT

For next two years…

- Improvement of the laboratory performances
- Significantly lowering outliers laboratories
- More complex samples (!)
Please help improving the PT…

• Submit everything requested (enough replica, mean)
• Answer clearly ISO 17025 accreditation question
• Give clearly the name of the method used for measurement, e.g. “ICP-OES”, “Fire Assay” or “Gravimetry”, not “Spectroscopy” or “By difference”
• Use the comment box to describe combined methods

Thanks to…

• LBMA staff and former staff, especially Neil Harby, Varsha Peiris, Ruth Crowell and Stewart Murray
• FAPAS
• All my referee colleagues