Organization of proficiency tests on physicochemical properties of plant protection products

Alain Dubois, Franck Defeijt and Geert De Poorter
BU PT Schemes
# Table of content

1. Introduction  
2. Origin of the project  
3. Time schedule  
4. Process  
5. Observations  
6. Further developments  
7. References  
8. Contact point
Table of content

1. Introduction
2. Origin of the project
3. Time schedule
4. Process
5. Observations
6. Further developments
7. References
8. Contact point
1. Introduction

In Belgium, it is the Federal Agency for the Safety of the Food Chain and in particular one of its labs based in Liège that is in charge of the analysis of plant protection products.

This lab as the most of those involved in the quality control of pesticides are ISO/IEC 17025 accredited. According to this standard (item 5.9.1), these laboratories must assure the quality of their test results.
1. Introduction

How?

• regular use of certified reference materials and/or internal quality control using secondary reference materials.
• participation in interlaboratory comparison or proficiency-testing programmes;
• replicate tests using the same or different methods;
• retesting of retained items.
1. Introduction

How?

- regular use of certified reference materials and/or internal quality control using secondary reference materials.
- participation in interlaboratory comparison or proficiency testing programmes;
- replicate tests using the same or different methods;
- retesting of retained items.

Biased results ???
Table of content

1. Introduction
2. Origin of the project
3. Time schedule
4. Process
5. Observations
6. Further developments
7. References
8. Contact point
2. Origin of the project

• In 2007, the DG Laboratories of FASFC wanted to develop by itself some PT needed for its own labs and not available from the usual providers.

• Such a missing PT was naturally pointed for the analysis of plant protection products.
2. Origin of the project

• During the 2007 CIPAC Meeting, discussions with other colleagues on this topic: it appears same problem occurs for all of them.
• Small survey on the opportunity to organize such a PT: received this proposal warmly.
2. Origin of the project

- In 2008, decision to organize this PT on conditions that:
  - Analysis of real formulations;
  - Available in Belgium and widely used;
  - Availability of a CIPAC method for a.i. determination (and alternation of analytical techniques: HPLC – GC for successive PT’s);
2. Origin of the project

- Restricted to European participants (to limit customs and logistics problems).
- Assisted by a technical working group of experts in plant protection products analysis.
- Limited registration fee.
- ISO/IEC 17043:2010 accreditation asap!

Obtained in 2011.
Table of content

1. Introduction
2. Origin of the project
3. Time schedule
4. Process
5. Observations
6. Further developments
7. References
8. Contact point
3. Time schedule

1. Technical working group briefing:
   Choice of formulation, parameters, conditions of analysis, sample amount, …
2. Formulation purchase.
3. Contact with potential participants (Information sheet and order form to save time).
3. Time schedule

4. Samples preparation (packaging).

5. Homogeneity tests.
3. Time schedule

6. Samples sending with Analysis Request form (all details of the analysis) + stability tests (2 months).

7. Receipt of results and writing of preliminary report (2 weeks).

8. Receipt of participants’ comments (2 weeks).

9. Follow-up meeting and writing of final report.
3. Time schedule

10. Sending of the final report (< 2 months) and satisfaction questionnaire.
11. Debriefing (including satisfaction survey evaluation) (+ briefing for the next PT).
Table of content

1. Introduction
2. Origin of the project
3. Time schedule
4. Process
5. Observations
6. Further developments
7. References
8. Contact point
3. Process

Results' evaluation

• Statistics according ISO 13528 (Algorithm A).
• Assigned value = Consensus value from participants’ results.

Chosen because no reference values available (no reference materials) and no historical data to design expert laboratories. None of the participants is accorded higher status.
3. Process

- Performance assessment:

\[ z\text{-score} = \frac{x - X}{\sigma} \]

where:
- \( x \) = the result reported by the participating laboratory;
- \( X \) = the robust mean;
- \( \sigma \) = the robust standard deviation for proficiency assessment.
3. Process

- The z-score can be interpreted as:
  
  \[
  \begin{align*}
  |z| \leq 2 & : \text{ satisfactory result;} \\
  2 < |z| \leq 3 & : \text{ questionable result;} \\
  |z| > 3 & : \text{ unsatisfactory result.}
  \end{align*}
  \]
3. Process

Background history of these PT’s
<table>
<thead>
<tr>
<th>Year</th>
<th>Number of Participants</th>
<th>Formulation</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>2008-2009</td>
<td>13</td>
<td>Soluble concentrate (SL)</td>
<td>5 : a.i. content (g/kg and g/l), density, pH of formulation and 1% dilution, foaming properties.</td>
</tr>
<tr>
<td>2010</td>
<td>14</td>
<td>Aqueous suspension concentrate (SC)</td>
<td>8 : a.i. content (g/kg and g/l), density, pH of formulation and 1% dilution, foaming properties, wet sieve test, suspensibility.</td>
</tr>
<tr>
<td>2011</td>
<td>17</td>
<td>Water dispersible granules (WG)</td>
<td>7 : a.i. content, pH of 1% dilution, foaming properties, wet sieve test, suspensibility, wettability, degree of dispersion.</td>
</tr>
<tr>
<td>2012</td>
<td>17</td>
<td>Aqueous suspension concentrate (SC)</td>
<td>9 : a.i. content (g/kg and g/l), density, pH of formulation and 1% dilution, foaming properties, wet sieve test, suspensibility, spontaneity of dispersion.</td>
</tr>
<tr>
<td>2013</td>
<td>17</td>
<td>Soluble granules (SG)</td>
<td>7 : a.i. content, water, acidity-alcalinity, pH of 1% dilution, foaming properties, dissolution degree and dilution stability, flowability.</td>
</tr>
</tbody>
</table>
Participants
Table of content

1. Introduction
2. Origin of the project
3. Time schedule
4. Process
5. Observations
6. Further developments
7. References
8. Contact point
5. Observations

• The results are generally very good;
• Some « simple » tests like pH can cause problems to participants;
• Non-respect of analytical conditions (water quality for dilution);
• Calculation errors.
• Methods are not always very clear.
5. Observations

• For some parameters, difficulty to evaluate the results because the distributions are not normal.
• Not follow blindly the statistics for the evaluation of the results.
• Not easy to find « poor » formulations:
  - with foam production, residues on sieve, suspensibility different from 100%, …
5. Observations

- We try as far as possible to give to the participants points of reflection to solve the encountered problems.
- The participants are globally very satisfied with the organization of these PT’s (4.5/5).
Table of content

1. Introduction
2. Origin of the project
3. Time schedule
4. Process
5. Observations
6. Further developments
7. References
8. Contact point
5. Further developments

- Continue to improve the quality of our PT’s.

- Increase the number of participants.

- Increase the frequency of PT or the number of samples per round
Table of content

1. Introduction
2. Origin of the project
3. Time schedule
4. Process
5. Observations
6. Further developments
7. References
8. Contact point
6. References

• General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005).

• Conformity assessment — General requirements for proficiency testing (ISO/IEC 17043:2010).

• Statistical methods for use in proficiency testing by interlaboratory comparisons (ISO 13528:2005)
# Table of content

1. Introduction
2. Origin of the project
3. Time schedule
4. Process
5. Observations
6. Further developments
7. References
8. Contact point
7. Contact point

Federal Agency for the Safety of the Food Chain
BU PT Schemes
Chaussée de Namur, 22
B-5030 Gembloux
Belgium
BUPTSchemes@afsca.be

Tel.: + 32 81 62 03 00
Fax: + 32 81 62 03 01
Acknowledgements

I want to thank all the participating laboratories that trusted us. All my gratitude goes also to the members of Technical Group and to the collaborators of the laboratories of Gembloux and Liège involved in the preparation of these PT’s.
Thank you for your attention!