

Federal Agency for the Safety of the Food Chain 58<sup>th</sup> CIPAC Symposium Liège, Belgium (2014/06/24)

Organization of proficiency tests on physicochemical properties of plant protection products

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Federal Agency for the Safety of the Food Chain

Our task is to preserve the safety of the food chain and the quality of food in order to protect the health of humans, animals and plants.

**Fhe Food Agency** 

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## **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;
  Biased results ???
- retesting of retained items.



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- 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.





- 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.





- 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);







- 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.





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# 3. Time schedule

- Technical working group briefing: Choice of formulation, parameters, conditions of analysis, sample amount, ...
- 2. Formulation purchase.
- 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

- 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).





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#### 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-score = (x-X)/σ

## where: x = the result reported by the participating laboratory; X = the robust mean; $\sigma =$ the robust standard deviation for proficiency assessment.



#### 3. Process

 $2 < |z| \le 3$ :

- The z-score can be interpreted as :
  - $|z| \le 2$ : satisfactory result;
    - questionable result ;
  - |z| > 3 : unsatisfactory result.







#### **Background history of these PT's**





Year	Number of Participants	Formulation	Parameters
2008-2009	13	Soluble concentrate (SL)	5 : a.i. content (g/kg and g/l), density, pH of formulation and 1% dilution, foaming properties.
2010	14	Aqueous suspension concentrate (SC)	8 : a.i. content (g/kg and g/l), density, pH of formulation and 1% dilution, foaming properties, wet sieve test, suspensibility.
2011 BEB LAC 014-PT	17	Water dispersible granules (WG)	7 : a.i. content, pH of 1% dilution, foaming properties, wet sieve test, suspensibility, wettability, degree of dispersion.
2012 BBB LAC 014-PT	17	Aqueous suspension concentrate (SC)	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.
2013 BBB LAC 014-PT	17	Soluble granules (SG)	7 : a.i. content, water, acidity- alcalinity, pH of 1% dilution, foaming properties, dissolution degree and dilution stability, flowability.

(11)

## **Participants**



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# 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.







- 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%, ...





- 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).





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## 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 ???



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# 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)



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## 7. Contact point

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#### Thank you for your attention !



