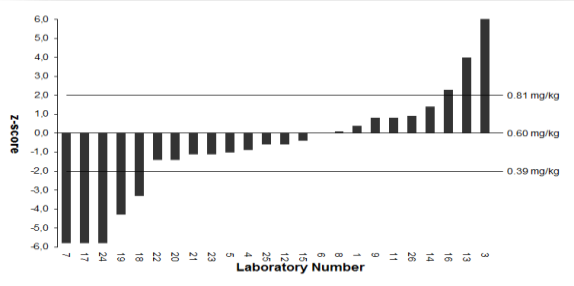
Proficiency studies, what we have learned? A review of the Proficiency Test conducted by the CORESTA Agrochemical Analysis Sub-Group

CORESTA Congress - Quebec
October 15th, 2014



Hans Braeckman – Fytolab
Marco Prat – Japan Tobacco International

Collaborative Workshop

Outline

Marco Prat

- Agrochemical Analysis Sub-Group objectives
- Proficiency Test history
- Laboratories' profile and participation over the years
- CPA tested over the years

Hans Braeckman

- Continuous participation to CORESTA-FAPAS Proficiency Test - a tool for better analytical quality

Agrochemical Analysis Sub-Group objectives

... laboratory performance improvement

- To perform regular Proficiency Testing of Multi-Residue Methods for the analysis of agrochemical residues on tobacco

- Undertake Joint Experiments to resolve unanswered questions arising from proficiency tests; to expand knowledge base on agrochemical residues and their analysis

- Produce and maintain a series of Technical Notes (on different agrochemical residue classes and selected individual compounds) to supplement the Technical Guideline and aid method development and improvement

AA SG Proficiency Test

History

2004 Pilot Run performed by the AA SG with the support of Swedish Match

2005
2006
2007
2008

Proficiency Test conducted in collaboration with FAPAS

- max. 10 CPAs
- analytical methods submitted to FAPAS and included in the official report

2009
2010
2011
2012
2013
2014

Proficiency Test conducted in collaboration with FAPAS

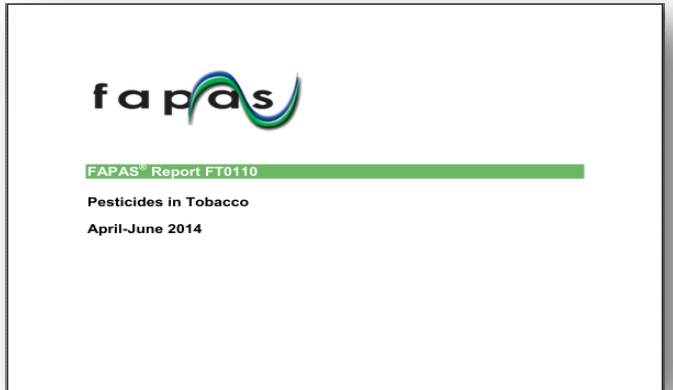
- max. 20 CPA s
- analytical methods collected by the AA SG and provided to the PT participant laboratories

CORESTA-FAPAS

Tobacco Proficiency Test

Food **A**nalysis **P**erformance **A**ssessment **S**cheme

- FAPAS is the largest and most comprehensive analytical chemistry proficiency testing scheme in the food sector. The scheme has more than 2,000 participants in over 100 countries
- FAPAS is an activity of the **F**ood and **E**nvironment **R**esearch **A**gency (Fera) → Executive Agency of the UK **D**epartment for **E**nvironment, **F**ood and **R**ural **A**ffairs (Defra)
- Proficiency Testing for Food Chemistry. Tests available include the following analytes in:
  - Food, feed and drink nutritional components
  - Food ingredients
  - Natural food contaminants
  - Organic & inorganic contaminants
  - Pesticides
  - Veterinary drug residues
  - Food additives
  - Migration from food packaging
  - Allergens
  - Authenticity

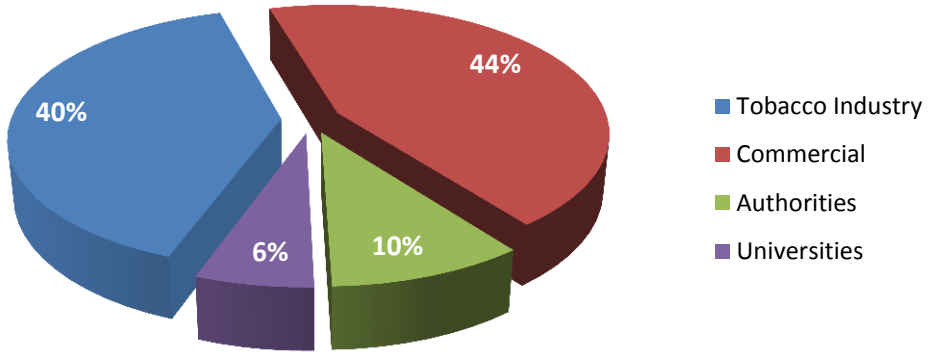


AA SG Proficiency Test

Laboratories' profile

52 CORESTA and non CORESTA members laboratories have taken part to the CORESTA-FAPAS Proficiency Test from 2004 to 2014

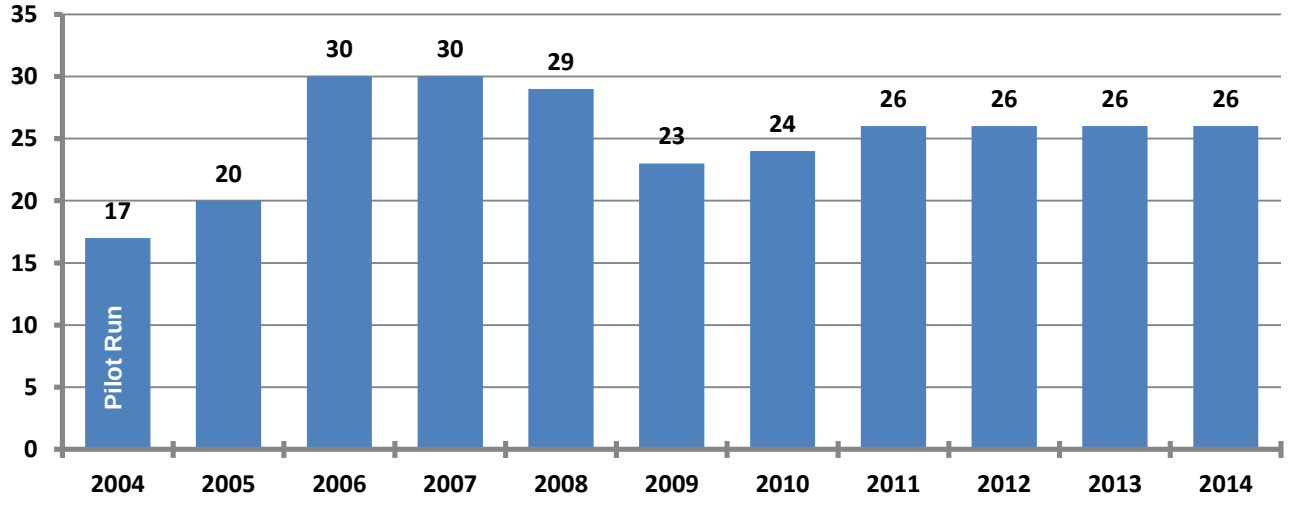
- Tobacco Industry → 21
- Commercial → 23
- Authorities → 5
- Universities → 3



AA SG Proficiency Test

Number of laboratories over the years

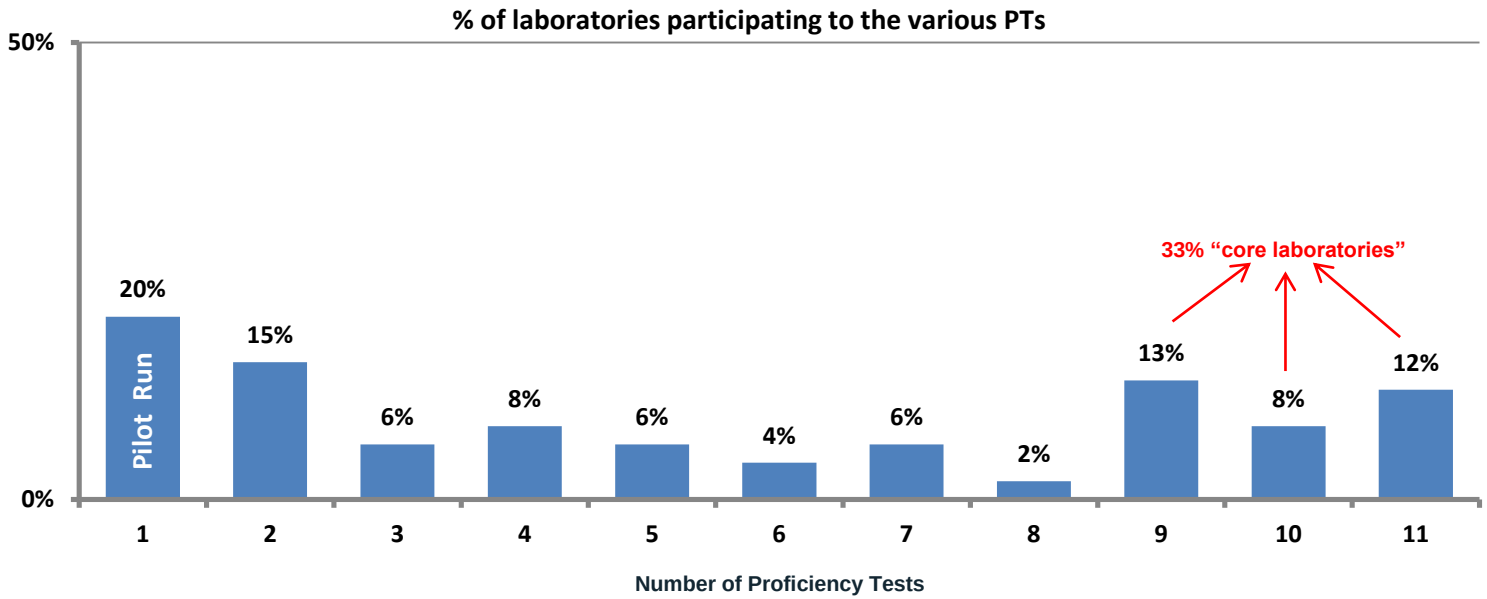
From 2009 the number of laboratories taking part to the PT every year is stable



AA SG Proficiency Test

Participation %

Only a small percentage of the laboratories has taken part to all PTs

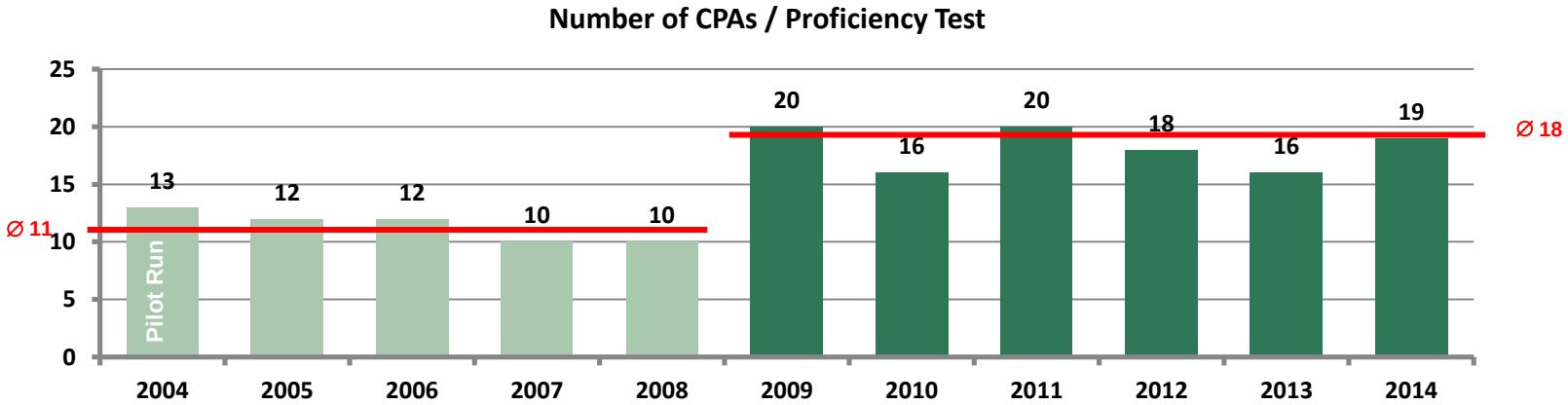


AA SG Proficiency Test

Number of CPAs included in the PTs

From 2009 the number of CPAs per PT has increased considerably

- Same FAPAS price
- Methods collection done by the Sub-Group

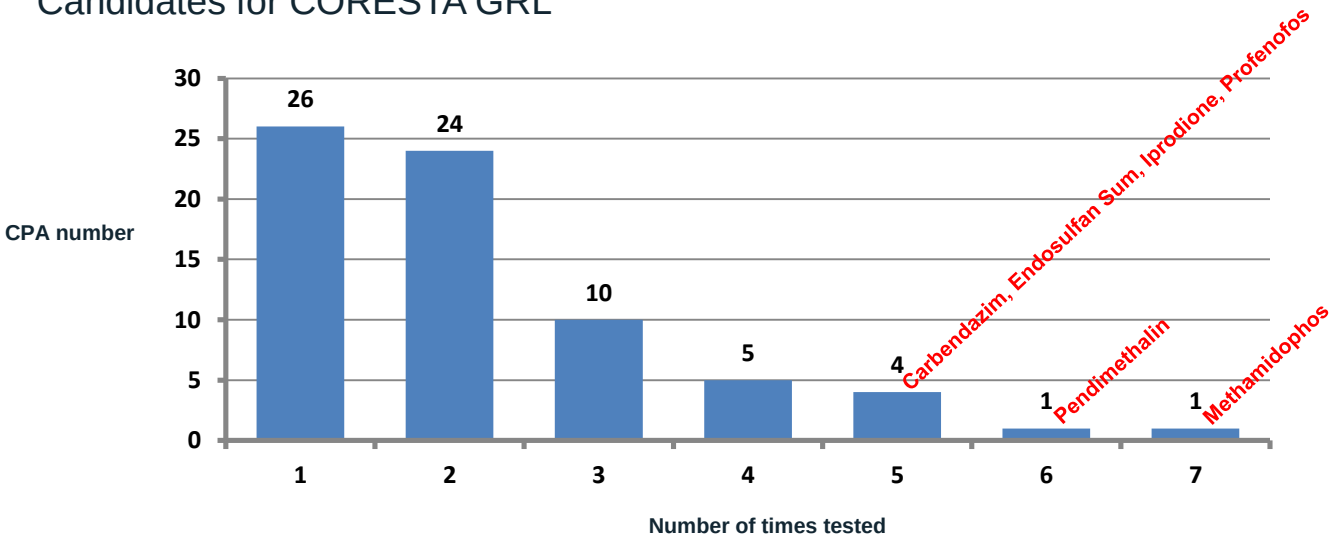


AA SG Proficiency Test

CPAs and number of times tested

2004-2014 → 71 CPAs tested

- Most detected in commercial processed tobacco
- Problematic in Proficiency Test
- Candidates for CORESTA GRL



Continuous participation to CORESTA-FAPAS Proficiency Test “a tool for better analytical quality”

Hans Braeckman

Fytolab cvba

Introduction

- Independant contract lab specialised in CPA residue analysis (95%) and other contaminants: mycotoxins, heavy metals, nitrosamines, ... Approx 45,000 samples a year
- Focus on challenging matrices like tobacco, hops, oils, essential oils, concentrates, ... with a mission towards reliability
- Spinoff University of Ghent, started in 2001, continuing the expertise and knowledge develloped at the University
- Headquartered in Belgium
- Sites in Bulgaria, Colombia and France

www.fytolab.com

Questions to be answered

After some years of work in the Agrochemical Analysis Sub-Group

- Can the quality of laboratories be made objective?
- Is continuous participation to proficiency tests a tool for quality improvement?

Starting point

- Anonymous data provided of all available proficiency tests
- Selection of the “core laboratories”
  - Participated 4 out of 5 proficiency tests in 2007-2011
  - 17 laboratories were selected as regular participants to proficiency test →”core laboratories”
- Using data provided by FAPAS

Pesticides quantified per PT

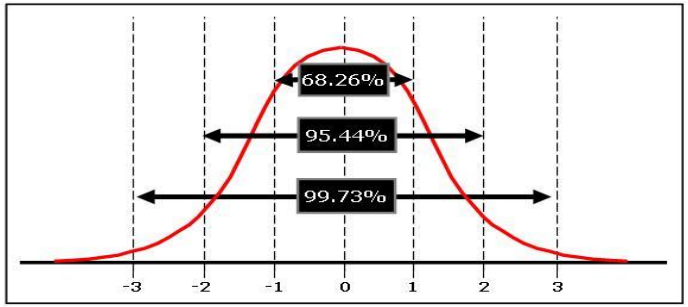
CPAs evaluated per PT

spiked	10	20	16	20	18	14	20
evaluated	5	14	13	16	17	12	18
laboratory	2008	2009	2010	2011	2012	2013	2014
B	5	14	11	15	16	12	18
D	4	14	12	15	17	12	18
E	5	14	12	16	17	12	18
F	4	6	11	12	15	9	12
G	5	9	11	10	11	10	17
H	4	7	11	15	16	12	18
I	4	6	11	9	8	9	13
L	2	3	5	3	3	-	10
M	3	12	13	16	17	12	18
N	5	14	13	16	17	12	18
O	5	14	13	16	17	12	18
Q	5	14	13	16	17	12	18
R	4	14	13	16	17	11	17
T	4	13	12	15	16	11	17
U	2	3	4	5	6	4	-
Z	4	14	13	16	16	12	18
Y	4	-	13	15	16	11	18
average	4.1	10.7	11.1	13.2	14.1	10.8	16.6
% quantified	81.3	76.3	85.6	82.4	83.1	90.0	92.4

Objective evaluation

Data used by the tool

Z-score:
- *Measure of precision of a measurement compared to the true value*
- *|Z| =< 2 : Acceptable*
- *2 < |Z| =< 3 : Questionable*
- *3 < |Z| : Unacceptable*



- Using only data of pesticides that had full evaluation resulting in a scientific relevant Z-score
- Using Z-scores per pesticide per laboratory as calculated by FAPAS
  - For false negative results, the LOQ has been used to calculate the Z-score
  - No penalties for false positive results
- For further calculations: any z-score values of |z| > 5 corrected to a value of '5' in order to limit the effect of important single deviations (see EU PT approach)

The tool: EU reference laboratories evaluation approach

Scientific accepted tool to evaluated lab-performance

- Calculated Average of squared z-score
- $AZ^2 = \frac{\sum Z\text{^}2}{n}$
- Used EU PT's to evaluate total lab performance
- Consequences:
  - Result independent of the number of pesticides analysed
  - The aim is to encourage laboratories to not only improve the accuracy of their results but also to analyse a greater number of pesticides.

The tool: Calculation examples

Assumption:

- A proficiency test with 5 pesticides
- One pesticide can not be evaluated (e.g. not enough data) → no z-score attributed
- Results reported by the two laboratories:

Pesticide	Lab One Z-score	Lab Two Z-score
A	Not analyzed	8 (corrected to value 5)
B	0.5	1
C	2	1.5
D	No z-score attributed	No z-score attributed
E	1	0.5

The tool: Calculation examples

Laboratory One

- Average $Z^2 = (0.5^2 + 2^2 + 1^2)/ 3 = 1.75$

Laboratory Two

- Average $Z^2 = (5^2 + 1^2 + 1.5^2 + 0.5^2)/ 4 = 7.1$

The tool: Average z^2 interpretation

- Usage of average z^2
  - Approach applied by all 4 EU reference laboratories for pesticide residue analysis in proficiency test evaluation
  - Used to evaluate the **overall performance** of a laboratory in a proficiency test
    - $AZ^2 =< 2$: Acceptable
    - $2 < AZ^2 =< 3$: Questionable
    - $3 < AZ^2$: Unacceptable

Average z² : the results on CORESTA PT's

Shows lab performance per lab per participation

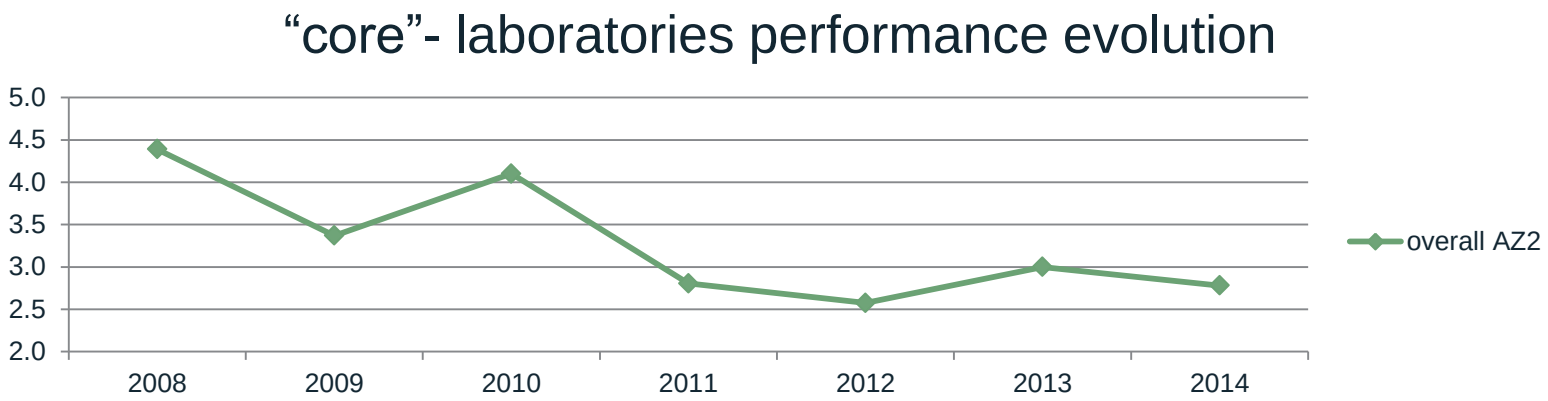
SEGMENT	LAB	2008	2009	2010	2011	2012	2013	2014	average
Contract	B	0.3	0.5	0.8	0.9	2.8	1.2	1.5	1.1
Contract	D	0.9	2.8	1.0	0.4	2.0	1.2	1.6	1.4
Industry	E	1.8	1.2	1.2	1.9	0.6	5.0	3.0	2.1
Authority	F	13.1	9.1	10.4	0.9	4.6	7.3	11.4	8.1
Industry	G	7.4	4.6	4.1	6.4	1.1	9.6	7.5	5.8
Industry	H	3.7	16.3	11.1	5.1	0.7	2.4	0.8	5.7
Industry	I	5.3	2.2	0.8	0.5	0.7	3.0	2.8	2.2
Contract	L	0.6	9.0	16.9	2.7	3.0		15.8	8.0
Authority	M	0.7	7.4	3.4	0.7	1.3	4.7	1.1	2.8
Contract	N	2.6	0.3	0.4	0.5	2.1	0.3	0.9	1.0
Industry	O	8.3	0.2	0.3	0.2	3.3	1.9	0.5	2.1
Contract	Q	10.4	0.5	0.5	0.6	0.3	0.2	1.0	1.9
Contract	R	4.3	1.0	1.0	4.8	0.8	0.3	3.1	2.2
Contract	T	6.6	2.3	8.1	7.4	8.5	8.2	0.9	6.0
University	U	1.0	9.4	19.3	11.4	6.2	3.0		8.4
Industry	Y	1.1	6.3	2.9	2.3	3.4	1.4	2.8	2.9
Contract	Z	0.9		6.8	7.1	4.2	0.5	0.7	3.4
	weighed average	4.4	3.4	4.1	2.8	2.6	3.0	2.8	3.3

Weighed average Z^2: Results of the 'core' labs

Big differences between laboratories

- Some continuous good performing: e.g. avg $AZ^2 <= 2$
- No laboratory shows only "acceptable" results
  - Even the best laboratories can have occasional "questionable" results
  - Tobacco is a difficult matrix to analyze!
- Some show almost always "unacceptable" results: e.g. avg $AZ^2 >= 5$
- Some are struggling and going up and down!

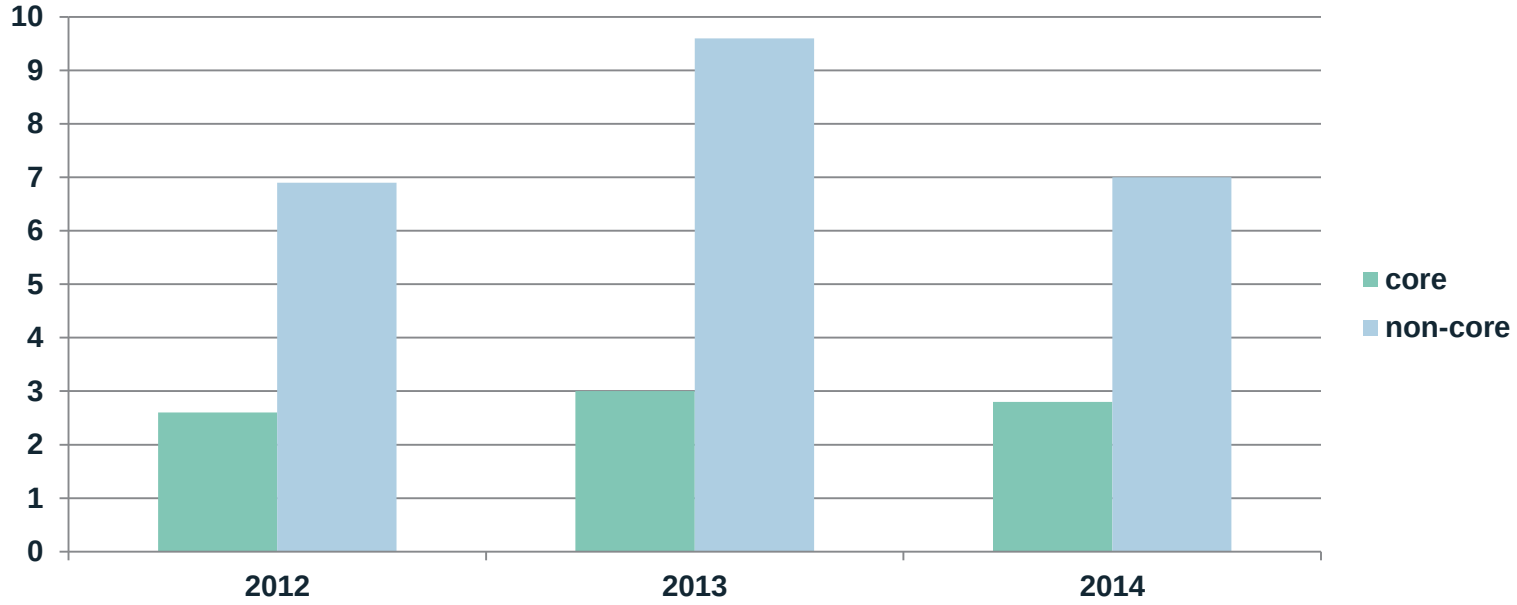
Weighed average Z^2: the trend



- Weighed Average AZ^2 of the 17 "core" laboratories is going down
- The average level went down from around 4.5 to a minimum of 2.6, and is stable between 2.6 and 3

→ laboratory quality was improving significantly in time and seems to have reached a maximum
→ further progress seems to be difficult with the current approach

Difference between “core” and “none-core” laboratories



“Core” laboratories show better performance than “non-core” laboratories

Overall conclusions

- The used tool allows monitoring of lab performance in time and in different groups
- Regular participating (“core”) laboratories show on average better results in proficiency tests → stimulate participation
- Within the group of “core” laboratories big differences in performance are demonstrated, room for improvement → stimulate to work in the lab with the obtained results to improve quality
- The quality of the core laboratories has increased significantly over the time. Further progress seems to be difficult at this moment → new approaches to stimulate further improvements

Thank you

Hans Braeckman – Fytolab
Marco Prat – Japan Tobacco International