

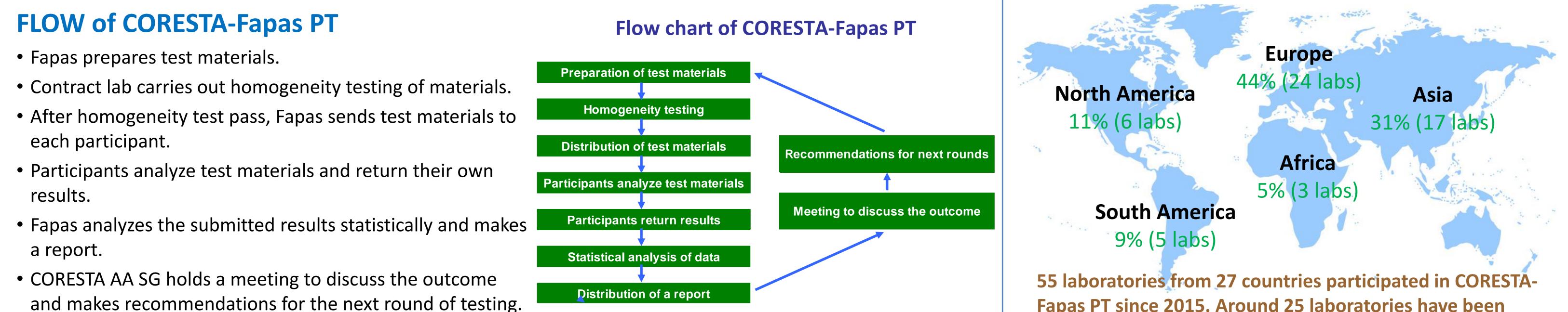
# **Proficiency Testing for Pesticide** fa **Residues Analysis in Tobacco** Proficiency Testing from

### Masahiro MIYOSHI<sup>1</sup>, Marco PRAT<sup>2</sup>, Dominic ANDERSON<sup>3</sup> and Mark SYKES<sup>3</sup>

1 Japan Tobacco Inc., Leaf Tobacco Research Center, 1900, Idei, Oyama, Tochigi 323-0808, Japan; E-mail: masahiro.miyoshi@jt.com 2 Japan Tobacco International, Germany GmbH, Diedenhofener Str 20, 54294, Trier, Germany 3 Fapas, Fera Science Ltd, Sand Hutton, York, YO41 1LZ, United Kingdom

## INTRODUCTION

There is an increasing demand for laboratories to demonstrate their performance and reliability in pesticide residue analysis. Proficiency Testing (PT) schemes provide an independent and unbiased assessment of performance. The Cooperation Centre for Scientific Research Relative to Tobacco (CORESTA) Agrochemical Analysis Sub-Group (AA-SG) has been implementing a PT on pesticide residues analysis in tobacco test materials every year since 2005 in collaboration with Fapas from Fera Science Ltd, UK (Fera), in order to evaluate the quality of an analytical laboratory's results.



Fapas PT since 2015. Around 25 laboratories have been taking part in every round.

#### **TEST MATERIALS**

- Artificially spiked and/or agronomically incurred test materials were provided for participants to determine the identity of and the levels of pesticide residues present in each test material.
- A total 80 pesticides which were chosen from those listed in CORESTA Guide No.1 and its candidates have been tested in 13 rounds of this PT since 2005 (Table 1).

### **EVALUATION**

- *z*-scores are calculated as:

#### Table 1. Tested analytes with rate of satisfactory z -scores in CORESTA-Fapas PTs since 2005 FT0112 FT0103 FT0104 PT No. FT0101 FT0102 FT0105 FT0106 FT0107 FT0108 FT0109 FT0110 FT0111 2016 2007 2008 2015 **Tested analytes** 2006 2014 2009 2010 2011 2012 2013 Year 2005 Sample type spiked spiked incurred spiked incurred spiked spiked spiked spiked spiked spiked spiked spiked incurred spiked incurred 2,4-D 80% 65% N/A 65% acetamiprid 80% 88% 67% acibenzolar-S-methyl 78% aldicarb (Σ) 60% 90% 91% azoxystrobin 100% 91% 64% 89% benalaxyl bifenthrin 77% 80% 89% bitertanol 89% butralin 76% 74% 76% 83% 79% 82% carbaryl 68% 63% 55% 62% carbendazim (Σ) 81% 67% 94% 73% carbofuran (Σ) 76% 85% 93% 82% chlorantraliniprole 70% 74% chlorothaloni 45% 45% N/A N/A chlorpyrifos 88% 75% 82% 89% chlorpyrifos-methyl 50% 65%

| N. of tested analytes                  |      |                |      |      |     |            |                           |                    |            |       |              |            |       |                    | 13     |
|--|------|----------------|------|------|-----|------------|---------------------------|--------------------|------------|-------|--------------|------------|-------|--------------------|--------|
| trifluralin                            |      |                | 86%  |      |     |            | 89%                       |                    |            |       |              |            |       |                    |        |
| triazophos<br>triflumuron              |      |                |      |      |     |            |                           | 94%<br>87%         |            |       | 94%          |            |       |                    |        |
| riadimefon                             |      |                |      |      |     |            |                           | 0404               | 94%        |       |              | 91%        | 83%   | 91%                |        |
| hiophanate-methyl                      |      |                |      |      |     |            |                           |                    | 72%        |       |              |            |       |                    | / v    |
| hiamethoxam                            |      |                |      |      |     | 79%        |                           |                    | 80%        |       |              | / 2 /0     |       | TOO \0             | 60%    |
| eflubenzuron<br>hiacloprid             |      |                |      |      |     |            |                           |                    |            |       |              | 81%<br>72% |       | 79%<br><b>100%</b> | 68%    |
| ebuconazole                            |      |                |      |      |     |            |                           |                    |            |       |              | 010/       |       | 89%                | 89%    |
| pirotetramat (Σ)                       |      |                |      |      |     |            |                           |                    |            |       |              |            |       | 50%                |        |
| juinalphos                             |      |                |      |      |     | 0270       |                           |                    |            |       |              |            |       | 88%                |        |
| bymetrozine                            |      |                |      |      |     | 62%        |                           |                    | 7 5 /0     | 7070  | 0070         |            | 01/0  | 10/0               | 0/ د د |
| profenofos<br>propamocarb              |      | 76%            |      |      |     |            | 72%                       |                    | 68%<br>79% | 76%   | 74%<br>68%   |            | 81%   | 70%                | 55%    |
| pirimiphos-methyl                      |      | 700/           |      |      |     |            | 770/                      |                    |            |       | 740/         |            |       | 95%                |        |
| piperonyl butoxide                     |      |                |      |      |     | 65%        |                           |                    |            |       |              | 80%        |       | 0 = 0 (            |        |
| permethrin (Σ)                         |      |                | 79%  |      |     |            | 70%                       |                    |            |       |              |            |       |                    |        |
| penconazole                            |      |                |      |      |     | 77%        |                           |                    |            |       |              | / 0        |       |                    |        |
| parathion                              | 0770 |                |      |      |     |            |                           |                    |            |       |              | 95%        |       |                    |        |
| oxamyl                                 | 67%  | 7070           |      |      |     |            | 0.070                     | 01/0               | 0070       |       | 02/0         | 90%        | 0070  | ,0/0               | ,070   |
| pendimethalin                          | 88%  | 76%            |      |      |     | 0070       | 65%                       | 81%                | 80%        | 70%   | 82%          | 05%        | 80%   | 78%                | 78%    |
| methamidophos<br>methomyl (Σ)          | N/A  | 44%            | 48%  |      |     | 53%<br>88% | N/A                       | 67%                |            | 70%   |              | 62%<br>83% |       | 78%                |        |
| metalaxyl (Σ)<br>methamidonhos         |      | 63%<br>11%     | 100/ |      |     | E 20/      | NI / A                    | 89%                |            | 90%   |              | 620/       | 92%   | 700/               |        |
| maleic hydrazide                       |      | C20/           | 50%  | %    |     |            |                           | 000/               |            | 0.007 |              |            | 0.207 |                    |        |
| prodione (Σ)                           |      |                |      | 55%  |     | 83%        |                           |                    | 78%        | 78%   | 84%          | 77%        | 78%   | 40%                |        |
| probenfos                              | /0   |                |      |      |     |            |                           | 92%                |            |       | / V          |            |       |                    |        |
| heptachlor (Σ)                         | 82%  |                |      |      |     |            | 02/0                      | , 570              |            |       | 52%          | 86%        |       |                    |        |
| indoxacarb (Σ)                         | 5070 | 04/0           |      |      |     |            | 82%                       | 75%                |            |       |              |            |       |                    | / U /0 |
| folpet<br>imidacloprid                 | 58%  | 64%            |      |      |     |            |                           |                    |            |       | N/A          |            |       |                    | 76%    |
| flumetralin<br>Folget                  |      | 61%            |      |      |     |            | 70%                       | 81%                |            | 86%   | NI / A       |            | 79%   | 79%                | 67%    |
| flubendiamide                          |      | <b>C</b> 4 0 ( |      |      |     |            | 700/                      | 040/               |            | 82%   | 87%          |            | 700/  | 89%                | 78%    |
| luazifop-butyl (Σ)                     |      |                |      |      |     | 100%       |                           |                    |            |       |              |            |       |                    |        |
| fenvalerate (Σ)                        | 69%  |                |      |      |     |            |                           |                    |            | 64%   |              | 68%        |       | 77%                |        |
| fenamiphos sulfoxide                   |      |                |      | 88%  |     |            |                           |                    | 70%        |       |              |            |       |                    |        |
| fenamiphos sulfone                     |      |                |      | 78%  |     |            |                           |                    |            |       |              |            |       |                    |        |
| fenamiphos (2)                         |      |                |      | 82%  |     |            | 0/ ف ف                    |                    |            | 01/0  |              |            |       |                    |        |
| fenamiphos (Σ)                         |      |                |      |      |     |            | 95%                       | 100%               |            | 81%   | 0770         |            |       | 0370               | 0470   |
| famoxadone<br>fenamidone               |      |                |      |      |     | N/A        |                           | 56%<br><b>100%</b> |            |       | 89%          |            |       | 89%                | 84%    |
| ethion<br>famovadone                   |      |                |      |      |     |            |                           | F <i>C</i> 0/      |            |       |              |            |       | 95%                |        |
| endosulfan sulfate                     |      | 60%            |      |      |     |            |                           |                    |            |       | 67%          |            |       |                    |        |
| beta-endosulfan                        |      | 75%            |      |      |     |            |                           |                    |            |       | <b>63</b> 0/ |            |       |                    |        |
| alpha-endosulfan                       |      | 68%            |      |      |     |            |                           |                    |            |       |              | 78%        |       |                    |        |
| endosulfan (Σ)                         | 59%  | 58%            |      |      |     | 71%        |                           |                    |            |       |              |            |       |                    |        |
| dithiocarbamates (as CS <sub>2</sub> ) |      |                |      |      | 65% |            |                           |                    |            |       |              |            | 54%   |                    |        |
| dimethomorph (Σ)                       |      |                |      | 58%  |     |            |                           | 89%                |            |       | 91%          |            |       |                    |        |
| dimethoate (Σ)                         |      |                |      |      |     |            |                           |                    | 61%        | N/A   |              | 57%        |       | 71%                |        |
| diflubenzuron                          |      |                |      |      |     |            | $\mathbf{J}\mathbf{L}$ /U | 78%                | //         |       |              |            | , 070 | 5570               |        |
| difenoconazole                         |      |                |      |      |     |            | 82%                       | 0470               | 100%       |       |              |            | 73%   | 90%                | 81%    |
| dicloran                               |      |                |      |      |     |            |                           | 84%                | 67%        |       |              |            |       | 79%                |        |
| deltamethrin (Σ)<br>dicamba            | 47%  | 59%            |      |      |     |            | 58%                       | 67%                | 670/       |       |              | 76%        |       |                    |        |
| pp'-DDT<br>doltomothrin (S)            | 470/ |                |      | 70%  |     |            |                           | <b>C7</b> 0/       |            |       | 71%          | 700/       |       |                    |        |
| op'-DDT                                |      |                |      | 68%  |     |            |                           |                    |            |       | 77%          |            |       |                    |        |
| DDT (Σ)                                |      | , •            |      |      |     |            | , •                       |                    |            | 52%   |              |            |       |                    |        |
| cypermethrin ( $\Sigma$ )              |      | 71%            |      |      |     | 67%        | 71%                       |                    |            |       |              |            | 77%   |                    |        |
| cymoxanil                              |      |                |      |      |     | 75%<br>75% |                           |                    | 0170       |       |              |            | 01%   |                    |        |
| cyfluthrin (Σ)<br>cyhalothrin (Σ)      |      |                |      |      |     | 75%        | 80%                       |                    | 81%        | 86%   | 88%          |            | 81%   |                    |        |
| clomazone                              |      |                |      |      |     | 94%        | 000/                      |                    | 95%        | 0.004 | 000/         |            |       |                    |        |
| clothianidin                           |      |                |      |      |     |            |                           |                    | 81%        |       |              |            |       |                    | 61%    |
| chlorthal-dimethyl                     |      |                |      | 00/0 |     | 73%        |                           |                    | 89%        |       | 0070         |            |       | 89%                |        |
| chiorpyritos-methyl                    |      |                |      | 50%  |     |            |                           |                    |            |       | 65%          |            |       |                    |        |

| $z=\frac{(x-x_a)}{x-x_a}$   |                             |  |  |  |  |  |  |
|---|-----------------------------|--|--|--|--|--|--|
| $\sigma_p$<br>where x: the participant's rep  | orted result                |  |  |  |  |  |  |
| $X_a$ : the assigned value<br>and $\sigma_p$ : the standard devia   | tion for PT.                |  |  |  |  |  |  |
| <ul> <li>Obtained z-scores can be inte</li> <li> z ≤2 : satisface</li> </ul>  | •                           |  |  |  |  |  |  |
| 2<  <i>z</i>  ≤3 : questio  | onable                      |  |  |  |  |  |  |
| z >3 : unsatis  | sfactory                    |  |  |  |  |  |  |
| RESULTS   | analysis in tobacco so that |  |  |  |  |  |  |
| <ul> <li>The rate of satisfactory z-<br/>scores for all participants has<br/>been increasing from a<br/>minimum of 63% to a<br/>maximum of 83% and is stable<br/>at around 80% in the last 5</li> </ul> | 70% -                       |  |  |  |  |  |  |
| <ul> <li>rounds.</li> <li>To resolve questions arising from each round of PT,</li> </ul>  | 60% -<br>50% -<br>40% -     |  |  |  |  |  |  |

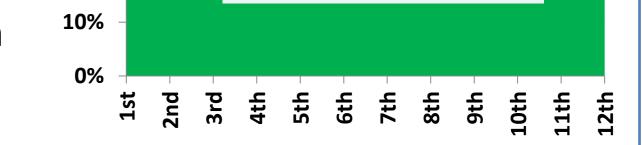
Unsatisfactory z-scores Questionable z-scores Satisfactory z-scores

Italics indicate for information only.

expands knowledge based on these pesticide residues

CORESTA AA-SG hold an

annual discussion which



### CONCLUSIONS

• PT for pesticide residues analysis in tobacco has contributed to improve overall laboratory performance over 12 years.

30%

20%

• Future PT could be more effective with an increase in the number of participating laboratories (e.g. from African or Latin American regions).

#### ACKNOWLEDGMENT

CORESTA Agrochemical AA-SG and all laboratories participating in the CORESTA-Fapas PTs.

**Reference:** Cooperation Centre for Scientific Research Relative to Tobacco (CORESTA), Guide No. 1: The Concept and Implementation of Agrochemical Guidance Residue Levels, July 2016, https://www.coresta.org/sites/default/files/technical\_documents/main/Guide-No01-GRLs4th-Issue-July16.pdf AA-138-CXP\_LAPRW2017-Poster\_170514