Agrochemicals Analysis (AA) Sub-Group Report

2019 CORESTA AP Conference

Victoria Falls, Zimbabwe

17th October 2019

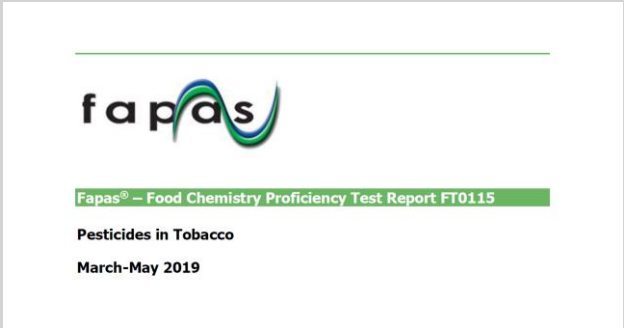
AA SG – Objectives

- To perform regular proficiency testing of Multi-Residue Methods for the analysis of agrochemical residues on tobacco.
- To undertake joint experiments to resolve unanswered questions arising from proficiency tests; to expand knowledge base on agrochemical residues and their analysis.
- To 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 – Activities

Proficiency testing 2019 (FAPAS FT0115)

- **107 CPAs listed in CORESTA Guide No.1 and its 23 GRL candidates**
- **Direction on reporting the sum of CPAs**
  - Residue definition and Conversion factor
- **Two test materials** (artificially spiked and agronomically incurred)
  - 17 CPAs spiked on blank Burley tobacco
  - 12 CPAs in incurred Oriental tobaccos (provided by RFT SG)
- **28 laboratories from 18 countries**
- **z-score evaluation**
- **FAPAS Report (June 2019)**
- **Discussion at SG meeting in July 2019**


fapas

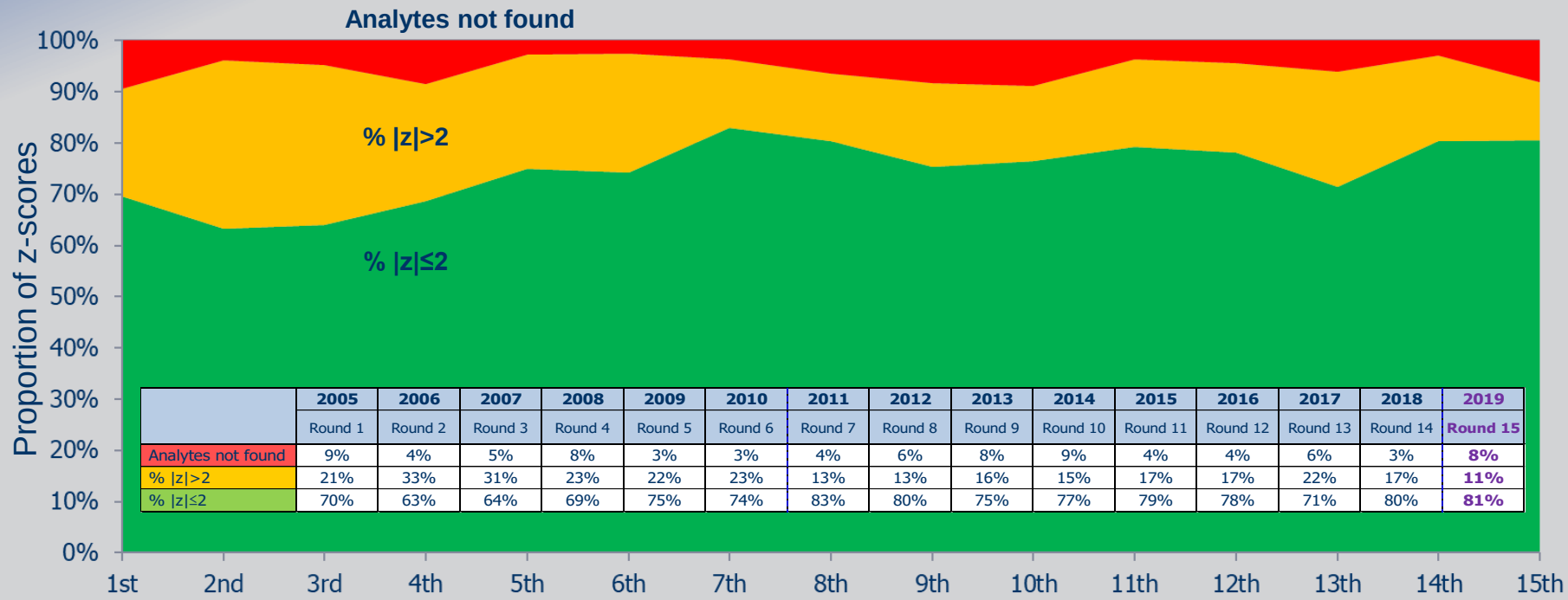
Fapas® – Food Chemistry Proficiency Test Report FT0115

Pesticides in Tobacco

March-May 2019

AA SG – Activities

z-score trend (FAPAS FT0101-FT0115)



	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019
	Round 1	Round 2	Round 3	Round 4	Round 5	Round 6	Round 7	Round 8	Round 9	Round 10	Round 11	Round 12	Round 13	Round 14	Round 15
Analytes not found	9%	4%	5%	8%	3%	3%	4%	6%	8%	9%	4%	4%	6%	3%	**8%**
% |z|>2	21%	33%	31%	23%	22%	23%	13%	13%	16%	15%	17%	17%	22%	17%	**11%**
% |z|≤2	70%	63%	64%	69%	75%	74%	83%	80%	75%	77%	79%	78%	71%	80%	**81%**

AA SG – Activities

Follow-up of Joint Experiment Technical Study (JETS) on Maleic Hydrazide

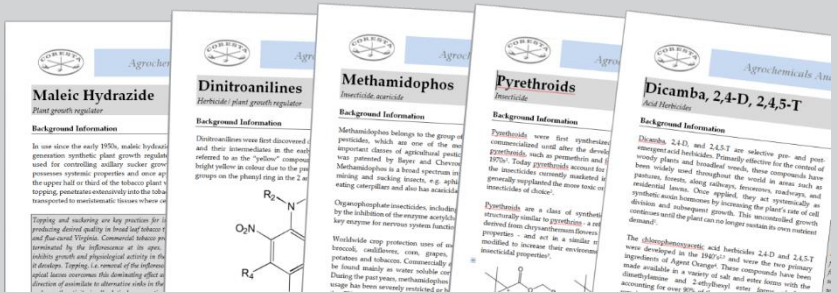
- During the systematic review of the ISO 4876:1980 method (Tobacco and tobacco products - Determination of maleic hydrazide residues) in 2011, two revision proposals were submitted to the ISO/TC 126/SC 2.
- AA SG conducted two rounds of JETS in 2016 and 2017 to evaluate the capacity of two proposed methods and concluded that the tested methods did not compare to the ISO method statistically and needed further development.
- The ISO/TC 126 adopted the Resolution No 94 in 2018 that SC 2 decides to wait for the work of CORESTA AA SG prior to initiate a new work item proposal.
- SG made a statement of the findings from two JETSs in 2018 for submitting to the ISO/TC 126/SC 2, instead of a third study for developing a new method.
- CORESTA submitted the statement in 2018 to the ISO/TC 126, in response to the Resolution No 94.
- The ISO/TC 126 mentioned the Resolution No 94 and decided not to initiate a new work item proposal in 2019, in accordance with the feedback from CORESTA.

AA SG – Activities

Revision of CORESTA Guide No.5 -Technical Notes (TNs)

- **Guide No.5 and TN #001: Maleic Hydrazide were revised in October 2018.**
- **Following the TN #001, AA SG decided to revise TNs #002-#005:**

  - TN #002 Dinitroanilines
  - TN #003 Methamidophos
  - TN #004 Pyrethroids
  - TN #005 Dicamba, 2,4-D, 2,4,5-T



- **Revised versions drafted and finalized by SG members**
- **To be published on the CORESTA website after reviewed by the Scientific Commission and the Board**

AA SG – Activities

2019 AA SG meeting

- Dubai, UAE on July 3-4, 2019
- Hosted by Premium Tobacco
- 23 participants from 14 countries
- 18 presentations by 10 speakers

AA SG – On going Activities

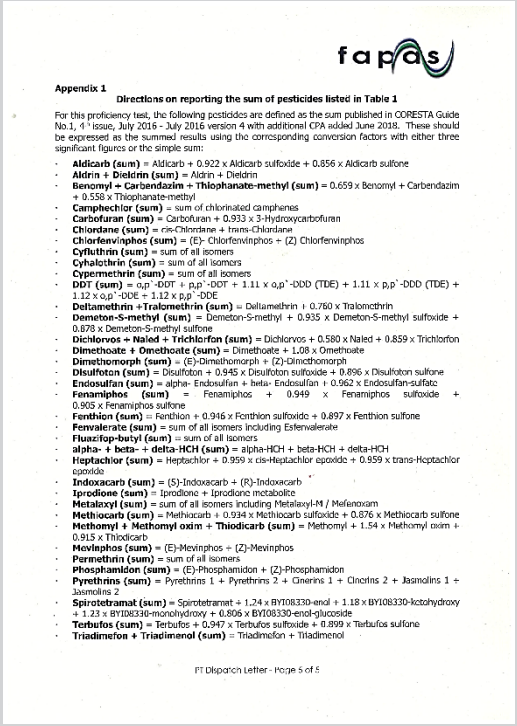
Joint Experiment Test Study (JETS 19/1) on Matrix Effects from DAC tobacco

- **Coordinator:** Masahiro Miyoshi (JT)
- **Background:**
  - Matrix effects are a major concern in CPA analysis on tobacco.
  - AA SG has conducted numerous proficiency testing and JETS using BLY (Burley) or FCV (Flue-cured Virginia), however DAC (dark air-cured) tobacco was never used.
- **Objective:**
  - To know if there are any differences in matrix effects among DAC, BLY and FCV
- **Study design:**
  - Comparing responses from solvent standard and three types of matrix-matched standards
- **11 laboratories registered**
- **Test materials (DAC, BLY, FCV) to be dispatched (November 2019)**
- **Outcome to be discussed at next SG meeting**

AA SG – Next Activities

Proficiency testing 2020 (FAPAS FT0116)

- **Meeting with FAPAS on 18 Sep 2019**
- **Study design confirmed**
  - Spiked and incurred tobacco samples
  - Trackable sample shipment
  - Reminding participants of reporting rule for the sum of CPAs
- **Timeline**
  - Registration: Jan 2020
  - Sample dispatching: Feb 2020
  - Submission of results: Apr 2020
  - FAPAS report: May 2020
  - Discussion: Jun 2020



Appendix 1

Directions on reporting the sum of pesticides listed in Table 1

For this proficiency test, the following pesticides are defined as the sum published in CORESTA Guide No.1, 4 th Issue, July 2016 - July 2016 version 4 with additional CPA added June 2018. These should be expressed as the summed results using the corresponding conversion factors with either three significant figures or the simple sum:

- **Aldicarb (sum)** = Aldicarb + 0.922 x Aldicarb sulfoxide + 0.856 x Aldicarb sulfone
- **Aldrin + Dieldrin (sum)** = Aldrin + Dieldrin
- **Benomyl ↔ Carbendazim + Thiophanate-methyl (sum)** = 0.659 x Benomyl + Carbendazim + 0.558 x Thiophanate-methyl
- **Camphechlor (sum)** = sum of chlorinated camphenes
- **Carbofuran (sum)** = Carbofuran + 0.933 x 3-Hydroxycarbofuran
- **Chlordane (sum)** = cis-Chlordane + trans-Chlordane
- **Chlorfenvinphos (sum)** = (E)- Chlorfenvinphos + (Z) Chlorfenvinphos
- **Cyfluthrin (sum)** = sum of all isomers
- **Cyhalothrin (sum)** = sum of all isomers
- **Cypermethrin (sum)** = sum of all isomers
- **DDT (sum)** = o,p`-DDT + p,p`-DDT + 1.11 x o,p`-DDD (TDE) + 1.11 x p,p`-DDD (TDE) + 1.12 x o,p`-DDE + 1.12 x p,p`-DDE
- **Deltamethrin +Tralomethrin (sum)** = Deltamethrin + 0.760 x Tralomethrin
- **Demeton-S-methyl (sum)** = Demeton-S-methyl + 0.935 x Demeton-S-methyl sulfoxide + 0.878 x Demeton-S-methyl sulfone
- **Dichlorvos + Naled + Trichlorfon (sum)** = Dichlorvos + 0.580 x Naled + 0.859 x Trichlorfon
- **Dimethoate + Omethoate (sum)** = Dimethoate + 1.08 x Omethoate
- **Dimethomorph (sum)** = (E)-Dimethomorph + (Z)-Dimethomorph
- **Disulfoton (sum)** = Disulfoton + 0.945 x Disulfoton sulfoxide + 0.896 x Disulfoton sulfone
- **Endosulfan (sum)** = alpha- Endosulfan + beta- Endosulfan + 0.962 x Endosulfan-sulfate
- **Fenamiphos (sum)** = Fenamiphos + 0.949 x Fenamiphos sulfoxide + 0.905 x Fenamiphos sulfone
- **Fenthion (sum)** = Fenthion + 0.946 x Fenthion sulfoxide + 0.897 x Fenthion sulfone
- **Fenvalerate (sum)** = sum of all isomers including Esfenvalerate
- **Fluazifop-butyl (sum)** = sum of all isomers
- **alpha- + beta- + delta-HCH (sum)** = alpha-HCH + beta-HCH + delta-HCH
- **Heptachlor (sum)** = Heptachlor + 0.959 x cis-Heptachlor epoxide + 0.959 x trans-Heptachlor epoxide
- **Indoxacarb (sum)** = (S)-Indoxacarb + (R)-Indoxacarb
- **Iprodione (sum)** = Iprodione + Iprodione metabolite
- **Metalaxyl (sum)** = sum of all isomers including Metalaxyl-M / Mefenoxam
- **Methiocarb (sum)** = Methiocarb + 0.934 x Methiocarb sulfoxide + 0.876 x Methiocarb sulfone
- **Methomyl + Methomyl oxim + Thiodicarb (sum)** = Methomyl + 1.54 x Methomyl oxim + 0.915 x Thiodicarb
- **Mevinphos (sum)** = (E)-Mevinphos + (Z)-Mevinphos
- **Permethrin (sum)** = sum of all isomers
- **Phosphamidon (sum)** = (E)-Phosphamidon + (Z)-Phosphamidon
- **Pyrethrins (sum)** = Pyrethrins 1 + Pyrethrins 2 + Cinerins 1 + Cinerins 2 + Jasmolins 1 + Jasmolins 2
- **Spirotetramat (sum)** = Spirotetramat + 1.24 x BYI08330-enol + 1.18 x BYI08330-ketohydroxy + 1.23 x BYI08330-monohydroxy + 0.806 x BYI08330-enol-glucoside
- **Terbufos (sum)** = Terbufos + 0.947 x Terbufos sulfoxide + 0.899 x Terbufos sulfone
- **Triadimefon + Triadimenol (sum)** = Triadimefon + Triadimenol

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Acknowledgments

Proficiency testing 2019 (FAPAS FT0115)

- Dominic Anderson (Fera)
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Revision of Technical Notes

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Follow-up of Joint Experiment Technical Study (JETS) on Maleic Hydrazide

- Keisuke Nakayama (JT)
- Pierre-Marie Guitton (CORESTA)

Thank you for your attention!