AOAC VDR Sub-Group Community Meeting

Tuesday, August 28, 2018

11:45 am – 1:15 pm

Room : Willow East

Sheraton Centre Toronto Hotel

Co-Chairs: Eric Verdon & Jian Wang

132nd Annual Meeting & Exposition
August 26-29, 2018
Toronto, ON CANADA

AOAC Educate Network Collaboration
Annual Meeting & Exposition
132nd Annual Meeting • August 26–29, 2018 • Toronto, Canada

ATTENDING LIST – Sign in

VDR Sub-Group Community Meeting
Attending List

AOAC Educate Network Collaboration
Annual Meeting & Exposition
132nd Annual Meeting • August 26–29, 2018 • Toronto, Canada

Co-Chairs: Eric Verdon & Jian Wang

E-mails: eric.verdon@anses.fr
jian.wang@inspection.gc.ca

Website: http://www.nacrw.org/Community/drugs_11-2207.l

Your name	Your Institute/ Company/ Laboratory	Your email	Your interest in this meeting or Comments

ATTENDING LIST – Sign in

Wang	Jian	CFIA
Verdon	Eric	ANSES
Yang	Charles	Thermofisher
Shia	Jeremy	Waters
Joseph	George	Asurequality
Denney	Dan	Ataraxis
Brunkhorst	Julie	Trilogy Analytical Lab
McGhee	Heather	Trilogy Analytical Lab
Kariuki	Solomon	Univ of Kentucky - Div Reg Services
Stebbins	Nathan	Tyson Foods
Yeung	Jupiter	Nestlé
Shringarpune	Jayant	Tyson Foods
Shelly	Don	LGC Standards
Siegel	Victoria	Eurofins CAL
Torres	Marina	LATU
Delatour	Thierry	Nestlé
Mastovska	Katerina	Eurofins FII
George	Ed	Thermofisher
McRae	Garnet	NRC
Kong	Jason	Ohio Dept of Agriculture
Krepich	Scott	Phenomenex
Sibanda	Liberty	Randox Laboratories
Yang	Dan-Hui Dorothy	Agilent
Lu	Meiling	Agilent
Wu	Jingcun	Perkin Elmer
Fujiera	Roberto	Cargill Sindiraçoês
Kaufmann	Anton	KLZH
Stevens	Jack	General Mills
Borts	David	Iowa State University
Wages	Travis	Tyson Foods

Agenda

- **1. Introduction** Sub-Group Co-Chairs: **Eric Verdon and Jian Wang**

  Sign the "sign-in sheet"

  to get all future subgroup updates and let us know you attended

  http://www.nacrw.org/Community/vet_drugs.html

- **2. Topic n°1:**

  Latest method performance criteria updates

  – ***International - Pesticides*** (Jian Wang):

  ✓ CCPR CAC/GL 90-2017 Guidelines on Performance Criteria for Methods of Analysis for the Determination of Pesticide Residues in Food and Feed

  ✓ SANTE/11813/2017 Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed

  – ***European*** (Eric Verdon):

  ✓ Update for Revision of European Decision 2002/657/EC on Performance Criteria for Methods of Analysis for the Determination of Vet Drug Residues in Food

Agenda

- **3. Topic n°2:**

  Update on multi-class multi-residue method on vet drugs in food and AOAC process to submit the proposal of method – by *Thierry Delatour*

- **4. Topic n°3:**

  AOB …

  ➢ The AOAC Official Method – is it time for a new approach?

- **5. Adjournment** of Contaminant Subgroups Meeting for Veterinary Drug Residues

Topic n°1

Latest method performance criteria updates

- ***International - Pesticides*** (Jian Wang):
  - ✓ CCPR CAC/GL 90-2017 Guidelines on Performance Criteria for Methods of Analysis for the Determination of Pesticide Residues in Food and Feed
  - ✓ SANTE/11813/2017 Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed.
- ***European*** (Eric Verdon):
  - ✓ Update for Revision of European Decision 2002/657/EC on Performance Criteria for Methods of Analysis for the Determination of Vet Drug Residues in Food

49th CCPR

CAC/GL 90-2017 Guidelines on Performance Criteria for Methods of Analysis for the Determination of Pesticide Residues in food and feed

Adopted at

Joint FAO/WHO Food Standards Program
40th Session of the Codex Alimentarius Commission
Geneva, Switzerland
17-22 July 2017

http://www.fao.org/fao-who-codexalimentarius/meetings-reports/en/

CCPR49 Codex Committee on Pesticide Residues	Beijing China	24/04/2017	29/04/2017	Agenda: Report:	✓ ✓ ✓ ⊘ ⊘ ⊘ ✓ ✓ ✓ ⊘ ⊘ ⊘

CAC/GL 90-2017

Table 1. Identification criteria for different MS techniques

MS detector / characteristics	Typical systems (examples)	Acquisition	Requirements for identification	
			minimum number of ions	other
Unit mass resolution	quadrupole, ion trap, TOF	full scan, limited m/z range, SIM	3 ions	S/N ≥ 3[e] Analyte peaks in the extracted ion chromatograms must fully overlap. Ion ratio within **±30% (relative)** of average of calibration standards from same sequence[f]
MS/MS	triple quadrupole, ion trap, Q-trap, Q-TOF, Q-Orbitrap	selected or multiple reaction monitoring, mass resolution for precursor-ion isolation equal to or better than unit mass resolution	2 product ions	
Accurate mass measurement	High resolution MS: TOF or Q-TOF Orbitrap or Q-Orbitrap FT-ICR-MS sector MS	full scan, limited m/z range, SIM, fragmentation with or without precursor-ion selection, or combinations thereof	2 ions with mass accuracy ≤ 5 ppm[a,b,c]	
		combined single stage MS and MS/MS with mass resolution for precursor-ion isolation equal to or better than unit mass resolution	2 ions: 1 molecular ion, (de)protonated molecule or adduct ion with mass acc. ≤ 5 ppm[a,c] *plus* 1 MS/MS product ion[d]	

a) preferably including the molecular ion, (de)protonated molecule or adduct ion

b) including at least one fragment ion

c) < 1 mDa for m/z < 200

d) ≤5 ppm

e) in case noise is absent, a signal should be present in at least 5 subsequent scans

f) if the mass accuracy of a precursor and its product ion is ≤ 5 ppm, ion ratio tolerance is optional.

SANTE/11813/2017

EUROPEAN COMMISSION
DIRECTORATE GENERAL FOR HEALTH AND FOOD SAFETY

Safety of the Food Chain
Pesticides and Biocides

SANTE/11813/2017
21 – 22 November 2017 rev.0

Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed.

SANTE/11813/2017

Supercedes

SANTE/11945/2015

SANTE/11813/2017

Table 4. Identification requirements for different MS techniques[2]

MS detector/Characteristics		Acquisition	Requirements for identification	
Resolution	Typical systems (examples)		minimum number of ions	other
Unit mass resolution	Single MS quadrupole, ion trap, TOF	full scan, limited m/z range, SIM	3 ions	S/N ≥ 3[d] Analyte peaks from both product ions in the extracted ion chromatograms must fully overlap. Ion ratio from sample extracts should be within **±30% (relative)** of average of calibration standards from same sequence
	MS/MS triple quadrupole, ion trap, Q-trap, Q-TOF, Q-Orbitrap	selected or multiple reaction monitoring (SRM, MRM), mass resolution for precursor-ion isolation equal to or better than unit mass resolution	2 product ions	
Accurate mass measurement	High resolution MS: (Q-)TOF (Q-)Orbitrap FT-ICR-MS sector MS	full scan, limited m/z range, SIM, fragmentation with or without precursor-ion selection, or combinations thereof	2 ions with mass accuracy ≤ 5 ppm[a, b, c]	S/N ≥ 3[d] Analyte peaks from precursor and/or product ion(s) in the extracted ion chromatograms must fully overlap. Ion ratio: see D12

[a] preferably including the molecular ion, (de)protonated molecule or adduct ion
[b] including at least one fragment ion
[c] < 1 mDa for m/z < 200
[d] in case noise is absent, a signal should be present in at least 5 subsequent scans

SANTE/11813/2017

D12 For accurate mass measurement / high resolution mass spectrometry, the variability of ion ratios is not only affected by S/N of the peaks in the extracted ion chromatograms, but may also be affected by the way fragment ions are generated, and by matrix. For example, the range of precursor ions selected in a fragmentation scan event ('all ions', precursor ion range of 100 Da, 10 Da, or 1 Da) results in different populations of matrix ions in the collision cell which can affect fragmentation compared to solvent standards. Furthermore, the ratio of two ions generated in the same fragmentation scan event tends to yield more consistent ion ratios than the ratio of a precursor from a full scan event and a fragment ion from a fragmentation scan event. For this reason, no generic guidance value for ion ratio can be given. Due to the added value of accurate mass measurement, matching ion ratios are less critical, however, they should be used as indicative. Deviations exceeding 30% should be further investigated and judged with care.

Process of Revision of European Commission Decision 2002/657/EC

COMMISSION

COMMISSION DECISION

of 12 August 2002

implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results

(notified under document number C(2002) 3044)

(Text with EEA relevance)

(2002/657/EC)

Under REVISION 2016-2019

http://eur-lex.europa.eu/legal-content/EN/ALL/?uri=CELEX%3A32002D0657

Process of Revision of European Commission Decision 2002/657/EC

- **Agenda and Objectives of this Revision :**

  ❖ To update and reorganize the currently 3 technical documents :

  => **Decision 657/2002/EC** (main piece)

  \+ => Doc SANCO/2726/2004

  \+ => Guidance CRLs 2010 for Screening Methods

Process of Revision of European Commission Decision 2002/657/EC

- **Agenda and Objectives of this Revision :**

  - **Confirmatory issues** : identification & quantitation
    - Criteria linked to the Exact Mass Measurement in HRMS
    - Ion Ratio Tolerances in LR-MS/MS and HR-MS/MS
    - LC Retention Times Tolerances
    - CCalpha for confirmation / LOQ and MRL

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  - **Screening issues** : for biological and chemical methods
    - Criteria for Validation on Screening in Multi-Class/Multi-Residue (100^+ analytes) by LC-MS/MS or LC-HRMS
    - HRMS data acquisition modes for screening with low FN rate
    - CCbeta for screening versus LOD

Process of Revision of European Commission Decision 2002/657/EC

- **Agenda for the updating :**

2016-2017

=> Workshops for Technical discussions within the network of National Reference Laboratories of the 28 EU-Member States

=> Headed by the cluster of VDR EU-RLs of

BVL-Berlin, Anses-Fougeres, & Rikilt-Wageningen

End 2018

=> Technical draft transfered to EU Commission and to the CVO of EU-Member States

End 2019 **=> Adoption of final version replacing Decision 657/2002**

Topic n°2

Update on multi-class multi-residue method on veterinary drugs in food and AOAC process to submit the proposal of analytical method

– by Thierry Delatour

Topic n°3

- **AOB … :**

The AOAC Official Method – is it time for a new approach?

Victoria Siegel, Cheryl Lassitter (AOAC Contaminants Community Co-chairs)

and Jo Marie Cook (NACRW working groups)

The AOAC Official Method – is it time for a new approach?

- **The AOAC Official Method – is it time for a new approach?**

The AOAC Official method is the gold standard when it comes to analytical test methods, and we all know that data produced using an AOAC method is less likely to be challenged and is easily defended when disputes arise over test results. We know what we like about them, and we also know that the journey to produce these officially recognized procedures takes a long time, involves a lot of support and is expensive! Many really great methods never achieve this much sought-after status. Perhaps we can also agree that for ISO 17025 accredited labs, use of an Official method is not as critical as it once was; we are verifying the performance of the test methods that we use on the samples that we are testing, whether we are using an Official method or not. Many Official methods are being adapted for use well beyond the initial scope of the original procedure. Perhaps the best example of this is AOAC 2007.01, "QuEChERS". The extraction procedure used in this test method has been applied successfully to countless applications, well beyond the 26 pesticides and three fruit and vegetable matrices of the Official Method. We are consummate modifiers, and in doing so we are improving the performance, and expanding the applicability of the methods while making efficient use of our resources. We can demonstrate that the methods perform as well for these new applications, but they no longer have the same status as the Official method. What if we develop an Official process to demonstrate equivalent performance of the method you used to the performance required of an Official method? Could we get regulators on-board?

What would be involved?

The AOAC Official Method – is it time for a new approach?

- Let's take a look at the key elements involved in the current process for developing an Official method.

  - **Support** – there is a recognized need for the method, and people are willing to invest resources (time, technical expertise, supplies and money) in the method
  - **Performance criteria** – requirements that are specific to the use of the data; agreed upon by experts
  - **Single lab validation (SLV)** – data that demonstrate potential method suitability that has been put together in a format for review
  - **Assessment of performance** – collaborative study with a single sample set or use of reference material; expert review of the data produced
  - **Publication** – performance criteria, SLV and collaborative study data are published; the method itself is written in AOAC format and published in OMA

The AOAC Official Method – is it time for a new approach?

- Can we demonstrate method suitability in a different, streamlined, efficient and less costly manner? An Official process can include many of these elements, but also would provide flexibility and allow tailoring to meet specific needs. Let's talk about the resources needed for these elements. Support comes from stakeholders and all of us in the Contaminants Community fall into that category. We need subject matter experts (SMEs) that have been vetted to ensure they have appropriate credentials. Developing and publishing templates for performance criteria, SLVs, and data packets to document performance assessment will be essential. Reference materials (RMs) are an absolutely critical component of this process. Development of suitable materials and provision of guidance on assessment of your performance using RMs is likely to be the most costly part of this enterprise, but will be extremely valuable.
- Are you interested in this idea? Would you be willing to work towards this goal? In collaboration with NACRW we can utilize expertize of our Community volunteers to develop this process. Will you join a working group to help move this forward? Elements of this process would require specialized working groups (such as developing an SME pool with vetted credentials who can be assigned to work on developing performance criteria, and criteria used to review performance or development of document templates to be consensus approved by the community and made available publically). Other activities will fit well into existing NACRW working groups.
  - Now is the time to sharpen your pencils! Will you join us?

- **Any Other Business or Request from the Sub-Group Attendance**

Questions ?

1. *Vicky announced her interest to put a scientific session next year dedicated to new approaches for validation of comparability of analytical methods*

2. To be completed with questions raised

3. To be completed with questions raised

132nd AOAC Meeting Scientific sessions of concern for the VDR sub-group

- **Monday,** August 27, 2018
  - 3:30 pm – 5:00 pm (Grand Centre): Symposium: Worldwide Perspectives on Contaminants Testing in Food and Environmental Samples Using Advanced Analytical Techniques
- **Tuesday**, August 28, 2018
  - 10:15 – 11:45 am (Grand West): Symposium: Which Multi-Class/Multi-Residue Method Strategies are Applicable for Veterinary Drug Residues Control in Foods? Multi-Target Screening and Multi-Confirmatory Quantification
- **Wednesday**, August 29, 2018
  - 8:15 – 9:45 am (Grand West): New Blood 2018 - Developing Methods for the Detection of Important Chemical Analytes, Residues and Contaminants

Up-coming events for 2019 …

- June 2019 : AOAC-Europe section workshop
  (3 – 4 June - Oslo – Norway)
- July 2019 : 56th NACRW
  (27-31 July - Naples – Florida)
- September 2019 : 133rd AOAC International Annual Meeting & Expo
  (8 – 11 September - Denver - Colorado)

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