

# 2019 AOAC Pesticide Sub-Group Community Meeting Minutes

11:45am – 1:15pm

Tuesday 9/10/19

Denver Downtown Sheraton Hotel

- Introduction from Chair: Ping Wan introduced herself and the new subgroup co-chair Katie Banaszewski from NOW Foods
  
- Update on nDATA collaborative study from Jon Wong and Jian Wang
  - Overview of the collaborative FDA/CFIA HRMS Pesticide Screening project
  - Study has been expanded globally to industry, federal and state labs
  - 2/3 of the results have been submitted and still waiting for data submission from 10 labs
  - Shipping internationally has been a challenge due to the number of samples and weight of the packages with dry ice but, most arrived with the samples in suitable condition
  - November is target for having all of the data in. CFIA to perform MS data processing
  - Early 2020 is target for having a manuscript prepared
  - Protocols are currently available for additional interested laboratories
  - nDATA approach is suitable for both quantitation and screening but the focus is on screening
  - Collab study covers 50 pesticides; the goal of database is to contain >1000 pesticides and metabolites
  - Collaborating with both LGC and Alta Scientific for mixed standards
  - Method is suitable for both QToF and Orbitrap platforms and as more manufacturers providing the capacity to perform the data acquisition it will become more universal
  - Still recommend the usage of QC standards with approximately 20 analytes for performance monitoring (a subset to save data processing time)
  - Comparing HRMS to QQQ - non-targeted method is faster for method development and transfer and saves time because each compound doesn't have to be optimized individually
  - Next Steps
    - Kate Mastovska asked if the method would be taken through the AOAC process, potentially sponsored by FDA?
    - Jon Wong would want community support before going through the process
    - It was mentioned that different HRMS platforms from different vendors would all have to meet the performance requirements
    - Jian Wang mentioned that the QToF and Orbitrap results align well from the data they have gotten so far
    - Amadeo Fernandez-Alba pointed out that a limit of detection for the screening still need to be defined

- Dr. Pang read comments from his technical staff who had participated in the study. The comments were all positive and he specifically mentioned the quality of the preparation (samples and training) and the documentation. They also liked the data processing software and how efficient it was. He also pointed out the satisfactory rates of false positives and false negatives.
- Update on Pesticides in Cannabis
  - Jessica Krank from Colorado Department of Agriculture pointed out that the main difficulty they are facing is finding a suitable extraction method for pesticide in cannabis that has good recoveries but doesn't dirty the instrument so much
  - Don Shelly from LCG asked Jessica K. about the increase in requests for isomeric compounds and she explained that many of the isomers are resolved peaks so need to be quantitated separately so the calibration curve expected concentration need to be adjusted based on the ratio of each component in the standard.
  - An attendee asked if the requirements were different depending on the mode of consumption and in Colorado, they are not.
  - Ping Wan mentioned that in Indiana they regulate pesticide products through their registration and usage and that some 25B exempt products have been found to be contaminated/adulterated
    - Inspectors received tips about products that were supposed to be organic oils (neem oil, garlic oil etc.) but were found to contain malathion
- Glyphosate
  - Ping W. introduced the topic mentioning the idea of a collaborative study since it is such a difficult compound and no AOAC official method available. She also mentioned the PERFORM working group that came out of NACRW as a way for labs to demonstrate acceptable method performance.
  - Jerry Zweigenbaum from Agilent stated that because Glyphosate is a chelating agent the system needs to be deactivated and that there needs to be more robust chromatographic techniques. He brought up ion exchange but mentioned that it is very matrix dependent and that metal ions ( $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ ) in the water can affect the chromatography
  - Amadeo F. suggested the use of IC as a sample introduction for glyphosate but said that the IC does not communicate well with the MS. He was asked and confirmed that he did use a suppressor on his IC. He reiterated that the main difficulty will be linking the two types of instrumentation.
  - Katie B. also likes IC coupled to MS because it is robust but, pointed out that it is prone to matrix interference that would need to be mitigated. She mentioned that the system does not need passivation and she doesn't see peak tailing as in HPLC. She said that in their lab only specific analysts are trained to run the method.
  - Katie B. said that her company struggles with the differences in regulation and regulatory limits between the US and the EU.
  - Jon W. mentioned that the tolerances are listed in 40 CFR and to make sure to differentiate between human and animal consumption

- Jerry Z. said his suggestion is just to assume zero tolerance regardless of the regulations
  - There was discussion about different methods (IC/MS, Ion exchange LC with MS on bioinert system, FMOC derivatization) so without a real consensus of the best method it wasn't really at the stage for study leaders and/or a collaborative study which led to the PERFORM working group
  - Vicki Siegel gave a brief description describing it as a way to harmonize proof of performance / "fit-for-purpose" regardless of the levels needing to be evaluated. She described it as a best practice approach with the goal of a blind reference material set for validation. There are some criteria in place but, it is still in early stages.
  - Don S. informed the group that they are working on incurred reference material with NIST (glyphosate in oats).
  - Melissa Phillips from NIST expanded on this and said that they hope to have the reference material ready for an inter-lab study next spring with the goal of it becoming a CRM.
- Acid Herbicides
    - Ping W. introduced the topic by asking the group about ideas for matrices and candidate methods that could be applied to the list of ~12 analytes from last year. She also brought up the possibility of applying the PERFORM approach to this group of analytes. It was confirmed by Melissa P. that there are no CRM's available from NIST.
    - Jerry Z. said that he had been having some success with a method based on the 8151 method from EPA using SPE and LC/MS
    - Don S. informed the group that from a reference materials provider standpoint they are not able to import their standards from Germany right now because chlorophenoxyacetic acids are considered "weapons of mass destruction" in neat or solution
    - Alta Scientific said that they have the standards and work with Sigma in the US
    - Joe Konschnik mentioned that RESTEK also has them available.
    - Kate M. brought up the need for reference materials in matrix because of the conjugated forms that occur
    - Amadeo F. agreed with Kate, pointing out that the recoveries from spiked samples are potentially not accurate
    - Vicki S. suggested submitting a proposal to the analytical solutions forum for consideration for next year's meeting to try and get some more traction and support from AOAC
    - FUTURE ACTION – Ping will circulate an email with a call for ideas to the attendees of the subgroup meeting
- NEW TOPICS
    - Don S. suggested people use the new TDRM database from AOAC which contains good quality reference materials from different vendors. The site was not live yet but, hopefully will be soon.

- Vicki S. asked about the level of interest in pesticides in dietary supplements. They are technically considered food by the FDA but, have no set limits so USP561 is being used and NSF has proposed using the organic list.
- Update from Dr. Pang on the Chinese multi-residue method for LC and GC/QToF for 970 multi-class pesticides and chemical contaminants in fruits and vegetables.
  - He gave a brief background of the group's history of establishing AOAC methods and then on the scope of the current project including identification criteria, scope of analytes and their use of digital standard fingerprints rather than neat standard material.
  - He presented data showing the results of national agriculture screening in China and the incidence of various categories and classes of pesticides identified on the sampled commodities.
  - He proposed an abridged collaborative study design in which a subset of analytes would be validated in a subset of matrices. 20 pesticides by each platform (GC/QToF and LC/QToF) in 2 fruits and 2 vegetables with two concentrations in each matrix. Each participating lab would receive 16 fortified and 8 incurred samples; limited sample amount only enough for one extraction. For a lab to be involved, they must have performed well and passed with the samples in the pre-collaborative study to be invited to the actual study.
  - The result of the study would be an official method paper.
  - Interested labs can email [cigpang@163.com](mailto:cigpang@163.com) and the goal is to have 30 collaborator labs in place by the end of the year.
    - Jian Wang pointed out that the 163.com email address provided may not work from secured emails in the US and suggested creating another.
    - Study details will be posted on the NACRW website.

### **Future Meeting**

- NACRW - July 26-29, 2020 Fort Lauderdale, FL
- AOAC - September 13-16, 2020, Orlando, FL