Magruder Committee Minutes

February 18, 2020 – New Orleans, LA

Meeting called to order at 10:20 am by Bill Hall

Attendance - Thirteen control officials and thirty-two industry

Introductions of committee members

Motion to approve agenda – Sharon Webb, seconded by Ametra Berry - approved

Minutes – motion to approve by Sharon Webb, seconded by James Bartos – approved

Treasurer's report – Jamey Johnson

Does not show any income for this February meeting. Statement is through January 31, 2020. There is some income on the methods forum. Finances are above where we were last year. Total assets are $123,280.49. Expenses were $28,696.13 and net income was $9,317.39. Jamey would like to give some credit to Frank who does not provide samples if payments are not paid after a period of time. This is the second year the Methods Forum funds are wrapped in with the Magruder funds. Forum has ~70 attendees registered; works well if we have at least 50 sign up.

Motion to approve Treasurer's report – Hugh Rodriques seconded by Sharon Webb – approved

Forum – format changed last year; good comments on improved format (more discussions, less breakout sessions)

Does anyone have any comments about the forum?

Tour last year was a good one; went to NM state university; looked at chemistry dept and mining museum; also went to the Energetic Materials Research Facility; got to see materials detonated; this year going to dead zone of Gulf of Mexico research area.

James Bartos – Value in using the Magruder program for method validation purposes; could be designed in advance to use data for these purposes. Issues identified with IAs, using data for identification of poor performing methods. Example, ICP for potash vs. gravimetric method. Can fold data from these methods in to address changes in IA. If there are poor performing methods, there are opportunities to address methods identified through Magruder in Lab Services Committee.

Bill Hall asked if anyone is not participating in Magruder and why. Kevin with Mosaic said they don't participate because there are not enough phosphate samples for their purposes. Ed Thomas asked if there could be a sub category of Magruder for phosphate manufacturers? Bill Hall indicated that this has been looked at before. Asked how many labs there may be. There are 5 phosphate labs and 4 potash labs. Asked if other producers here would be interested in participating in this?

Sharon Webb said that AAFCO addresses this by determining how many labs they need to have to break even

James Bartos – AAFCO isn't monthly, could be less frequent

Frank Sikora – would we be competing with AFPC?

Kevin Sapp from AFPC/Mosaic Co - No, it gives us something a little different.

Bill Hall – anyone interested in working group to determine minimum number of labs.

Bill Hall - How many labs in AAFCO?

Sharon Webb – 20 labs; needed for money reasons and statistics

Kevin Sapp – Question on number of labs; also maybe some labs that are in AFPC are not in Magruder; Would some of the Magruder folks also participate in AFPC?

Sharon Webb – Described how AAFCO does it quarterly.

Bill Hall – Some regulatory labs also look at raw materials. May not be getting enough check samples for these purposes. For statistics, need to have a minimum number of 7. Definitely something to consider. Based on feedback from this meeting, will put together a proposal to report at summer meeting.

James Bartos – Challenge to us as managers, opportunities to participate may be shrinking due to other project responsibilities; adding more can complicate things regarding participation.

Bill Hall – any problematic analytes or matrices? Will discuss more later.

Bio-Stimulants and Beneficial Substances

Bill Hall – Biostimulants; people are working to develop methods; determine what is and is not a biostimulant; regulatory challenges; is there an active ingredient and a method to test for it; its early yet; have had one go through (humic Acid); Frank Sikora has some data to share on this today (results recently submitted). Another point, if you are in the program, run and report as many samples as you can so we can have better statistics.

Bill Hall - Other than humic acids, is anyone ready to dive into biostimulants? Don't have any methods yet. Need to start thinking about this. EU is already doing so. While we are not ready yet, there will be a great need to have something out there and available for them.

James Bartos – another opportunity for a subset group for PT samples. Might blend these in as well.

Sharon Webb – said that this was difficult for AAFCO (micro testing)

Frank Sikora – Maybe we could get advice from experts in field.

Sharon Webb – Feed ships microbiological samples frozen overnight.

Ed Thomas– Get methods through ISO ring test. Does AAPFCO want to endorse ISO methods for biostimulants (cost and time considerations). If you have an active champion for AOAC, that can work. And if we can get a better ring test. Do we want to consider moving in that direction (especially with the number of potential methods out there)?

Hugh Rodriques – had a look at EU methods; same thing that is in biological manual; general plate count/yeast and mold; has EN number; established methods for basics coming out right now; challenges going forward, looking at speciation.

Bill Hall – asked if anyone in here has experience in manufacturing biostimulants; need help in how to obtain, store and ship these types of samples. AAPFCO is in the center of this regarding management

Dave Howard from CXI – not microbial biologist; sampling will take sterile sample of product; shipping product all over country so could do so elsewhere. Standardization of that; what does industry need to know. Would be glad to sit in and assist with this.

Frank Sikora – Regarding Ed's point for ISO, we (Magruder) could assist with sending samples out for the ring study.

Administrative Update – Frank Sikora (see Powerpoint for details)

Number of active clients – currently have 138 labs in program and new application a couple weeks ago – up to 139. In 2015, had 119 labs (historical reference). We get a number of international labs. US public = 43, US private = 36 and International = 59. See chart for number of labs who have paid and number of labs reporting.

Frank Sikora sent out a customer survey last week – received 63 responses; on average received 4.3 stars ranking. Will send out information for review. Fact sheet on Certificate of Analysis – In 2018, Magruder starting reporting a COA for each sample on website; showed example on screen. Provides robust mean for all lab's results with standard uncertainty. Number of labs are also shown; if at least 17 labs report, then MU is shown. Discussed it would be good to have a fact sheet on what is being shown; is now available. Under resources and articles, there is a fact sheet available for this. Went through review with Andy. Committee can review and provide feedback to Frank. Any questions?

Has anyone found these useful? Value added? Can further discuss when we talk about reserve samples. Can submit COA with these samples (valuable). Also, cost of reserve samples.

Frank Sikora- Would like to get approval from the committee on (and discussion) is how we advertise purchasing reserve samples? We don't receive many of these (probably 5-6 per year). We have these samples and labs could benefit for QC samples. Some samples have had homogeneity work done on them. Andy does a pseudo-homogeneity test using N (does same thing for AAFCO). Frank would propose charging $50 for all samples. Right now we charge $28 for each and $50 for certified ones.

Bill Hall – if not homogeneous, we would not provide a COA.

Jamey Johnson - $50 would not cover international shipping; only for US samples

James Bartos – proving homogeneity is only for some analytes (N); don't have for all.

Bill Hall – Need to update website with Jamey's new contact info

Frank Sikora – Made a motion to charge $50 for all samples with additional cost for international shipping; Lawrence Mayhew seconded

Discussion – need to find a way to advertise; how do we let other fertilizer samples know we have these available. Hugh suggested having it on the left-hand side of the webpage to see instead of having to scroll through. Click on it and it would go to application. Bill – don't know if we want to go so far as to say it is a reference material; Lawrence said using word "Reserve sample" is not as familiar; Recommend using the term "Quality Reference Material" (QRM). This is used by AAFCO. Question from

Lawrence Mayhew regarding uniformity, would there be reason to assume there would be differences for various nutrients; answer is yes, there are a number of reasons they might not be homogeneous.

Motion approved by majority vote; no vote – think cost is too high

Frank Sikora would like to ask for committee approval to purchase fertilizer for the program. Sometimes we get contacts for samples and Frank coordinates with Bob Able. It would be

Sharon Webb made a motion to give Frank authority to have authority to purchase fertilizer samples up to $500 per year to use for situations where promised fertilizers fall through. Seconded by Lawrence Mayhew.

Discussion: Regarding mechanism, as reimbursement. Bill – concern over getting SDS. Sharon said she pays for it with UK funding and gets reimbursed.

Motion approved unanimously.

Frank Sikora – New method codes. Showed location on website. Notes are placed at bottom of codes when changes made. This past August, made a phosphorus code obsolete to improve # of labs reporting appropriate code and added soluble silicon and urea and humic/fulvic acids.

Bill Hall – reminder to labs to be sure to use correct codes when reporting results.

See slide showing DAP Sample 170111 and DAP Sample 191011. James will be providing additional info on this. This data would be used in place of a collaborative study.

Discussion on method codes

Hugh Rodriques – May want to look at putting through Lab Services to verify that product registration certificates are done correctly.

Frank Sikora reviewed results for Humic Acids; may needs some education on telling labs where and how to report results. Good participation on humic/fulvic acids.

Questions – Hugh Rodriques asked who claimed 20% for these?; answer was manufacturer.

Lawrence Mayhew – in process of generating training video on procedures; helpful with ring study; were able to educate and walk them through the process; video may help with outliers.

Frank Sikora – hope that people look at their results and if they are an outlier, then investigate why they are and take steps to address.

Bill Hall – benefit of having a reference material; provides method performance information. Asked if there will be a reference material? Frank said that if there are 17 labs; thinks we will have. Lawrence said that there will be 2 different methods (COA based on all results).

Frank Sikora – if lab consistently has a high bias, they would need to explore why.

Bill Hall – this goes back to a review of the data; newsletter recommendations.

Patty Lucas – Regarding laboratory evaluation of their performance on check samples, shared that review of check sample results is standard procedure for ISO17025 accredited laboratories (or labs that

have a Quality Management System if not accredited) during annual management review and upon result receipt to determine corrective actions. Bill asked how many state and industry labs were ISO accredited (several state and one industry).

Frank Sikora - Range is reported for two values.

Jamey Johnson - asked if reason that so few labs reported due to cost.
Sharon said that for states, it depends if they have that program. Bill asked Lawrence if humic/fulvic labs are familiar with Magruder.

Validation Support for AOAC 2017.02 Using Magruder Data – Sharon Webb

Use data generated from Magruder to get data that would be obtained from collaborative study. Use specific method code for study.

Bill asked if there were enough labs –

Patty Lucas – recommend informing labs that this is how the collaborative study will be conducted and to report results using this method number AOAC 2017.02.

Bill Hall – brought up an issue that we could run out of sample if we start doing this and using reserve quantities.

Bill Hall – make an effort to use 2017.02 for this purpose.

Use of IA Index – Need More Explanation? Acceptance?

Frank Sikora – Method IA ratio on reports is measure of robust std dev/IA at analyte assigned value

Bill Hall – good indicator of where we need to focus our efforts

Break for Lunch – 12:00 – 1:00 pm

Magruder Committee Meeting continued…

Sample Selection Discussion – Criteria include low, medium and high ranges of primary, secondary and micronutrients; liquid samples, eg. UAN; organic matrix samples (eg. Plant/biosolids based); humic acids; some simple compounds/blends, ammonium sulfate, DAP and MOP with sulfate micros; some complex compounds/blends, methylene urea, high Cl phosphates, SOP/SPM; some elemental and combined S, oxy-sulfate micros; ingredients (MAP, DAP, MOP); more than 12 samples a year, a few duplicate samples are acceptable.

See spreadsheet for sample selection information – compiled during meeting

Discussion on sample preparation will occur in the Methods Forum.

A Process for Collecting and … - James Bartos – see Powerpoint presentation

Determine if the Magruder program…

Need a well-defined method, Magruder method code for reporting, at least 7 valid data points (more will allow for removal of outliers), a range of fertilizer/product types, range of matrices and sources, range of concentrations, variety of lab types, enough samples.

Questions:

Frank Sikora - What is difference between % repeatability and % reproducibility and standard deviation repeatability and reproducibility. Multiply by 2.8 (2.77 rounded). 1.96 is actually the 95% confidence interval. (see slide)

See slide for % r & % R (ISO) vs. RSD(r) & RSD(R) AOAC

cv of 10 vs. 3

Advantages of process include less of an imposition on study director, data is there to be mined, don't need to prepare samples (high quality study materials), less effort to recruit collaborators, overall costs are less if running samples anyway, overall time savings

Disadvantages include less control by study director, no practice samples to pre-evaluate or exclude a lab, can influence but can't control who collaborates, collaborators unknown to study directors (harder to know if deviating from method), lab types not known (state, industry, private, etc.), etc. (see slides)

Final thoughts – this may represent future of method validation, most tools in place, allows for comparison to other methods, could move very quickly to N, P or K type methods, etc. (see slides)

Motion to adjourn by James Bartos with second by Ametra Berry. Motion approved.

Magruder committee meeting adjourned at 2:02 pm.