MAGRUDER NEWSLETTER

Spring 2023 Edition

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CHAIRMAN NOTES

After being scolded by my parents for doing something bad as a child, I would often claim, "Everyone else is doing it". My parents would then remark, "If your friends jumped off a bridge, would you do it, too?". I am reminded of that saying after reviewing data over the last eight years in the Magruder Fertilizer Proficiency Testing Program. When our PT Program evaluates a Z-score for your result, the score is partly based on how widely distributed all the lab results are. If there is high variability from all the labs, you can receive a Z-score that seems okay. However, your lab may be jumping off the bridge with others. Investigational Allowances in the Association of American Plant Food Control Officials (AAPFCO) Official Publication (OP) were derived several years ago from expected variability across labs that were lower than current variability for the same type of fertilizers. Being like everyone else may not be a good thing. An article is presented in this issue that discusses how your Lab Report from the Magruder Program can help you evaluate if your result could cause a decision of falsely claiming a deficiency based on AAPFCO Investigational Allowances (IAs).

As the heading on our website states, the goal of the Magruder Program is to strive for analytical excellence. Tools and information are provided in the Program to help accomplish this goal. This issue includes information from SPECTRO that can be used to improve accuracy using ICP for analysis of P and K. Also, Bill Hall, the catalyst behind the Methods Forum for many years, shares the history of the Forum and its goal to educate analysts and improve method performance in fertilizer testing. We also recap new features in the Lab Portal including payment for enrollment.

- *Frank Sikora*

2023 SAMPLE SCHEDULE

The Magruder Program is a proficiency testing program for fertilizers that aims to provide an external quality control samples for the laboratories around the World. Each subscribing laboratory receives one or two samples each month. The samples contain varying levels of plant nutrients as well as trace metal contaminants.

Within the sample schedule, the overall goal for the Program is to select a wide range of fertilizer samples to broadly cover the requirements of industry, commercial, and regulatory laboratories.

Here is a calendar of fertilizer samples offered in the first half of the current subscription year across the Regular, N, P, and K schemes.

Sample number	Sample month	Description	Scheme
230111	January	Organic	Regular scheme
230213	February	UAN	Regular & N scheme
230311	March	NPK	Regular scheme
230341	March	P rock	P scheme
230411	April	High micros w/NPK	Regular scheme
230451	April	Potash	K scheme
230511	May	Si product	Regular scheme
230611	June	Coated product	Regular scheme
230631	June	Urea	N scheme
230714	July	DAP	Regular & P scheme

Subject to change

- *Job Fugice*

NEW METHODS OF PAYMENT AND THE NEW LAB PORTAL

While the function of entering laboratory PT data has remained the same, there have been significant changes to how payments are made to the Magruder Program. Payments are now processed via "Dashboard," a companion website that is managed by FASS. This is different than how Magruder payments were managed prior to 2022. Instructions for accessing and using Dashboard have been added to the Magruder homepage. In brief, each laboratory identifies the subscription manager for the lab. That individual acquires a username and password for the Dashboard and can add or remove users to the Lab Portal.

Once in the Dashboard, sample schemes and shipping methods for international labs can be selected to create an invoice. The Dashboard is the same site where payments can be made on-line via credit card, check, money order, purchase order, and wire transfer.

It is important to realize that samples are not shipped until payment has been received. If a laboratory submits payment after the annual subscription begins, the laboratory still receives samples from previous rounds. However, these samples may be received too late for data to be included in statistical reports. As a reminder, PT data cannot be entered in the Magruder Lab Portal after the due date has passed.

The updated Lab Portal is now active for 2023 Magruder PT samples. Instructions for navigating the Lab Portal are available here. New features in the Lab Portal include:

- Welcome screen with current news alerts with the "Information" button.
- Update postal address for sample shipments and emails for shipment alerts with "Lab Admin" button.
- View of shipment tracking information with the "Shipments" button.

- *Frank Sikora*

IS YOUR LAB REPORTING A FALSE DEFICIENCY?

Reports from the Magruder Fertilizer Proficiency Testing Program provide two different measures of a lab's result for plant nutrients. One measure is well-known among proficiency testing programs as the Z-score. The Z-score measures how well the lab result compares with the robust mean considering the distribution of results from all other labs. Another measure important for the regulation of fertilizer in the United States is evaluating if the lab result is within the Investigational Allowance range as defined in the AAPFCO OP.

A laboratory's report is shown below for diammonium phosphate fertilizer (18-46-0). The Z-score for available P_2O_5 was -1.71. The negative Z-score indicates the lab value was less than the robust mean of 46.35%. The magnitude of the Z-score indicates the lab value was between 1 and 2 standard deviations away from the robust mean. The green Z-score indicates an acceptable lab value because it was not greater than 2 standard deviations of the distribution of all lab results.

A quality control manager or ISO 17025 auditor could evaluate this Z-score and conclude that the laboratory's performance in testing available P_2O_5 is adequate. However, in the realm of fertilizer regulation based upon the AAPFCO IAs, more scrutiny is required. The other comparative measure on the report evaluates if the lab result falls within the AAPFCO IA range, which is equal to the robust mean plus or minus the IA. For the case of the lab report below, the IA at the robust mean of 46.35% is 1.00% for available P_2O_5. The lab value of 45.15% has a footnote next to it indicating it is less than the robust mean minus the IA which is 45.35%. The report also highlights results that are higher than the robust mean plus the IA, which in this case is 47.35%

This second measure with the red value and footnote alerts labs that accuracy of their testing needs improvement. It is especially important for US regulatory labs to recognize the special footnote and critically analyze their laboratory practices so results are produced closer to the robust mean to avoid reporting a false deficiency in their regulatory samples. One tool to help with improving accuracy is included in a fact sheet from SPECTRO in Vendor's Corner (page 6). This fact sheet provides guidance on improving the accuracy of available P_2O_5 and soluble K_2O results using Inductively Coupled Plasma (ICP) spectroscopy.

magruder fertilizer check sample program

STRIVING FOR EXCELLENCE IN ANALYSIS

Sample # 210812: 18-46-0 DAP

Analyte Report Card for Lab Code 0999

Proficiency Testing For 5 Analytes — **Issue Date : 09/30/2021**

Analyte Group	Analyte Group (Units)	Lab 0999 Data		Analyte Values				Magruder Z Score	Your Method	Flag
		Value	range	Rob Mean	Rob SD	R-bar	# Tests			
010	Total N (18%)	17.65	0.7000	17.90	0.2676	0.1338	72	-0.92	010.12	0
041	Direct Available P2O5 (46%)	45.15†	0.3000	46.35	0.7034	0.3366	55	-1.71	041.21	0
050	Soluble K2O (%)	0.1600	0.0000	0.1772	0.0166	0.0068	10	-1.04	050.51	0
101	Acid Soluble Ca (%)	0.2060	0.0020	0.1947	0.0499	0.0044	9	0.21	101.30	0
121	Acid Soluble Mg (%)	0.6275	0.0350	0.8114	0.1242	0.0224	11	-1.39	121.30	0

Interpreting Z Scores: Red indicates a normally distributed Z value >3 or <-3 (requires action), Orange = Z between 2 and 3 or -2 and -3 (warning) and Green = Z < 2 and >-2 (OK at 95%). Flags indicate data usage: 0 = Used, 1 = rejected for duplicates too far apart, 2 = rejected as extreme outlier, 3 = removed from stats, 4 = rejected due to 0s submitted, 5 = LOD. The 9 Flag indicates a data problem - scores not calculated. Robust statistics not used if < 6 labs used in calculations, in this case the Z Scores are grey and included for information only. Your offset values are higher (‡) or lower (†) than the Robust Analyte value ± Investigational Alowance. Analyte codes in light green indicate a guaranteed analyte. Lab values below detection limits reported solely for this PT program. Your offset values are higher (‡) or lower (†) than the Robust Analyte value ± Investigational Alowance.

- *Frank Sikora*

MAGRUDER CELEBRATES 100 YEARS!

Attendees of the 2022 AAPFCO Summer Annual Meeting (St. Louis, MO) celebrated the 100-year anniversary of the Magruder Fertilizer Proficiency Testing Program. Chairman Frank Sikora shared Program highlights with the group as the venue sliced the cake to be served during the Presidential Reception.

- *Sally Flowers*

METHODS FORUM OVER THE YEARS – PART 1

Fit for purpose analytical methods are a vital part of fertilizer production, regulation, distribution, and consumer protection. While this seems obvious, often the process of regulation development outpaces the development of needed methods. Further complicating the issue is assigning the responsibility of method development and validation. Ultimately it is the responsibility of all stakeholders to determine the method requirements and ensure that the needs of the entire community are met. It can take some time for this reality to sink in across the entire community.

The fertilizer community has many stakeholders: producers, distributors, regulators, consumers, and other interested organizations. It is up to this community to organize and determine together the method needs and priorities assuring that methods developed are "fit for purpose". Historically, methods were developed by regulators and/or industry chemists, who often worked in virtual silos, without the full input of the entire community. This process relied on the experience and perspective of the method developer to understand the needs of all the stakeholders. This process began to break down and become inadequate in the 1970s and 1980s as resources (manpower and funding) within the states and industry were overextended.

The birth of the Fertilizer Methods Forum was a result of imminent regulatory action in a few states in the late 1990s. This action proposed upper limits for trace or "heavy" metals in fertilizers. Regulations and regulatory limits had been developed based on the public perception that many fertilizers contained dangerous levels of some metals. Risk assessments were undertaken; but those took time and the public wanted action. Few existing methods were designed for the unique matrix and problems that fertilizers present. Consequently, the first methods that were applied were not developed for, or validated, using fertilizer materials. Typically, they were environmental, water, or soil methods.

Fertilizer materials are generally of two types – minerals and organic-based products. Mineral fertilizers are unique because they often are composed almost entirely of salts, such as potassium chloride,

ammonium phosphate and similar compounds. Organic-based products are composed of remnants of plant or animal-based materials. Both present different extraction and detection problems for methods that are based on traditional methodology. Applying traditional methods for solids or environmental matrices caused bias or unusually high variability in the data. Thus, there was a need for methods designed specifically for fertilizer materials to provide the accuracy and precision required for the analytes and concentrations subjected to regulation.

The first forum was organized in 2002 to address the need for appropriate trace metal methods. Consequently, the first few forums were titled, "Fertilizer Metals Forums". As the need for more methods were identified to address other fertilizer analytes, the name changed to the "Fertilizer Methods Forum". In the United States, the primary path to method development and validation was and is the standards organization, AOAC International. AOAC International is an organization with its roots in North American agriculture and fertilizers. However, the organization entered a period of financial difficulty and, to continue its mission, began to require substantial fees to use their method validation infrastructure. This coincided with a depressed fertilizer market and reduced funding levels to state regulatory laboratories, which made it very difficult to raise the funds necessary to put needed methods through the AOAC validation process. Method authors needed to gain financial backing and ensure that the methods that enter the system meet the uppermost priority of the community and garner universal community support.

Early contributors to the Methods Forum

The first and primary method to be developed through the Forum was AOAC 2006.03 arsenic, cadmium, chromium, cobalt, lead, molybdenum, nickel and selenium in fertilizers by microwave digestion and inductively coupled plasma emission spectroscopy. This effort took four years to complete and was a result of many hours of volunteer work and laboratory support based on Forum input.

Over the years Forum meetings were held annually in conjunction with the winter meeting of AAPFCO. While the face-to face-meetings were only held once a year, a lot of work was done by forum volunteers during the year outside the meeting. Thanks to the efforts of method champions and session leaders of the Workout® process the work was guided through prioritization and completion of action items.

The Forum has continued for 22 years despite its initial driver (i.e., a trace metals method) being satisfied after only four years. That is because many methods used by the community relied on older instrumentation and technology that had changed. A survey of nearly 100 community stakeholders was conducted to identify method needs. This list guided the prioritization of fertilizer methods to be developed, validated, and funded by the community. Over the years this list has been revised several times to reflect changing priorities, funding support, and the needs of the community. It was determined that some high priority issues were not new methods but simply revisions of current methods. Through the efforts of The Fertilizer Institute (TFI), its members, and method champions, over $250,000 was committed to support the method development and validation process through AOAC International. The final list was finalized in the 2008 Forum and a contract was signed with AOAC in 2009 to begin validation of six new methods and modifications of four existing methods. Over the next three to four years the development and validation work continued through the Forum format. This work culminated in the publication of several articles in the *Journal of AOAC International* (Vol. 97, No. 3, 2014). In fact, a whole section was devoted to the Forum's work and summarized the community efforts through the first 13 years of the Forum. This section was edited and compiled by Nancy Thiex, who contributed a paper entitled, "New and Improved Methods of Analysis for Plant Food Materials". All the articles in this section were made open access thanks to the efforts of TFI and the Methods Forum.

Part 2 "New Challenges" to follow in the next edition of the Magruder Newsletter

- *Bill Hall, N-P-K Consulting, LCC, Method Forum Organizer*

VENDOR'S CORNER

When determining a concentration in an analytical method based on a calibration function developed from calibration standards, several errors can occur due to the function representing an average of the calibration standard results and a drift in measurement that can occur over time. These errors are minor most of the time. The use of ICP to determine P and K with AOAC method 2015.18 is prone to have larger errors than the older methods of gravimetry for P and titrimetry for K due to the use of a calibration function with the ICP method that is not used in the older methods. Spectro has developed a method termed the "Bracketing Technique" to reduce the error associated with a calibration function. This method has been used successfully in the precious metals arena and can potentially be used to improve accuracy for P and K determined by ICP. Quality Reference Materials (i.e., past samples purchased from the Magruder Fertilizer PT Program) can be useful in this technique. Past PT samples come with a statistical report that can act as a certificate of analysis with well documented concentrations, uncertainties, and other sample attributes. A publication highlighting the details of the method is available online courtesy of SPECTRO Analytical Instruments.

- *Dion Tsourides, SPECTRO Analytical Instruments*

UPCOMING MEETINGS

TFI: The InfoAg Conference (St. Louis, MO) June 27-28, 2023
More information available here

97th Southwestern Fertilizer Conference (Denver, CO) July 16-20, 2023
More information available here

2023 AAPFCO Summer Annual Meeting (Baltimore, MD) August 3-4, 2023
Online registration available from:
2023 Summer Annual Meeting

2024 AAPFCO Winter Annual Meeting (Mobile, AL) February 18-20, 2024
More information to come

2024 TFI Annual Business Conference (Palm Springs, CA) February 26-28, 2024
Meeting information available here

2024 AAPFCO Summer Annual Meeting (San Antonio, TX) August 5-6, 2024
More information to come

MAGRUDER COMMITTEE ROSTER

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