MAGRUDER NEWSLETTER

www.magruderchecksample.org

Summer 2021 Edition

CONTENTS:

CHAIRMAN NOTES

Changes are occurring in the Magruder program to improve our service in offering samples and data to laboratories for supporting their quality assurance. Starting in the next enrollment year of 2022, we will be offering separate sample schemes for nitrogen, phosphate, and potash fertilizers. These targeted schemes should be attractive to fertilizer manufacturers that are not interested in the wide variety of blended materials currently offered or to labs wanting to focus on materials that are most like the samples they receive. To assist us with planning, please enroll in schemes desired for 2022 by visiting https://uky.az1.qualtrics.com/jfe/form/SV_aaWHiV5qupEi3zM.

Billing will occur differently for the 2022 season. Laboratories will be billed early starting in August as soon as we know which schemes are selected. The due date for payment will be December 1, 2021. Samples will be sent after payment is made.

We have also improved information on our website for purchasing past samples in the program. Magruder quality reference materials (QRMs) can be used as quality control samples in your lab. Robust mean and other statistical data are available for each QRM, which has already been analyzed by many laboratories. Be sure to read the article entitled “Quality Reference Materials” for information on how to incorporate these samples into your quality assurance program.

Another upcoming change is a new program name. The Magruder program has historically been called the “Check Sample” program. These words have become out of date in describing a program such as ours. Consistent with ISO 17025 and related references, the modern term for an interlaboratory comparison is “Proficiency Testing”. You will begin seeing this name change on our website and reports.

- *Frank Sikora*

INVESTIGATIONAL ALLOWANCES ARE NOT TOLERANCES

A common question control officials receive is how much can my product or blend deviate from its guarantee before a penalty is assessed? Alternatively, this is sometimes stated as what "tolerance" does AAPFCO allow? Since many fertilizer products are now produced and sold internationally, this question is not unusual since some countries or regions allow or describe this as a tolerance. From ISO FDIS 8157, the definition of a tolerance is the permitted deviation of the measured value of a nutrient content from its declared value. Since it is not always clear what has been incorporated into the "tolerance," for example: sampling variability, within lab repeatability, among lab reproducibility, product, manufacturing, blending variability, etc., AAPFCO does not use or accept the word tolerance. Rather, AAPFCO recommends adherence to truth-in-labeling and suggests that products be consistent with their labeled values. If there is variability in the type, manufacturing, or blending of the product(s), then the goal should be to decrease the variability to the extent possible and to ensure the lower end of the range of expected values serves as the labeled guarantee. A "worst-case" example may be a non-uniform product with a range of 3% to 5% Nitrogen. Should this be labeled as the mid-point of the range or possibly the average of several test results? The short answer is "no" with the better approach to label it as the lowest point of the expected range. The expected range can be determined from the mean minus two times the standard deviation for the 95% confidence level from different test results, different batches in processing, or both.

Though this approach may seem unfair to the manufacturer, the purchaser of the product expects it to contain at least its labeled value. Also, the application rate and value of the product is based upon the guaranteed value. AAPFCO similarly uses its lower statistical range of variability for determining when to suggest penalties rather than targeting a preferred result. Then, for example, why does AAPFCO not take regulatory action for a 5% Nitrogen product until after the determined lab value falls below 4.49% N? Is this difference in value, or 0.51, a tolerance? The difference between the guaranteed value and the value at which a penalty is assessed is defined by AAPFCO as the Investigational Allowance (IA), which was discussed in the Winter 2021 Magruder Newsletter. The IA encompasses variability associated with the collection, testing and confirmation of analytical test results. In this example, we are not saying that a value of 4.49% N is acceptable, but rather that we do not have sufficient certainty (i.e., 98% statistical confidence) that the reported test result is not 5.00% until after the test result falls below 4.49% N. Should a company mistakenly interpret the IA as a tolerance and accidentally or intentionally make a product that is 4.49% N and guarantee it at 5%, then the variability associated with collecting and testing a product that is even slightly below its guarantee will result in some states assessing penalties.

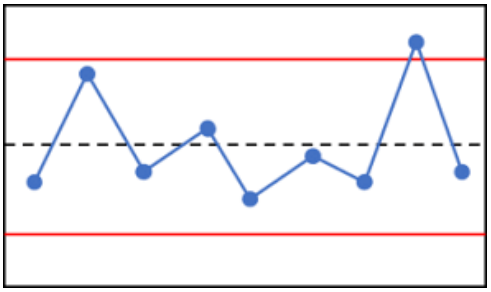
Here are a couple of final thoughts. Investigational Allowances are not all the same for all fertilizer nutrients since all test methods do not perform equally. Similarly, the absolute IA values generally increase as the guaranteed value increases, although on a percent relative value basis, IAs are actually highest for the lower guarantees because low values are harder to detect and replicate. Finally, some states may adopt IAs that are different from those recommended by AAPFCO to reflect "uncertainty" associated with their experiences. As products, test methods and technologies change, IAs may also have to be updated to reflect these changes. In closing, AAPFCO does not recognize a tolerance, products should be guaranteed to reflect their known value or the lower end of their statistically validated concentration range, and products that do not meet or exceed their labeled guaranteed value will likely result in some states taking regulatory action.

- *James Bartos*

QUALITY REFERENCE MATERIALS

How do you know that your lab is performing an analytical method correctly? Each laboratory should have an active quality program in place. This should include personnel that are adequately trained and competent, traceability of materials and instruments, and participation in a proficiency testing program, to name a few. There should be a paper trail demonstrating that the instruments used are calibrated and performing as they should by using reference materials. Each analytical method that is used in the laboratory should have a quality reference material (QRM) in each set and be matched to what is in the set; i.e., the concentration of the analyte of interest and the matrix are similar to the samples.

Definitions for reference materials can be found at www.nist.gov/srm/srm-definitions. First, consider the term, "Reference Material." This term is commonly used in the laboratory and refers to material for which the analyte concentration and uncertainty of the concentration is well defined. Wherever relevant and available, Certified Reference Materials (CRMs) should be used to calibrate instruments and testing processes. A CRM is a material that has an analyte concentration certified to be within a specific range of uncertainty. It may also be used to do one or more of the following: calibrate analytical instruments; qualify a secondary reference material; verify calibration in instrumental analyses where a non-ISO Guide 34 Certified Reference Material is used to generate a calibration curve. A Certificate of Analysis (COA) from the CRM supplier will specify the analyte concentration and range of uncertainty for the concentration. Ideally, the supplier should be accredited to ISO Guide 34 or ISO 17034:2016. Reference materials should be stored according to the manufacturer recommendations and not used beyond the expiration date. So, what is a QRM? A QRM is used to check instrument performance, review laboratory techniques and calculations, and monitor analyst performance. It is a material characterized by collaborative studies, third party testing, proficiency testing programs, or analyzing the sample multiple times and is matched to the sample matrix being tested. QRMs should be monitored using control charts.



Including QRMs in each set of data will provide reliable and defensible analytical results. It will provide a measure of the precision and accuracy of the analytical method. Using a QRM with known statistics allows for monitoring the accuracy and precision of the analyst. It may assist in identifying problematic methods and identifying training needs within the laboratory. Using a QRM with known statistics over time will provide a permanent record of instrument performance when validating data, projecting instrument repair or replacement needs. QRMs may be used not only to verify the uses above but can also be used to monitor accuracy. They may also be used to monitor batch to batch or day to day variance. It also documents the effectiveness of laboratory performance and the quality assurance program.

The data obtained from each QRM should be put into a control chart. This graphical representation of the data over time is very useful in visualizing trends. Control charts of all the QRMs should be maintained and be the basis for corrective and preventable actions when they indicate potential problems with methods. The control chart should be used once the analysis is complete. Control charts may be constructed by hand, statistical software, or even a spreadsheet program. There are several types of control charts that can be employed and will be discussed in a future newsletter.

The Magruder proficiency program should become an important part of a fertilizer laboratory quality assurance program. Comprehensive statistical reports are prepared based on ISO 13528:2015. Reports for each sample are available on the web for participating laboratories to evaluate their performance in testing the various analytes. Once the report for the sample is on the web, the sample is available for purchase. Go to http://www.magrudercheckssample.org/ and choose "Purchase Samples" under the lab portal on the left side of

the screen. You can review the available QRMs by clicking on links to the excel files. It lists Magruder sample numbers, the portions available for purchase, analyte concentrations, and certificate of analyses. Once you have chosen the QRM you would like to incorporate into your laboratory's quality program, click on "QRM Request Form.pdf". Complete this form and email it to the address provided on the form. Upon receipt, the sample will be removed from the inventory. An invoice is then emailed to you with instructions on how to proceed with payment. Once payment is received, the QRM will be shipped to you. Please make sure you complete all the forms with correct information.

The samples are reasonably priced at $50 each. Magruder is an international program with over 150 active participants. If you have not used Magruder QRMs in the past, I encourage you to do so now.

- *Sharon Webb*

NEW SAMPLE SCHEMES INTRODUCED IN 2022

The samples offered in the Magruder program are well suited for a laboratory that analyzes a wide variety of fertilizer types. The variety of samples offered is not attractive to laboratories that support fertilizer manufacturers that concentrate on analysis of a narrow range of fertilizer types. To accommodate the needs of these laboratories, new sample schemes are being included in the Magruder program in 2022. Three sample schemes will be added to include samples important to N, P, and K fertilizer manufacturers. In each of these sample schemes, one sample will be offered every four months for a total of three samples in the year. Typical fertilizer types to be included in each scheme are shown below.

N scheme: urea, UAN, (NH4)2SO4, NH4 polyphosphate, NH4 thiosulfate
P scheme: MAP, DAP, P rock, NH4 polyphosphate
K scheme: KCl, K2SO4, KMag, K thiosulfate

The regular variety of fertilizer types will continue to be offered in the regular scheme. One sample in each of the new sample schemes will be shared with the regular scheme. Since samples will be shared with the regular scheme, cost for enrolling in one of the new schemes will depend on whether you enroll in the regular scheme. The pricelist for the various enrollment options is shown below. The cost for enrollment in all the schemes is $780.

Regular scheme: $360
N scheme: $200 if not enrolled in the regular scheme, $140 if enrolled in the regular scheme
P scheme: $200 if not enrolled in the regular scheme, $140 if enrolled in the regular scheme
K scheme: $200 if not enrolled in the regular scheme, $140 if enrolled in the regular scheme

A survey is available for labs to specify the schemes they choose to enroll in for 2022 at the following link.
https://uky.az1.qualtrics.com/jfe/form/SV_aaWHiV5qupEi3zM

Labs that are new to the Magruder program will be asked to provide contact information and shipping address. Invoices will be sent out in August based on the schemes selected in the survey. Payment will be due December 1 and samples will be shipped after payment is received.

- *Frank Sikora*

UPCOMING MEETINGS:

ISO TC-134 Fertilizers, Soil Conditioners and Beneficial Substances Working Group 3 – Labeling and Marking Virtual Meeting (August 17 8:00 am to 11:00 am and possibly August 18)

- The meeting is open to participants, who are currently registered with ANSI.
- An executive summary/business plan can be found here: https://isotc.iso.org/livelink/livelink/fetch/2000/2122/687806/ISO_TC_134__Fertiliers_and_soil_conditioners_.pdf?nodeid=12254155&vernum=-2

2022 AAPFCO Winter Annual Meeting/Methods Forum (February 13-18; Tentatively: Mobile, AL)
Check https://www.aapfco.org/meetings.html for meeting updates.

2022 AAPFCO Summer Annual Meeting (July 31 - August 3; St. Louis, MO)
Check https://www.aapfco.org/meetings.html for meeting updates.

MAGRUDER COMMITTEE ROSTER

Name	Organization
Frank Sikora, Chairman	University of Kentucky, Division of Regulatory Services, Lexington, KY
Job Fugice, Vice Chair	IFDC, Muscle Shoals, AL
Patricia Lucas, Secretary	Florida Department of Agriculture and Consumer Services, Tallahassee, FL
Jamey Johnson, Treasurer	Arkansas State Plant Board, Little Rock, AR
Andy Crawford, Statistician	Crawford Science Consulting, Hacienda Heights, CA
Robert Kieffer, Sample Preparation	Able Laboratory, Inc., Pikeville, TN
Sally Flowers, Newsletter Editor	Kansas Department of Agriculture, Manhattan, KS
Ametra Berry	Georgia Department of Agriculture, Atlanta, GA
Bill Hall	Retired
David Collier	Nutrien, Aurora, NC
Deion Tsourides	Spectro Analytical Instruments, NJ
Hugh Rodrigues	Thornton Laboratories, Tampa, FL
James Bartos	Office of Indiana State Chemist, West Lafayette, IN
Lawrence Mayhew	Humic Products Trade Organization, WI
Lise-Anne Prescott	Canadian Food Inspection Agency
Maryam Khosravifard	California Department of Food and Agriculture
Michael Hojjatie	Tessenderlo Kerley, Tucson, AZ
Nadia Guagliardo	CF Industries, Inc., Donaldsonville, LA
Scott Roalofs	Colorado Department of Agriculture, Broomfield, CO
Sharon Webb	University of Kentucky, Division of Regulatory Services, Lexington, KY

Thanks to Allyssa Davis, Kansas Department of Agriculture Laboratory, for creative input in this newsletter.

Association of American Plant Food Control Officials
MAGRUDER

Contact Us

Frank J. Sikora, Ph.D.
University of Kentucky, Division of Regulatory Services
859-218-2452
fsikora@uky.edu
http://www.magruderchecksample.org