MAGRUDER NEWSLETTER

www.magruderchecksample.org

Summer 2022 Edition

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CHAIRMAN NOTES

One hundred years ago, Egbert Watson Magruder sent a sample out to 29 laboratories for comparing results. This was the start of our proficiency testing program. We still recognize the scientist who started the program with his namesake.

Science is not only a compilation of facts and figures. The biography of scientists provides valuable insight into the human element behind scientific achievements. We learn that hopes, aspirations, frustrations, and challenges are factors common in all of us as we work for advancements and improvements in our own area of work. Recognizing the human element acknowledges that mistakes will be made. The Magruder Fertilizer Proficiency Testing Program is a valuable tool that alerts laboratories when mistakes are made and gives them data to help them improve their analytical performance.

This edition includes an article on the life of Egbert Watson Magruder providing insight to the man behind our program. It is hoped that the article provides information on the man behind the name we still use to identify our program 100 years after its inception.

Other articles in this edition includes information on more improvements ahead for the Magruder program. We are updating the Lab Portal to include several new features. Articles are also included on the value of the program to a regulatory lab and information on the International Network on Fertilizer Analysis.

A common feature you will continue to see in our newsletter is a "Vendor's Corner" with tips and techniques for improving analytical performance. Information in this edition includes tips from CEM on the criticality of glassware cleanliness to reduce contamination when determining analytes at low concentrations.

- *Frank Sikora*

CHANGING OUR NAME TO PROFICIENCY TESTING

You may have noticed a change in our name on the website and reports. The Magruder Fertilizer Check Sample Program is now the Magruder Fertilizer Proficiency Testing Program. The term, check sample, is an older term used for samples sent out to various labs for testing. Thus, the program was labeled a "Check Sample Program". More recent terminology for the type of program we administer is "Proficiency Testing Program". This term is referenced in many ISO documents, in particular the ISO 17025:2017 document used for laboratory accreditation. Proficiency testing programs like ours also can obtain accreditation using ISO/IEC 17043. Our program is not currently an accredited program under the ISO/IEC 17043 standard. Although we are not an ISO accredited proficiency testing program, we do follow statistical analysis and reporting guidelines set forth in the ISO 17043 standard.

- *Frank Sikora*

MAGRUDER SAMPLE SCHEDULE

The Magruder program is a proficiency testing program for fertilizers. 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 cover a wide range of sample types to meet the requirements of in-house, commercial, and regulatory laboratories that subscribe to the program.

For this current year, the list of fertilizer samples chosen to be distributed within the Regular, N, P, or K schemes is shown below:

Sample ID	Sample Month	Description	Scheme
220111	**Jan**	soil supplement	Regular scheme
220213	**Feb**	urea	Regular & N scheme
220311	**Mar**	ammonium thiosulfate	Regular scheme
220341	**Mar**	DAP	P scheme
220411	**Apr**	24-25-4	Regular scheme
220451	**Apr**	K-Mg-SO_4	K scheme
220511	**May**	21-7-14	Regular scheme
220611	**Jun**	organic with humic acids	Regular scheme
220631	**Jun**	UAN	N scheme
220714	**Jul**	MAP	Regular & P scheme
220811	**Aug**	NP 20S with organics	Regular scheme
220851	**Aug**	K2SO4	K scheme
220911	**Sep**	NPK blend	Regular scheme
221011	**Oct**	high micros	Regular scheme
221031	**Oct**	ammonium sulfate	N scheme
221111	**Nov**	organic w humic acids	Regular scheme
221141	**Nov**	P rock	P scheme
221215	**Dec**	KCl	Regular & K scheme

- *Job Fugice*

MAGRUDER TURNS 100!

The Magruder program reaches a huge milestone with a 100-year anniversary this fall. You may wonder where the name "Magruder" came from that so prominently identifies our proficiency testing program. Magruder is the name of the chemist who began the program in 1922.

Egbert Watson Magruder was born in 1868. He grew up in rural Virginia with three older brothers. After he received a B.A. degree from Hampden-Sydney College in 1891, he attended University of Virginia and later Johns Hopkins University where he received his Ph.D. degree in Organic Chemistry.

His love of soil along with his rural background and education made him keenly interested in what it takes to increase crop yields. After some work in the Mississippi College of Agriculture and A&M College in Raleigh, North Carolina, he returned to Virginia to become Chief Chemist for the State Department of Agriculture and director of the Virginia test farm at Saxe in Charlotte County. In 1915, he became Chief Chemist for the Royster Guano Fertilizer Company of Norfolk, Virginia. The company recognized the importance of quantifying nutrients in fertilizer ingredients to ensure consistency in the products that they sold to farmers. Egbert Magruder oversaw Royster chemical laboratories to ensure accurate testing of fertilizers. His main laboratory was on the top floor of the 14-story Royster building in Norfolk and was considered to be the finest in the country. He worked with the Royster Company until retiring in 1945 at the age of 77.

Marriage came soon after he started work with Royster in 1916 when he married Frances Byrd Alvey. They did not have any children; but, they had several nieces and nephews. Egbert had a good sense of humor and liked to tease. He was deeply devoted to family by celebrating moments of joy and sharing grief in times of sorrow. Egbert Magruder was a member in several scientific organizations such as the American Chemical Society, Association of Official Agricultural Chemists, American Association for the Advancement of Science, and the Virginia Academy of Science. He also was a member of and served in several civic organizations.

Egbert was said to have an exceptionally fine mind and possessed a remarkable memory. A story was told of him visiting his brother late at night. He was not quite sure if the taxi took him to the right place. Since it was late, he did not want to wake anyone in case it was not his brother's home. He noticed two chairs that looked like the ones he and his brothers made in their youth. He investigated the construction and remembered the type of screws used in one the arms. He then knew he was at the right place.

His keen mind and attention to detail served him well in his career as a chemist. While working at Royster, he recognized the need for sharing samples across labs to improve a lab's performance in analytical testing of fertilizer. To fulfill that need, he began the Magruder Check Sample Program sending out the first sample to 29 laboratories in November 1922. Below is an excerpt of a paper he presented at the American Chemical Society in 1924 regarding the initial years of the Magruder program. With the frequent use of personal pronouns, the paper provides a good glimpse into Egbert Magruder's thought process and reasoning for starting the program. It is interesting to note that the objectives he wrote for 33 inaugural chemists 100 years ago are still applicable to the 150 Magruder lab participants today. We seek to perpetuate the vision of Egbert Watson Magruder for another 100 years.

- *Frank Sikora*

...THE...

AMERICAN FERTILIZER

"That man is a benefactor to his race who makes two blades of grass to grow where but one grew before."

Vol. 61 OCTOBER 4, 1924 No. 7

Results of Check Fertilizer Samples for 1923-1924

By E. W. MAGRUDER, Chief Chemist, F. S. Royster Guano Co., Norfolk, Va.

Published by courtesy of the American Chemical Society. Paper presented at the Ithaca Meeting, Sept. 8-13, 1924.

During the past year I conducted the check fertilizer analysis work, begun in 1922. The work consists in the analysis of samples sent out monthly to collaborating chemists. The samples are thoroughly dried and sent out in sealed containers at the first of each month. During the first year, 33 chemists took part in the work. Some of them have dropped out, but others have come in. At the beginning of the work in 1923, 35 chemists started in, and the number has gradually increased until now we have 45 on the list. The chemists participating in the work are nearly equally divided between State laboratories, commercial fertilizer laboratories, and laboratories of fertilizer manufacturers, so that all classes of fertilizer chemists are represented.

The chemists have shown more interest during the second year than they did the first, and the work on the whole has been of rather higher grade. We still have differences between the various chemists which are entirely too great. In phosphoric acids, the difference of one per cent. on goods containing not more than 10 per cent. of phosphoric acid is not uncommon, and a difference of 0.7 per cent. is the rule. On goods containing not over three per cent. of ammonia or potash, there is usually a difference of as much as 0.5 per cent. between different chemists.

In several instances, when the sample was made up, enough material was prepared for two samples and all put in the containers and sealed at the same time, one sample being sent out at that time and the other sent out about six months later. In many cases, the same chemist has gotten widely varying results on the two samples analyzed at different times, the differences being much greater than should occur.

Last year in my report I called attention to the fact that the different analysts used methods which varied considerably from one another, and also varied more or less from the official methods of the Association of Official Agricultural Chemists. I am convinced that part of the difficulty in securing concordant results by different chemists is due to variations in the method employed. I believe that all of the methods for fertilizer analysis should be standardized more accurately than they are, and that the methods should be studied more closely, and the causes of the variations discovered, and the means for their elimination worked out. I am convinced that as long as we use so many different methods, and so many variations of the same method, we will continue to have great difficulty in getting concordant results on samples prepared as accurately as possible.

The work on the check samples has served to call attention of many chemists to some faults in their procedure, and I know in some instances has served to give the chemists warning of the fact that they were beginning to get bad results. This has thus caused them to look into their methods, and has enabled them to get back to correct results.

UPGRADE OF LAB PORTAL WEBSITE

Improvements are coming to the Lab Portal website to include additional features to benefit labs in the program. Current features are limited to entering lab data and accessing reports. The additional features, as shown below, will give labs much greater control and information on their involvement in the program.

Invoicing
- Select sample schemes and generate invoice
- Make payments
- Choose shipment options
- Enroll into program as a new lab

Sample Shipments
- Update address for sample shipments
- View shipment tracking information

Laboratory Contacts
- Update contacts for access to Lab Portal
- Update email contacts for shipment notices

Quality Reference Materials
- Access inventory of samples
- Purchase samples

The new website will be similar to the program used by the Association of American Feed Control Officials (AAFCO) Proficiency Testing Program. Completion of the improved website is planned for the end of 2022 so that it can be available for invoicing for the 2023 program year.

- *Frank Sikora*

VALUE OF THE MAGRUDER PROGRAM IN A REGULATORY LABORATORY

Agriculture plays a critical role in California's economy and our nation's food supplies and security. It is essential that fertilizer products are both safe and effective as farmers rely on nutrient availability to achieve desirable crop yields while protecting our environment and natural resources. The California Department of Food and Agriculture (CDFA) Center for Analytical Chemistry Branch (CAC) is housed within the Division of Inspection Services. As a regulatory laboratory, the CAC provides analytical services to support the CDFA Fertilizing Material Inspection Program (FMIP), which also is housed in the same division. Analytical data provided by CAC informs on FMIP strategic direction for inspection activities, product registration, and research to ensure safe and effective distribution and environmentally sound use of fertilizing materials in accordance with California laws and regulations.

The CAC has long been a proponent and participant of the Magruder Fertilizer Proficiency Testing (PT) Program due to the external validation and value it provides. On an annual basis, CAC analyzes 10 to 12 proficiency samples that include a wide variety of fertilizer types. CAC's participation in the Magruder Program demonstrates capability and competency to perform reference analytical tests while also meeting ISO 17025 accreditation requirements. Further, CAC uses the established consensus values of past proficiency

samples for quality control and to evaluate instrument performance, new methodologies, and accuracy of standards. These past samples also are used to demonstrate staff competency for specific sample preparation procedures and analyses.

The Magruder Program has helped the CAC team to evaluate performance criteria of their test methods. As the agriculture industry continues to grow and evolve, manufacturers develop new and exciting fertilizing materials and ingredients. The Magruder Program can assist labs around the world with the standardization and quality control for these emerging materials. The program provides a tool for comparison of performance with peers in other states and commercial laboratories while helping identify measurement issues and staff training deficits. Participation in a nationally recognized proficiency testing program, such as the Magruder Program, aligns with the CAC's strategic direction and continuous improvement in its testing effectiveness.

- *Maryam Khosravifard*

GOALS OF THE INTERNATIONAL NETWORK ON FERTILIZER ANALYSIS (INFA)

The International Network on Fertilizer Analysis (INFA) is administered through the Food and Agriculture Organization (FAO) of the United Nations. The aim of INFA is to build and strengthen the capacity of laboratories that analyze fertilizers and to harmonize fertilizer quality standards to promote the efficient and sustainable use of fertilizers worldwide.

Although it is challenging to design and implement solutions to avoid over- and misuse of fertilizers, these efforts will reduce human impact on soil, atmosphere, water, and biodiversity. If excess nutrients reach these pools, extraction and removal is complex, expensive, or simply not feasible. At this point, preventive solutions are better alternatives to improve the knowledge of what, when and how we add amendments to the soil. Laboratory analyses are the first step in assessing the quality of fertilizers and amendments that have major impacts on soil quality. For this reason, INFA was formed in December of 2020 as a subnetwork of The Global Soil Laboratory Network (GLOSOLAN). INFA was developed to support GLOSOLAN and cover a similar scope of activities while replacing the focus on soil with fertilizers. Both networks are initiatives of the FAO Global Soil Partnership (GSP) to promote and support sustainable soil management from the point of view of soil and fertilizer analysis. INFA supports the implementation of the International Code of Conduct for the Sustainable Use and Management of Fertilizers, an instrument to provide a locally adaptable framework and a voluntary set of practices to serve the different stakeholders, who are directly or indirectly involved with fertilizers.

INFA has grown to include 159 laboratories with a presence in 84 countries. This network supports laboratories that are planning to or already performing fertilizer testing. The network met in June 2021 to identify and endorse three main objectives that cover technical, supportive, and legislative aspects of fertilizer activities. Each of these objectives has been assigned to a working group as described below.

Working Group 1 (WG1) covers the harmonization of fertilizer testing methodologies. The goal of WG1 is to produce comprehensive harmonized standard operating procedures (SOPs) for each of the different testing methodologies according to laboratory equipment and other available resources. The initial testing focus is on mineral/synthetic fertilizers for the analysis of nitrogen, phosphorus, and potassium (NPK). The INFA network recently has highlighted the importance of organic fertilizers, especially in the context of the global fertilizer shortage. Hence, organic fertilizers are included as a testing matrix within WG1. Over time, WG1 will expand its scope to include additional sample matrices and testing methods for additional parameters, such as micronutrients and heavy metals.

Working Group 2 (WG2) covers the capacity building and strengthening of existing and emerging fertilizer testing laboratories. WG2 initially is focused on fertilizer sample preparation and quality assurance for laboratory application. These topics will be developed as supporting documentation to guide laboratories on the best practices. A mid-term goal is to develop training materials in video or other media format to capture best practices, statistical analysis, and quality assurance procedures. Additionally, WG2 will develop videos to illustrate harmonized methodologies that comes from WG1. These forthcoming videos will provide a tool for transferring technical knowledge and observing best practices in action.

Working Group 3 (WG3) covers fertilizer regulation and legislation across the globe. Initially, the focus of WG3 is to gather and consolidate existing legislation from multiple countries on imports and exports as well as labeling, storage and safety. The proposed database will serve industry, academia, and commercial operations to identify the requirements for import and export, at regional and global levels. This will aid INFA in preparing more laboratories to participate in proficiency testing, which is a long-term goal. Knowledge of import requirements for each country is critical to increasing the successful distribution of proficiency testing samples to laboratories that currently experience challenges with the receipt of international shipments.

INFA was established to support all fertilizer stakeholders: industry, academia, laboratories, users, and outreach specialists through its vast network and access to the resources of the FAO/GSP. The network is still in its 'start-up' phase and is beginning to progress and develop to achieve outcomes relating to its defined objectives. The network is flexible and will be able to adapt to meet the needs and changes of the fertilizer industry over time.

For more information about INFA:

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https://www.fao.org/global-soil-partnership/infa/en/

- *INFA*

VENDOR'S CORNER

Every step in the analytical process is important, from obtaining a representative sample in the field to the final analysis in lab. Everything you do can make or break your data. Every step you perform can add uncertainty and contamination to your samples. Controlling contamination in your laboratory and process is an important step in controlling the quality of your data.

In the metals laboratory, sample handling, from balance to analysis, is key in obtaining quality data. Part of that process includes digesting the samples for analysis by ICP. Acid digestion of samples in a closed vessel microwave system provides reproducible digestion conditions from batch to batch and reduces the likelihood of contamination of samples and method blanks.

Contamination issues and vessel cleaning techniques are discussed in a video featuring CEM Applications Chemist, Sam Heckle. This presentation takes a look at sources of contamination, gives tips for identifying and avoiding them.

> Achieving Low Detection Limits Through Clean Chemistry: https://cem.sharefile.com/d-s2871663bdd6248cebf9d50a935691b51

After the samples have been digested and quantitatively transferred for final dilution, how do you clean the vessels before the next use? How do you ensure there is no carryover of analyte? There are four basic techniques for vessel cleaning. Which one you use depends on your sample matrix, the analytes of interest, and the concentration of analyte (ex., ppt or %).

> Option #1: Deionized Water Rinse - https://cem.com/en/cleaning-vessels-di-water-rinse-mars-6
>
> Option #2: Detergent Wash Followed by Deionized Water Rinse - https://cem.com/en/cleaning-vessels-detergent-mars-6
>
> Option #3: Soak Teflon Vessel Liners in Dilute Acid - https://cem.com/en/cleaning-vessels-acid-soak-mars-6
>
> Option #4: Hot Acid Clean (sealed vessel heated in the microwave) - https://cem.com/en/cleaning-vessels-microwave-clean-mars-6

The best gauge for determining the appropriate cleaning method is to monitor your method blanks. If your blanks are not low enough, then the cleaning technique needs to be more aggressive.

For more tips and tricks on general microwave care and use, sample prep, and vessel care, check out https://cem.com/en/in-the-lab or contact our Applications Team at Elemental.Support@cem.com or 800-726-3331.

- *Courtesy of Leanne Anderson and Elaine Hasty, CEM Corporation*

We introduced Vendor's Corner in the Winter 2022 edition as a place to discover training opportunities and other resources that could be helpful to your laboratory team. Many thanks go to CEM Corporation for contributing to this edition. Ideas for future articles are welcomed.

- *Sally Flowers*

MAGRUDER COMMITTEE ROSTER

Please join us in welcoming two new members to the Magruder Committee.

Wendy Zellner is an Associate Professor at The University of Toledo where she also holds an Adjunct Professor position in Clinical Molecular Biology. She has conducted research with silicon and molecular biology in agroecosystems. Her microbiological expertise is of value for evaluating organic fertilizers that have been increasing in the market.

Mélanie Titley is the Section Head of the Feed and Fertilizer Chemistry laboratory of the Canadian Food Inspection Agency. She has experience in method development and validation for mycotoxins, veterinary drug residues and guarantees in animal feed. She is a member of the Association of American Feed Control Officials (AAFCO) Laboratory Methods and Services Committee and the International Cooperation for the Convergence of Technical Requirements for the Assessment of Feed Ingredients (ICCF Feed) Working Group on Analytical Methods.

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
Matt Pearson, Treasurer	Office of Indiana State Chemist, West Lafayette, IN
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	N-P-K Consulting, LLC
Wendy Zellner	The University of Toledo, Toledo, OH
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
Mélanie Titley	Canadian Food Inspection Agency
Maryam Khosravifard	California Department of Food and Agriculture
vacant	vacant
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

UPCOMING MEETINGS:

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

2023 AAPFCO Winter Meeting (Santa Fe, NM)
Visit https://www.aapfco.org/meetings.html

2023 AAPFCO Summer Annual Meeting (August 3-4, 2023; Baltimore, MD)
Visit https://www.aapfco.org/meetings.html

2024 AAPFCO Summer Annual Meeting (August 5-6, 2024; San Antonio, TX)
Visit https://www.aapfco.org/meetings.html

Association of American Plant Food Control Officials
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