

Magruder Newsletter April 2014

Continued dialog with Laboratories in the Magruder Check Sample Program

1. Investigational Allowance Study by James Bartos (IN) and Patricia Lucas (FL) – both have been reviewing data from the Magruder Check Sample Program for trace nutrients over the past years. A review of data and action recommendations from the IA Investigators Committee will be in the future. Listed below are the preliminary findings:

What's good?

- B – (acid-soluble) spectrophotometric and “other” - all labs
- Cu – AAS, all labs
- Mg – AAS, all labs
- Mn – AAS, 972.02a, all labs
- Mo – AAS, all labs

What's needs minor improvement?

- Cu – ICP, regulatory labs
- Mn – ICP 972.02a, regulatory labs
- Mo – ICP, all labs
- S – gravimetric (some labs appear to have a method reporting issue)

What needs moderate improvement?

- Fe – AAS, all labs
- Mg (> 3%) – ICP, all labs
- Zn (< 10%) – ICP, regulatory labs

What needs major improvement or adjustment?

- Ca – AAS, all labs
- Ca – ICP, all labs
- Fe – ICP, all labs
- Mn – ICP 972.02a, non-regulatory labs
- Mn – ICP 972.02b, all labs
- S – “other,” all labs
- Zn – AAS, all labs
- Zn (>10%)– ICP, all labs

2. Latest ICP work – explanation for low DAP and other concentrates is forthcoming with recommendations.
3. News:
 - a. The May-June 2014 Journal of the AOAC will feature eleven (11) articles on fertilizer and will be dedicated to Bill Hall of Mosaic.
 - b. A video of Sepor sample splitters in action preparing Magruder Check Samples.
<http://www.youtube.com/watch?v=wXldJOSqTu4>

- c. Videos are being created by AAPFCO and TFI on proper techniques for preparation of samples and ICP analytical work. The videos are free to watch and learn from the ICP manufacturers and experts regarding the techniques for proper analytical work.

4. What are Investigational Allowances found on pages 44-46 in the No. 67 volume of the Official Handbook of AAPFCO? Investigational Allowances (IA's) were determined in the 1960's by Dr. Quackenbush and others (JAOAC 1966, Vol 49, No 5, pp 915 - 943) using a study with approximately 40 labs for NPK ranging from 0 to 32%. The laboratories were a mixture of State and Industrial Laboratories with equal numbers from each. All methods were official methods of AOAC (Association of Agricultural Chemists) in computing the IA's which include the sampling, inter & intra lab results variances. The IA is square root of the sum of the three variances times 2.33 from 4-32% NPK. In 2012 the IA's were changed to reflect higher concentrations from 40 to 60%. Potash at 60-62% has been changed from 1.44 to 1.8% and DAP and MAP phosphate have been changed from 0.7% AP to 1.0 – 1.1%

How do you interpret your laboratory results in comparison to the IA's? Let's take the check sample 201308B a concentrate Zn product for review. The average value with 39 labs participating was 39.57 +/- 3.28%. Reported values had a wide range from 30.8 to 45.7%. If Lab X reported a value of 32.70 %, they received a grade of "C" and yet this lab was below the average of 39.57% by 6.87%. The IA for Zn in the AAPFCO Handbook is 0.005% + 10% of the guarantee which is 4.16%, but the maximum allowed is 1%. By Lab X's value, a penalty would be levied on the manufacturer for being low. The Horwitz 2 standard deviation calculated is 1.8% for this value of 39.57%. So any lab outside of 37.77 to 41.37 is outside of the 2 STD by Horwitz. Sixteen (16) labs were outside this window and still may have received a "C" grade.

Another example would be the check sample 201402 average for total nitrogen of 17.76 +/- 0.26 with 68 laboratories participating. Lab Y analyzed the sample at 18.05% and was 0.29% high and received a grade of "C". The IA for N at 17.76% is 0.70% with Lab Y within the IA. Horwitz 2 standard deviation for this product is 0.93%. There were only two (2) laboratories outside of the IA range of 17.06 – 18.46% among the 68 laboratories analyzing this product.

Two different contrasting results for a grade of "C" are given for your review. Your comments and questions are welcomed.

5. Update of coming events:
 - a. IFA meeting will be held in Florida on April 15th and 16th at Mosaic.
 - b. Association of Southern Control Officials meeting in Montgomery, AL June 17-20, 2014
 - c. AAPFCO meeting in Sacramento, CA July 29-31, 2014
6. Magruder Check Samples for Sale – **Fire Sale** – any date beyond five years reduced to ½ prices for a limited time. Place order with Jamey Johnson of AAPFCO.
7. Free or reduced rate for Magruder Check Sample Program - Your laboratory invites another lab to join the check sample program and when they join your fee will be reduced to ½ price – if you sign up two new members to the check program your lab will have one free year of service.

8. Reference Materials for fertilizer laboratories
 - a. LQCRM MAP 11-52-0 (Caution - moisture loss or gain may occur during handling)
 - b. LQCRM MAP (10-40-0-10-1) with sulfur and zinc – (Caution - moisture loss or gain may occur during handling) Certificate reported on the dry basis. Recommend to use as is and check moisture level.
 - c. Magruder reagent grade MAP 12-61-0 (in progress)
 - d. NIST 120c and 694 – Phosphate Rock
 - e. NIST 194 and 194a – reagent grade MAP (194a may be available by April 2014)
 - f. NIST 193 – potassium nitrate
 - g. SRM 695 – mixed fertilizer with minor and trace elements
 - h. NIST 200b – potassium phosphate (do not use for ICP)
 - i. SRM 2141 – Urea
 - j. SRM 999b – Potassium Chloride
 - k. Aldrich 99.99% pure materials, KCl, NH_4NO_3 , MAP & others
9. Questions about the Magruder Check Sample Program:
 - Would you participate in electronic/on line data reporting?
 - Any methods you would like to see added to the code list?
 - Would it be of value to you to have certified homogeneous Magruder samples? (FYI we believe they are, but doing the work to verify this on every sample would be an extra cost)
 - Any blends or new materials you would like to see added to the program?
10. The Newsletter will print any testimonies on how you use the samples and data to manage your quality systems in your lab. So, let us hear from you with your testimony.
11. Nutrient of the Month - Sulfur

Sulfur (S)

- Essential plant food for production of protein.
- Promotes activity and development of enzymes and vitamins.
- Helps in chlorophyll formation.
- Improves root growth and seed production.
- Helps with vigorous plant growth and resistance to cold.
- Sulfur may be supplied to the soil from rainwater.
- It is also added to some fertilizers.
- The use of gypsum also increases soil sulfur levels.

Taken from the Agricultural Dept. of North Carolina

Sulfur comes in many forms such as sulfate, thiosulfate, organically bound sulfur and elemental sulfur. When analyzing these different sulfur material and reporting result to the Magruder Check Sample Program, due to the multiple codes (which are under revision) some labs have been reporting in the total sulfur section. When multiple sulfur forms are present in the sample, and labs report total sulfur by methods that

measure only sulfate and/or thiosulfate and do not analyze elemental sulfur, the results are skewed. There are several methods for analyzing elemental sulfur such as bromine digestion followed by the gravimetric or ICP method. Carbon disulfide also measures elemental sulfur by dissolution, but does not measure sulfate, thiosulfate or organic sulfur. Combustion is a method that can measure all forms of sulfur. The ICP cannot measure acid-insoluble elemental sulfur unless it has been converted into the sulfate form. Example: A sample of 10-40-0-10-1 (2009-04) was a mixture of 5% sulfate sulfur and 5% elemental sulfur. Results were reported with 27 labs reporting total sulfur near 10% and 14 labs analyzing only sulfate sulfur and reporting 5% in the column as total sulfur. This reporting of Sulfur with any improper code resulted in an unusual mean and standard deviation. Changing the codes in the very near future will help with this problem.