



## Magruder Newsletter June 2013

Continued dialog with Laboratories in the Magruder Check Sample Program

1. Magruder Check Sample 2012-5A and 5B included two samples - UAN 32 and Potash. The UAN sample (A) average nitrogen values was 32.02% with 0.53% as 1 STD. AAPFCO's IA is 0.88%, which suggests that 1 STD should be  $\leq 0.44$ . One lab was above the IA value and nine labs were below, which would result in a penalty for 14.5% of the labs. Most labs (50/69) analyzed N by the combustion method with an average of 32.05% with 1 STD at 0.38%. For the potash sample (B), average values were 61.69% +/- 1.45 as the standard deviation. Five labs were above the IA of 1.80% and 14 labs were below the IA, which would result in a penalty for 25% of the labs reporting. The theoretical value of pure KCl is **63.14% K<sub>2</sub>O**. Fourteen labs found values above theoretical for this mined product.

2. Data in Table Form – taken from Recent Magruder Check Samples

Product	Sample	Element	Average +/- 1 STD	Est. IA from 1 STD	Current IA
KCl	2012-09A	K <sub>2</sub> O	60.67 +/- 1.35	<b>2.70</b>	1.80
ZnSO <sub>4</sub>	2012-09B	Zn	36.20 +/- 2.56	<b>5.12</b>	1.00
		S	17.16 +/- 0.56	<b>1.12</b>	1.00
GTSP	2012-11	Total P <sub>2</sub> O <sub>5</sub>	46.62 +/- 0.86	<b>1.72</b>	1.53
		Indirect P <sub>2</sub> O <sub>5</sub>	42.44 +/- 0.54	1.08	1.53
		Direct P <sub>2</sub> O <sub>5</sub>	43.11 +/- 2.34	<b>4.68</b>	1.53
MAP-S	2013-01	Total P <sub>2</sub> O <sub>5</sub>	40.85 +/- 0.58	<b>1.16</b>	0.90
		Indirect P <sub>2</sub> O <sub>5</sub>	39.99 +/- 0.93	<b>1.86</b>	0.90
		Direct P <sub>2</sub> O <sub>5</sub>	40.22 +/- 0.55	<b>1.10</b>	0.90
MAP	2013-03	Total P <sub>2</sub> O <sub>5</sub>	51.61 +/- 0.75	<b>1.50</b>	1.05
		Indirect P <sub>2</sub> O <sub>5</sub>	50.86 +/- 1.08	<b>2.16</b>	1.05
		Direct P <sub>2</sub> O <sub>5</sub>	50.82 +/- 1.01	<b>2.02</b>	1.05

AAPFCO IA is approximately 2 STD – **Red values over current IA**

3. If you have any comments about the Newsletter, contact James Bartos at [jbartos@purdue.edu](mailto:jbartos@purdue.edu) or any member of the Magruder Committee. Members are listed on the bottom of the first page of the website. <http://www.magruderchecksample.org/>
4. NIST SRM 695 is a mixed fertilizer (13.9-16.5-11.65) product with micronutrients and trace metals. When using this material, it may not be obvious about the micronutrients and trace metals listed on the document from NIST. A comparison of the “acid-soluble” trace metals vs. total metals are listed below:

<b>Element</b>	<b>*Average, 1 STD</b>	<b>Range of Values</b>	<b>NIST Total Value**</b>
As	193 +/- 19	171 – 235	200 +/- 5
Cd	16.1 +/- 2.9	12.4 – 23.2	16.9 +/- 0.2
Co	47.5 +/- 12.3	27.4 – 65.7	65.3 +/- 2.4
Cr	174 +/- 19	136 – 192	244 +/- 6
Mo	14.0 +/- 2.0	10.2 – 16.8	20.0 +/- 0.3
Ni	112 +/- 15	85 – 131	135 +/- 2
Pb	257 +/- 15	231 - 281	273 +/- 17

Results average\* was taken from 14 laboratories using microwave nitric acid digestion compared to NIST results\*\* using other techniques which includes total metals. See website for more explanation of methods used. [https://www-s.nist.gov/srmors/view\\_detail.cfm?srm=695](https://www-s.nist.gov/srmors/view_detail.cfm?srm=695)

5. Future Check Samples. Past Magruder samples, especially for the high concentrates such as DAP, MAP, GTSP, KCl, Zn Oxy Sulfate, have shown higher standard deviations that are often above the AAPFCO recommended IA’s, as seen in Item 2 above. Sample 2013-05 is a pure reagent grade MAP, which should help with future samples of high concentrates when compared with other MAP fertilizer samples. After ISO certification of this sample, it will be on sale by the Magruder Committee – watch website. <http://www.magruderchecksample.org/>. Future schedule for 2013-2014  
<http://www.magruderchecksample.org/SampleSchedule20132014WOR.pdf>

Recommendations for analyzing high concentrates are as follows:

- a. Use a known reference material for phosphate when analyzing an unknown such as Magruder 2013-05, NIST 194, NIST 200b, and ISO certified MAP by SGS or use a previous check sample with known values.
- b. Follow approved analytical methods when analyzing these products. Any deviation(s) from the method should be validated by multiple labs before implementation.
- c. For direct available phosphate (AP), the ammonium citrate used by the method needs to be destroyed before analyzing. After digesting the sample in EDTA and ammonium citrate for one hour at 65°C the AOAC method is unclear about the next step. Should gravimetric, colorimetric or ICP be used after the sample has been brought to volume, shaken and let

settle then taking an aliquot out of the flask? Once the aliquot is taken the sample should be acidified before determine the analytical results.

- d. Pure KCl is 63.15%  $K_2O$  – the highest value it can be. Most commercial products are between 60 and 62%. Use a previous check sample such as 2012-09B, 2012-05B and 2011-03B as a reference material. The official AOAC procedure for Zn (965.09) is by using HCl only with Atomic Absorption. Currently there is no approved Zn standard commercially available in the 36% range. Using a reference from a previous Magruder check sample, such as 2011-02B, or a pure reagent material of comparable value is recommended.
  - e. Sulfur – currently there is no sulfur standard, but one is being developed by SGS as an ISO certified standard with approximately 5% elemental S and 5% sulfate sulfur, for a total of 10%. Combustion appears to be the best method for analyzing total sulfur, and if differences are required for elemental sulfur and sulfate sulfur, in a weak HCl digest solution, the ICP will only measure sulfate sulfur.
  - f. GTSP – recommend analyzing on a certain week (sample when submitted should state analyzing a certain week) – if you wait until the last minute to complete the data – the product continues to react and the %AP will increase.
6. LQCRM MAP 11-52 by SGS is a reference material analyzing 52.78% total phosphate (as  $P_2O_5$ ) and 52.69% available phosphate on an as-is basis. The Nitrogen value is 11.16% as is, with other values of metals listed. The samples contains 2.25% moisture (as is), but dry basis values are reported as most standards. Caution in drying material – sample should be dried by AFPC XI-2, or AOAC 965.08B. This is an excellent ISO reference material at a reasonable cost. [Go to SGS website to purchase.](#)
7. Meeting of Interest for Fertilizer Chemist:
- a. [ASFFPCO meeting in Wilmington, NC from June 18 – 21<sup>st</sup>, 2013](#)
  - b. [ISO meeting in Washington, DC August 5-8<sup>th</sup>, 2013.](#)
  - c. [AAPFCO meeting in St. Petersburg, FL August 14-16<sup>th</sup>, 2013](#)
  - d. [AOACI meeting in Chicago, IL August 25-29<sup>th</sup>, 2013.](#)