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# AFFPCO Summer Meeting 2018
# Laboratory Services Committee

Sanford Siegel

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# Limitations Observed with Organic Sulfur Compounds by AOAC 980.02 and Alternatives



## Agenda

Our Product

Challenges

Solutions

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# ANUVIA Plant Nutrients

## Our Product

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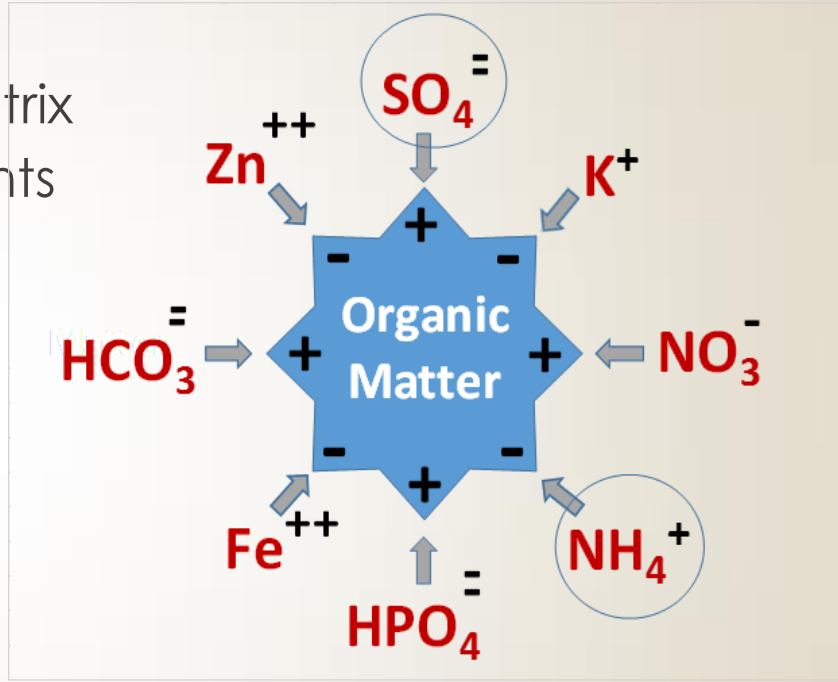
# Enhanced Organic Complex Fertilizer

- A Novel Slow Release Product
- Product utilizes an Organic Matrix
  - To Bind and Release Nutrients
  - A mechanism perfected in nature
- Homogenous Multi-Nutrient Product
- Can utilize organic resources available from a variety of sources




***A Progressive Way to Deliver Nutrition to Plants***

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# The Challenge

- Monthly composite sample analyzed by Anuvia and independent commercial Lab differed by 1-2 wt %, with low bias. Regulatory Laboratory performing standard methods of analysis for sulfur were reporting low determinations as well.
- Anuvia standard sample preparations: 1 gram of sample in a 250 ml volumetric flask, digest @65 C, 100 ml DI water, 5 ml 37% HCL, minimum 45 minutes in hot water shaker bath.
- Analysis by ICP, using matrix adjusted standards, scandium internal standard, ionization buffer included in the internal standard.

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# The Scope of the Challenge
## Magruder Check Sample Program
## (Anuvia value, 19.7 % sulfur)

| May 2017 Magruder Results for Sulfur ( 20% Nutrient Guarantee) | | | | |
|---|---|---|---|---|
| Method | # of Labs | range | robust mean | robust SD |
| Total sulfur , HCl soluble, gravimetric sulfate | 5 | 17.23-19.70 | 18.87 | 0.9526 |
| Total sulfur, combustion | 6 | 18.40-20.24 | 19.13 | 0.8525 |
| Total sulfur , HCl soluble, gravimetric, sulfate and elemental | 13 | 18.32-20.30 | 19.35 | 0.6904 |
| Total sulfur, ICP | 11 | 18.71-20.17 | 19.03 | 0.8235 |
| total sulfur, other | 11 | 18.19-27.17 | 18.89 | 1.277 |

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# Meeting the Challenge:

Proposed Anuvia Strategy for Resolution of Sulfur Analysis

Collaborate with Thornton Laboratory to determine whether Anuvia method was flawed or there was a low bias from standard method, AOAC 980.02(a)

Define corrective action for root cause of bias, whether internal or external.

Develop a Standard Reference Material(SRM) to distribute on request to any laboratory interested in analyzing the Anuvia complex organic nutrient products.

Confirm the conclusions of Thornton Laboratories with AAPFCO, using the SRM as a keystone for comparison.

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# Thornton Laboratories Recommended Sample Preparation for Sulfur Determination[1] by Gravimetric Method AOAC 980.02(a)

1. After appropriate sample preparation, weigh 1 g of sample (to 0.1 mg accuracy) into a 250ml beaker,
2. Add 125 ml DI water + 30 ml 1:1 HCl, bring to a boil on hotplate, and let simmer for 30 mins.
3. Remove from heat after desired time, cool and quantitatively transfer with filtering (using warm water) to a 250 ml vol. flask.
4. Make to volume, shake and remove a suitable aliquot (into 250 ml beaker) for precipitation with BaCl2. Adjust volume in beaker to 200 ml and warm on hotplate for minimum of 2 hours prior to filtering of precipitate.
5. Filter through a tared crucible (with suitable prepared filtering pad - < 4 um retention) washing precipitate with hot water (90 oC) at least 10 times.
6. Dry crucible & precipitate @ 250 C for minimum of 1 hour.
7. Remove from drying oven, cool, reweigh and calculate sulfur content.

[1]This sample preparation can also be used for Total Sulfur analysis on the ICP – Note, calibration standards will have to be matrix matched.

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# Request for Assistance from James Bartos, Office of the Indiana State Chemist, Chairman of the LSC, AAPFCO

Anuvia requested James to review the conclusions of Thornton Laboratory regarding the incomplete digestion using AOAC 980.02, the most common method of sulfur determination. James provided technical assistance as time permitted, and offered to be a referee lab, if needed during the interim.

James established his research goal as the evaluation of different digestion conditions on the determinations using ICP vs. gravimetric methods.

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| Methods Evaluated | total sulfur | | |
|---|---|---|---|
| | range | mean | n |
| ICP Sulfur, Microwave 9 ml nitric, 3 ml HCL | 19.46-19.75 | 19.61 | 4 |
| Modified AOAC 980.02 | | | |
| AOAC 980.02, vigorous boiling for 15 minutes | 19.86-20.72 | 20.29 | 4 |
| AOAC 980.02, Microwave 9 ml nitric, 3 ml HCL | 22.42-22.94 | 22.73 | 4 |
| AOAC 980.02, normaldigestion with addition of 30% H2O2 during digestion | 18.67-20.20 | 19.57 | 4 |
| AOAC 980.02, 1:1 addition of HCl@ 15 ml, brought to boil, +30% H2O2 over 20 minutes, volume to 200 ml, and boil | 20.07-20.25 | 20.16 | 4 |

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# Conclusions and Recommendations from James Bartos and Tim Byers

1. ICP Sulfur determination following mixed acid digestion (soon to be official, Dr. Sharon Webb).

2. 1:1 HCl : water addition @15 ml each, brought to a boil, then 3 additions of $H_2O_2$, then brought to volume of approx. 200 ml, proceed with BaSO4 ppt portion of AOAC 980.02.

3. Do not follow a mixed acid procedure, as that produced a high bias.

True value for total sulfur in the SRM is 19.6-20.2 %

The SRM is available from Anuvia Nutrients
Contact Sanford Siegel
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We at Anuvia wish to acknowledge the advice and guidance received from Thornton Laboratories and the support and technical contributions Office of Indiana State Chemist.

We wish to extend  our gratitude to Tim Byers, Hugh Rodrigues, and James Bartos for identifying the best sample preparations for complex matrices for sulfur analyses.

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