

Update--Validation for Nutritive and Nonnutritive Metals Method

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Background

- ❑ 2008 James led interlaboratory study
- ❑ Comparing Official AOAC methods with proposed method
- ❑ AOAC 965.09 for nutritive metals as basis
- ❑ AOAC 2006.03 for nonnutritive metals as basis
- ❑ 9 Magruder samples
 - Dynamic range in metals concentration
- ❑ Reagent Grade KCl
- ❑ 5 samples with more dynamic Fe range

Goals Inter-Laboratory Study

- ❑ Determine if universal method is viable for both nutrients and non-nutrients in fertilizer
- ❑ Evaluate if using mixed acids rather than one acid enhances recoveries

Conclusions of Study

- ❑ Study shows universal method for both nutritive and nonnutritive metals in fertilizer is viable
- ❑ Mixed acid digestions can enhance recovery of nutritive and nonnutritive metals in fertilizer, *versus Nitric alone*

Validation Materials

ID	2006.03	Description
A	4321/2025	Metal Fe oxysulfate
B	4031/5938	Magruder 2002-09B
C	5488/5890	Zinc Oxysulfate
D	2818/7669	Granulated mine waste
E	1615/2056	Metal oxysulfate
F	3313/6267	Western MAP
G	7999/3375	DAP from North African rock
H	6501/4812	NC MAP
I	7738/7418	China DAP
J	3917/8165	Magruder 2003-11
K	4459/8931	Magruder 2004-07
L	8873/9469	N-P-K lawn product blend
M	9886/9774	Organic biosolid
N	4626/8088	Organic mixed fertlizer + biosolid
O	6411/3401	Composted manure
P	3716/4606	Fe humate
Q	NA	NIST SRM 695
R	NA	Magruder 2009-06

Term Changes

❑ Nonnutritive Metals

❑ Nutritive Metals

❑ Group A

❑ Group B

❑ As, Cd, Cr, Co, Mo, Ni,
Pb, Se

❑ Ca, Cu, Fe, Mg, Mn, Zn

LOD and LOQ

Group A

	As, mg/L	Cd, mg/L	Cr, mg/L	Co, mg/L	Mo, mg/L	Ni, mg/L	Pb, mg/L	Se, mg/L
LOD	0.61	0.12	2.26	1.03	0.16	0.52	0.63	0.48
LOQ	2.03	0.39	7.55	3.43	0.53	1.74	2.09	1.60

Group B

	Ca, %	Cu, mg/L	Fe, %	Mg, %	Mn, %	Zn, %
LOQ	0.0045	0.49	0.0018	0.0015	0.00003	0.0009

Completed to Date

- ❑ Method has been published in Journal of AOAC Intl. May/June 2014
- ❑ Open Access is available
- ❑ FREE access
- ❑ Collaborative Study Packet has been submitted
- ❑ External Review Panel (ERP) Reviewed September 2015
- ❑ Received comments from reviewers and ERP

Comments Received

- ❑ Expansion of method for inclusion of “heavy” metals
 - ❑ Hg & Al not included
- ❑ Total versus plant available nutrients/nonnutrients
 - ❑ Acidic vs alkali digestion to mimic metals’ reactions acids vs. bases
 - ❑ Adjust pH of digestion to mimic pH of soils that most crops are grown (pH ~5.5-6.5)
 - ❑ Use “leachable metals test” to mimic metal plant availability
 - ❑ Inclusion of macro- and micronutrients is misleading for label guarantees and not user-friendly to the end user

Comments, cont'd

□ Statistician:

- Incorrect calculations
- Conclusions not reflect statistical parameter estimates
- Poor precision for As, Se, Pb ($H_{orRat} > 2$)
 - Results of 2006.03 not correct either
 - AOAC 2006.03 not best choice for As, Se, & Pb
- Units different for different elements (% vs ppm)
- No assessment of values (?) presented in paper
 - No assessment of values presented in 2006.03 paper
- J AOAC Int. 2007; 90: 844-56
 - ICP/MS of As, Cd, Hg, & Pb of foods by Nordic Committee on Food Analysis

Although I felt like this...



**SCIENCE
CAN DO IT!**



...we will move on

Next Steps

- Address ERP comments
- Address statistician comments
- Address reviewer's comments
- Proceed forward like planned

Questions or comments?

