

Method Validation for Nutritive and Nonnutritive Metals---Update

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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 fertilizer + 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

Conclusions and Next Steps

- ❑ Method has been published in Journal of AOAC Intl.
May/June 2014
- ❑ Open Access is available
- ❑ FREE access
- ❑ Collaborative Study Packet has been submitted

- ❑ We need YOUR HELP to move forward

HELP

- ❑ AOAC suggested their experts on various analytes to serve on the Expert Review Panel
- ❑ None had experience with fertilizer or fertilizer materials or any agriculture products
- ❑ We need your expertise!
- ❑ www.aoac.org
- ❑ “Call for Experts” tab
- ❑ “Call for Experts-AOAC Research Institute”

HELP

- ❑ “Metals”
- ❑ “Phosphorus & Potassium”
- ❑ “Total Sulfur”
- ❑ “Urea”

- ❑ “Please submit your information by clicking the link below”
- ❑ CV, summary of expertise, and references

Questions / Comments?