

ICP PK Updates

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Status

- Another year flew by
- Have an AOAC *First Action* method:
AOAC Official Method 2015.18
Phosphorus and Potassium
in Commercial Inorganic Fertilizers
Inductively Coupled Plasma–Optical Emission Spectrometry
First Action 2015
- Working on a revised Committee Draft (CD) for *ISO TC-134* with suggested modifications
 - *EU – separate extraction (citrate-EDTA) from determination (ICP-OES)?*
 - *Suggested modification for low-flow instruments – anyone using?*
- Found a cheaper source of commercial stock standards
 - *Big deal as standards need to be made up frequently (~ 3 wks)*
- Have 24 study samples selected, prepared, in vials and ready to ship; received request to add a few more samples
- Looking a bit into extraction conditions
 - Time: some labs test samples immediately, some wait until next day
 - Temp: deviations from 65C
 - Type: air bath vs. water bath

New Developments?

- Two states contacted me about ICP-PK method
 - both acquired new ICP's & switching to ICP-PK method
 - we are providing technical support on method
- Switched to commercial custom stock standards
 - Can still use $\text{NH}_4\text{H}_2\text{PO}_4$ and KCl salts, if prefer
- Trying a non-concentric nebulizer
 - Forgiving of suspended solids ($\leq 1\text{mm}$) without clogging, slightly higher RSD
 - OptiSolids nebulizer
 - TEXAS SCIENTIFIC PRODUCTS LLC, www.txscientific.com; tel. 888.268.6037; fax. 972.850.7659
- We (Indiana) acquired a new ICP dedicated to this analysis
 - *Different model, different design, different results*

New Standard Supplier

Exaxol Corporation

Phone 727-524-7732

www.exaxol.com

- P: custom standard stock number SP-6170
 - 2,000 mg/L P from $\text{NH}_4\text{H}_2\text{PO}_4$
- K: custom standard stock number SP-6202
 - 3,000 mg/L K from KCl
- Water matrix, preserved with biocide
 - *Alkylbenzyl ammonium chloride (little foamy)*
- ~ \$85 per liter
- One-year shelf-life
- Guaranteed, or will remake free
- Help reduce variability?

New ICP Lessons (Agilent 5100 SVDV)?

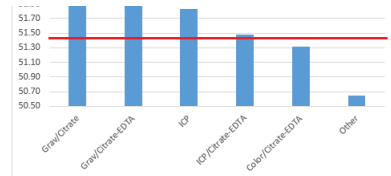
- Got low P/P₂O₅ results even with new method?
- Trying different injector types/diameters
 - 2.4 mm – high salts/solids
 - 1.8 mm – sometimes standard and/or recommended for organic matrices
 - 1.4 mm – semi-volatile organics
 - **Note: injecting less of a problem matrix and a high conc can help, but switch back to bigger injector for trace level analyses**
- With non-concentric nebulizer (and possibly others), may be “flooding” nebulizer with large volume of sample and IS into less efficient situation
 - Slowed pump speed during analysis
 - Increased neb pressure slightly (~10%)
- Looking into radial viewing height for both P and K
 - P prefers “hotter” zone, while K prefers “cooler” zone
- Currently using axial view for P and radial for K
 - Gives stronger signal for P (views entire plasma) and can set K to preferred viewing height in radial view
- Looking at baffled/double path options to decrease vol injected

Magruder Comparison – P₂O₅

Magruder 160911, MAP (P2O5)

Method	Description	Result	Stdev	# of Labs
41.10	Grav/Citrate	51.92	0.20	5
41.11	Grav/Citrate-EDTA	52.19	0.40	5
41.50	ICP	51.83	1.52	6
41.51	ICP/Citrate-EDTA	51.48	0.94	5
41.60	Color/Citrate-EDTA	51.31	0.75	14
41.99	Other	50.65	1.85	4

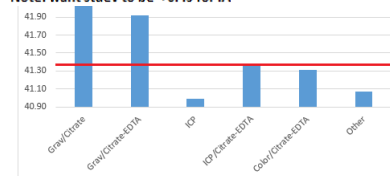
Note: want stdev to be < 0.5 for IA



Magruder 161211, MAP+S (P2O5)

Method	Description	Result	Stdev	# of Labs
41.10	Grav/Citrate	42.17	0.47	7
41.11	Grav/Citrate-EDTA	41.92	0.94	5
41.50	ICP	40.99	0.72	7
41.51	ICP/Citrate-EDTA	41.37	0.46	5
41.60	Color/Citrate-EDTA	41.31	0.80	14
41.99	Other	41.07	1.19	5

Note: want stdev to be < 0.45 for IA



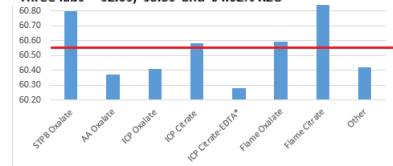
Magruder Comparison – K₂O

Magruder 151111, MoP (K₂O)

Method	Description	Result	Stdev	# of Labs
50.00	STPB Oxalate	60.80	0.74	11
50.30	AA Oxalate	60.37	0.94	7
50.50	ICP Oxalate	60.41	0.85	6
50.51	ICP Citrate	60.58	0.72	8
50.52	ICP Citrate-EDTA*	60.28	1.02	6
50.60	Flame Oxalate	60.59	0.19	3
50.61	Flame Citrate	61.00	3.12	4
50.99	Other	60.42	1.14	19

Note: want stdev to be < 0.9 for IA

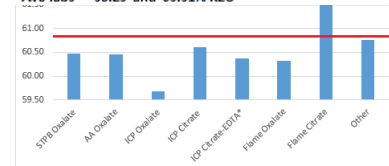
* Three labs = 52.03, 58.83 and 64.62% K₂O

Magruder 161011, MoP (K₂O)

Method	Description	Result	Stdev	# of Labs
50.00	STPB Oxalate	60.48	0.58	12
50.30	AA Oxalate	60.45	0.86	16
50.50	ICP Oxalate	59.68	0.49	8
50.51	ICP Citrate	60.61	1.10	4
50.52	ICP Citrate-EDTA*	60.37	1.63	7
50.60	Flame Oxalate	60.33	1.34	6
50.61	Flame Citrate	62.03	0.93	3
50.99	Other	60.76	1.58	28

Note: want stdev to be < 0.9 for IA

* Two labs = 58.23 and 63.31% K₂O



Summary

- Want to send out practice samples to collaborators now that have a few more options/suggestions for improving data
- Need to switch pump tubes and may have to switch nebulizer, injector and possibly introduction system for PK method vs. acid digestion method(s) for secondaries and micros
 - Note: with “quick-connects,” simple and easy in spite of complaining
- Accept that may not get accuracy and precision of gravimetric and STPB methods and/or that may need to raise IA, **but ICP PK is an increasing, unstoppable trend**
- We (Indiana) have received an additional support person, so we can hopefully recommit to existing and new projects
- Not opposed to other modifications, **if documented**
- If improvement with practice samples is obtained (expected result), then gauge when collaborators would like to conduct the study

Acknowledgements

- OISC fertilizer staff:
 - Tim Byers (NEW)
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