

Colorimetric P Speciation Analysis

Long Path Lengths and Model Compounds

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1. Important Issues in P Analysis in Waste Water

1 Low Levels:

regulatory requirements on TP potentially below detection using one cm light path

Can long path lengths help?

2 Speciation:

to optimize P removal need to understand dynamics of P speciation across treatment technologies

for example: DOP is resistant to traditional removal methods.
Can it be converted to other (more treatable) forms?

Does Standard Methods recover speciation of model compounds relevant to wastewater ?

2. Path length Study: Method

- 1 orthophosphate solutions
- 2 fixed concentrations to give absorbance of 0.4 for 10 cm or 1 m pathlengths
- 3 vary mixed reagent volume
- 4 vary colour development time
- 5 Standard Methods, 1998. *Standard methods for the examination of water and wastewater*, American Public Health Association, Washington, DC, USA, 20th ed.

3. Ascorbic Acid 10 cm

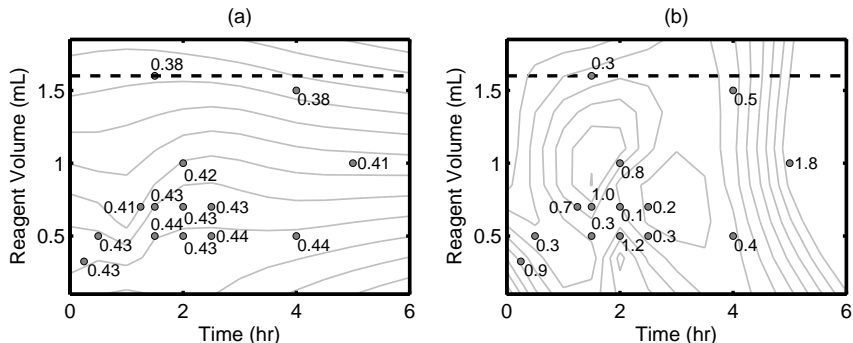


Figure: Contour plot of absorbance (a) and relative (%) standard deviation (b) by the ascorbic acid method with 10 cm light path. Dashed line corresponds to the volume used in Standard Methods.

4. Ascorbic Acid 1 m

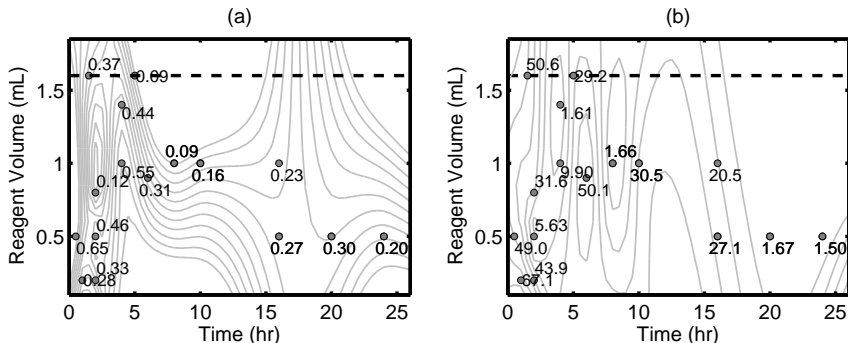


Figure: Contour plot of absorbance (a) and relative (%) standard deviation (b) by the ascorbic acid method with 1 m light path. Dashed line corresponds to the volume used in Standard Methods.

5. Kinetic slice. Ascorbic Acid. 1 m

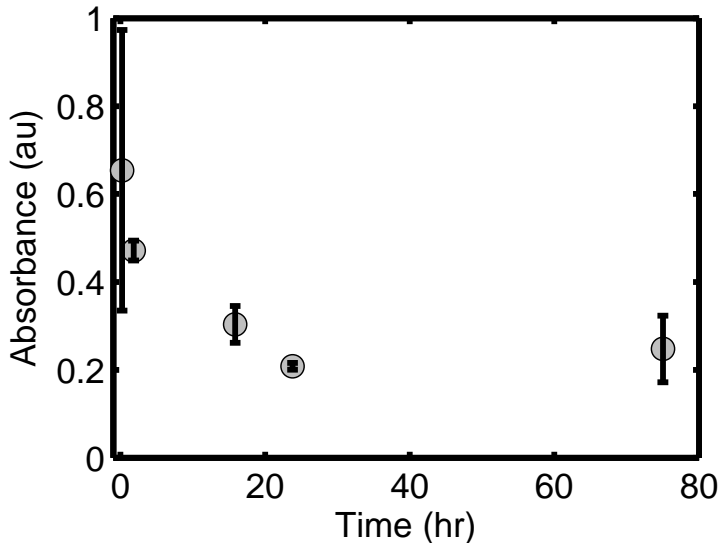


Figure: 8 μg P/L standard, 0.5 mL mixed reagent volume.

6. Path length Study: Summary

- 1 no surprise = long path lengths work fine
- 2 reduced colour forming reagents = modest 10% increase in sensitivity
- 3 1 m pathlength requires long colour development for reproducible signal
- 4 **suggests FIA would be useful !**

7. Matrix Effects?

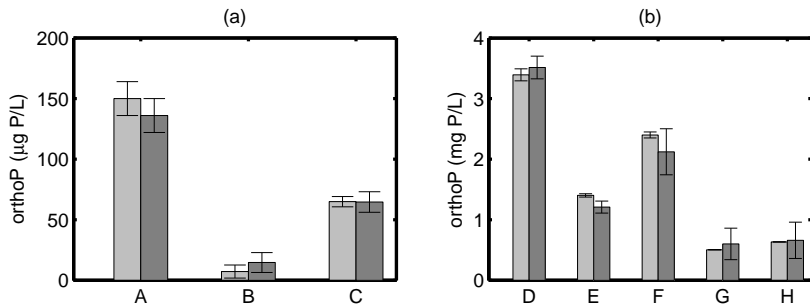


Figure: Orthophosphate determinations using external calibrations (light gray bars) and standard addition calibration (dark gray bars). A. Grand River water, B. laboratory tap water, C. final treated wastewater effluent. Subplot (b) correspond to 5 wastewater samples.

8. Anecdote for need to study speciation methods

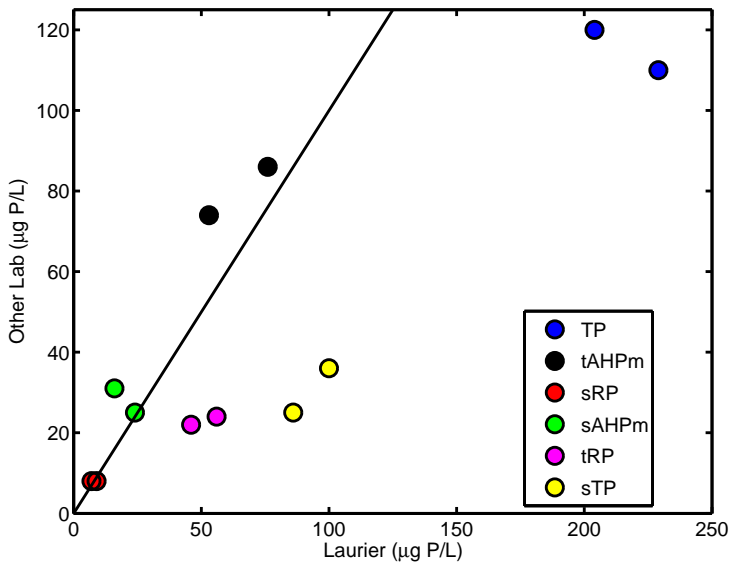
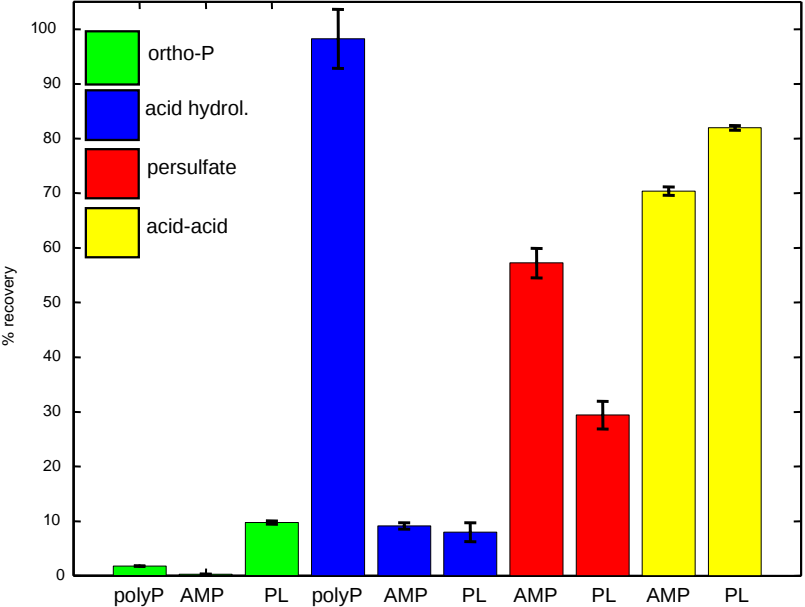


Figure: caption

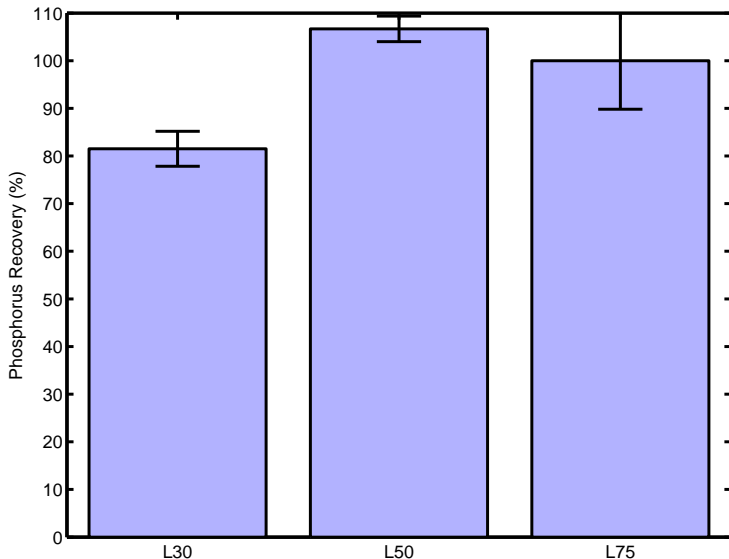
9. P Speciation Study: Method

- 1 Both labs = Standard Methods, 1998. *Standard methods for the examination of water and wastewater*, American Public Health Association, Washington, DC, USA, 20th ed.
- 2 **but different interpretations of Standard Methods!**
- 3 Ascorbic acid method
- 4 Various digestion methods (always orthophosphate analysis at the end)
- 5 polyP
- 6 AMP
- 7 phospholipid

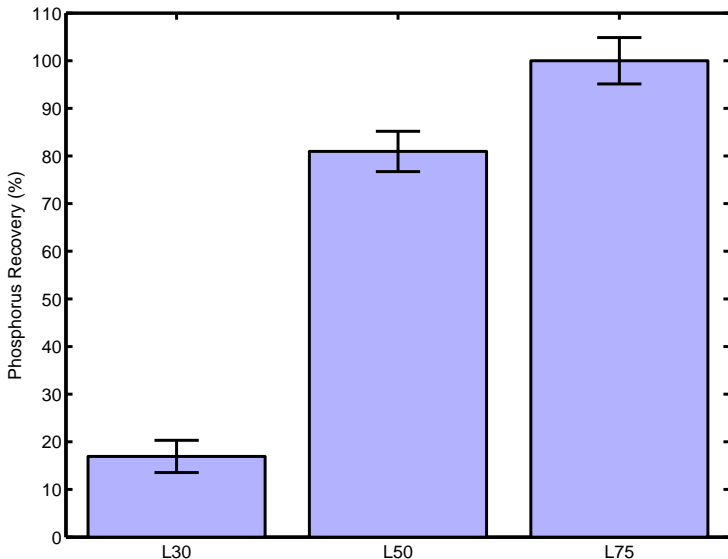
10. Recoveries



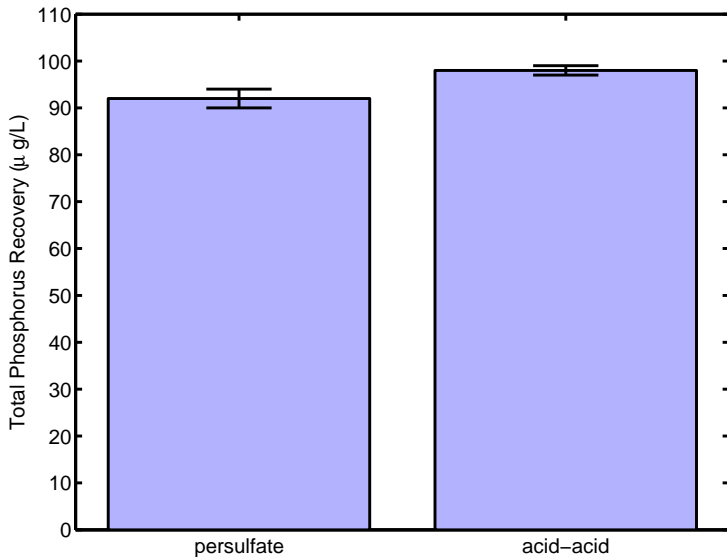
11. Persulfate Autoclave Program: AMP



12. Persulfate Autoclave Program: Phospholipid



13. Digestion Method and real waste water



14. P Speciation Study: Results

- 1 some model compounds are not recovered by typical methods
- 2 effect on TP results depends on actual speciation of sample
- 3 autoclave program needs optimization
- 4 **analysis of model P compounds is important area for future research**
- 5 **analysis of real waste water by different digestion methods is important**

15. P Acknowledgements

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