

Optimizing the Sensitivity of Particulate Silica Quantification by Molybdenum Blue Absorbance Spectrometry

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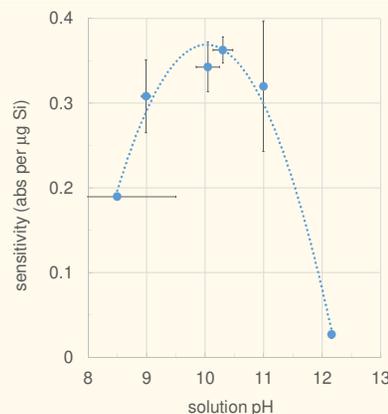
INTRODUCTION AND



FACTORS CONSIDERED TO INCREASE ANALYSIS

SENSITIVITY Initial pH of Silica Suspension

- The optimum initial pH (before addition of molybdate) for the dissolution of silica to form HPA is approx. pH 10
- This may be due to the balance between enhanced silica solubility at high pH and the optimal (low) pH of the reaction to form HPA
- As the acidic molybdate solution is added to the basic silica suspension to initiate the reaction, the initial solution pH quickly falls to approx. pH 1-2 for the formation of heteropoly blue.

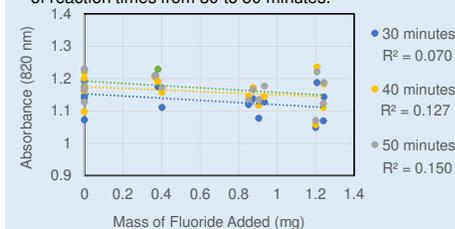


Ionic Strength Adjustment and Addition of EDTA

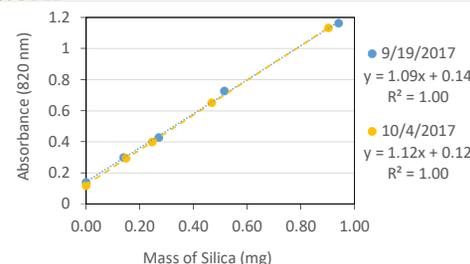
- It was initially believed that EDTA would increase analytical sensitivity due to how it chelates metals in solution, enhancing dissolution.
- After multiple trials were carried out, it was observed that the absorbance at 820 nm decreased as more EDTA that was added to solution.
- Ionic strength adjustment was also explored to see if higher ionic strength would increase silica dissolution.
- Sodium Chloride was added to silica solution to see if absorbance would increase. This additive also proved to be ineffective.

Addition of Fluoride

- Because hydrofluoric acid can dissolve silica, sodium fluoride was tested as an additive to increase analytical sensitivity.
- ~10 ppm F⁻ was added to the silica suspension along with the acidic molybdate reactant at a highly acidic pH of 1 – 2.
- After testing various amounts of fluoride in silica solution, it was observed that fluoride had no significant positive effect on absorbance. There may be a weak inhibiting effect for a range of reaction times from 30 to 50 minutes.



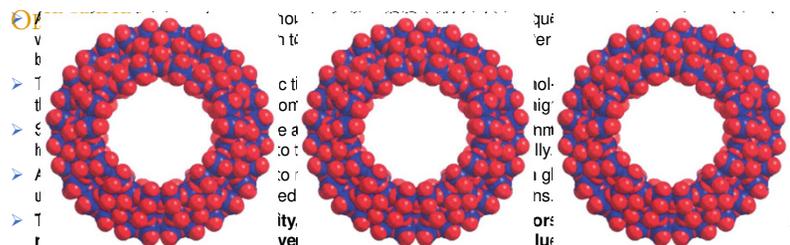
CONCLUSIONS AND FUTURE



Next steps...

- In collaboration with WiSys a provisional patent has been filed for an instrument design utilizing a chemically similar analysis.
- We have received an AR/WITAG grant to build a prototype of this instrument and evaluate its analysis sensitivity to the respirable fraction of airborne particulate silica.
- Contact Dr. Boulter if you are interested in working on this project.

METHOD AND



Works Cited

- [1] Department of Labor, Occupational Safety and Health Administration, *Occupational Exposure to Respirable Crystalline Silica*, vol. 78, 2013, pp. 56274-56504.
- [2] R. Key-Schwartz, P. Baron, D. Bartley, F. Rice and P. Schlecht, "Determination of Airborne Crystalline Silica," in *NIOSH Manual of Analytical Methods*, National Institute of Occupational Safety and Health, 2003, pp. 260-280.
- [3] T. Fujiwara, K. Kurahashi, T. Kumamaru and S. H., "Luminescence Chemiluminescence with Heteropoly Acids and its Application to the Determination of Arsenate, Germanate, Phosphate and Silicate by Ion Chromatography," *Applied Organometallic Chemistry*, vol. 10, pp. 675-681, 1996.
- [4] J. Woods and M. Mellon, "The Molybdenum Blue Reaction: A Spectroscopic Study," *Industrial and Engineering Chemistry*, vol. 13, no. 11, pp. 760-764, 1941.
- [5] D. Boltz and M. Mellon, "Determination of Phosphorous, Germanium, Silicon, and Arsenic by the Heteropoly Blue Method," *Analytical Chemistry*, vol. 19, no. 11, pp. 873-877, 1947.