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

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This research is supported by WiSys and UW System applied research funding programs (AR-WITAG) and the UW-Eau Claire Office of Research and Sponsored Programs differential tuition funds (OBS and F/ESC grants). The views expressed herein are those of the authors and are not necessarily those of WiSys, UW-Eau Claire, or UW System.

INTRODUCTION AND METHODS

**FACORS 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 pmol 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.

**METHOD AND RESULTS**

**Summary of Optimized Reaction Conditions and Conclusions**
- Analytical sensitivity is highest for aqueous suspensions of silica at pH 11 (previous work).
- Optimal reaction times for the analysis are: 15 minutes following addition of molybdate solution and 45 minutes after reductant is added (experimental studies not shown).
- There is a consistent, positive correlation between silica concentration in solution and visible absorbance at 820 nm by the compound heteropoly blue.
- We have optimized the reaction conditions to convert silica to heteropoly blue and maximize the analysis sensitivity of the procedure.

**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.

Works Cited