Ion Analysis

2011 IMPROVE Steering Committee Meeting
Frostburg, Maryland

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RTI International is a trade name of Research Triangle Institute.

NPS Ions Contract Awarded to RTI
March 2011



Ion Analysis for IMPROVE

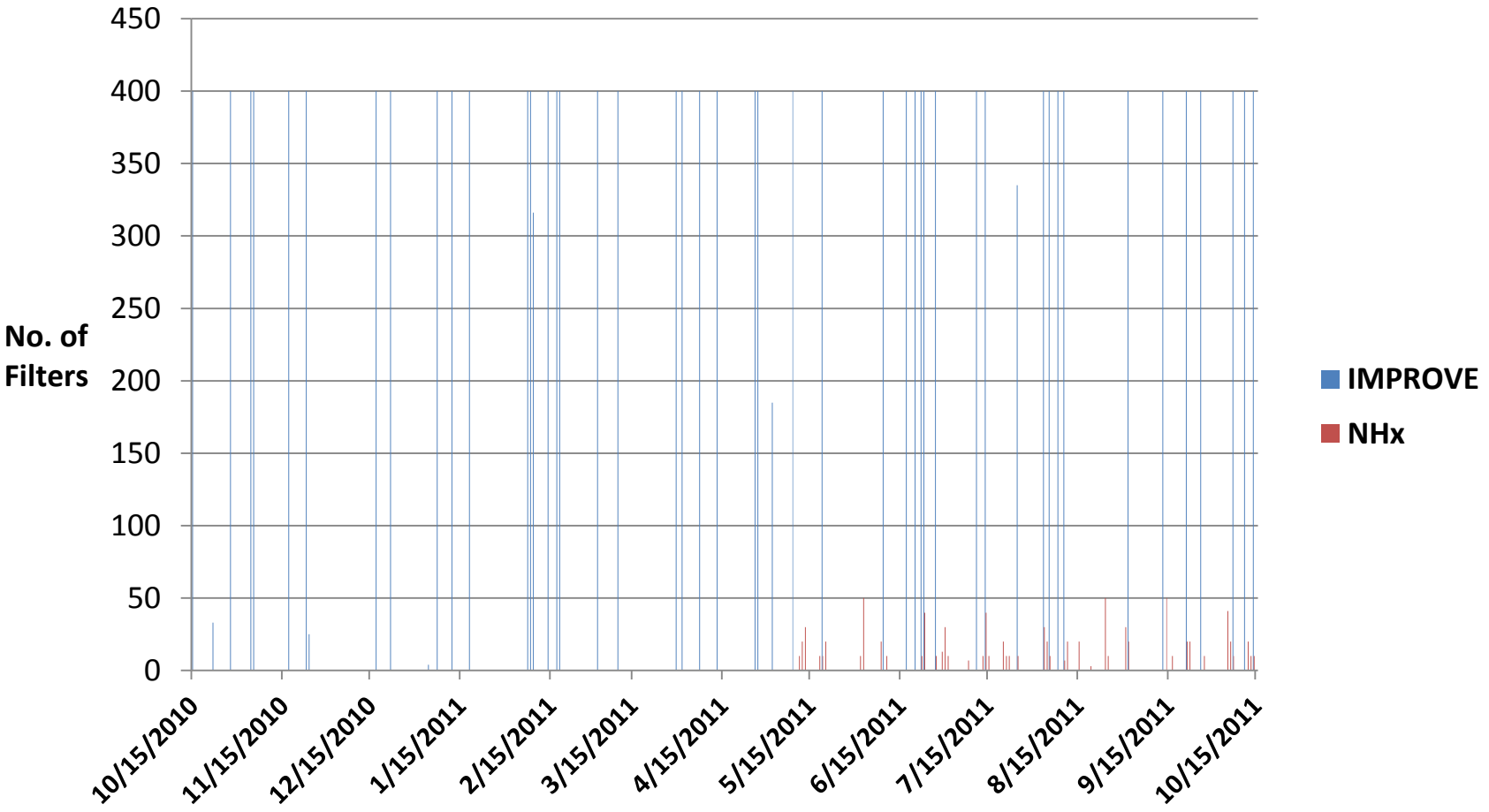
- Analysis and reporting of ion masses on nylon filters from the IMPROVE network.
- Preparation of impregnated filters for SO_2 sampling and analysis and reporting of sulfate ion mass on the exposed filters.
- Preparation of phosphorous acid-impregnated filters for inorganic reduced nitrogen (NH_x) and methylamine sampling and analysis, and reporting of NH_4^+ and methylamine mass on the exposed filters.
- Occasional studies related to ion analysis:

  Analysis of Teflon research filters prepared by UC-Davis for calibration of XRF systems.

Filters Received Since the Last Steering Committee Meeting

Filter Type/ Analytes	10/15/10 - 3/31/11	4/1/11- 10/15/11	Total
Nylon/Anions	8798	10920	19718
Phosphorous Acid-Coated Cellulose/NH_4^+ and MeA	--	861	861
Nylon/NH_4^+	--	380	380
Teflon/Na^+, Cl^-, NH_4^+, $SO_4^=$	--	56	56

Filters Received Since the Last Steering Committee Meeting



NH_x Study Update

2010 NH_X Study Review

- Ion analyses performed in 2010 revealed an unknown peak in some samples that precluded the quantitation of ammonium ion. The peak was identified as methylamine.
- Subsequent experiments performed at CSU determined that the observed methylamine was an artifact of the sampling, most likely through reaction of NH_4^++ formaldehyde.
- UCD prepared sample cartridges from a plastic that is non-reactive with phosphorus acid.

Analysis of NH_4^+ in Filter Extracts: Comparison of Methods

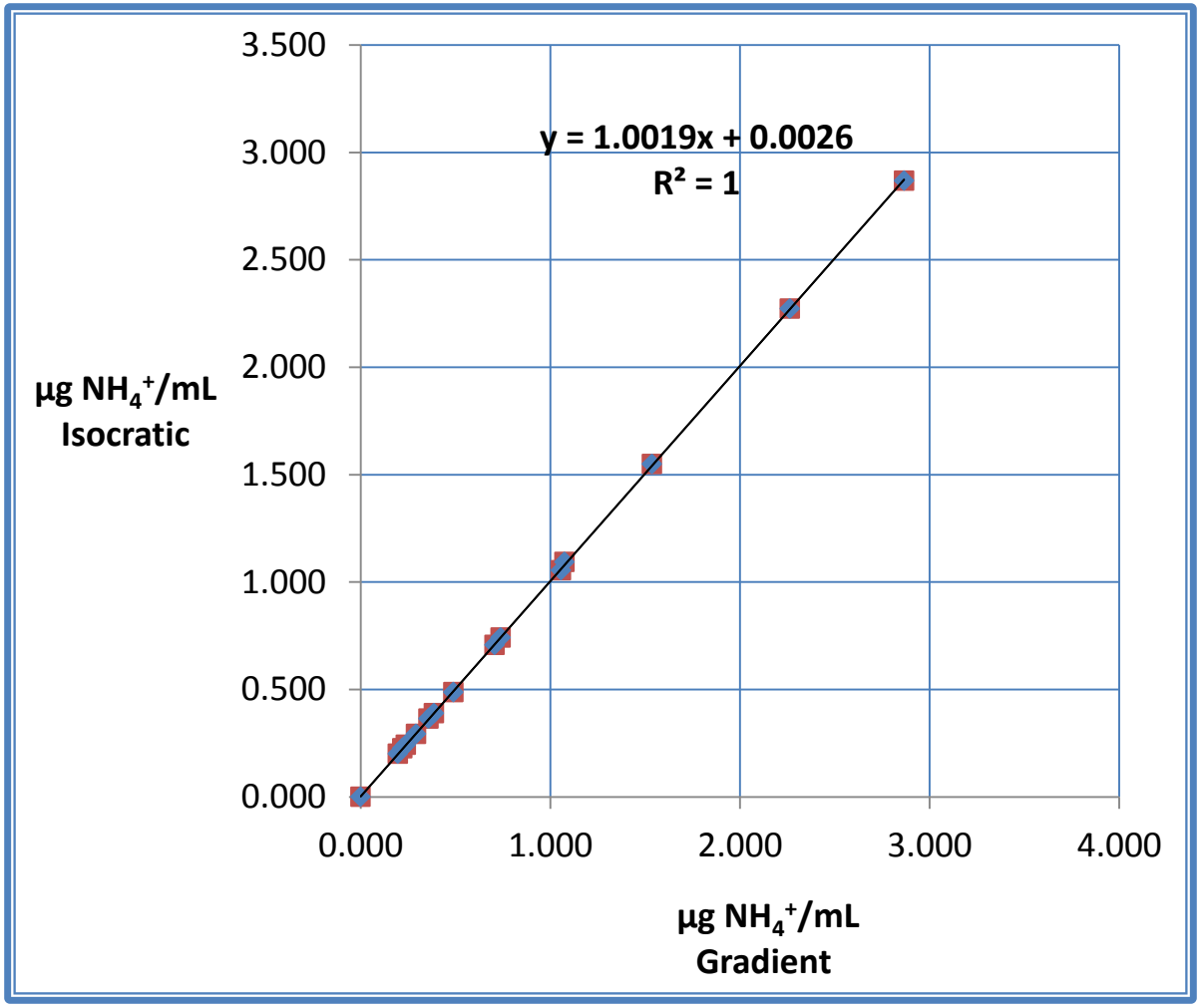
Gradient

- Dionex ICS-3000 IC (dedicated for NH_x)
- CS18 Cation Separator, 2 mm
- CSRS-300 Auto Regen Mode
- Eluent: 100 mM methanesulfonic acid (MSA); programmed with gradient from 0.5 to 8 mM
- Flow rate: 0.3 mL/min
- **Baseline separation of NH_4^+ and MeA**
- **Analysis time = 40 min**

Isocratic

- Dionex Ion Chromatograph (multiple)
- CS12A Cation Separator, 4 mm
- CSRS-300 Auto Regen Mode
- Eluent: 22mN sulfuric acid
- Flow rate: 1.0 mL/min
- **NH_4^+ and MeA are not separated**
- **Analysis time = 12 - 14 min**

Comparison of Isocratic vs. Gradient IC Methods for NH_4^+ (15 nylon filters)



NH_x Sampling Start Dates (H_3PO_3-impregnated cellulose filters)

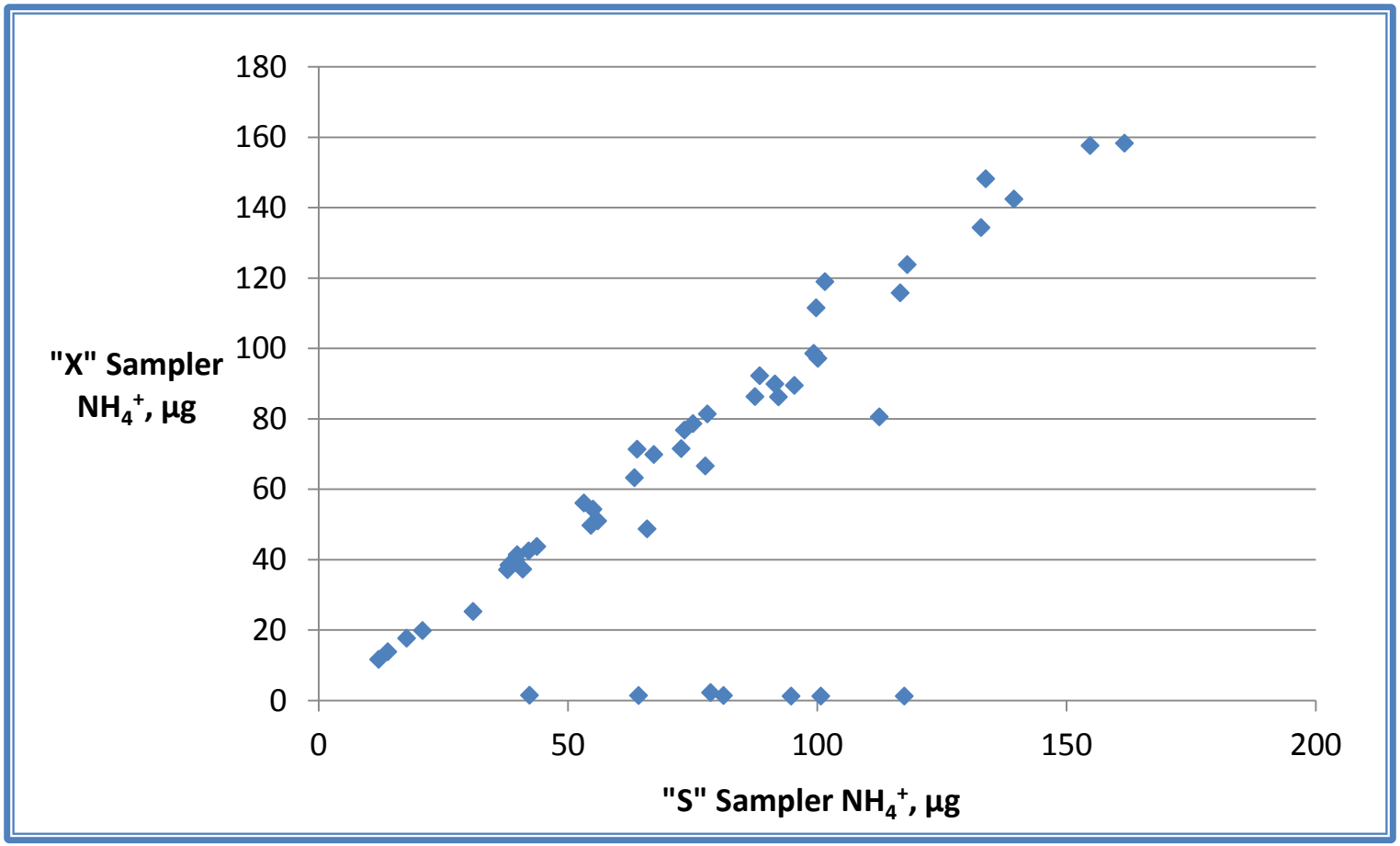
Site	Sampling Start Date
BANDS	4/27/11
BONDS*	4/21/11
CEBLS	4/27/11
CHIRS	4/27/11
GLACS	4/21/11
MEVES	4/27/11
ROMOS*	4/21/11
WICAS	4/21/11
YELLS	6/8/11

* Collocated samplers at these sites

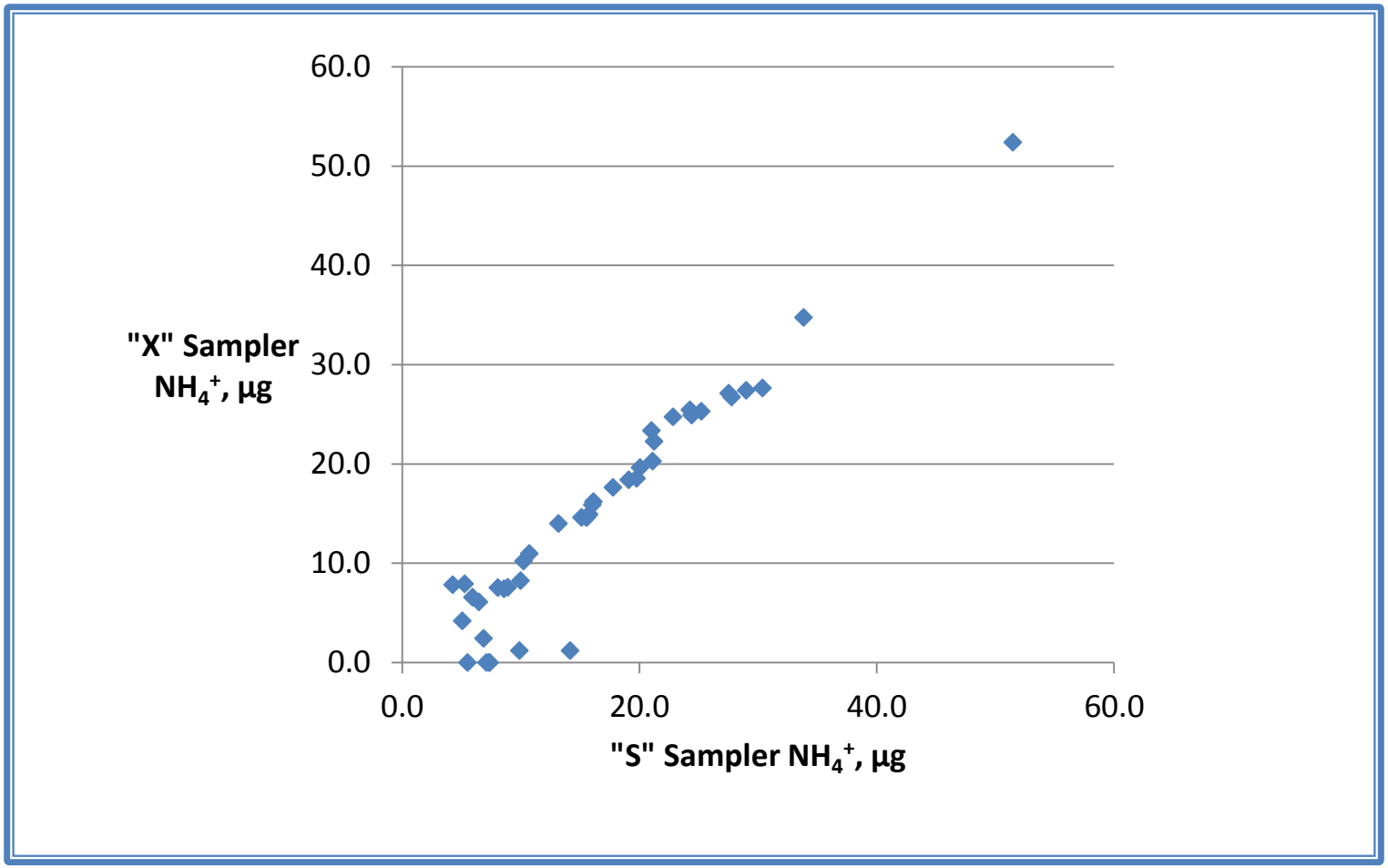
2011 NH_x Data Observations

- In the beginning, the collocated ("X") samplers at BOND and ROMO sometimes sampled improperly (or not at all).
- Field blanks generally are below 2 µg/filter, but show some increase over time.
- No methylamine has been detected in samples analyzed to date.
- The corresponding nylon filter samples that are analyzed for NH_4^+ are received later than the cellulose filters. How will this affect the data?

BONDS Collocated Samplers NH_4^+



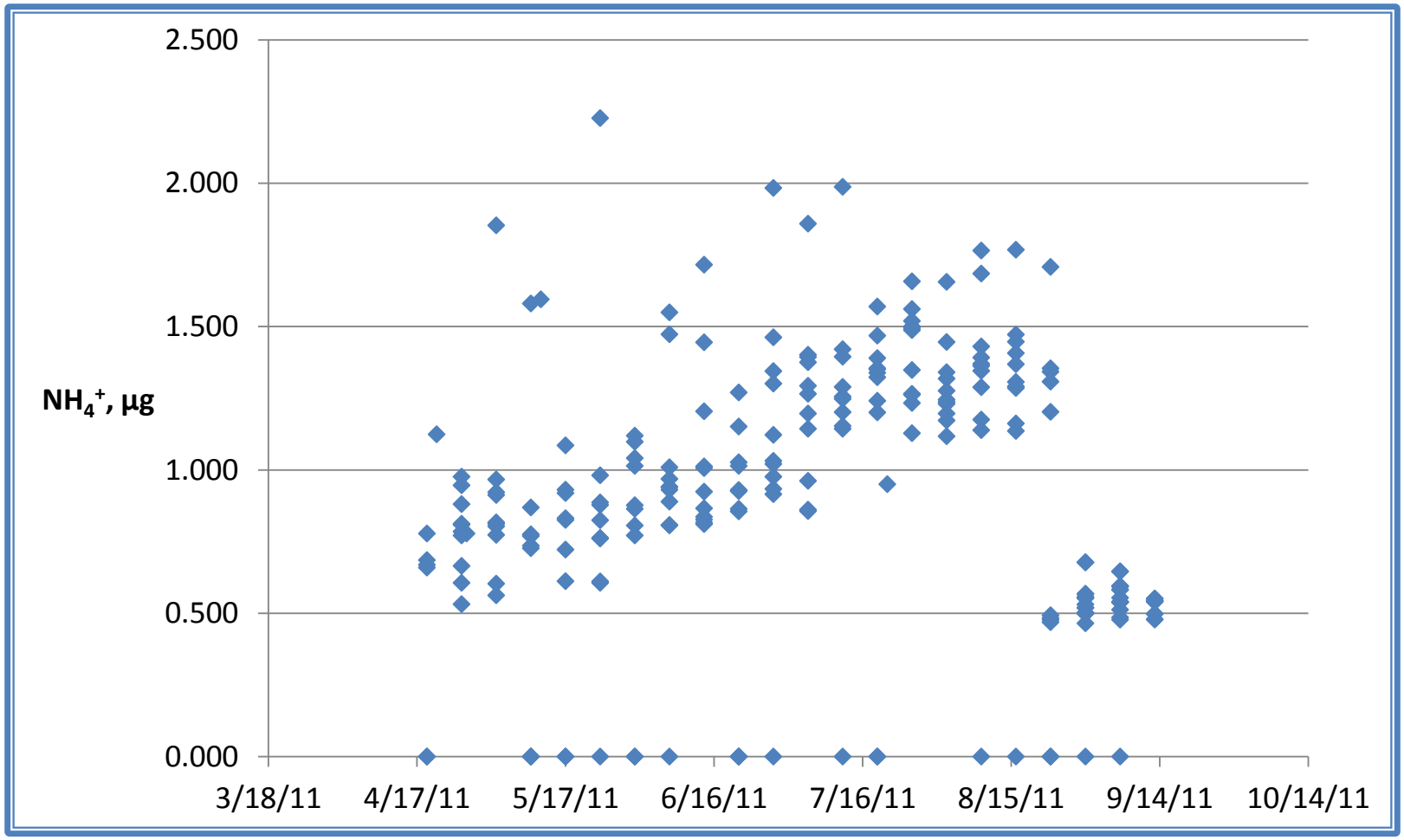
ROMO2S Collocated Samplers NH_4^+



Nylon/Cellulose Filters: Elapsed Time from Sampling to NH_4^+ Analysis

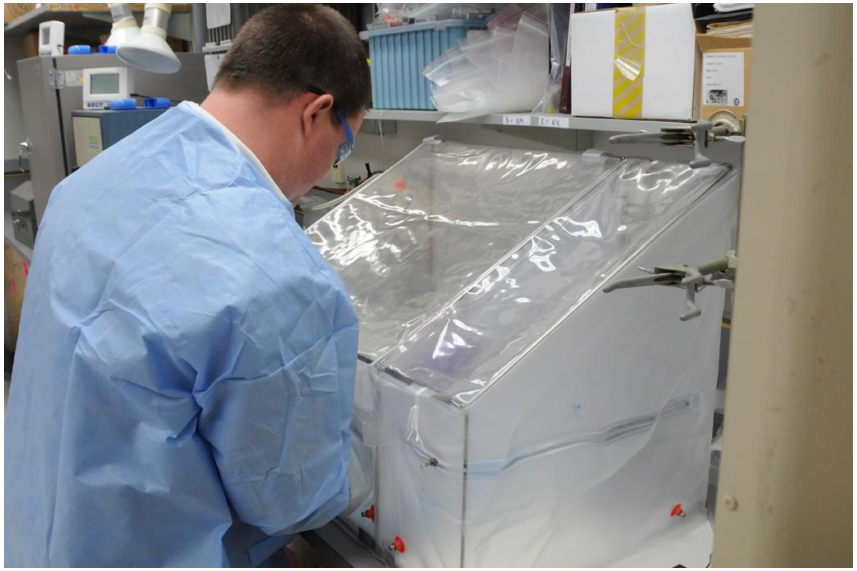
	Sampling to Receipt by RTI, days		Receipt by RTI to Analysis, days	
	Nylon	Cellulose	Nylon	Cellulose
Average	41	12.5	21	5
Minimum	18	2	7	1
Maximum	68	30	35	12

NH_x Cellulose Filter Field Blanks

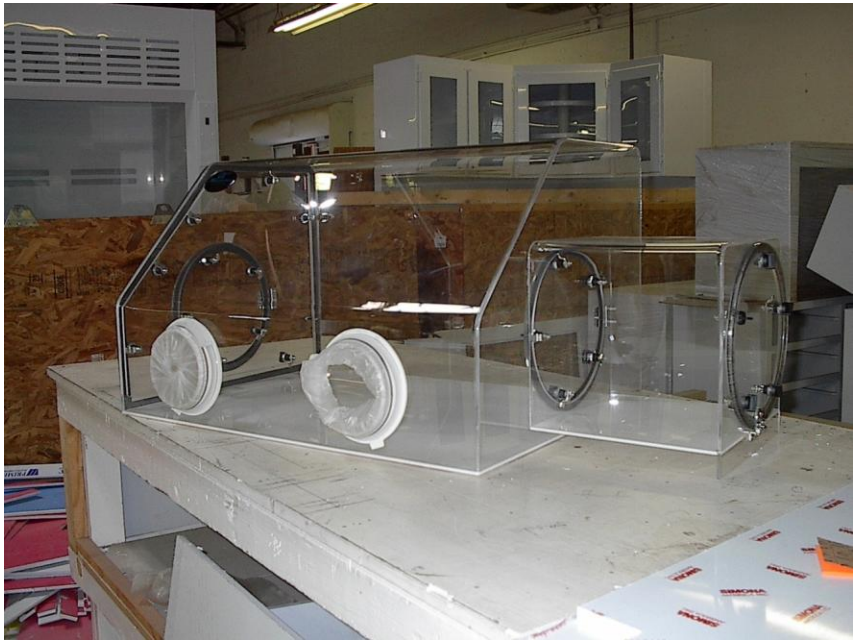


NHx Sample Storage and Extraction

Older, collapsible glove box

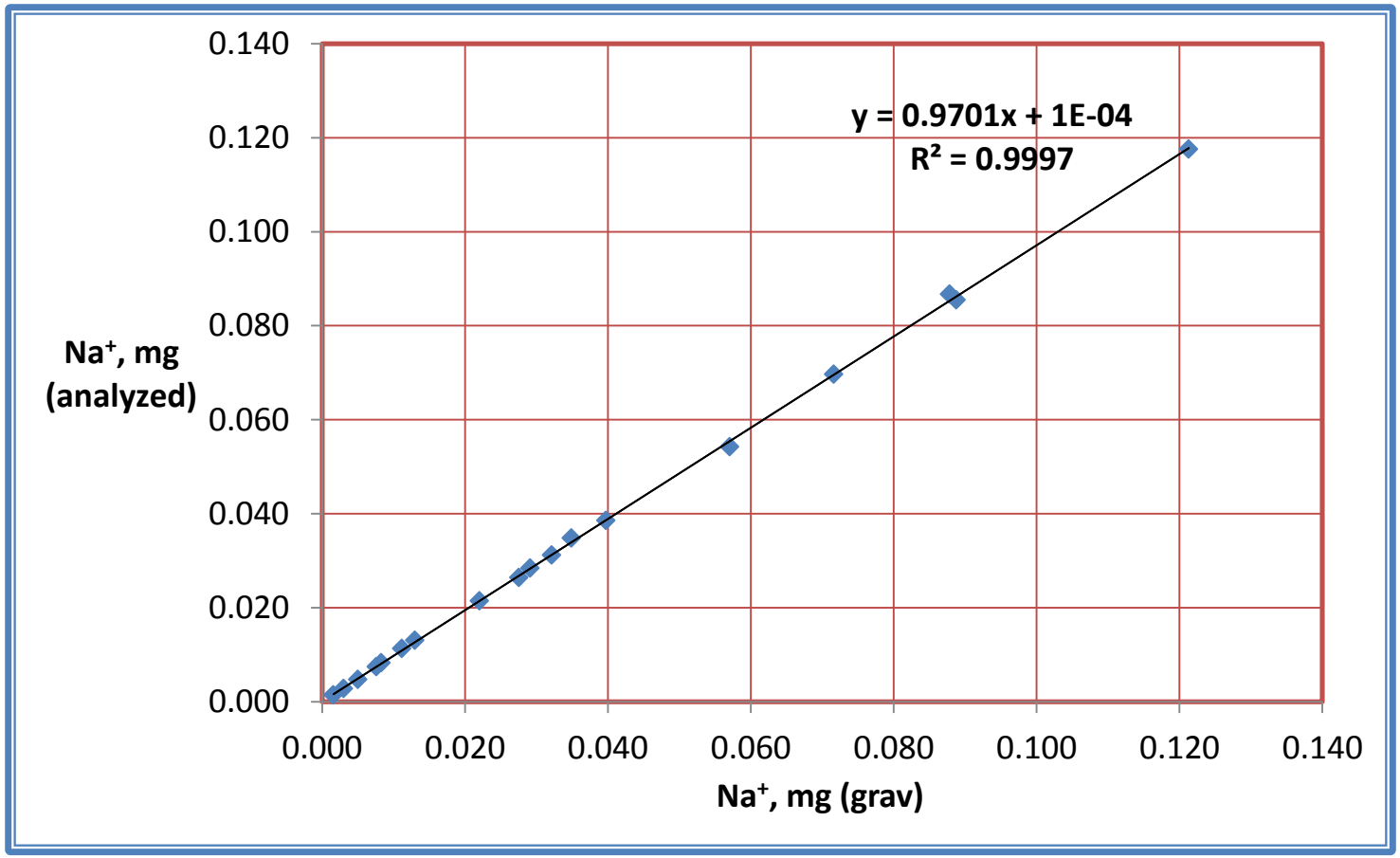


New glove box

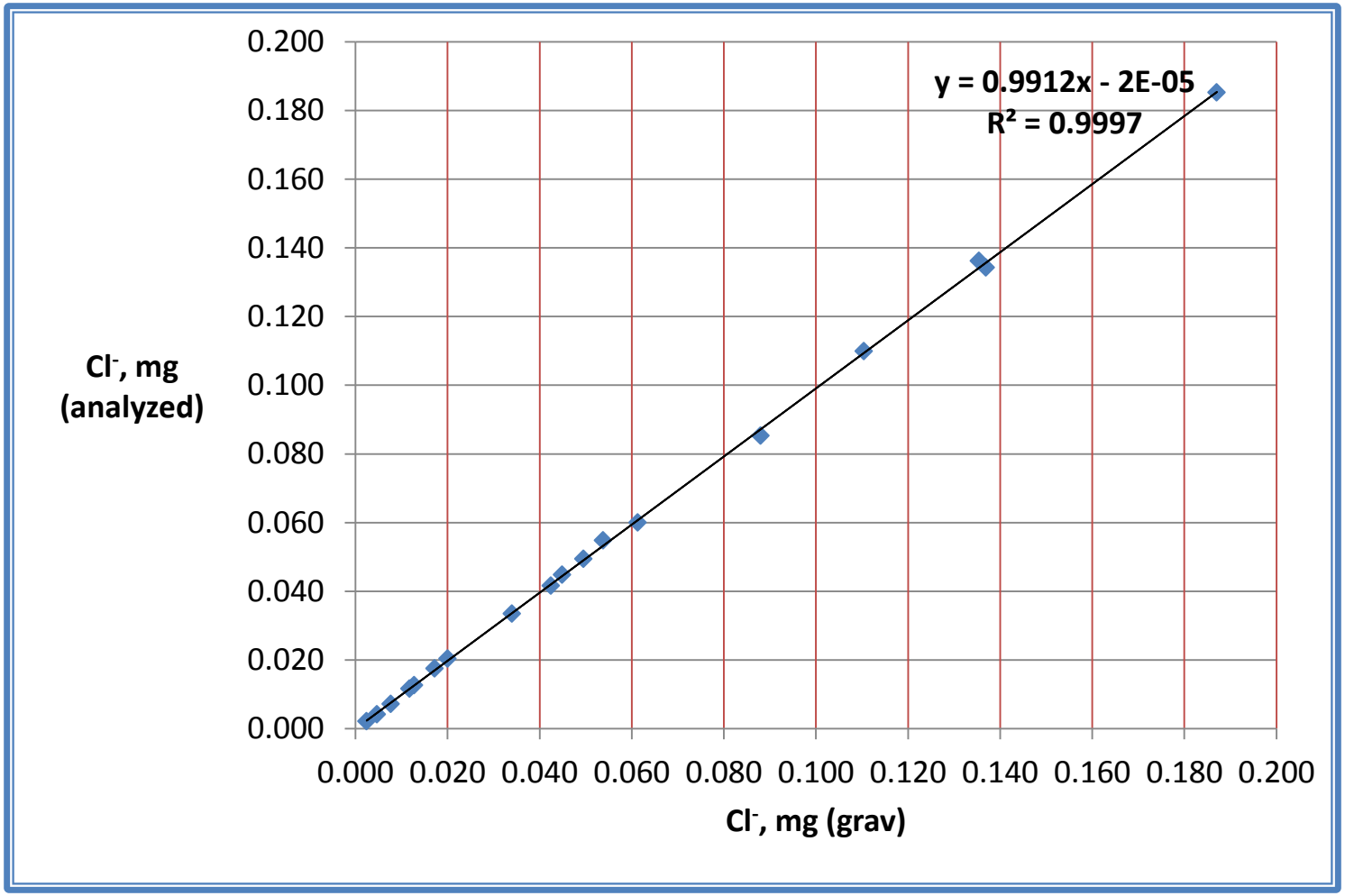


Special Studies: UCD Research Filters for Calibration of XRF Systems

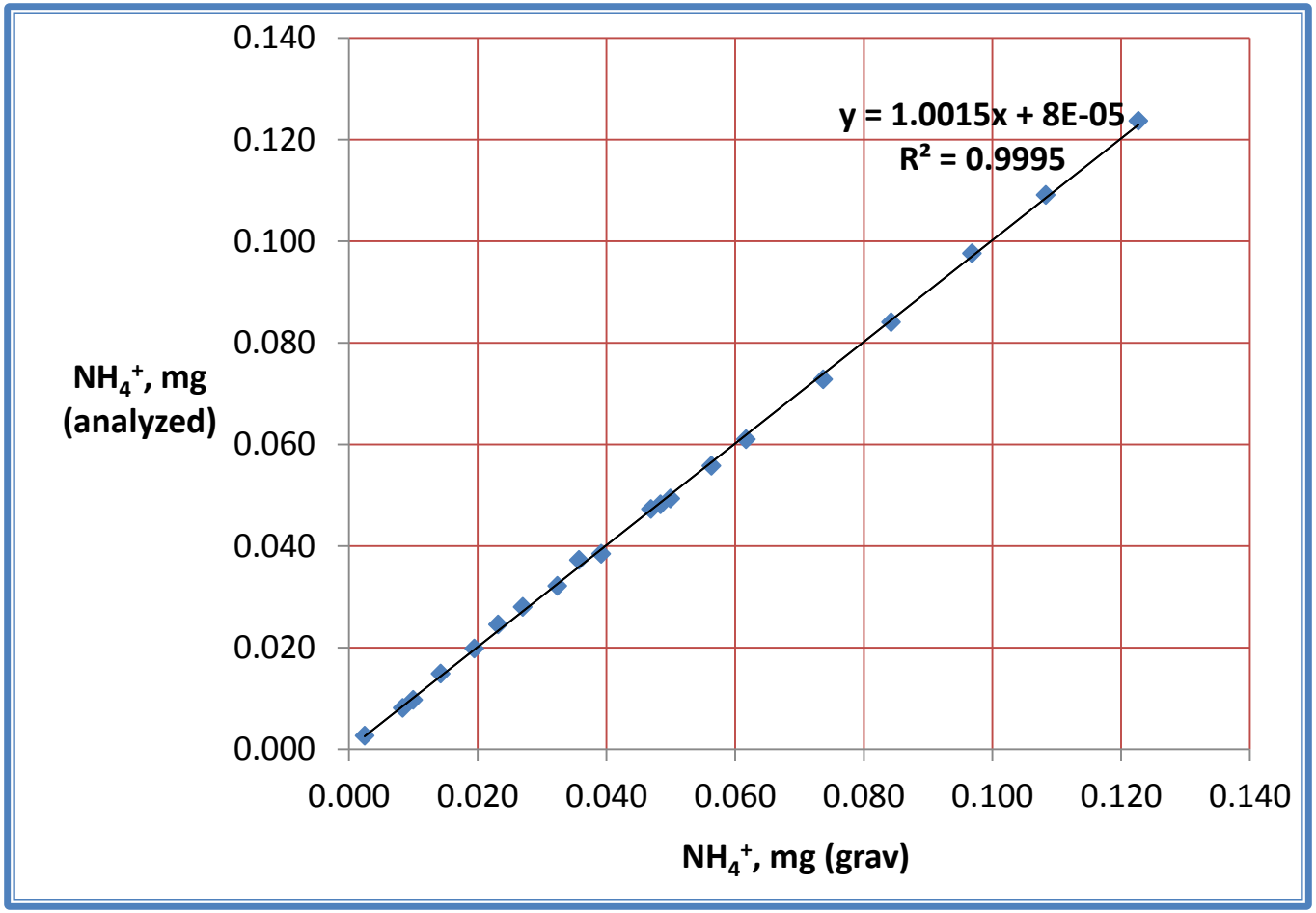
XRF Reference Filters: Sodium Ion Loadings



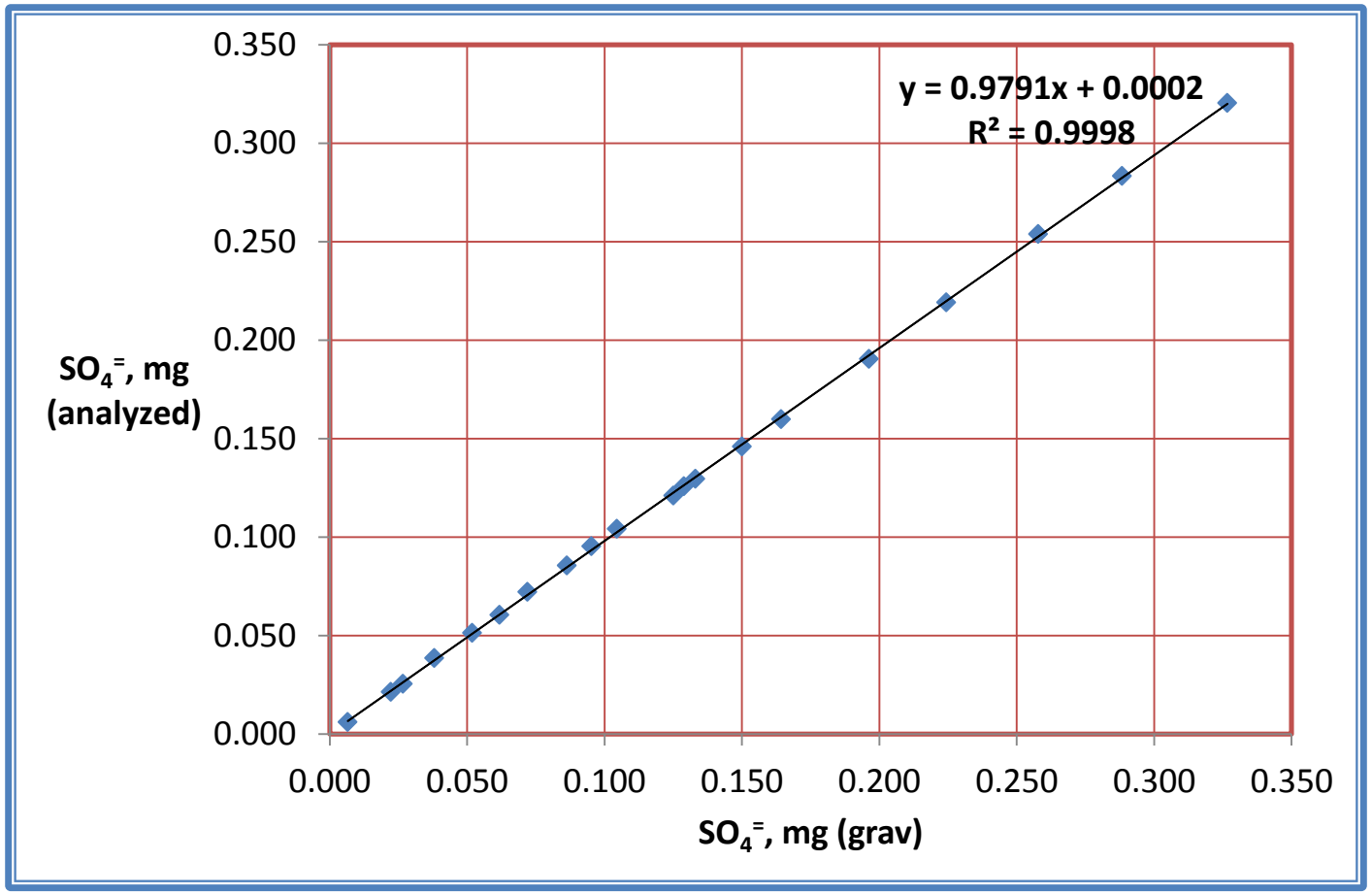
XRF Reference Filters: Chloride Ion Loadings



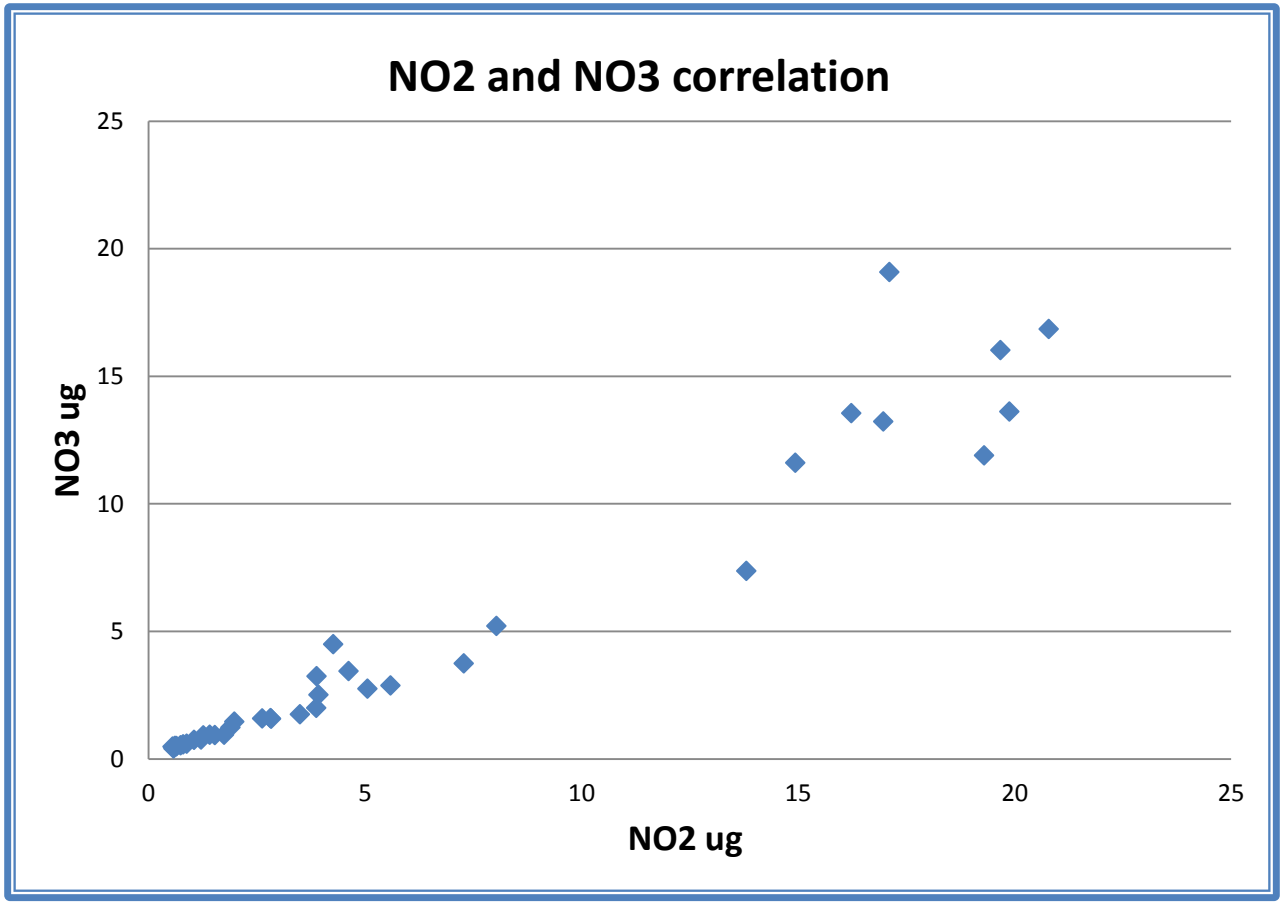
XRF Reference Filters: Ammonium Ion Loadings



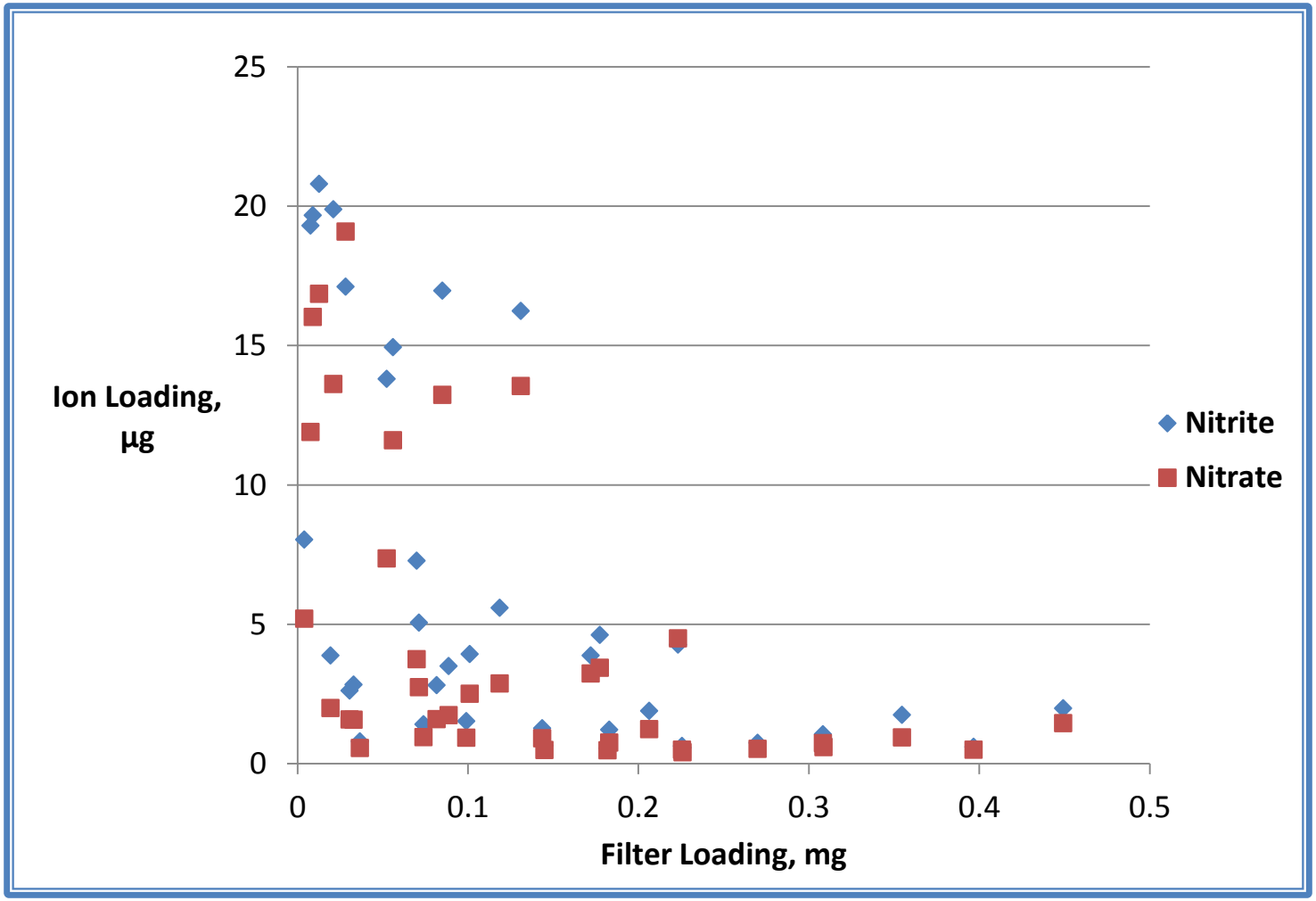
XRF Reference Filters: Sulfate Ion Loadings



XRF Reference Filters Also Contain NO_2^- and NO_3^-

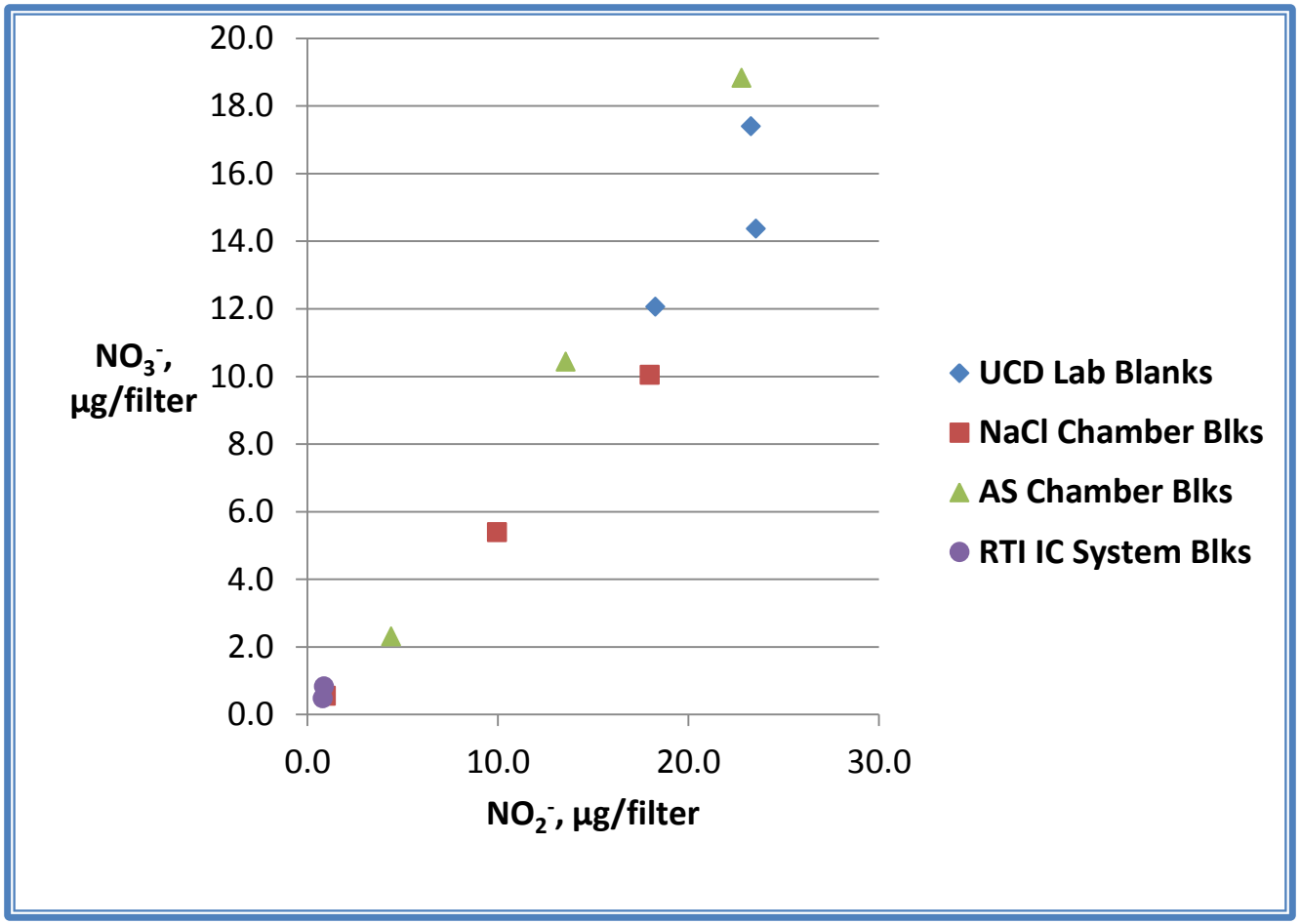


XRF Reference Filters



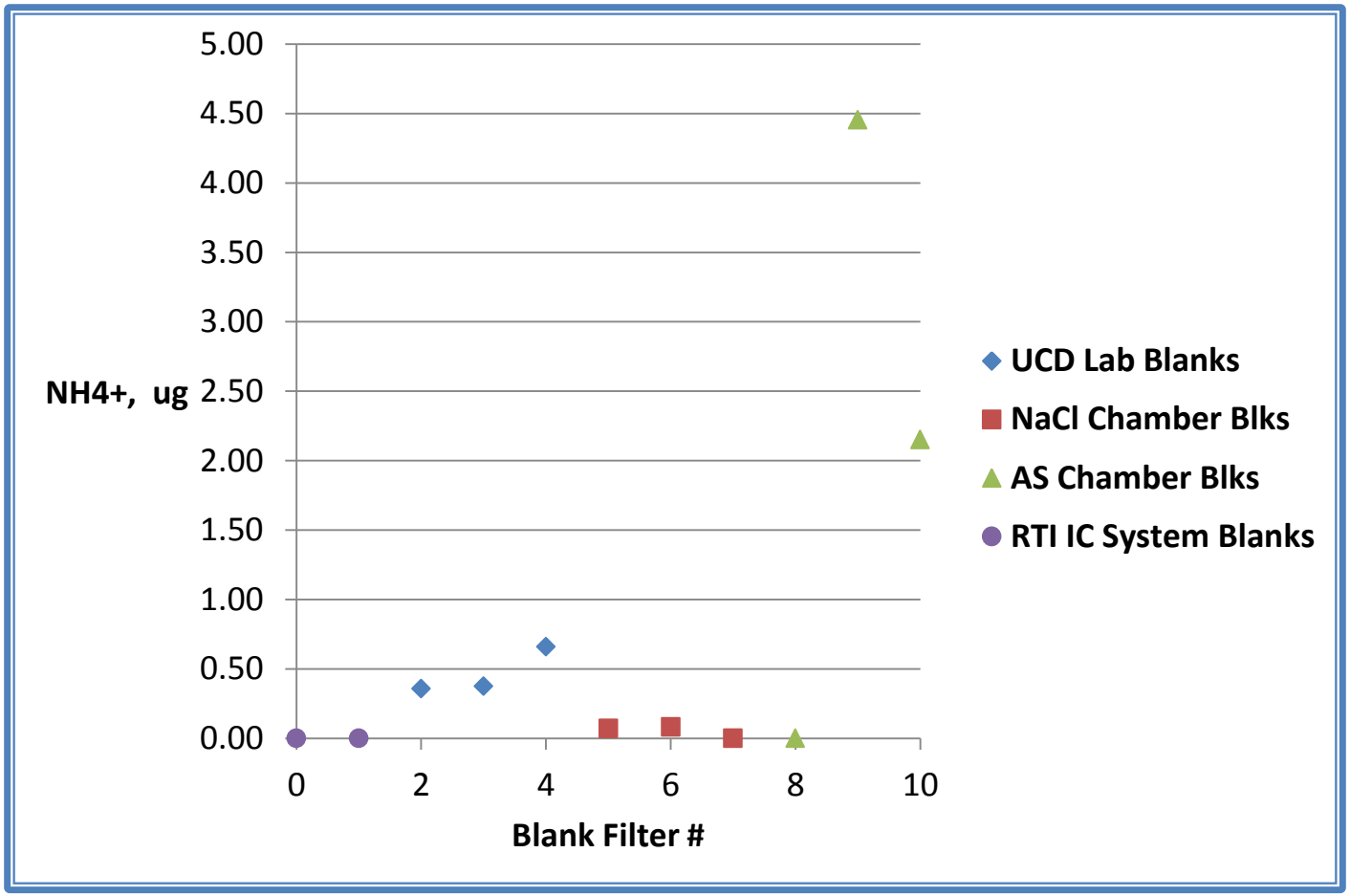
Teflon Filter Blanks

NO_2^- and NO_3^- Correlation



Teflon Filter Blanks

NH4+ Detected, µg



More News from the RTI Ions Lab

- A Dionex (Thermo Fisher Scientific) Model 3000 Ion Chromatograph has been set up and dedicated for levoglucosan analyses.

Fire at Chiricahua
June 8, 2011



Filter ID	Levo-glucosan, µg	K^+, µg
CHIR1 6/8/11	0.3	6.2
CHIR1 6/11/11	76.2	18.0
CHIR1 6/14/11	2.1	8.2
CHIR1 6/17/11	4.7	24.9
Extracts analyzed 9/13/11		

Announcements

- Dr. Prakash Doraiswamy, Research Scientist at the University at Albany, will join the Environmental Chemistry Department at RTI on October 31, 2011.
- If you are attending the AWMA Symposium on Air Quality Measurement Methods and Technology in Durham, NC, April 23-26, 2012, you are welcome to visit/tour RTI's laboratories.

Thank you, and…

