

Low-concentration Nutrient Determination Needs and Challenges—Which Ones? How Low? What Applications?

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Tuesday, August 16

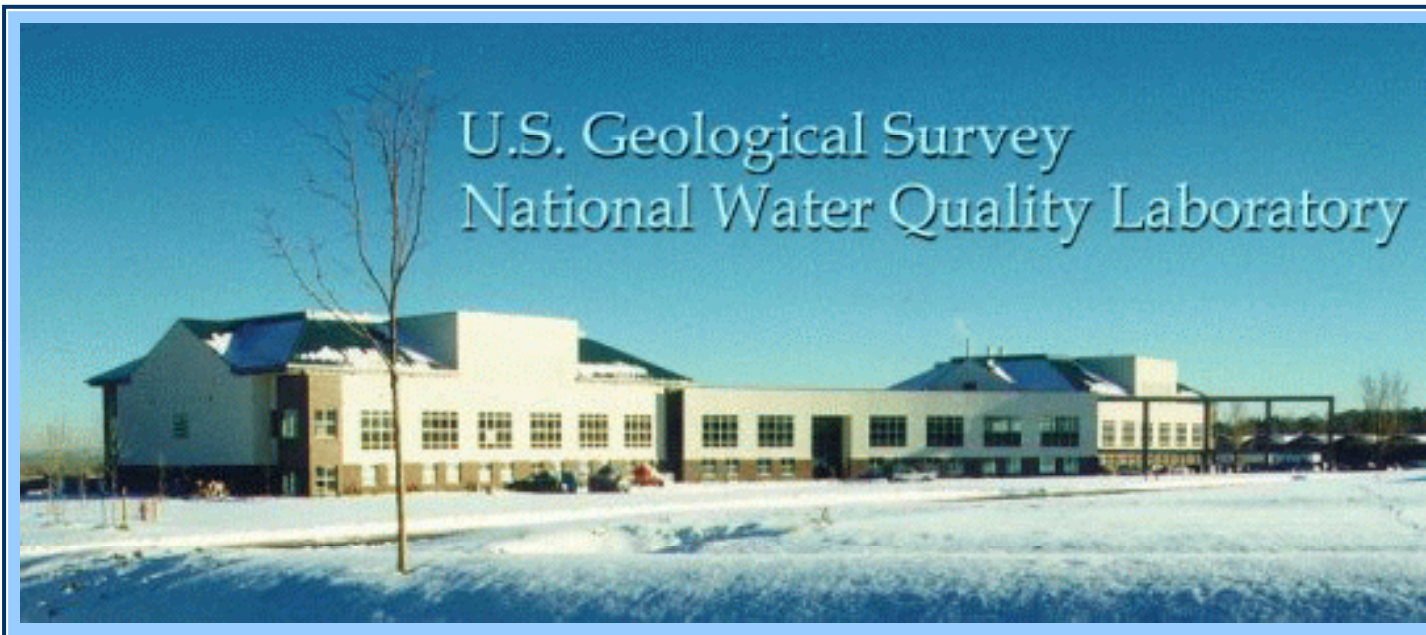
*2011 NEMC, Bellevue, WA
Challenges in Low-concentration Nutrient Analyses*

Speakers for Challenges of low-concentration nutrient analysis morning and afternoon sessions

Speaker Name	Time	Title
Charles Patton	9:00	Low-concentration Nutrient Determination Needs and Challenges—Which Ones? How Low? What Applications?
JB Neethling	9:30	Variability in Wastewater Effluent Phosphorus Measurements
Morning Break		
Gertrud Nurnberg	10:30	Importance of low-level analysis in comparison to sample timing, handling and other methods to obtain representative phosphorus measurements in lake water
Carl Zimmermann	11:00	Accurate Measurement of Particulate Nitrogen and Phosphorus in Environmental Water Samples
Wei Ning Yap	11:30	Monitoring and Fractionation of Low-Level Phosphorus in Water and Environment
Lunch Break		
Nancy Simon	1:30	The importance of analytical methods in the interpretation of data from natural systems- Accounting for all forms of P in the water and sediment of Upper Klamath Lake, OR
Scott Smith	2:00	Evaluation of Colorimetric Phosphate Speciation Analysis Using Long Path Lengths and Model Compounds
Edward Askew	2:30	Low Level Determination of Ammonia by Membrane Extraction-Conductivity
Afternoon Break		
William Lipps	3:30	Low-concentration Nutrient Determinations in Water—An Overview of Approved- and Candidate-method Capabilities
William Lipps and Charles Patton	4:00	Speaker/SessionAttendee Round Table Discussion

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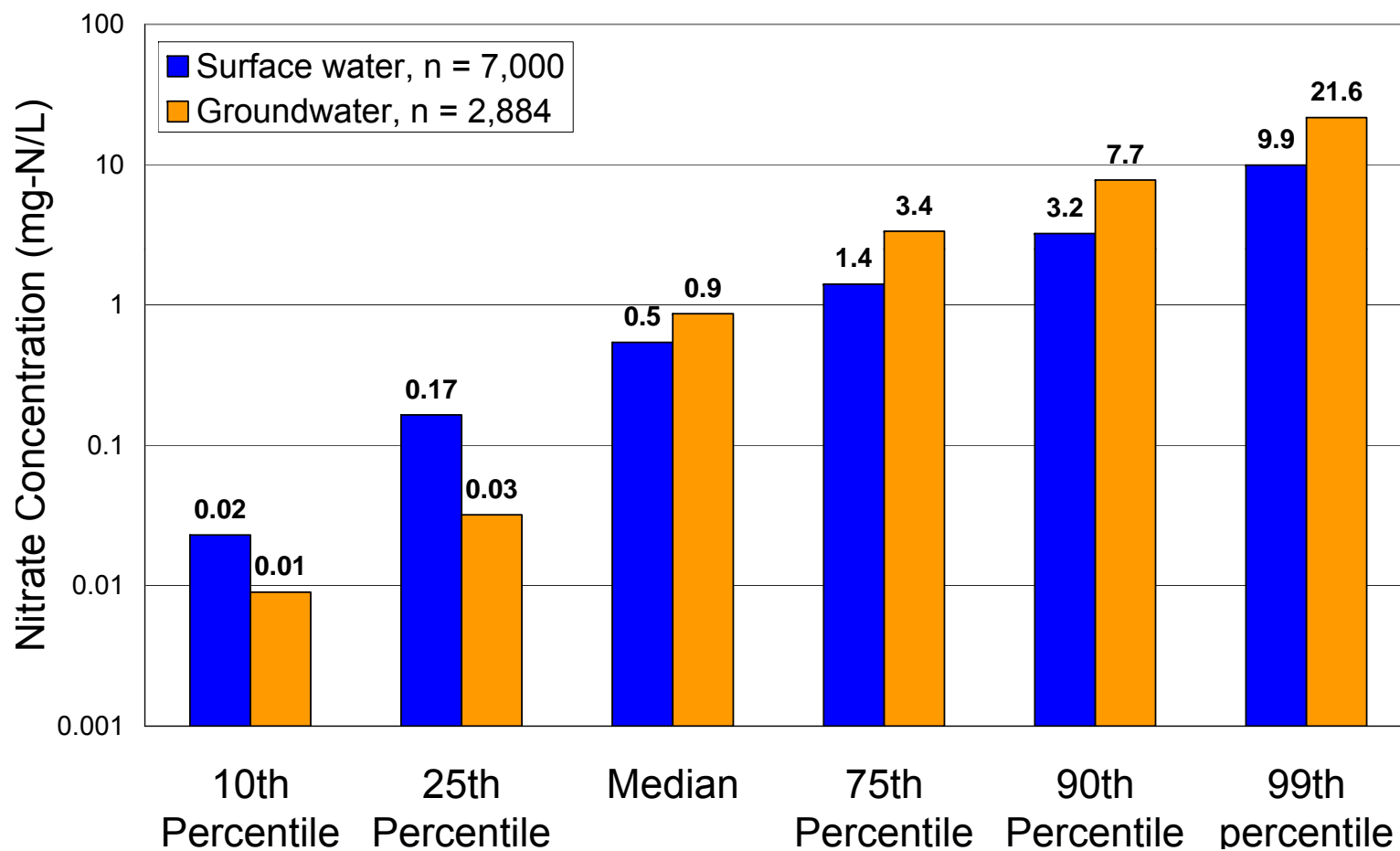


- Analytical service laboratory
- Production sample processing ~ 30,000 samples/year, ~ 1.5×10^6 determinations
- Quality Assurance/Quality Control
- New methods development including laboratory automation

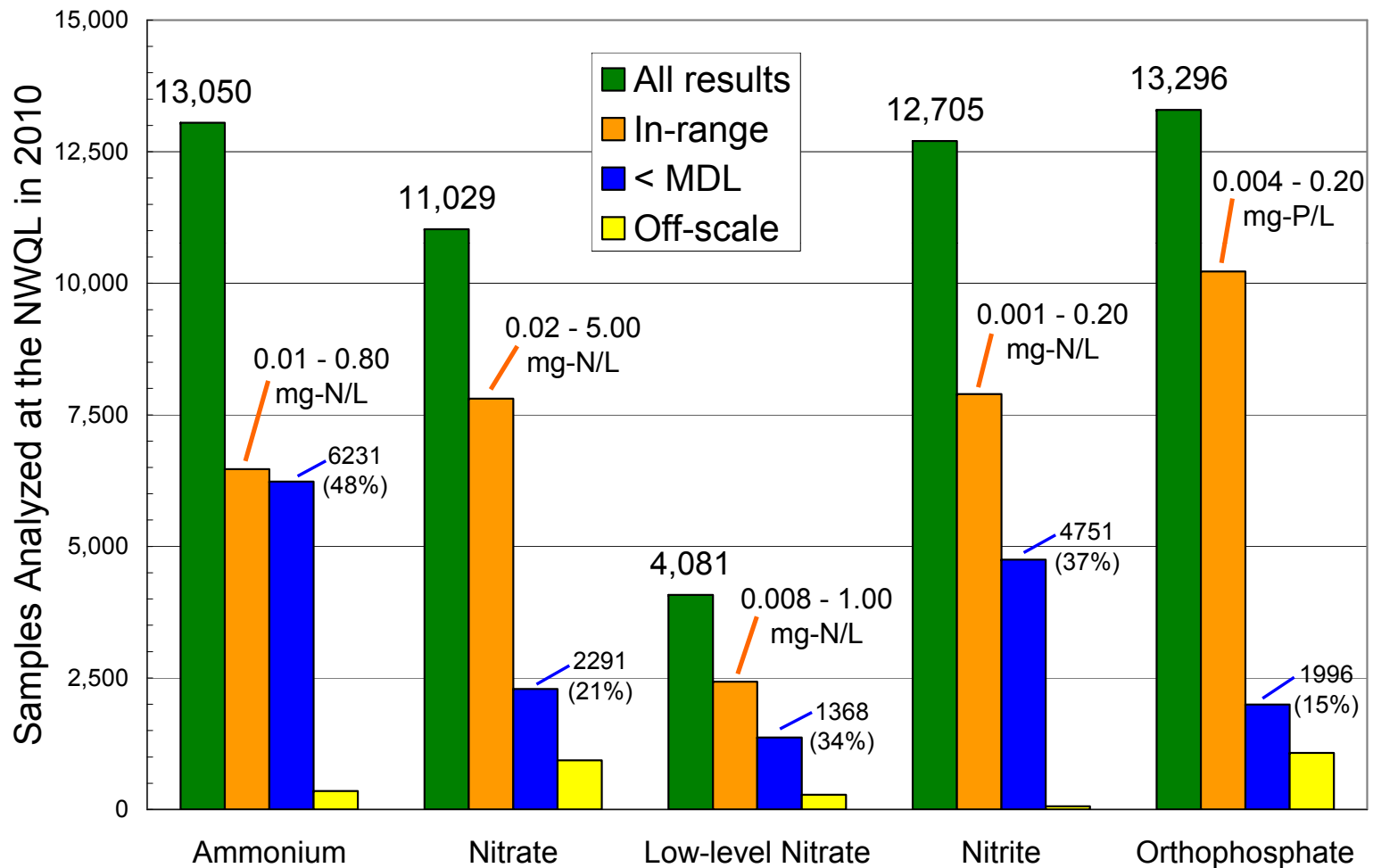
Overview of Yearly of Nutrient Concentrations in Surface Water and Groundwater in the United States

- Nitrogen- and Phosphorus-containing Nutrients
 - Filtered, Undigested
 - Filtered, Digested
 - Whole-water, Digested
 - Detection limits
 - Laboratory blanks
 - Blind blanks
 - Field blanks

Differences in concentration distributions for different water types



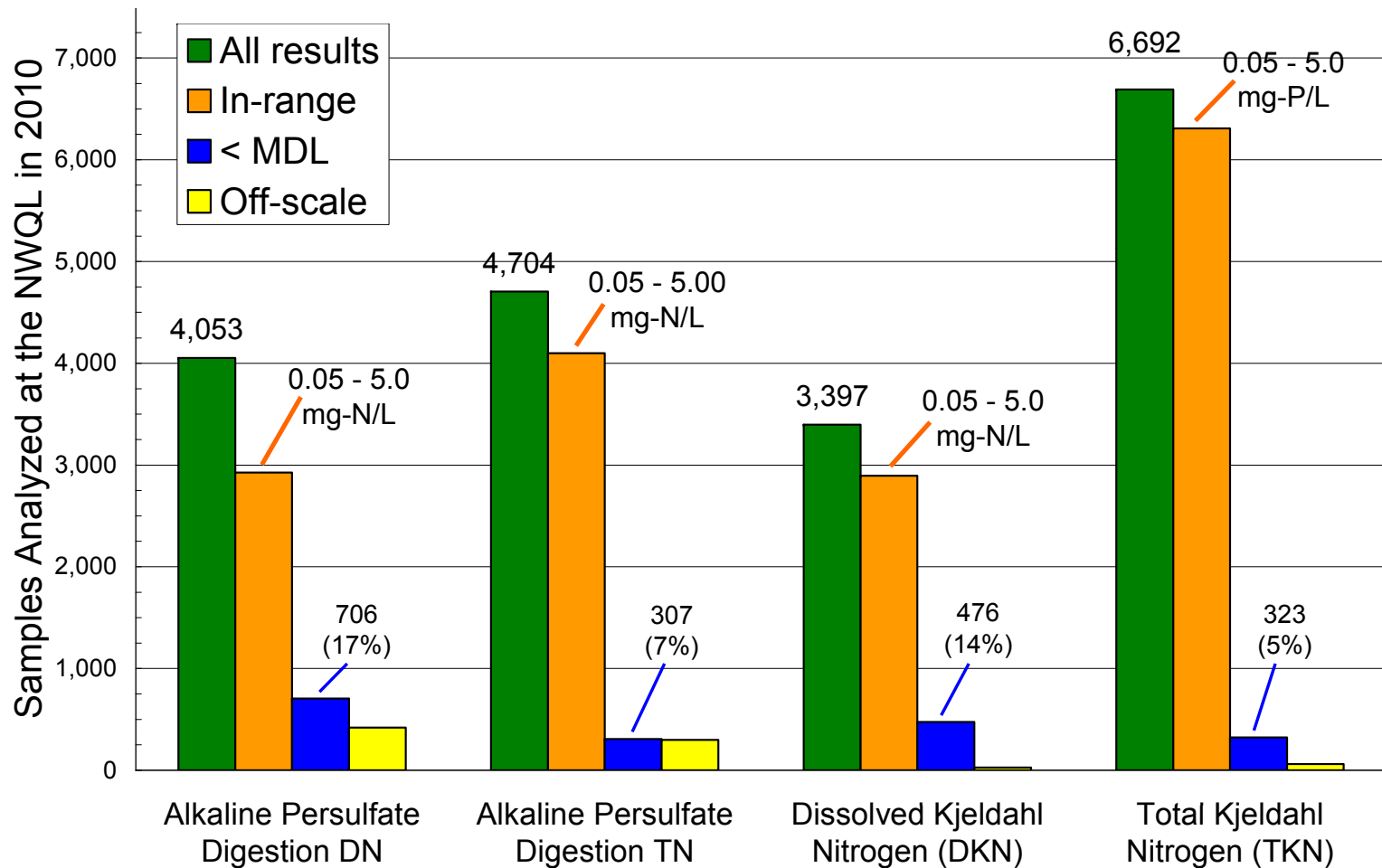
NWQL Dissolved Nutrient Analyses in 2010



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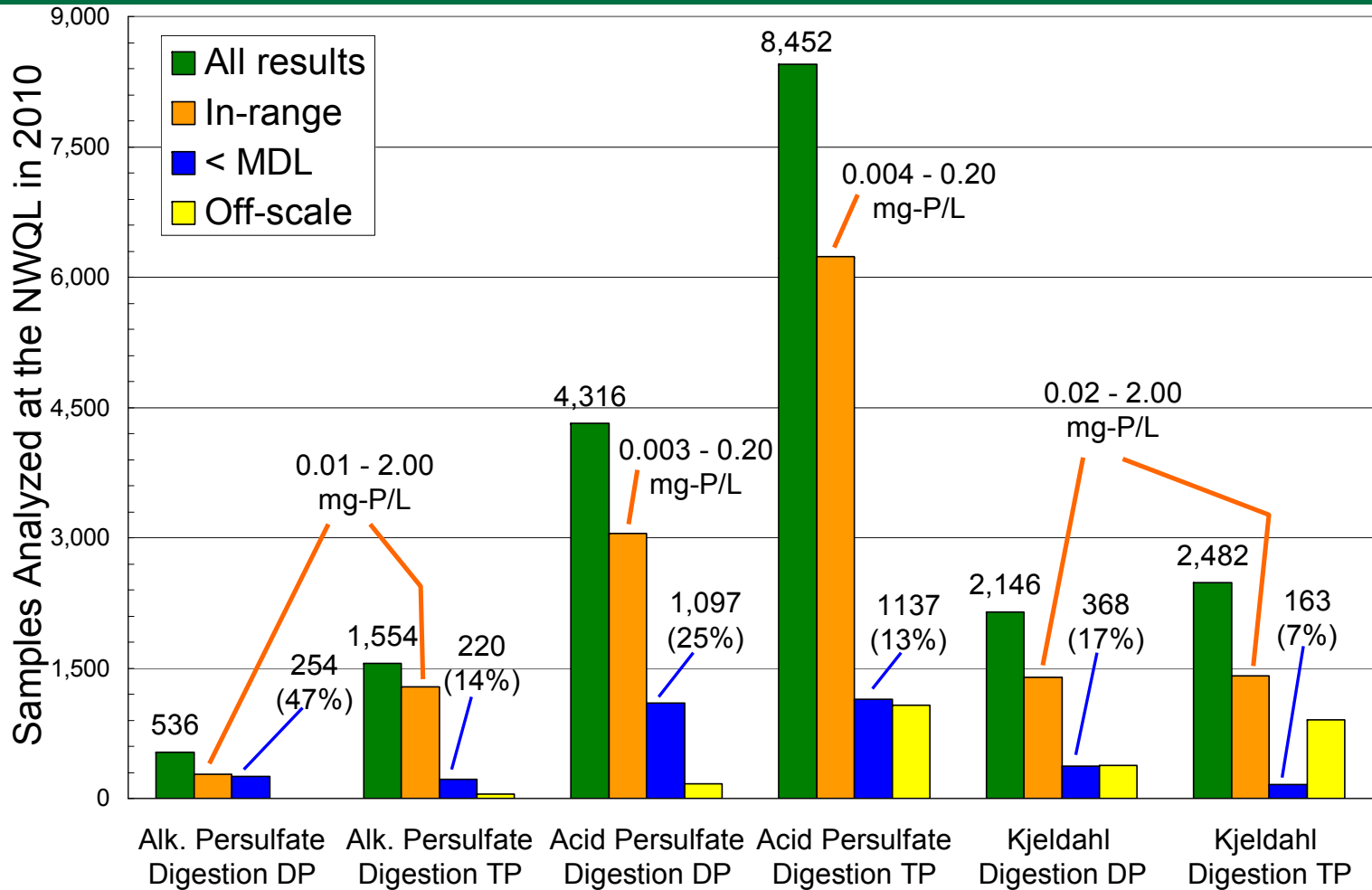
NWQL Dissolved and Total Nitrogen Analyses in 2010



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NWQL Dissolved and Total Phosphorus Analyses in 2010

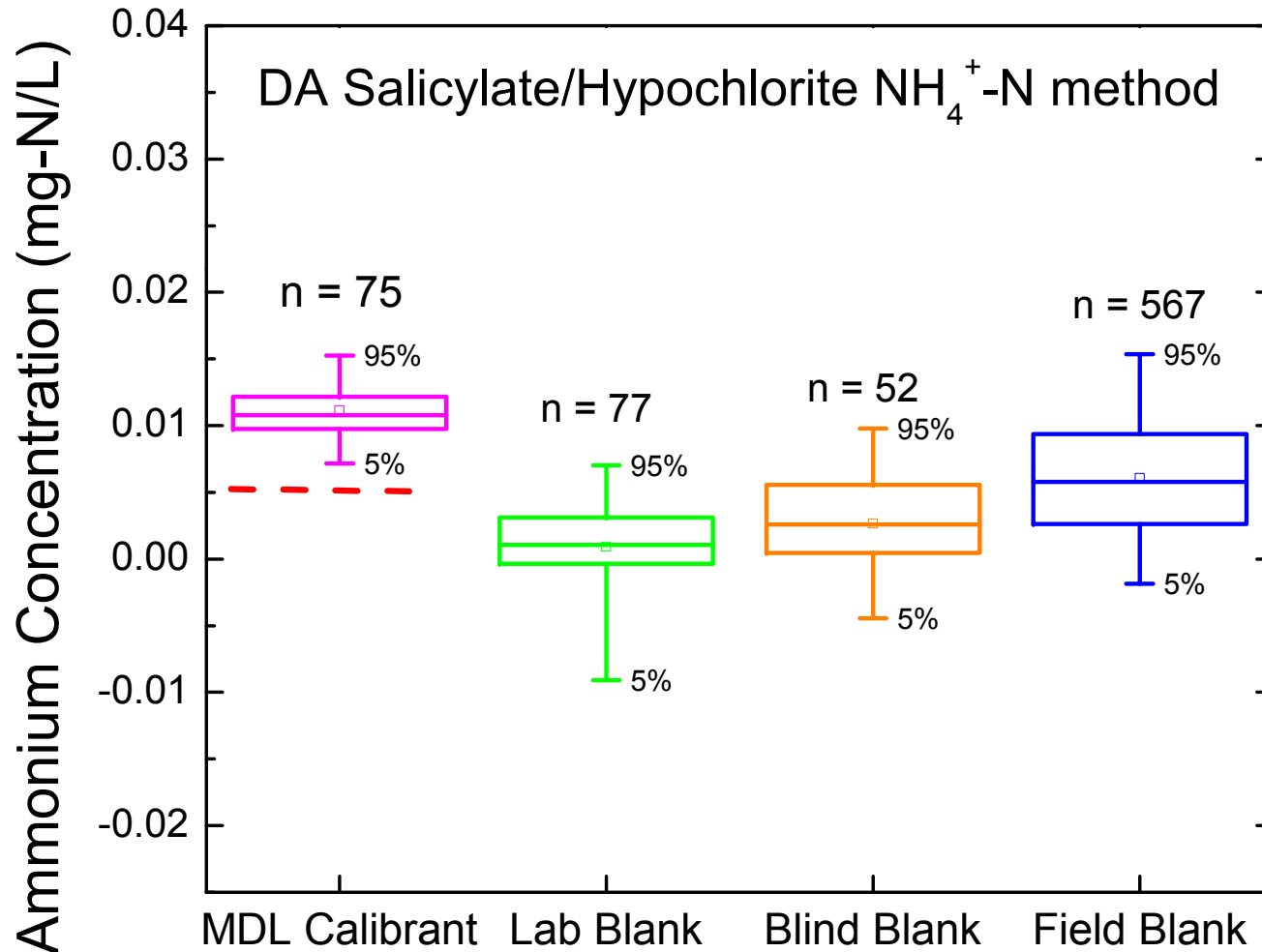


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NWQL Ammonium Analyses 2010 (Discrete Analyzer)

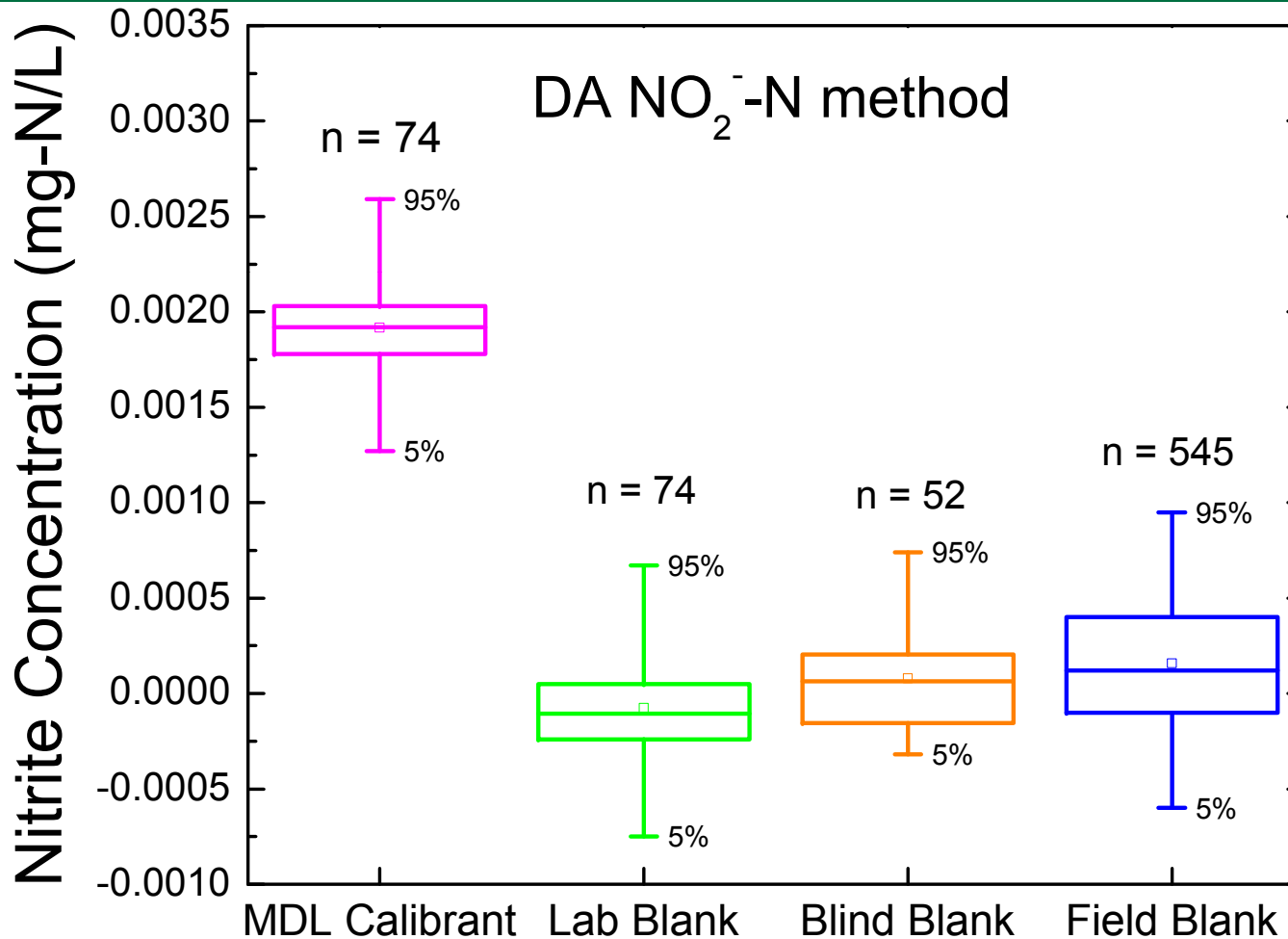


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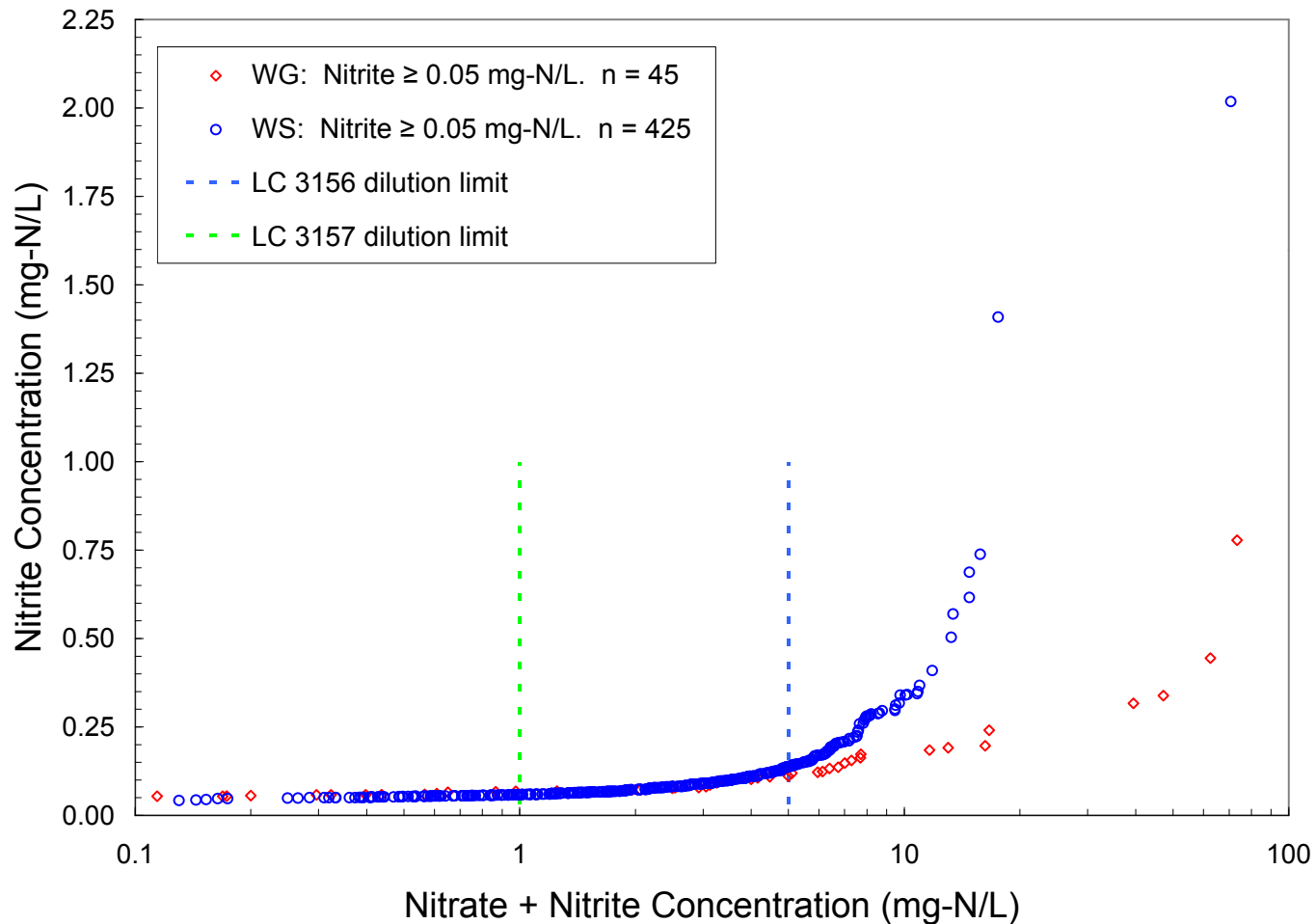
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NWQL Nitrite Analyses 2010 (Discrete Analyzer)



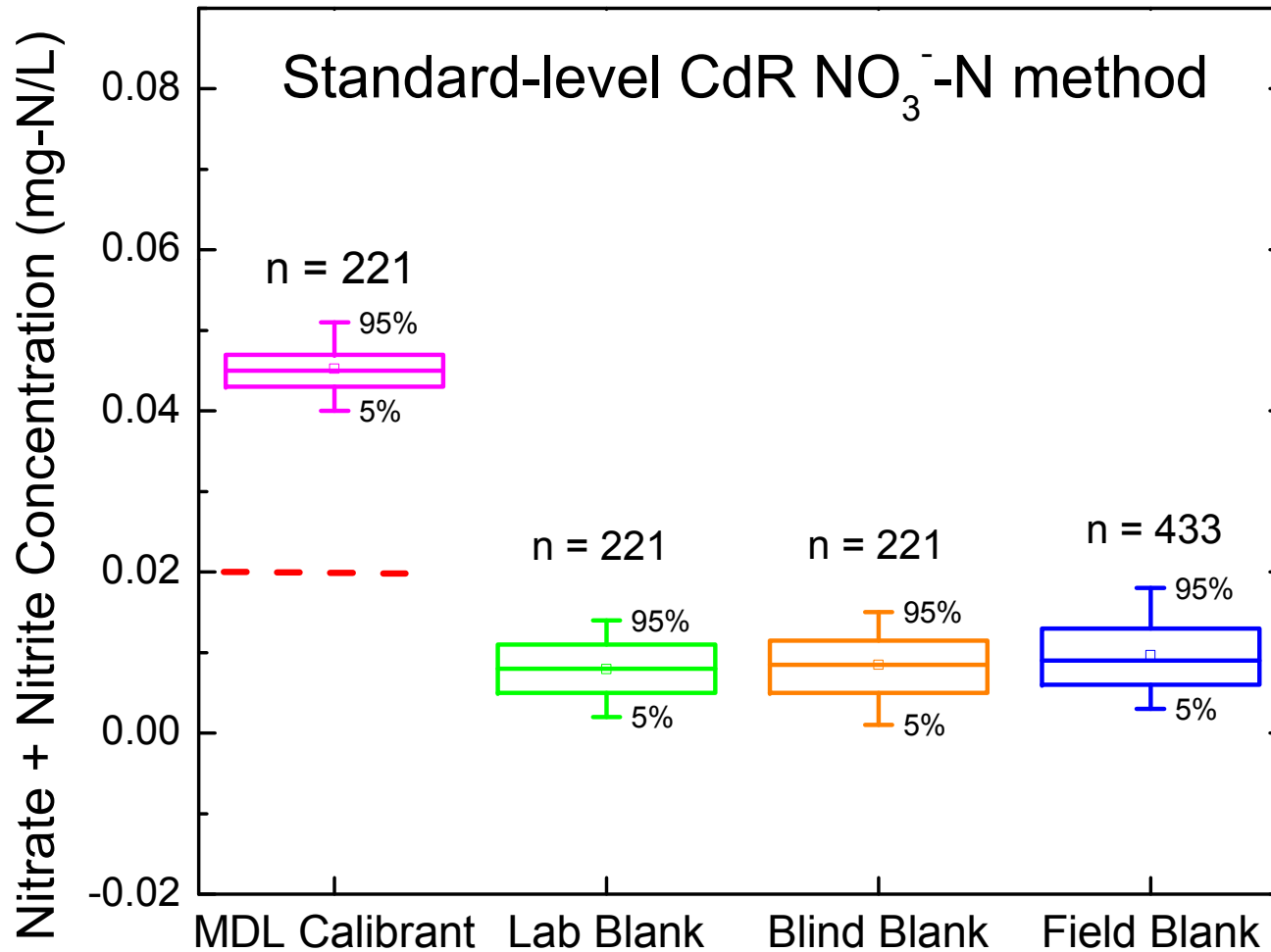
Relationship between nitrate + nitrite and nitrite concentrations analyzed at the NWQL in 2010



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NWQL Standard-level Nitrate Analyses 2010 (Continuous-flow Analyzer)

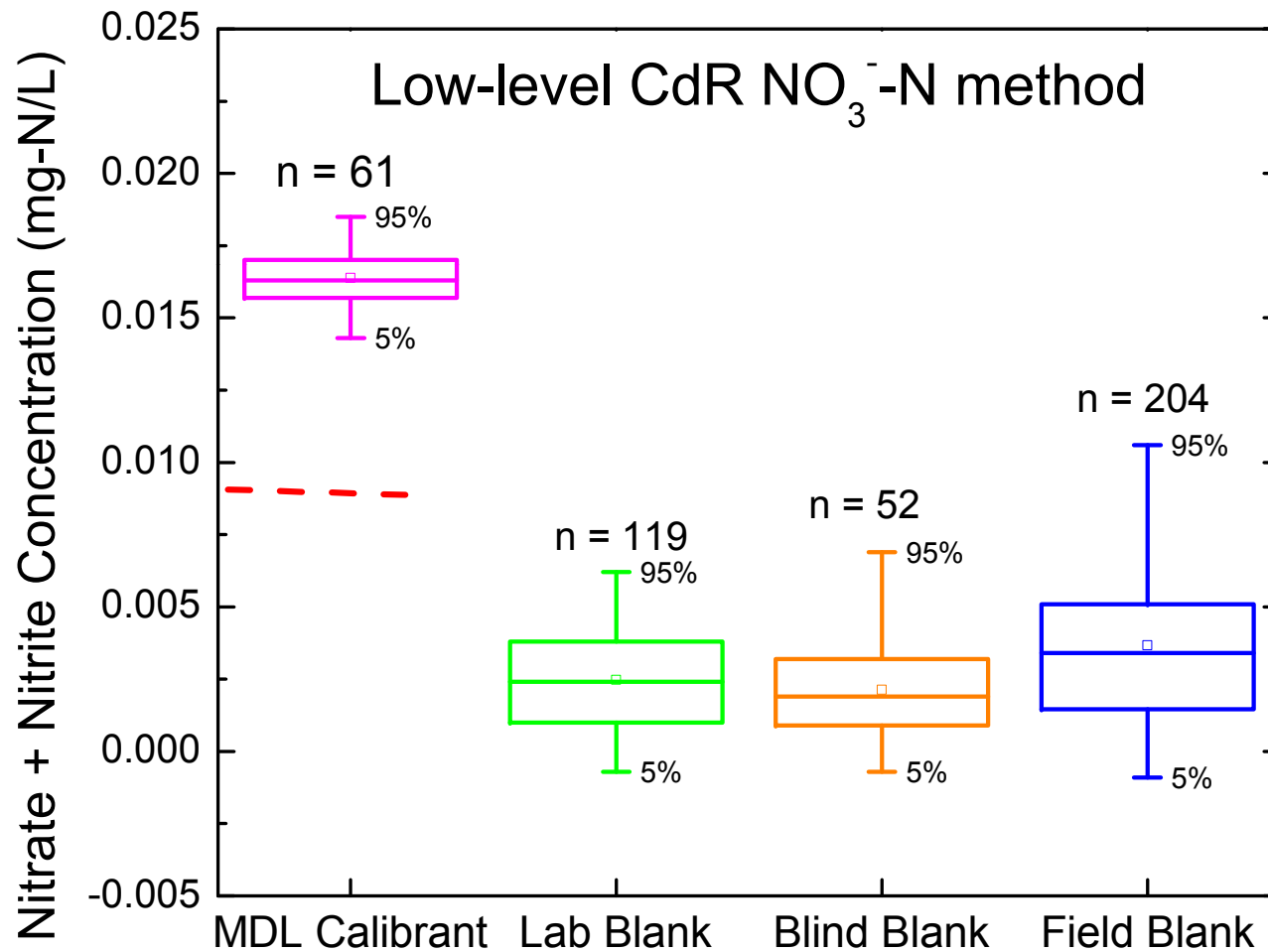


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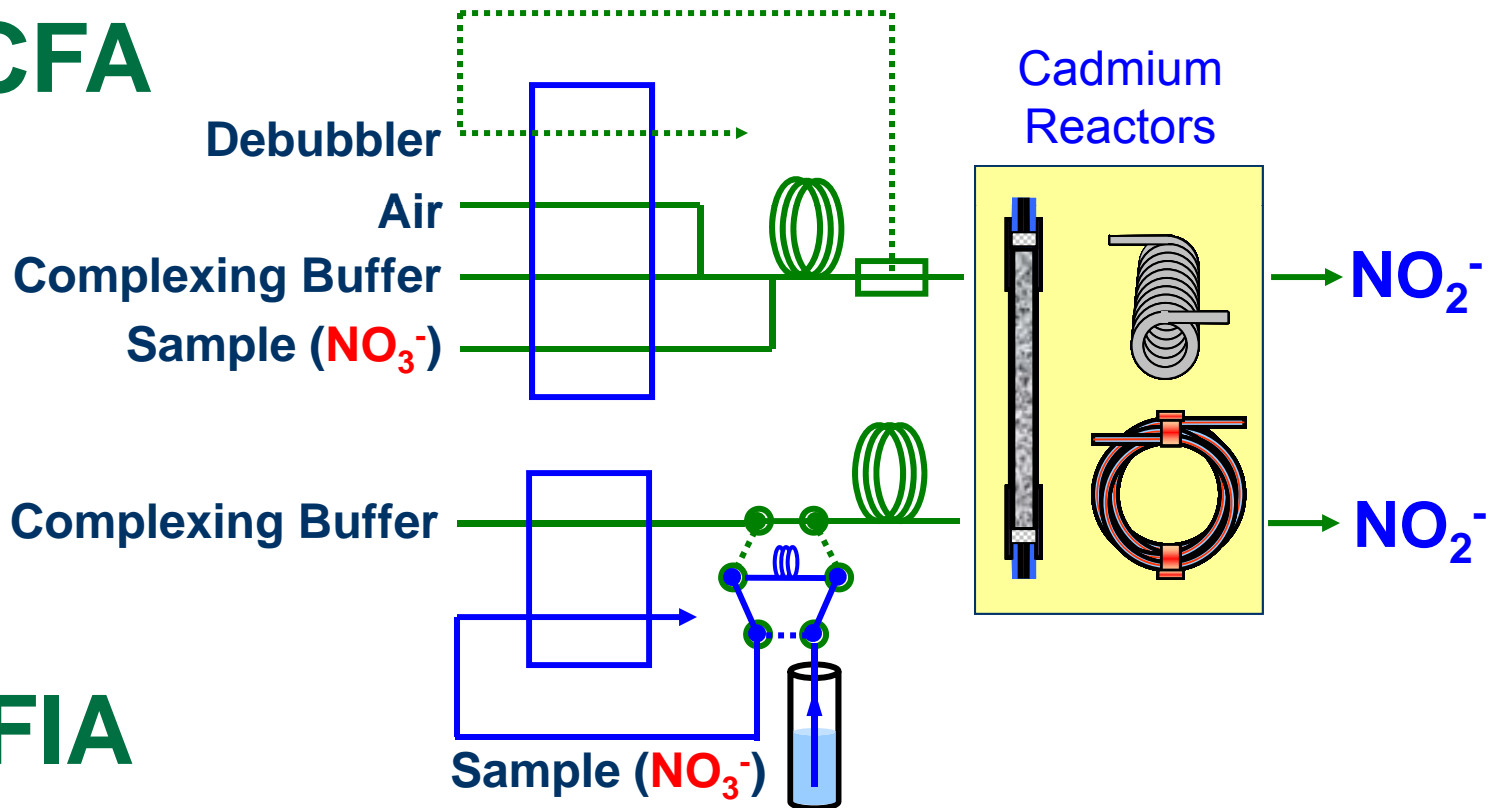
Challenges of Low-concentration Nutrient Analyses

NWQL Low-level Nitrate Analyses 2010 Analyses 2010 (Continuous-flow Analyzer)



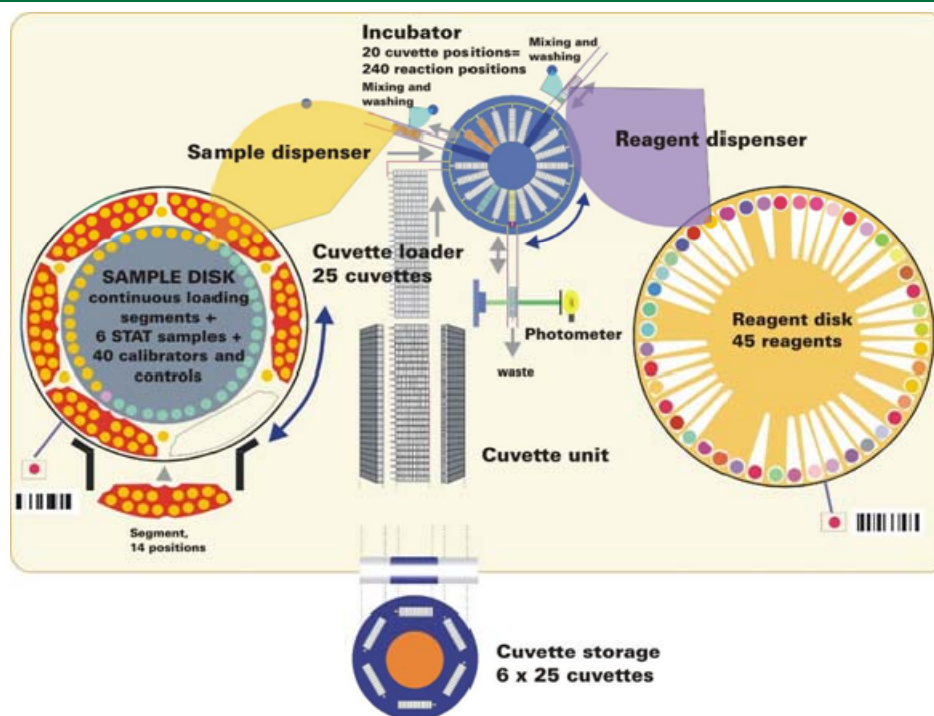
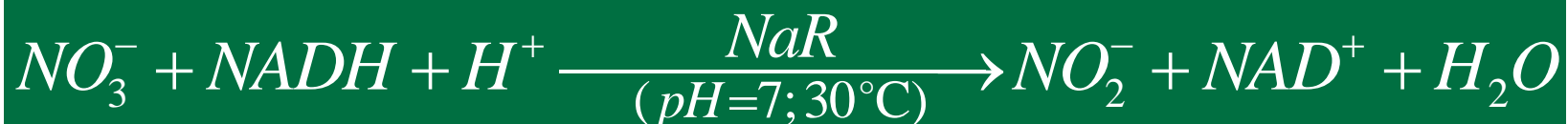
Cadmium reactors are easily incorporated into continuous flow analyzers

CFA

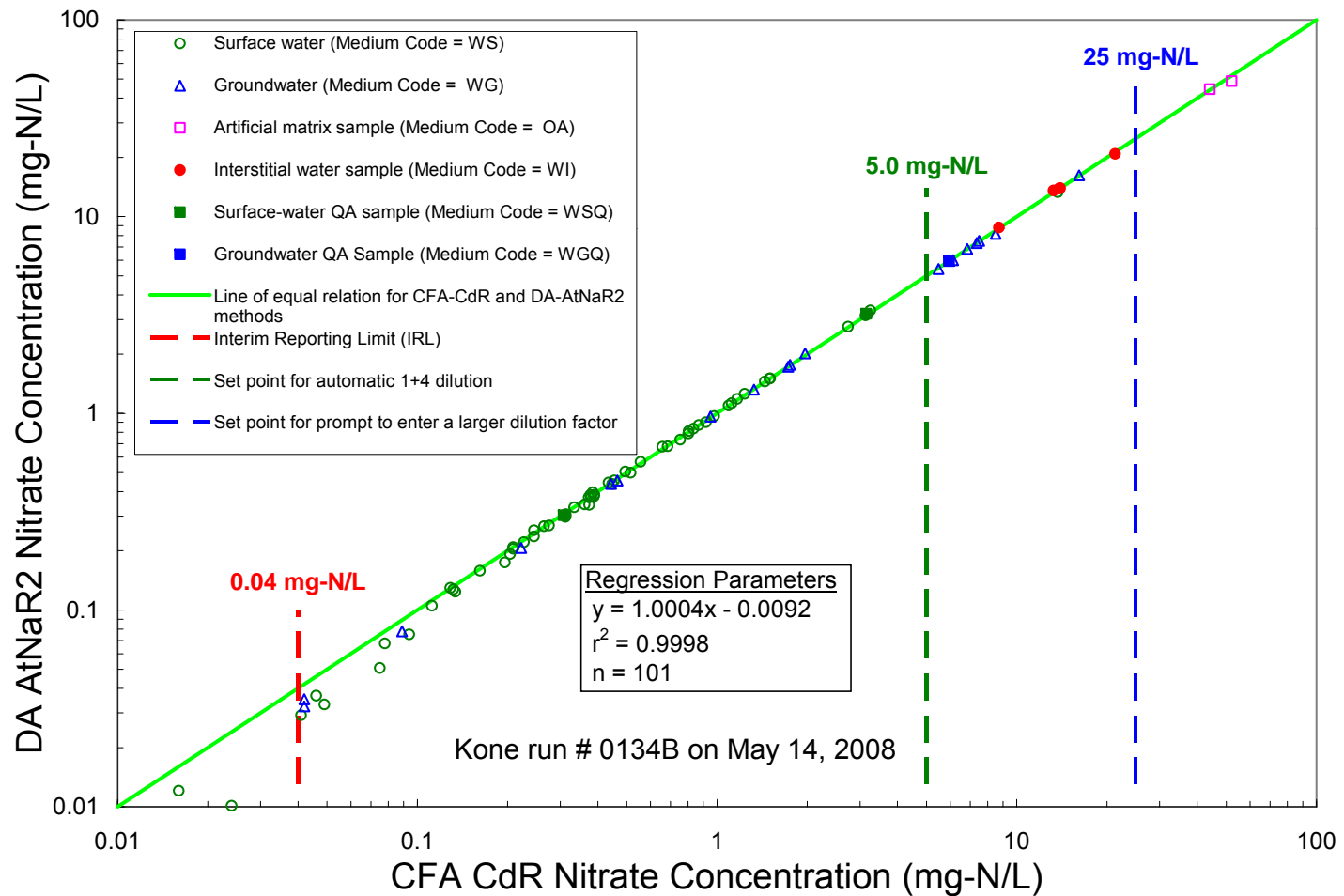


FIA

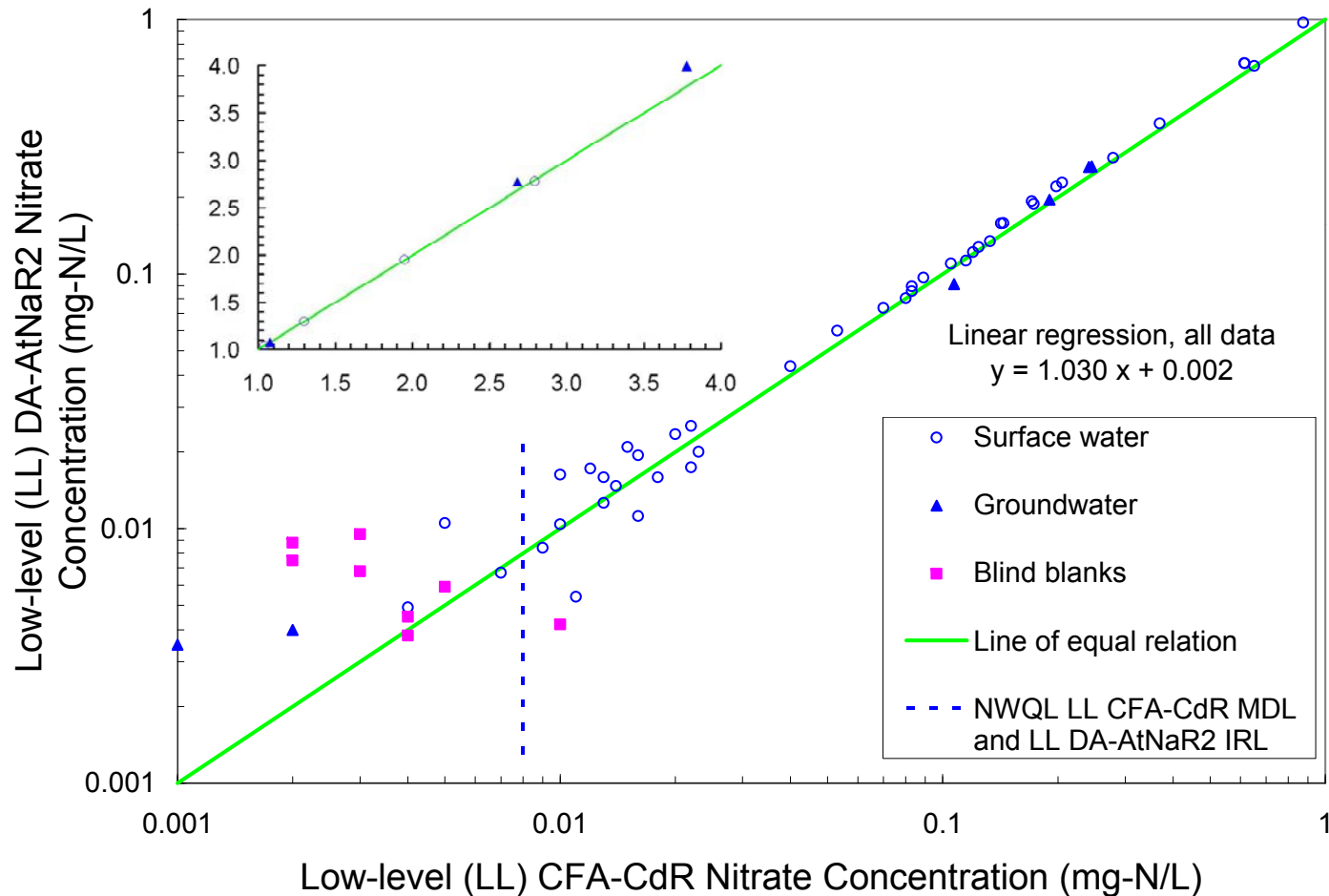
Discrete analyzer nitrate reductase (AtNaR2) nitrate assay (37°C)—now USGS approved



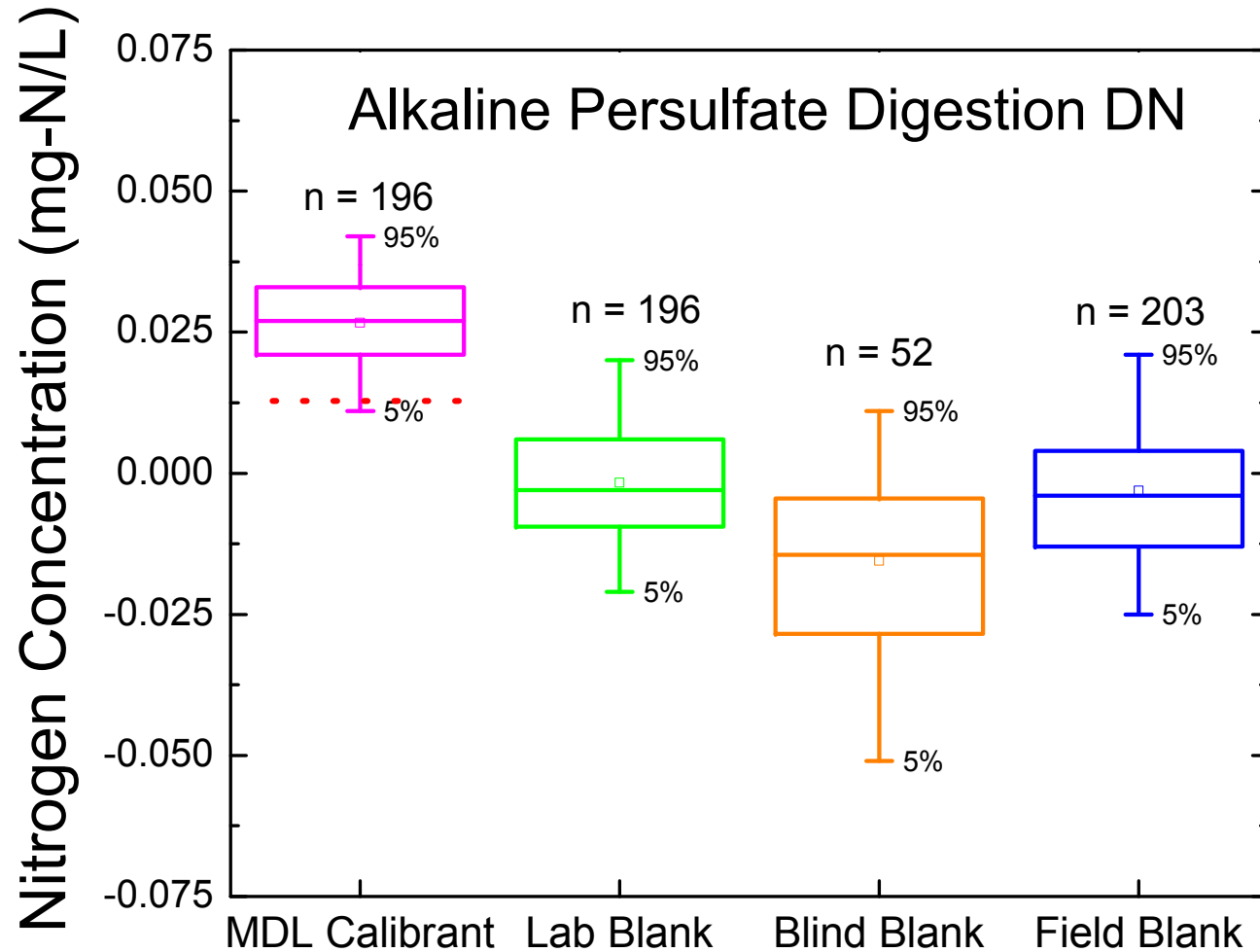
CFA cadmium-reduction and DA enzyme-reduction nitrate concentration data pairs plotted about the line of equal relation



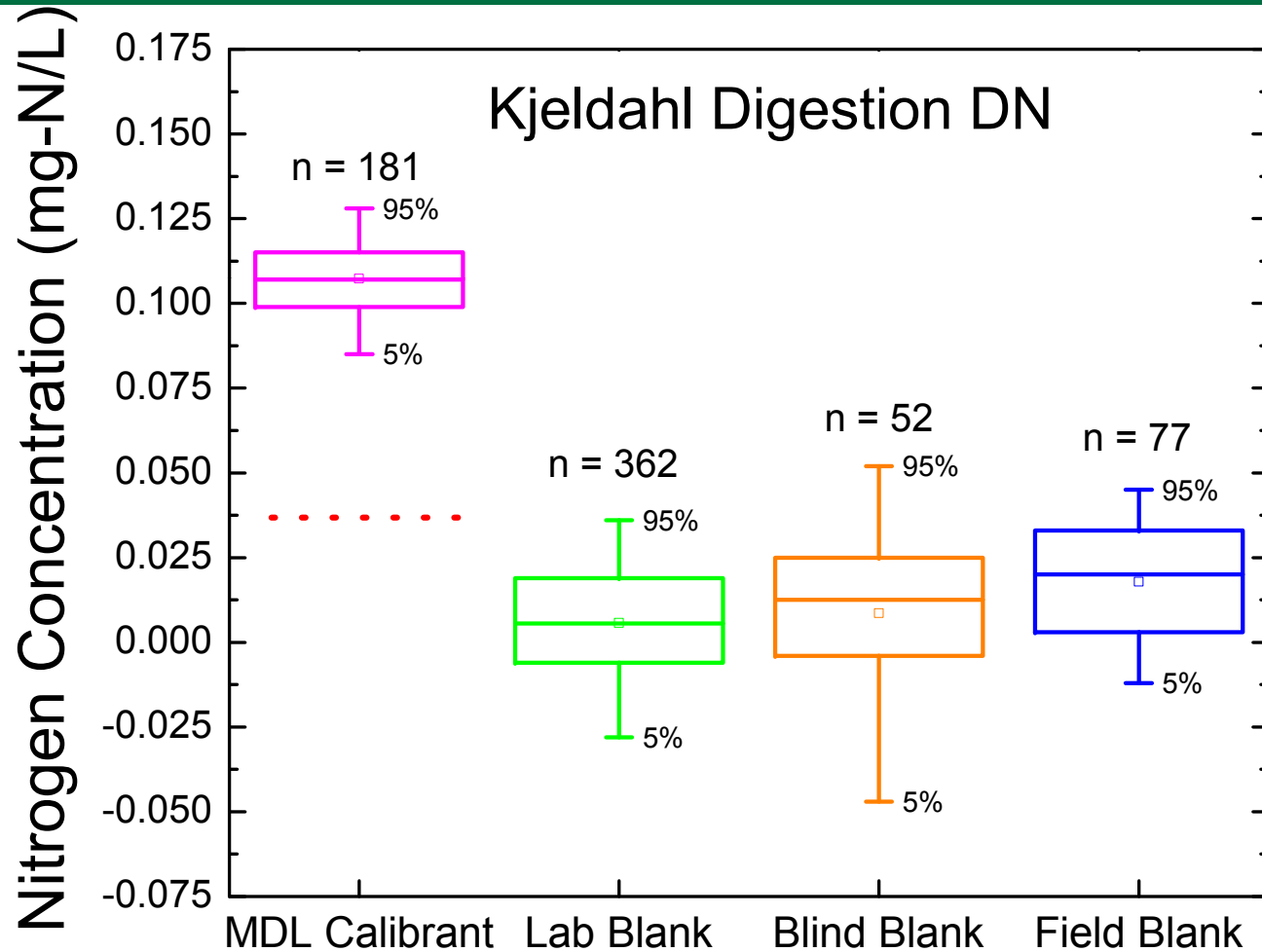
CFA cadmium-reduction and DA enzyme-reduction low-level nitrate concentration data pairs plotted about the line of equal relation



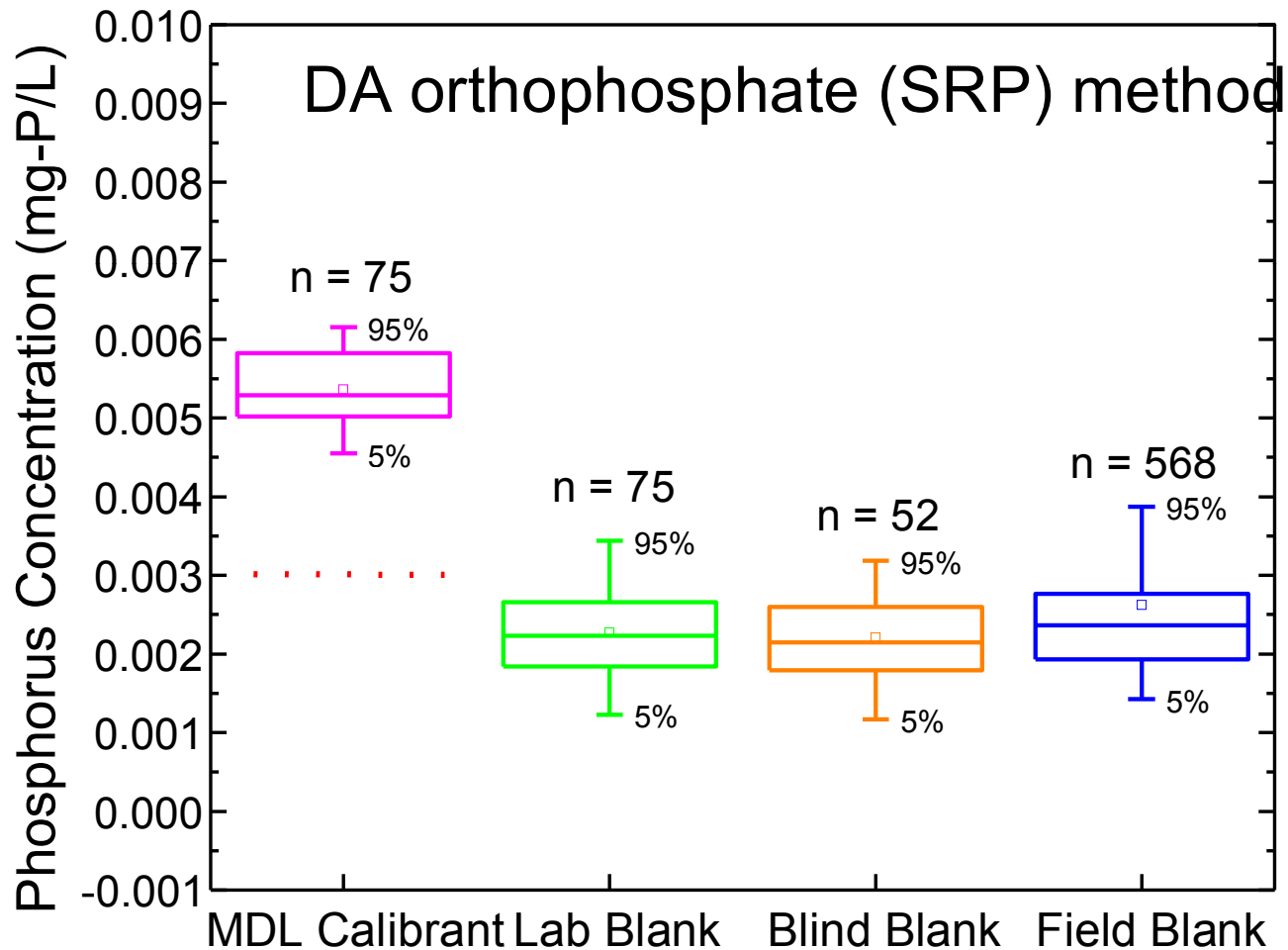
NWQL 2010 Alkaline-persulfate-digestion Dissolved Nitrogen (Continuous-flow Analyzer; determined as nitrate)



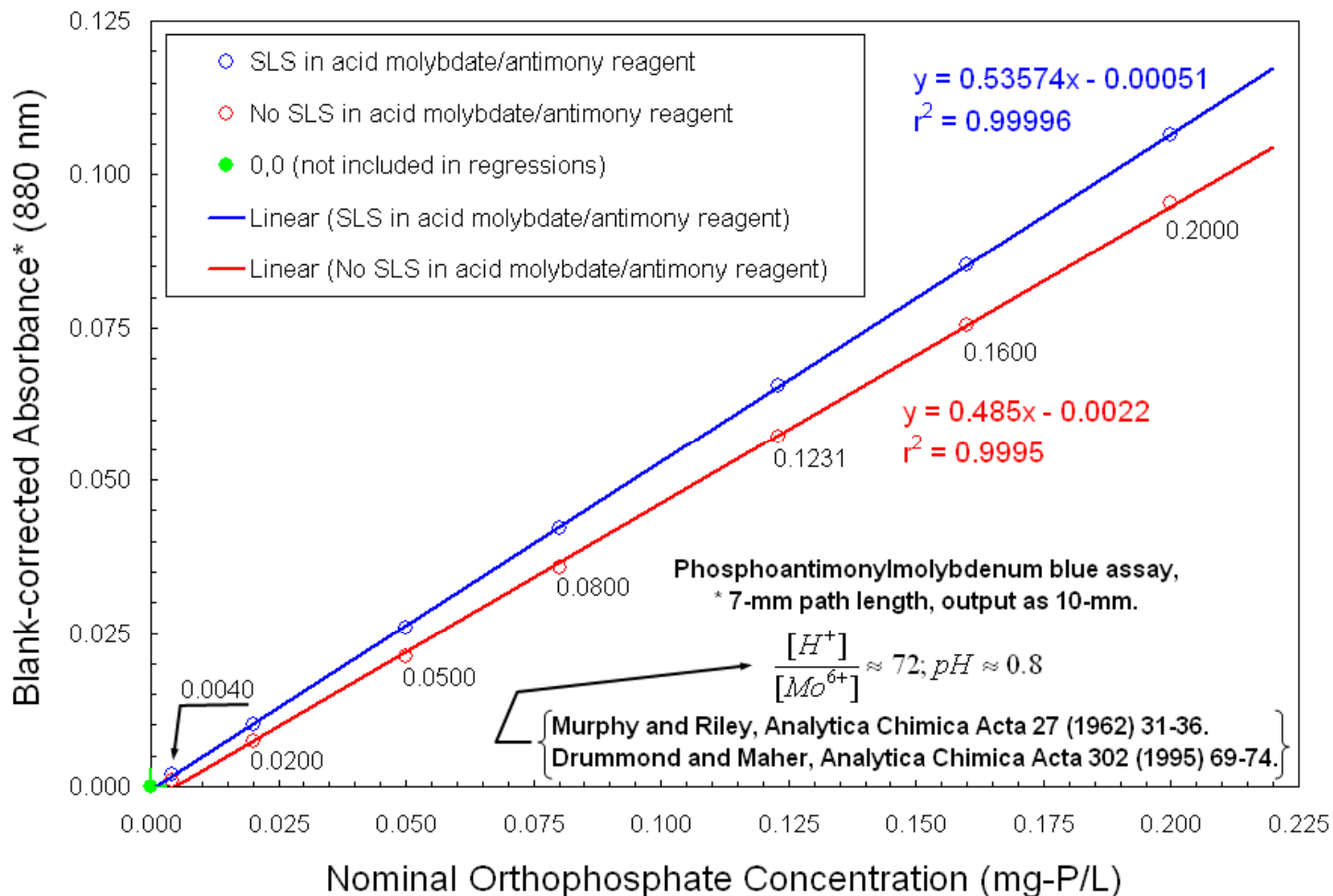
NWQL Kjeldahl Nitrogen 2010 (ammonium + organic nitrogen; continuous-flow analyzer; determined as ammonium)



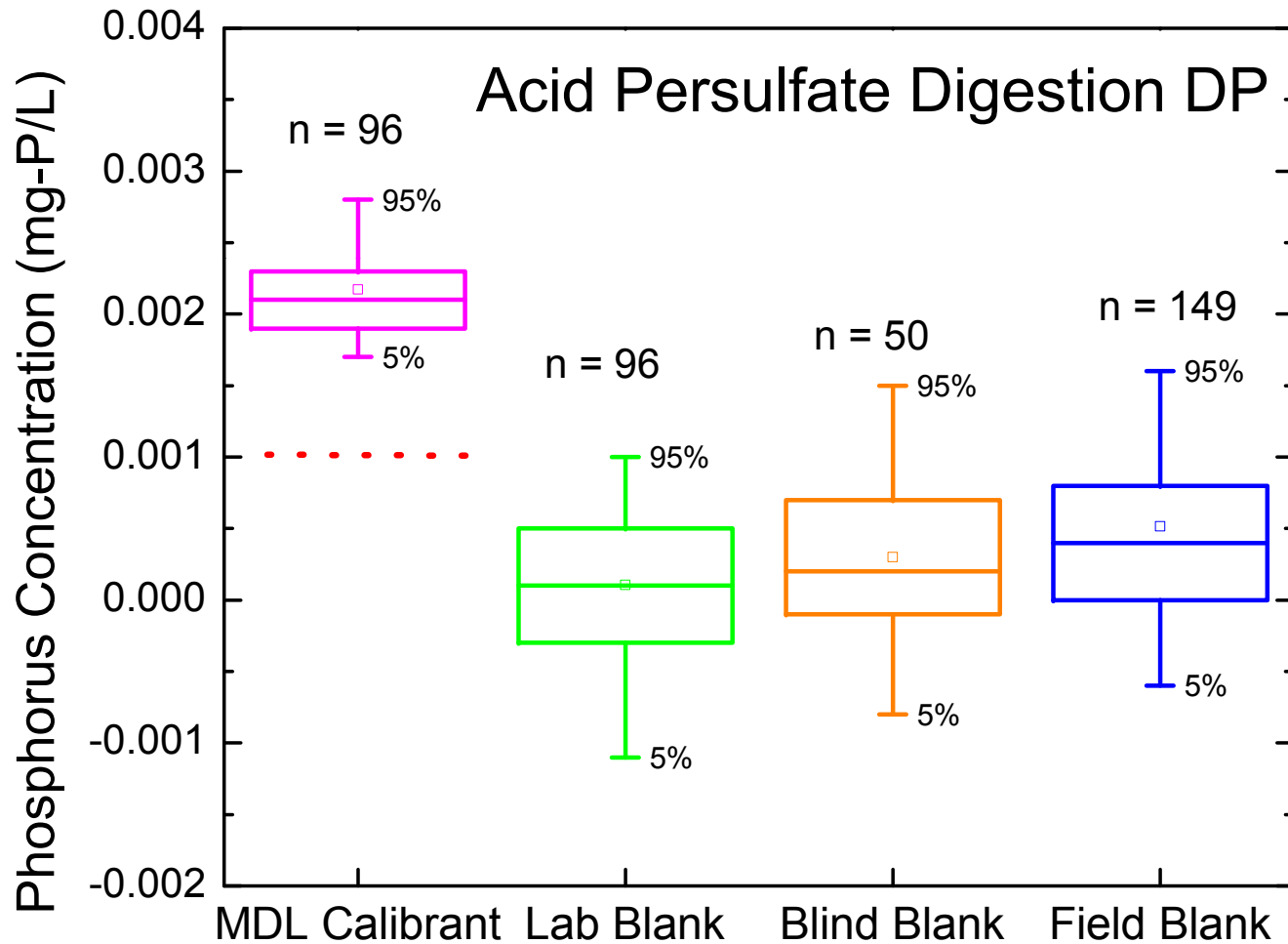
NWQL Orthophosphate Analyses 2010 (Discrete Analyzer)



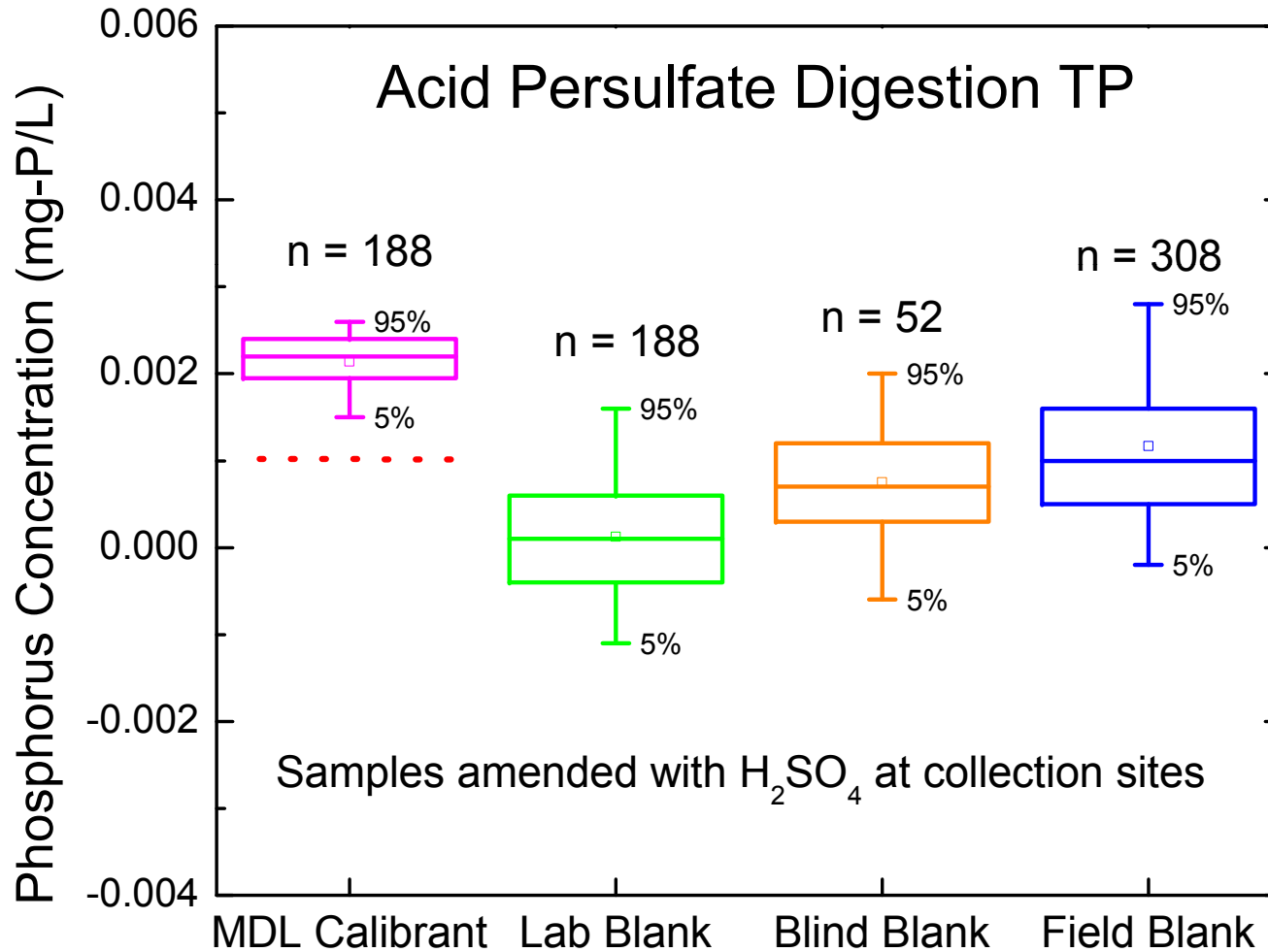
Murphy & Riley phosphate assays need sodium lauryl sulfate (SLS) even on discrete analyzer platforms



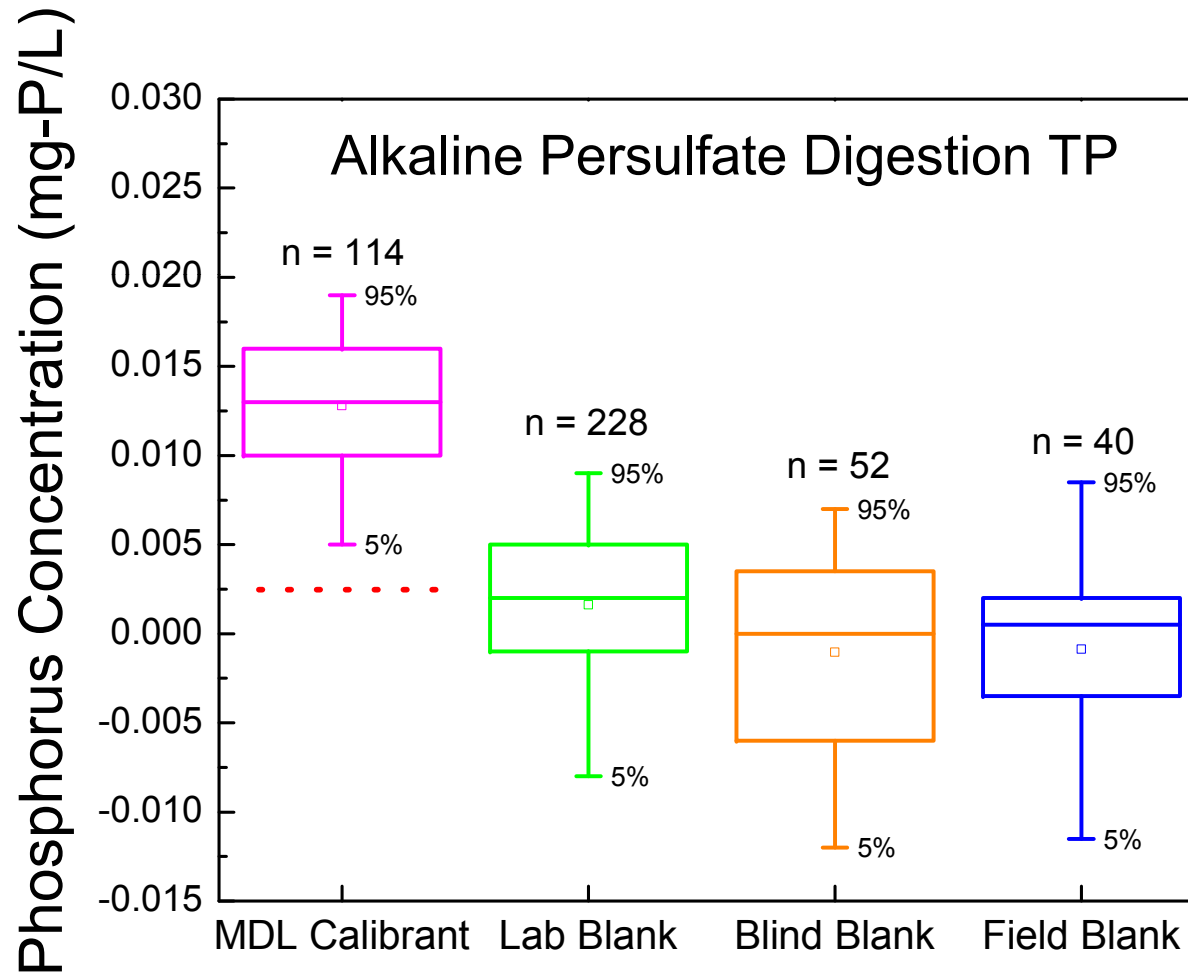
NWQL Low-level DP 2010 (Continuous-flow Analyzer)



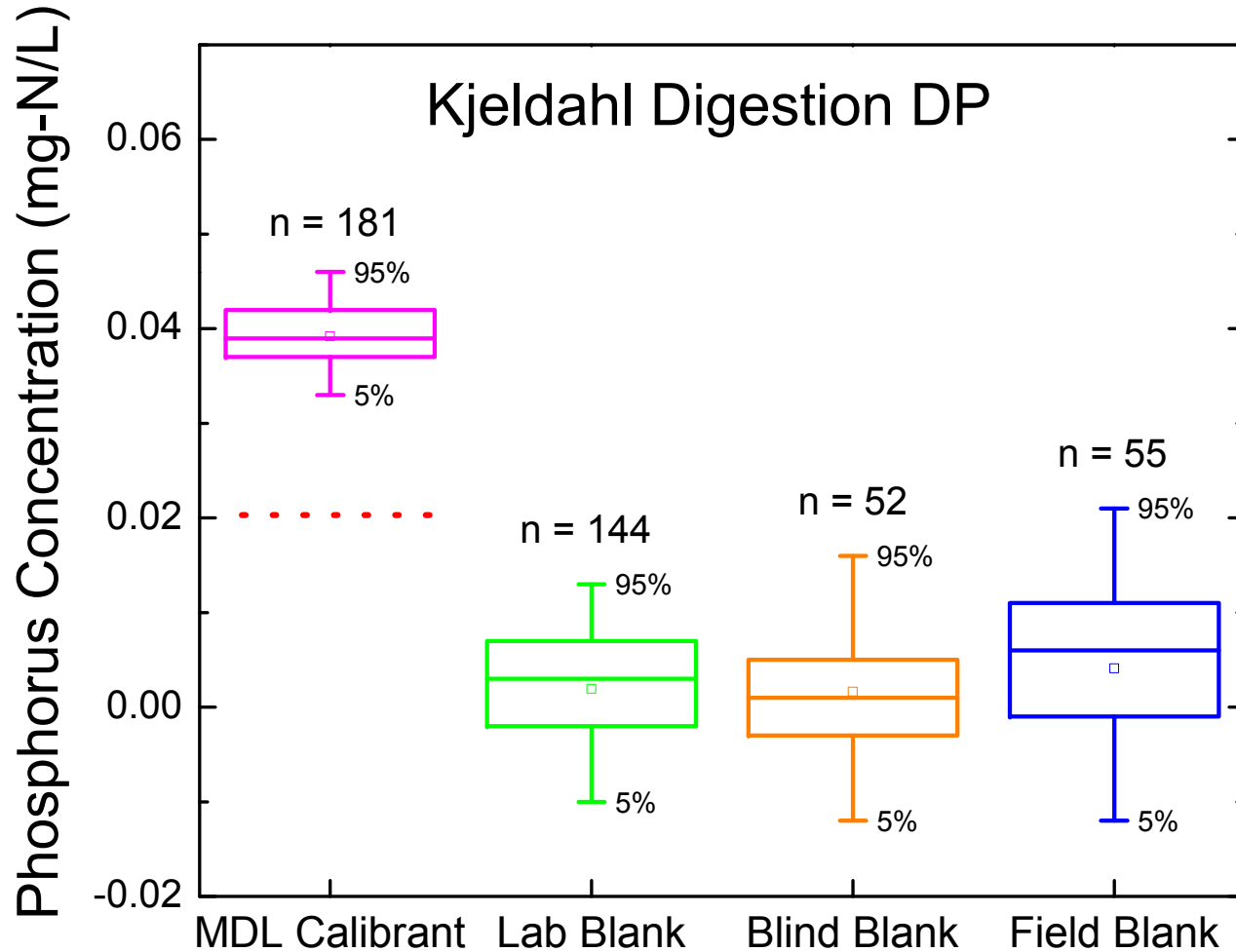
NWQL Low-level TP 2010 (Continuous-flow Analyzer)



NWQL Alkaline-persulfate-digestion DP 2010 (Continuous-flow Analyzer; determined as orthophosphate)



NWQL Kjeldahl Phosphorus 2010 (Continuous-flow analyzer; determined as orthophosphate)



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NWQL Detection limit summary

- **Nitrite: 2 $\mu\text{g NO}_2^-$ -N/L**
 - With lower calibration range, reagent and field blank limited at about 0.5 ppb
- **Orthophosphate (SRP): 4 $\mu\text{g PO}_4^{3-}$ -P/L**
 - 2 $\mu\text{g PO}_4^{3-}$ -P/L w/ SLS in acidic molybdate/antimony reagent; reagent and field blank limited at about 1 ppb)
- **Acid-persulfate-digestion DP and TP: 2 $\mu\text{g-P/L}$**
 - With lower-P persulfate reagent, perhaps 1 ppb before field blank limited
- **Alkaline-persulfate-digestion DP and TP: 10 $\mu\text{g-P/L}$**
 - Lower-P persulfate reagent needed
- **Kjeldahl-digestion DP and TP: 20 $\mu\text{g-P/L}$**
 - Reagent blank limited
- **Ammonium: 10 $\mu\text{g NH}_4^+$ -N/L**
 - Field and reagent blank limited
- **Standard- (low-) range Nitrate: 20 (10) $\mu\text{g NO}_3^-$ -N/L**
 - Field and reagent blank limited at about 5 ppb
- **Alkaline-persulfate-digestion DN and TN: 20 $\mu\text{g-N/L}$**
 - If nitrate could be removed prior to digestion to permit lower calibration range and lower-N persulfate reagent used, field and reagent blank limit at about 5 ppb
- **Kjeldahl-digestion nitrogen: 50 $\mu\text{g-N/L}$**
 - Reagent blank limited

Acknowledgements

- **TNI and NEMC**
 - Earl Hansen
 - Jerry Parr
- **USGS Office of Water and National Water Quality Laboratory (NWQL) for financial support and encouragement of numerous ongoing method development and laboratory automation projects**
 - NWQL Nutrients Unit
- **Speakers to follow for their time and effort to attend and contribute to this session**