

Isotopic Radiological Analysis by ICP-MS

May 21, 2008

Perkin Elmer Users Group Meeting

RT Preston
BJ Maes

Radiation Protection Sample Diagnostics (RPSD) Laboratory

Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.



Objective

Rapid Determination
of U-235, U-236, and U-238 in
Urine/Water/Soil/Filters/Vegetation

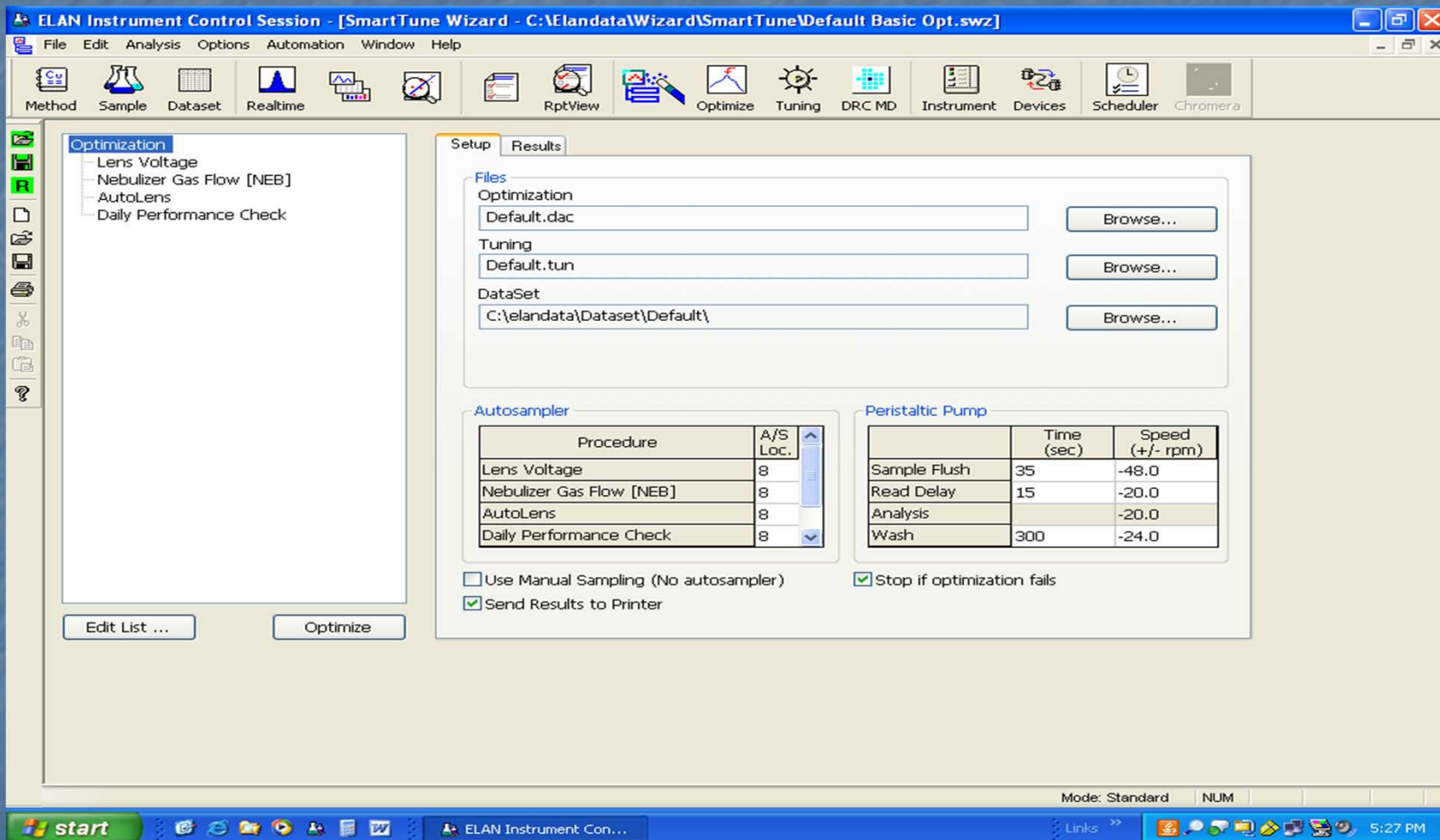
Overview

- ❑ ELAN DRC II with cyclonic chamber
- ❑ DOELAP Accredited for Isotopic Uranium in urine (only three laboratories in the USA)
- ❑ Proficiency testing through MAPEP program (soil, water, filter)
- ❑ In-Vitro results are reported within 24-hours

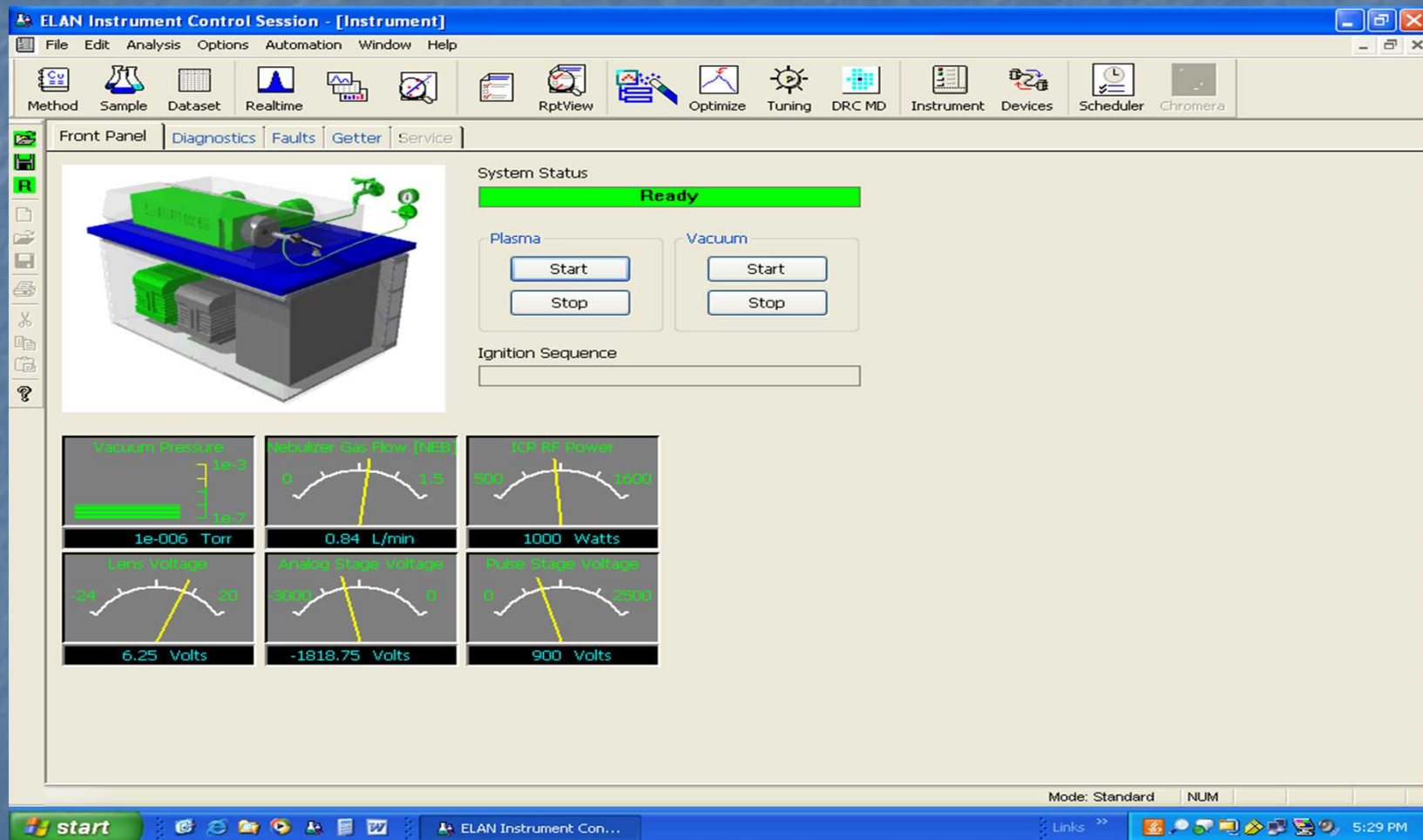
Elan DRC II



Software



Software



Uranium Minimum Detectable Concentration (MDC)

	Filter ($\mu\text{g}/\text{F}$)	Water ($\mu\text{g}/\text{L}$)	Soil ($\mu\text{g}/\text{g}$)	Urine ($\mu\text{g}/\text{L}$)
U-235	0.000238	0.00509	0.000999	0.00584
U-238	0.00104	0.0162	0.01498	0.01621
U-236	0.00021	0.00495	0.00101	N/A

Total Uranium – 0.01624 $\mu\text{g}/\text{L}$ (MDC), 0.00812 $\mu\text{g}/\text{L}$ Detection Level (DL)

Radiological Capability

- ❑ Isotopes with long half-lives yield required detection limits
 - ❑ Not feasible for U-232 ($T_{1/2} = 74$ years)
- ❑ Hydrides not formed for isotopes of interest
- ❑ No mass interferences
- ❑ Potential matrix interferences exist
 - ❑ Presence of dissolved solids
 - ❑ Possible solution - smaller aliquots and dilutions

Specific Activity vs Minimum Detectable Activity

Isotope	Specific Activity (Ci/g)	T _{1/2} (years)	Atomic Mass Unit (AMU)	Total U MDA (pCi/L)
Y-90	5.45E+05	7.31E-03	90	8.86E+09
Sr-85	2.38E+04	1.78E-01	85	3.86E+08
Fe-55	2.42E+03	2.70E+00	55	3.93E+07
Sr-90	1.39E+02	2.86E+01	90	2.27E+06
Ni-63	5.69E+01	1.00E+02	63	9.24E+05
U-233	9.68E-03	1.59E+05	233	1.57E+02
Pu-242	3.95E-03	3.76E+05	242	6.41E+01
U-236	6.50E-05	2.34E+07	236	1.06E+00
U-235	2.17E-06	7.04E+08	235	3.53E-02
U-238	3.38E-07	4.47E+09	238	5.48E-03

Aliquot/Chemistry

Matrix	Method	Aliquot	Dilution
Urine	Direct	0.5mL	Up to 10mL 0.1M HNO ₃
Water	Direct	1.0mL	Up to 10mL 0.1M HNO ₃
Soil	Microwave Dissolution	0.25 – 1.0g	Final dilution ~1.0L 0.1M HNO ₃
Filter	Microwave Dissolution	Entire Filter	Final dilution ~1.0L 0.1M HNO ₃

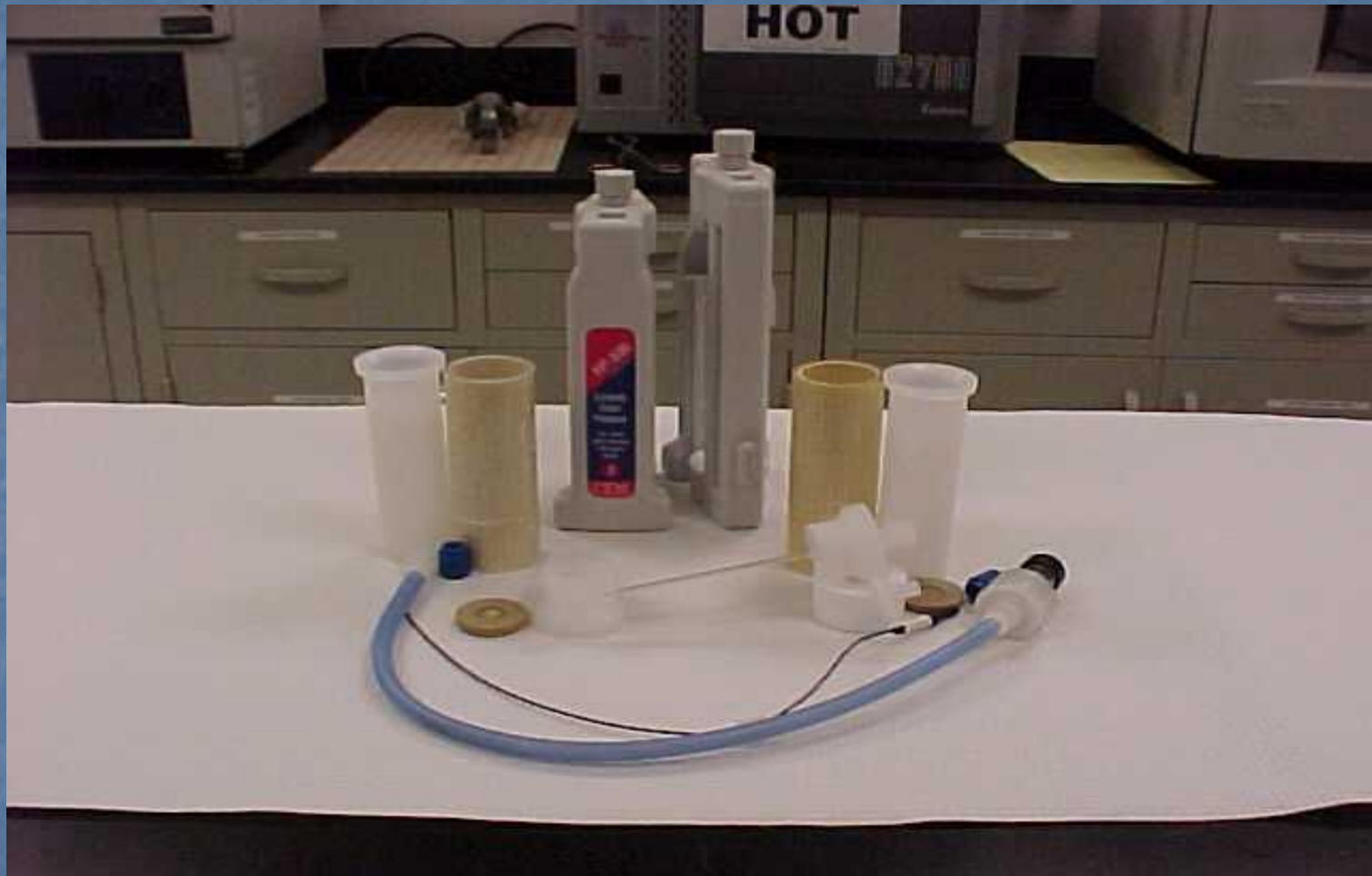
Microwave Digestion System



Microwave 14 Vessel Load



Control Vessel/Sample Vessel



Control Vessel/Sample Vessel



Soil Samples ready for Acid Load

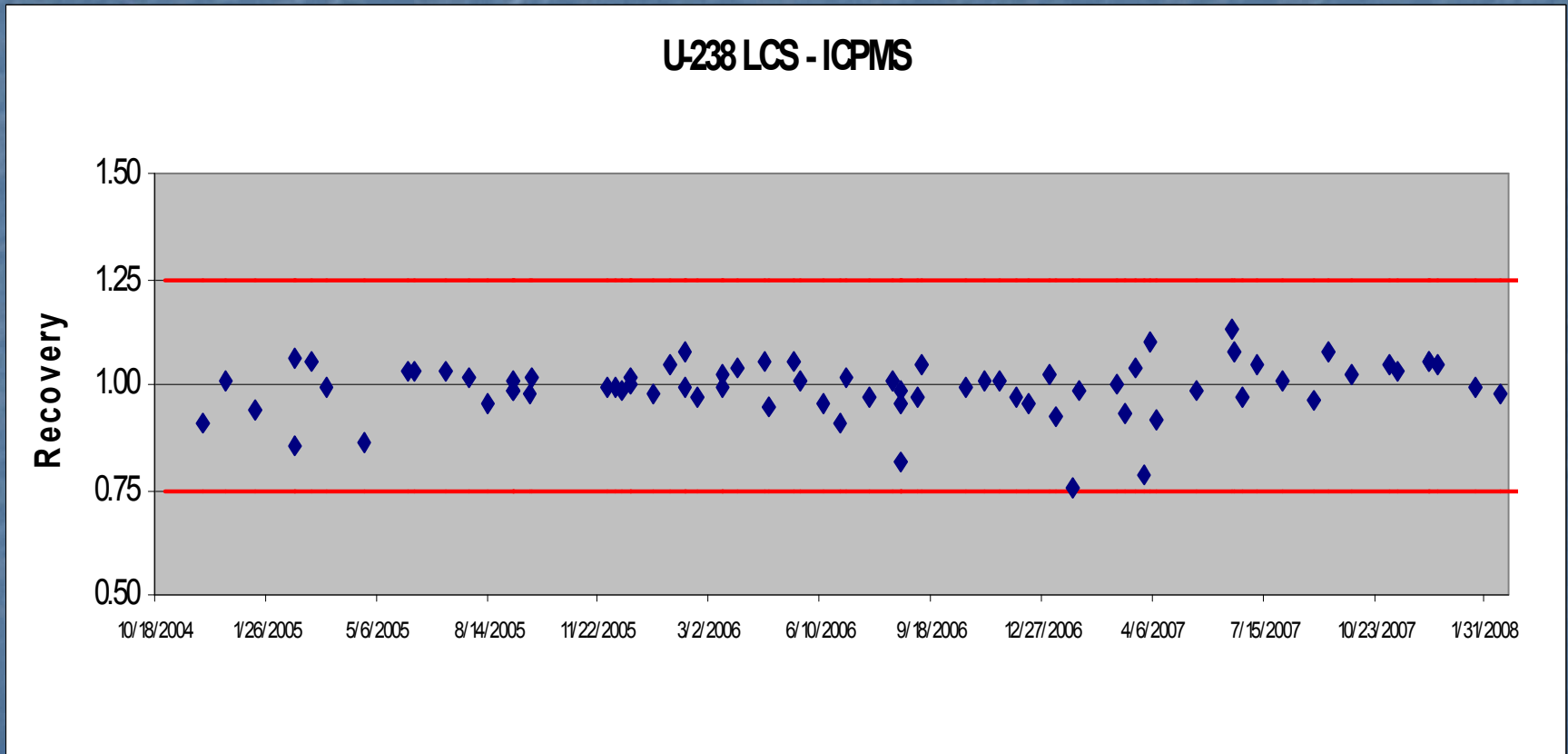


Quality Control

- ❑ Calibration Standards
 - ❖ U-Nat (0.5 – 55 ug/L)
- ❑ Laboratory Control Standards
 - ❖ LCS
 - ❖ ICV/CCV
- ❑ Tracers
 - ❖ Ho internal system tracer
 - ❖ U-233 internal sample tracer
- ❑ Blanks
 - ❖ Reagent Blank
 - ❖ Matrix Blank
 - ❖ CCB
- ❑ Performance Check Standard (PE 5030)

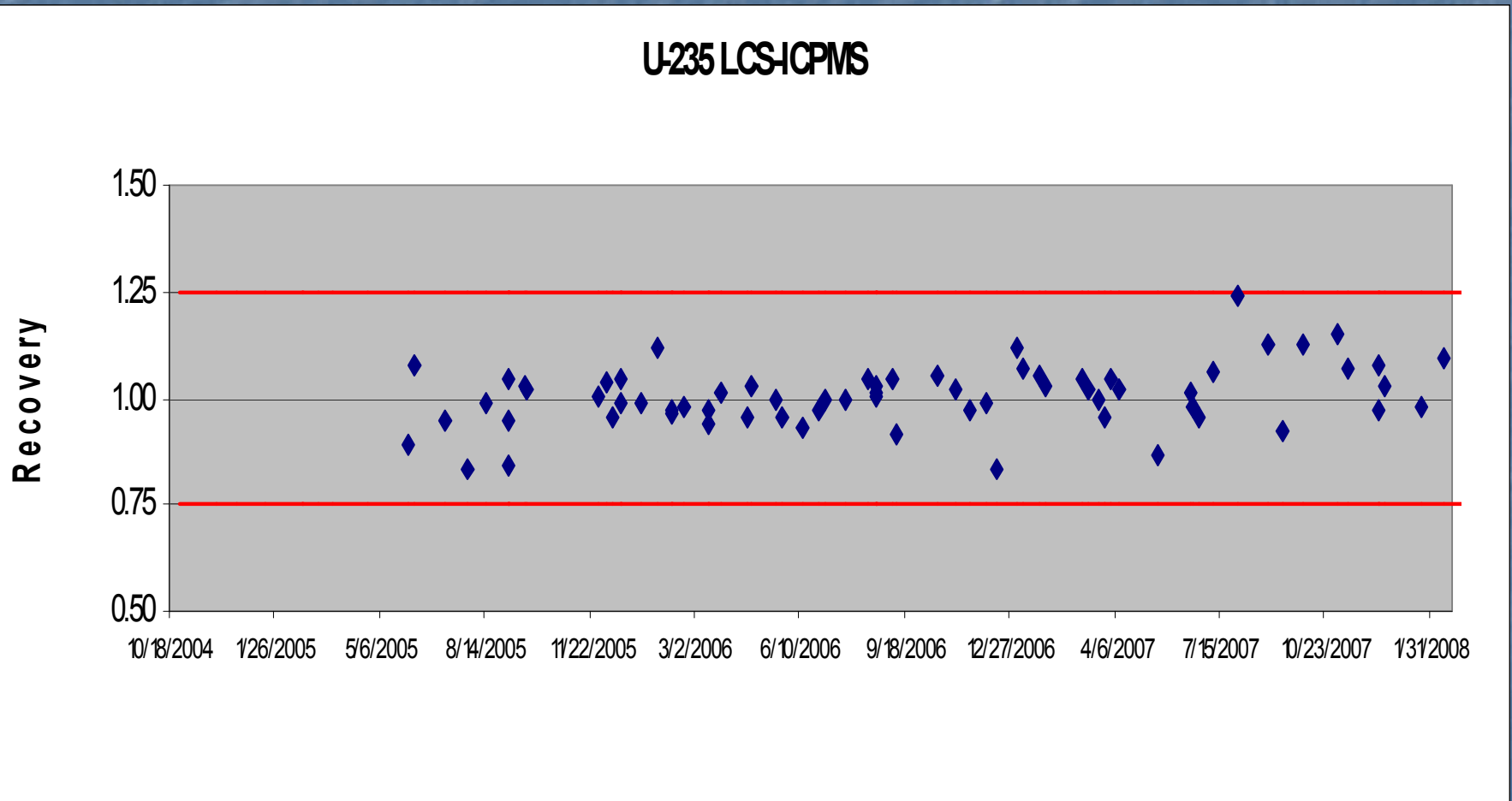
Tracking Charts

Lab Control Sample (Urine)



Tracking Charts

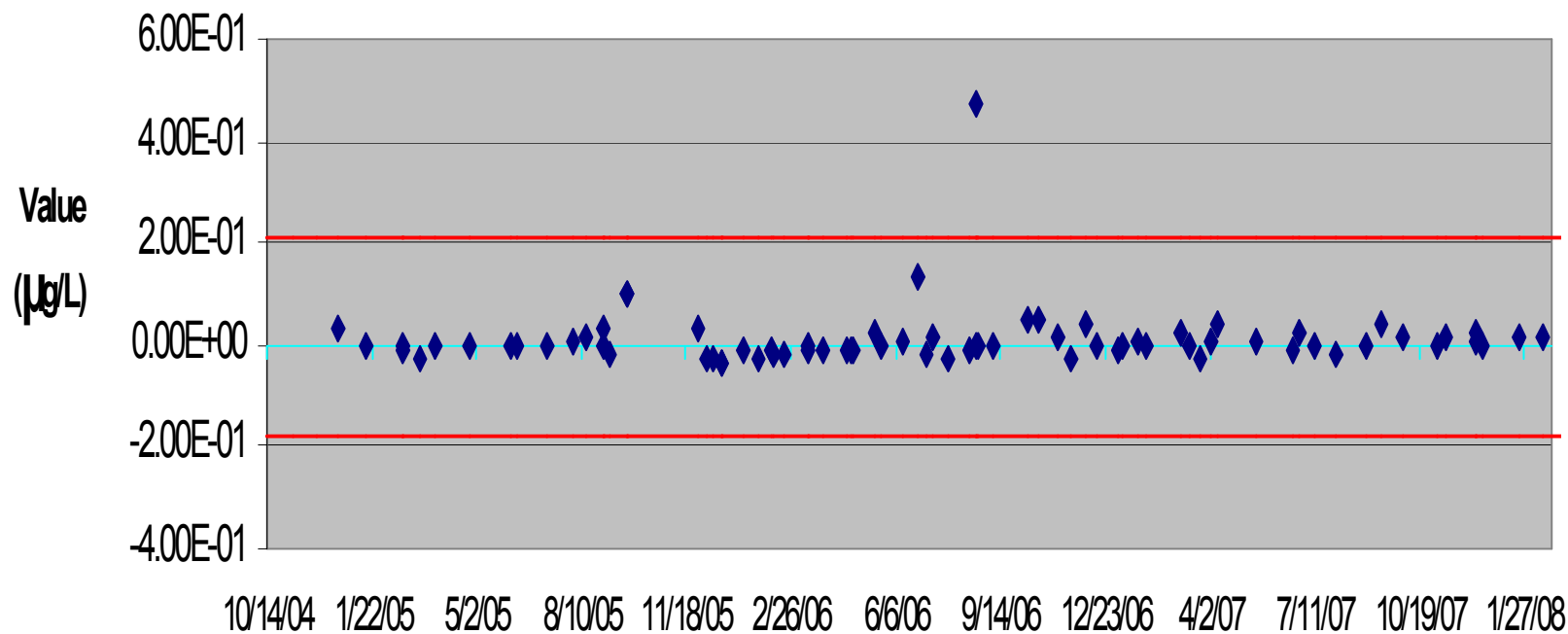
Lab Control Sample (Urine)



Tracking Charts

Blank Sample (Urine)

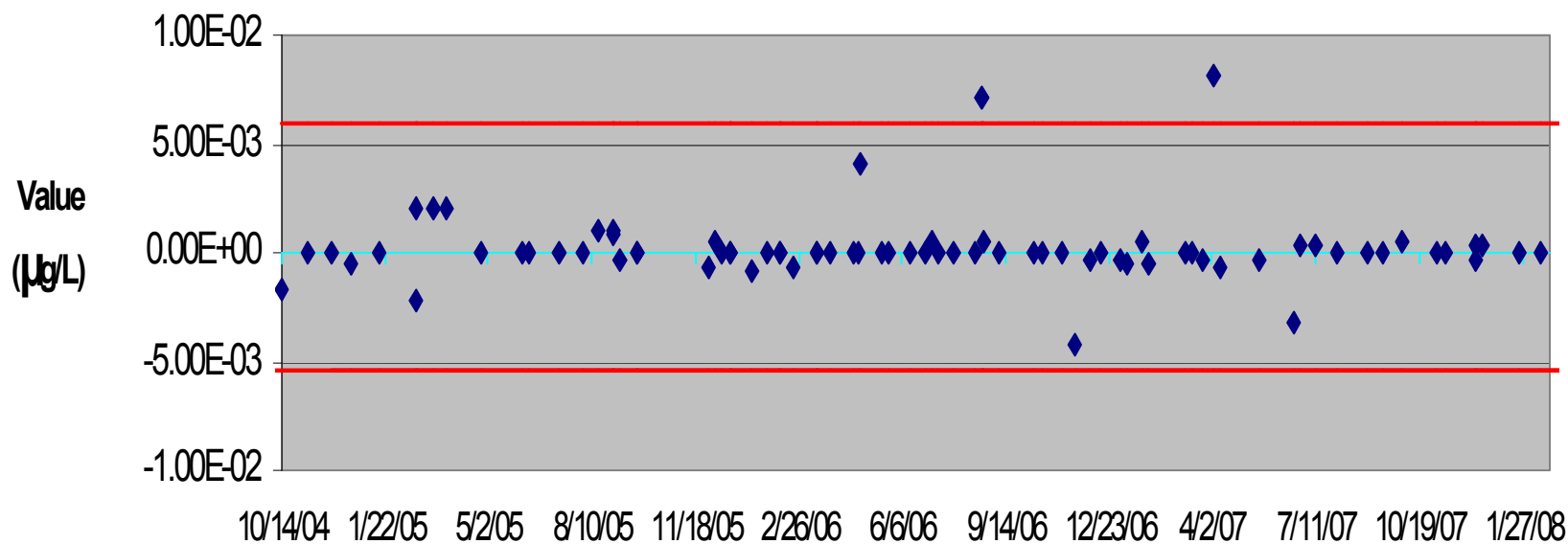
U-238 Blank Samples - ICPMS



Tracking Charts

Blank Sample (Urine)

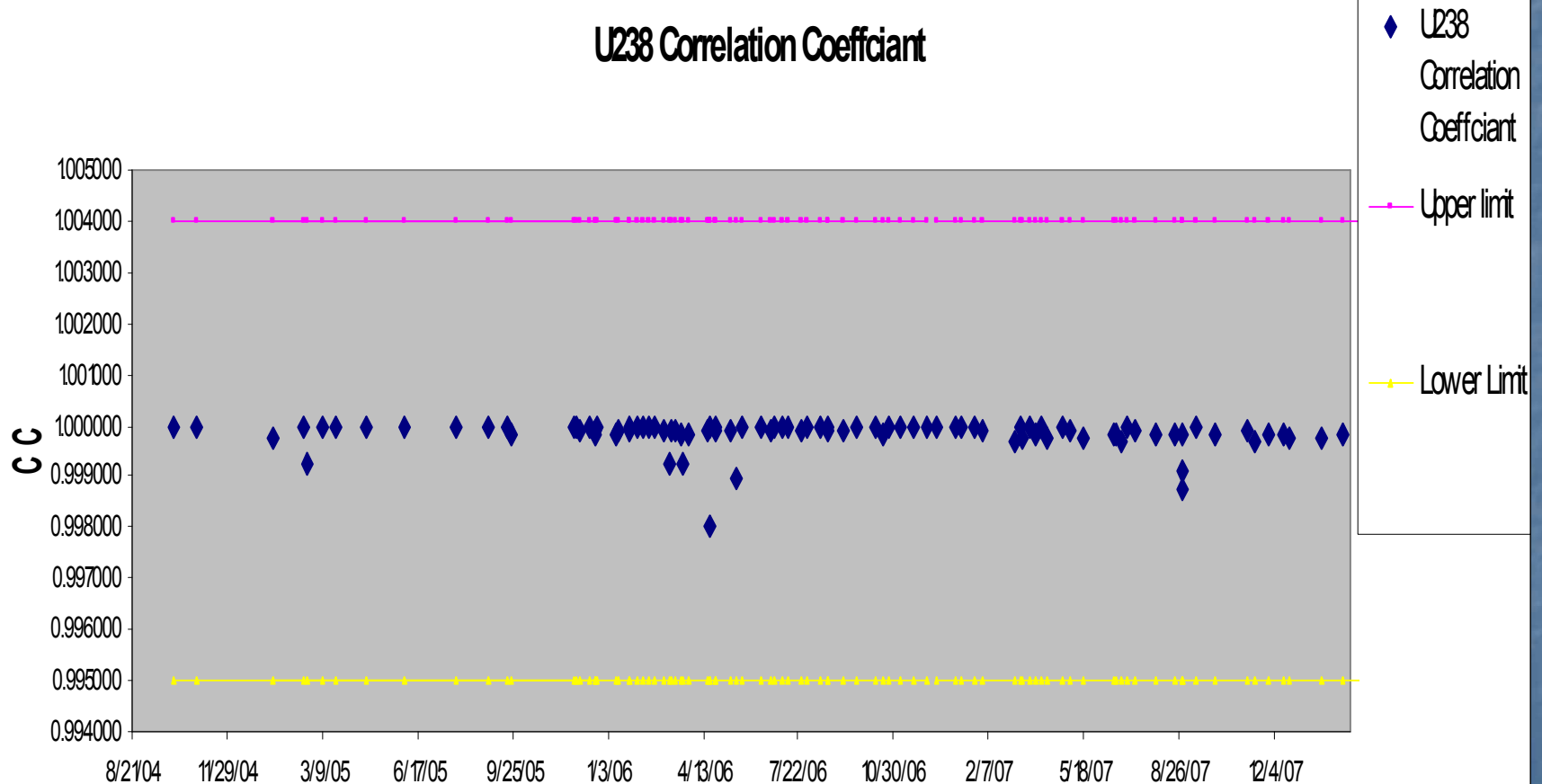
U-235 Blank QC - ICPMS



Tracking Charts

Correlation Coefficient

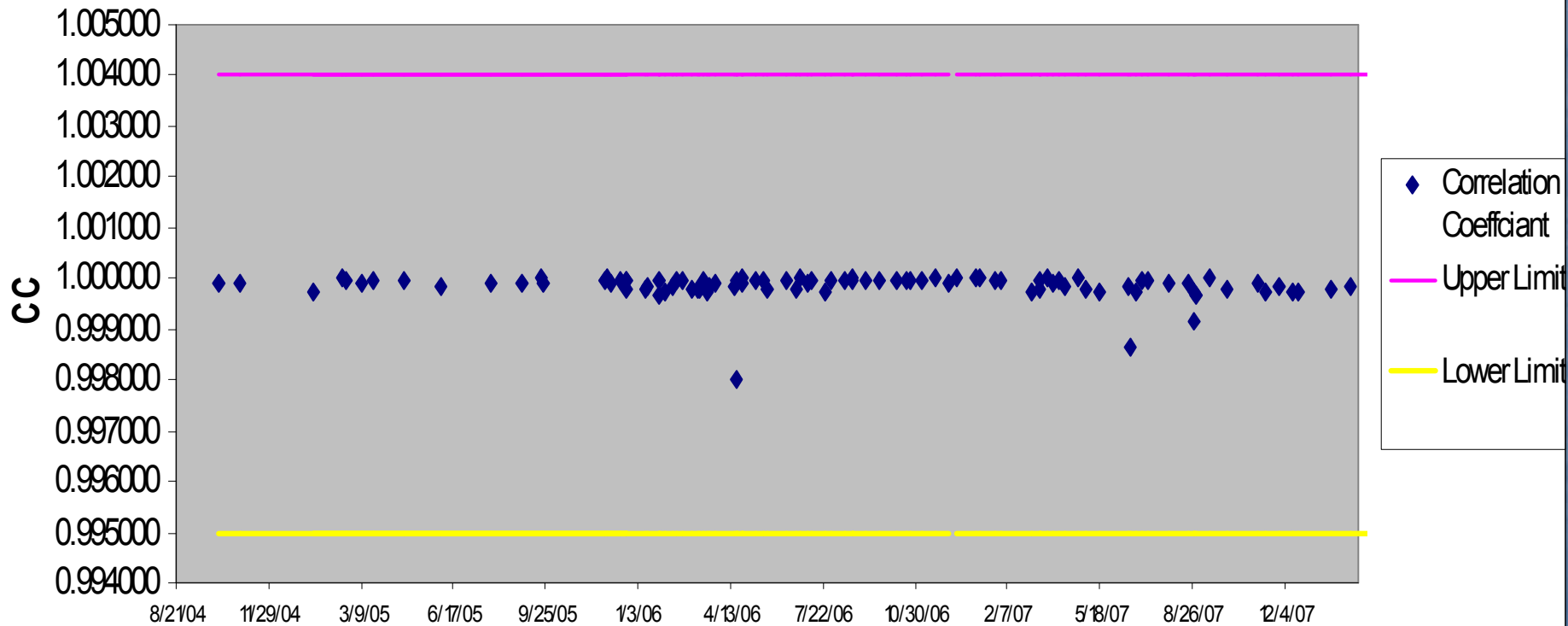
U238 Correlation Coefficient



Tracking Charts

Correlation Coefficient

U235 Correlation Coefficient



Additional Tracking Charts

- Vacuum Pressure
- Daily Checklist
- Daily Performance Tests
- DAC Values
- Uranium Intensities
- Uranium RSD

Procedure

- ❑ Sample log-in
- ❑ Preparation
 - ❖ Equilibration
 - ❖ Homogenization
 - ❖ Chemistry (if applicable)
- ❑ Set tubing
- ❑ Start ICP-MS
- ❑ Warm-up: 1-2 hours
- ❑ Preparation
 - ❖ Calibration Standards
 - ❖ LCS (including ICV/CCV)
 - ❖ Blanks
- ❑ Aliquot Samples

Urine Samples



Preparation of Samples/QC/Calibration Standards/Blanks



Standards



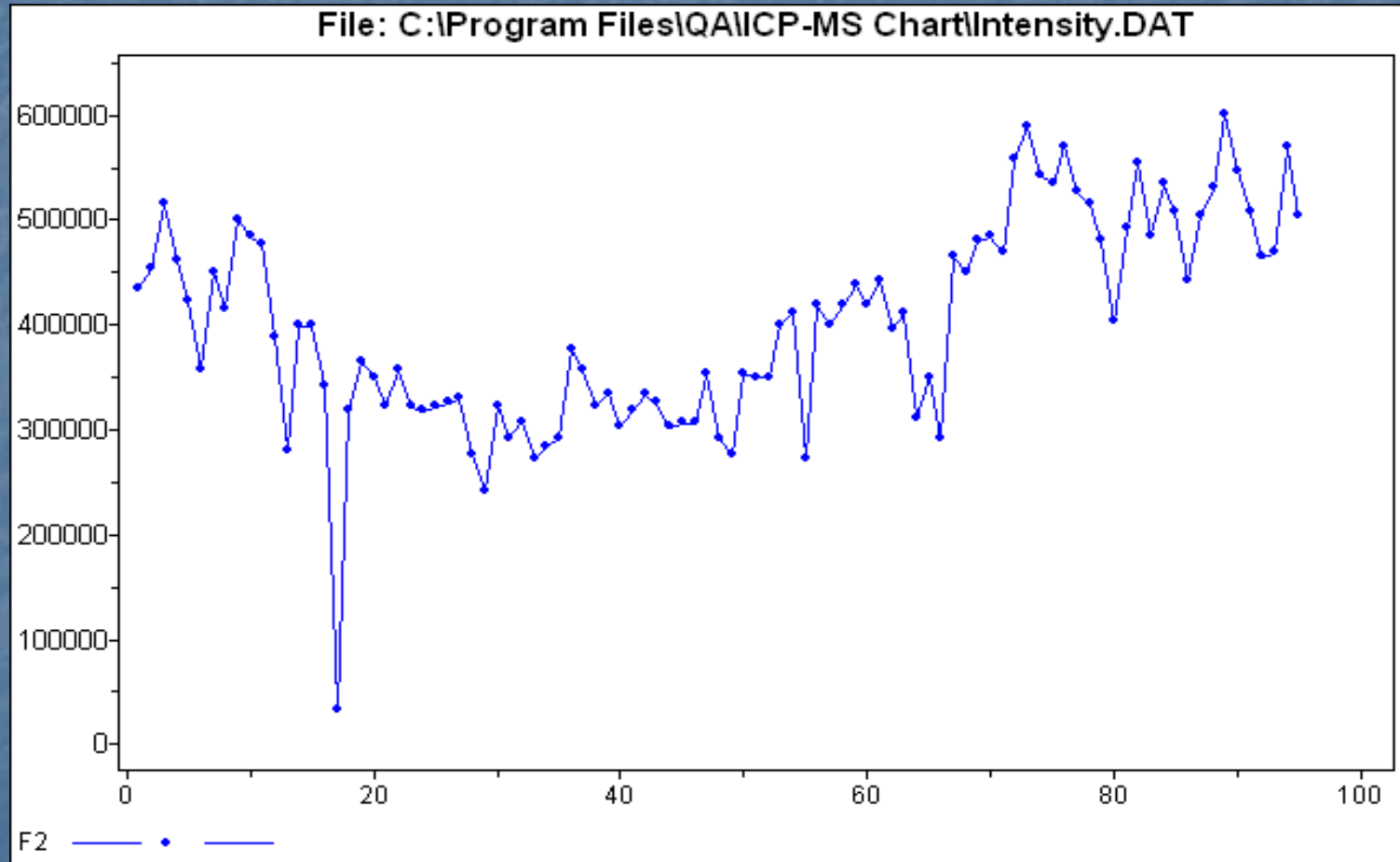
ICP-MS Analysis

- Smart Tune
 - ❖ Auto Lens
 - ❖ Daily Performance Check
 - ❖ Lens Voltage
 - ❖ Nebulizer Flow
- Set-up Method, Sample Batch set, Data Set workspace
- Run batch using specified method
- Prepare RPSD datasets and client reports
- Send reports to the RPSD Project Lead for Review and Signature
- Distribution of results

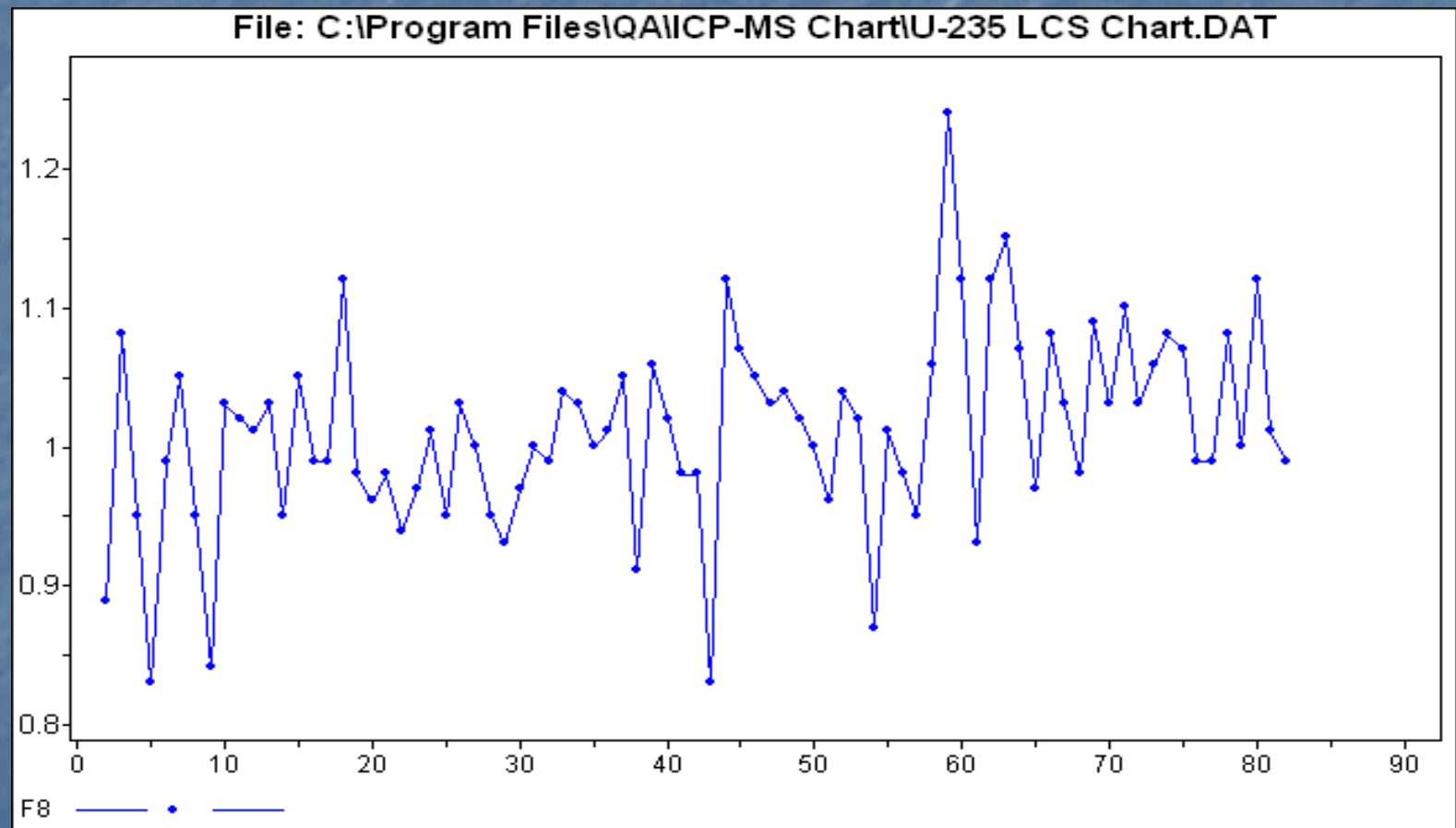
Currently in Development

- ❑ Utilize Labworks and generate a Crystal Report
 - ❖ Use for log-in
 - ❖ Use for tracking QC and other charts
 - ❖ Generate a more professional client report
 - ❖ Interface with SNL/RPSD database(s)
 - ❖ Cut down on >90% of hand entries

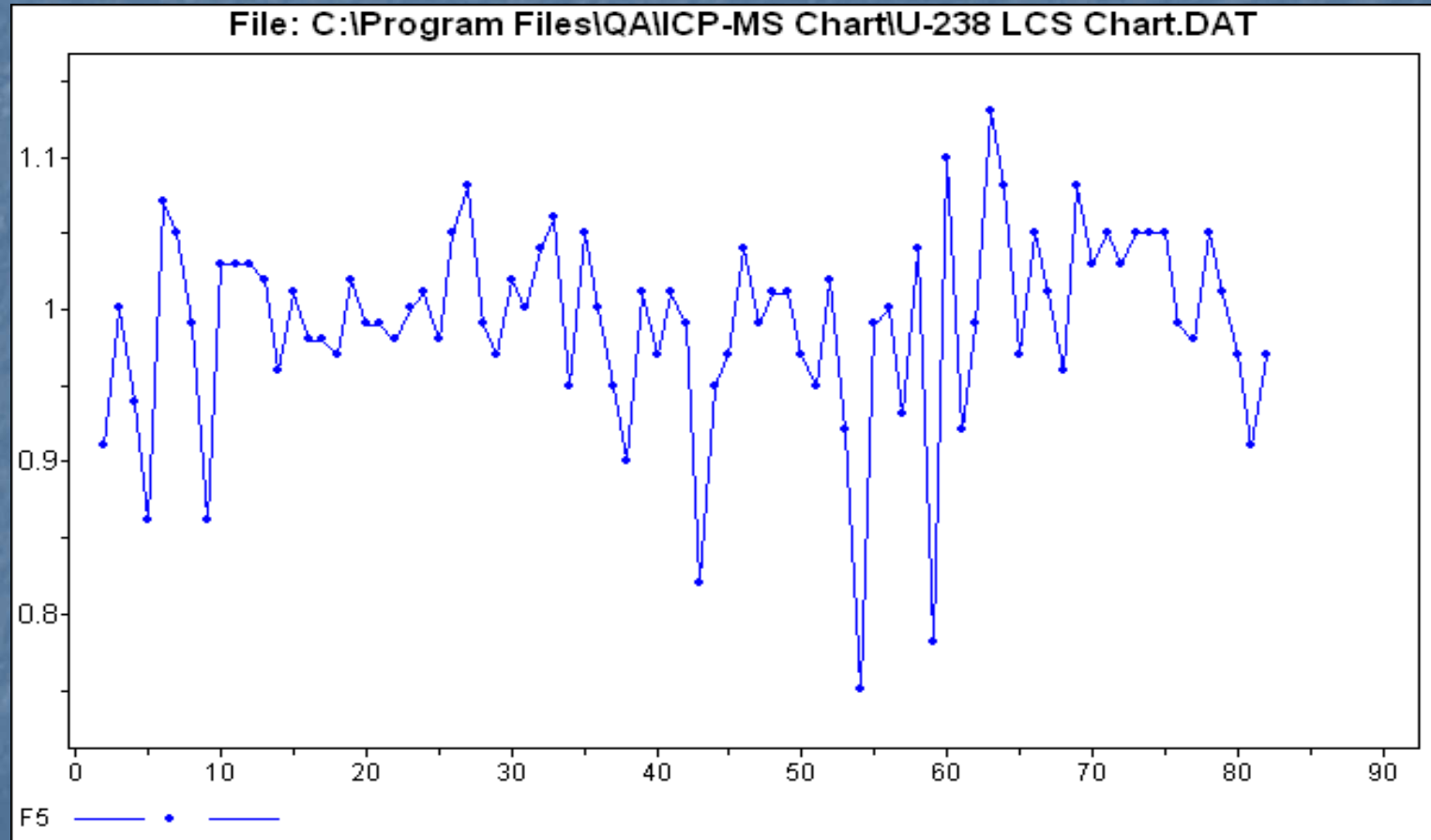
Labworks Uranium Intensity Chart



Labworks Uranium-235 LCS Chart



Labworks Uranium-238 LCS Chart



Labworks Crystal Sample Report

Customer: MAES B.

Customer Address:

MAES B.

4121

MS 1103

MAES

The samples were received in good condition and prepared under using procedure RPSD-09-07, "ICP-MS Sample Preparation." All related QC passed requirements. Refer to QC report.

RPSD Batch ID: G00588
Survey / COC # 050008



LOCKHEED MARTIN

Radiological Protection Sample Diagnostics

P.O. Box 5800 MS1103

Albuquerque, New Mexico 87185-1103

Phone: (505) 844-4069

Sample ID#: G0058801 Method RPSD-09-07 Date Analyzed 4/22/08 Analyst: BJM

Customer ID#: 001

Parameter	Result	Units
Aliquot	0.50	mL
U_235 LCS Recovery	96.82	%
U_235 Matrix Blank	0.00077	ug/L
U_235 Method Blank	0.00016	ug/L
U_238 LCS Recovery	97.27	%
U_238 Matrix Blank	0.02212	ug/L
U_238 Method Blank	0.00874	ug/L
U-235 CL	4.95E-05	ug/L
U-235 Concentration	0.00945	ug/L
U-235 MDA	9.904E-05	ug/L
U-238 CL	0.000462	ug/L
U-238 Concentration	1.277	ug/L
U-238 MDA	0.0009241	ug/L

End of 001

Sample ID#: G0058802 Method RPSD-09-07 Date Analyzed 4/22/08 Analyst: BJM

Customer ID#: 002

Parameter	Result	Units
Aliquot	0.50	mL
U-235 CL	2.74E-05	ug/L
U-235 Concentration	0.00053	ug/L
U-235 MDA	5.484E-05	ug/L
U-238 CL	6.64E-05	ug/L
U-238 Concentration	0.01296	ug/L
U-238 MDA	0.0001327	ug/L

Report Date: 5/12/2008 10:16:36

Page 1 of 2

r:\rpsd\labworks\rpsdlwdata\CRYSTAL\RPSD_TEST.rpt

RPSD Batch ID: G00588	
End of	002
COMMENTS:	
RPSD Analyst(s)	Date
RPSD Project Lead	Date
Report Date: 5/12/2008 10:16:37	
r:\rpsd\labworks\rpsdlwdata\CRYSTAL\RPSD_TEST.rpt	

Summary

- ❑ Rapid
- ❑ Accurate
- ❑ Low Detection Concentration
- ❑ Minimal waste generation

Issues

- ❑ Extensive Maintenance
- ❑ Urine Matrix interference with Ho internal tracer
- ❑ Cannot use for shorter lived isotopes
 - ❖ Results in unacceptably high MDA
 - ❖ Can potentially use in an emergency situation
- ❑ Hydride creation for Pu-239 (interference U-238)

Potential Future Use

- ❑ Perform radiochemistry separation allowing for analysis of Pu-239/238
- ❑ Explore and develop a rapid method to do direct analysis in urine for Isotopic Pu, Isotopic Am, Isotopic Th and Isotopic Np using different reaction cell gases

Many Thanks

- ❑ Amir Mohagheghi – Original development
- ❑ Sonoya Shanks – Continuing development/QC
- ❑ Robert Reese – Photography and Intuition
- ❑ Mike Buvinghausen – Internal tracer modifications
- ❑ Joe Zigmond – Labworks assistance
- ❑ Brenda Maes - (co-author)
- ❑ Larry Gugliotta – Personality/Problem Solving