

MDN
Annual QA Report

1997

FRONTIER GEOSCIENCES INC.

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MDN 1997 ANNUAL QA REPORT

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Quality Assurance Officer

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I. Introduction

The Mercury Deposition Network (MDN) has been analyzing weekly precipitation samples for total mercury since January of 1996 (contract period began in March, 1996). The study now incorporates 30 sites from around the United States, an increase of 9 since 1996. MDN is expected to add an additional 10 sites, bringing the total to 40 in 1998.

Bob Brunette took over the MDN project from Joe Tokos in March of 1997 and now coordinates all analytical processes from sample receipt to scientific reports. The Atmospheric laboratory technician processes incoming samples and analyzes them on a weekly basis, freeing Mr. Brunette up for client consulting, scientific reports, site liaison and project coordination.

Quality control is adhered to both in the laboratory and in the field. The MDN project saw an increase in quality control points from 301 to 444, which corresponds to the increase in number of sites in 1997 and demonstrates Frontier's commitment to quality assurance. The site liaison, Mr. Brunette, reviews weekly rain gauge charts, verifying chart dates and times. Site equipment failures or procedural errors are addressed quickly and sufficient replacement stocks of field supplies are maintained at Frontier.

Quality control protocols within the laboratory were overseen in 1997 by Frontier's Quality Assurance Officer, Toni GauthierDickey. Analysis records are kept on file in a fireproof cabinet and are readily available for review. In addition, Mr. Brunette prepares quarterly quality assurance reports, with quality control parameters listed in tabular format.

II. A General Description of Frontier's QA/QC

A. QA/QC

Quality Assurance (QA) is a system for ensuring that all information, data and interpretation resulting from an analytical procedure are technically sound, statistically valid and appropriately documented. Quality Control (QC) is the mechanism used to achieve quality assurance.

B. Data Quality Objectives

Data quality is achieved through Frontier's Data Quality Objectives (DQO's). Our DQO's consist of five components: precision, accuracy, representativeness, comparability and completeness (PARCC).

- Precision is a measure of data repeatability; it is measured by using sample replicates.
- Accuracy is a measure of how close the data is to the actual, or real value, and is measured by certified reference materials and matrix spikes.
- Representativeness is a measure of how typical a sample is compared to the sample population. It is achieved by accurate, artifact-free sampling procedures and appropriate sample homogenization.
- Comparability is a measure of how variable one set of data is to another.
- Completeness is a measure of how many data points collected are usable; Frontier strives for 95% completeness.

III. QC Procedures

A. Lab-Run Bottle Blanks

Lab sample bottle blanks are expected to be at or near the method detection limit (MDL). In cases where the blanks are significantly higher than the MDL, the situation is examined. Possible sources of contamination are looked for and identified. Once the problem has been found and corrected, the run continues. Lab blanks are control charted on an ongoing basis, helping to identify trends or anomalies.

The mean average for 1997 lab sample bottle blanks is 0.045 ng/L (n=21). The standard deviation is 0.019 ng/L. Numbers may be found in tabular format in Appendix A, while control charts are listed in Appendix B.

B. BrCl Reagent Blanks

BrCl reagent blanks are a measure of how much analyte may be found in the bromine monochloride used for oxidizing the samples. The BrCl reagent blanks help to narrow down possible sources of contamination.

The mean average for 1997 BrCl blanks is 0.143 ng/L (n=53). The standard deviation is 0.067 ng/L. Numbers may be found in tabular format in Appendix A, while control charts are listed in Appendix B.

C. Replicate Samples

A replicate sample is run with each analytical set. The relative percent difference (RPD) is calculated and is expected to be less than 25%. If the result is higher than 25% then the analysis is re-run. If the result is still higher than 25%,

then the problem is looked into and possible causes are identified and noted on the report.

The mean average for 1997 RPD's is 3.9% (n=152). The standard deviation is 4.52%. One result was out-of-control at 26.4 RPD. Two other sample replicates were run with that set with both results falling within acceptable quality control limits. Numbers may be found in tabular format in Appendix A, while control charts are listed in Appendix B.

D. Reference Samples

Reference samples are a way to compare the sample results with a known, certified value. This is a useful tool for comparing matrix spikes and for validating the analytical curve. The acceptance range for the reference samples is 75-125%. If the percent recovery lies out of this range the problem is identified, corrected and the instrument is recalibrated. If the percent recovery is within the acceptance range the analysis continues.

The mean average for 1997 SRM's is 97.2% recovery (n=78). The standard deviation is 5.8%. All reference samples fell within the designated parameters in 1997. Numbers may be found in tabular format in Appendix A, while control charts are listed in Appendix B.

E. Matrix Spikes

Matrix spikes are a tool for determining if and how the matrix of a sample interferes with the analyte. Matrix spikes tell us two things: does the analyte in the sample go through the analytical system the same way the analyte in the standards does, and are we able to carry analyte through the analytical system without significant losses.

Matrix spikes falling with 75-125% recovery are considered valid. Matrix spikes falling outside these parameters must be rerun. If the spike continues to fall outside of 75-125% recovery then possible causes must be looked for and identified.

The mean average for 1997 matrix spikes is 101.1% recovery (n=140). The standard deviation is 8.1%. There was just one incident of a matrix spike falling outside quality control limits. Numbers may be found in tabular format in Appendix A, while control charts are listed in Appendix B.

G. Quarterly Reports

Once per quarter Mr. Brunette prepares a quality control report, outlining quality control results in tabular format. Trends and anomalies are looked for, and if found, are identified. Possible causes must then be looked for by

examining the original dataset. Conclusions are then summarized and included in the report.

H. Performance Evaluation Studies

Frontier routinely performs outside performance evaluation samples at least twice per year. Studies that Frontier participated in 1997 include EPA's WP037 and WP038 studies, Analytical Products Group, National Water Institute and USGS. Frontier made PE studies a priority in 1997, as is evidenced by the decrease of "check for error" and "unacceptable" results in the EPA studies. Results from the most recent EPA study (WP038) have been included in Appendix B.

IV. 1998 Outlook

MDN experienced a period of change and growth during 1997. Several aspects are still being fine-tuned and will be worked on with the QA Officer in 1998. The 1997 QA Officer is leaving Frontier Geosciences Inc. to pursue other opportunities and the new QA Officer is Beverly Heaphey, formerly lead Aquatics analyst. Ms. Heaphey's extensive analytical experience with mercury will be a great benefit to the MDN project. Ms. GauthierDickey will continue to be available to Ms. Heaphey as a consultant to smooth over the transition and to answer any QA-specific questions.

Other areas that will be undergoing change and improvement in 1998 include:

- addition of 10 sites
- bi-annual blind-spike analysis
- weekly control charting of QC samples
- complete implementation of weekly air and water monitoring system started in 1997 (systems are currently monitored, but not on a set schedule)

V. Appendix A
QC Table

Period: 1997

LAB DATE	CALIBRATION			REPLICATES			BrCl Blank			MATRIX SPIKES			SRM (DORM-2 @4.64 ng/g)			LAB-RUN Bottle Blank		
	n	r	Bottle ID	ng/L gross	Spike (ng)	Bottle ID	% Recov.	ng/g	% Recov.	Bottle ID	ng/g	% Recov.	Bottle ID	ng/g	% Recov.	Bottle ID	ng/g	% Recov.
Dataset #	Lab Date																	
97-1	1/6/1997	6	0.99972	247	1.70	1.00	25	101.80	4.46	96.12	69	0.025						
97-2	1/6/1997	8	0.99985	63	4.10	1.00	221	97.80										
97-3	1/20/1997	8	0.99979	219	5.90	0.070	62	149.07*	4.60	99.14								
97-4	1/20/1997	7	0.99906	51	25.00	0.074	1.00	109	76.40									
97-5	2/3/1997	7	0.99993	461	16.30		1.00	451	102.30	4.64	100.00							
97-6	2/3/1997	7	0.99993	251	1.00		1.00	52	103.20									
97-7	2/17/1997	7	0.99991	460	0.30	0.142	1.00	454	87.80	4.21	90.73							
97-8	2/17/1997	7	0.99936	212	1.90		1.00	206	94.00									
97-9	3/3/1997	7	0.99998	52	2.20	0.077	1.00	254	96.00	4.26	91.81							
97-10	3/3/1997	7	0.99930	99	6.00		1.00	53	103.20									
97-11	3/17/1997	9	0.99972	216	1.90	0.085	1.00	207	95.80	4.57	98.49	203	0.022					
97-12	3/30/1997	8	0.99993	SB H ₂ O	4.30		1.00	202	112.30									
97-13	3/30/1997	8	0.99993	201	4.10		1.00	4	98.20	4.52	97.41							
97-14 (#4)	4/21/1997	9	0.99907	208	3.50	0.146												
				219	0.90		1.00	456	98.00									
				30	4.30	0.133	1.00	203	94.80	4.67	100.65	14	0.016					
				69	7.80		1.00	26	96.70									
				243	1.10													
				206	1.20	0.104	1.00	24	98.50	4.60	99.14							
				95	1.30		1.00	67	99.50									
				250	3.40	0.164	1.00	459	107.40	4.75	102.37							
				237	1.90		1.00	146	105.00	4.41	95.04	48	0.042					
				51	2.60		1.00	63	97.60									
				108	20.60		1.00	203	102.50									
				74	2.30	0.210	1.00	478	96.70	4.62	99.57							
				49	16.50		1.00	98	100.30									
				B3	10.00		1.00	461	99.70									
				215	0.40	0.166	1.00	1	97.80	4.60	99.14	99	0.025					
				236	5.20		1.00	221	100.20									
				469	4.20		1.00	88	100.20									
				MDN45Q	1.30	0.252	1.00	MDN45Q	118.68	4.66	100.43							

C 120 | 1.00 | MDN069 | 105.25 | 4.58 | 98.71

	4.52	97.41
	4.46	96.12
	4.49	96.8
	3.75	80.8
	3.66	78.9
	4.75	102.4
	4.55	98.1
	4.96	106.9
	4.39	94.6
	3.74	80.6
	4.46	96.1
	4.30	92.7
	4.58	98.7
	4.66	100.4
	4.90	105.6
	4.56	98.3
	4.57	98.5
	4.39	94.6
	4.72	101.7
	4.54	97.8
	4.67	100.6
	4.59	98.9
	4.65	100.2
	4.33	93.3
	4.42	95.3

1.00	MDN245	94.57
1.00	MDN527	104.98
1.00	MDN231	104.50
	FGB110	98.15
	MDN231	102.41
1.00	MDN506	100.70
1.00	MDN528	100.30
1.00	MDN553	106.90
1.00	MDN614	111.0
1.00	MDN459	99.6
1.00	MDN109	113.4
1.00	MDN254	94.4
1.00	MDN202	113.4
1.00	MDN607	97.7
1.00	MDN447	110.9
1.00	MDN519	108.6
1.00	MDN539	92.1
1.00	MDN573	112.2
1.00	MDN605	83.8
1.00	MDN548	99.7
1.00	MDN067	101.7
1.00	MDN557	93.0
1.00	MDN009	109.0
1.00	MDN240	94.0
1.00	MDN451	109.6
1.00	MDN063	96.3
1.00	MDN106	101.4
1.00	MDN224	101.6
1.00	MDN100	103.2
1.00	MDN226	103.4
1.00	MDN216	94.7
1.00	MDN612	103.9
1.00	MDN235	104.4
1.00	MDN553	99.4
1.00	MDN570	99.2
1.00	MDN108	102.3
1.00	MDN471	102.9
1.00	MDN534	100.9

0.141
0.133
0.116
0.039
0.148
0.188
0.086
0.179
0.125
0.168
0.142
0.189
0.096
0.184
0.111

MDN245	1.50
MDN508	2.80
MDN527	0.30
MDN231	0.00
FGB110	1.90
MDN231	14.70
MDN506	0.80
MDN528	3.10
MDN553	5.40
MDN614	0.1
MDN614	0.5
MDN459	1.4
MDN109	7.2
MDN254	15.5
MDN202	6.3
MDN607	0.5
MDN447	3.7
MDN519	4.2
MDN539	10.0
MDN573	12.3
MDN605	0.8
MDN548	5.6
MDN667	0.1
MDN557	1.8
MDN009	6.0
MDN240	8.5
MDN451	0.7
MDN536	0.2
MDN063	2.0
MDN106	2.2
MDN224	0.6
MDN100	0.7
MDN224	3.7
MDN216	1.8
MDN612	4.1
MDN235	1.6
MDN553	5.6
MDN570	4.8
MDN108	0.2
MDN471	3.7
MDN534	1.2

10	0.99980
10	0.99973
8	0.99993
8	0.99611
8	0.99981
10	0.99697
7	0.99997
7	0.99928
7	0.99923
7	0.99979
7	0.99989
7	0.99905
7	0.99984
7	0.99977
9	0.99976

4.33	93.3
4.42	95.3
4.56	98.3
4.32	93.1
4.69	101.1
4.68	100.9
4.61	99.4
4.59	98.9
4.63	99.8
4.53	97.6
4.53	97.6
4.64	100.0
4.56	98.3
4.54	97.8
4.72	101.7
4.93	106.3
4.53	97.6
4.55	98.1
4.55	98.1
4.75	102.4
4.73	101.9
5.15	111.0
4.51	97.16
0.27	5.78

1.00	MDN403	110.8
1.00	MDN108	102.2
1.00	MDN471	104.6
1.00	MDN534	100.4
1.00	MDN403	111.0
1.00	MDN516	94.4
1.00	MDN597	100.1
1.00	MDN206	90.1
1.00	MDN101	97.8
1.00	MDN232	98.5
1.00	MDN088	102.3
1.00	MDN537	101.6
1.00	MDN545	96.6
1.00	MDN092	76.0
1.00	MDN581	84.8
1.00	MDN228	101.3
1.00	MDN213	86.9
1.00	MDN551	99.8
1.00	MDN034	102.0
1.00	MDN581	105.1
1.00	MDN512	95.4
1.00	MDN601	110.3
1.00	MDN290	98.9
1.00	MDN231	98.7
1.00	MDN579	101.5
1.00	MDN596	97.4
1.00	MDN215	104.5
1.00	MDN247	96.5
1.00	MDN577	97.1
1.00	MDN079	98.7
1.00	MDN629	100.3
1.00	MDN545	95.0
1.00	MDN227	97.7
1.00	MDN036	107.9
1.00	MDN105	99.1
1.00	MDN548	108.4
1.00	MDN547	108.8
		100.74
		7.02

0.111
0.188
0.085
0.204
0.217
0.174
0.288
0.292
0.080
0.072
0.043
0.058
0.143
0.067

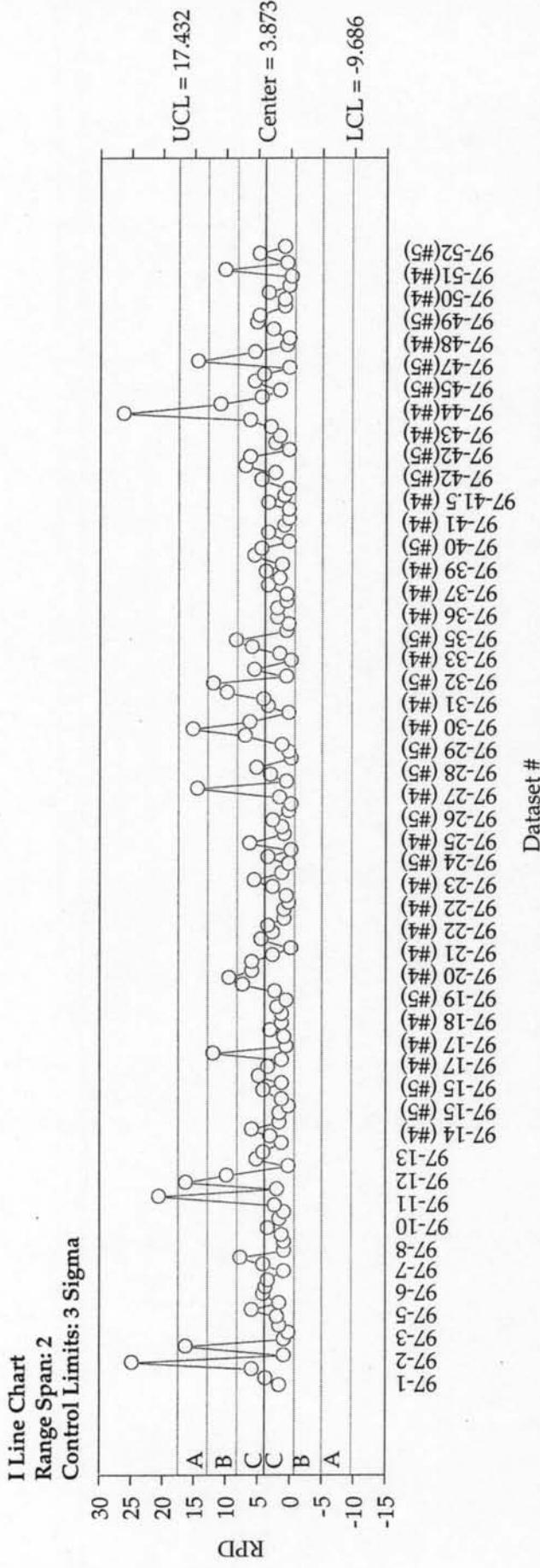
MDN403	0.2	MDN108	0.2	MDN471	3.1
MDN534	1.2	MDN403	0.2	MDN516	4.1
MDN597	2.6	MDN206	7.7	MDN101	6.6
MDN232	0.4	MDN088	2.5	MDN537	1.7
MDN092	6.5	MDN581	26	MDN545	3.2
MDN213	4.7	MDN228	10	MDN551	1.8
MDN034	5.6	MDN551	1.8	MDN512	0.5
MDN581	4.1	MDN601	14	MDN290	5.6
MDN231	0.4	MDN579	0.5	MDN614	3.0
MDN555	5.5	MDN247	5.7	MDN577	1.0
MDN079	0.9	MDN629	3.5	MDN545	0.4
MDN227	0.1	MDN036	10.0	MDN105	0.8
MDN548	5.0	MDN547	0.9	MDN547	3.8
					4.5

9	0.99976
9	0.99953
10	0.99979
8	0.99962
8	0.99933
7	0.99984
8	0.99913
7	0.99995
8	0.99994
8	0.99995
8	0.99986
8	0.99970
0.99943	
0.00084	

	97-41.5 (#4)	10/21/1997
	97-41.5 (#4)	
	97-42(#5)	10/21/1997
	97-42(#5)	
	97-43(#4)	11/11/1997
	97-43(#4)	
	97-44(#4)	11/22/1997
	97-44(#4)	
	97-45(#5)	11/22/1997
	97-45(#5)	
	97-46#4)	11/24/1997
	97-47(#5)	12/2/1997
	97-47(#5)	
	97-48#4)	12/2/1997
	97-49#5)	12/9/1997
	97-49#5)	
	97-50#4)	12/9/1997
	97-50#4)	
	97-51#4)	12/31/1997
	97-51#4)	
	97-52#5)	12/31/1997
	97-52#5)	
	Mean	
	Std. Dev.	

**VI. Appendix B
Control Charts**

Replicate Analysis

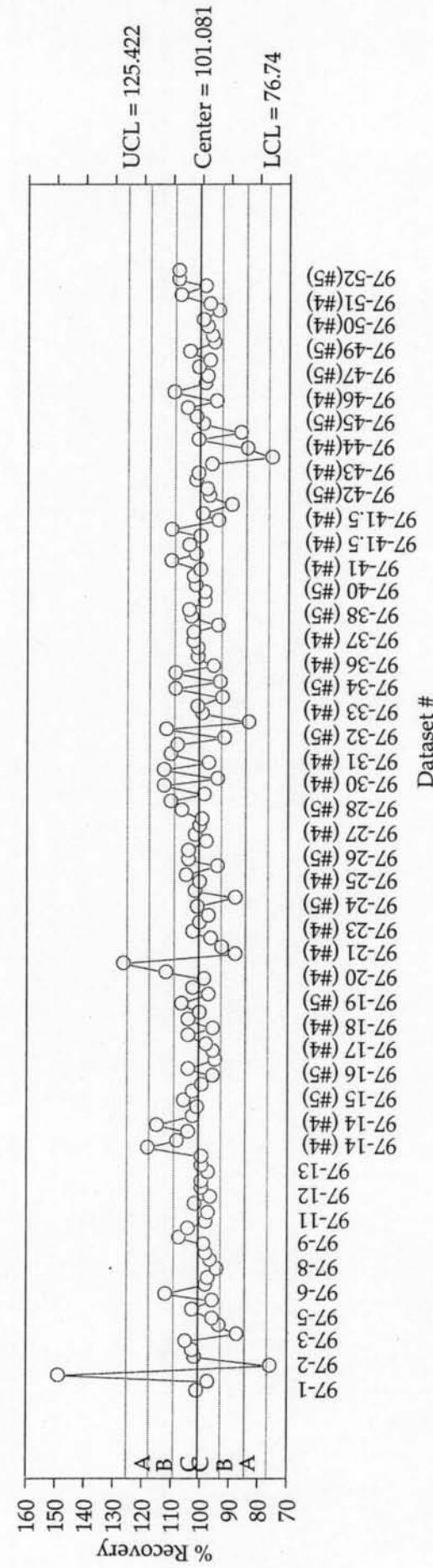


Summary Table for RPD
Grouping Variable: Dataset #
Range Span: 2

K Sigma	3.000
Alpha	*
Sigma	4.520
Xbar	3.873
MRbar	4.056
# Obs.	152
# Missing	0

Matrix Spike % Recovery

I Line Chart
Range Span: 2
Control Limits: 3 Sigma



Summary Table for % Recovery
Grouping Variable: Dataset #
Pancreas

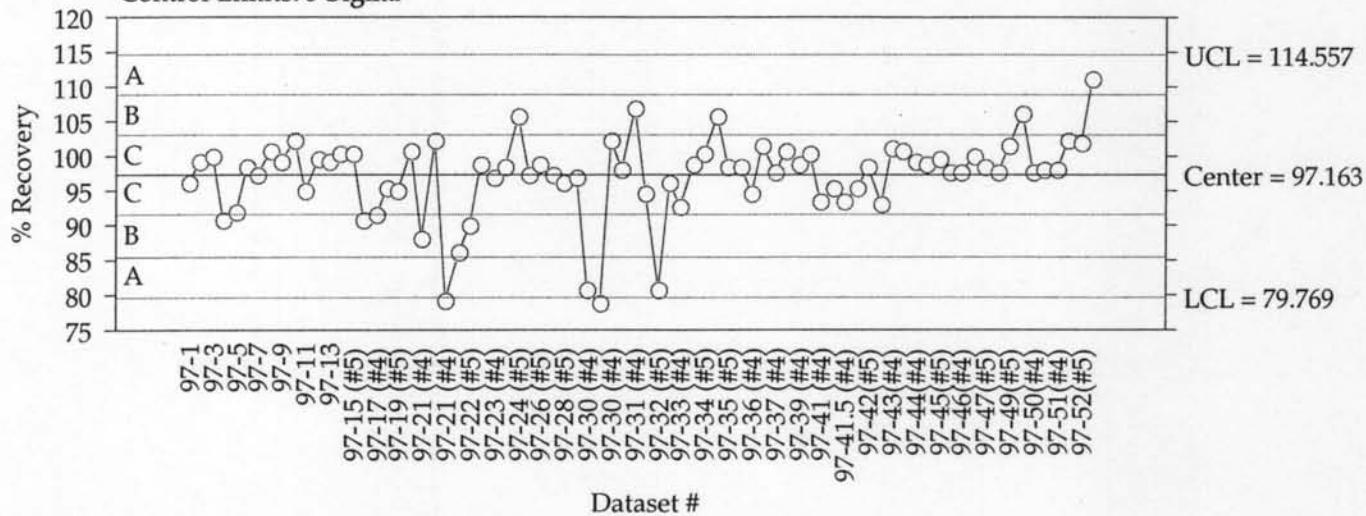
Range Span: 2	K Sigma	3.000
Alpha	•	
Sigma	8.114	
Xbar	101.081	
MRbar	8.101	
# Obs.	140	
# Missing	0	

Standard Reference Material % Recovery

I Line Chart

Range Span: 2

Control Limits: 3 Sigma



Summary Table for % Recovery

Grouping Variable: Dataset #

Range Span: 2

K Sigma	3.000
Alpha	•
Sigma	5.798
Xbar	97.163
MRbar	4.798
# Obs.	78
# Missing	0

**VII. Appendix C
EPA WP038 Results**

