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FINAL
Data Summary Report for Year 1 of the Chemical Speciation of PM2.5 Filter Samples Project

February through September 2000

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1.0 Introduction

1.1 Program Overview

In 1997, the U.S. Environmental Protection Agency (EPA) promulgated the new National Ambient Air Quality Standards (NAAQS) for particulate matter. The regulations (given in 40 CFR Parts 50, 53, and 58) apply to the mass concentrations (μg/cubic meter of air) of particles with aerodynamic diameters less than 10 micrometers (the PM10 standard) and less than 2.5 micrometers (the PM2.5 standard). Establishment of a 1500-site mass measurements network and a 300-site chemical speciation monitoring network is now under way.

The ambient air data from the network, which measures solely the mass of particulate matter, will be used principally for NAAQS comparison purposes in identifying areas that meet or do not meet the NAAQS criteria and in supporting designation of an area as attainment or non-attainment.

The smaller chemical Speciation Trends Network (STN) will consist of a core set of 54 trends analysis sites and some 250 other sites. Chemically speciated data will be used to serve the needs associated with development of emission mitigation approaches to reduce ambient PM2.5 concentration levels. Such needs include emission inventory establishment, air quality model evaluations, and source attribution analysis. Other uses of the data sets will be regional haze assessments, estimating personal exposure to PM2.5 and its components, and evaluating potential linkages to health effects.

Research Triangle Institute (RTI) is assisting in the PM2.5 STN by shipping ready-to-use filter packs and denuders to the field sites and by conducting gravimetric and chemical analyses of the several types of filters used in the samplers. The details of the quality assurance (QA) activities being performed are described in the RTI QA Project Plan (QAPP) for this project. This QAPP focuses on the QA activities associated with RTI's role in performing these analyses, as well as in validating and reporting the data, and should be considered a companion document to this annual QA report.

Prior to operation of the core and additional sites, EPA ran a prototype network informally known as the "mini-trends" network. This network was composed of approximately 13 monitoring stations at sites throughout the U.S. Each site had two or more PM2.5 chemical speciation monitors to enable various sampler intercomparisons. The mini-trends network ran from February 2000 to July 31, 2000. The 54-site STN is scheduled to be fully on-line in early 2001.

1.2 Project/Task Description

The STN laboratory contract involves four broad areas:

1. Supplying each site or state with sample collection media (loaded filter packs, denuders, and absorbent cartridges) and field data documentation forms. RTI ships the collection media to monitoring agencies on a schedule specified by the Delivery Order Project Officer (DOPO).

2. Receiving the samples from the field sites and analyzing the sample media for mass and for an array of chemical constituents including elements (by EDXRF), soluble anions and cations (by ion chromatography), and carbonaceous species (using the Sunset thermal degradation/laser transmittance system). Analysis of semi-volatile organic compounds and examination of particles by electron or optical microscopy will not be performed initially; however, these analyses may be included later in the full STN program.

3. Assembling validated sets of data from the analyses, preparing of data reports to EPA management and the states, and entering data to the Aerometric Information Retrieval System (AIRS) data bank 60 days after initial data reports are first submitted to the DOPO and the states.

4. Establishing and applying a comprehensive quality assurance/quality control (QA/QC) system. RTI's Quality Management Plan, QAPP, and associated Standard Operating Procedures (SOPs) provide the documentation for RTI's quality system.

1.3 Schedule

The initial portion of the STN program was a six-month pilot project at 13 different sites. This "mini-trends" project was conducted from February 2000 to July 2000. This period gave all participants an opportunity to work out technical and logistical problems. Additional sites are now coming on line and the full 54-station STN will be operational around the first of 2001. This QA report covers the first year of collection and analysis of samples, from about February 2000 through September 2000.

1.4 Major Laboratory Operational Areas

This report addresses the operation of the Sample Handling and Archiving Laboratory (SHAL) and QA/QC for the four major analytical areas active this past year. These analytical areas are the: (1) gravimetric determination of particulate mass on Teflon® filters;

(2) determination of 48 elements on Teflon® filters using X-ray fluorescence spectrometry; (3) determination of nitrate, sulfate, sodium, ammonium and potassium on nylon using ion chromatography; and (4) determination of organic carbon, elemental carbon, carbonate carbon, and total carbon on quartz filters using thermal optical transmittance. Also addressed is denuder refurbishment, data processing, and QA and data validation.

1.5 Significant Corrective Actions Taken

Over this past year, several significant corrective actions have been taken.

Individual corrective actions are described in Section 2 under each activity or laboratory. Some of the most significant corrective actions for the overall quality of the program were the following:

- Development of a revised procedure for cleaning nylon filters to reduce the background levels of ions below 1µg per filter.
- Installation of a new gravimetric laboratory with superior environmental controls.
- Remediating problems with leaking ice packs using ziplock bags.
- RTI personnel worked in close cooperation with EPA to fine-tune the carbon analysis procedure.

2.0 Laboratory Quality Control Summaries

2.1 Gravimetric Laboratory

2.1.1 Facilities

Gravimetric analyses are performed by personnel from RTI's Center for Environmental Measurement and Quality Assurance (CEMQA) Earth and Mineral Sciences Department (E&MSD). The Gravimetric Laboratory consists of a self-contained, climate-controlled weigh chamber in which Teflon® filters are inspected, conditioned, and weighed. The chamber is equipped with its own environmental controls consisting of an air conditioning, filtration, humidification, and dehumidification system. A dehumidifier located adjacent to the chamber removes excess humidity via a slowly turning desiccant wheel. Additional humidity, if needed, is added via a steam generator. Sensors mounted on the wall inside the chamber report to an environmental panel located outside the chamber. The panel, which includes a chart recorder, provides a means of monitoring the system and of changing the setpoint for temperature and relative humidity (RH). A data logger is also used inside the chamber to monitor temperature and RH, and is considered the laboratory's official recording device. Readings are downloaded from the data logger daily. Both the chamber's wall-mounted sensors and the data logger have NIST-traceable calibrations. The chamber's design performance is rated for temperature uniformity of ±1°C and humidity uniformity of ±5% RH. In a review of another contract to provide Federal Reference Method (FRM) laboratory support to selected states, Ms. Ann Marie Carleton of EPA Region 2 examined the chamber and performed a technical systems audit on October 5, 2000. Ms. Carleton's audit included verification of the chambers RH and temperature with her own monitoring device. She found the chamber to be operating within acceptable parameters.

The chamber was installed by an environmental specialty contractor and pronounced complete during the week of November 22, 1999. RTI HVAC personnel monitored the temperature/humidity system and informed laboratory personnel the next week that the chamber had been maintaining a steady temperature and relative humidity. The chamber and chamber furnishings were then precleaned by laboratory personnel. The newly purchased Microbalance B was relocated to the chamber, inspected, calibrated, and certified by a Mettler Toledo Service Representative. At that time, the Laboratory Supervisor commenced daily weighing of the working mass standards to be used with the balance and five blank Gelman Teflon® filters to monitor chamber stability. Following a final wipe of the floor with deionized (DI) water on December 13, 1999, the Gravimetric Laboratory was pronounced operative by laboratory personnel. The original microanalytical balance, Microbalance A, was relocated to the chamber in February 2000, and calibrated and certified before use by a Toledo Service Representative.

Several incidents related to failure of the chamber's dehumidification system occurred during its first seven months of operation. These incidents resulted in water leakage into the chamber with subsequent increases in RH and minor water damage to chamber furnishings. No filters or analytical equipment sustained water damage. Although all problems were reported

immediately to RTI HVAC personnel and the installation contractor, repeated diagnostic checks of the system were required before the installation contractor could isolate, identify, and correct the problems. All repairs during this time were coordinated by the RTI HVAC project manager and performed by the installation contractor under warranty. With the expiration of the chamber's one-year warranty, RTI HVAC personnel have assumed responsibility for maintenance and repair. It appears as of this writing that all issues related to the new construction/installation have been suitably addressed and that the chamber is operating within the parameters of its design performance. Corrective actions taken in response to system failures are described more completely in Table 1.

Table 1. Gravimetry Laboratory - Corrective Actions in Response to Facility Problems

Duration of Problem	Nature of Problem	Corrective Action
12/14/99 - 02/21/00	Humidity increases noted on chart recorder, water leakage from ceiling air plenum	02/09/00 - Installation contractor disassembled and cleaned a solenoid valve controlling water flow to the dehumidifier, which was not closing properly 02/10/00 - Installation contractor determined that leakage was caused by an improperly installed drain line 02/21/00 - Installation contractor changed the steam injection point and condensation drain point from the humidifier
07/01/00 (RTI Holiday Weekend)	RTI Security personnel discovered massive water leakage in the gravimetry chamber	07/01/00 - In response to notification by RTI Security personnel, Dr. Philip Lawless of CEET relocated CEMQA's filter racks to CEET's gravimetry chamber 07/05/00 - Installation contractor replaced a malfunctioning thermostat
07/17/00 - 07/19/00	Large fluctuations in RH, excessively high RH, Microbalance B autocalibration function unable to respond to sudden changes in RH	07/17/00 - RTI HVAC personnel removed a filter that they had recently placed on the chamber's HVAC system explaining that the filter was too constrictive and had caused the system's regeneration cycle to malfunction 07/19/00 - Installation contractor replaced the drive motor on the dehumidifier 07/20/00 - Mettler Toledo Service Representative adjusted internal calibration factor on Microbalance B and performed external calibration
10/9/00	Low RH	10/09/00 - RTI HVAC personnel replaced blown fuse in chamber's control panel

2.1.2 Description of Quality Control Checks Applied

QC checks applied to the gravimetric analysis of Teflon® filters for the PM2.5 STN are summarized in Table 2. The QC checks have been developed from guidance provided in Section 2.12 of the *EPA Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Ambient Air Specific Methods* (Guidance Document 2.12), and from our experience in providing federal reference method (FRM) laboratory support to various consulting firms, states, U.S. territories, and EPA since the inception of the compliance (mass) monitoring portion of the nationwide PM2.5 network.

2.1.3 Statistical Summary of QC Results

A total of 2,441 Teflon® PM2.5 Chemical Speciation filters were tared in the Gravimetric Laboratory between January 24, 2000, and September 1, 2000; 33 (~1.4%) of these filters were retained by the Gravimetric Laboratory for use as laboratory blanks. The remainder (2408) of the filters were transferred to the SHAL to be loaded into sampler modules. The SHAL returned 2,311 of these filters were returned to the Gravimetric Laboratory between February 23, 2000, and October 6, 2000, for a return rate of approximately 94.7%. Two of the filters returned to the Gravimetric Laboratory (~0.1%) were voided by the Gravimetric Laboratory Supervisor. One filter was voided due to Gravimetric Laboratory error. One filter was voided after consultation with the SHAL due to a labeling discrepancy.

2.1.3.1 Working Mass Reference Standards - The Gravimetric Laboratory has identified a negative bias associated with the laboratory's working mass reference standards. The bias, clearly present on laboratory control charts (see Figures 1 through 4), is possibly attributable to deformation or damage of the standards from the rigid plastic forceps historically used to handle them, or to having been dropped from these forceps. In response, the Gravimetric Laboratory has recently moved to the use of more specialized forceps with Teflon® tips to minimize damage to the standards. A 100-mg and a 200-mg working mass reference standard were delivered to the North Carolina Department of Agriculture and Consumer Services (NCDA&CS) Standards Laboratory for calibration on November 15, 2000. As of this writing, RTI has not been notified that the calibration is complete.

2.1.3.2. Laboratory Blanks - Guidance Document 2.12 suggests that replicate measurements of laboratory blanks should agree within 15 µg. The Gravimetric Laboratory performed 167 replicate weighings of the 33 filters retained as laboratory blanks (see Figure 5). The mean weight change for the laboratory blanks was 5 µg. Only five replicate weighings (3.0%) exceeded the 15 µg acceptance criterion. Four of the weighings in excess of 15 µg were performed on one filter. One of the weighings, performed well over four months after the filter's initial weighing, was not considered part of routine batch QC. It was performed when previously analyzed filters were returned to the Gravimetric Laboratory by the QA Officer for confirmatory analysis. Elevated laboratory blanks were attributed to contamination, probably resulting from chamber repair and building renovation activities. In response to the elevated laboratory blanks, the chamber was thoroughly cleaned.

Table 2. Gravimetric Analysis of PM2.5 Filters - QC Checks

QC Check	Frequency	Requirements	Comments
Working standard reference weights (mass reference standards)	After every 10th filter (at least one working standard - preferable to weigh standards bracketing weight of filter)	Verified value ± 3 µg	Reference - Guidance Document 2.12, Section 7.0, Filter Preparation and Analysis Calibrated annually at the NCDA&CS Standards Laboratory in Raleigh, NC Verified via primary standards maintained by EQSD
Blanks			Reference - Guidance Document 2.12, Section 7.0, Filter preparation and Analysis
‣ Laboratory (filter) blanks	At least one lab blank every weigh session	Initial weight ± 15 µg	Assigned by Gravimetric Laboratory for determination of contamination in the weighing environment
‣ Lot (stability filter) blanks	At least three filters from every new (manufacturer's) filter lot	Initial weight ± 5 µg (Guidance Document 2.12 suggests ± 15 µg)	Assigned by Gravimetric Laboratory for determination of period that filters from lot must be conditioned before tare weighing
‣ Field (filter) blanks	See below	See below	See below
Balance Audits	Semiannually	Satisfactory per RTI HERL audit checklist	Performed by personnel from EQSD
Replicates	Every 10th filter	Initial weight ± 15 µg	Reference - Guidance Document 2.12, Section 7.0, Filter Preparation and Analysis
Calibrations			
‣ Working Mass Standards	Annually		Performed by NCDA&CS Standards Laboratory in Raleigh, NC
‣ Balance	Auto (internal) calibration daily External calibration annually or as needed		Balances have internal calibration weights and are self-calibrating via an auto-calibrate function. RTI maintains a service contract with Mettler Toledo for annual inspection, calibration, and preventive maintenance of the balances. Mettler Toledo is also contacted on an as-needed basis for adjustment or repair.
‣ RH/T Data Logger	Annually	± 2% RH ± 0.25°C	Performed by Dickson Calibration Services
Field (filter) blanks	At least three field blanks every weigh session	Initial weight ± 30 µg	Reference - Guidance Document 2.12, Section 7.0, Filter preparation and Analysis Assigned by field for determination of contamination occurring during sampling **Field blanks are not identified to gravimetric laboratory staff.**

2.1.3.3 Lot Blanks (Lot Stability Filters) - Guidance Document 2.12 suggests that Teflon® filters should be conditioned before routine sampling until their 24-hour weight change is less than 15 µg. The Chemical Speciation filters tared in the Gravimetric Laboratory between January 24, 2000, and September 1, 2000, were taken from three Whatman manufacturing lots (00299, 00025, and 0159007). Lot stability tests were performed on each of the three lots as summarized in Table 3. The 24-hour weight changes monitored for all three lots were well within the suggested 15 µg acceptance criterion.

Table 3. Gravimetric Analysis of PM2.5 Filters - Lot Stability Tests

Filter Lot	Mean Weight Change After 24 Hours (µg)	Mean Weight Change After 48 Hours (µg)	Mean Weight Change After 72 Hours (µg)	Mean Weight Change After 96 Hours (µg)
00299	-1	5	-2	0
00025	-1	4	0	0
0159007	0	0	1	1

2.1.3.4 Field Blanks - The evaluation of field blanks is outside the scope of the Gravimetric Laboratory's QA program.

2.1.3.5 Balance Audits - Balance audits were performed on each of the two microanalytical balances on March 27, 2000, and October 11, 2000, by personnel from CEMQA's Environmental Quality Systems Department (EQSD). Each audit included the following tests: environmental evaluation level test, scale-clarity test, zero-adjustment test, display drift test, off-center (corner load error) test, precision test, and accuracy test. The weighing environment and both balances were evaluated as satisfactory by the auditor. The auditor also verified the Gravimetric Laboratory's working mass reference standards at this time with his (primary) standards.

2.1.3.6 Replicates - Guidance Document 2.12 suggests that replicate measurements of filters used for routine sampling should agree within 15 µg. Two hundred thirty-two pre-sampling (tared) Chemical Speciation replicates were weighed in the Gravimetric Laboratory between January 24, 2000, and September 1, 2000, with an average difference of 2 µg (Figure 6a). The maximum difference between pre-sampling replicates was 6 µg. One hundred seventy-seven post-sampling Chemical Speciation replicates were weighed in the Gravimetric Laboratory between February 23, 2000, and October 6, 2000, with an average difference of 2 µg (Figure 6b). The maximum difference between post-sampling replicates was 10 µg. None of the replicates exceeded the acceptance range of 15 µg.

Guidance Document 2.12 suggests that internal QC checks should be applied after every tenth filter weighed. Since sampled filters are neither returned from field sites in numerical order nor in complete sets of 10 filters, gravimetric analysts often perform post-sampling weighing of only a few filters from each of several data (spreadsheet) files. Consequently, the rhythm of every-tenth-filter QC is disrupted and an insufficient number of post-sampling replicates are weighed. In response to the low number of post-sampling replicates, the Gravimetric Laboratory has instituted a laboratory policy of performing at least one replicate measurement in every weigh session for every data (spreadsheet) file.

2.1.3.7 Calibrations - The Gravimetric Laboratory's working mass reference standards, microanalytical balances, and temperature/RH data logger are calibrated as part of routine laboratory operation. All calibrations are performed by qualified providers who are independent of the laboratory's data generation.

Working mass reference standards were calibrated by the NCDA&CS Standards Division on November 15, 1999; March 16, 2000; and November 15, 2000.

In addition to daily auto- (internal) calibrations, the microanalytical balances were inspected and calibrated by a Mettler Toledo Service Representative on December 10, 1999; February 17, 2000; and July 20, 2000. The Service Representative also adjusted Microbalance B's internal calibration factor on July 20. The calibration factor had drifted as a result of the microbalance's inability to respond to rapidly changing RH in the weighing environment during repairs on the dehumidifier. In a review of E&MSD's contracts to provide FRM laboratory support to selected states, Ms. Ann Marie Carleton of EPA Region 2 examined the laboratory's two microanalytical balances and performed a technical systems audit on October 5, 2000. Ms. Carleton's audit included verification of balance accuracy with her own calibrated mass standards. She found both balances to be operating within acceptable parameters.

The temperature/RH data logger was calibrated by Dickson Calibration Services on February 11, 2000.

2.1.4 Data Validity Discussion

Filters are inspected in the Gravimetry Laboratory before conditioning and tare-weighing. Damaged or anomalously labeled filters are rejected at this time. No damaged filters are supplied to the SHAL for loading into sampler modules. Sampled filters are often returned to the Gravimetric Laboratory; however, with visible damage, including tears, holes, scratches, and smudges. Such damaged filters are not voided by the Gravimetric Laboratory. They are flagged with a descriptive comment pertaining to the damage and returned to the SHAL for further processing and analyses. Only two of the 2,311 filters returned to the Gravimetric Laboratory by the SHAL between February 23, 2000, and October 6, 2000, were voided by the Gravimetric Laboratory.

2.1.4.1 Invalid Data Due to Gravimetric Laboratory Errors — One filter was voided due to electronic data loss when the gravimetric analyst failed to press the microbalance's print

button to enter the filter's post-sampling weight into the data spreadsheet. The weighing could not be replicated because the filter had already been transferred to and processed by the SHAL.

2.1.4.2 Invalid Data Due to Other Causes — Several filters were covered with impactor oil, which dissolved and obliterated the stamped filter identification numbers. The exact number of filters contaminated with impactor oil is not known because gravimetric analysts did not realize until August 2000, that the presence of oil is something that they should report to the Gravimetric Laboratory Supervisor. In almost all cases, the filter could be identified by comparing the aliquot barcode label applied to the Petrislide® by the SHAL to the Chain of Custody prepared by the SHAL. One such filter was voided, in coordination with the SHAL Supervisor, when two different aliquot barcode labels were used to refer to the same filter number such that the Chain of Custody could not be used to identify the filter.

2.1.5 Summary of Audit Findings and Recommendations

The laboratory audit performed by EPA-NAREL and EPA-NE personnel on December 14, 1999, identified no major deficiencies in the Gravimetric Laboratory. Auditors made two recommendations for the Gravimetric Laboratory, as indicated in the audit report letter excerpted below.

> *A regular system of calibration checks should be made of all balances, using Class 1 weights. The results of the checks should be documented in a balance logbook. Weights for the calibration checks should span the range of weights of expected samples.*
>
> *Criteria for a balance being out of calibration should be established, and procedures for taking a balance out of service and corrective action should be created.*

2.1.6 Corrective Actions Taken

A regular system of calibration checks has always routinely (initial, after every tenth filter, final) been made of all microbalances using Class 1 working weights that bracket the mass range of a typical 46.2-mm diameter Teflon® filter. The results of the checks have been recorded in the QA/QC Data Sheet for each weigh session. An additional calibration check with the laboratory's primary Class 1 standards has always been routinely made during the balance audit. In response to EPA's laboratory audit, the Gravimetry Laboratory's SOP has been revised to more clearly reflect this. In addition, the SOP has been revised to include criteria for judging a microbalance to be out of calibration and a written procedure for taking a microbalance out of service and taking corrective action.

2.2 Ion Analysis Laboratory

2.2.1 Facilities

Ion chromatographic analyses are performed by personnel from CEMQA's Environmental and Industrial Chemistry Department (EICD). Four ion chromatographic systems were used for performance of the measurements. These are described in Table 4. The use of the systems was determined by the workload. A new Dionex Model 600 was recently purchased to meet the work load anticipated with the expansion of the STN.

Table 4. Description of Ion Chromatographic Systems used for Analysis of PM2.5 Filter Samples

System No.	Dionex IC Model	Ions Measured	Period of Use	Comments
1	Model 500, Purchased	SO_4, NO3	9/15/00 - 9/25/00	
2	Model 500, Purchased	SO_4, NO3	2/15/00 - 9/30/00	
3	Model 500, Purchased	SO_4, NO3	6/1/00 - 9/15/00	
4	DX-500	Na, NH4, K	2/15/00 - 9/30/00	

2.2.2 Description of QC Checks Applied

QC checks for ion analyses are summarized in Table 5. For ion analyses, a daily multipoint calibration (7 points for cations; 8 points for anions) is performed over the range 0.05 to 25.0 ppm for each ion (Na^+, NH_4^+, and K^+ for cation analyses; NO_3^- and $_{SO4}^{2-}$ for anion analyses) followed by QA/QC samples including (1) a QC sample containing concentrations of each ion in the mid- to high-range of the calibration standard concentrations, (2) a QC sample containing concentrations of each ion at the lower end of the calibration standard concentrations, and (3) a commercially prepared, NIST-traceable QA sample containing known concentrations of each ion.

The regression parameters (a,b,c and correlation coefficient, r) for the standard curve for each ion are compared with those obtained in the past. Typically, a correlation coefficient of 0.999 or better is obtained for each curve. If the correlation coefficient is <0.999, the analyst carefully examines the individual chromatograms for the calibration standards and reruns any

Table 5. Ion Analysis of PM2.5 - Quality Control/ Quality Assurance Checks

QA/QC Check	Frequency	Requirements
Calibration Regression Parameters	Daily	r $\geq$ 0.999
Initial QA/QC Checks:		
- QC sample at mid to high range concentration	Daily, immediately after calibration	Measured concentrations within 10% of known values
- QC sample at lower end concentration	Daily, immediately after calibration	Measured concentrations within 10% of known values
- Commercially prepared, NIST traceable QA sample	Daily, immediately after calibration	Measured concentrations within 10% of known values
Periodic QA/QC Checks:		
- Duplicate sample	Every 20 samples	RSD = 5% at 100x MDL* RSD = 10% at 10x MDL* RSD = 100% at MDL*
- QA/QC sample	Every 20 samples	Measured concentrations within 10% of known values
- Matrix spiked sample extract	Every 20 samples	Recoveries within 90 to 100% of target values

* MDL = Minimum Detectable Limit

standard that is judged to be out of line with respect to the other standards or to values (peak area and/or height) obtained in the past for the same standard. Possible causes for an invalid standard run include instrumental problems such as incomplete sampling by the autosampler. If necessary, a complete recalibration is performed.

When all individual calibrations have been judged acceptable, the results for the QA/QC samples are carefully examined. If the observed value for any ion being measures differs by more than 10 percent from the known value, the problem is identified and corrected any field samples are then analyzed.

During an analysis run, a duplicate sample, a QA/QC sample, and a spiked sample are analyzed at the rate of at least one every 20 field samples. Precision objectives for duplicate

analyses are ±5 percent for concentrations that equal or exceed 100 times the minimum detectable limit (MDL), ±10 percent for concentrations at 10 times the MDL, and ±100 percent for concentrations at the MDL. The observed value for any ion being measured must be within 10 percent of the known value for the QA/QC samples, and ion recoveries for the spiked samples must be within 90 to 110 percent of the target value. If these acceptance criteria are not met for any QA/QC or spiked sample, the problem is identified and corrected. All field samples analyzed since the last acceptable check sample are reanalyzed.

2.2.3 Statistical Summary of QC Results

2.2.2.1 IC System 1 – QC checks performed on System 1 included:

- Percent recovery for QC samples
- Percent recovery for QA samples
- Relative percent difference (RPD) for replicates
- Spike recovery tests
- Reagent blank tests

Figures 7a through 7f show recoveries for NO_3 and SO_4 with low, medium and high concentration QC samples. Recoveries for all six combinations of ion and level ranged between 96% and 103% over the one month period of use of this system. Figures 8a through 8d show recoveries for NO_3 and SO_4 with low and medium high concentration QA samples. Recoveries for all four combinations of ion and level ranged between 98% and 103% over the one month period of use of this system. Figures 9a and 9b show RPD for replicate NO_3 and NO4 measurements, respectively. The RPD values above 5% correspond to actual solution concentrations below 0.05 µg/mL. Figures 10a and 10b show recovery for NO_3 and SO_4 spikes, respectively. Values were in the range of 97% to 102%. Figures 11a through 11d show total ion levels in normal extraction volumes (25 mL for nylon filters and 10 mL for quartz filters) of deionized water reagent blanks and eluent reagent blanks. Only one value above 0.5 µg was measured.

2.2.2.2 IC System 2 – QC checks performed on System 2 included:

- Percent recovery for QC samples
- Percent recovery for QA samples
- RPD for replicates
- Spike recovery tests
- Reagent and filter blank tests

Figures 12a through 12f show recoveries for NO_3 and SO_4 with low, medium and high concentration QC samples. Recoveries for all six combinations of ion and level ranged between 96% and 103% over the eight month period of use of this system. Figures 13a through 13d show recoveries for NO_3 and SO_4 with low and medium high concentration QA samples. Recoveries for all four combinations of ion and level ranged between 98% and 103% over the eight month period of use of this system. Figures 14a and 14b show RPD for replicate NO_3 and NO4 measurements, respectively. The RPD values above 5% correspond to actual solution concentrations all below 0.1 µg/mL and with the majority below 0.05 µg/mL. Figures 15a and 15b show recovery for NO_3 and SO_4 spikes, respectively. Values ranged between 96% and 102%. Figures 16a through 16h show total ion levels in normal extraction volumes (25 mL for nylon filters and 10 mL for quartz filters) of deionized water reagent blanks, deionized water-extracted filter blanks, eluent reagent blanks, and eluent-extracted filter blanks. Except in one case, all deionzed water reagent blanks were less than 1 µg NO_3 or SO_4. Deionized water-extracted filter blanks were less than 1 µg for NO_3 and less than 2 µg for SO_4. Eluent reagent blank solutions were less that 1 µg for both NO_3 and SO_4. Eluent-extracted filter blanks were less than 1 µg for NO_3 and less than 2.5 µg for SO_4.

2.2.2.2 IC System 3 – QC checks performed on System 3 included:

- Percent recovery for QC samples
- Percent recovery for QA samples
- RPD for replicates
- Spike recovery tests
- Reagent and filter blank tests

Figures 17a through 17f show recoveries for NO_3 and SO_4 with low, medium and high concentration QC samples. Recoveries for all six combinations of ion and level ranged between 96% and 103% over the seven month period of use of this system. Figures 18a through 18d show recoveries for NO_3 and SO_4 with low and medium high concentration QA samples. Recoveries for all four combinations of ion and level ranged between 98% and 103% over the eight month period of use of this system. Figures 19a and 19b show RPD for replicate NO_3 and NO4 measurements, respectively. The RPD values above 5% correspond to actual solution concentrations all below 0.05 µg/mL, except for one SO_4 solution where the concentration was 0.06 µg/mL. Figures 20a and 20b show recovery for NO_3 and SO_4 spikes, respectively. Values ranged between 97% and 103%. Figures 21a and 21b show total ion levels in normal extraction volumes (25 mL for nylon filters and 10 mL for quartz filters) of deionized water reagent blanks. Except for one day, all deionzed water reagent blanks for NO_3 were below the MDL. The levels for SO_4 (Figure 21b) were all less than 0.6 µg.

2.2.2.3 IC DX-500 – QC checks performed on the DX-500 IC System included:

- Percent recovery for QC samples
- Percent recovery for QA samples
- RPD for replicates
- Spike recovery tests
- Reagent and filter blank tests

Figures 22a through 22c show recoveries for 2.0 ppm QC samples for Na, NH4 and K. Recoveries were in the range of 94% to 109%, all within the 10% limits, over the seven month period of use of this system. The decline in recovery near the end of the term has shown an improvement. Figures 22d through 22f show recoveries for 5.0 ppm QC samples; values ranged between 94% and 109%. Figures 23a through 23c show recoveries for 0.4 ppm QA samples. Except for two sodium recoveries, all were within the 10% limits; the two low sodium values were still greater than 88%. Figures 23d through 23f show recoveries for 4.0 ppm QA samples; values were in the range of 93% to 108%. Figures 24a through 24b show RPDs for replicate Na, NH4 and K measurements. The RPD values above 5% correspond to actual solution concentrations all below 0.1 µg/mL, with the majority below 0.05 µg/mL. Figures 25a through 25c show spike recoveries for the three ions. Except for one Na spike recovery, all values ranged between 90% and 110%. Figures 26a through 26c show total ion levels in normal extraction volumes (25 mL for nylon filters and 10 mL for quartz filters) of deionized water reagent blanks. All values were less than 1 µg. Figures 26d through 26f show total ion levels in normal extraction volumes (25 mL for nylon filters and 10 mL for quartz filters) of deionized water extracted filter blanks. Again, all values were less than 1 µg.

2.2.4 Data Validity Discussion

To date, no data have been invalidated as a result of errors in the ion chromatography laboratory. Any inconsistencies that are observed in the filter samples are flagged on the ion chromatography data report when it is submitted for entry into the database. For example, on one occasion, two filters were found in one petri dish. The filters were extracted and analyzed as one, and this was noted on the data report for that batch of samples.

2.2.5 Summary of Audit Findings and Recommendations

All audit findings and recommendations are described in Appendix A to this report.

2.2.6 Corrective Actions Taken

Nylon filters are used for the collection of PM2.5 material in the chemical speciation particulate samplers. These filters are analyzed for anions (nitrate and sulfate) and/or for cations (ammonium, sodium, and potassium). At the beginning of the project, the filters, as purchased and received from different manufacturers, were found to contain unacceptable levels of one or more of these ions, often exceeding the maximum level of 1µg per filter for a particular ion.

This prompted development of a SOP for cleaning the nylon filters prior to their use for field sampling. Filters cleaned using this procedure have been demonstrated not to exceed the acceptable levels of the ions of interest. The SOP is included in the latest revision of the QAPP, Volume II.

2.3 Organic and Elemental Carbon Laboratory

2.3.1 Description of QC Checks Applied

QC checks, acceptance criteria, and corrective actions for the organic and elemental carbon (OC/EC) Laboratory are summarized in Table 6.

2.3.2 Statistical Summary of QC Results

The OC/EC Laboratory has two carbon analyzers designated as the New analyzer (because it is the newer of the two) and the Retrofit analyzer (because it is an old analyzer that has been retrofit to be equivalent to the New analyzer).

Table 6. Summary of OC/EC QC Checks, Acceptance Criteria, and Corrective Actions

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Detection Limit	annually	MDL $\leq$ 0.5 µg C/cm^2	Investigate the source of the problem and initiate corrective action, if necessary, to correct the problem before analyzing samples.
Calibration Peak Area	every analysis	Within 90% to 110% of average calibration peak area for that day	Discard the results of that analysis and, if necessary, repeat the analysis with a second punch from the same filter.
Instrument Blank	daily	Blank $\leq$0.3 µg/cm^2 (Prior to July 17, 2000, blank criterion was $\leq$1 µg/cm^2.)	Determine if the problem is with the filter or the instrument, and, if necessary, initiate corrective action to identify and solve any instrument problem before analyzing samples.
Three-Point Calibration	weekly	Correlation Coefficient (R^2) $\geq$0.99	Determine the cause of the nonlinearity, and initiate actions that will identify and solve any problem that may have arisen. Then repeat the three-point calibration, which must yield satisfactory results before samples are analyzed.
Calibration Check	daily	(1) 90% to 110% recovery, and (2) calibration peak area 90% to 110% of average for the weekly 3-point calibration.	Initiate corrective action, if necessary, to solve the problem before analyzing samples.
Duplicate Analyses	10% of samples	(1) TC Values greater than 10 µg/cm^2-- Less than 10% RPD, (2) TC Values 5 - 10 µg/cm^2-- Less than 15% RPD, (3) TC Values less than 5 µg/cm^2-- Within 0.5 µg/cm^2.	Flag analysis results for that filter with non-uniform filter deposit (LFU) flag.

The method detection limit for total carbon (TC) is determined annually. Both the OC/EC carbon analyzers met the required limit of $\leq$0.5 μg C/cm^2 with MDLs of 0.29 μg C/cm^2 for the New analyzer and 0.24 μg C/cm^2 for the Retrofit analyzer.

Calibration peak area, which is the response of the FID to the internal standard, is plotted for every analysis run on a given day. Any filter analysis for which the calibration peak area is outside the range of 90% to 110% of the average calibration peak area for that day is repeated with a second punch on the following day.

Routine QC samples analyzed in the OC/EC Laboratory include (1) daily instrument blanks, (2) weekly three-point calibration standards, (3) daily mid-level calibration check standards, and (4) duplicate analyses on 10% of quartz filter samples analyzed. Each of these is described separately below.

Figure 27 shows measured total carbon (TC) for daily instrument blanks run on the New and Retrofit OC/EC analyzers. The 137 daily instrument blanks run on the New OC/EC analyzer gave a mean total FID response corresponding to 0.060 μg C/cm^2 with a standard deviation of 0.040 μg C/cm^2, and the 121 daily instrument blanks run on the Retrofit OC/EC analyzer gave a mean total FID response corresponding to 0.055 μg C/cm^2 with a standard deviation of 0.044 μg C/cm^2. None of the daily instrument blanks run on either instrument exceeded the acceptance criterion even after the acceptable blank level was reduced from 1 μg C/cm^2 to 0.3 μg C/cm^2 on July 17, 2000.

Figure 28 shows linearity (as R^2) for all 3-point calibrations run on both instruments. Both the New and Retro OC/EC analyzers met the $R^2 \geq 0.99$ requirement for every 3-point calibration.

Percent recovery of standards is used to make sure the instruments are functioning properly and are still calibrated correctly. Figure 29 shows percent recovery on the New OC/EC analyzer for each of the three (low, middle, and high) calibration standards used for each three-point calibration. Figure 30 shows the same data for the Retrofit OC/EC analyzer.

Response factors for the flame ionization detector (FID) are used to monitor FID performance. Figures 31 and 32 show FID response factors for each of the three calibrations standards and the average FID response factor for each three-point calibration on the New and Retrofit instruments, respectively.

Duplicate measurements are used to monitor the uniformity of filter loading and to indicate instrument stability. The acceptance criteria for duplicate measurements (in the Table above) are based on a significant absolute uncertainty at low (< 5 μg C/cm^2) TC loadings and the relative uncertainty at higher TC loadings. Figures 33 and 34 show RPD of duplicate measurements versus filter concentration (μg C/cm^2) for the New and Retrofit instruments, respectively. Insets on the two figures show total number of duplicates run on each instrument and the numbers of filters that passed and that failed the appropriate duplicate criteria. Filters that failed to meet the appropriate duplicate acceptance criterion were flagged as having a nonuniform filter deposit.

2.3.3 Data Validity Discussion

2.3.3.1 Invalid Data Due to OC/EC Laboratory Errors – The ability to take a second or third punch from a quartz filter for analysis allows the OC/EC analyst to avoid invalidating data due to OC/EC Laboratory error except in extreme cases when an entire filter (or half-filter aliquot) is involved in an error. So far, this has occurred only when a half-filter aliquot arrived at the OC/EC Laboratory in pieces so small that a full punch could not be taken as a single piece. Quartz filters are almost always torn around the edges during removal from the cassette filter holder in the SHAL but are only flagged as torn (1) by SHAL personnel if they arrive at RTI damaged or (2) by the OC/EC analyst if there is no portion of the filter large enough for the removal of a full punch for analysis as a single piece. The second occurrence has happened only twice and, so far, has happened only when a quartz filter was cut in half for analysis by two different laboratories.

2.3.3.2 Invalid Data Due to Other Causes – The OC/EC Laboratory simply analyzes filters that are delivered from the SHAL without any knowledge of the sampling or other field and transport data associated with those filters. OC/EC Laboratory personnel do not know if data for a filter will be invalidated for causes other than those associated with the OC/EC analysis.

2.3.4 Summary of Audit Findings and Recommendations

A copy of the audit findings and recommendations are given in Appendix A, and RTI's responses to those findings and recommendations are given in Appendix B.

2.3.5 Corrective Actions Taken

Major changes were made to both New and Retrofit OC/EC carbon analyzers during the period August 7 through 29, 2000. On August 7, 2000, the original Teflon® gas supply lines on both instruments were replaced with Alltech NO-OX™ tubing, and the quartz oven in the New OC/EC analyzer was replaced. On August 8, the software used to control both systems was upgraded to prevent the computers from crashing, and oxygen traps were installed in the helium supply lines for both instruments. On August 24, new inline regulators with stainless steel diaphragms were installed in the helium supply lines for both instruments. On August 29, 2000, indicating oxygen traps were installed downstream from the non-indicating oxygen traps installed earlier.

The changes to the systems caused a dramatic change in the OC-EC split time but no change in TC measurements. The EC to TC and OC to TC ratios did not change for the Retrofit instrument and changed only slightly for the New instrument. The slight change for the New instrument could have been caused by a defective quartz oven that was installed by instrument manufacturer's technician on March 21, 2000, and used until August 7, 2000.

2.4 X-ray Fluorescence Laboratory

2.4.1 Description of QC Checks Applied

QC elements for the analysis of elements by EDXRF, their frequency of application and control limits, and corrective actions are shown in Table 7.

Table 7. QC Procedures Used to Analyze EDXRF Elements

QC Element	Frequency	Control Limits	Corrective Action
Calibration	as needed	--	--
Calibration verification	weekly	within NIST uncertainties	recalibrate
Instrument precision	once per batch of $\leq$ 15	95–105% recovery	batch reanalysis
Excitation condition check	every sample	within analysis uncertainty	sample reanalysis
Sample replicate precision	10%	± 5 RPD	batch reanalysis

The two-sigma (95 percent confidence level) detection limits in units of ng/cm^2 are calculated from the analysis of a blank Teflon filter as follows:

$$\text{detection limit for element i} = 2\delta_i = \frac{2(2B_i)^{1/2}}{s_i t}$$

where,

B_i is the background counts for element i,
s_i is the sensitivity factor for element i,
and t is the counting lifetime.

Theoretically, detection limits may be decreased by simply increasing the counting lifetime. In practice, a point of diminishing returns is reached for real-world samples in which the background increases along with the analyte signal. At this point, further improvement in detection limits by increasing the counting time is not possible.

2.4.2 Statistical Summary of QC Results

2.4.2.1 Precision – The precision is monitored by the reproducibility of the XRF signal in counts per second. The counts for a select element are measured for each of the targets used. The comparison of the counts during calibration and during the run gives the measure of reproducibility or precision. Figures 35a through 35e show the following:

- Precision recovery for Si(0) with Rh L - ranges between 97% and 107%
- Precision recovery for Si(1) with Ti target - ranges between 91% and 110%

- Precision recovery for Ti(2) with Fe target - ranges between 98% and 106%
- Precision recovery for Fe(3) with Ge target - ranges between 97% and 105%
- Precision recovery for Pb(4) with Rh K - ranges between 93% and 107%

2.4.2.2 Recovery - Recovery or system accuracy is determined by the analysis of a series of NIST Standard Reference Materials filters. Recovery is calculated by comparison of a measured and expected values. Figures 36a through 36m show recovery for 12 select elements spanning the range of the 48 elements normally measured. Except for one value for lead, all recovery values for all elements ranged between 92% and 108%. One lead value was at about 111%. Note that lead was giving values generally greater than 105% but now is giving values between 95% and 105%.

2.4.2.3 Replicates – Ten percent of the filters are reanalyzed and the results for select elements are compared. Figures 37a through 37f compare replicate values for six elements through regression analysis. Note that slopes are all greater than 0.99 except for one value at 0.98 and correlation coefficients are all greater than 0.995, indicating acceptable replication.

2.4.3 Data Validity Discussion

The data presented in Section 2.4.2 indicate no problems with the XRF data. Lead has shown a change in recovery but it has remained in the ±10% window and is currently within a ±5% window. The only problems encountered were occasional tears and/or pinholes in the filters. If these were minor, and not considered to have a significant impact on the analysis results.

2.4.4 Summary of Audit Findings and Recommendations

All audit findings and recommendations are described in Appendix A to this report.

2.4.5 Corrective Actions

No changes were made in the analytical procedures used by the XRF laboratory, Chester LabNet (LabCor). No substantive corrective actions were taken,.

2.5 Sample Handling and Archiving Laboratory (SHAL)

2.5.1 Description of QC Checks Applied

Numerous QC checks are built into the SHAL procedures. These include:

- Bar-code readers are used to input identification numbers from modules, bins, containers, data forms to virtually eliminate data transcription errors.
- Barcoded labels with identification numbers are generated by computer and the ID numbers include a check-digit.
- The training of new employees includes a reciprocal check procedure, in which other SHAL technicians check the contents of each other's coolers before they are closed for shipment. This cross-checking procedure is also used when an excessive number of packing errors is reported.

2.5.2 Corrective Actions Taken

Problem: A significant number of coolers, approximately 17%, are being returned at greater than 4°C. Figure 38 illustrates the distribution of temperatures that were measured was the coolers were unpacked. The figure includes only routine shipments, and includes some shipments made after minitrends concluded. **Corrective Action:** No corrective actions have been taken by RTI. EPA may wish to consider whether additional expense is justified to reduce the number of coolers received below the target temperature of 4°C.

Problem: A major problem in the SHAL has been sites not returning coolers on the designated shipping date. This interrupts the flow of work in the SHAL laboratory and requires extra effort to determine which coolers are missing. **Corrective Action:** After discussions with EPA, it has been decided that when a cooler does not arrive on time, the SHAL supervisor will e-mail the state contact and copy the DOPO, the EPA contracting officer and the RTI Project manager. The state contact will then be responsible for notifying RTI of the coolers' status.

Problem: Early in the program some sites were complaining about leaking blue ice packs. RTI responded by purchasing small ziplock bags. **Corrective Action:** All icepacks are now packaged in ziplocks. RTI also changed the way the icepacks are loaded in the coolers. The new orientation of the icepacks attempts to keep as many as possible upright to minimize leakage.

Problem: Sites which have been scheduled and sent a set of filters for a sampling event, but do not run the sampler have been returning filters unexposed. **Corrective Action:** RTI has converted these filters to blanks and have been analyzing the filters.

Problem: One particular site was operating collocated URG samplers. This site continually returned modules in incorrect bins or returned coolers late. Because the two samplers had different style modules eventually due to the sites mixing of modules, RTI could not supply one sampler with the correct type of module. **Corrective Action:** The SHAL had to

wait until inventory was returned from the site in order to commence shipping filters for both samplers.

Problem: Early in the program, it was found that the Andersen cassette rings were too small. **Corrective Action:** New cassette rings were supplied by Andersen and placed into the SHAL inventory. The old rings were removed from service.

Problem: Unlike the other samplers used in the minitrends program, the Andersen filters are shipped without a sampling module. The filters are installed in the field. There was concern that these filters would become contaminated. **Corrective Action:** Modules were supplied to the SHAL. All Andersen filters are being installed into their modules in the SHAL, as with the other types of samplers.

2.6 Denuder Refurbishment Laboratory

The Denuder Refurbishment Laboratory was first located in RTI Building No. 6, Laboratory 206, near the SHAL. It is now located in RTI Building No. 3, Laboratory 220. The purpose of the laboratory is to clean and refurbish the coatings on various acid-gas-removing denuders used in the chemical speciation networks operated by EPA and various State and local agencies which utilize the RTI/EPA contract. The laboratory follows these protocols:

- SOP for Coating Annular Denuders with Magnesium Oxide
- SOP for Coating and Extracting Annular Denuders with Sodium Carbonate
- SOP for Coating R & P Speciation Sampler ChemComb© Denuders with Sodium Carbonate
- SOP for Coating Annular Denuders with XAD-4 Resin

2.6.1 Operational Summary

To date, only magnesium oxide denuder refurbishments have been performed. We are just beginning to coat R & P brand honeycomb denuders with sodium carbonate. Thus far, the use of XAD-4 resin coated denuders for removal of organic vapors has not been specified by EPA/OAQPS.

The laboratory has refurbished frosted glass annular denuders for the Andersen RAAS speciation sampler and the URG MASS-400 speciation sampler at three-month intervals since the program began. The Met One SASS speciation sampler aluminum honeycomb denuders have not yet been refurbished since these denuders are part of the sampling module and receive only one-sixth the exposure of the annular denuders which are part of the sampler itself and are exposed to ambient air each sampling event.

The laboratory is experienced in the preparation of sodium carbonate-coated denuders for Andersen, URG, Met One, and R&P speciation samplers. Citric acid-coated denuders can also be prepared, should they be required to removal and quantification of basic gases (i.e., ammonia and amines).

2.6.2 Problems and Corrective Actions

Only one significant problem was encountered in the first nine months of operation. One URG downtube denuder arrived at RTI broken. In RTI's opinion, the device had not been packaged properly. The site operator was alerted to the proper procedures for packaging and shipping. Since it could not be clearly established how and when the denuder had been broken, RTI had URG repair it at one-half the price of a new denuder, and thus promptly returned the denuder to service.

2.6.3 Quality Assurance Activities

Since no analysis of denuder extracts are presently conducted, the QA activities for magnesium oxide-coated denuders are confined to the following three topics.

- Obstruction-free annuluses. After coating and drying the interior, each denuder is inspected by holding it to a strong light and viewing down the tube. Thus far, only a few "bridges" of magnesium oxide coating have been noted. These bridges were removed with a thin piece of plastic film.

- Adherence of coating to surfaces. The dried coated denuder is subjected to a blast of nitrogen gas to remove non-adhering particles. The denuder is then gently tapped against a dark laboratory bench surface to ensure no visible coating particles fall out. If some do, the nitrogen gas blast is repeated and the tapping test repeated until no particles are seen.

- Uniformity of coating. Each coated denuder is visually examined to see that all surfaces have been coated. Because it is impossible to clearly see all interior surfaces, a net weight of coating is established and compared to other coated denuder weight increases. The clean, dry denuder is first weighed to the nearest 0.01 g to establish a tare weight. After coating and drying, the denuder is reweighed to the nearest 0.01 g. The uniformity of coating from denuder to denuder is approximated by comparing the net weights of magnesium oxide applied. The typical URG downtube denuder retains 1.0 g of magnesium oxide. The typical Andersen annular denuder retains 0.7 g of magnesium oxide. The amount retained by the Met One aluminum honeycomb denuder has not yet been determined.

2.7 Data Processing

2.7.1 Operational Summary

Data processing supports the following program operations:

- Forms generation in the SHAL, including generation of unique, check-digited bar code labels
- Input of bar coded information in the SHAL and other locations to reduce the frequency of input errors
- Menu-driven applications for data entry in the SHAL and for input of the Field Sampling Chain of Custody (FSCOC) sheets.
- Input of data provided in spreadsheet form by the laboratories:
  - OC/EC laboratory
  - Gravimetric laboratory
  - IC (ions) laboratory
  - XRF (elements) laboratory
- Resolution of data attribution problems between data sources
- Support of data validation, including:
  - Data reporting
  - Data screening and application of data quality flags
  - Generation of statistical summaries for QC purposes
- Generation of monthly data reports
- Input of changes requested by the state monitoring agencies based on review of monthly data reports.

2.7.2 Problems and Corrective Actions

Despite the relatively brief startup time allotted for system design and development before sampling was started in February, 2000, a working data processing system has been developed that allowed data to be acquired. Although many improvements have been made to this system during the first year of the program, the basic data base structure has remained intact, and it has not been necessary to make any major restructurings of the data base. The following describes specific problems and corrective actions related to data processing.

Problem: State monitoring agency personnel complained of difficulty understanding the data spreadsheet sent with the monthly data reports. **Corrective Action:** RTI devised two additional data reporting spreadsheets that are organized in a way that is easier for a human to read. The original spreadsheet is still being provided because it is more suitable for importing into a data base for computer analysis.

Problem: It was discovered that the Andersen samplers were calculating flow rates incorrectly, which caused all concentration results reported for the Andersen samplers to be in error. **Corrective Action:** Calibration equations were supplied by the various state agencies that were applied to the flow rate and volume data by RTI's data processing group. Corrections were made piecemeal, as the agencies installed the corrected software and recalibrated their instruments. RTI subsequently recalculated and resubmitted all the affected data.

Problem: Work stations in the SHAL were locking up frequently, resulting in inefficient operation and waste of personnel time. **Corrective Action:** Work stations are being upgraded to more modern computers and Windows NT or Windows 2000 is being installed.

Problem: Many state monitoring agency contacts, DOPOs, and other recipients of the data reports were finding that the files were too large to be accommodated by their e-mail systems. **Corrective Action:** RTI implemented a secure Web server to distribute the monthly data reports. Users are assigned passwords that entitle them to access only the data that they have been authorized to see.

2.8 Quality Assurance and Data Validation

2.8.1 QA Activities

QA activities directly related to data validation are described in the PM2.5 Chemical Speciation Laboratory QAPP (December 2000), and include the following:

- Review of monthly data reports sent to the state monitoring agencies and EPA
  - Verification of data attribution to the correct site, POC, and date
  - Review of report formats
  - Troubleshooting when discrepancies are found
  - Running manual and partially-automated range checks
  - Reviewing validation flags

- Review of each data batch before it is sent to AIRS
  - Verification of data attribution to the correct site, POC, and date
  - Verification that changes requested by the state monitoring agencies have been correctly made by the Data Processing personnel
  - Review of data format to be sure that records and individual fields are of the correct length

- Development of Level 1 outlier screening criteria (application of Level 1 checks were done manually by the QA Officer during much of the Minitrends program, but these are being automated so that they can be run by data processing personnel in the future).

- Troubleshooting of sample and data problems that cross the boundaries between laboratories, the SHAL, and/or the data processing function

2.8.2 Data Validation Procedures (data summaries will be provided in section 3)

The full scope of the Level 0 and Level 1 procedures carried out by RTI before data are delivered to the state monitoring agencies each month are described in the Laboratory QAPP (December 2000). Data validation procedures, particularly those at Level 1, evolved significantly during the Minitrends study. The following is a brief overview of the data validation procedures carried out for the data processed during Minitrends:

Validation Step	Performed by	Type of Check	Criteria
Review of operator comments and data values on the Field Sampling Chain of Custody forms	Data input personnel	Manual (many of the Level 0 range checks are being changed to automated procedures)	Set criteria for average flow rate, exposure time, temperature, etc. are defined by EPA. These criteria were evaluated by data entry personnel during much of the Minitrends program. These flagging procedures are now being automated.
Examination of filters and modules upon return to SHAL	SHAL personnel	Manual	Filter condition, module condition, return of correct part numbers in the correct container are reviewed. Specific flags have been defined for various problems such as shipment temperature too high, damaged filter, etc.
Laboratory QA/QC criteria	Laboratory personnel	Manual	Method specific QC criteria are applied by the analysts and the appropriate data flags are transmitted along with the laboratory data. These include criteria such as: recovery percentage, duplicate agreement, blank levels, detection limits, and calibration linearity
Reconciliation of ID numbers, scheduling information, etc. in the data management system	Data Processing personnel	Automated, with manual follow-up of discrepancies	Data base integrity criteria must be met, including referential integrity, and uniqueness of primary keys. Troubleshooting is performed until the report generation software reports no data attribution errors. Follow-up procedures may include review of hardcopy documentation and/or contact with the monitoring agency.
Acceptance Range Screening (Level 0)	Data Processing Personnel	Automated, with automated flagging	Data validation codes assigned by EPA are assigned based primarily on fixed limits.
Statistical Outlier Screening (Level 1)	Data Processing and QA personnel	Semi-automated; these are currently being made fully automated	Outlier flagging criteria are defined based on percentiles of the frequency distributions of the Minitrends data. (Specific criteria were not in effect during most of the Minitrends program.)
Review of monthly data reports	State monitoring agency personnel	varies by agency	Agencies approve the Level 0 and Level 1 validation flags applied by RTI and may perform their own validation. Corrections are sent to RTI before the AIRS data are delivered.
Insertion of corrections requested by the state monitoring agencies	RTI data processing personnel	manual or semi-automated	Agencies may request that AIRS codes be changed, or that data be made valid or invalid. In one instance, data values were recomputed based on revised flow rate calibration data for the Andersen samplers following a firmware replacement on the sampler.
Review of AIRS data	RTI data processing and QA personnel	manual or semi-automated	Integrity of the data set is checked by verifying the counts by site and date and other simple procedures. Additional data validation steps are not performed.

Level 1 data validation procedures, also known as outlier checks, were under development during the Minitrends study. These include the following:

- Lower Limit on PM2.5 Mass - routine samples only
- Reconstructed Mass Balance - routine samples only
- Anion/Cation ratios - routine samples only
- Sulfur/Sulfate ratios - routine samples only
- Upper Limit on PM2.5 Mass - blank samples only.

2.8.3 Problems and Corrective Actions

Problem: It was found that additional informational flags needed to be defined to define unanticipated conditions. **Corrective Action:** The following flagging conditions were defined by RTI:

- Filter installed backwards
- Correction(s) made on data form by RTI data input personnel
- Different flow channel used (channel flow rate reassigned).

In addition, flags were added by EPA resulting in the list of applicable AIRS codes shown in Tables 8 and 9.

Problem: EPA data analysts noted that some samples were showing excessive imbalance between the reconstructed mass and particulate mass. **Corrective Action:** RTI changed from a visual check of a plot of reconstructed mass vs. particulate mass to an automated procedure that screens outliers based on historical minitrends mass ratio results. All samples that are above the 98th percentile or below the 2nd percentile, based on minitrends results, are automatically flagged. These flags are being implemented manually at present, but fully automated range screening will be added to the software soon. Flagged values will be investigated manually because it is necessary to decide whether to assign a validity status flag for suspicious data, or to assign a null value code for data considered invalid.

Problem: EPA personnel screening data noticed some data inconsistencies, including high blank concentrations, collocated concentration values that do not agree, unreasonable values for temperature and barometric pressure, and negative concentration values. **Corrective Action:** Additional automated data screening checks are being implemented. Details of the screening levels are provided in the Laboratory QAPP, December 2000. The following summarizes the changes made to the screening process as a results of the EPA comments:

- Blanks are being screened such that any concentrations above the 95th percentile of the minitrends concentration distribution are flagged.

- Where possible, collocated concentrations will be compared during data validation; however this includes only a small minority of sites.

Table 8. Airs Validity Status Codes Applicable to PM2.5 Chemical Speciation

AIRS Validity Status Code	Flag Name
1	Critical Criteria Not Met
2	Operational Criteria Not Met
3	Possible Field Contamination
4	Possible Lab Contamination
5	Outlier - Cause Unknown
6	Data Prior to QAPP Approval
A	High Winds
E	Forest Fire
F	Structural Fire
H	Chemical Spills & Indust. Accidents
I	Unusual Traffic Congestion
J	Construction/demolition
K	Agricultural Tilling
L	Highway Construction
M	Rerouting of Traffic
N	Sanding/salting of Streets
O	Infrequent Large Gatherings
P	Roofing Operations
Q	Prescribed Burning
R	Clean up after a Major Disaster
S	Seismic Activity
T	Multiple Flags; Misc.
V	Validated Value
W	Flow Rate Average out of Spec.
X	Filter Temperature Difference out of Spec.
Y	Elapsed Sample Time out of Spec.

Table 9. AIRS Null Value Codes Applicable to PM2.5 Chemical Speciation

AIRS Code		Flag Name
Original AIRS	Re-engineered AIRS	
9964	BI	Sample Lost or Damaged in Shipment
9967	AA	Sample Pressure out of Limits
9968	AB	Technician Unavailable
9969	AC	Construction/repairs in Area
9970	AD	Shelter Storm Damage
9971	AE	Shelter Temperature Outside Limits
9972	AF	Scheduled but Not Collected
9973	AG	Sample Time out of Limits
9974	AH	Sample Flow Rate out of Limits
9975	AI	Insufficient Data (Can't Calculate)
9976	AJ	Filter Damage
9977	AK	Filter Leak
9978	AL	Voided by Operator
9979	AM	Miscellaneous Void
9980	AN	Machine Malfunction
9981	AO	Bad Weather
9982	AP	Vandalism
9983	AQ	Collection Error
9984	AR	Lab Error
9985	AS	Poor Quality Assurance Results
9987	AU	Monitoring Waived
9988	AV	Power Failure (Powr)
9989	AW	Wildlife Damage
9993	BA	Maintenance/routine Repairs
9994	BB	Unable to Reach Site
9997	BE	Building/site Repair

- Range checks are being implemented for gross acceptability of temperature and barometric pressure.

- Range checks have been implemented to exclude negative concentration values, except for temperatures.

Problem: State monitoring agency personnel complained of difficulty understanding the data spreadsheet sent with the monthly data reports. **Corrective Action:** RTI devised two additional data reporting spreadsheets that are organized in a way that is easier for a human to read. The original spreadsheet is still being provided because it is more suitable for importing into a database for computer analysis.

3.0 Data Validity and Completeness

3.1 Summary of all Scheduled Samples

Routine samples were scheduled on a 1-in-6 day schedule between February 9 and April 1, 2000, after which they were sampled at a 1-in-3 day schedule. Data were batched at monthly intervals for reporting. Table 10 shows the batches that were reported between April and October. The end of the Minitrends program was July 29, 2000, but several sites continued sampling on a 1-in-3 day schedule after that date. The sampling dates comprising a batch are approximate because sample reporting sometimes lagged for individual samples due to late analysis results and other problems. To avoid confusion, RTI did not report partial results for any exposure session.

Table 11 lists the sampling sites operated during Minitrends by site name, state, and POC.

Table 12 summarizes the routine sampling events by site, date and batch number in which the data were first reported to the state monitoring agencies. If a site/date cell is missing, the exposure was not performed as a routine sample. In many cases these were converted to "unsampled blanks" which can often be used as a source of additional Trip Blank information.

Table 10. Reporting Batches for Minitrends Data

Batch Number	Sampling Dates	Report (mid-month)
1	2/9/2000 (only)	March
2	2/15 - 3/10	April
3	3/16 - 4/9	May
4	4/12 - 5/9	June
5	5/12 - 6/8	July
6	6/11 - 7/8	August
7	7/11 - 8/10	September
8	8/13 - 9/6	October

Table 11. Minitrends Site Summary

Site Name	State	AIRS_CODE	POC
Phoenix Supersite	AZ	040139997	5
Phoenix Supersite	AZ	040139997	6
Phoenix Supersite	AZ	040139997	7
Sacramento - Del Paso Manor	CA	060670006	5
San Jose - Fourth Street	CA	060850004	5
Lewis	FL	120571075	5
Lewis	FL	120571075	6
Chicago - SE Police Station	IL	170310050	5
Chicago - SE Police Station	IL	170310050	6
Roxbury (Boston)	MA	250250042	5
Roxbury (Boston)	MA	250250042	6
Roxbury (Boston)	MA	250250042	7
Blair Street	MO	295100085	5
Blair Street	MO	295100085	6
NY Botanical Gardens	NY	360050083	6
NY Botanical Gardens	NY	360050083	7
Bismarck Residential	ND	380150003	5
Bismarck Residential	ND	380150003	6
Portland - SE Lafayette	OR	410510080	6
PHILA - AMS Laboratory	PA	421010004	5
Deer Park	TX	482011039	5
Deer Park	TX	482011039	6
Hawthorne	UT	490353006	5
Hawthorne	UT	490353006	6
Georgetown	WA	530330032	5

Table 12. Summary of Routine Exposure Reporting Batches

SITE NAME	ST	POC	2/9	2/15	2/21	2/27	3/4	3/10	3/16	3/22	3/28	4/3	4/6	4/9
Phoenix Supersite	AZ	5			2	2	2	2	3	3	3	3	3	4
Phoenix Supersite	AZ	6			2	2	2	2	3	3	3	3	3	4
Phoenix Supersite	AZ	7			2	2	2	2	3	3	3	3	3	3
Fresno - First Street	CA	5	1	2	2	2	2	2	3	3	3	3	3	3
Fresno - First Street	CA	6												
Sacramento - Del Paso Manor	CA	5	1	2	2	2	2	2	3	3	3	3	3	3
San Jose - Fourth Street	CA	5	1	2	2	2	2	2	3	3	3	3	3	3
Lewis	FL	5	1	2	2				3	3	3	3	3	3
Lewis	FL	6	1	2	2	2	2	2	3	3	3	3	3	4
Chicago - SE Police Station	IL	5	1	2	2	2	2	2	3	3	3	3	3	3
Chicago - SE Police Station	IL	6	1	2	2	2	2	2	3	3	3	3	3	
Roxbury (Boston)	MA	5	1		2	2	2	2	3	3	3	3	3	
Roxbury (Boston)	MA	6	1	2	2	2	2	2	3	3	3	3	3	
Roxbury (Boston)	MA	7	1	2	2	2	2	2	3	3	3	3	3	
Blair Street	MO	5	1	2	2	2	2	2	3	3	3	4	3	3
Blair Street	MO	6	1	2	2	2	2	2	3	3	3	3	3	3
NY Botanical Gardens	NY	5												
NY Botanical Gardens	NY	6	1	2	2	2	2	2	3	3	3	3	3	3
NY Botanical Gardens	NY	7	1			2	2	2	3	3	3	3	3	3
Bismarck Residential	ND	5	1	2	2	2	2	2	3	3	3	3	3	3
Bismarck Residential	ND	6	1	2	2	2				3	3	3	3	4
Portland - SE Lafayette	OR	5	1	2	2	2	2	2	3	3	3	3	3	3
Portland - SE Lafayette	OR	6	1	2	2	2	2	2	3	3	3	3	3	3
PHILA - AMS Laboratory	PA	5	1	2	2	2	2	2	3	3	3	3	3	3
PHILA - AMS Laboratory	PA	6	1	2	2	2	2	2	3	3	3	3	3	4
Deer Park	TX	5		2	2	2	2	2		3	3	3	3	
Deer Park	TX	6		2	2	2	2	2		3	3	3	4	
Hawthorne	UT	5	1	2	2	2	2	2	3	3	3	3		3
Hawthorne	UT	6	1	2	2	2	2	2	3	3	3	3	3	3
Georgetown	WA	5												
Beacon Hill	WA	5	1	2	2	2	2	2	3	3	3	3	3	3
Beacon Hill	WA	6	1	2	2	2	2	2	3	3	3	3	3	4

Table 12 (continued)

SITE NAME	ST	POC	4/12	4/15	4/18	4/21	4/24	4/27	4/30	5/3	5/6	5/9	5/12	5/15	5/18	5/21	5/24	5/27	5/30	6/2	6/5
Phoenix Supersite	AZ	5	4	4	4	4	4	4	4	4	4	4	5	5		5	5	5	5	5	
Phoenix Supersite	AZ	6	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	
Phoenix Supersite	AZ	7	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	
Fresno - First Street	CA	5	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	5
Fresno - First Street	CA	6							4	4	4	4	5	5	5	5	5	5	5	5	5
Sacramento - Del Paso Manor	CA	5	4		4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	5
San Jose - Fourth Street	CA	5	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	
Lewis	FL	5	4	4	4	4	4	4	4	4	4	4	5	5	5	5		5	5	5	5
Lewis	FL	6	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	5
Chicago - SE Police Station	IL	5	4	4	4	4	4	4	4	4	4	4	5		5	5	5	5	5	5	5
Chicago - SE Police Station	IL	6	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	5
Roxbury (Boston)	MA	5	4	4		4			4		4	4	5		5	5	5	5		5	5
Roxbury (Boston)	MA	6	4	4		4			4		4	4	5		5	5	5	5		5	
Roxbury (Boston)	MA	7	4	4		4		4	4		4	4	5		5	5	5	5		5	
Blair Street	MO	5	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5		5	5
Blair Street	MO	6		4	4	4	4	4	4	4	4	4	5	5	5	5	5	5		5	5
NY Botanical Gardens	NY	5		4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	5
NY Botanical Gardens	NY	6	4	4	4	4	4	4	4	4		4	5	5	5	5	5	5	5	5	5
NY Botanical Gardens	NY	7	4	4	4	4	4	4	4	4		4	5	5	5	5	5	5	5	5	5
Bismarck Residential	ND	5	4			4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	5
Bismarck Residential	ND	6	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	6	5
Portland - SE Lafayette	OR	5	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	5
Portland - SE Lafayette	OR	6	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5				
PHILA - AMS Laboratory	PA	5	4	4	4	4	4	4	4	4	4	4	5	5	5		5	5	5	5	5
PHILA - AMS Laboratory	PA	6	3	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	5
Deer Park	TX	5	4	4		4		4	4	4	4	4	5		5		5	5		5	
Deer Park	TX	6	4	4		4	5	4	4	4	4	4	5	5	5		5	5		5	
Hawthorne	UT	5		4	4	4		4	4	4	4	4	5	5	5	5	5	5	5	5	5
Hawthorne	UT	6	4	4	4	4		4		4	4	4	5	5	5	5	5	5	5	5	5
Georgetown	WA	5							4	4	4	4	5	5	5	5	5	5	5	5	6
Beacon Hill	WA	5	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	5
Beacon Hill	WA	6	4	4	4	4	4	4	4	4	4	4	5	5	5	5	5	5	5	5	6

Table 12 (continued)

SITE NAME	ST	POC	6/8	6/11	6/14	6/17	6/20	6/23	6/26	6/29	7/2	7/5	7/8	7/11	7/14	7/17	7/20	7/23	7/26	7/29
Phoenix Supersite	AZ	5	6	6	6	6	6	6	6	6	6	6	6	7	7	7	7		7	7
Phoenix Supersite	AZ	6	6	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Phoenix Supersite	AZ	7	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Fresno - First Street	CA	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Fresno - First Street	CA	6	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Sacramento - Del Paso Manor	CA	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
San Jose - Fourth Street	CA	5	5	6	6	6	6	6	6	6	6	6	6	7	7		7	7	7	7
Lewis	FL	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Lewis	FL	6	6	6	6	6	6		6	6	6	6	6	7	7	7	7	7	7	7
Chicago - SE Police Station	IL	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Chicago - SE Police Station	IL	6	6	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	8	7
Roxbury (Boston)	MA	5			6	6		6		6	6			7	7		7	7	7	7
Roxbury (Boston)	MA	6	5	6	6	6	6	6		6	6		6	7	7	7	7	7	7	7
Roxbury (Boston)	MA	7	6	6	6	6	6	6		6			6	7	7		7	7	7	7
Blair Street	MO	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Blair Street	MO	6	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
NY Botanical Gardens	NY	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
NY Botanical Gardens	NY	6	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
NY Botanical Gardens	NY	7	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Bismarck Residential	ND	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Bismarck Residential	ND	6	6	6	6	6	6	8	6		7						7	7	7	7
Portland - SE Lafayette	OR	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Portland - SE Lafayette	OR	6	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
PHILA - AMS Laboratory	PA	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
PHILA - AMS Laboratory	PA	6	6	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Deer Park	TX	5	5	6		6		6		7			7	7	7		7	7	7	7
Deer Park	TX	6	6	6	6	6	6	6		7			6	7	7		7	7	7	7
Hawthorne	UT	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Hawthorne	UT	6	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Georgetown	WA	5	6	6	6	6	6	6	6	6	6	6	6	7	7					
Beacon Hill	WA	5	5	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7
Beacon Hill	WA	6	6	6	6	6	6	6	6	6	6	6	6	7	7	7	7	7	7	7

Table 12 (continued)

SITE NAME	ST	POC	8/1	8/4	8/7	8/10	8/13	8/16	8/19	8/22	8/25	8/28	8/31
Phoenix Supersite	AZ	5	7	7	7	7		8	8	8	8	8	8
Phoenix Supersite	AZ	6											
Phoenix Supersite	AZ	7											
Fresno - First Street	CA	5											
Fresno - First Street	CA	6	7	7	7	7	8	8	8	8	8	8	8
Sacramento - Del Paso Manor	CA	5	7	7	7	7	8	8	8	8	8	8	8
San Jose - Fourth Street	CA	5	7	7	7	7	8	8	8	8	8	8	8
Lewis	FL	5											
Lewis	FL	6	7	7	7	7	8	8	8	8	8	8	8
Chicago - SE Police Station	IL	5											
Chicago - SE Police Station	IL	6	7	7	7	7	8	8	8	8	8	8	8
Roxbury (Boston)	MA	5											
Roxbury (Boston)	MA	6											
Roxbury (Boston)	MA	7	7	7									
Blair Street	MO	5											
Blair Street	MO	6	7	7	7	7	8	8	8	8	8	8	8
NY Botanical Gardens	NY	5	7	7	7	7	8	8	8	8	8	8	8
NY Botanical Gardens	NY	6											
NY Botanical Gardens	NY	7											
Bismarck Residential	ND	5											
Bismarck Residential	ND	6	7	7	7	7	8	8	8	8			8
Portland - SE Lafayette	OR	5	7	7	7	7	8	8	8	8	8	8	8
Portland - SE Lafayette	OR	6											
PHILA - AMS Laboratory	PA	5											
PHILA - AMS Laboratory	PA	6	7	7	7	7	8	8	8	8	8	8	8
Deer Park	TX	5											
Deer Park	TX	6	7	7		8				8			8
Hawthorne	UT	5											
Hawthorne	UT	6	7	7	7	7	8	8	8	8	8	8	8
Georgetown	WA	5				7	8	8	8	8	8	8	8
Beacon Hill	WA	5											
Beacon Hill	WA	6	7	7	7	7	8	8	8	8	8	8	8

Blanks were scheduled by the SHAL and were run as shown in Table 13. Blank data are not submitted to AIRS, but are reported to the state monitoring agencies and to EPA for statistical analysis.

Table 13. Summary of Scheduled Blanks for Minitrends

Type of Blank	Dates Run
Trip Blanks	• 2/9/2000 • 6/17/2000
Field Blanks	• 2/27/2000 • 4/3/2000 • 5/12/2000 • 6/2/2000 • 7/14/2000 • 8/1/2000 (only sites continuing after Minitrends)

3.2 Scheduled Samples Not Exposed

Samples scheduled for routine exposures were sometimes not exposed as planned and were sent back to RTI. Most of these were analyzed and were designated "unsampled blanks." Table 14 summarizes the unsampled blanks created between February 9 and July 29, 2000.

Table 15 summarizes the unsampled blanks by scheduled date that the sample was to have been run.

Table 14. Unsampled Blanks

SITE NAME	STATE	POC	Count of Unsampled
Phoenix Supersite	AZ	5	1
Phoenix Supersite	AZ	6	1
Phoenix Supersite	AZ	7	1
Sacramento - Del Paso Manor	CA	5	1
San Jose - Fourth Street	CA	5	2
Lewis	FL	5	1
Lewis	FL	6	1
Chicago - SE Police Station	IL	5	1
Chicago - SE Police Station	IL	6	1
Roxbury (Boston)	MA	5	12
Roxbury (Boston)	MA	6	9
Roxbury (Boston)	MA	7	11
Blair Street	MO	5	1
Blair Street	MO	6	1
NY Botanical Gardens	NY	6	1
NY Botanical Gardens	NY	7	1
Bismarck Residential	ND	5	2
Bismarck Residential	ND	6	0
Portland - SE Lafayette	OR	6	4
PHILA - AMS Laboratory	PA	5	1
Deer Park	TX	5	15
Deer Park	TX	6	10
Hawthorne	UT	5	3
Hawthorne	UT	6	2
Georgetown	WA	5	3

Table 15. Unsampled Blanks by Scheduled Date

Date	Count	Date	Count
2/9	1	5/30	8
2/15	1	6/2	1
3/16	2	6/5	9
4/6	1	6/8	1
4/9	6	6/11	1
4/12	1	6/14	1
4/15	2	6/20	1
4/18	6	6/23	1
4/24	6	6/26	5
4/30	1	7/2	3
5/3	3	7/5	5
5/6	2	7/17	5
5/15	5	7/20	1
5/21	3	7/23	1
5/24	1	7/26	1
5/27	1	7/29	1

3.3 Completeness Summaries and Frequency of AIRS Null Value Codes

AIRS Null Value Codes indicate exposures that have been invalidated either in the field, in the laboratory, or by the state monitoring agency. Table 16 shows the total number of records in each delivery batch group that were flagged with an AIRS Null Value Code. Table 17 expresses this as a percentage, and also shows the percentage of data not invalidated. The descriptions of the null value codes and the dates corresponding to the delivery batches are given in Section 2.8.

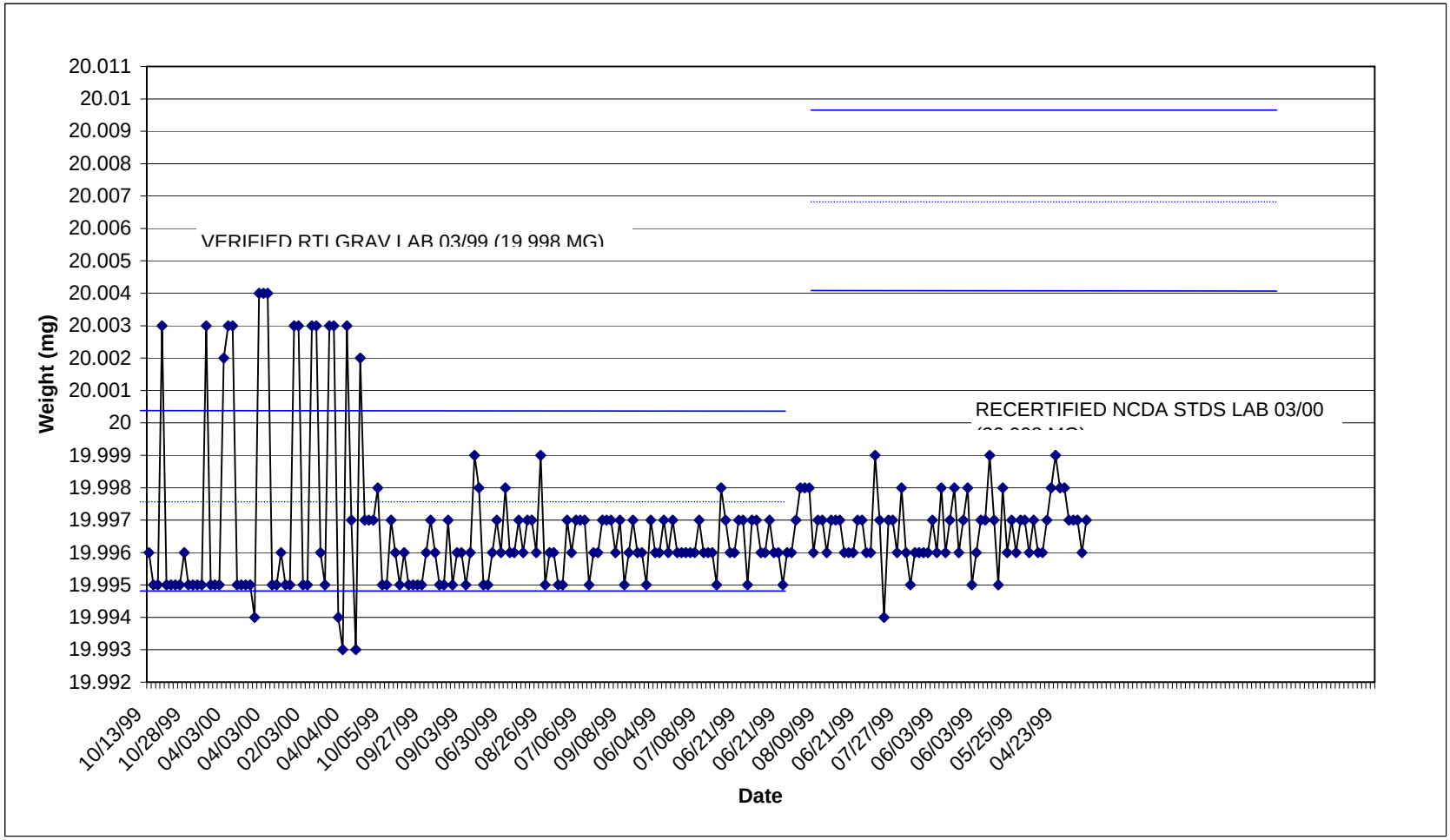
Table 16. Summary of Invalidated AIRS Data by Data Delivery Batch

AIRS CODE	Delivery Batch Number						
	1	**2**	**3**	**4**	**5**	**6**	**7**
9973		185		52	57	116	60
9974	148	192	85	81	579	377	120
9976		6	3	3	3	9	3
9978			3				
9979	70	292	475	206	393	166	209
9980		55	55	67	114	10	
9983				388	69	187	450
9984		3		63	11		3
9985				9			
9988						175	57
None	794	7241	8615	16541	15033	16682	15153
Total	1012	7974	9236	17410	16529	17722	16055

Table 17. Percentage of Invalidated AIRS Data by Data Delivery Batch

AIRS Code	Delivery Batch Number						
	1	**2**	**3**	**4**	**5**	**6**	**7**
9973		2.3%		0.3%	0.4%	0.7%	0.4%
9974	14.6%	2.4%	0.9%	0.5%	3.6%	2.1%	0.7%
9976		0.1%	0.0%	0.0%	0.0%	0.1%	0.0%
9978			0.0%				
9979	6.9%	3.7%	5.1%	1.2%	2.4%	0.9%	1.3%
9980		0.7%	0.6%	0.4%	0.7%	0.1%	
9983				2.2%	0.4%	1.1%	2.8%
9984		0.0%		0.4%	0.1%		0.0%
9985				0.1%			
9988						1.0%	0.4%
none	78.5	90.8	93.4	94.9	92.4	94	94.4

Figures 1 through 38 are contained in the following pages.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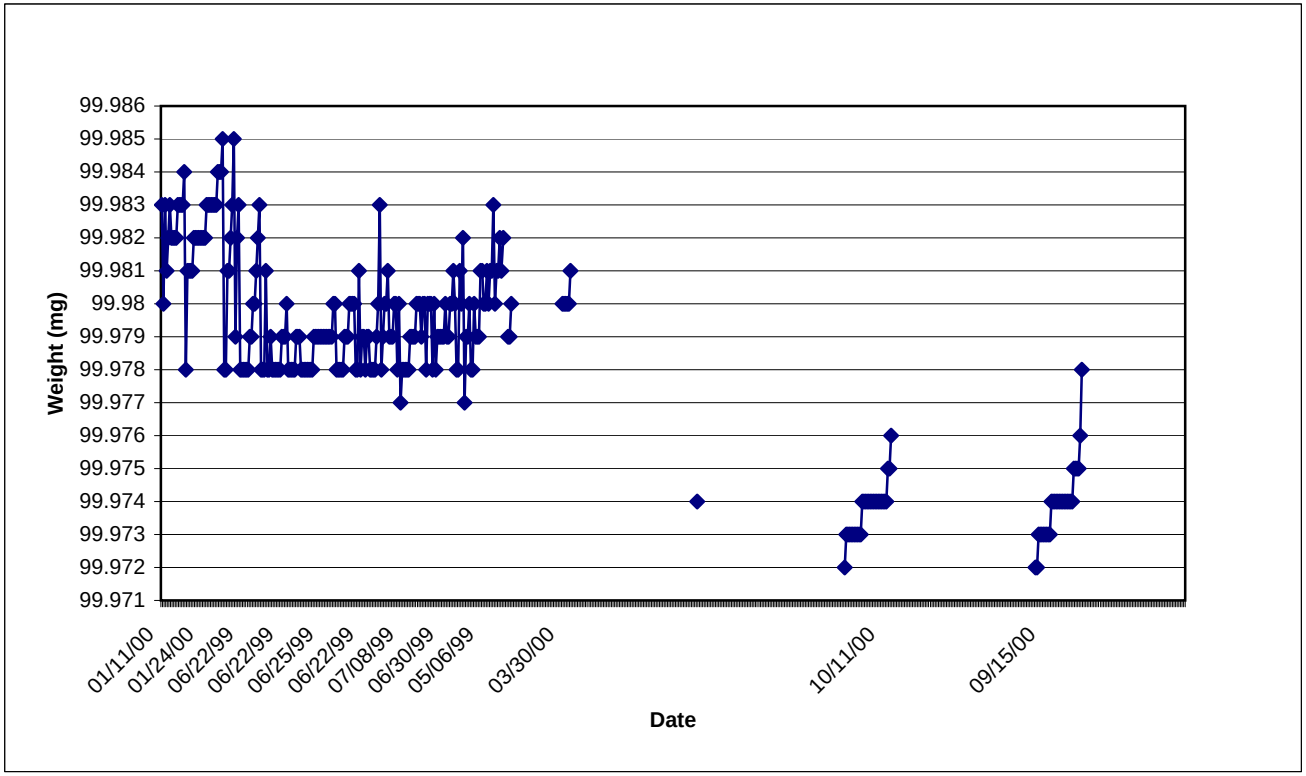
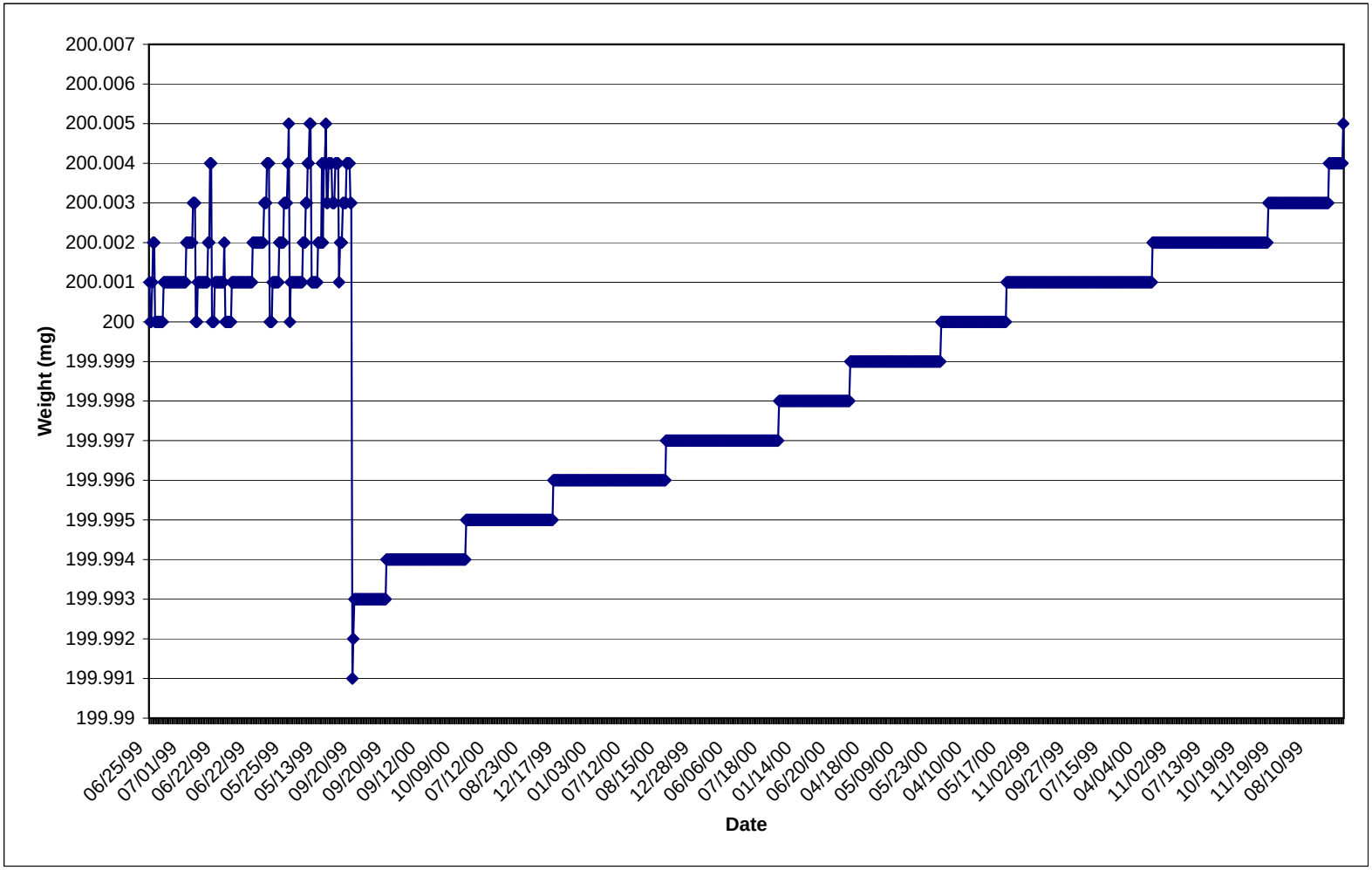
20.009
20.008
20.007
20.006
20.005
20.004
20.003
20.002
20.001
20
19.999
19.998
19.997
19.996
19.995
19.994
19.993
19.992
Weight (mg)
VERIFIED RTI GRAV LAB 03/99 (19.998 MG)
RECERTIFIED NCDA STDS LAB 03/00
10/13/99
10/28/99
04/03/00
04/03/00
02/03/00
04/04/00
10/05/99
09/27/99
09/03/99
06/30/99
08/26/99
07/06/99
09/08/99
06/04/99
07/08/99
06/21/99
06/21/99
08/09/99
06/21/99
07/27/99
06/03/99
06/03/99
05/25/99
04/23/99
Date


99.986
99.985
99.984
99.983
99.982
99.981
99.98
99.979
99.978
99.977
99.976
99.975
99.974
99.973
99.972
99.971
Weight (mg)
01/11/00
01/24/00
06/22/99
06/22/99
06/25/99
06/22/99
07/08/99
06/30/99
05/06/99
03/30/00
10/11/00
09/15/00
Date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200.006
200.005
200.004
200.003
200.002
200.001
200
199.999
199.998
199.997
199.996
199.995
199.994
199.993
199.992
199.991
199.99
Weight (mg)
06/25/99
07/01/99
06/22/99
06/22/99
05/25/99
05/13/99
09/20/99
09/20/99
09/12/00
10/09/00
07/12/00
08/23/00
12/17/99
01/03/00
07/12/00
08/15/00
12/28/99
06/06/00
07/18/00
01/14/00
06/20/00
04/18/00
05/09/00
05/23/00
04/10/00
05/17/00
11/02/99
09/27/99
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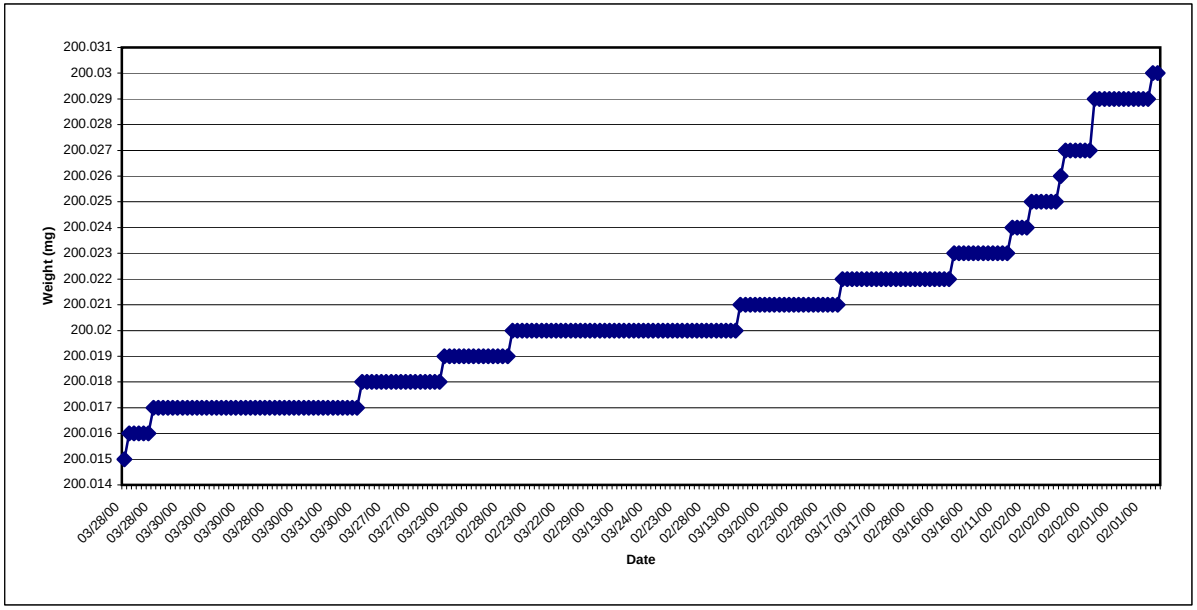
11/02/99
07/13/99
10/19/99
11/19/99
08/10/99
Date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200.03
200.029
200.028
200.027
200.026
200.025
200.024
200.023
200.022
200.021
200.02
200.019
200.018
200.017
200.016
200.015
200.014
03/28/00
03/29/00
03/30/00
03/30/00
03/30/00
03/28/00
03/30/00
03/31/00
03/30/00
03/27/00
03/27/00
03/23/00
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02/23/00
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02/29/00
03/13/00
03/24/00
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02/28/00
03/16/00
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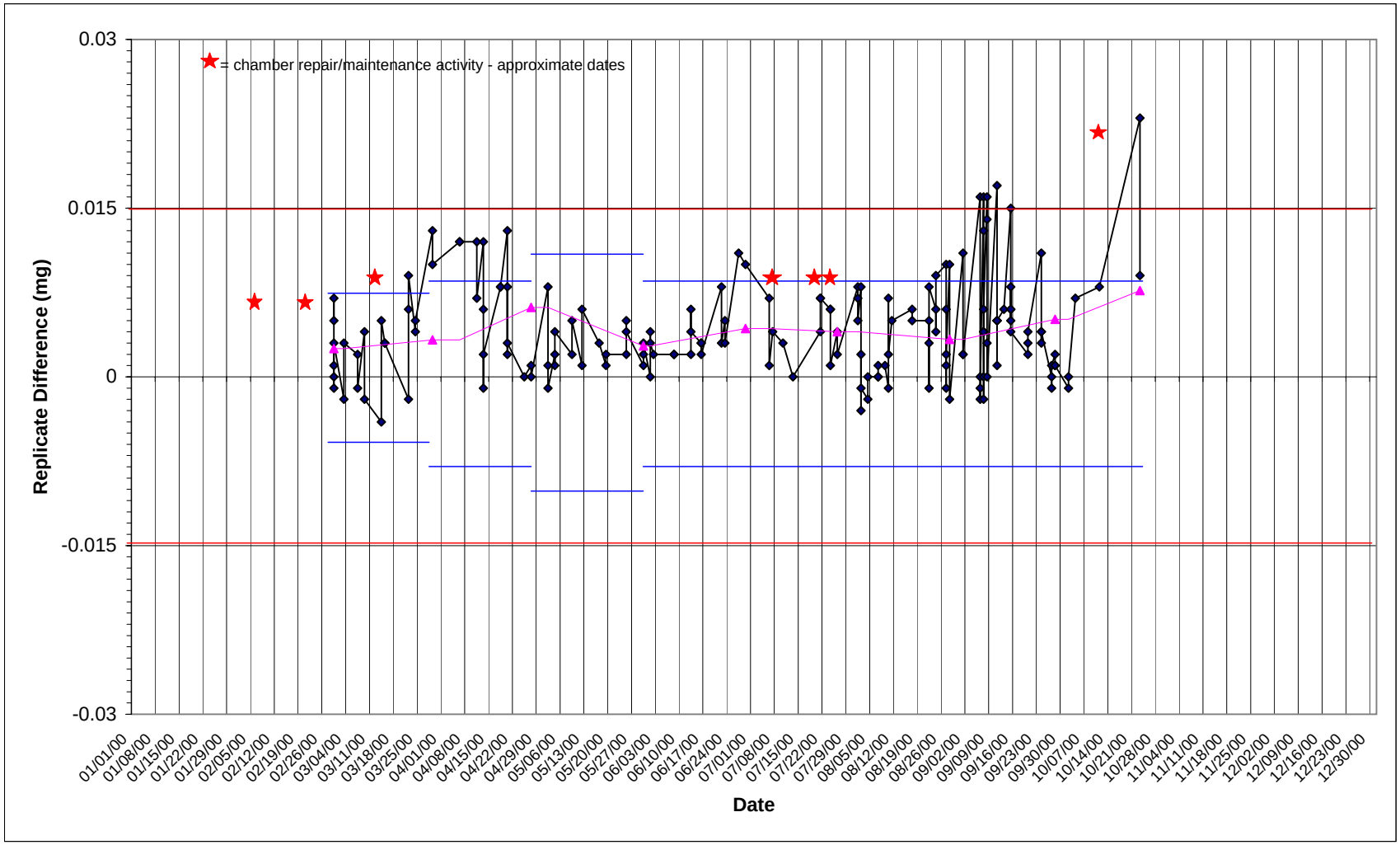
02/02/00
02/02/00
02/02/00
02/01/00
02/01/00
Date


= chamber repair/maintenance activity - approximate dates
0.03
0.015
0
-0.015
-0.03
Replicate Difference (mg)
01/01/00
01/08/00
01/15/00
01/22/00
01/29/00
02/05/00
02/12/00
02/19/00
02/26/00
03/04/00
03/11/00
03/18/00
03/25/00
04/01/00
04/08/00
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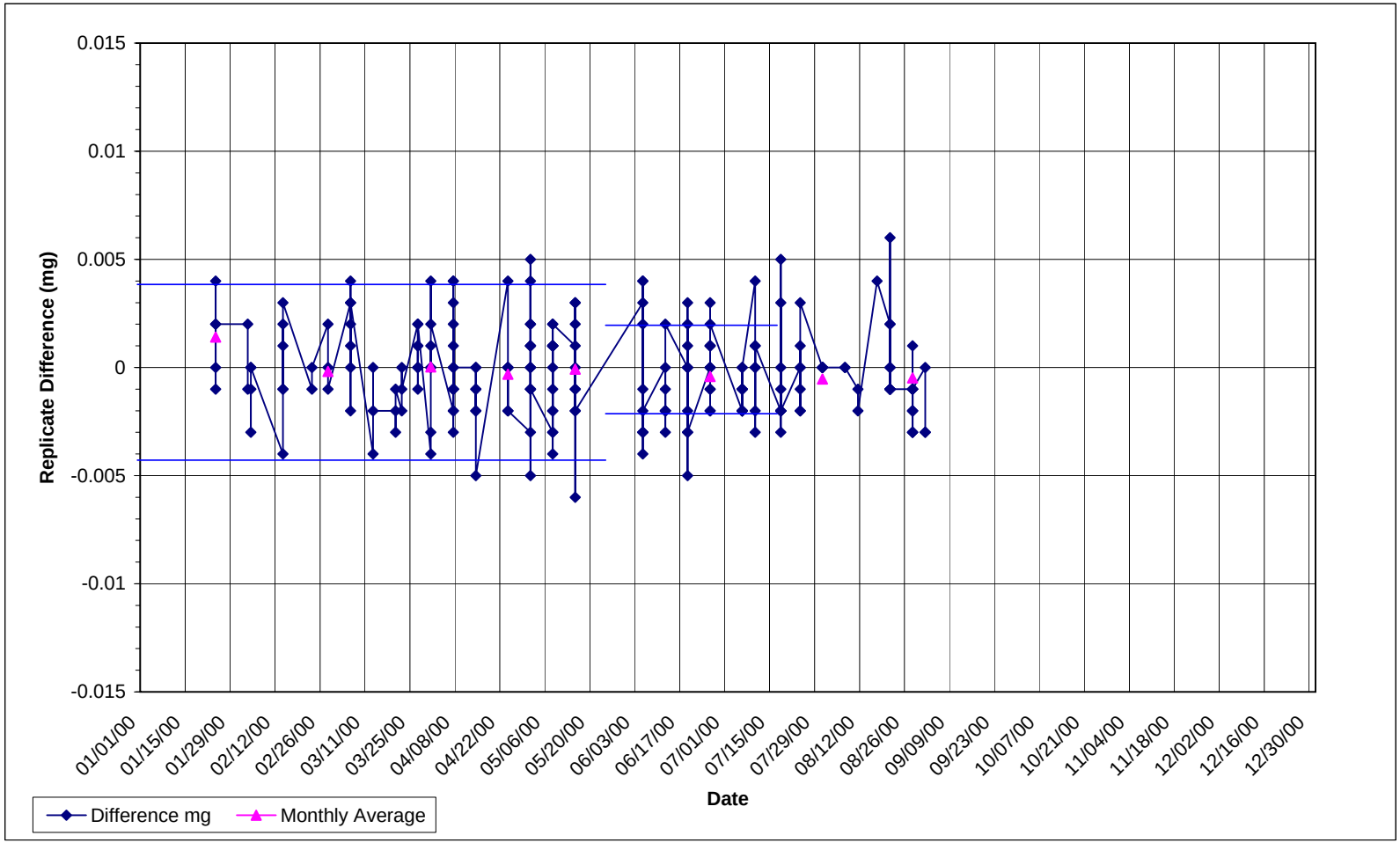
11/04/00
11/11/00
11/18/00
11/25/00
12/02/00
12/09/00
12/16/00
12/23/00
12/30/00
Date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0.01
0.005
0
-0.005
-0.01
-0.015
Replicate Difference (mg)
01/01/00
01/15/00
01/29/00
02/12/00
02/26/00
03/11/00
03/25/00
04/08/00
04/22/00
05/06/00
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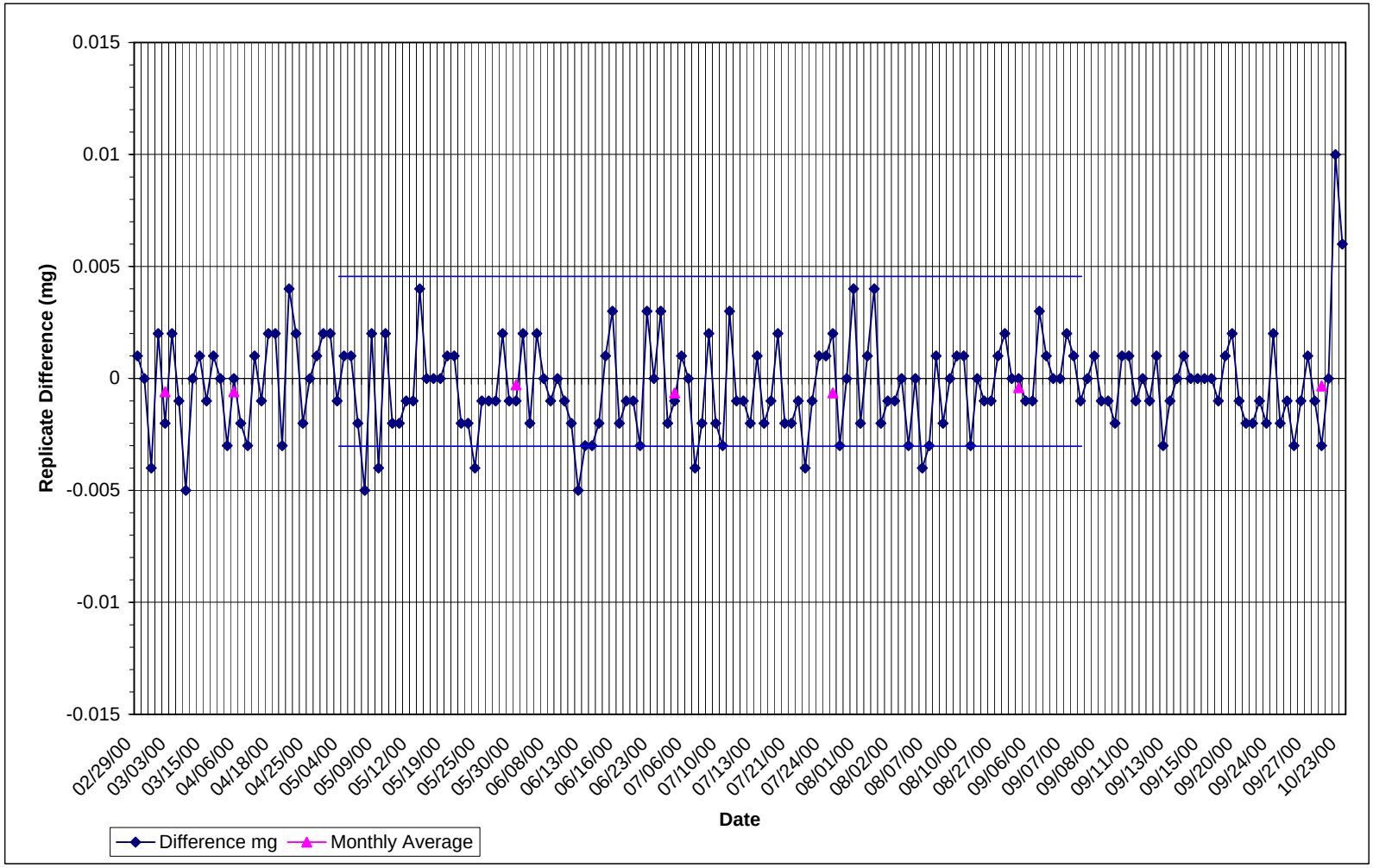
06/17/00
07/01/00
07/15/00
07/29/00
08/12/00
08/26/00
09/09/00
09/23/00
10/07/00
10/21/00
11/04/00
11/18/00
12/02/00
12/16/00
12/30/00
Date
Difference mg
Monthly Average


0.015
0.01
0.005
0
-0.005
-0.01
-0.015
Replicate Difference (mg)
02/29/00
03/03/00
03/15/00
04/06/00
04/18/00
04/25/00
05/04/00
05/09/00
05/12/00
05/19/00
05/25/00
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08/02/00
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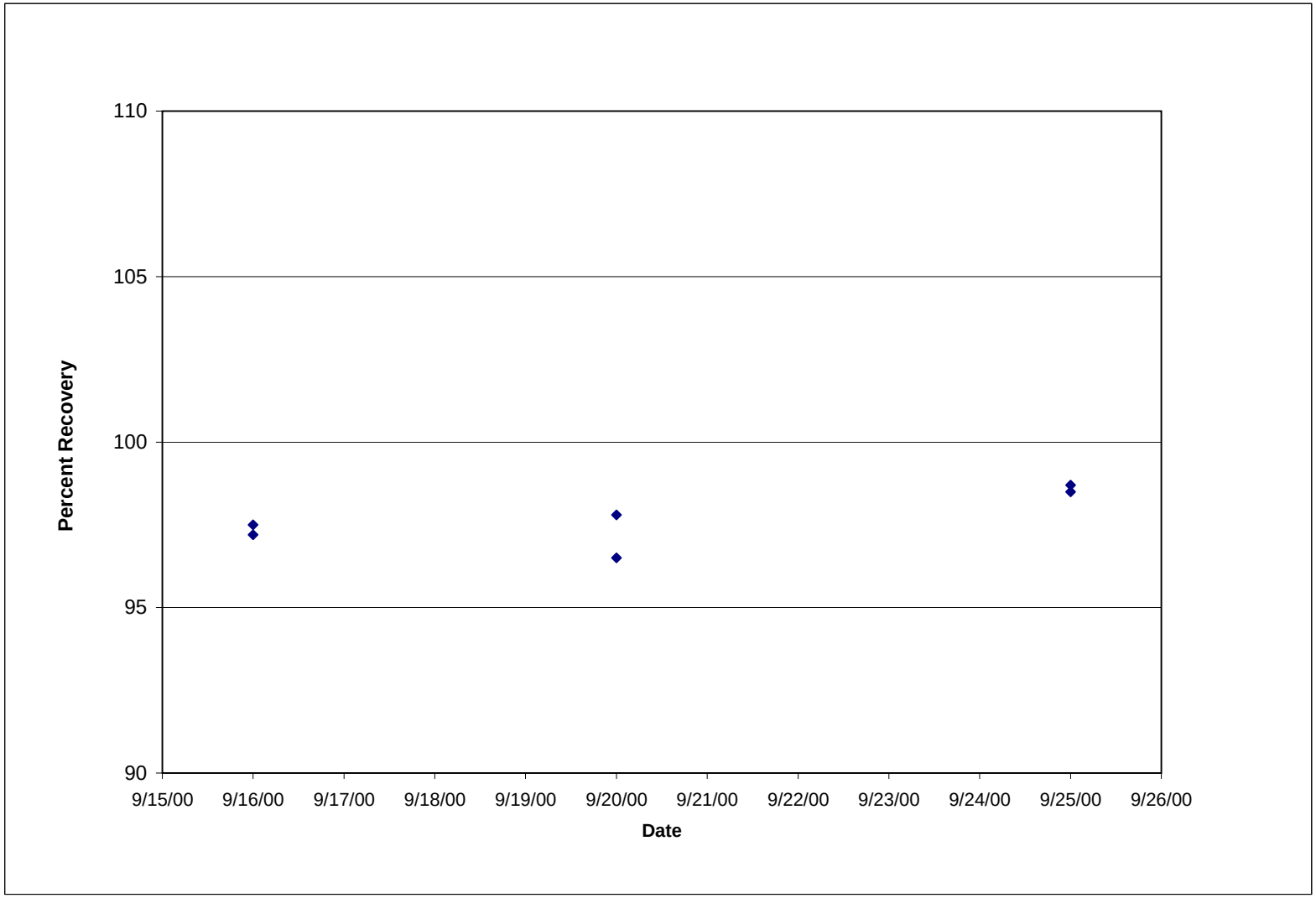
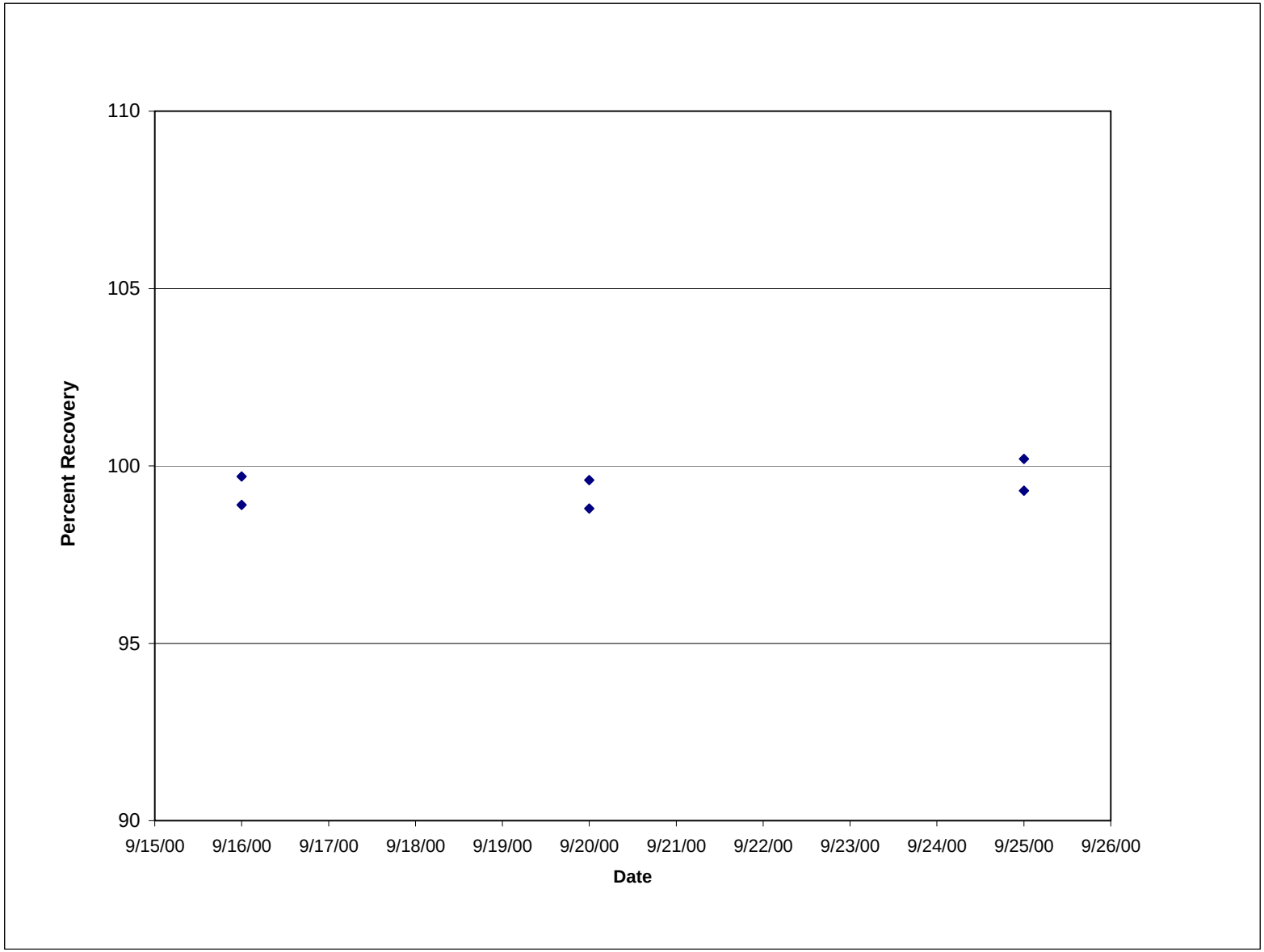
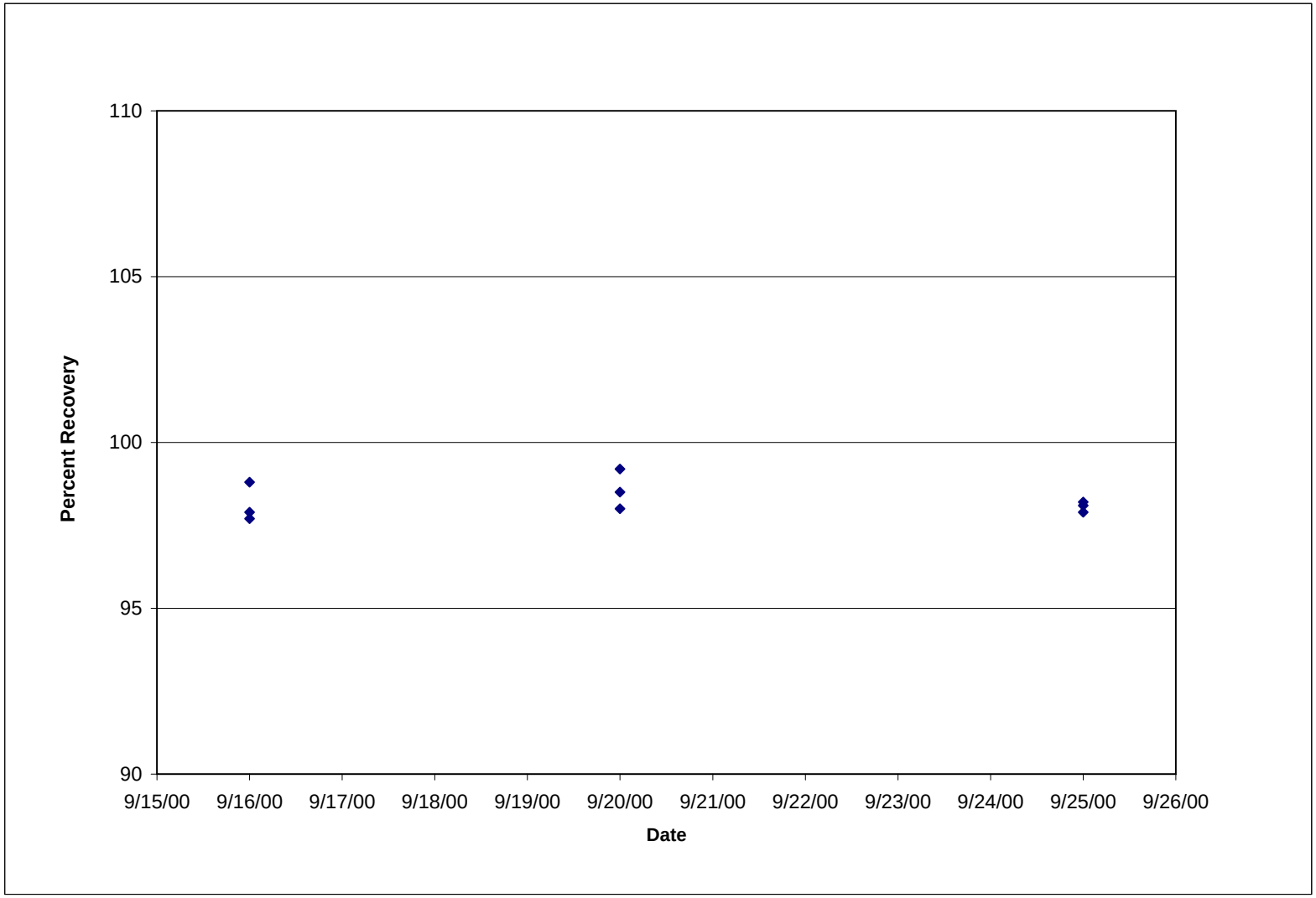
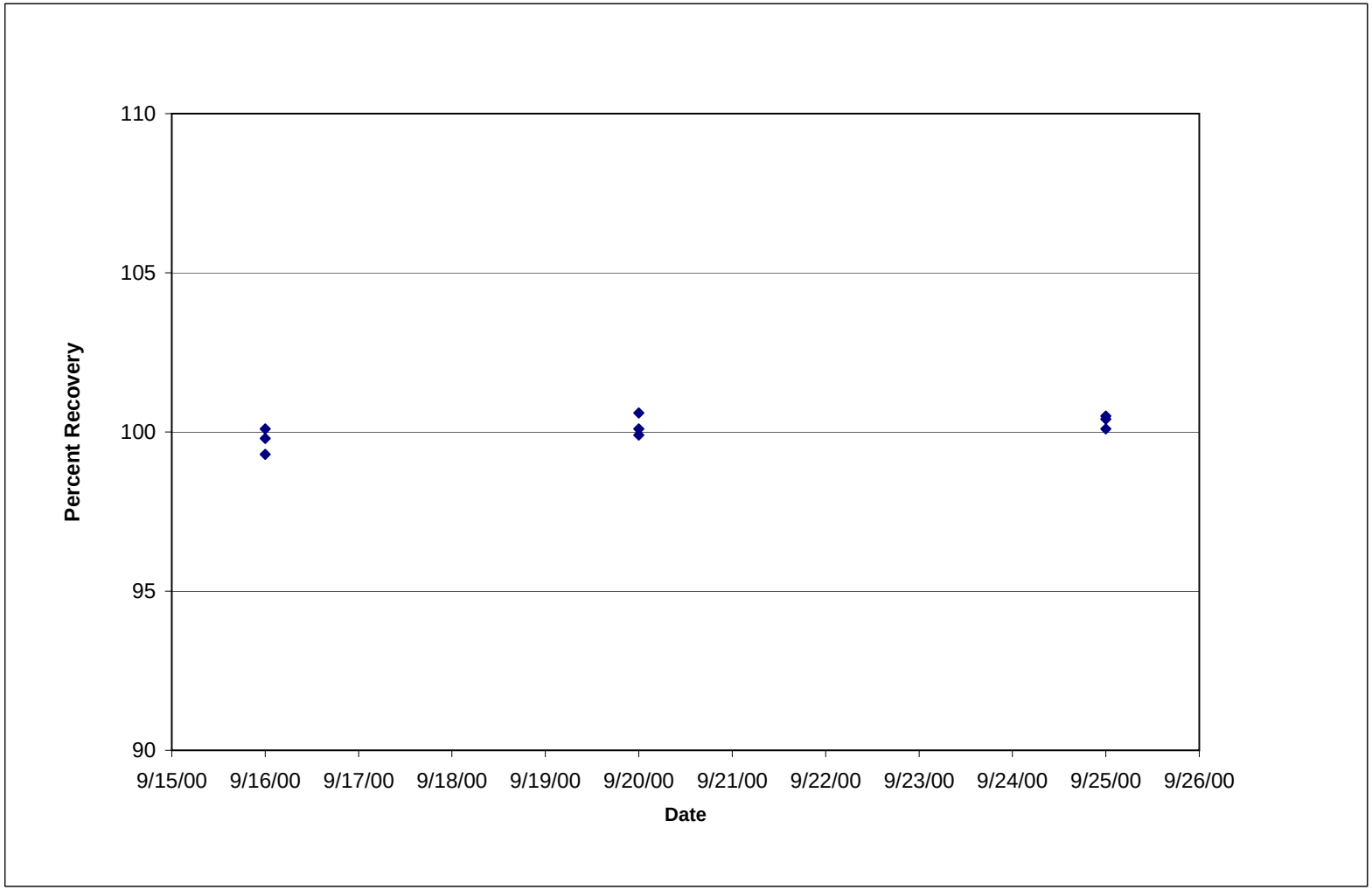
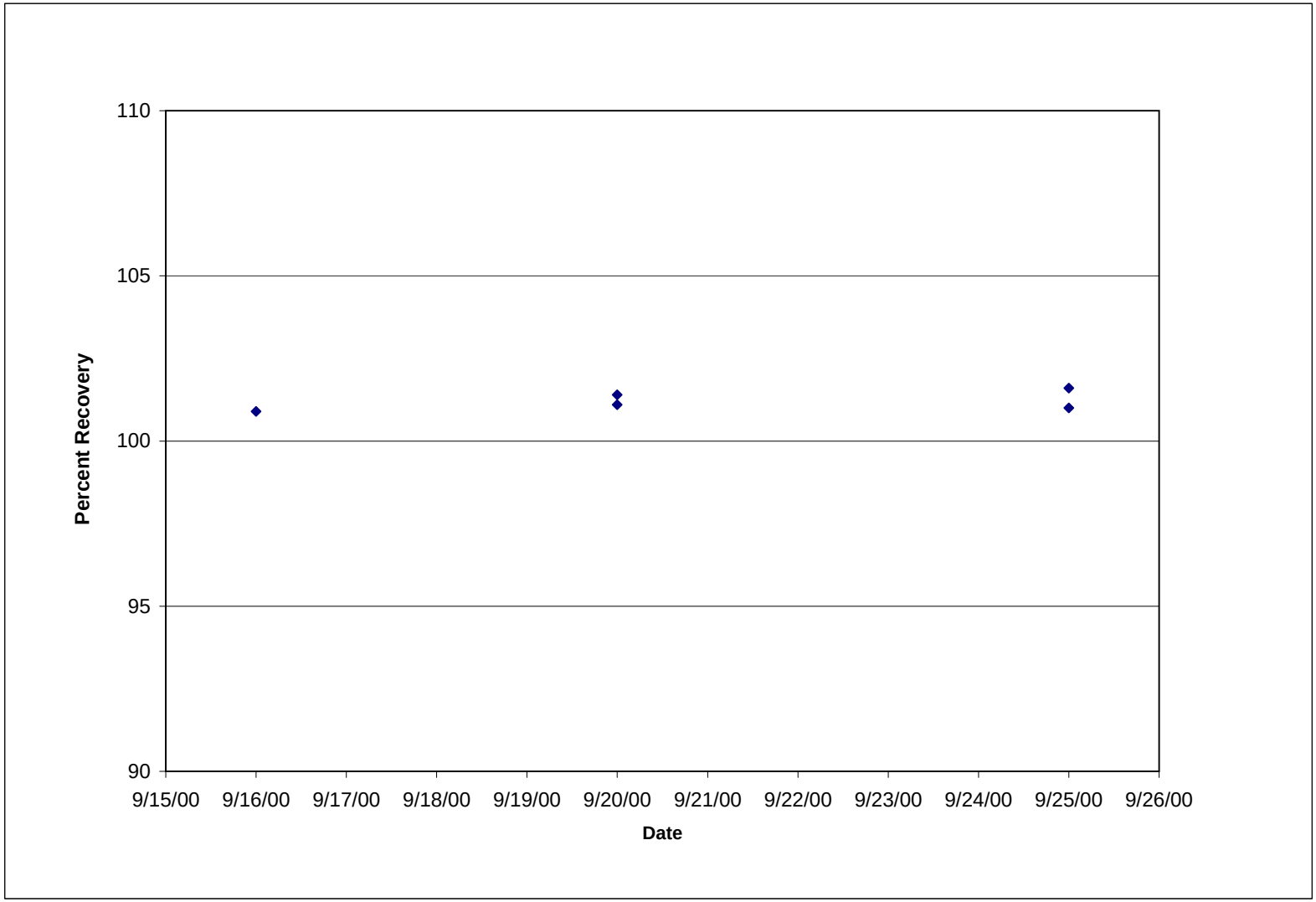
09/07/00
09/08/00
09/11/00
09/13/00
09/15/00
09/20/00
09/24/00
09/27/00
10/23/00
Date
Difference mg
Monthly Average

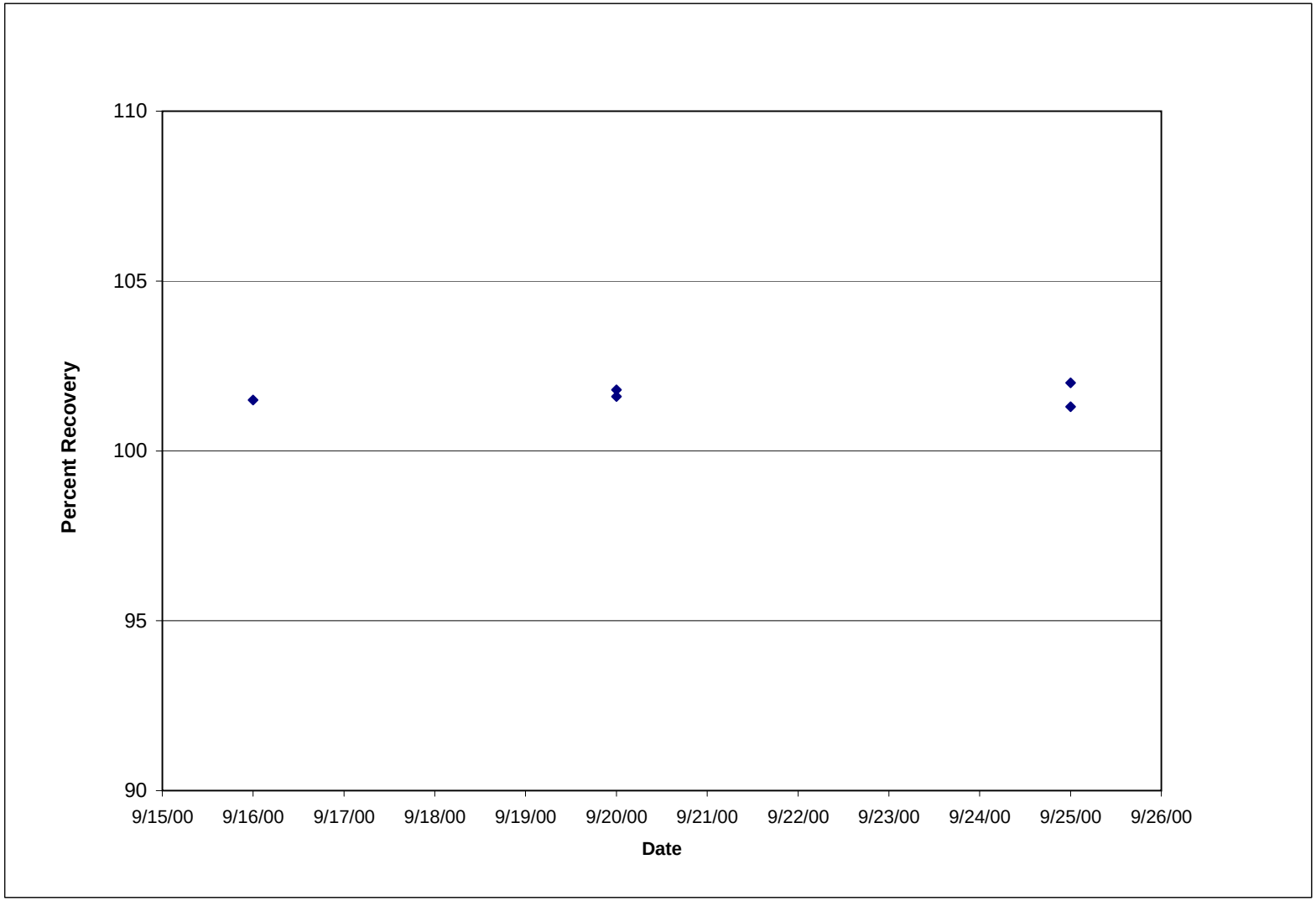

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

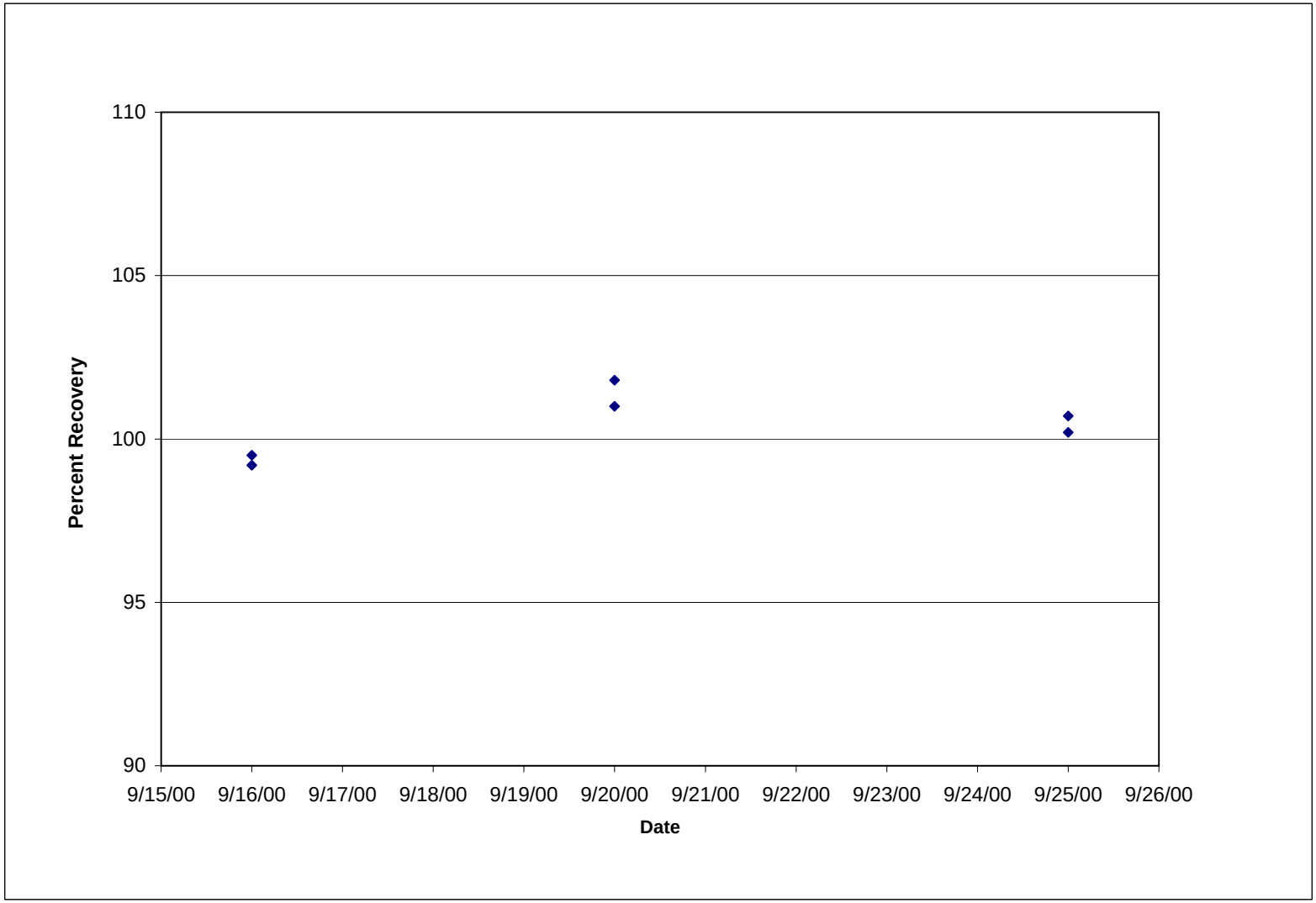

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

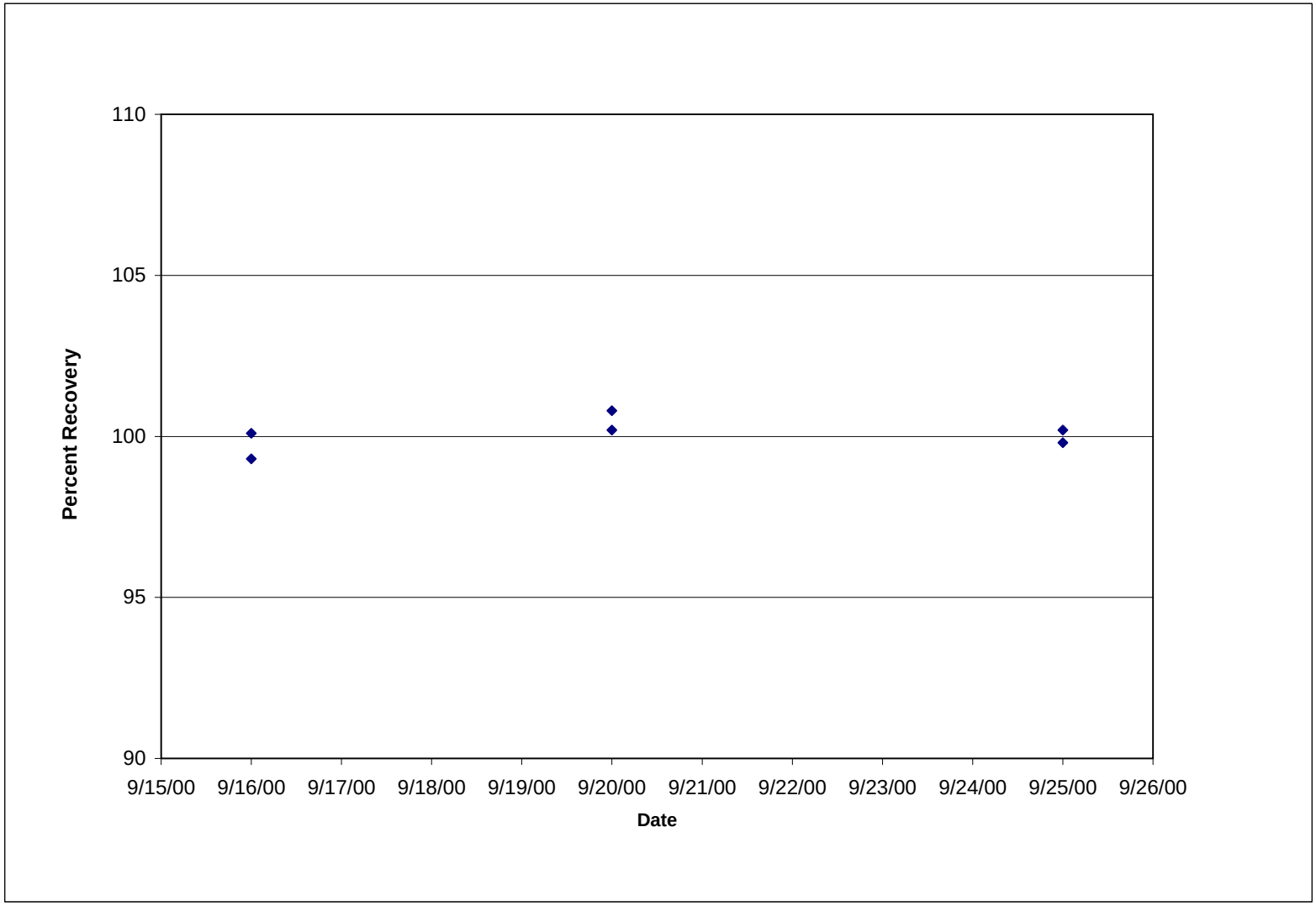

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

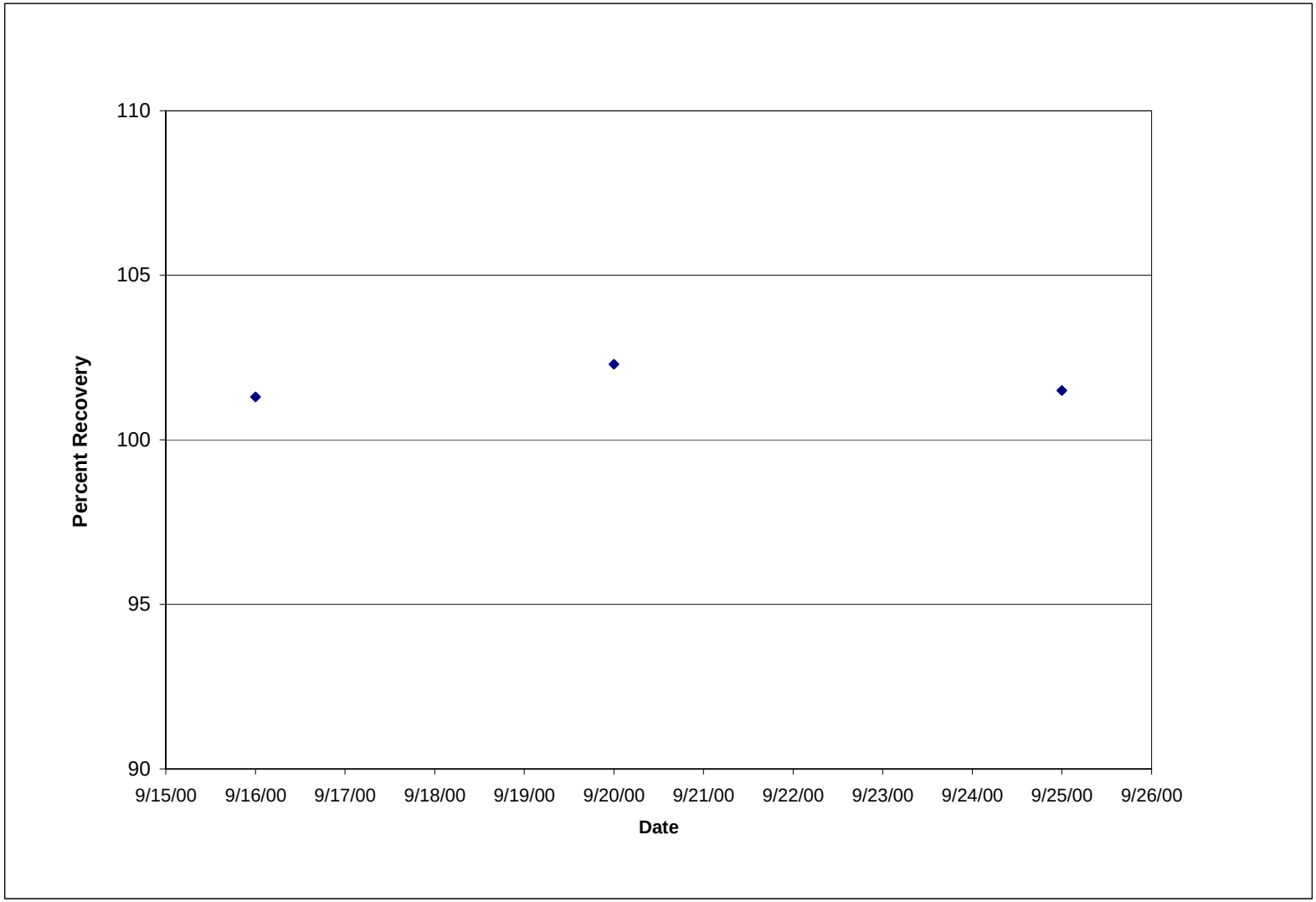

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

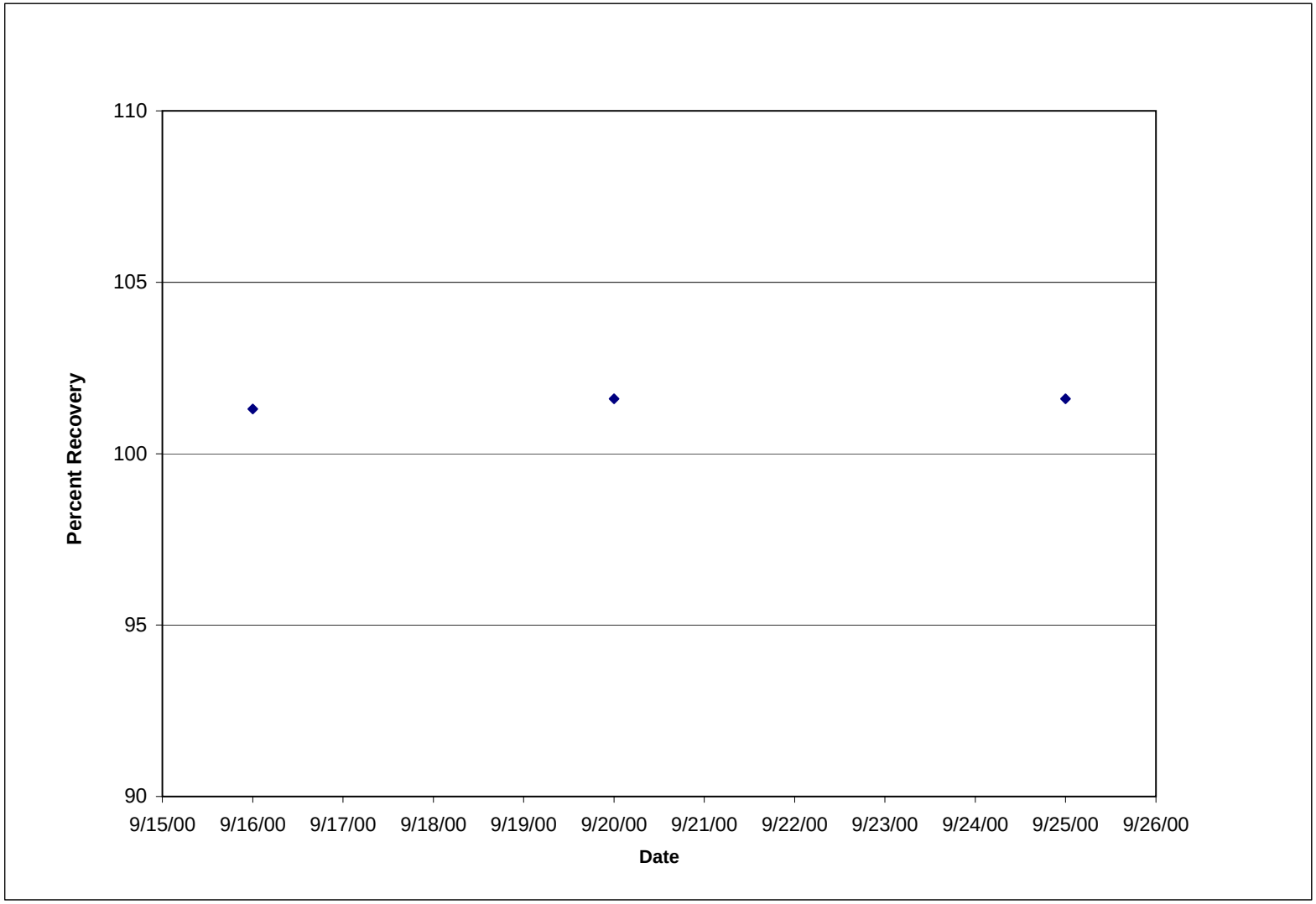

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

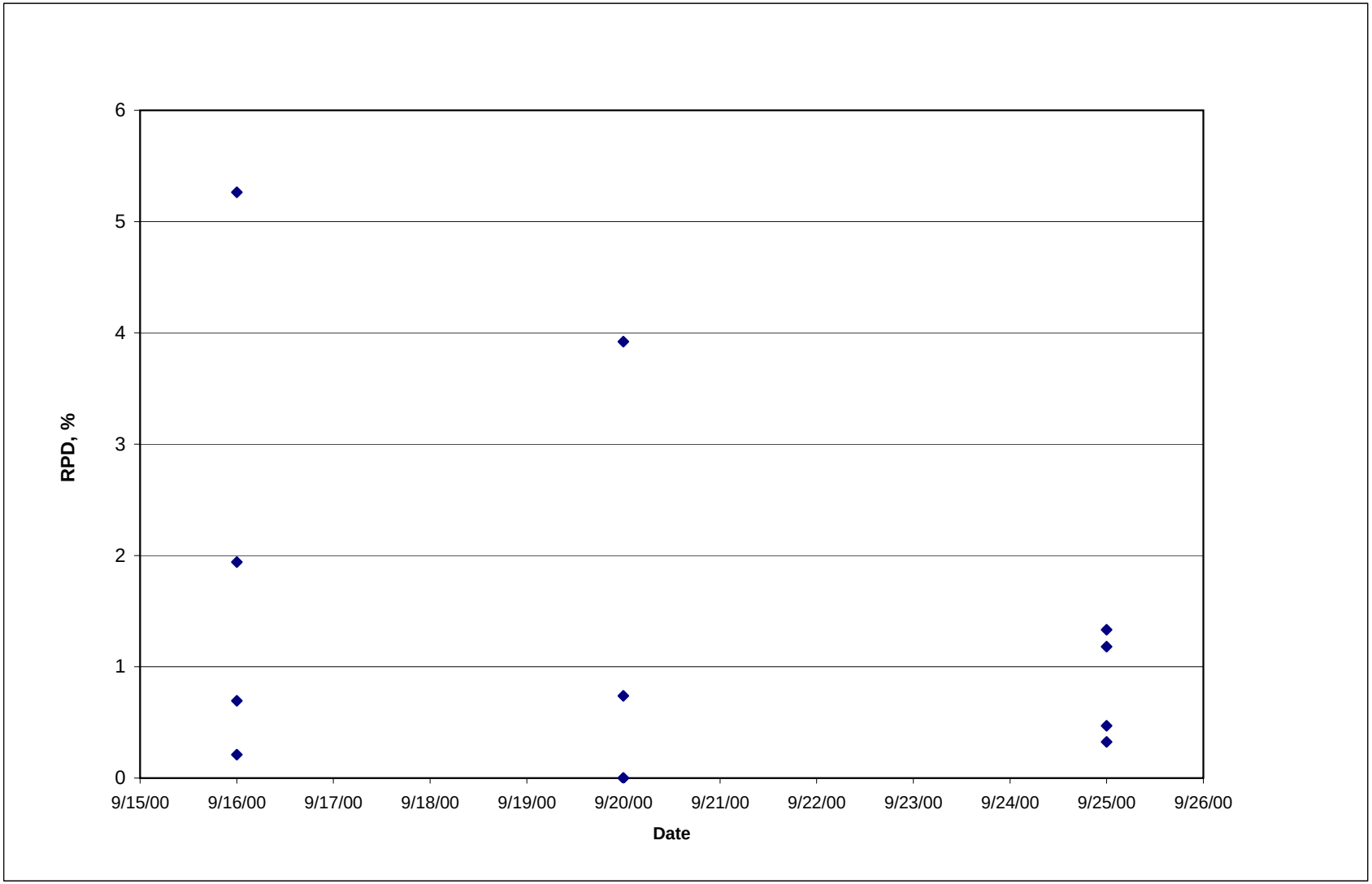

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

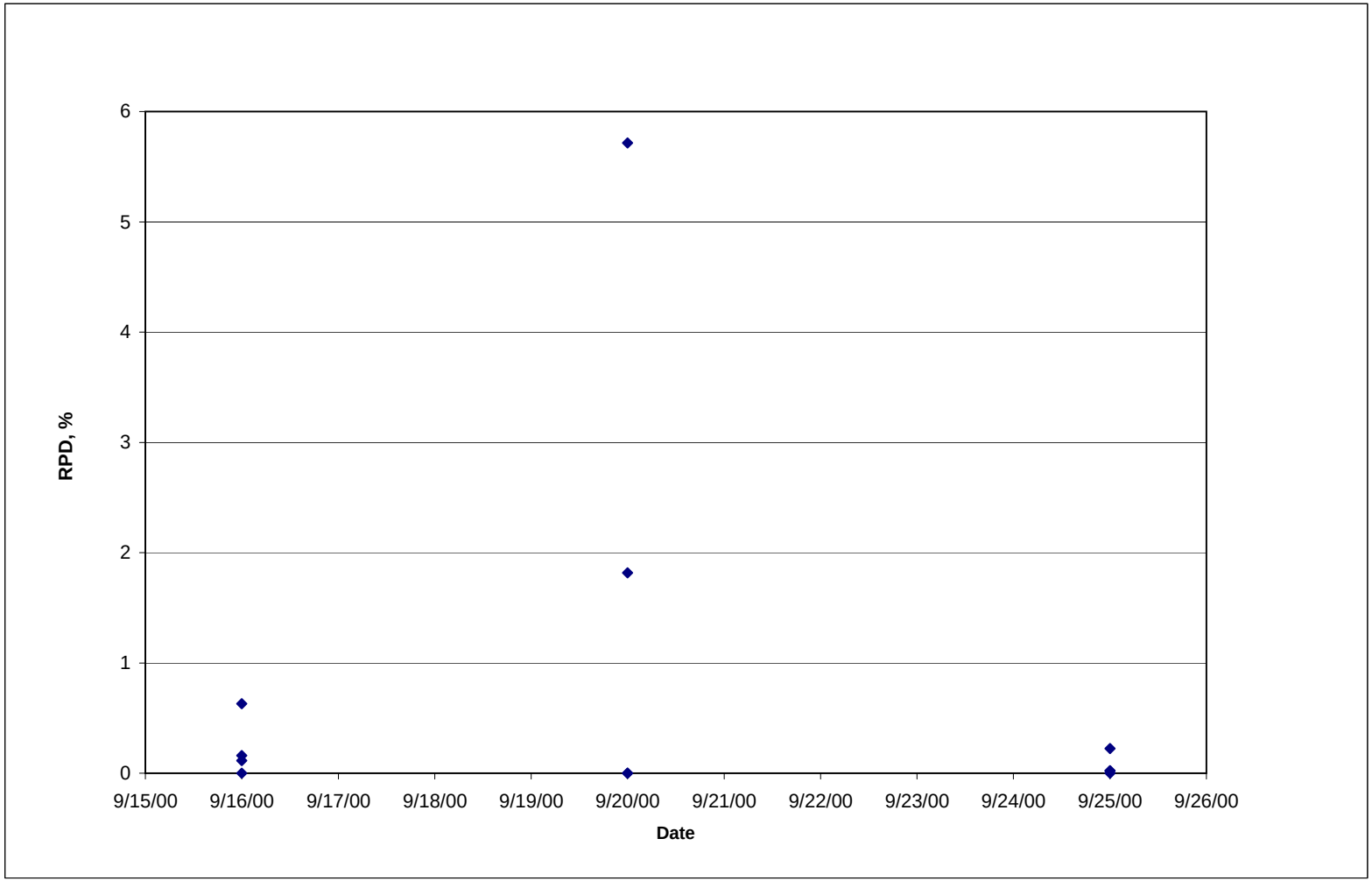

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

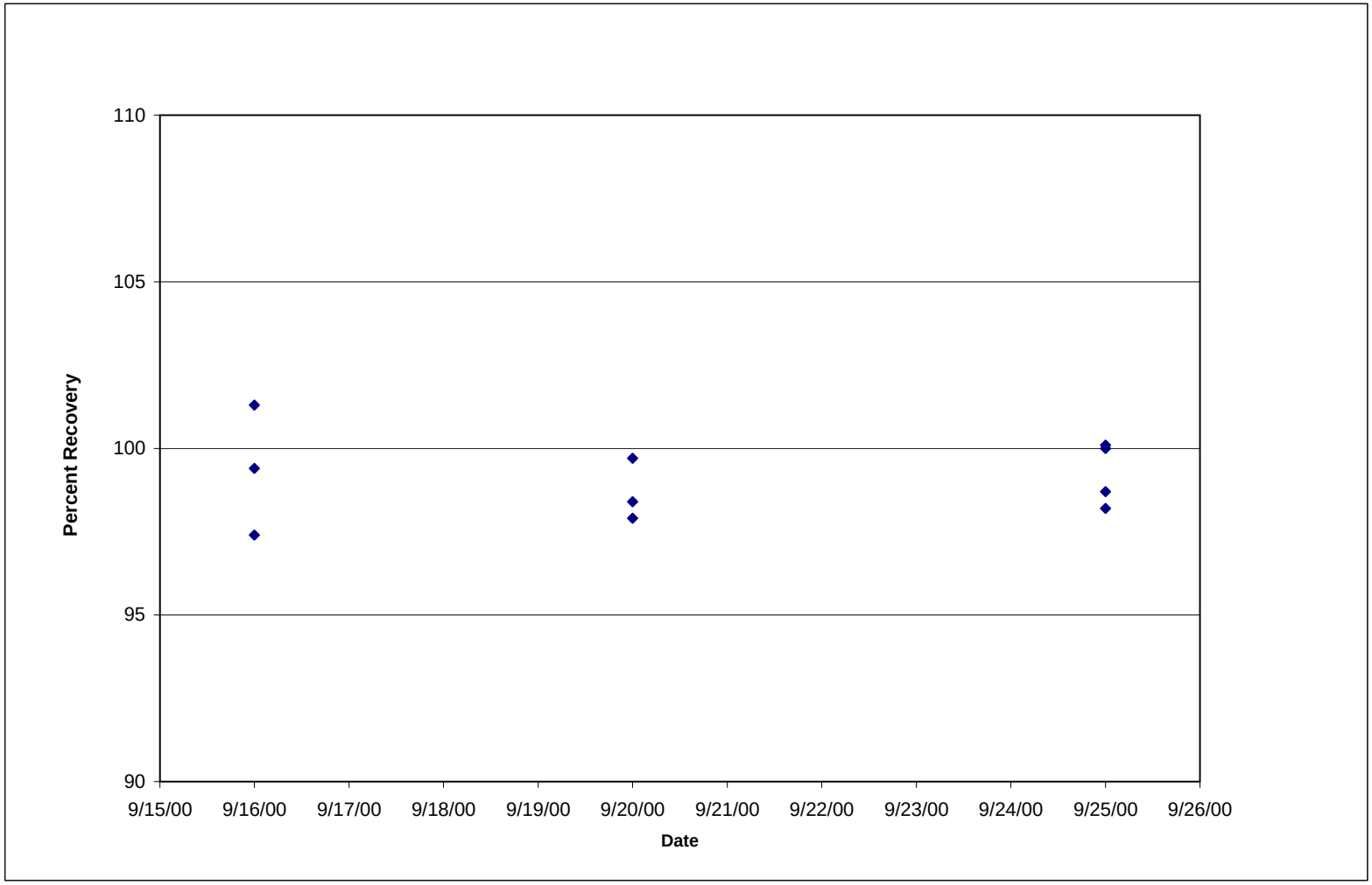

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

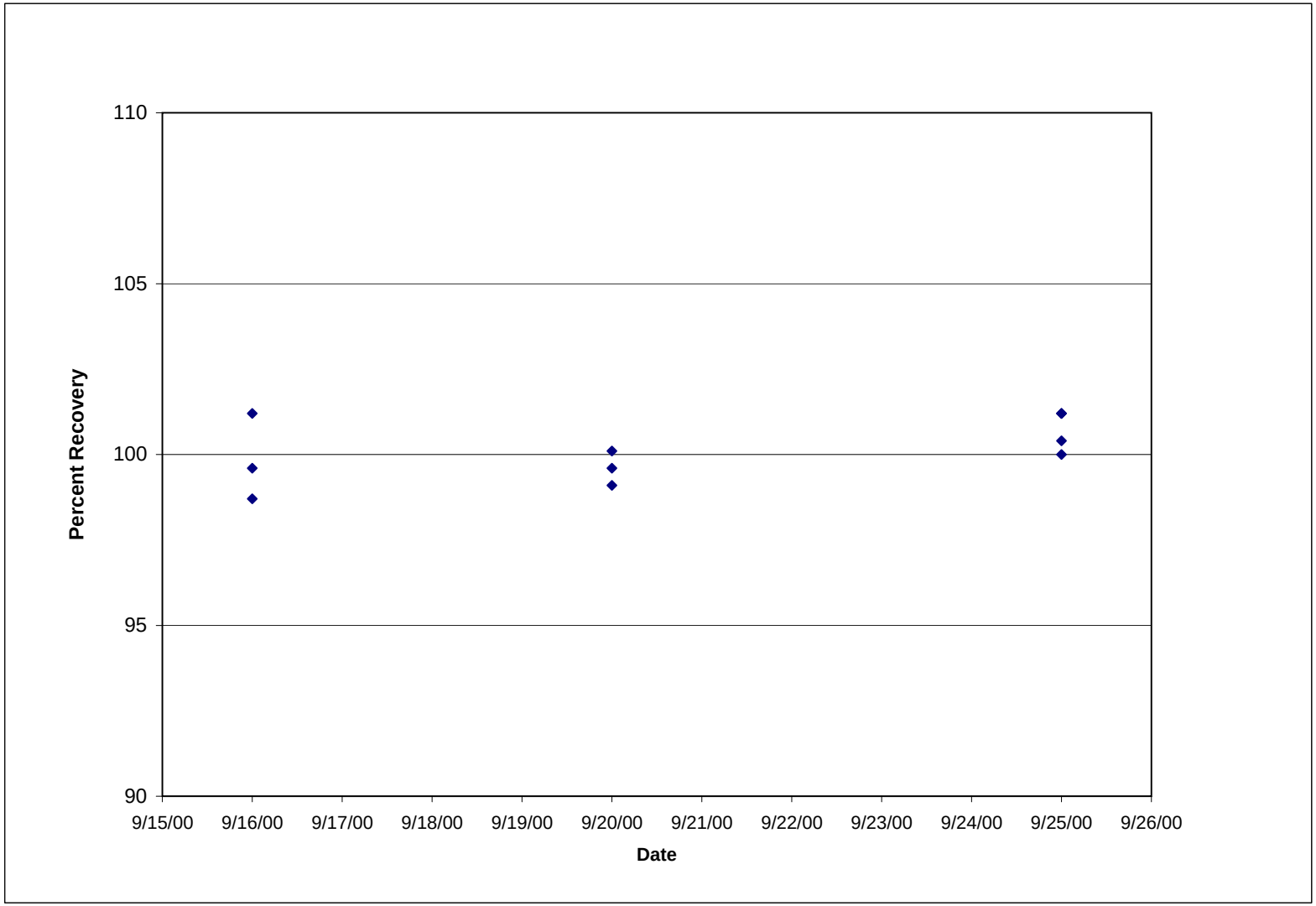

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

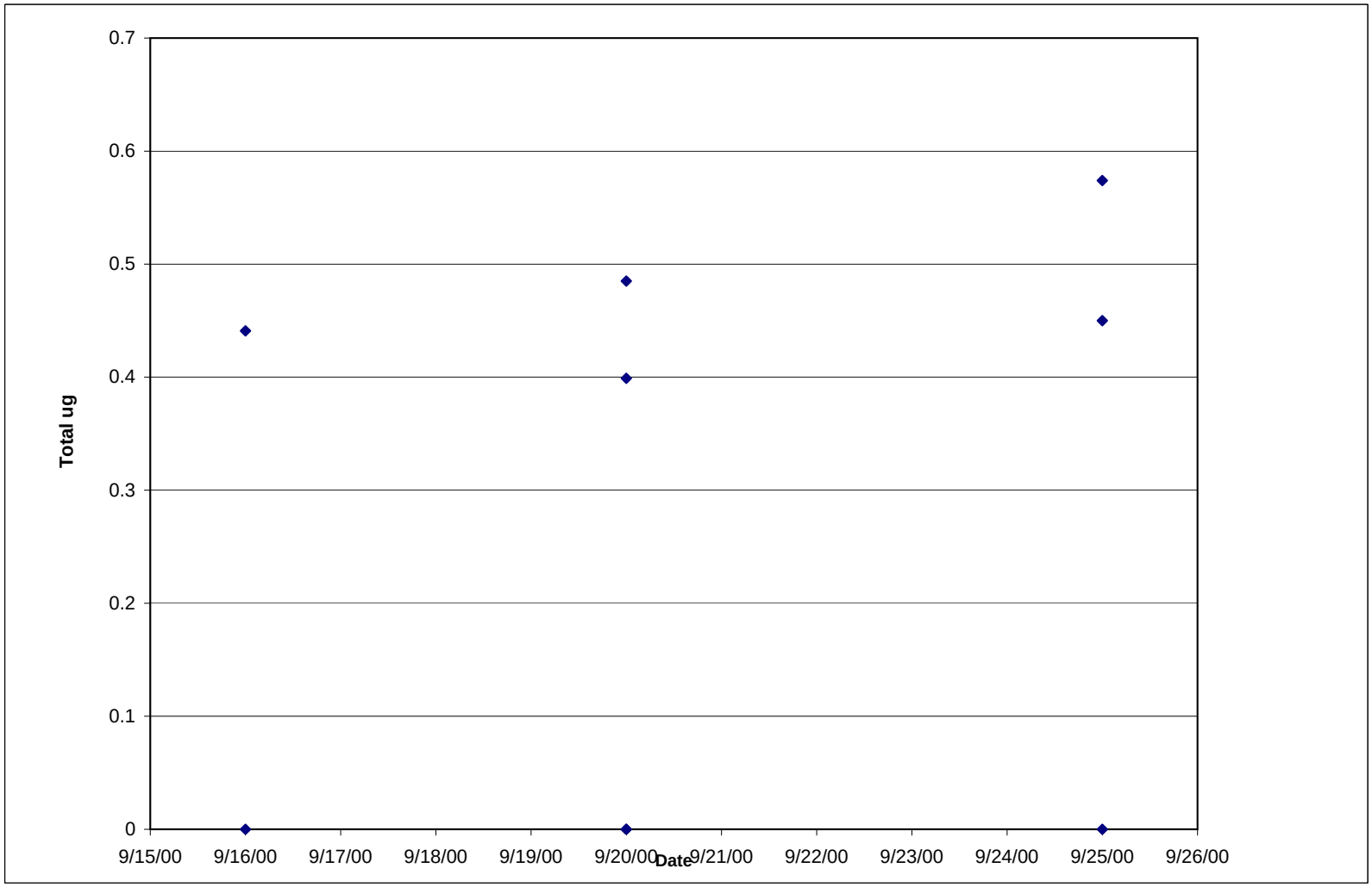

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

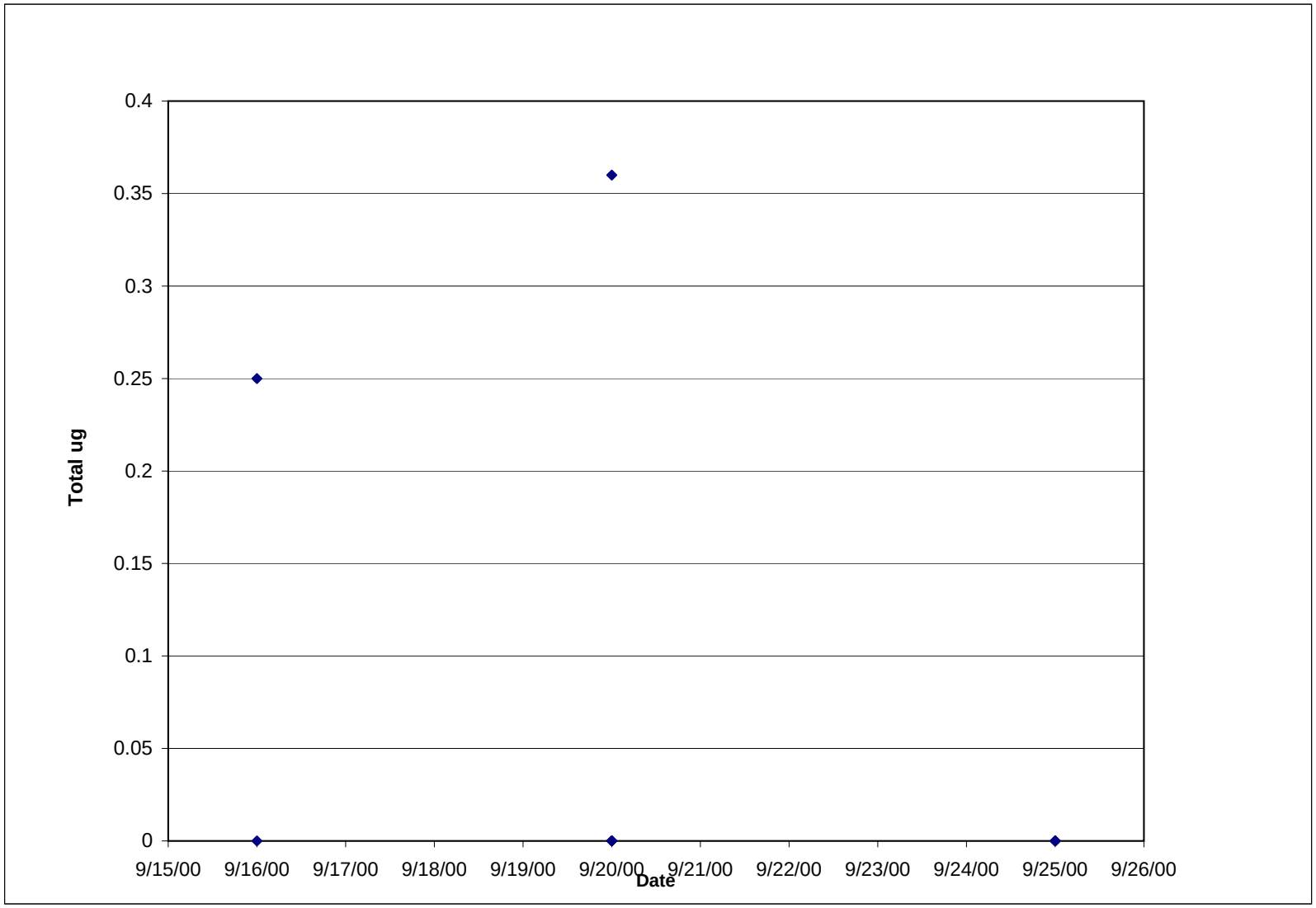

6
5
4
3
2
1
0
RPD, %
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

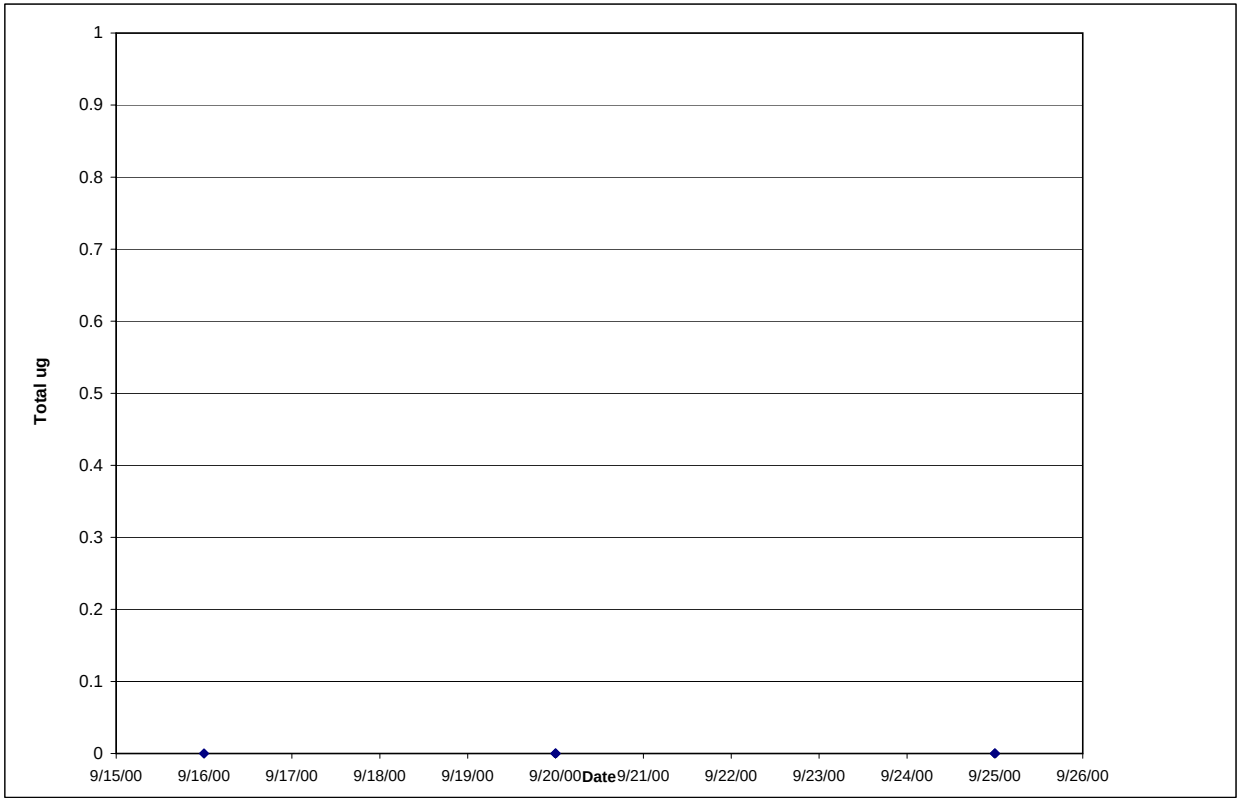

6
5
4
3
2
1
0
RPD, %
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

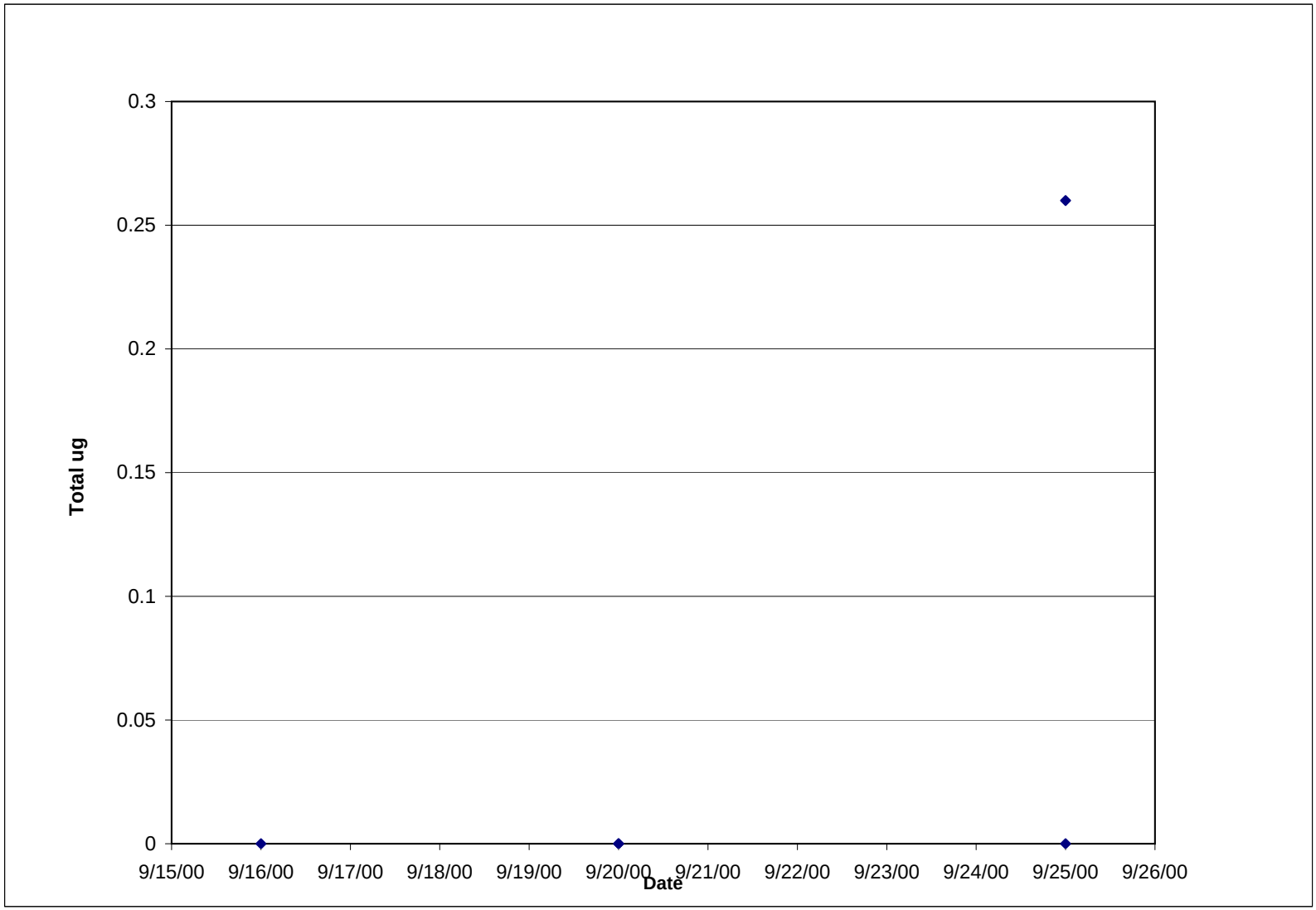

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

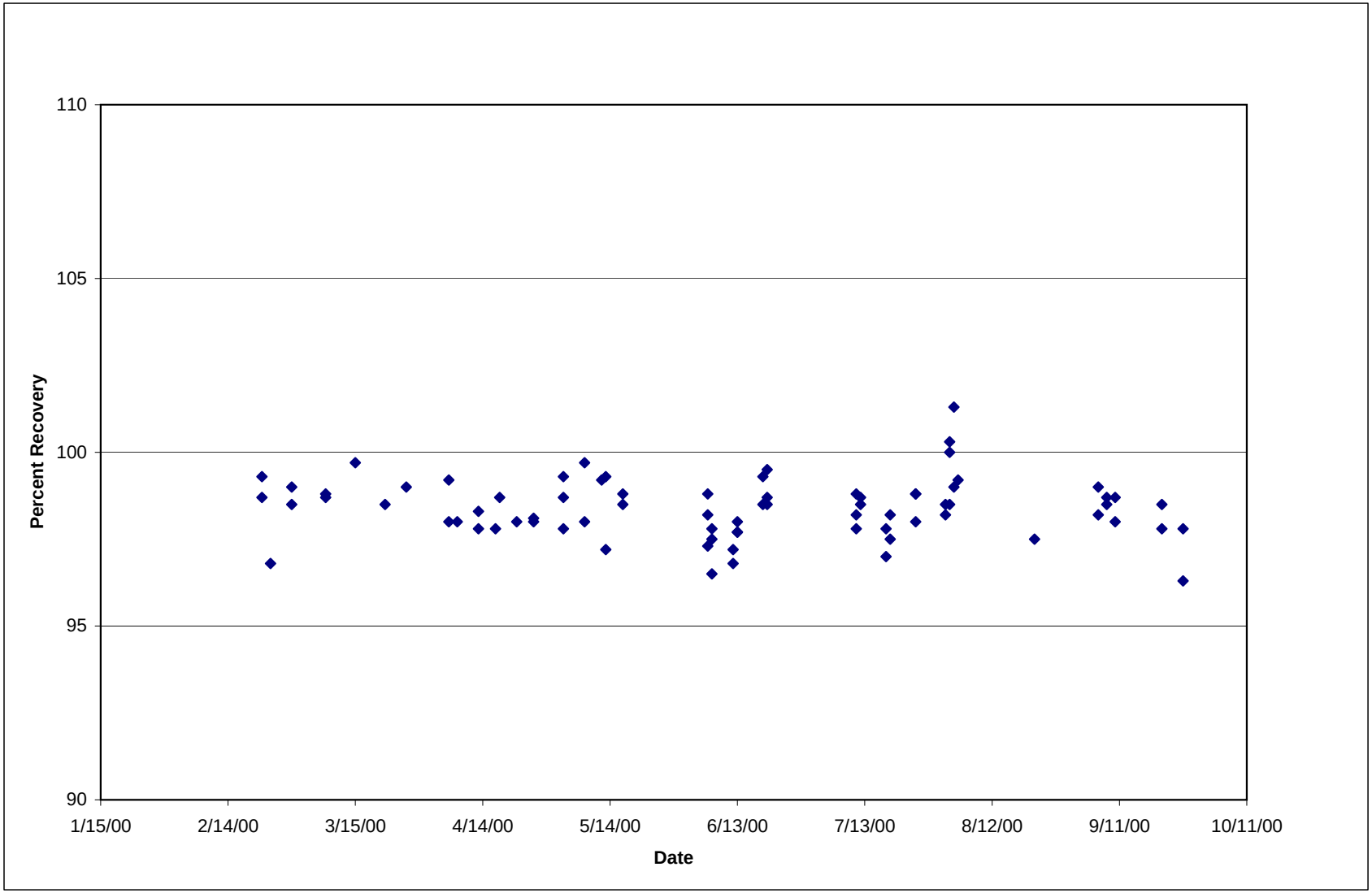

110
105
100
95
90
Percent Recovery
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

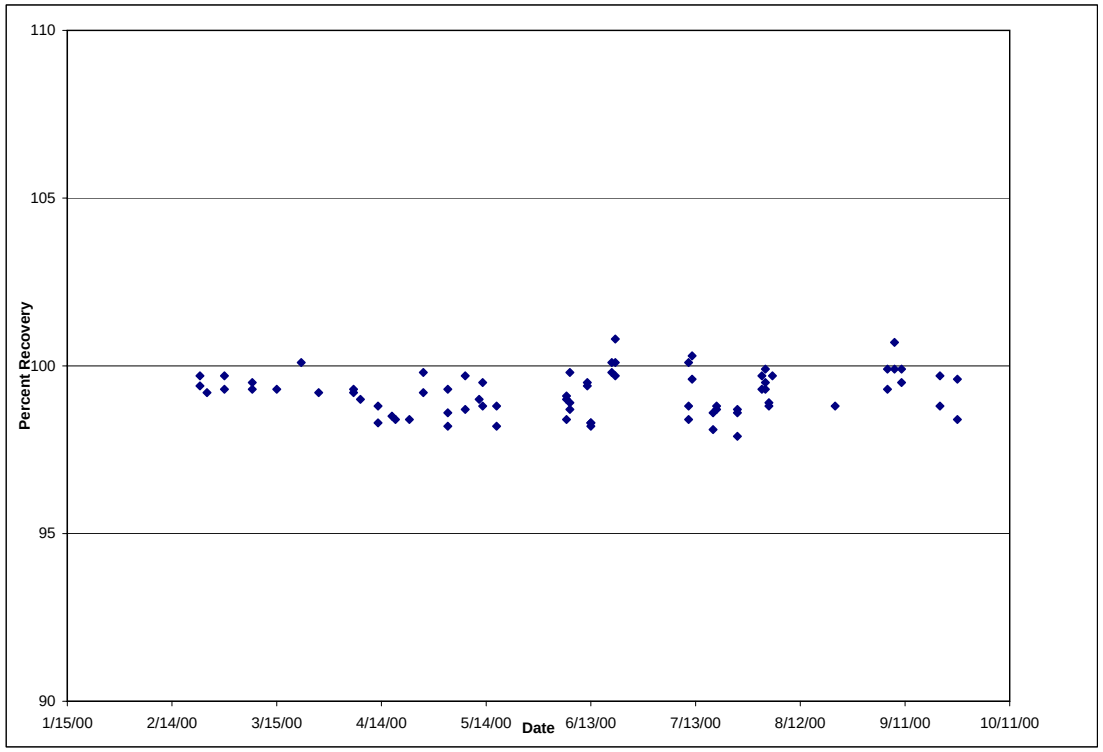

0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
Total ug
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

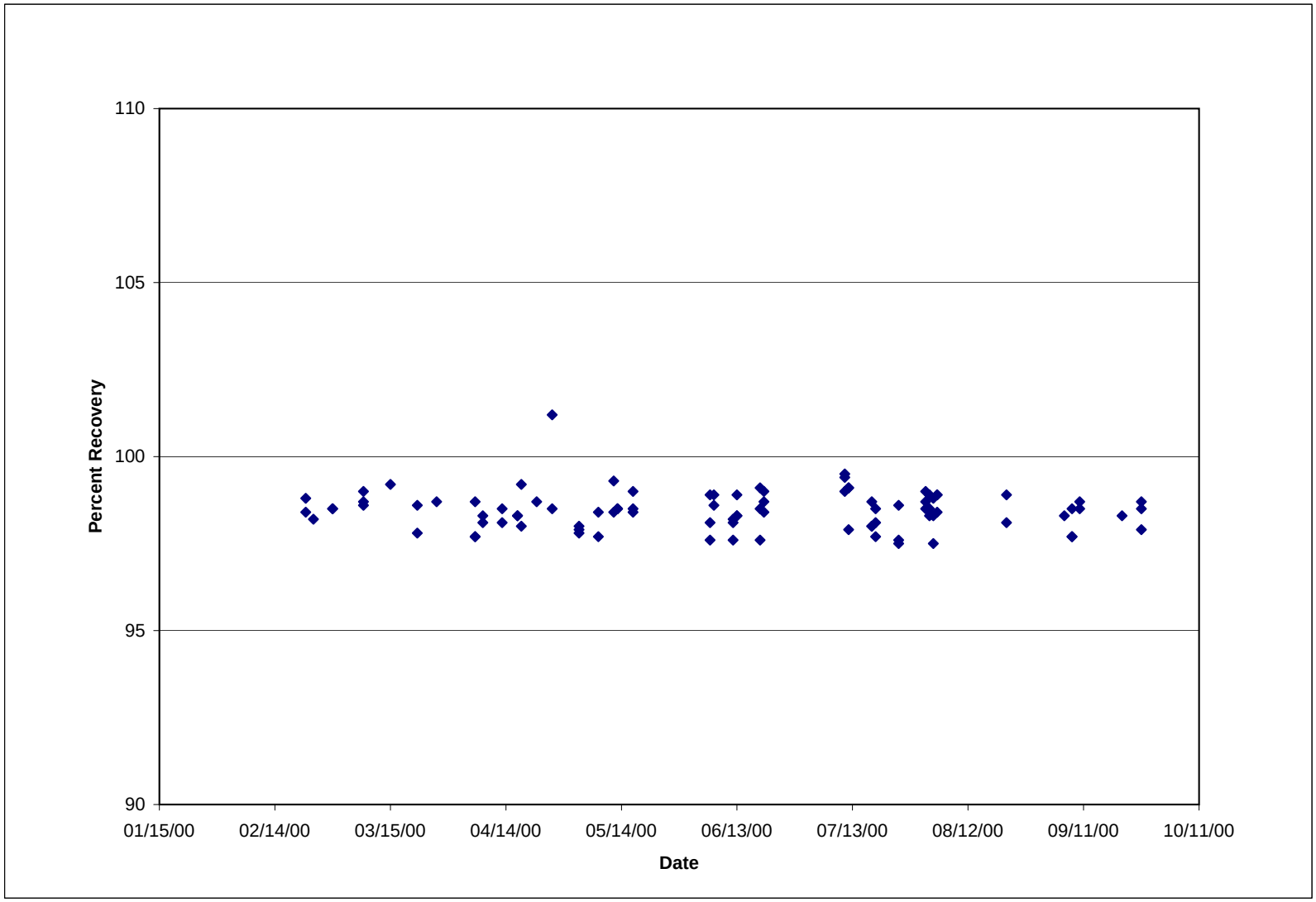

0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
Total ug
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

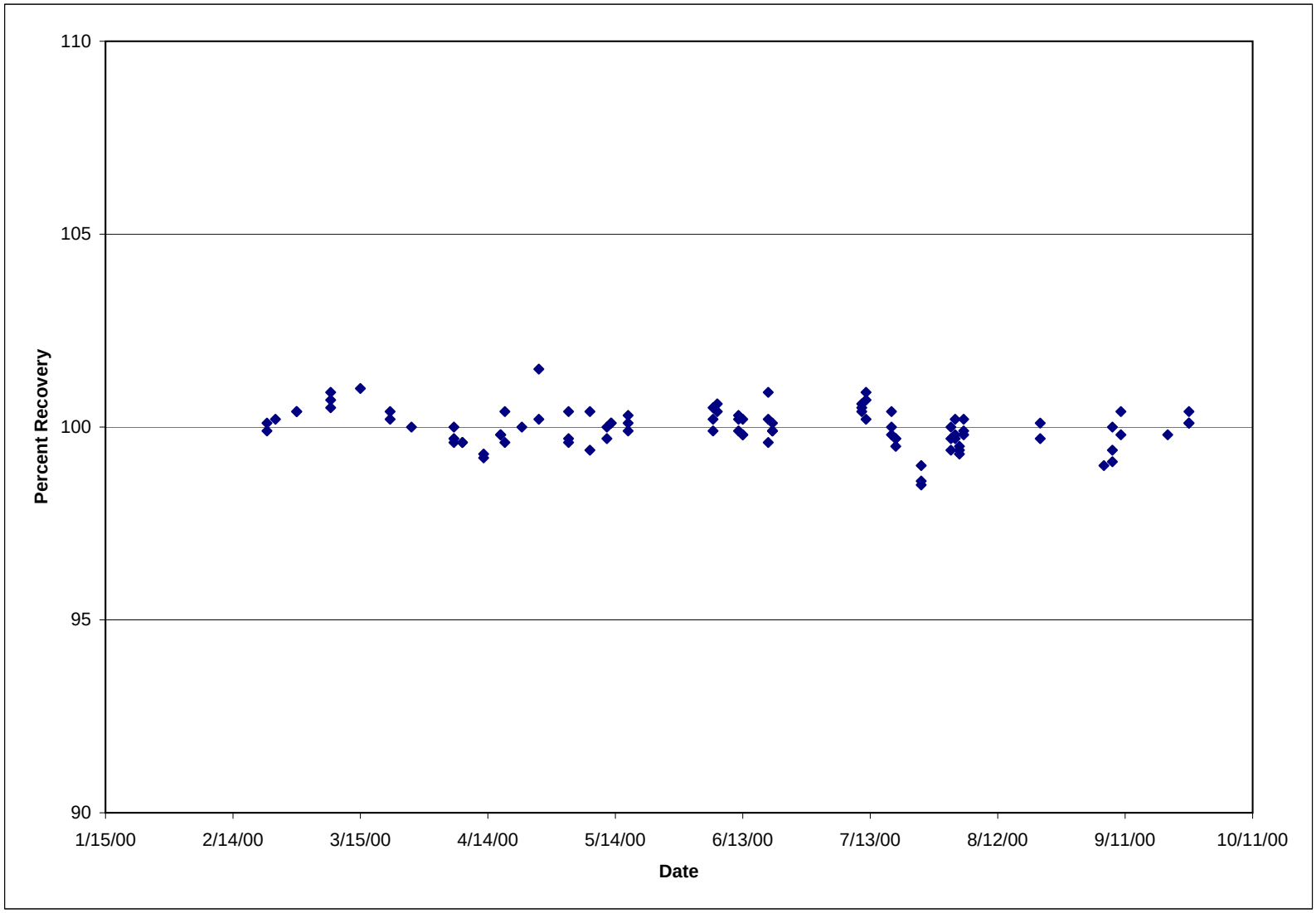

1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
Total ug
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
Date
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00

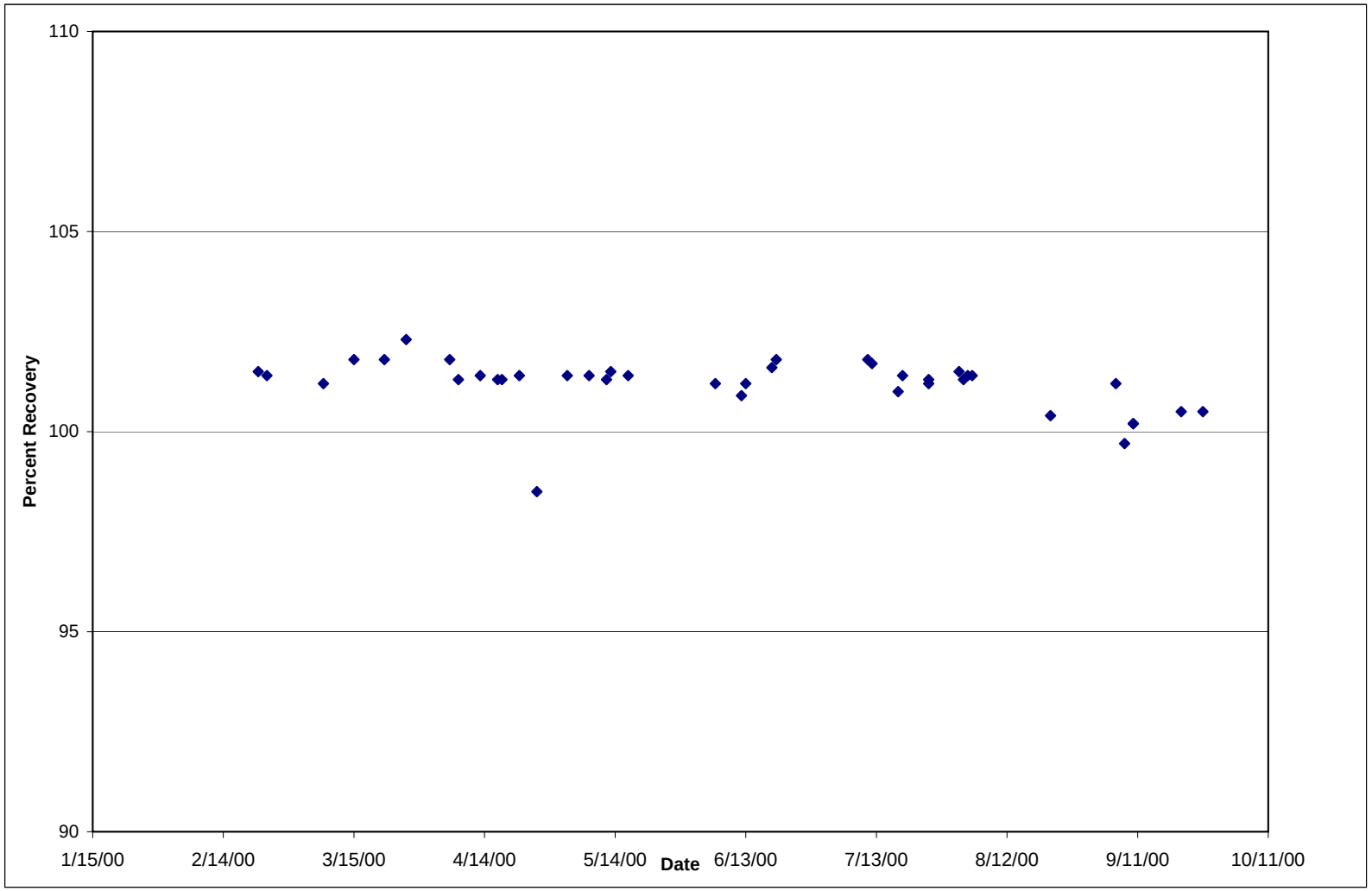

0.3
0.25
0.2
0.15
0.1
0.05
0
Total ug
9/15/00
9/16/00
9/17/00
9/18/00
9/19/00
9/20/00
9/21/00
9/22/00
9/23/00
9/24/00
9/25/00
9/26/00
Date

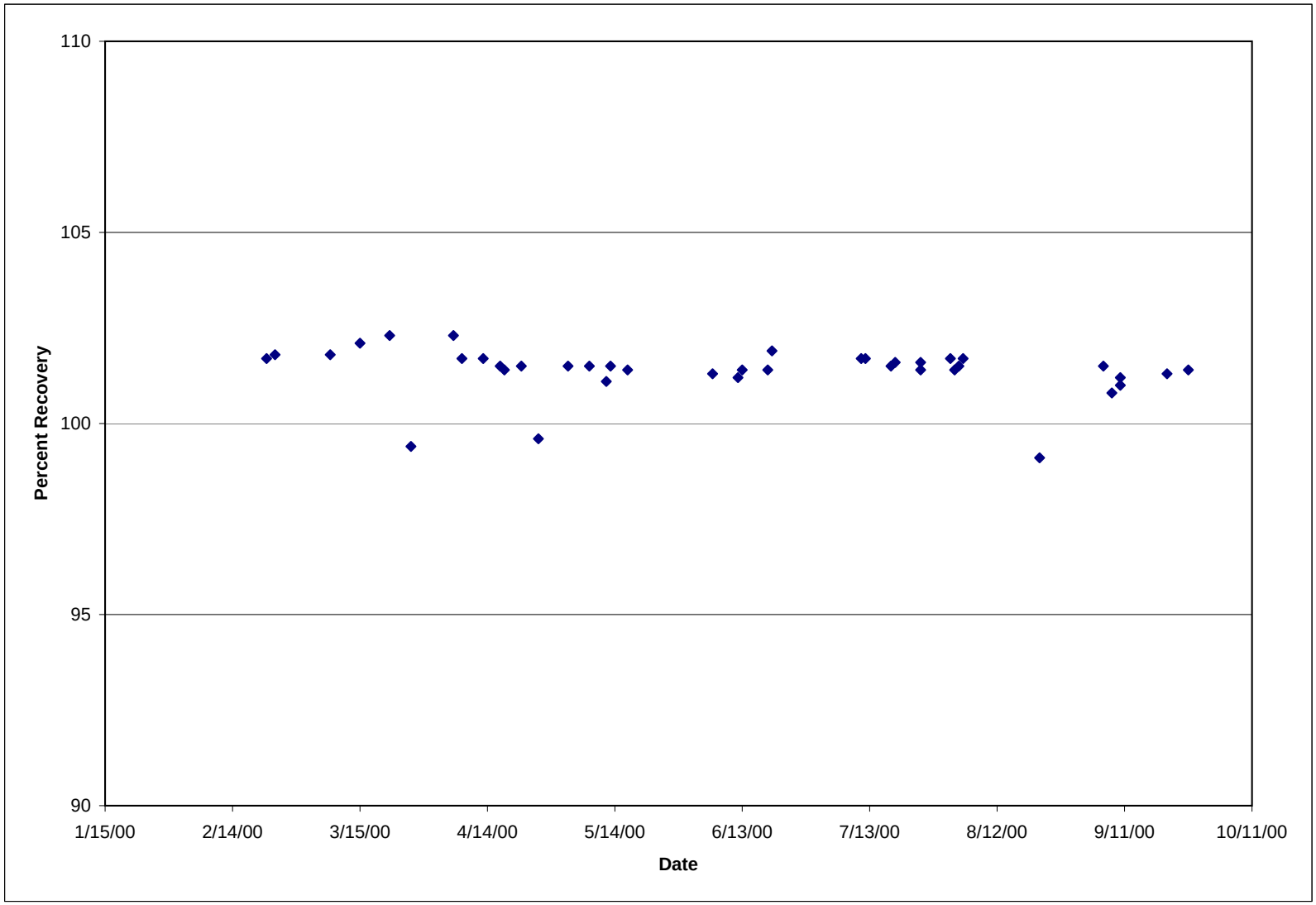

110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

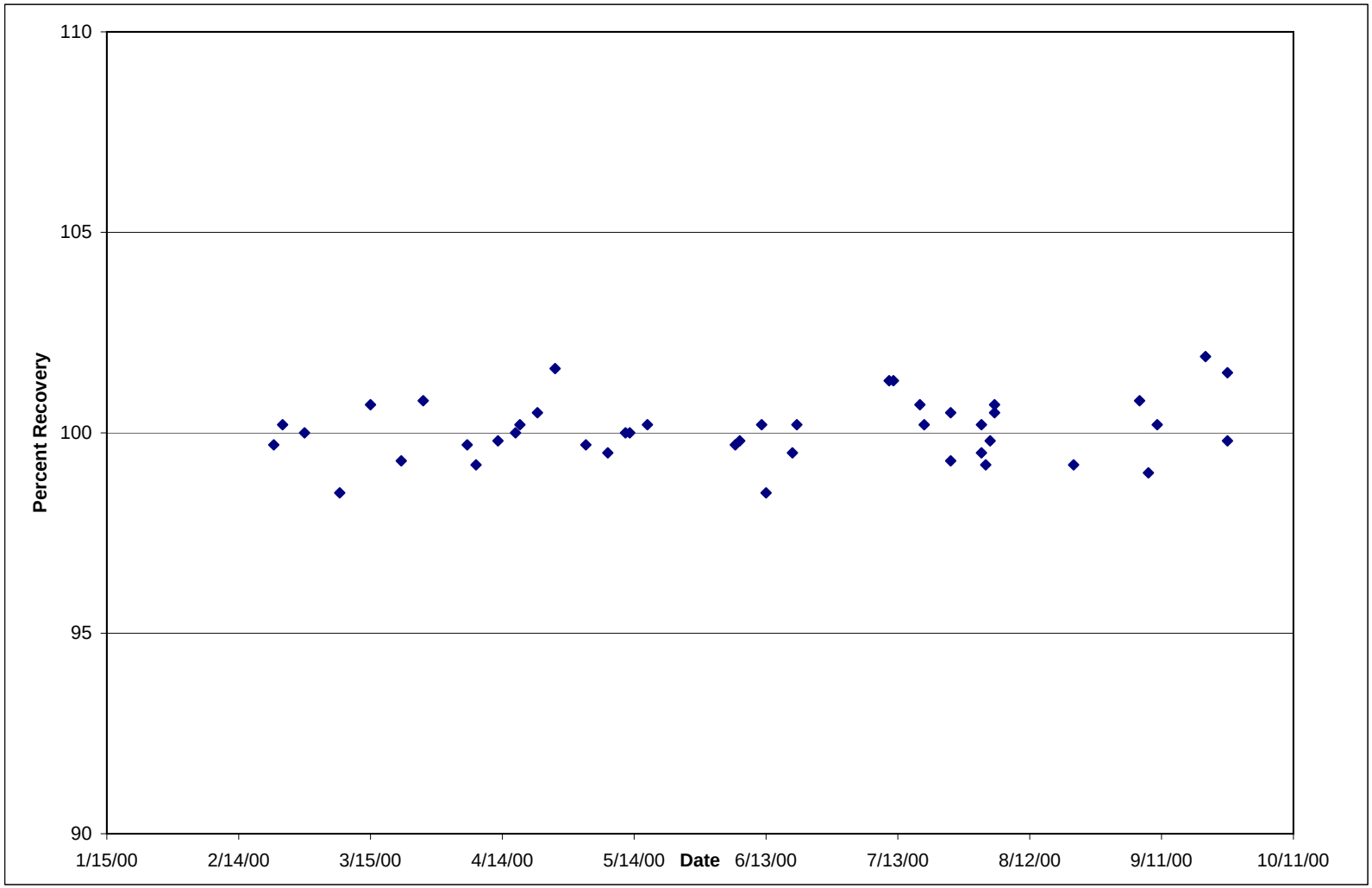

110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

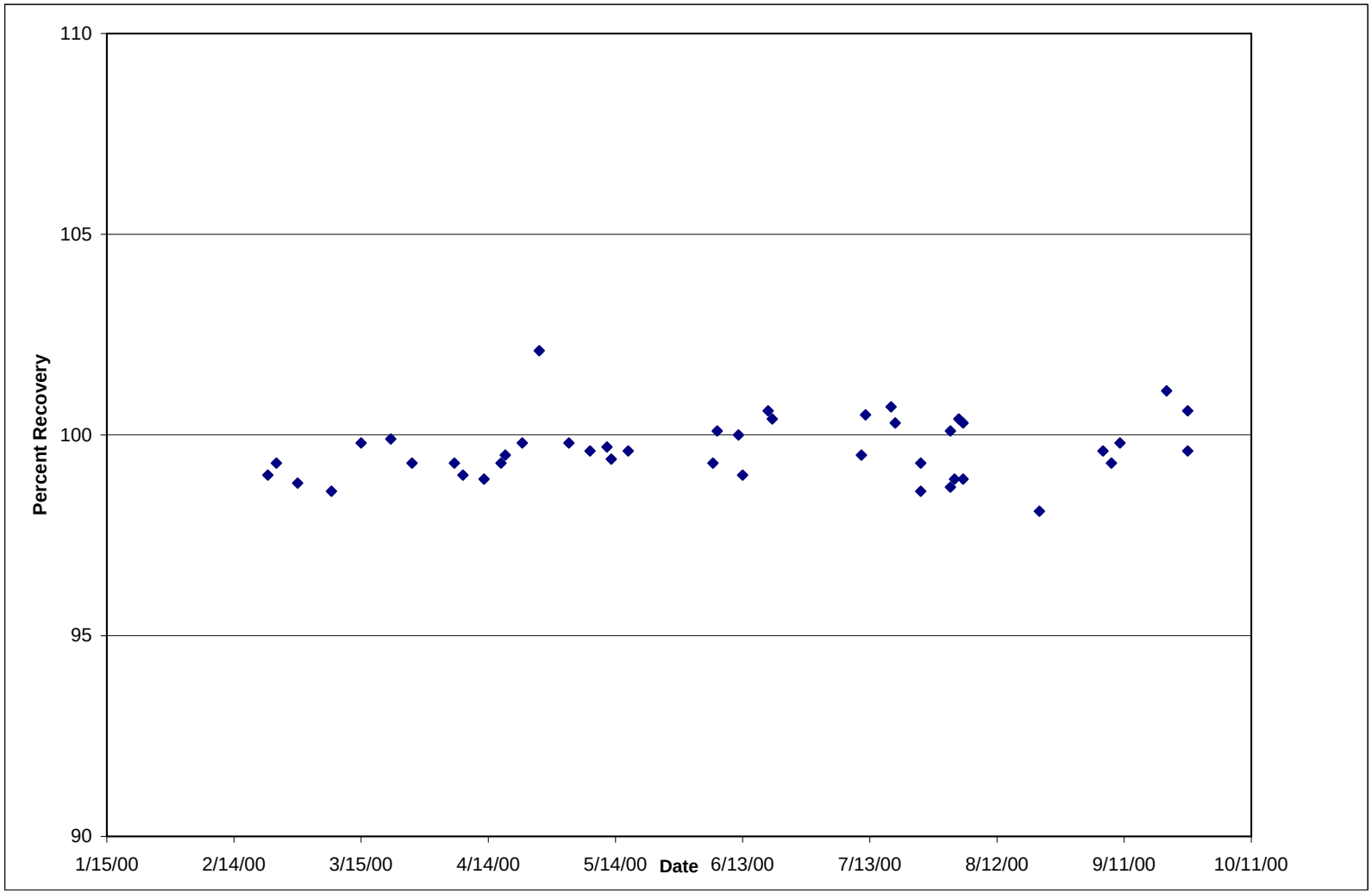

110
105
100
95
90
Percent Recovery
01/15/00
02/14/00
03/15/00
04/14/00
05/14/00
06/13/00
07/13/00
08/12/00
09/11/00
10/11/00
Date

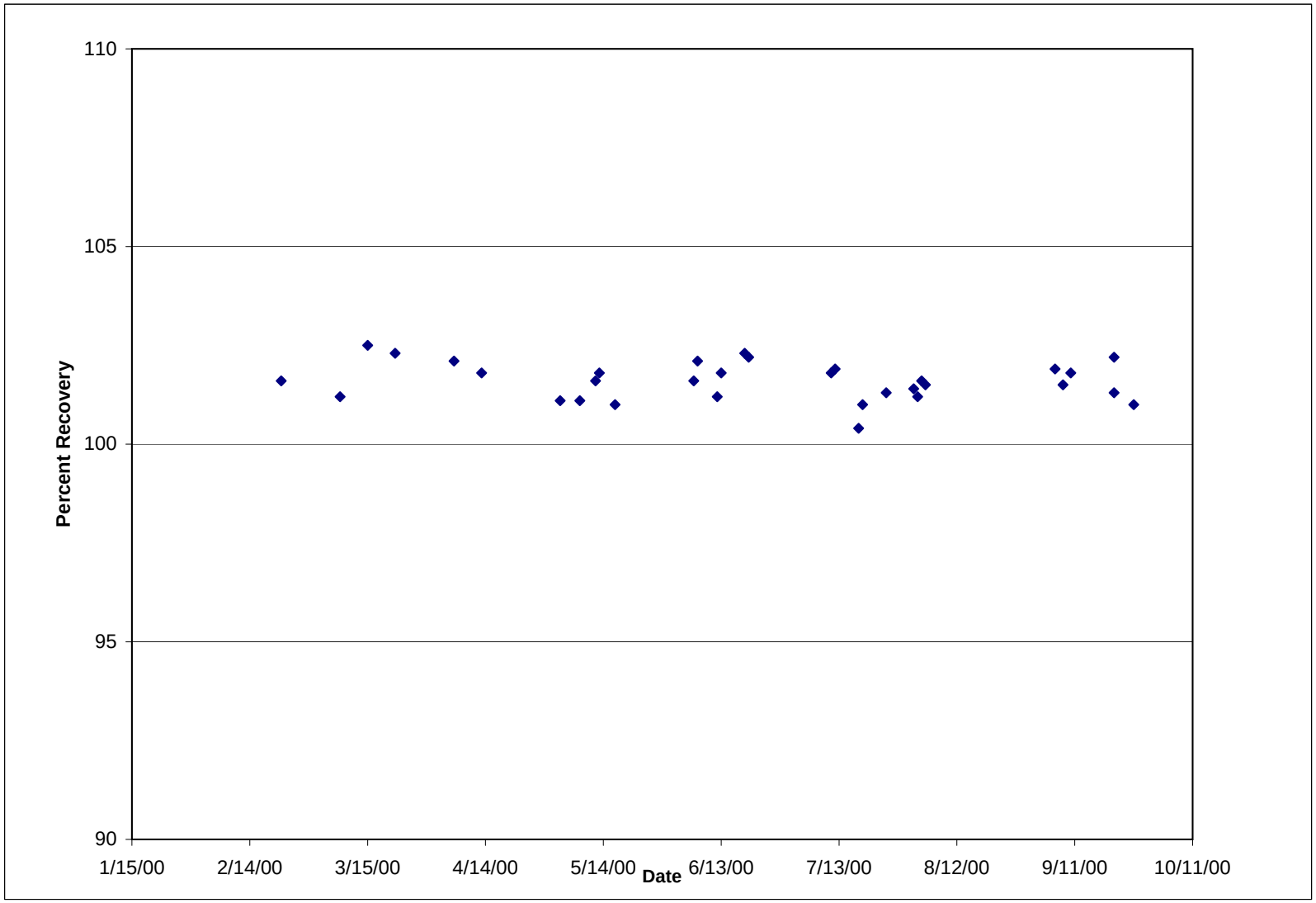

110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

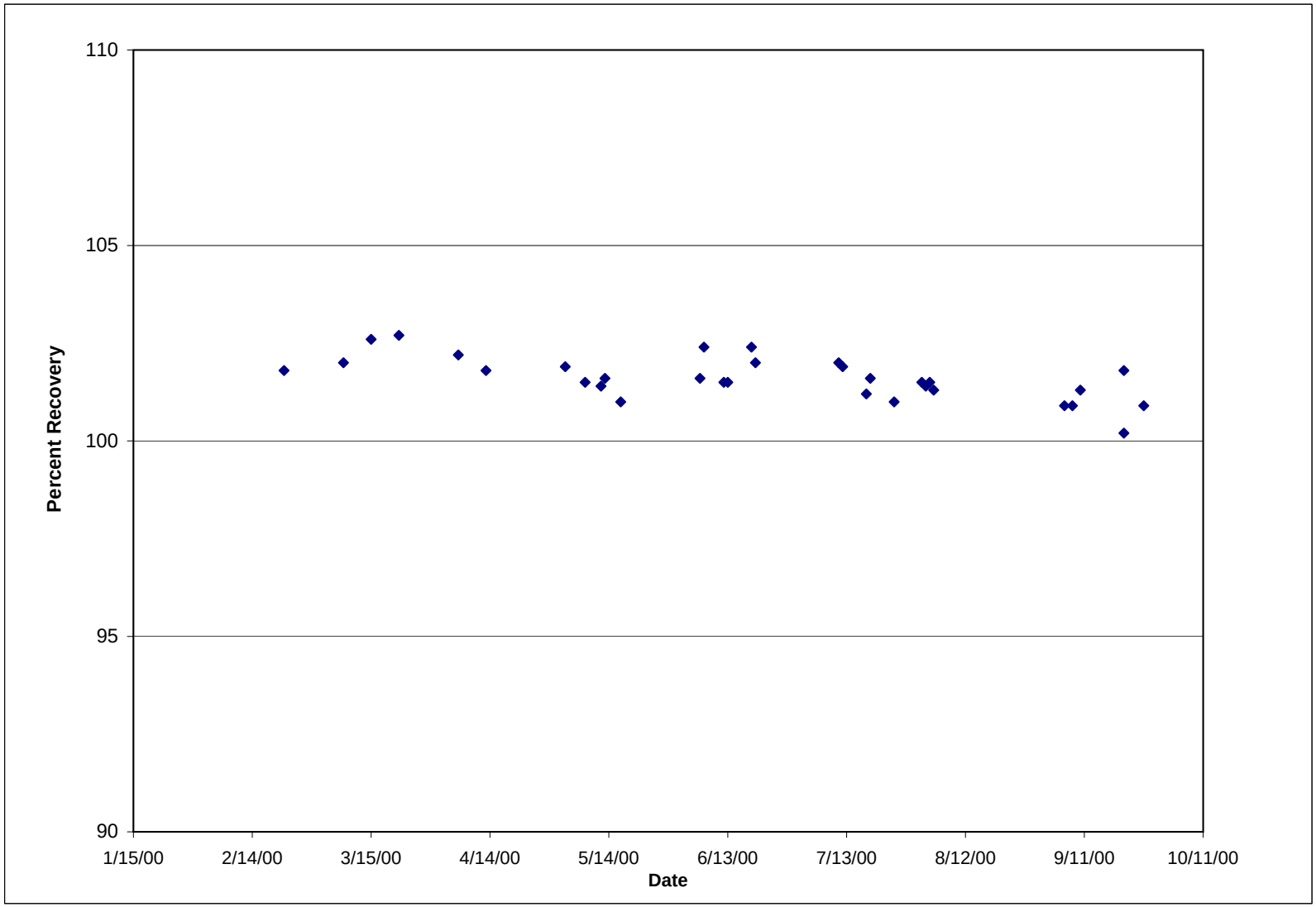

110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

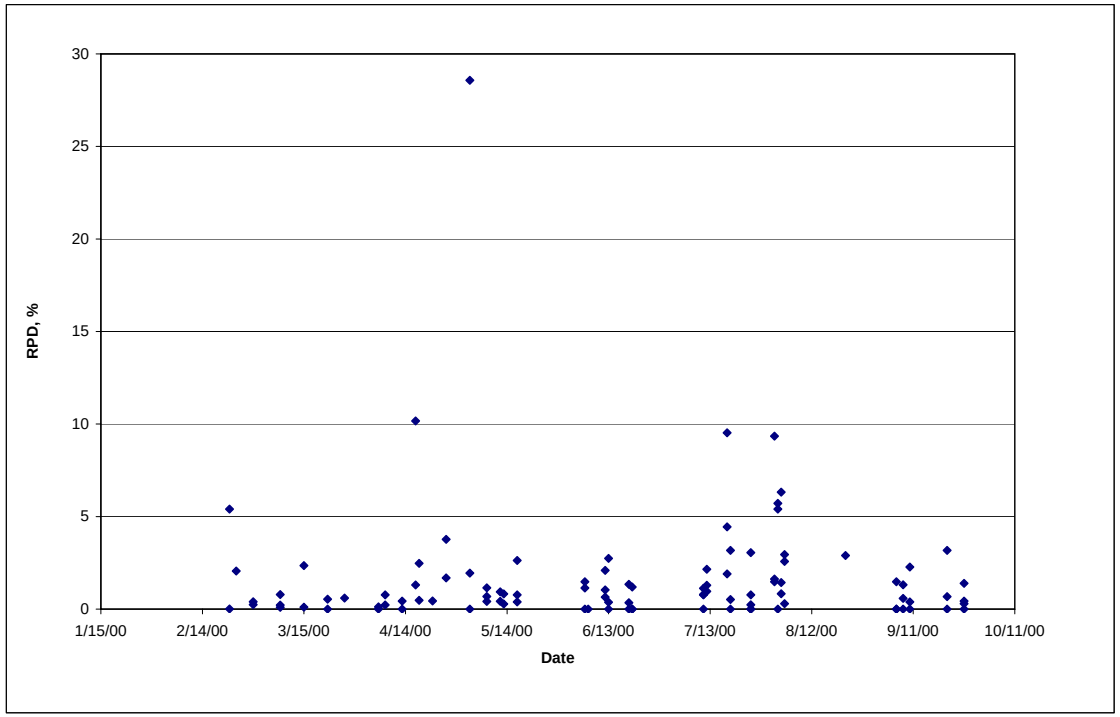

110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

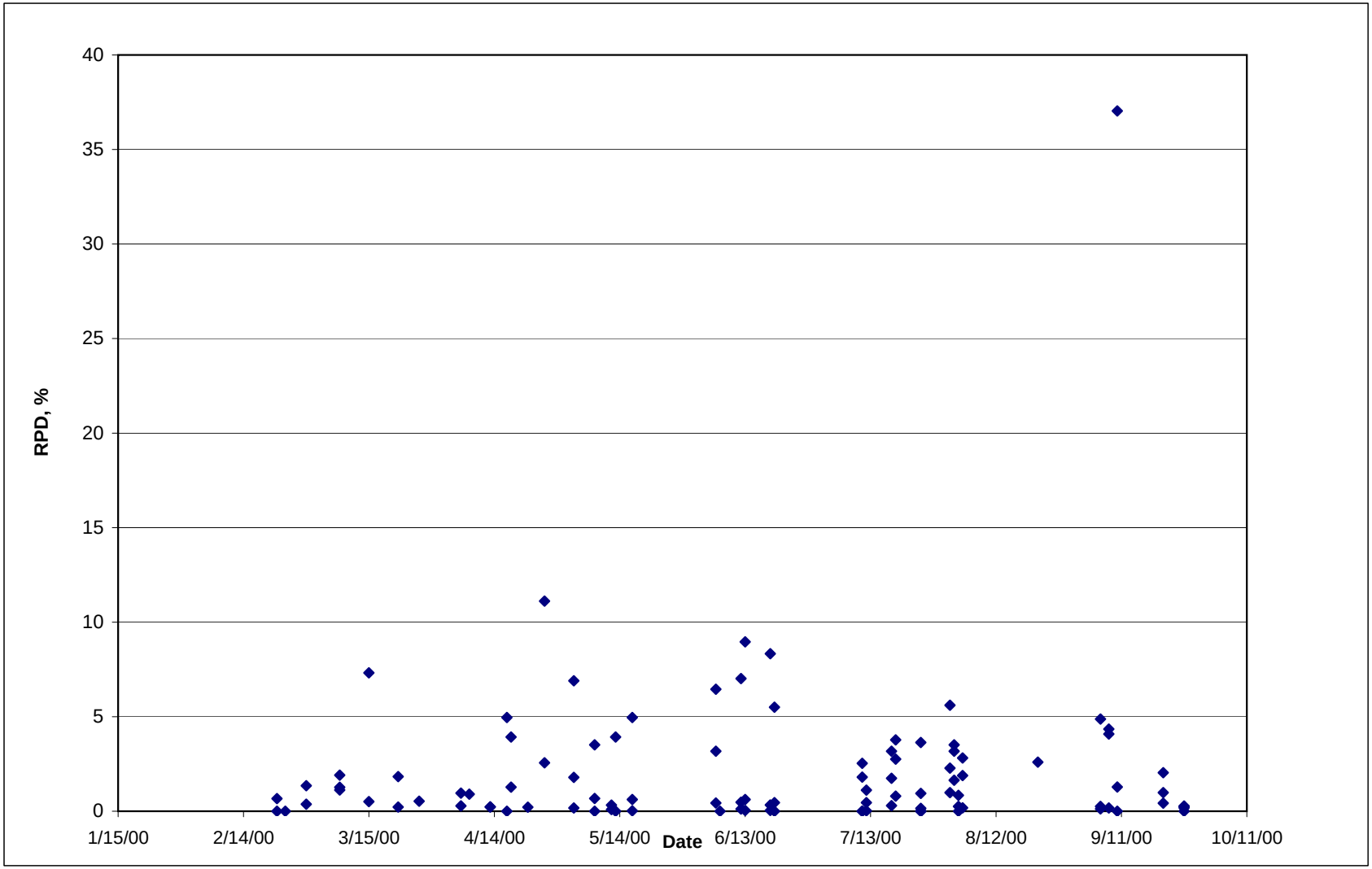

110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
Date
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00

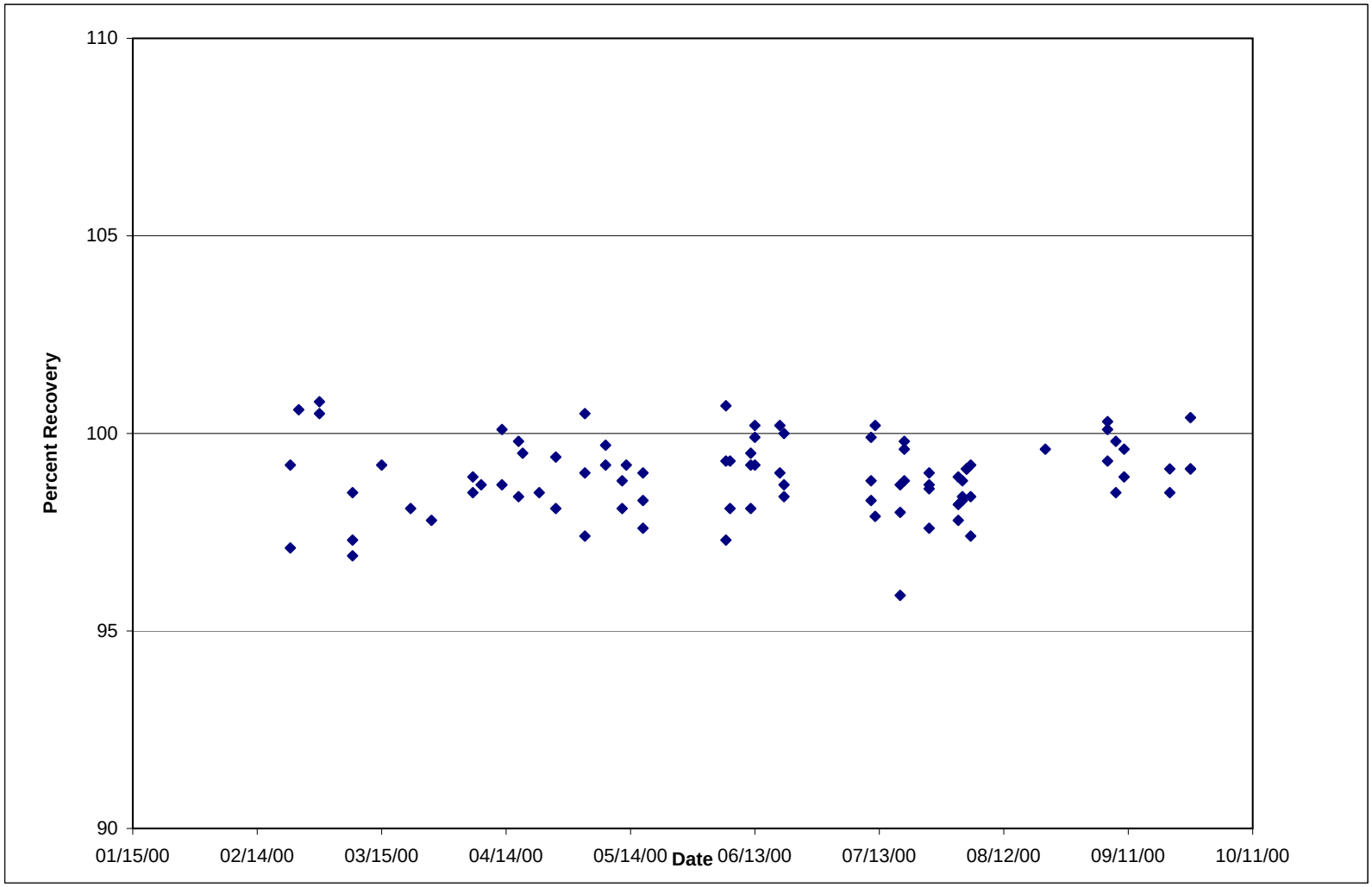

110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
Date
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00

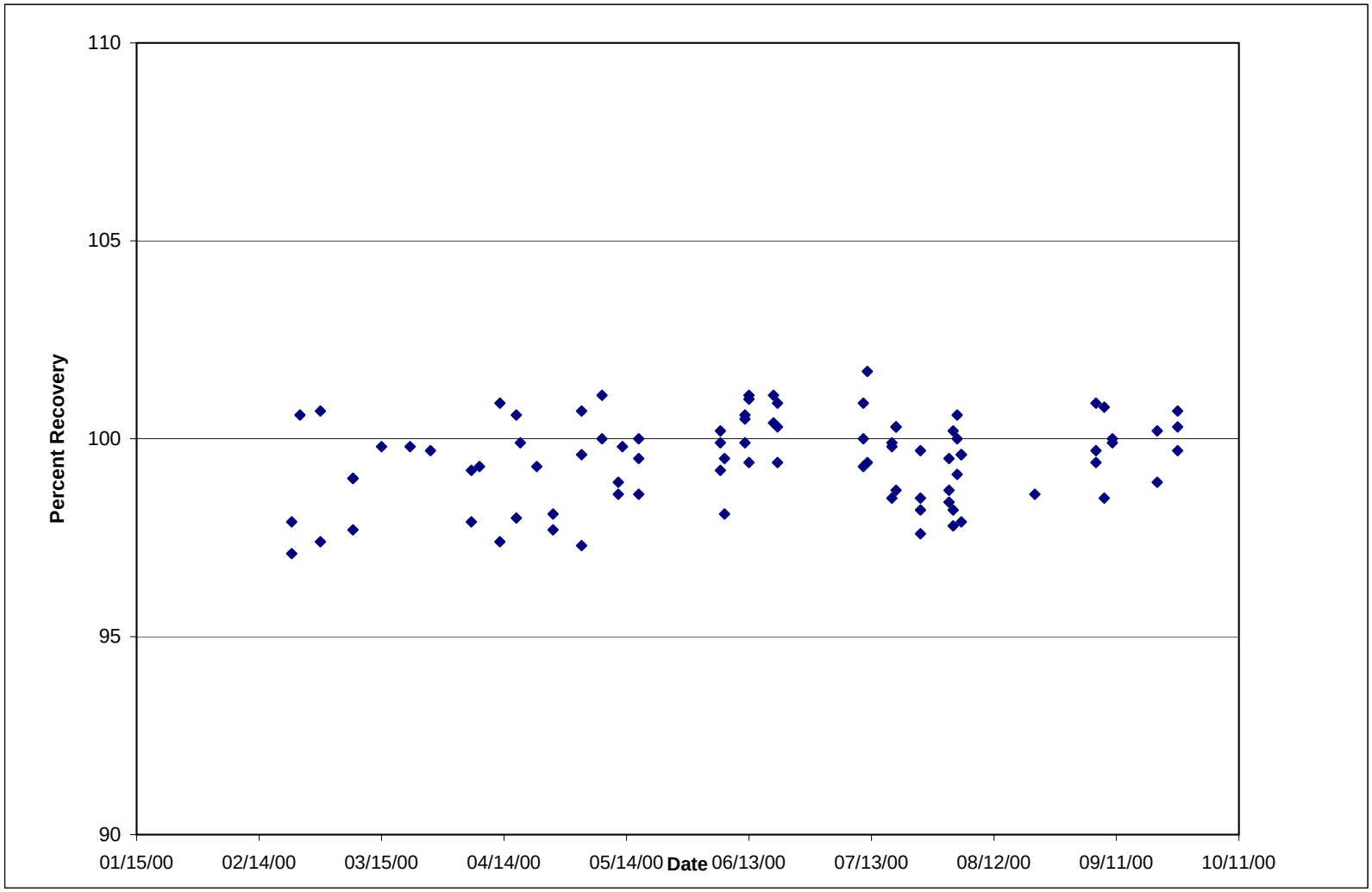

110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

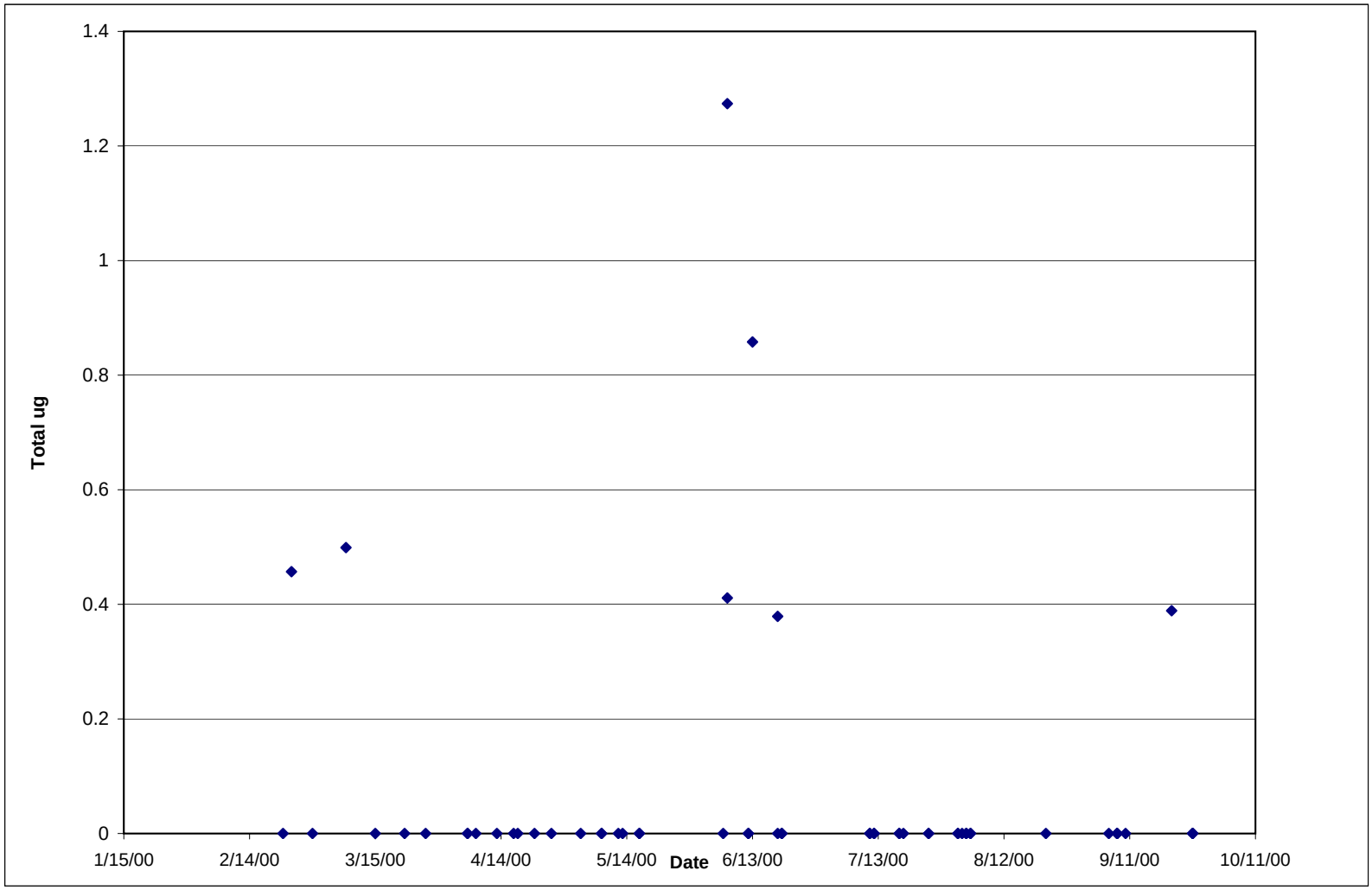

110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

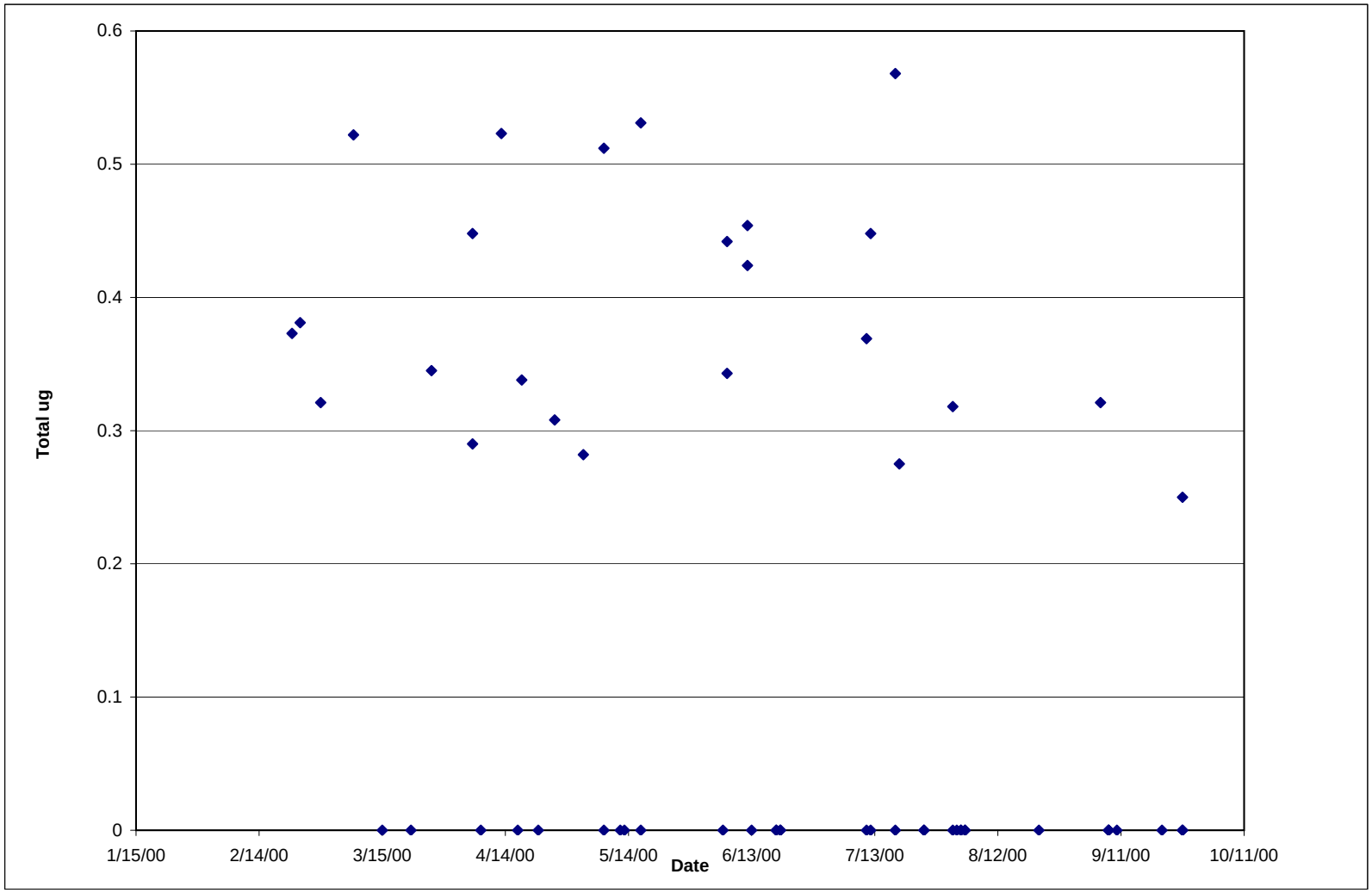

30
25
20
15
10
5
0
RPD, %
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

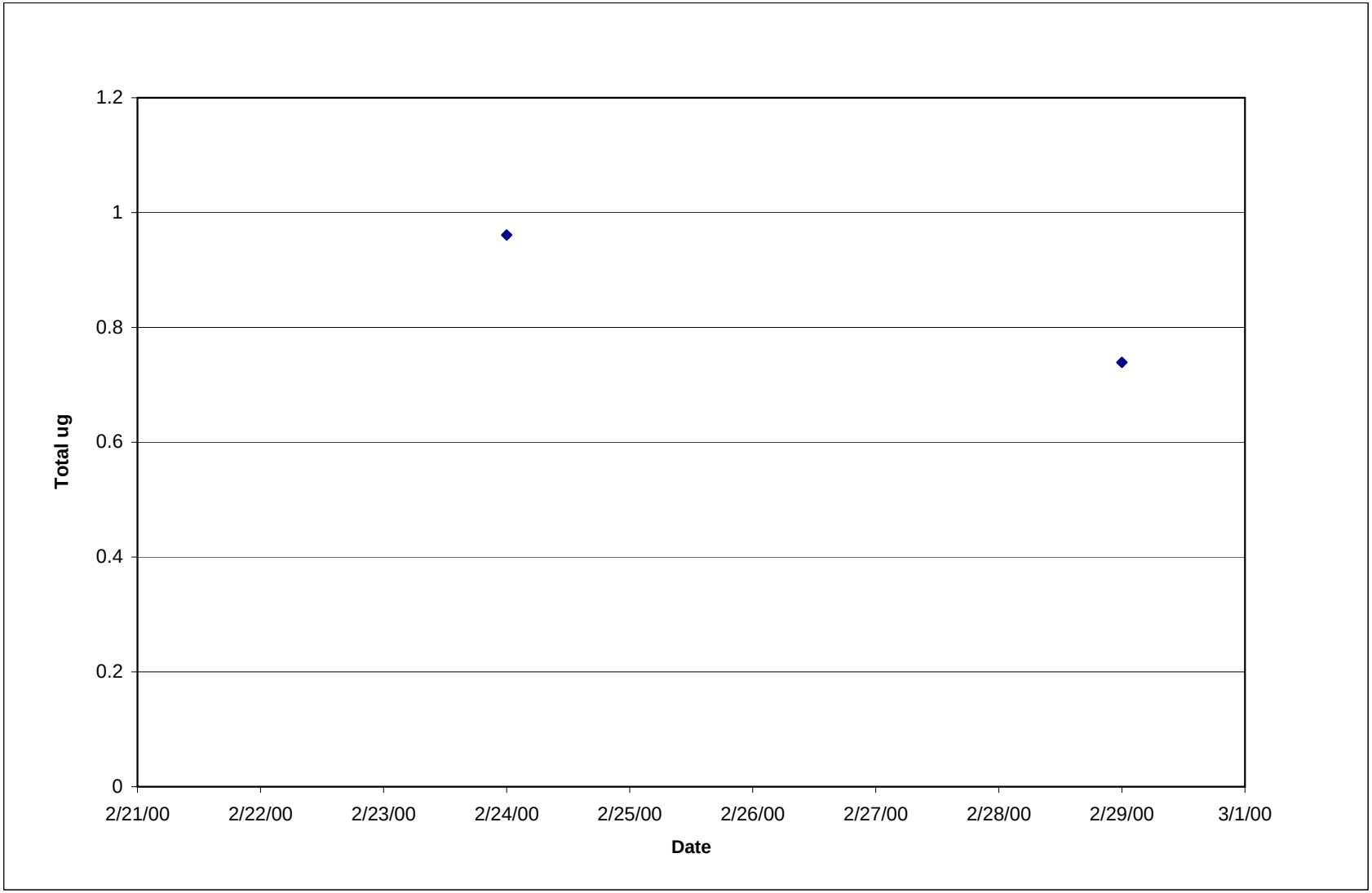

40
35
30
25
20
15
10
5
0
RPD, %
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

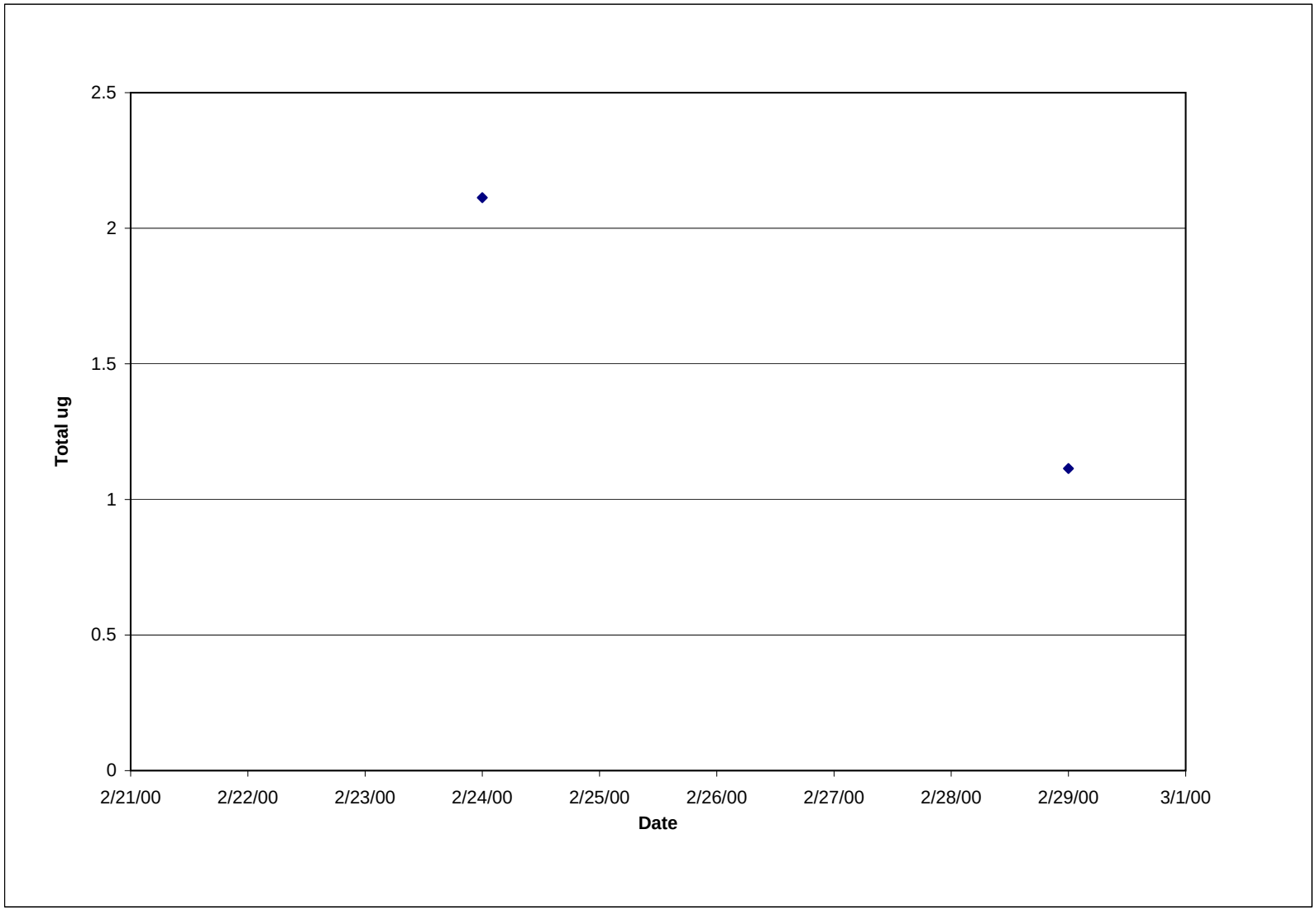

110
105
100
95
90
Percent Recovery
01/15/00
02/14/00
03/15/00
04/14/00
05/14/00
06/13/00
07/13/00
08/12/00
09/11/00
10/11/00
Date

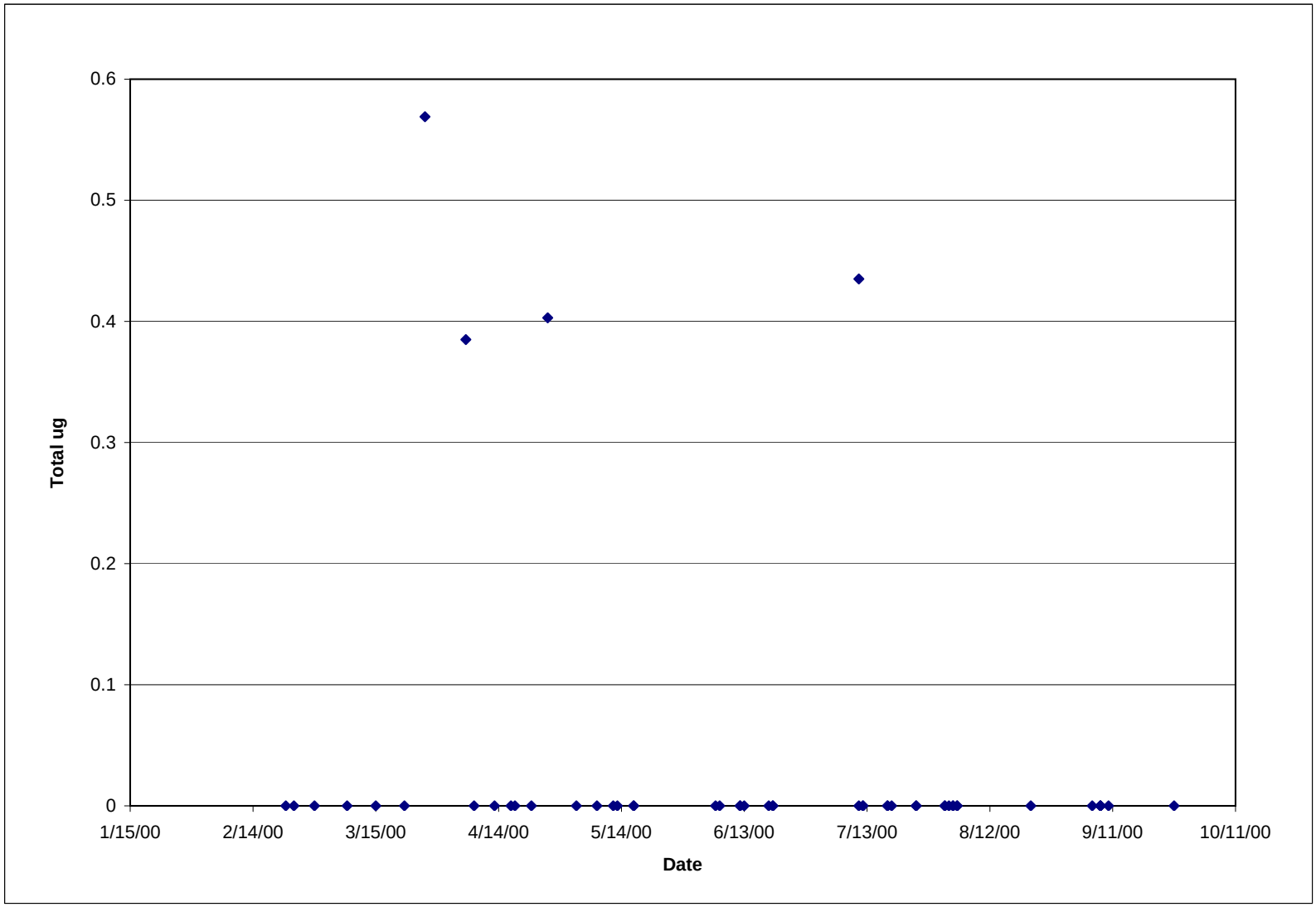

110
105
100
95
90
Percent Recovery
01/15/00
02/14/00
03/15/00
04/14/00
05/14/00
Date
06/13/00
07/13/00
08/12/00
09/11/00
10/11/00

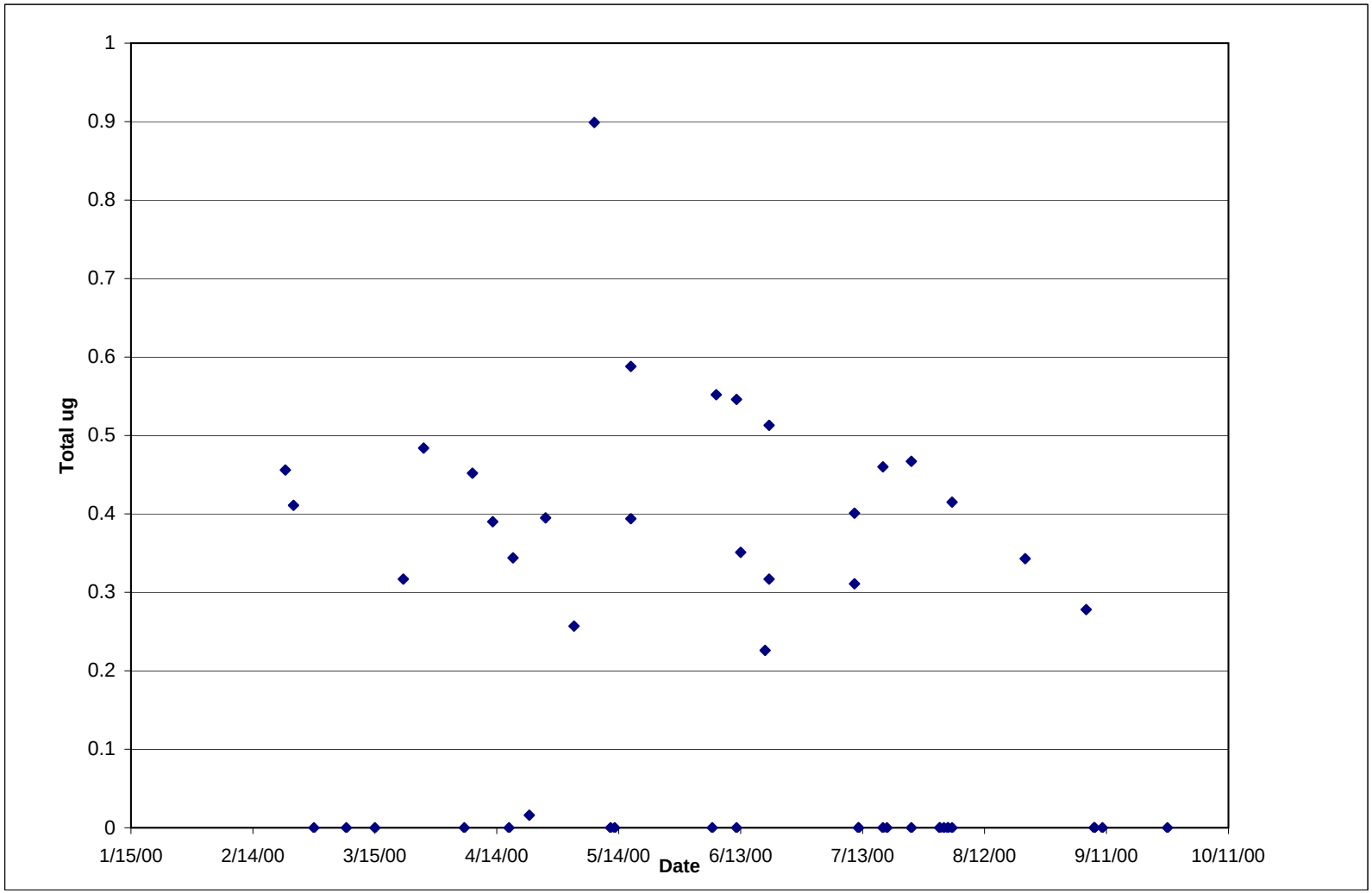

1.4
1.2
1
0.8
0.6
0.4
0.2
0
Total ug
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
Date
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00

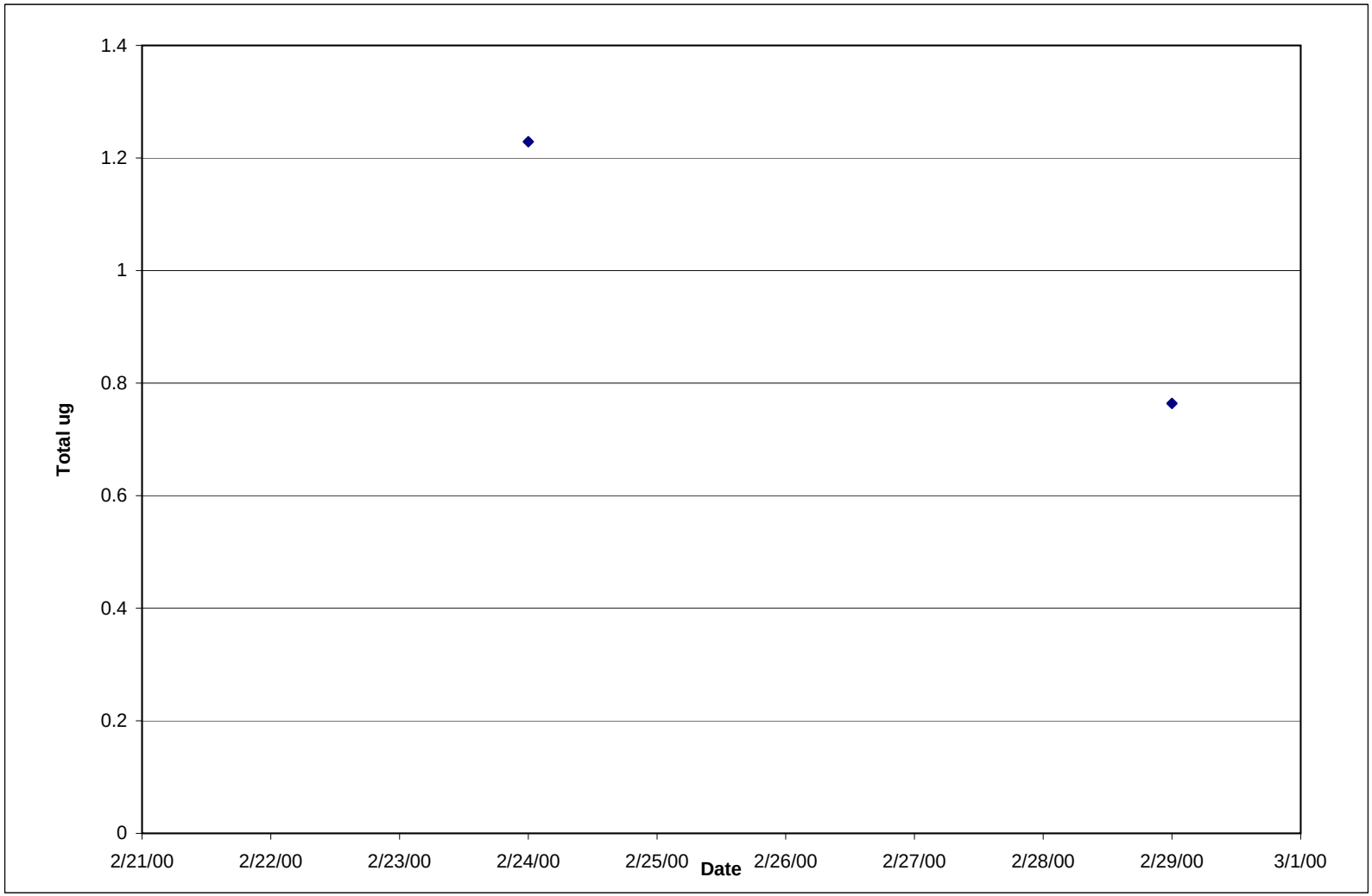

0.6
0.5
0.4
0.3
0.2
0.1
0
Total ug
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

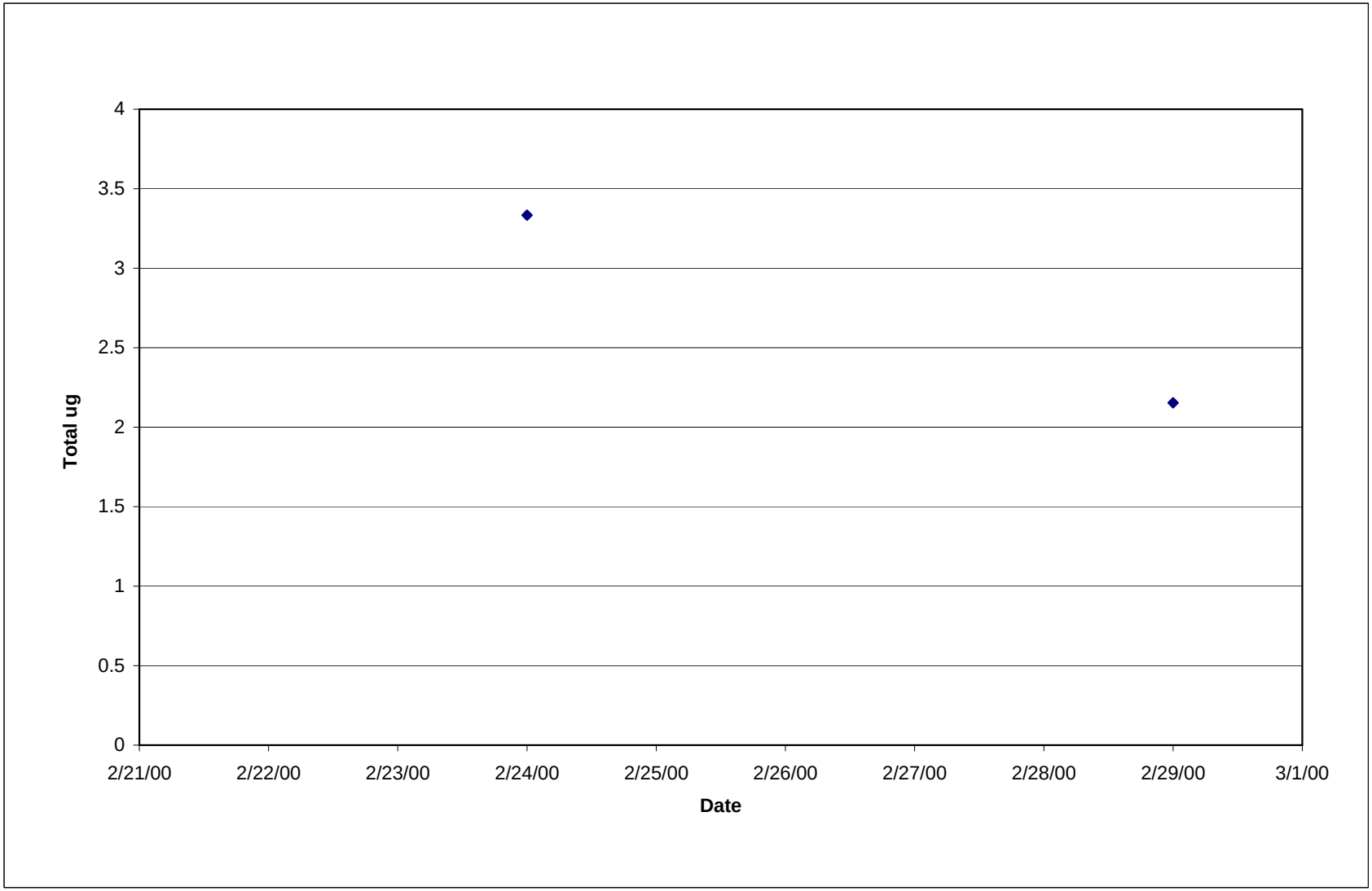

1.2
1
0.8
0.6
0.4
0.2
0
Total ug
2/21/00
2/22/00
2/23/00
2/24/00
2/25/00
2/26/00
2/27/00
2/28/00
2/29/00
3/1/00
Date


2.5
2
1.5
1
0.5
0
Total ug
2/21/00
2/22/00
2/23/00
2/24/00
2/25/00
2/26/00
2/27/00
2/28/00
2/29/00
3/1/00
Date


0.6
0.5
0.4
0.3
0.2
0.1
0
Total ug
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date


Total ug
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date


1.4
1.2
1
0.8
0.6
0.4
0.2
0
Total ug
2/21/00
2/22/00
2/23/00
2/24/00
2/25/00
2/26/00
2/27/00
2/28/00
2/29/00
3/1/00
Date


4
3.5
3
2.5
2
1.5
1
0.5
0
Total ug
2/21/00
2/22/00
2/23/00
2/24/00
2/25/00
2/26/00
2/27/00
2/28/00
2/29/00
3/1/00
Date

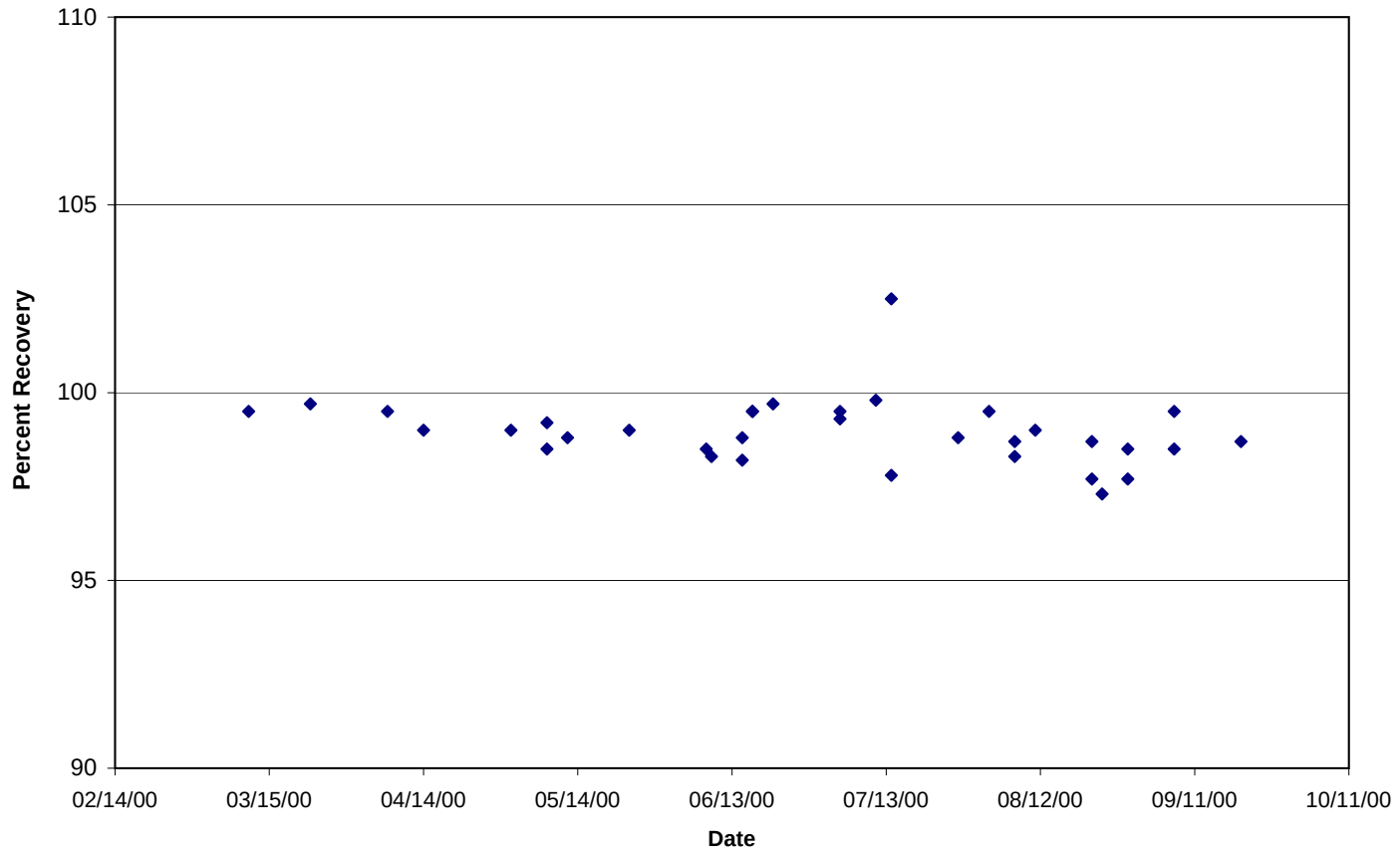


Figure 17a. IC System 3 -- Percent Recovery for Low NO_3 QC Sample
110
105
100
95
90
Percent Recovery
02/14/00
03/15/00
04/14/00
05/14/00
06/13/00
07/13/00
08/12/00
09/11/00
10/11/00
Date

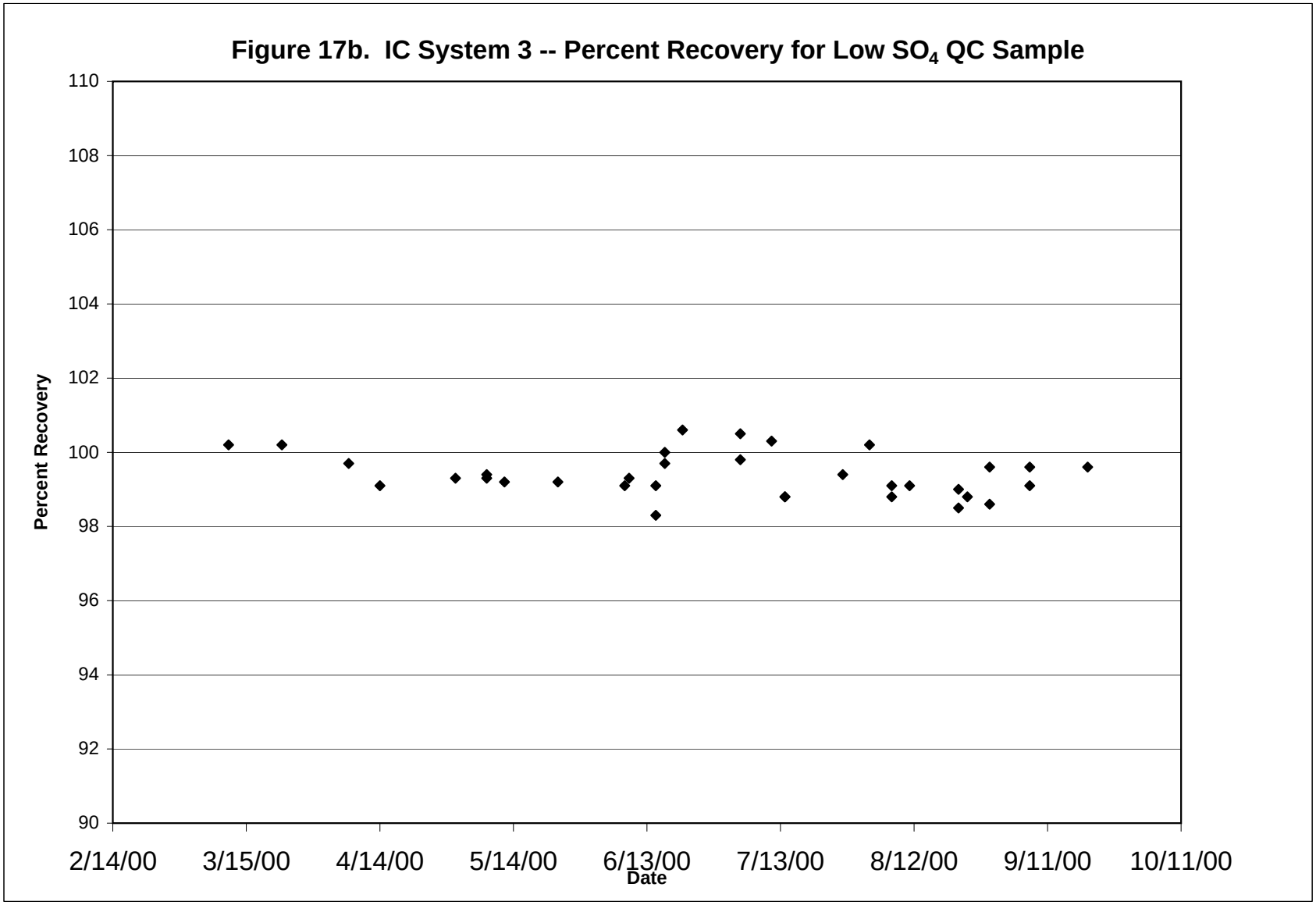


Figure 17b. IC System 3 -- Percent Recovery for Low SO_4 QC Sample
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

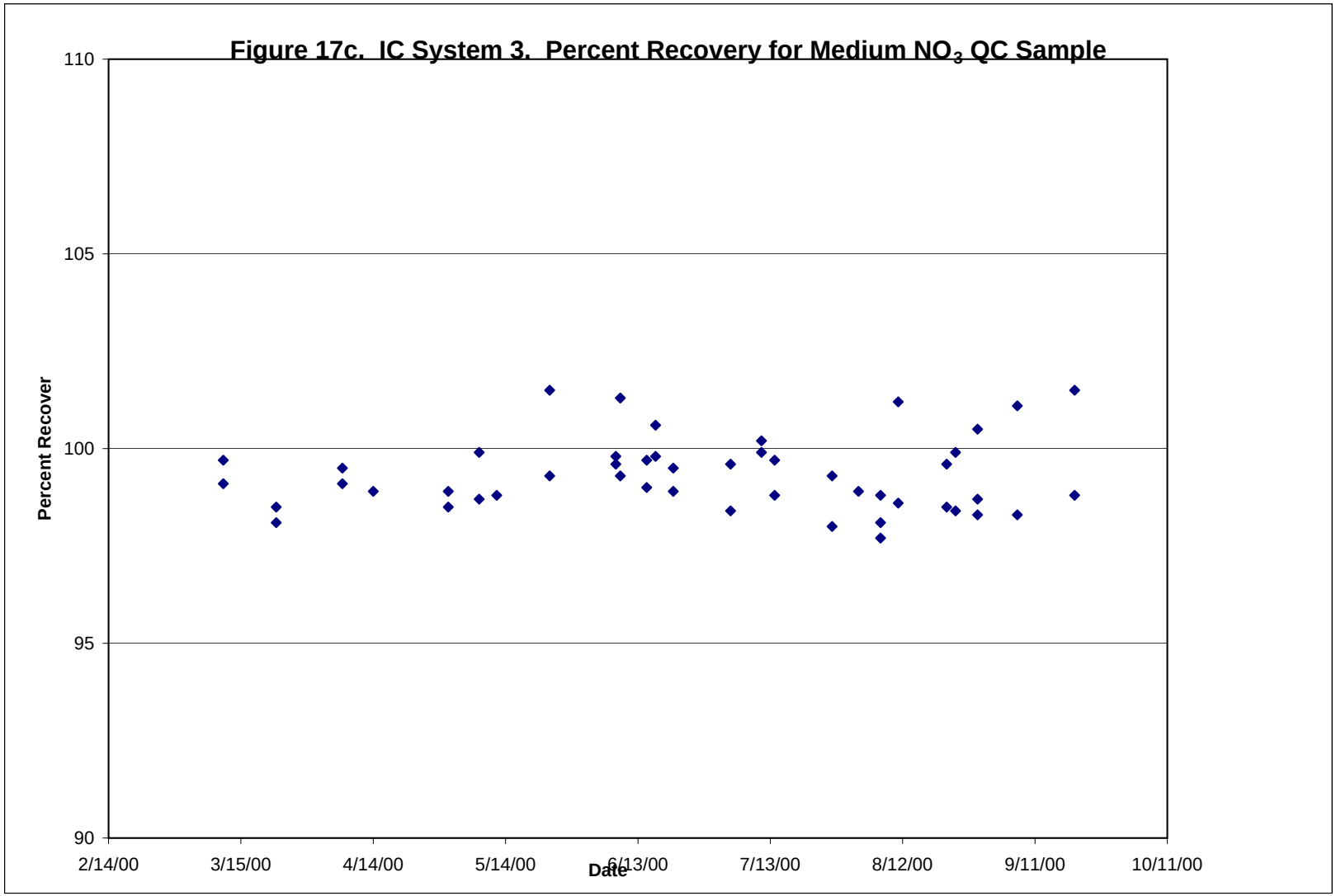


Figure 17c. IC System 3. Percent Recovery for Medium NO_3 QC Sample
110
105
100
95
90
Percent Recover
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

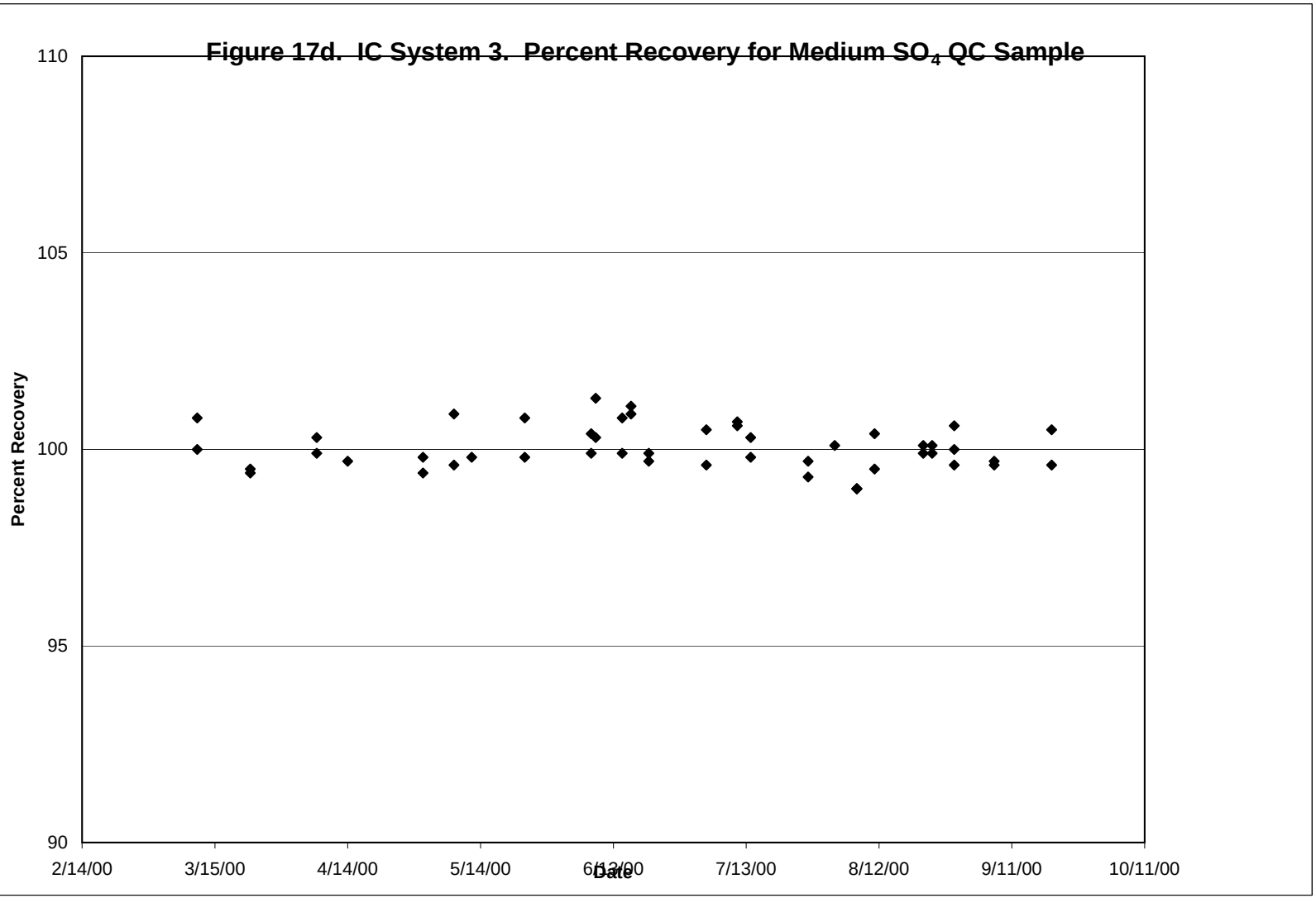


Figure 17d. IC System 3. Percent Recovery for Medium SO_4 QC Sample
110
105
100
95
90
Percent Recovery
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

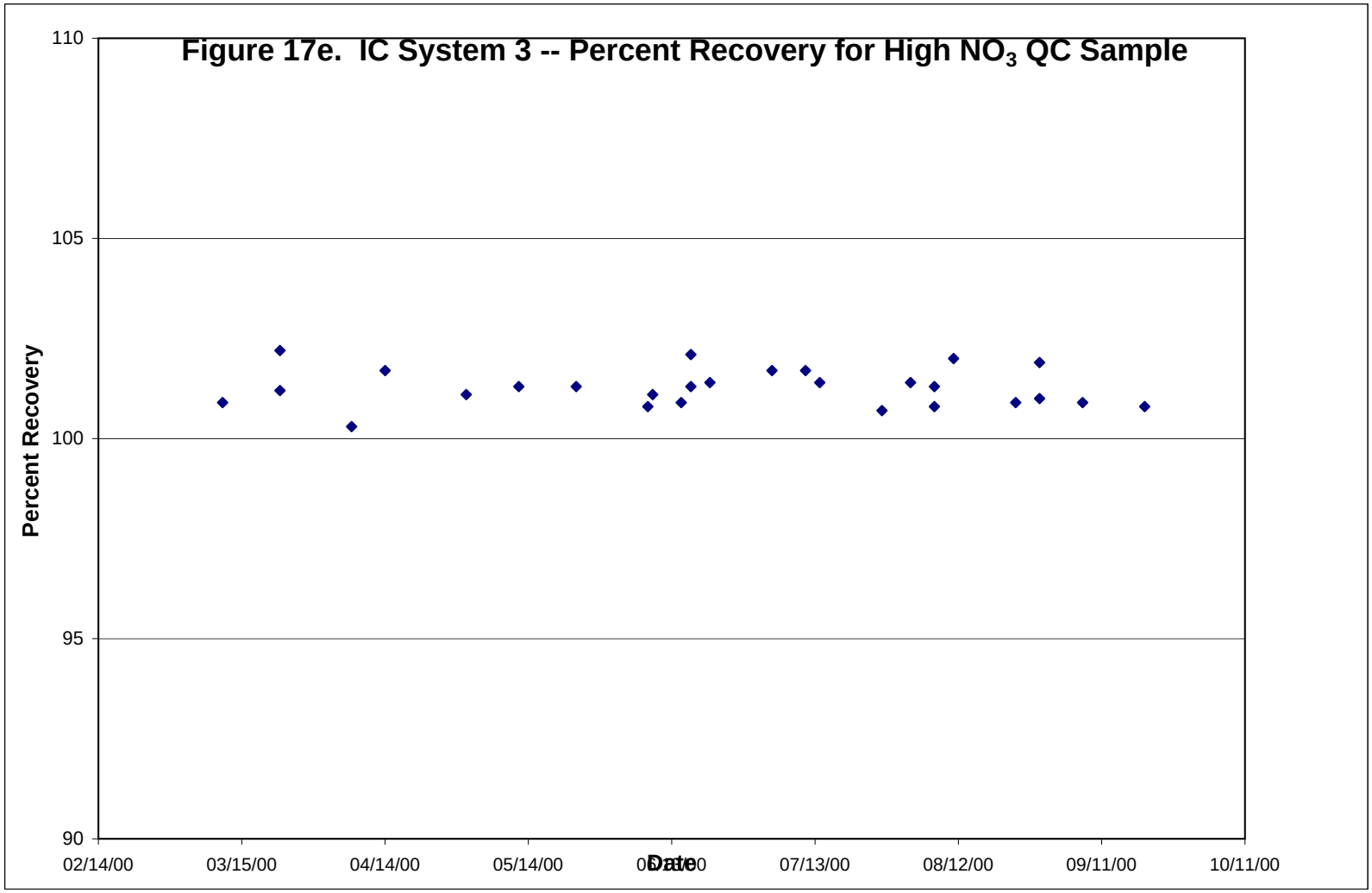


Figure 17e. IC System 3 -- Percent Recovery for High NO_3 QC Sample
Percent Recovery
110
105
100
95
90
02/14/00
03/15/00
04/14/00
05/14/00
06/13/00
07/13/00
08/12/00
09/11/00
10/11/00
Date

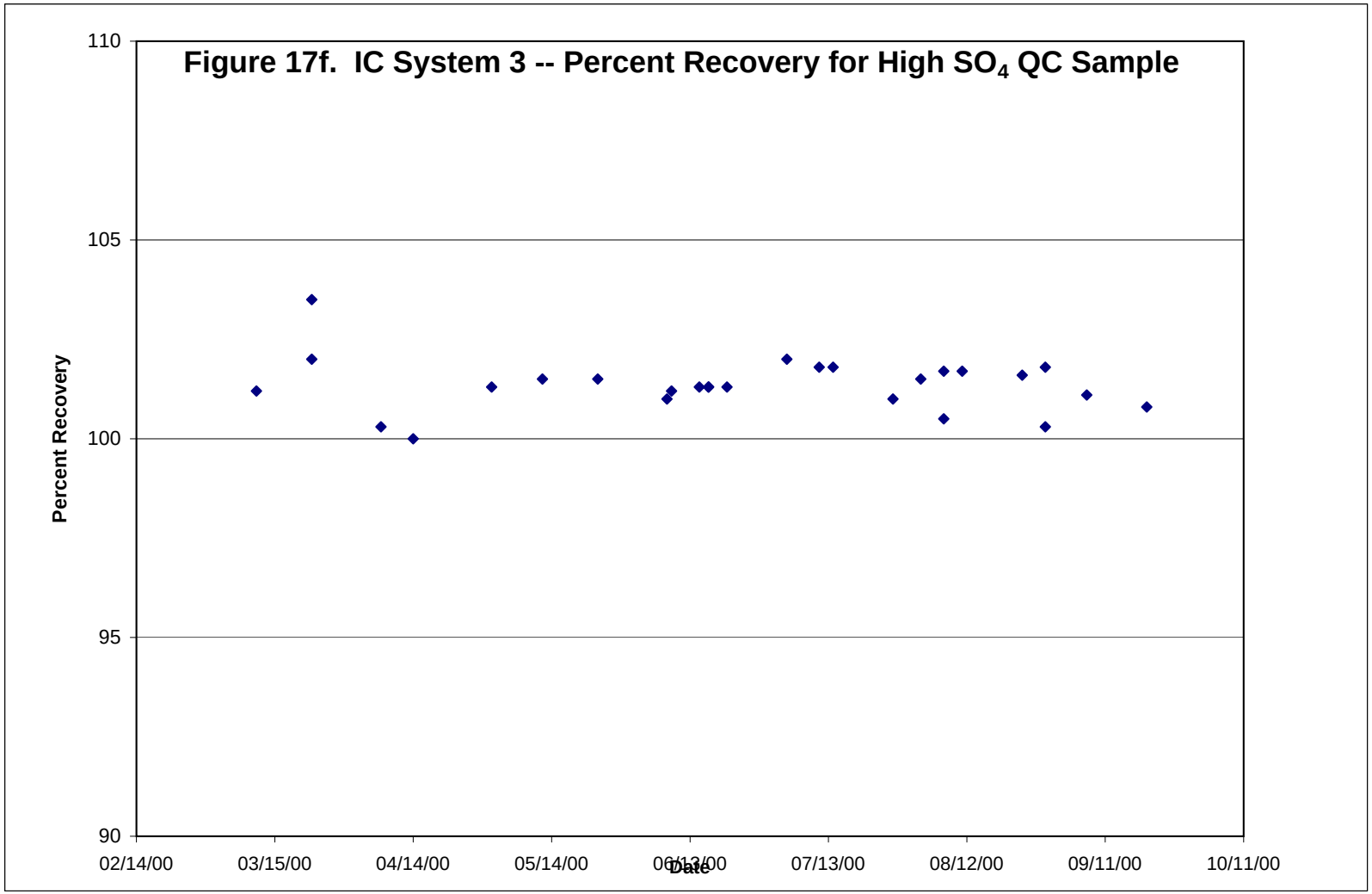


Figure 17f. IC System 3 -- Percent Recovery for High SO_4 QC Sample
Percent Recovery
110
105
100
95
90
02/14/00
03/15/00
04/14/00
05/14/00
06/13/00
07/13/00
08/12/00
09/11/00
10/11/00
Date

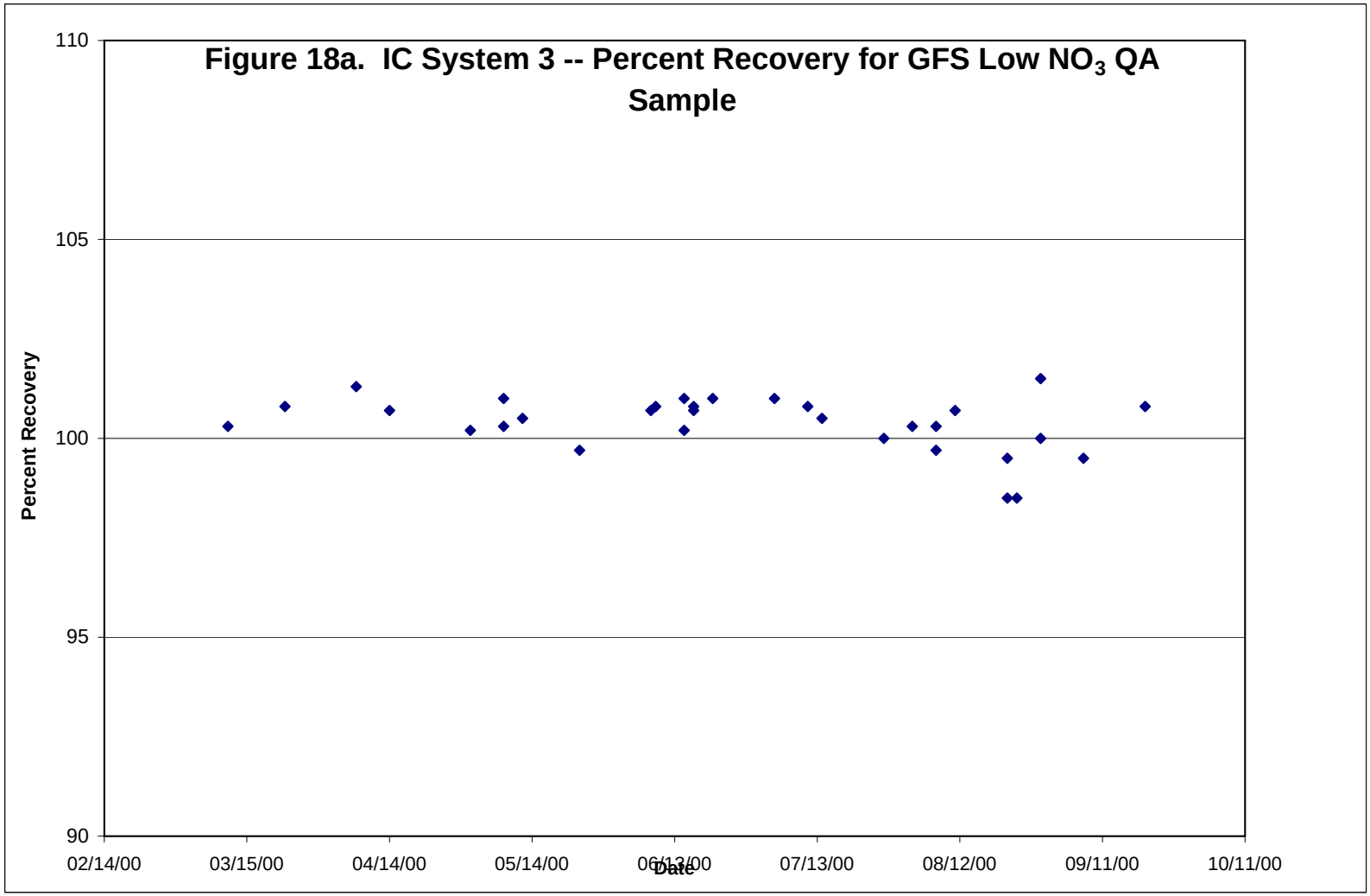


Figure 18a. IC System 3 -- Percent Recovery for GFS Low NO_3 QA Sample
Percent Recovery
110
105
100
95
90
02/14/00
03/15/00
04/14/00
05/14/00
06/13/00
07/13/00
08/12/00
09/11/00
10/11/00
Date

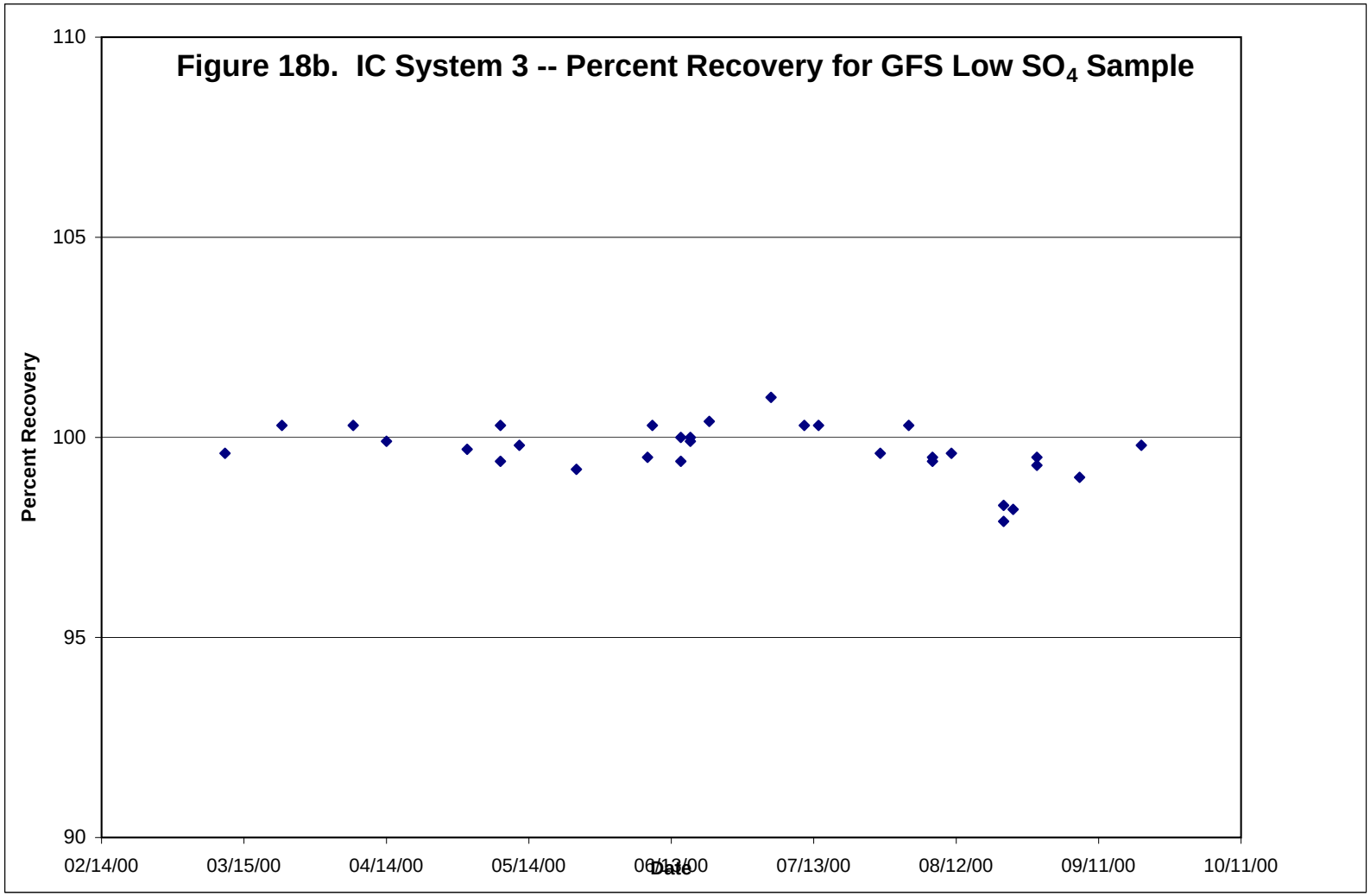


Figure 18b. IC System 3 -- Percent Recovery for GFS Low SO_4 Sample
Percent Recovery
110
105
100
95
90
02/14/00
03/15/00
04/14/00
05/14/00
06/13/00
07/13/00
08/12/00
09/11/00
10/11/00
Date

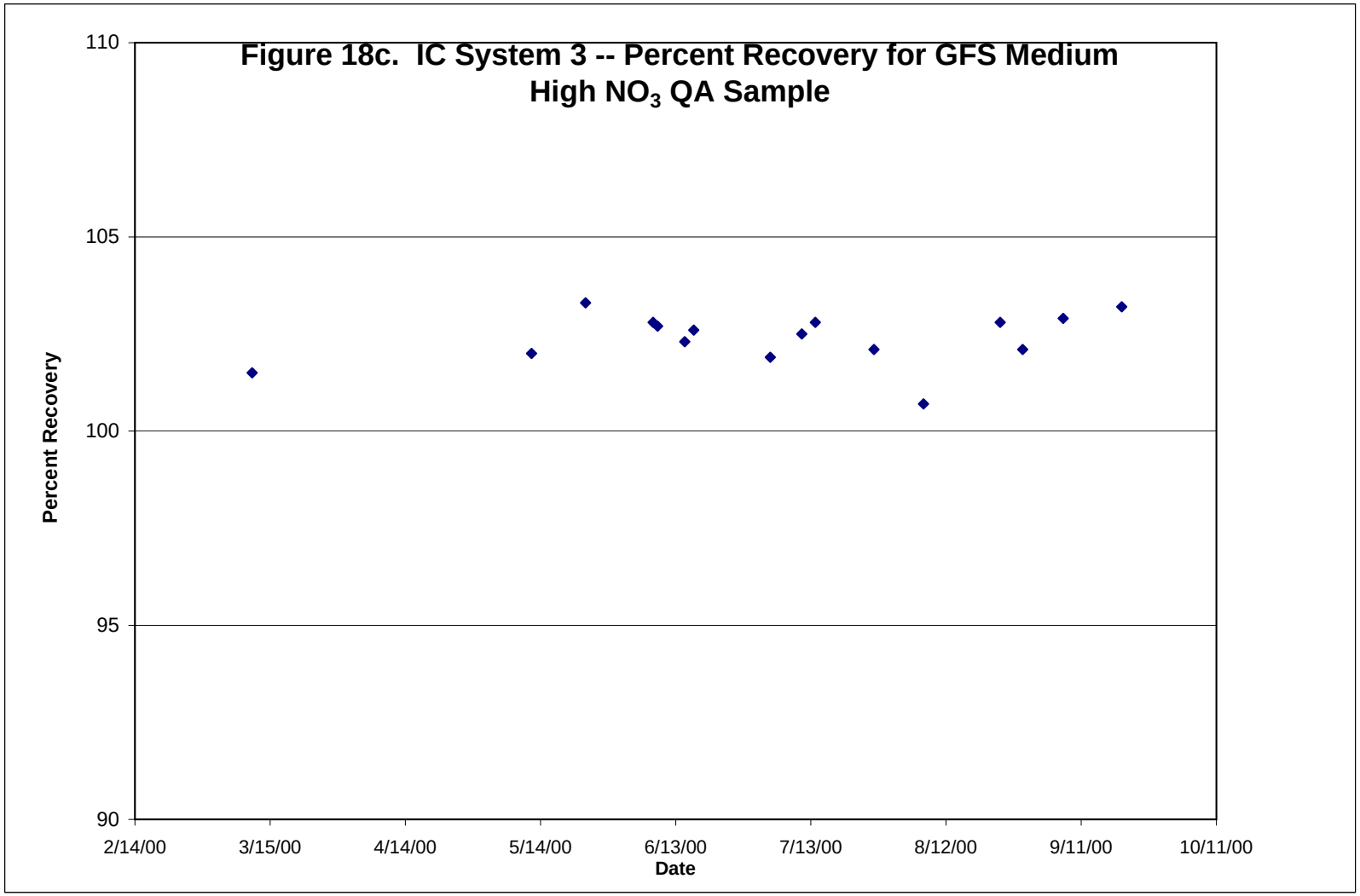


Figure 18c. IC System 3 -- Percent Recovery for GFS Medium High NO_3 QA Sample
110
105
100
95
90
Percent Recovery
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

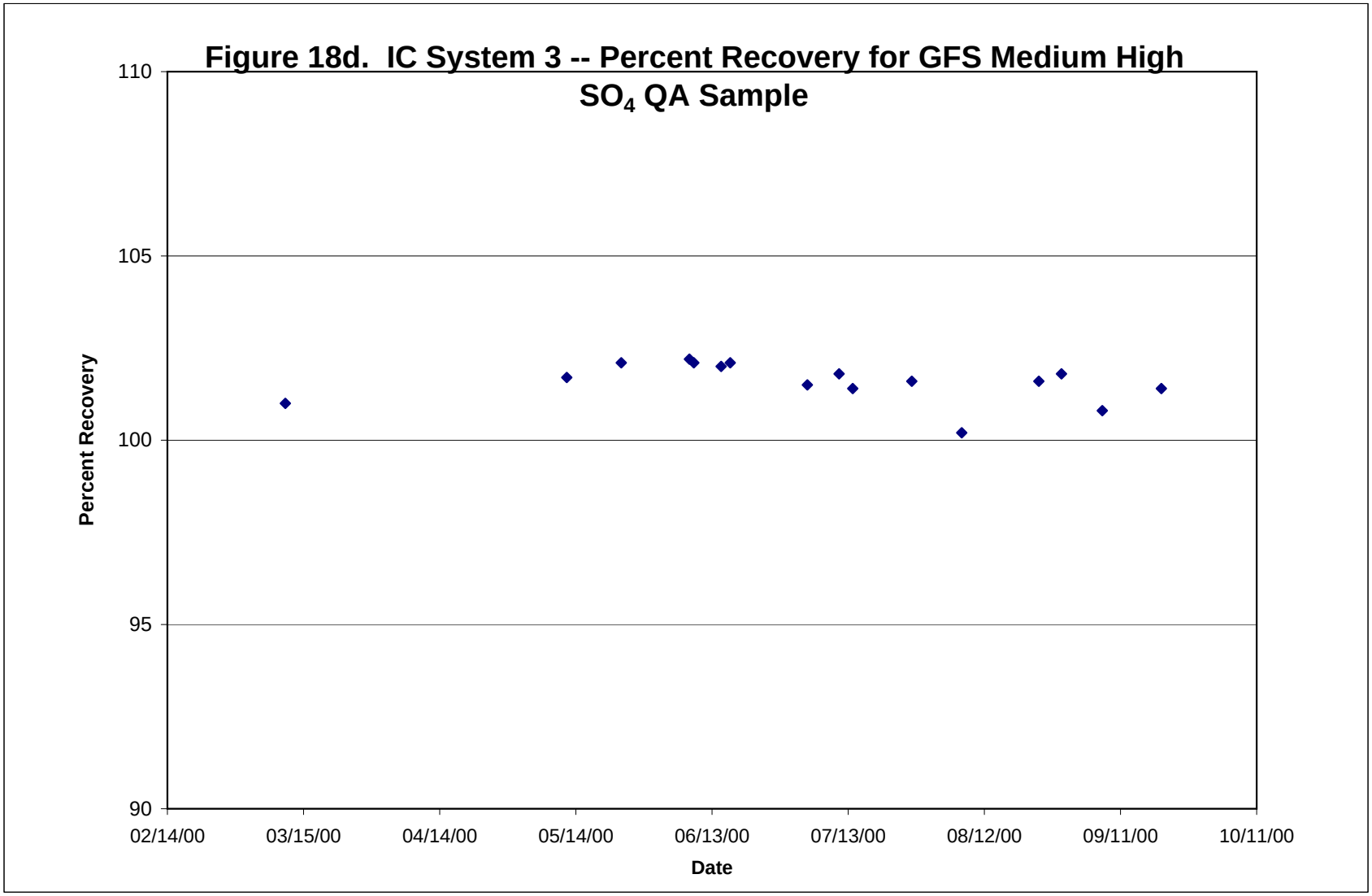


Figure 18d. IC System 3 -- Percent Recovery for GFS Medium High SO_4 QA Sample
110
105
100
95
90
Percent Recovery
02/14/00
03/15/00
04/14/00
05/14/00
06/13/00
07/13/00
08/12/00
09/11/00
10/11/00
Date

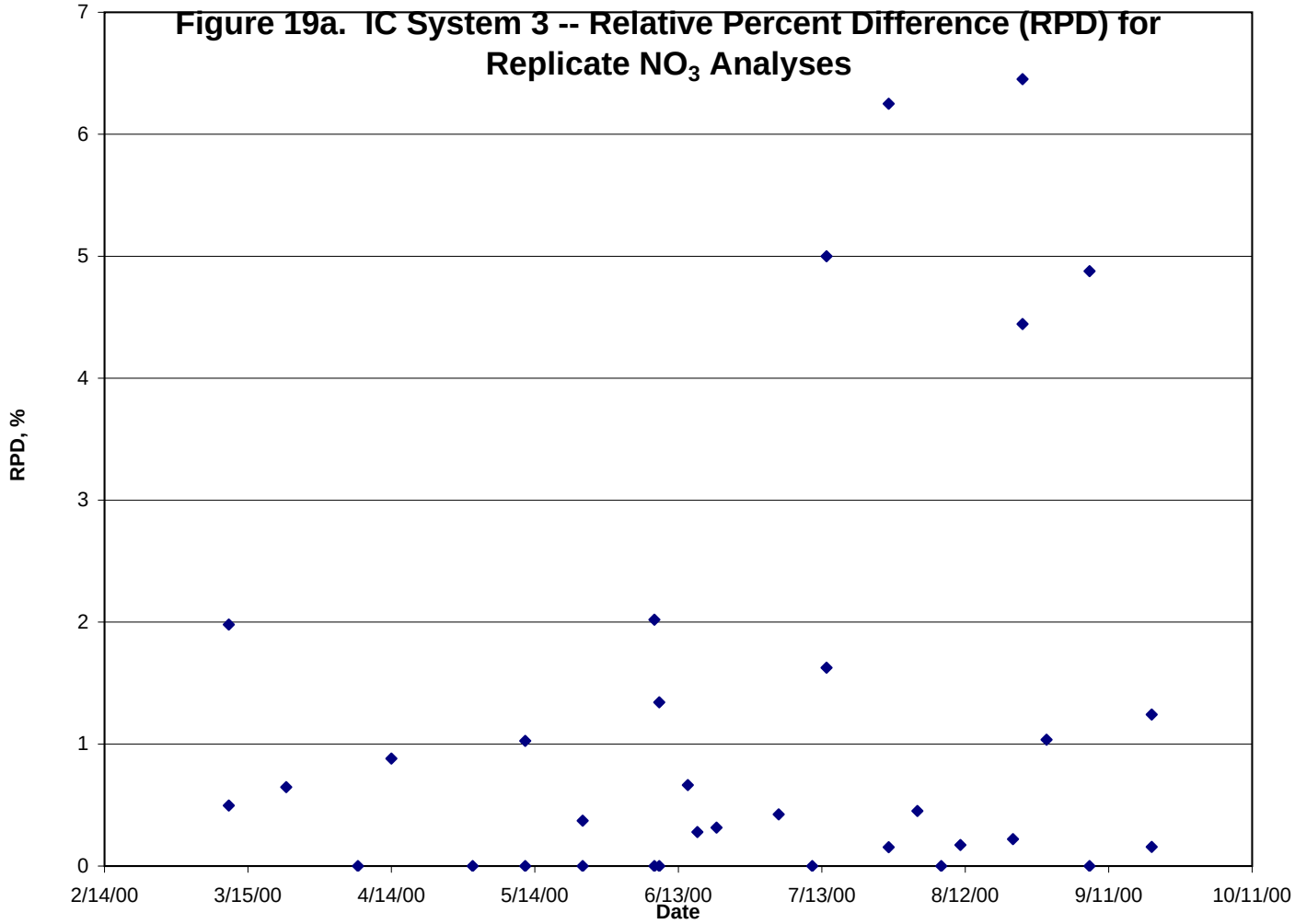


Figure 19a. IC System 3 -- Relative Percent Difference (RPD) for Replicate NO_3 Analyses
RPD, %
0
1
2
3
4
5
6
7
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

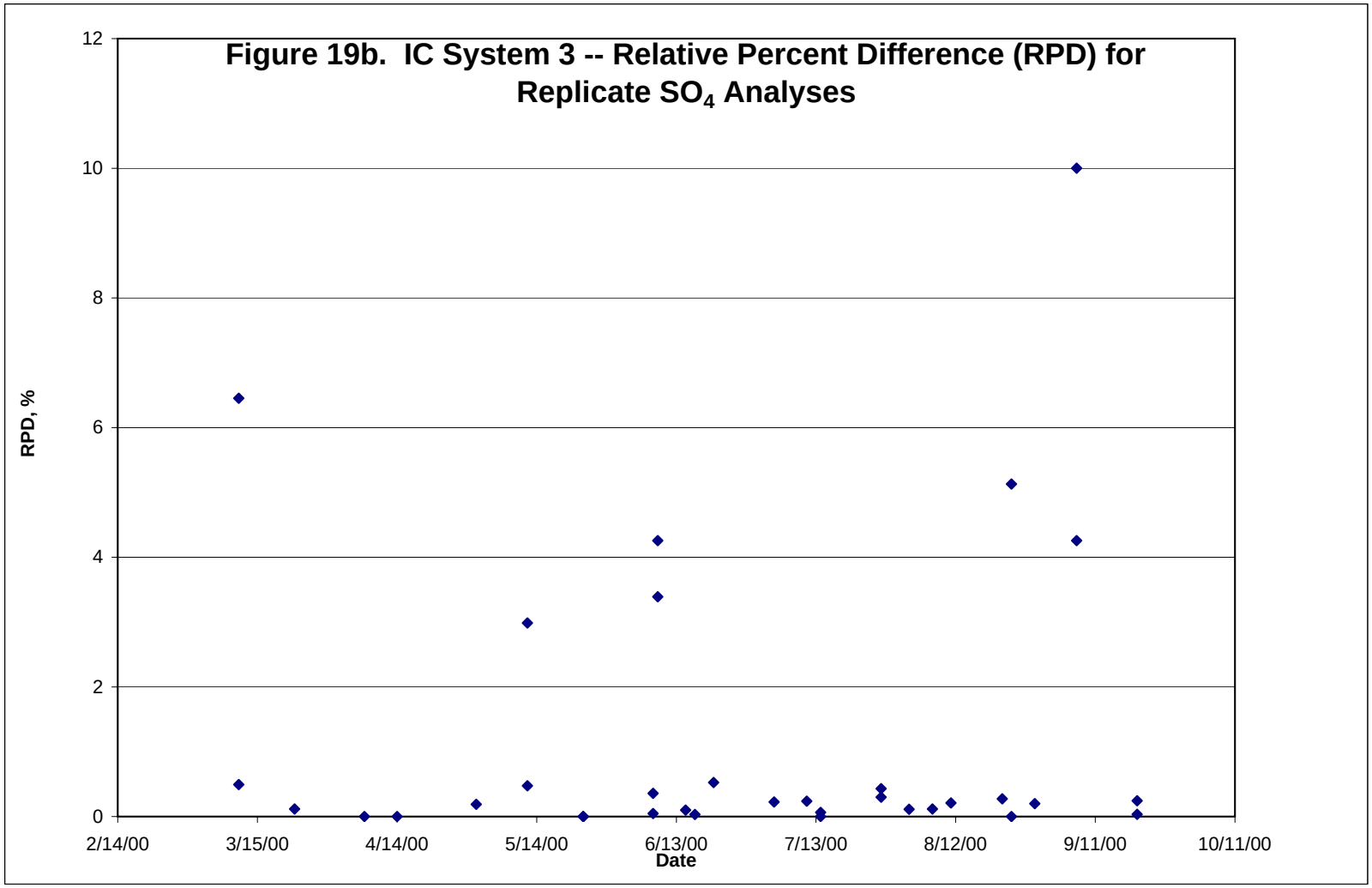


Figure 19b. IC System 3 -- Relative Percent Difference (RPD) for Replicate SO_4 Analyses
RPD, %
12
10
8
6
4
2
0
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

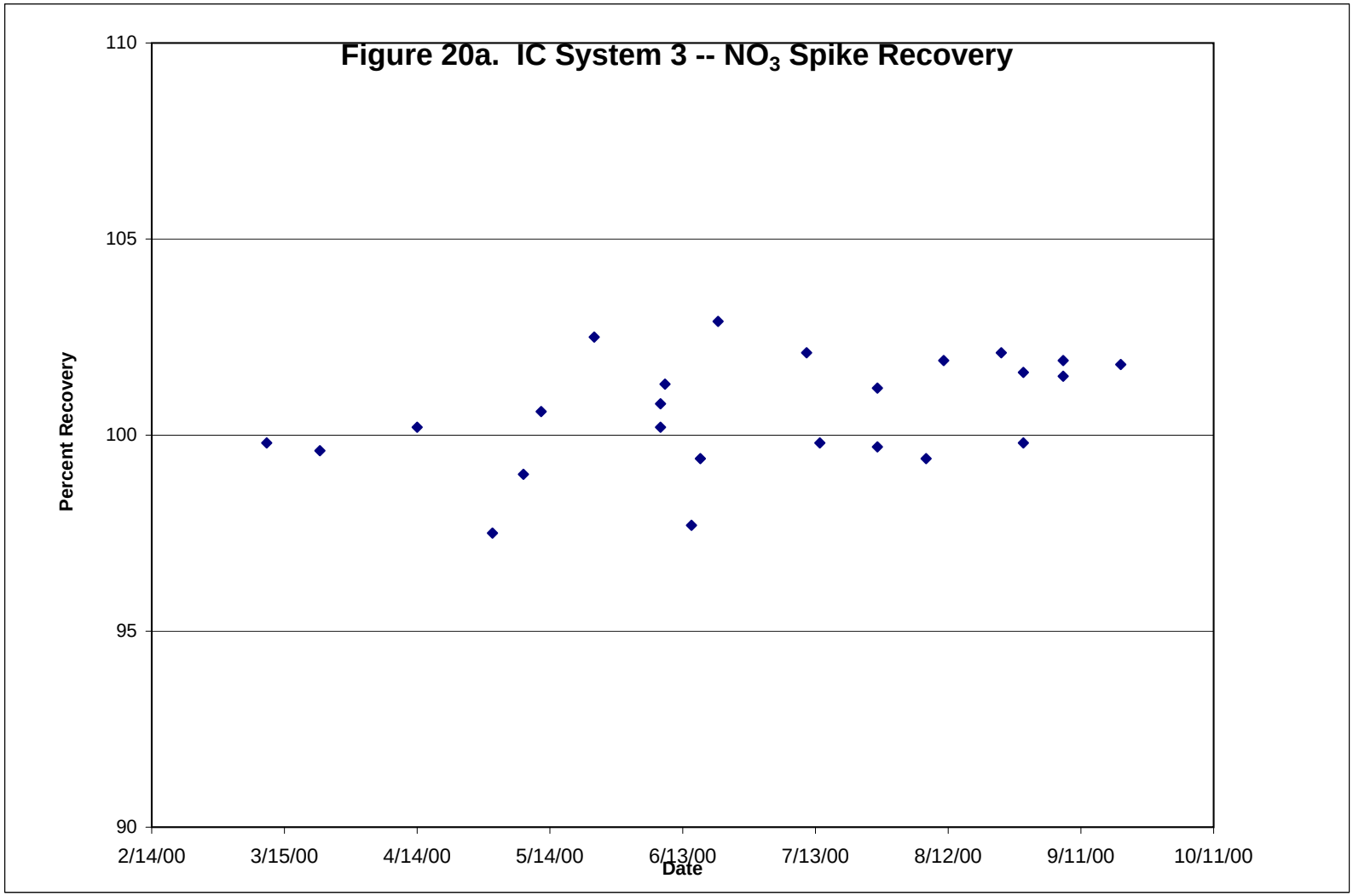


Figure 20a. IC System 3 -- NO_3 Spike Recovery
110
105
100
95
90
Percent Recovery
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

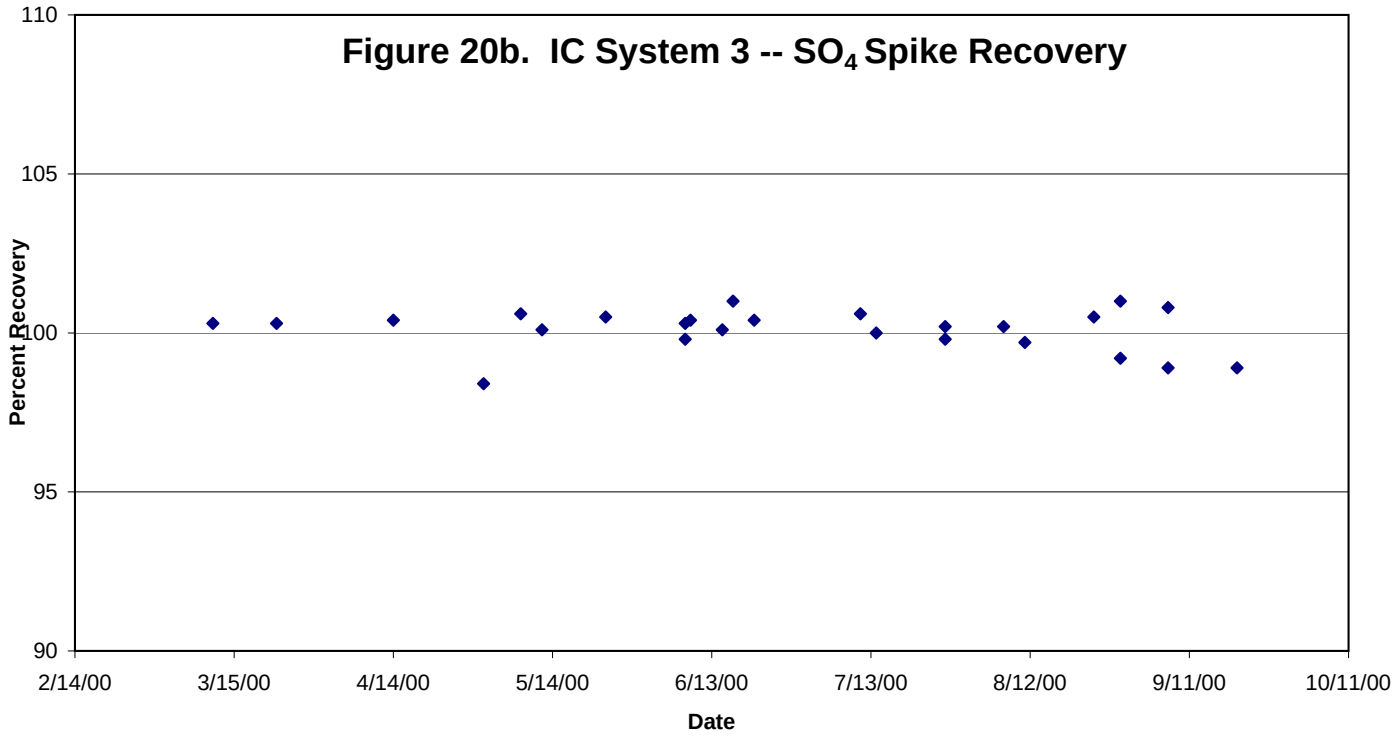


Figure 20b. IC System 3 -- SO_4 Spike Recovery
110
105
100
95
90
Percent Recovery
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

Figure 21a. IC System 3 -- NO_3 in Deionized Water Blank

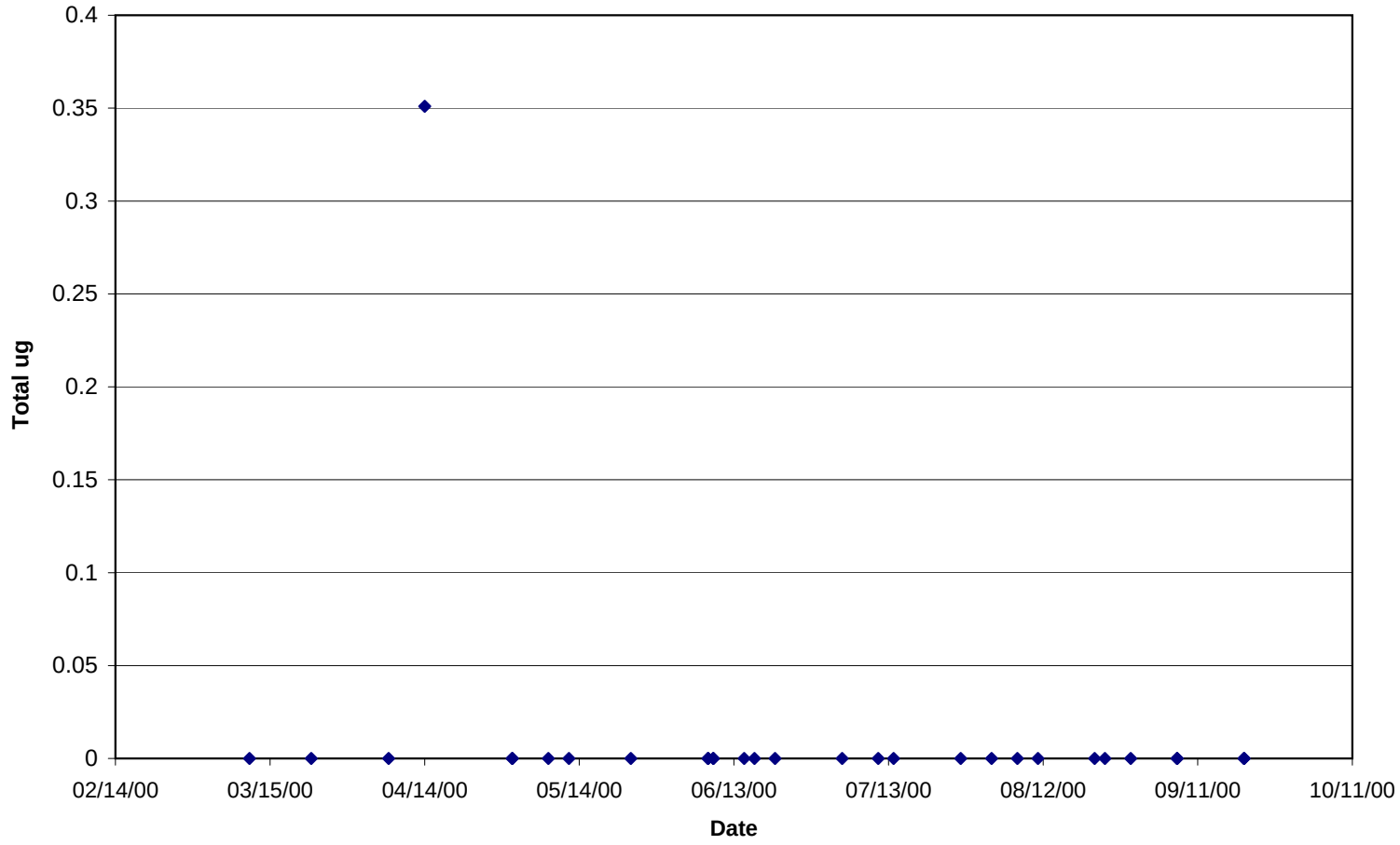
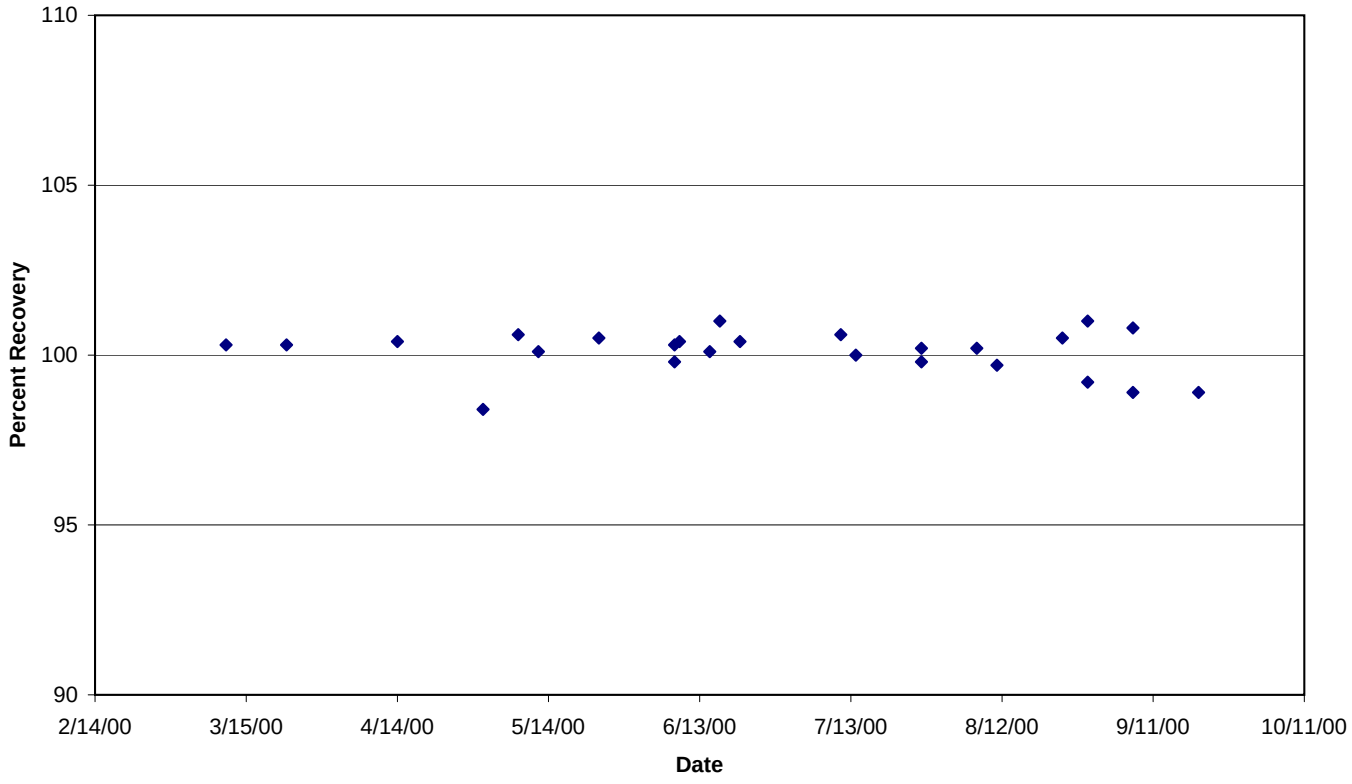


Figure 21b. IC System 3 -- SO_4 Spike Recovery



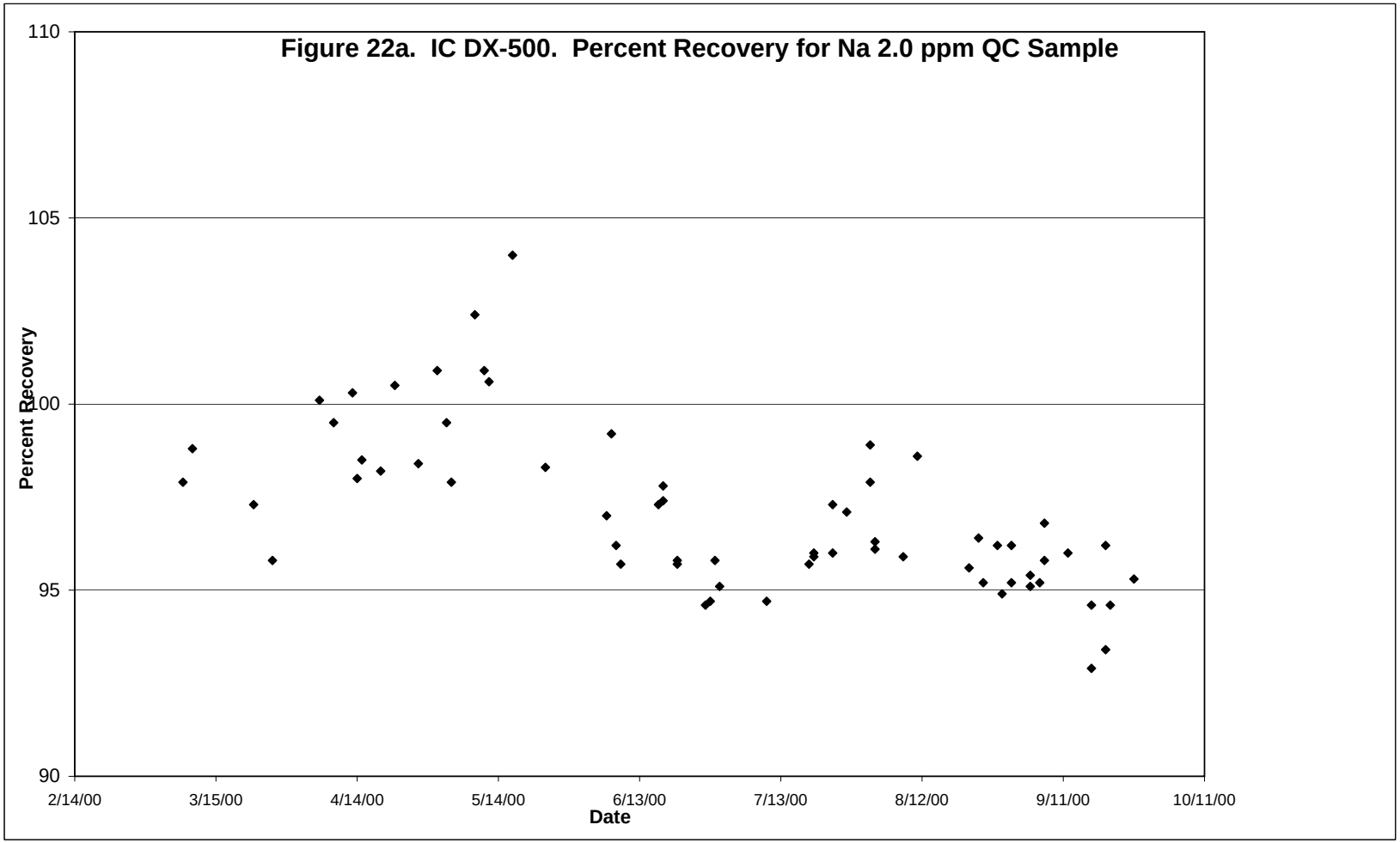


Figure 22a. IC DX-500. Percent Recovery for Na 2.0 ppm QC Sample
110
105
100
95
90
Percent Recovery
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

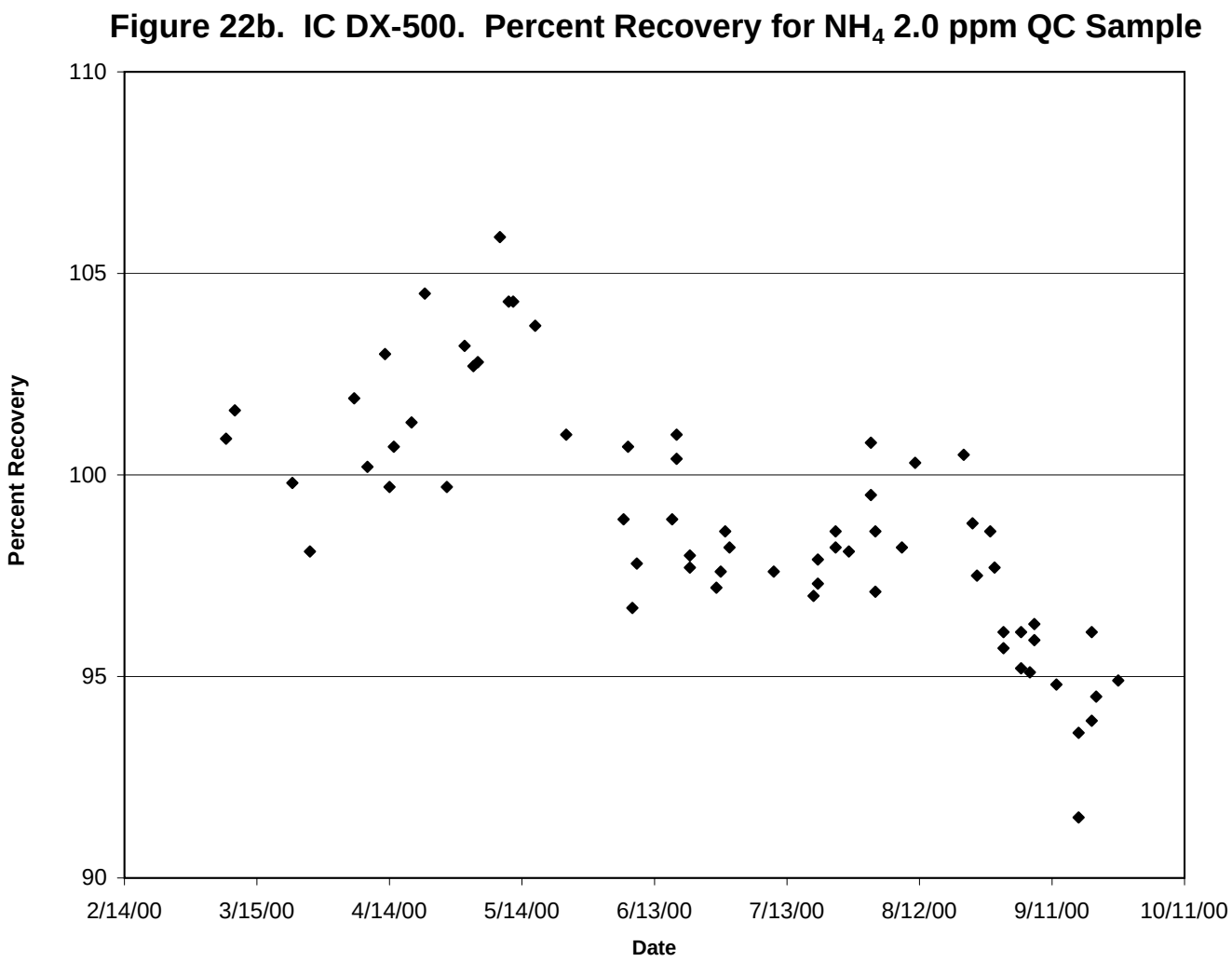


Figure 22b. IC DX-500. Percent Recovery for NH_4 2.0 ppm QC Sample
110
105
100
95
90
Percent Recovery
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

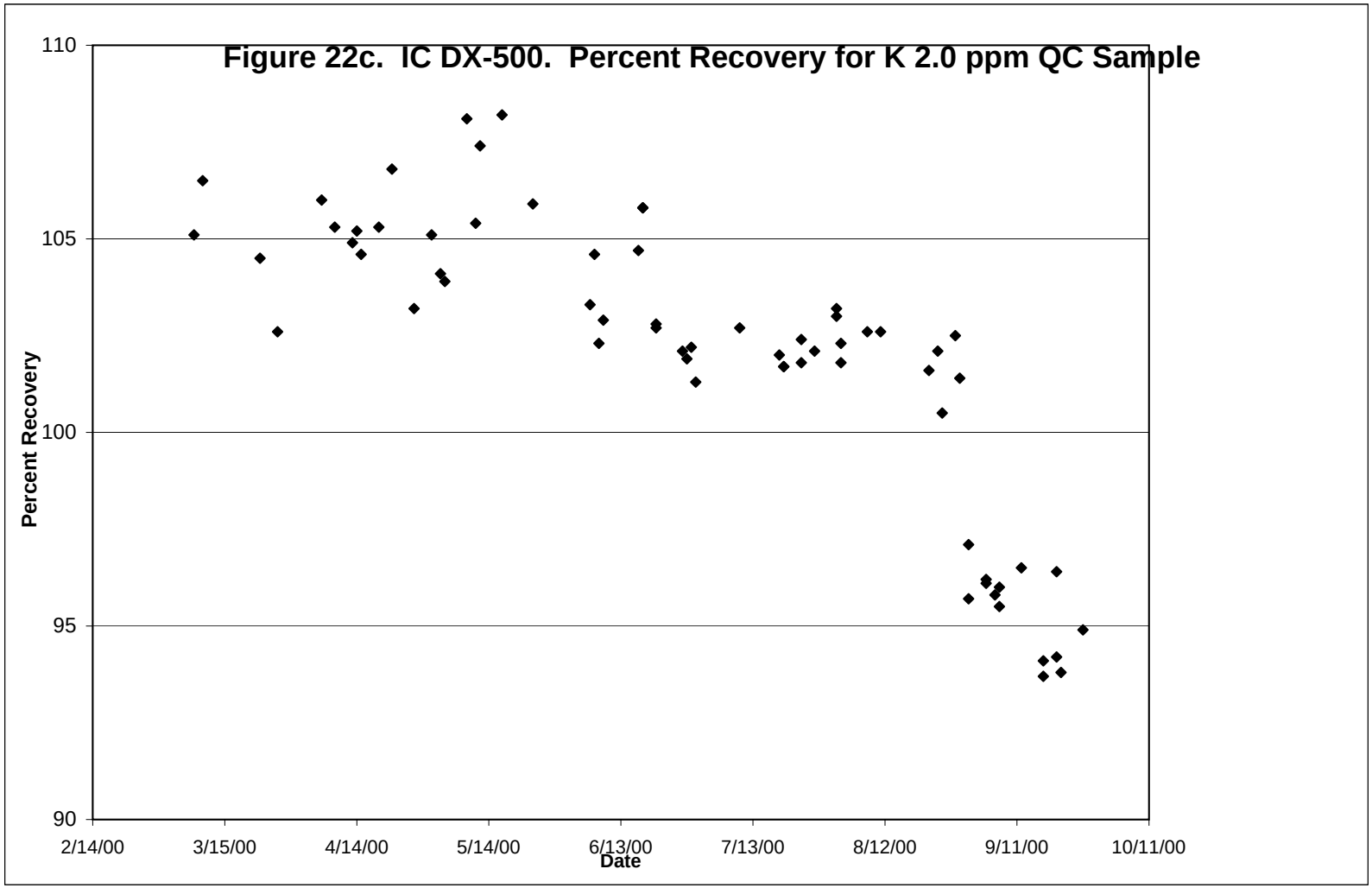


Figure 22c. IC DX-500. Percent Recovery for K 2.0 ppm QC Sample
110
105
100
95
90
Percent Recovery
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

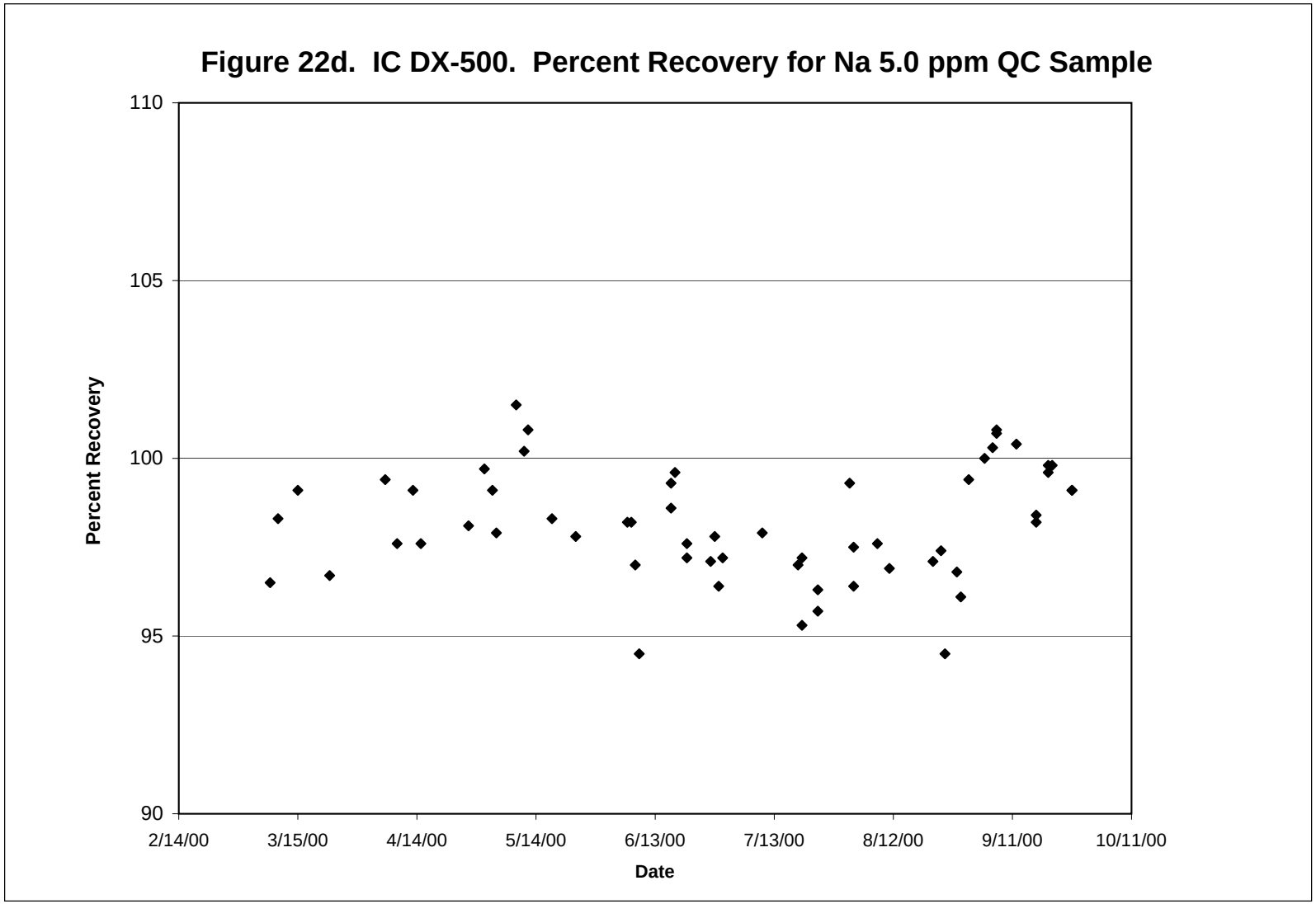
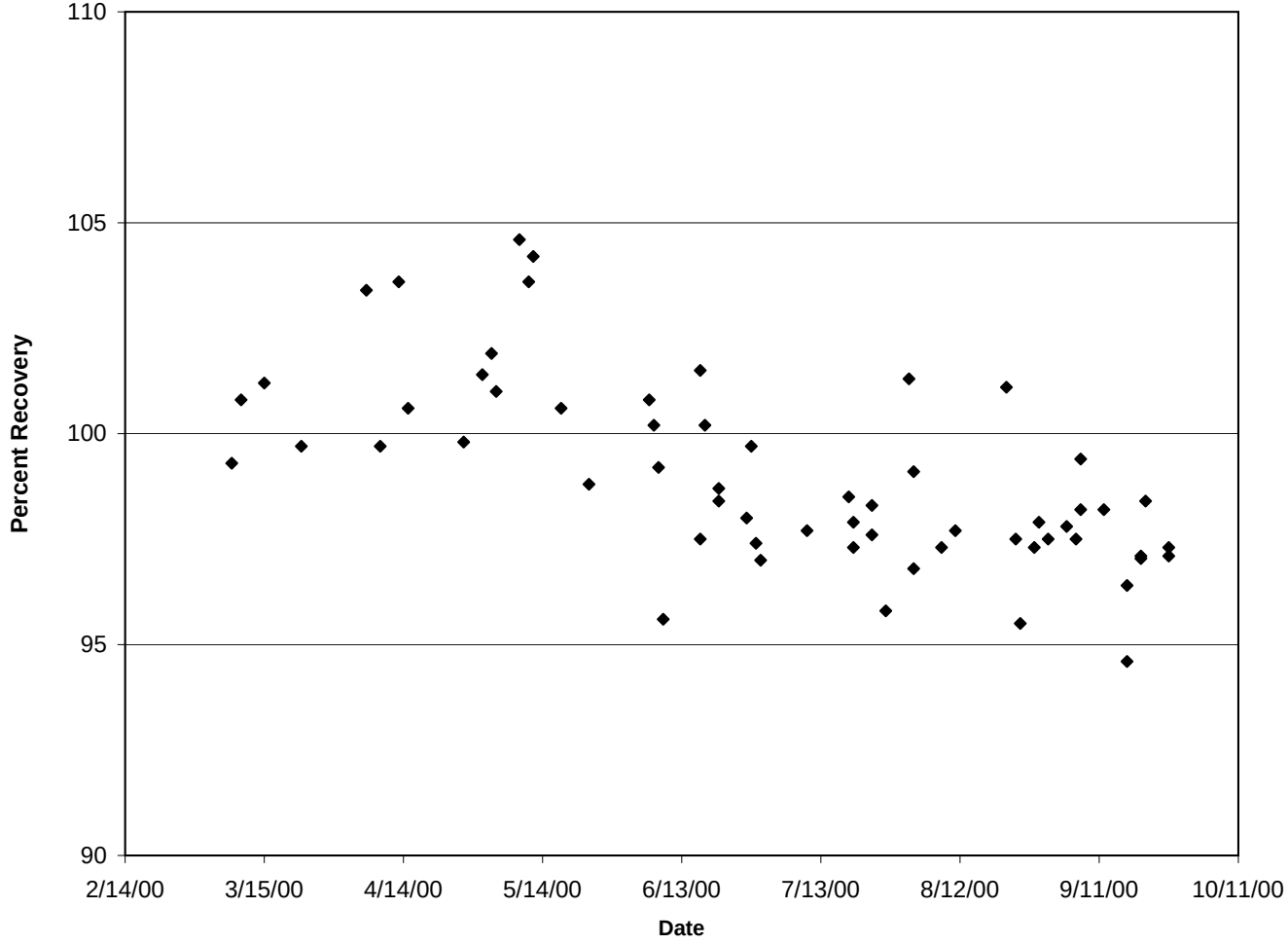


Figure 22d. IC DX-500. Percent Recovery for Na 5.0 ppm QC Sample
110
105
100
95
90
Percent Recovery
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

Figure 22e. IC DX-500. Percent Recovery for NH_4 5.0 ppm QC Sample



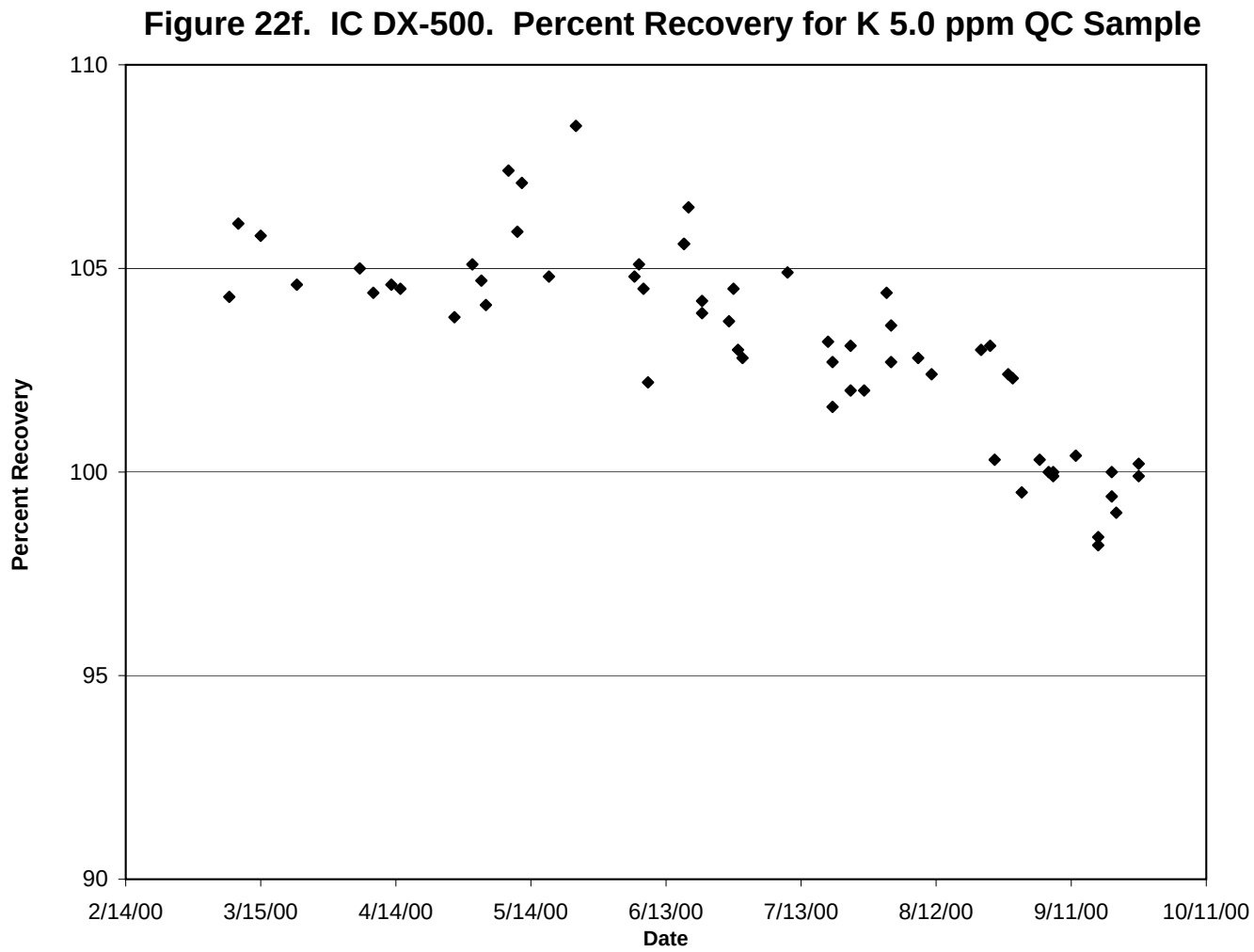


Figure 22f. IC DX-500. Percent Recovery for K 5.0 ppm QC Sample
110
105
100
95
90
Percent Recovery
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

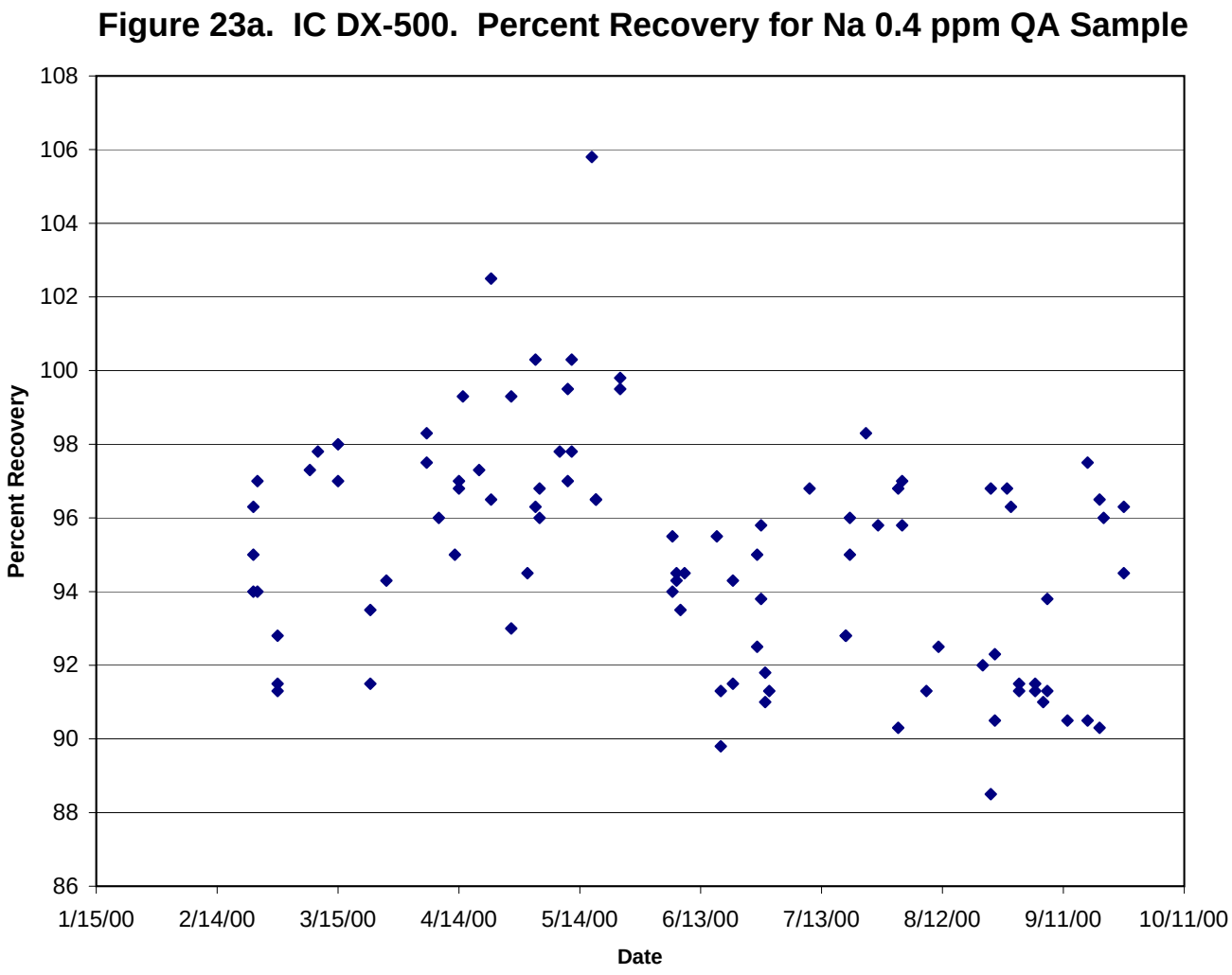


Figure 23a. IC DX-500. Percent Recovery for Na 0.4 ppm QA Sample
Percent Recovery
108
106
104
102
100
98
96
94
92
90
88
86
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

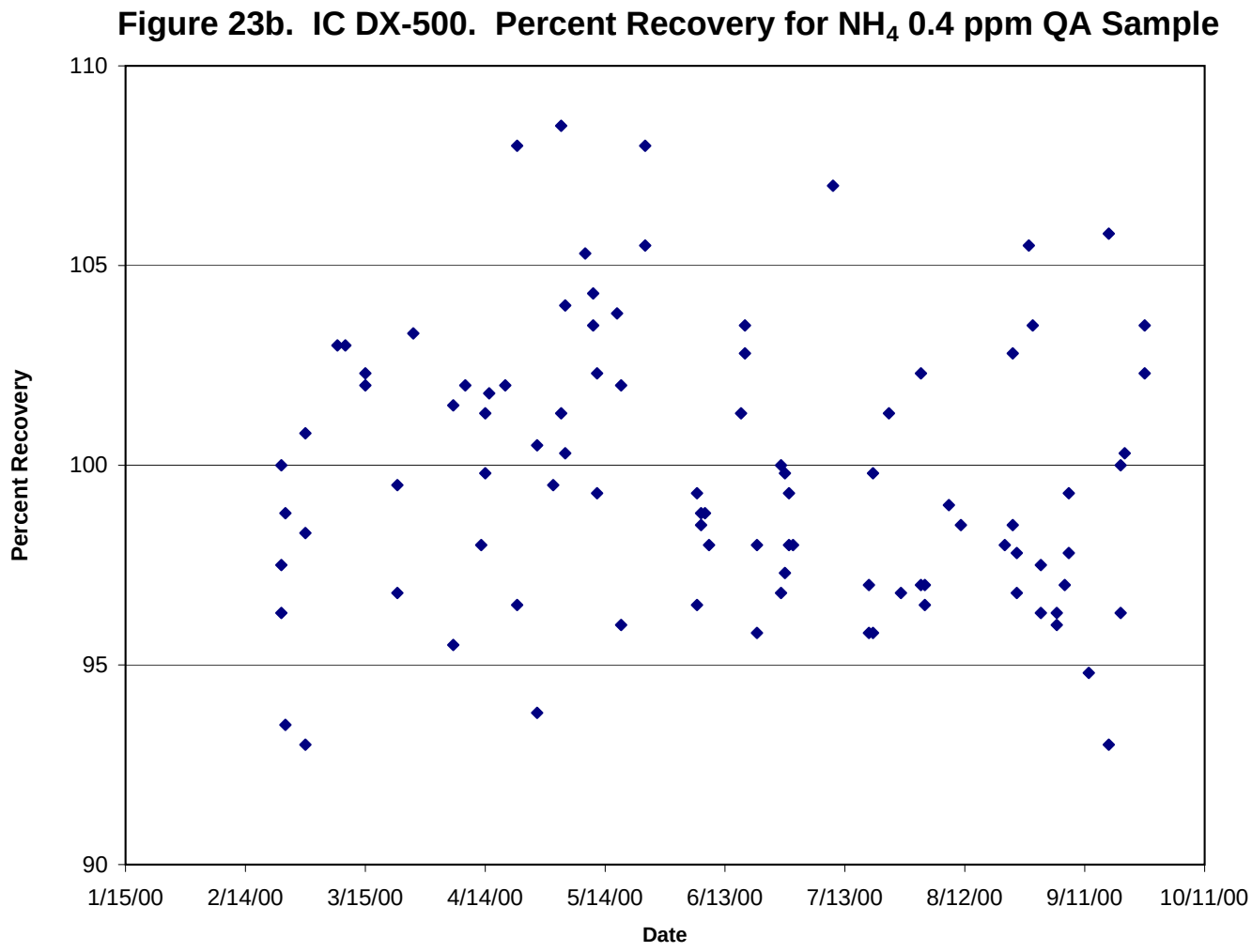


Figure 23b. IC DX-500. Percent Recovery for NH_4 0.4 ppm QA Sample
110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

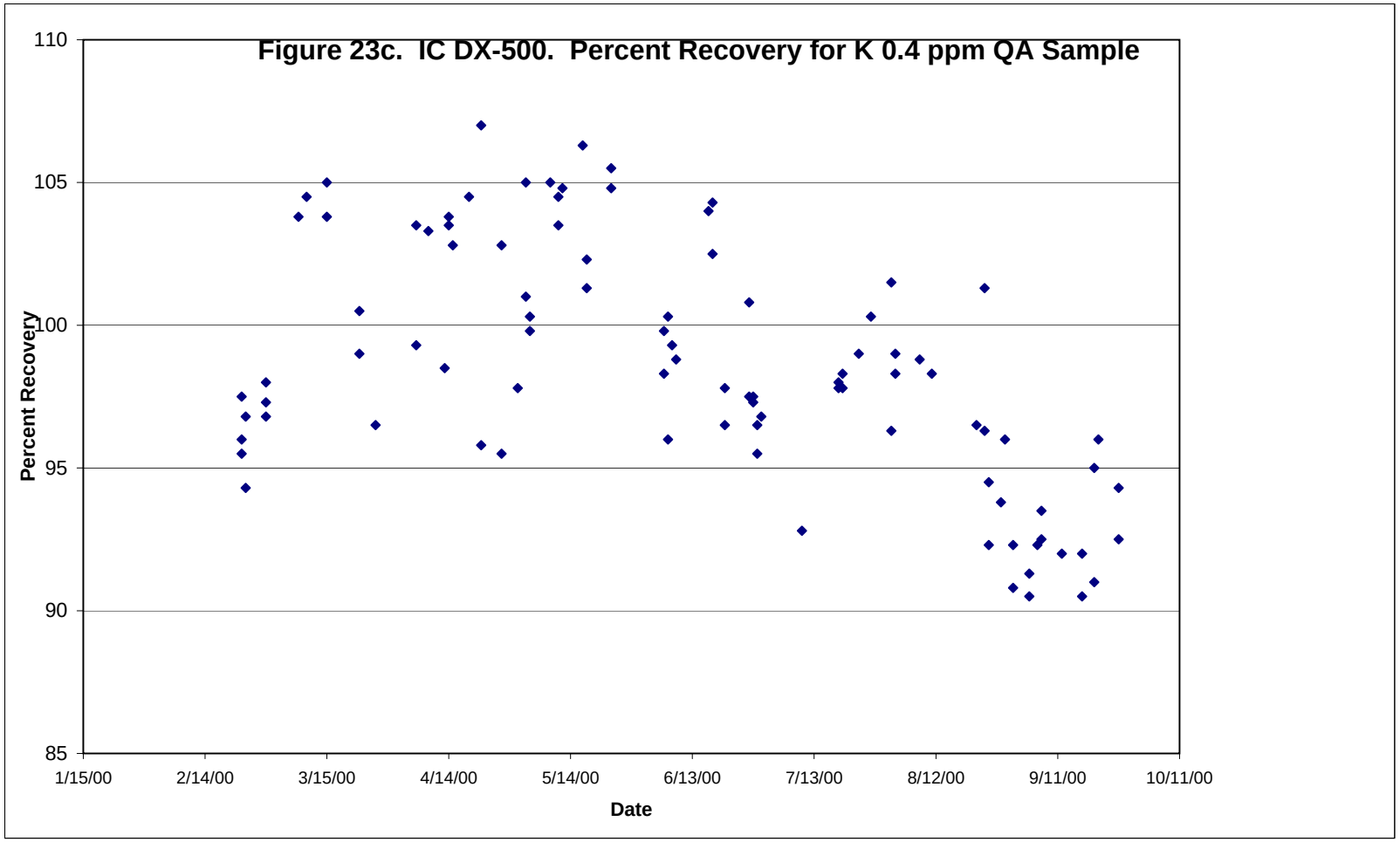


Figure 23c. IC DX-500. Percent Recovery for K 0.4 ppm QA Sample
Percent Recovery
110
105
100
95
90
85
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

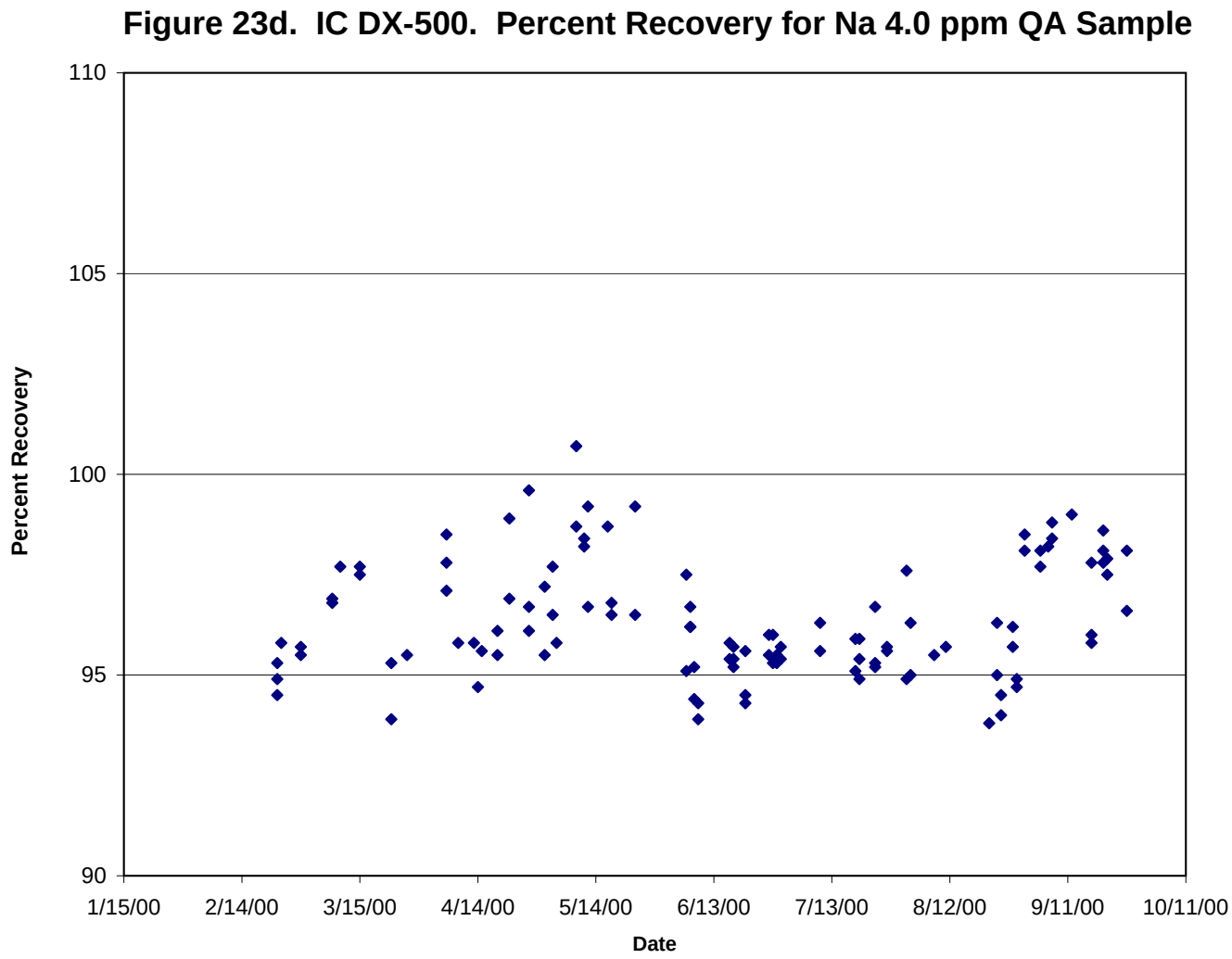


Figure 23d. IC DX-500. Percent Recovery for Na 4.0 ppm QA Sample
110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

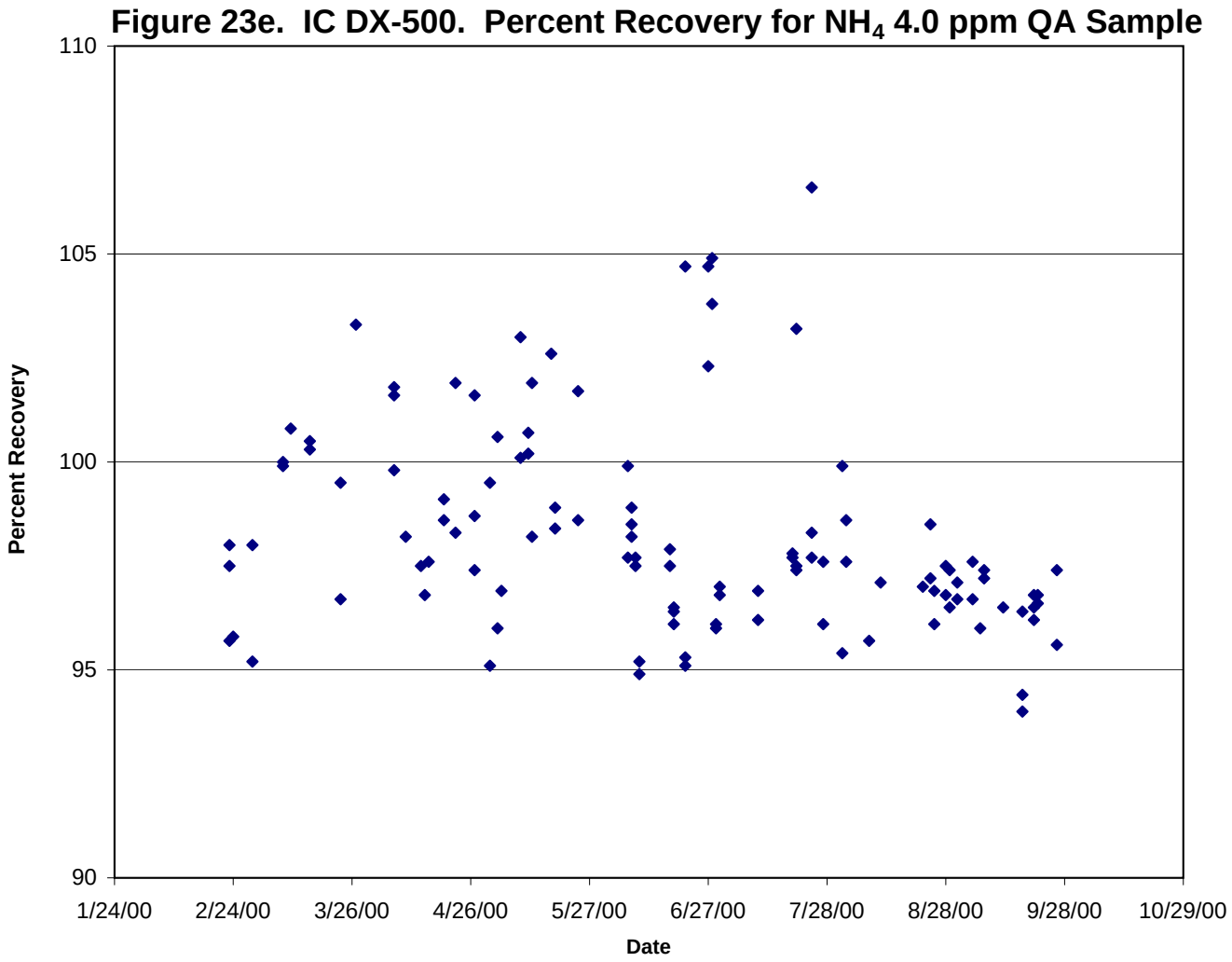


Figure 23e. IC DX-500. Percent Recovery for NH_4 4.0 ppm QA Sample
110
105
100
95
90
Percent Recovery
1/24/00
2/24/00
3/26/00
4/26/00
5/27/00
6/27/00
7/28/00
8/28/00
9/28/00
10/29/00
Date

Figure 23f. IC DX-500. Percent Recovery for K 4.0 ppm QA Sample

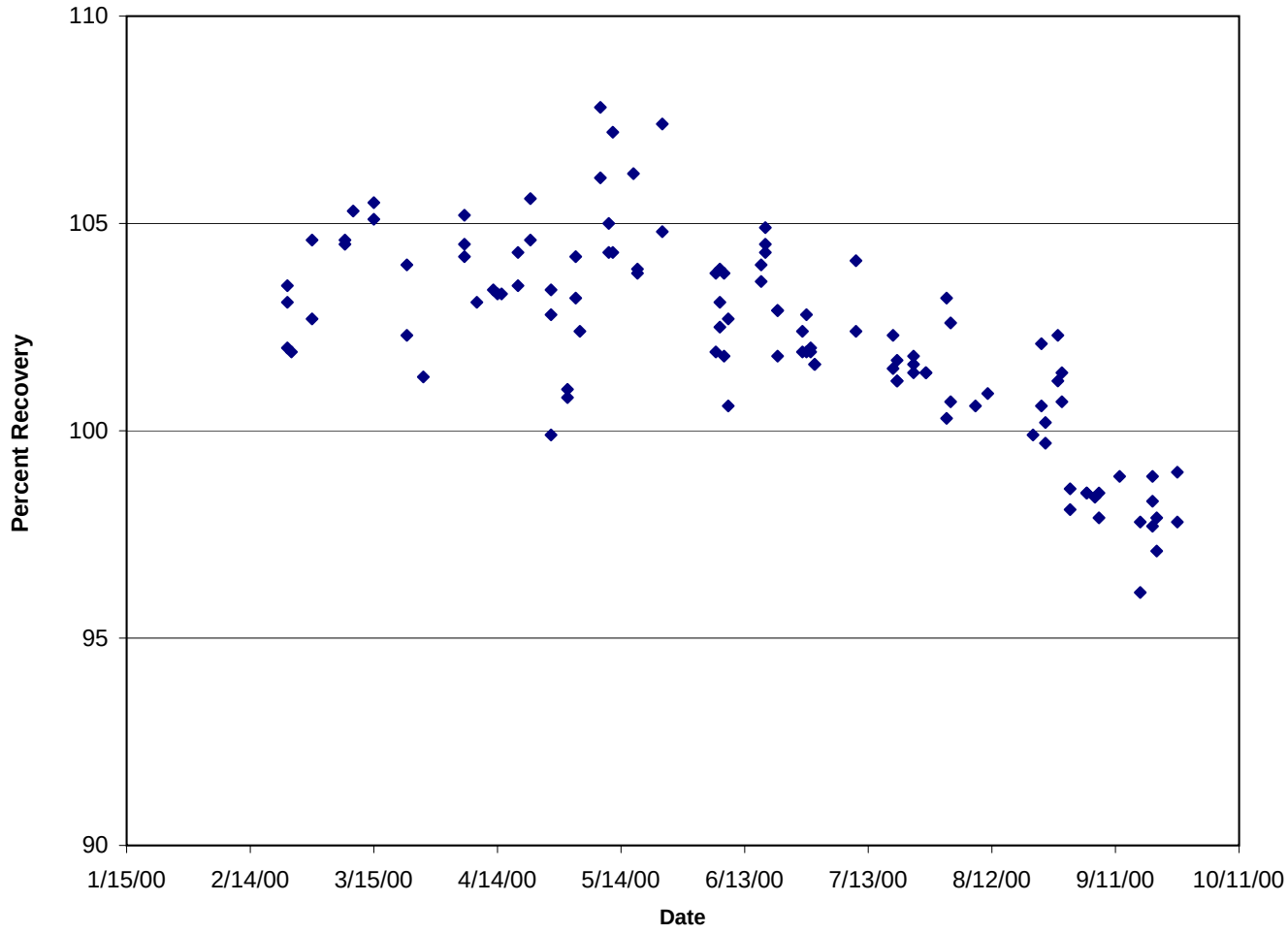


Figure 24a. IC DX-500. Relative Percent Difference (RPD) for Replicate Na Analyses

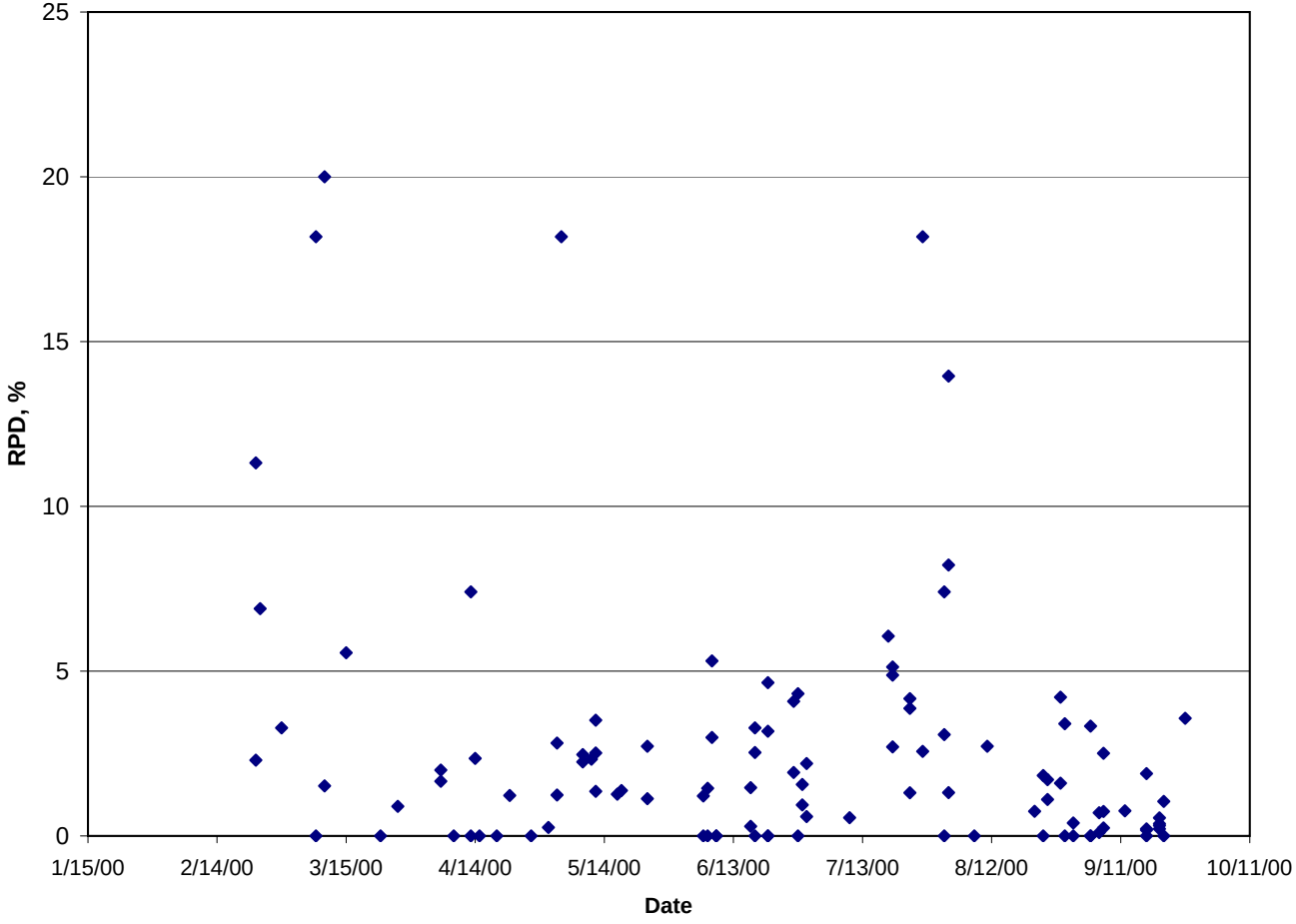


Figure 24b. IC DX-500. Relative Percent Difference (RPD) for Replicate NH_4 Analyses

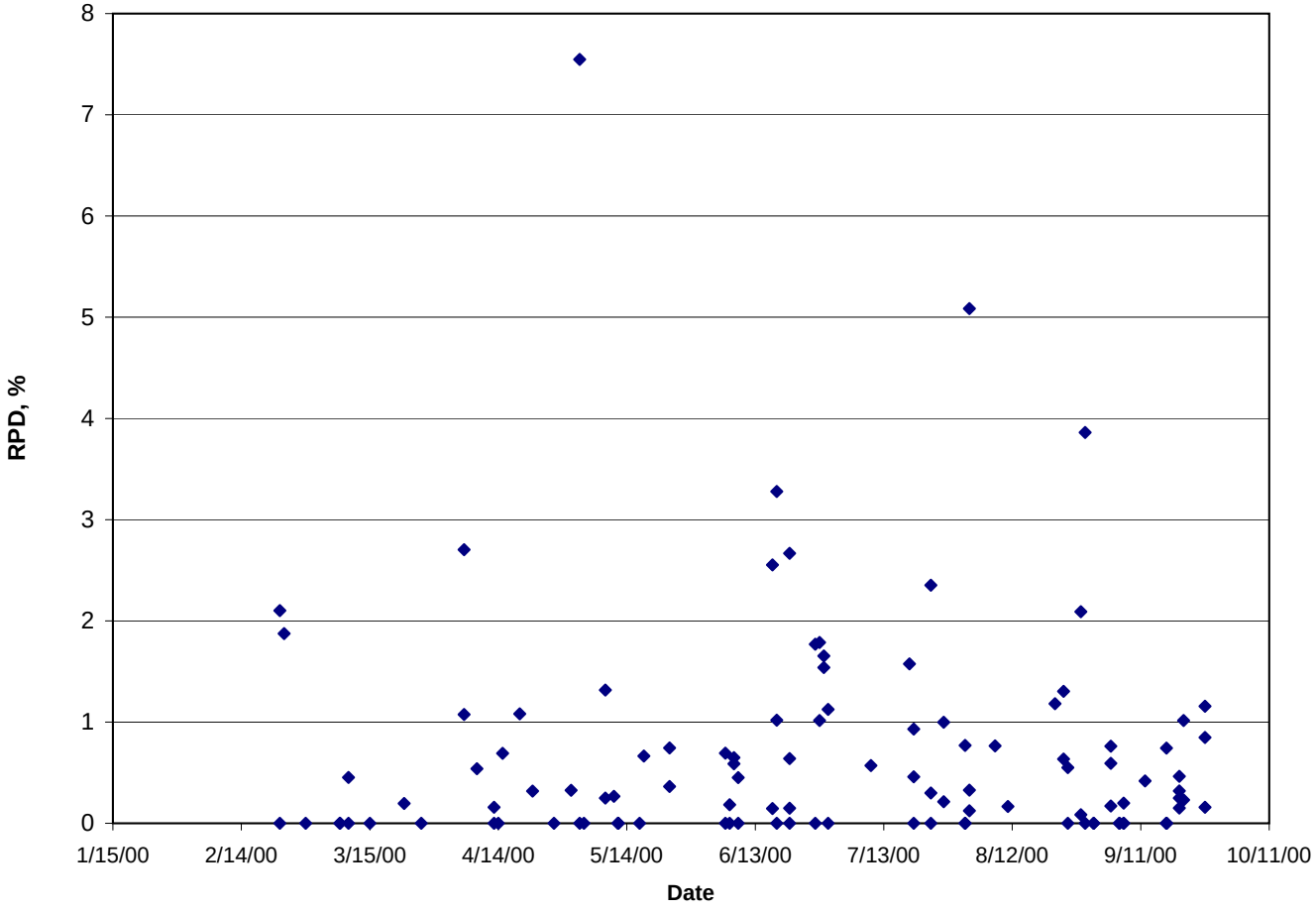
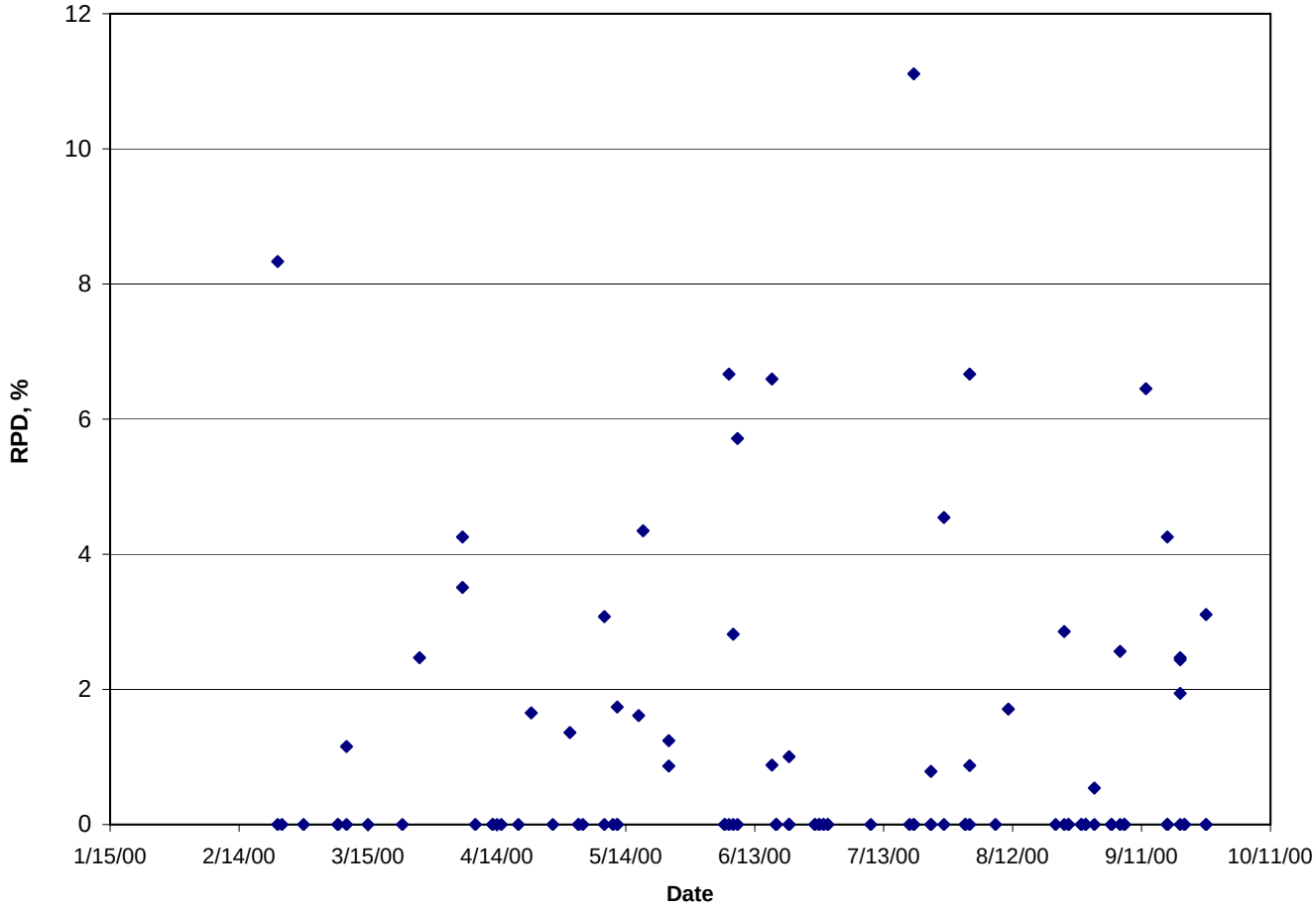


Figure 24c. IC DX-500. Relative Percent Difference (RPD) for Replicate K Analyses



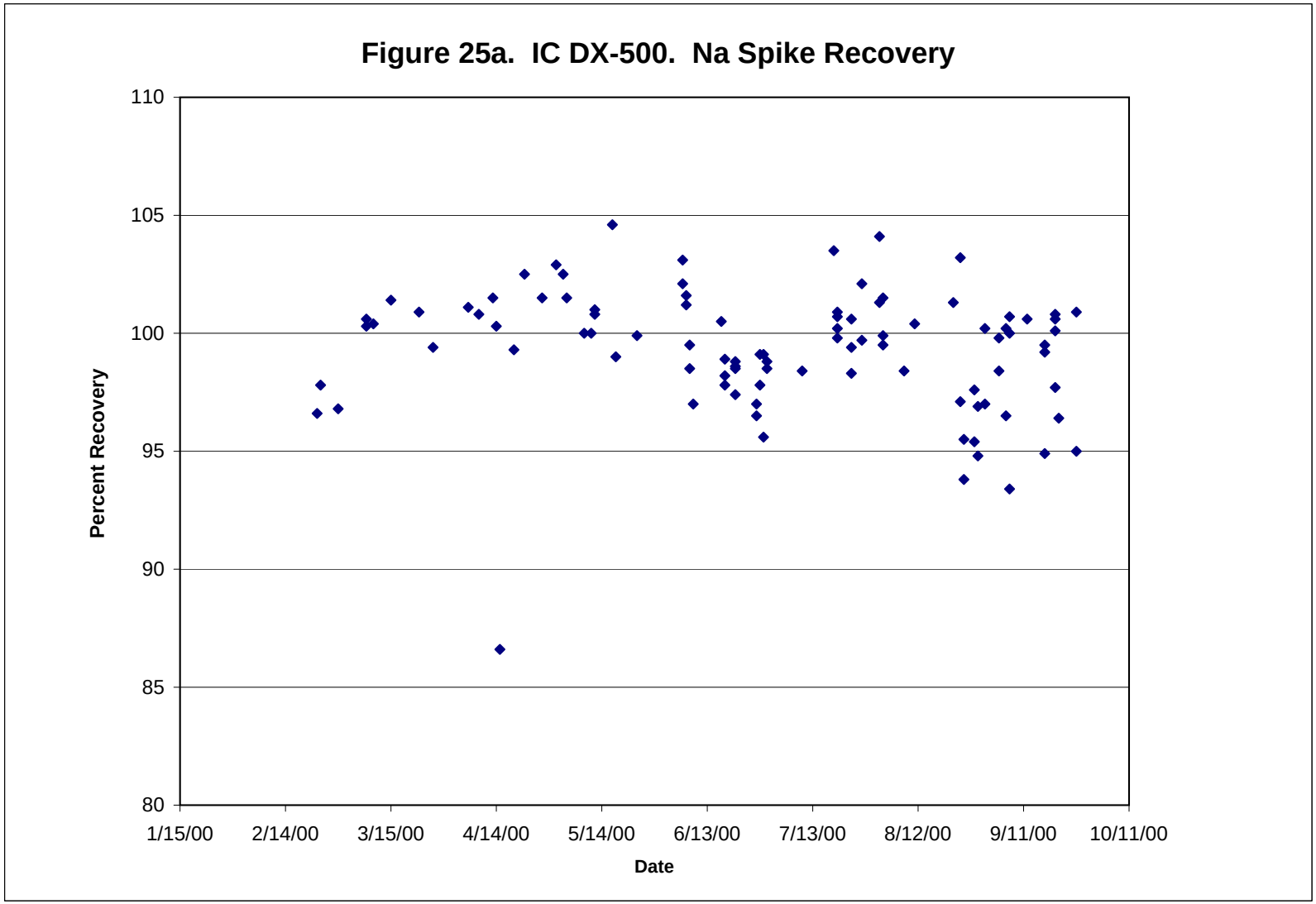


Figure 25a. IC DX-500. Na Spike Recovery
110
105
100
95
90
85
80
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

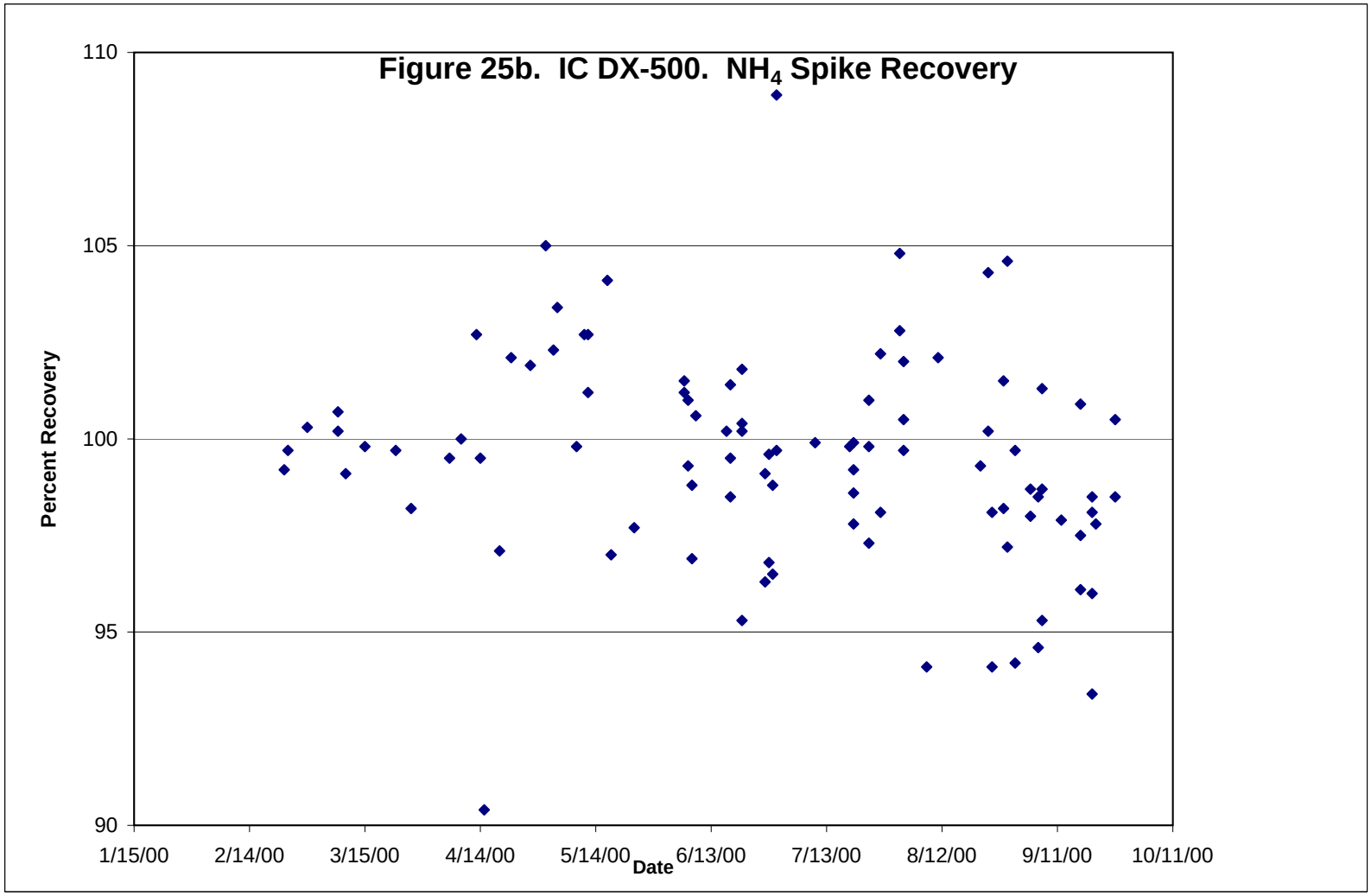


Figure 25b. IC DX-500. NH_4 Spike Recovery
110
105
100
95
90
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

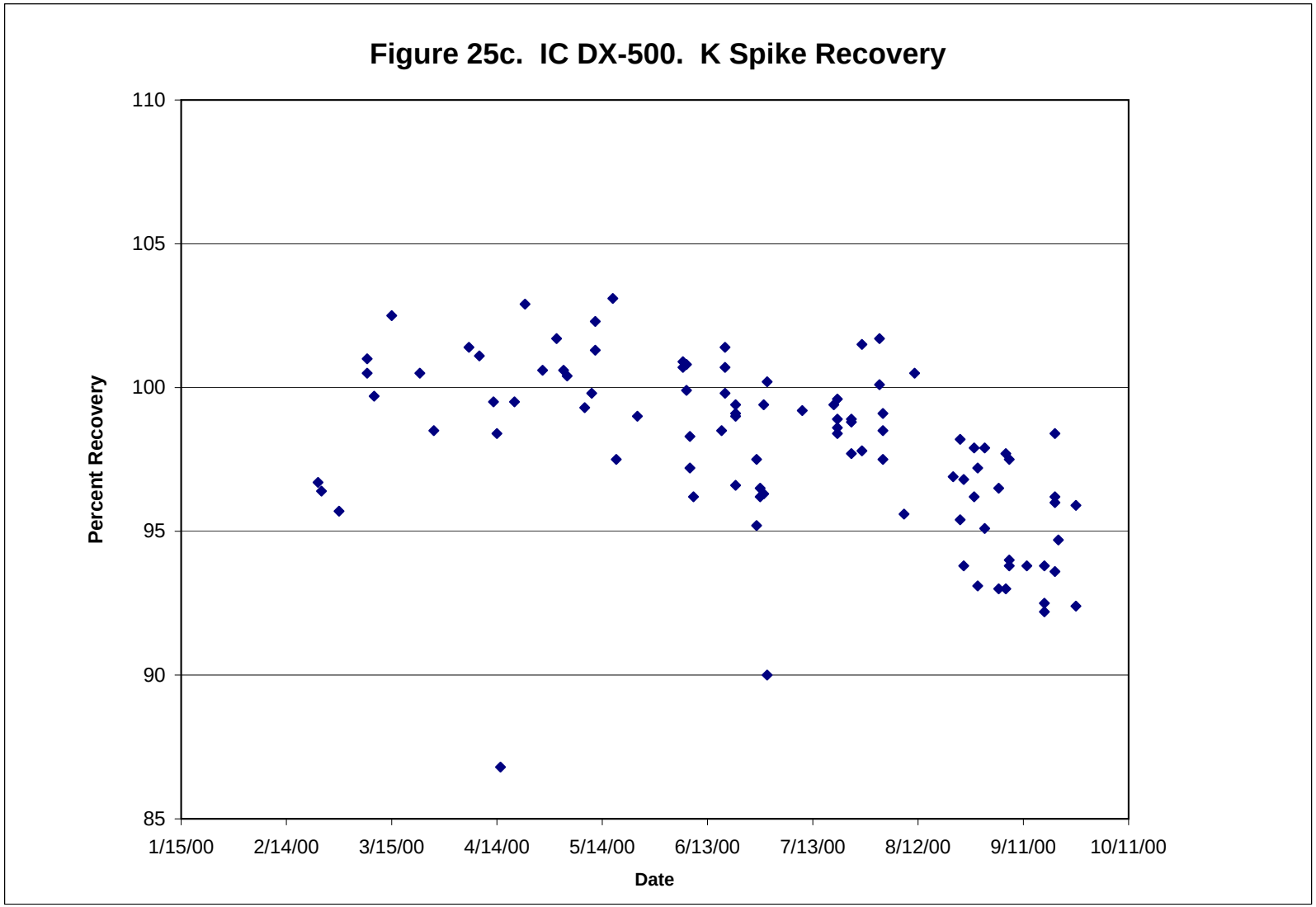


Figure 25c. IC DX-500. K Spike Recovery
110
105
100
95
90
85
Percent Recovery
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

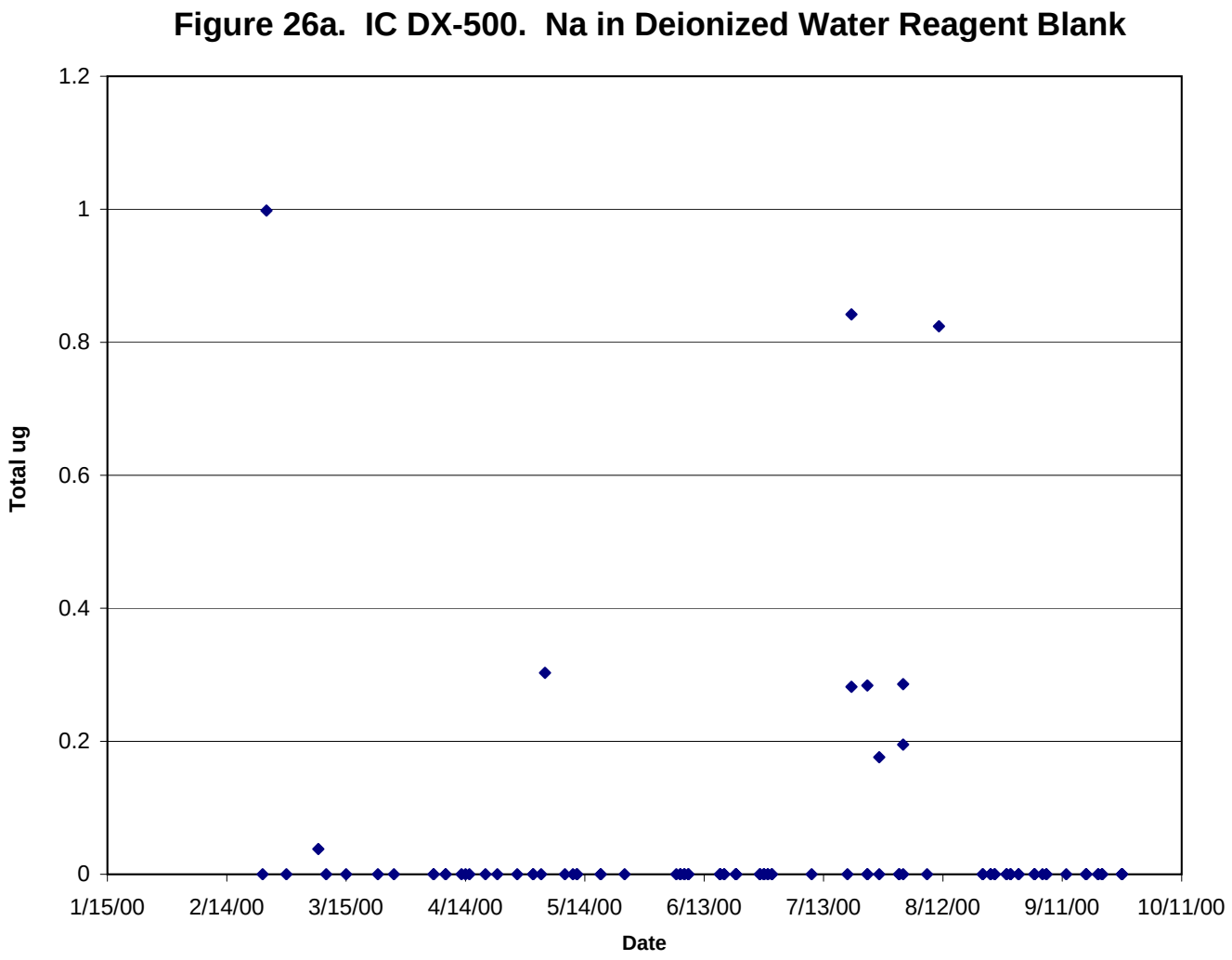
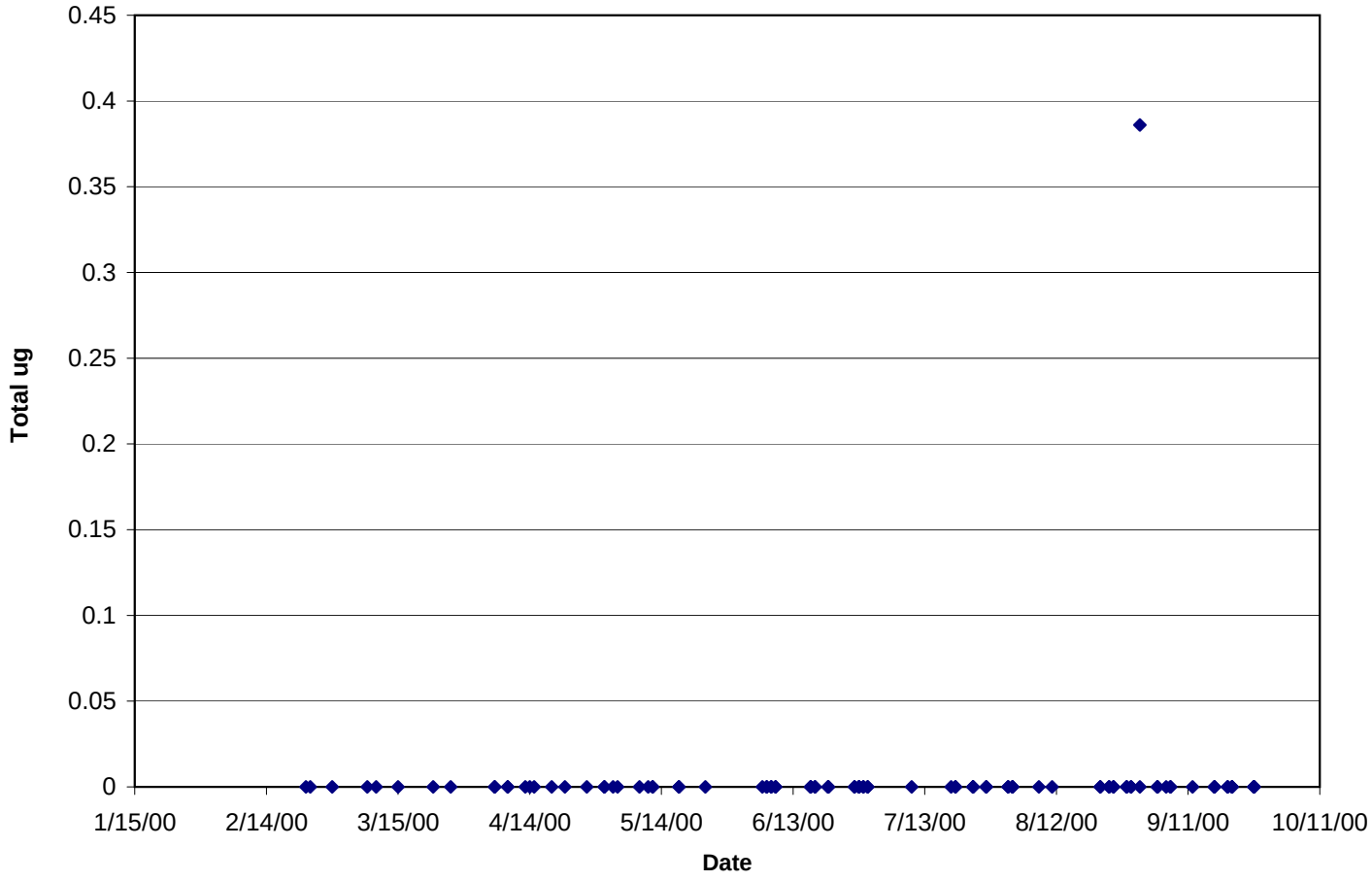


Figure 26a. IC DX-500. Na in Deionized Water Reagent Blank
1.2
1
0.8
0.6
0.4
0.2
0
Total ug
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

Figure 26b. IC DX-500. NH_4 in Deionized Water Reagent Blank



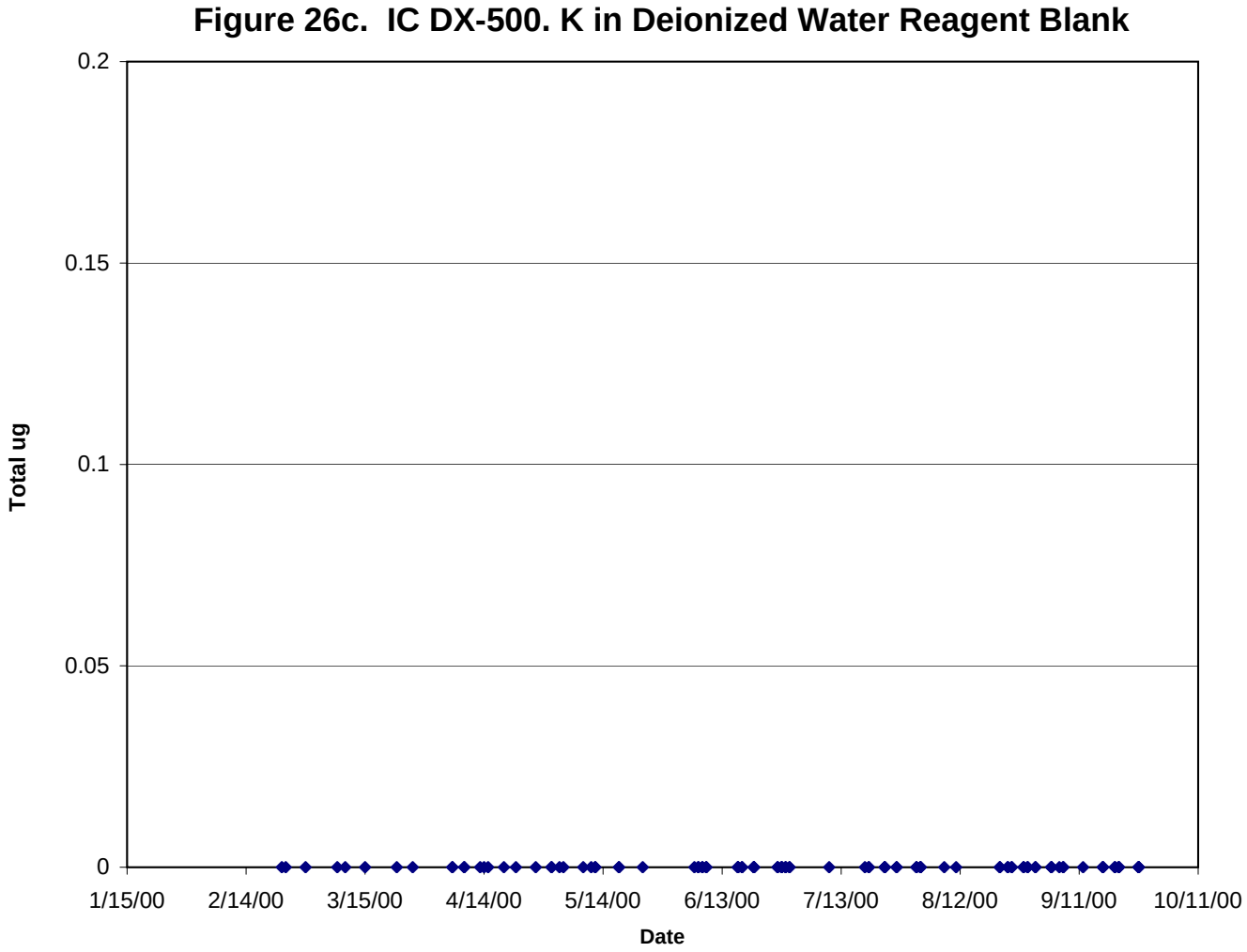


Figure 26c. IC DX-500. K in Deionized Water Reagent Blank
0.2
0.15
0.1
0.05
0
Total ug
1/15/00
2/14/00
3/15/00
4/14/00
5/14/00
6/13/00
7/13/00
8/12/00
9/11/00
10/11/00
Date

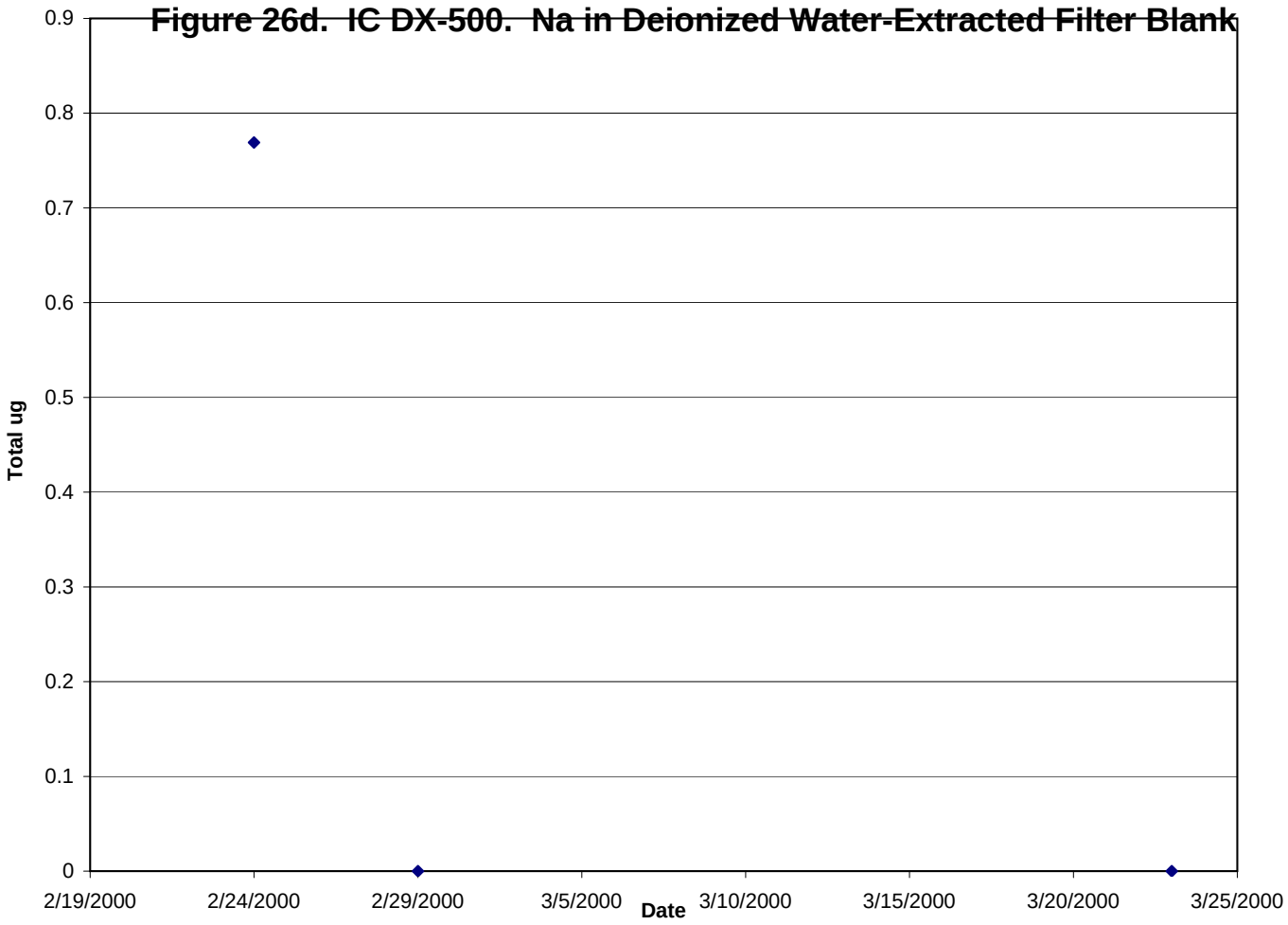


Figure 26d. IC DX-500. Na in Deionized Water-Extracted Filter Blank
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
Total ug
2/19/2000
2/24/2000
2/29/2000
3/5/2000
3/10/2000
3/15/2000
3/20/2000
3/25/2000
Date

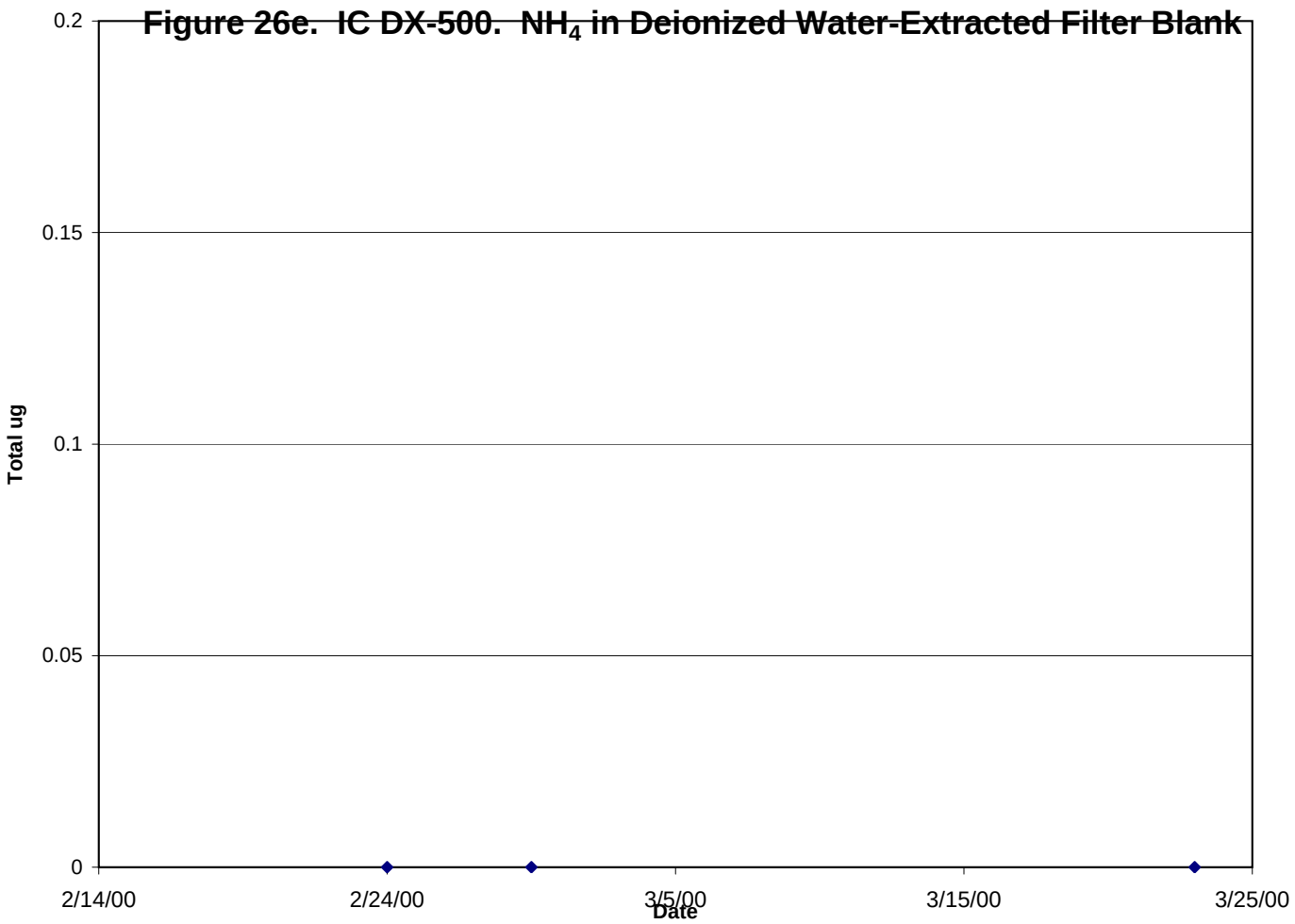
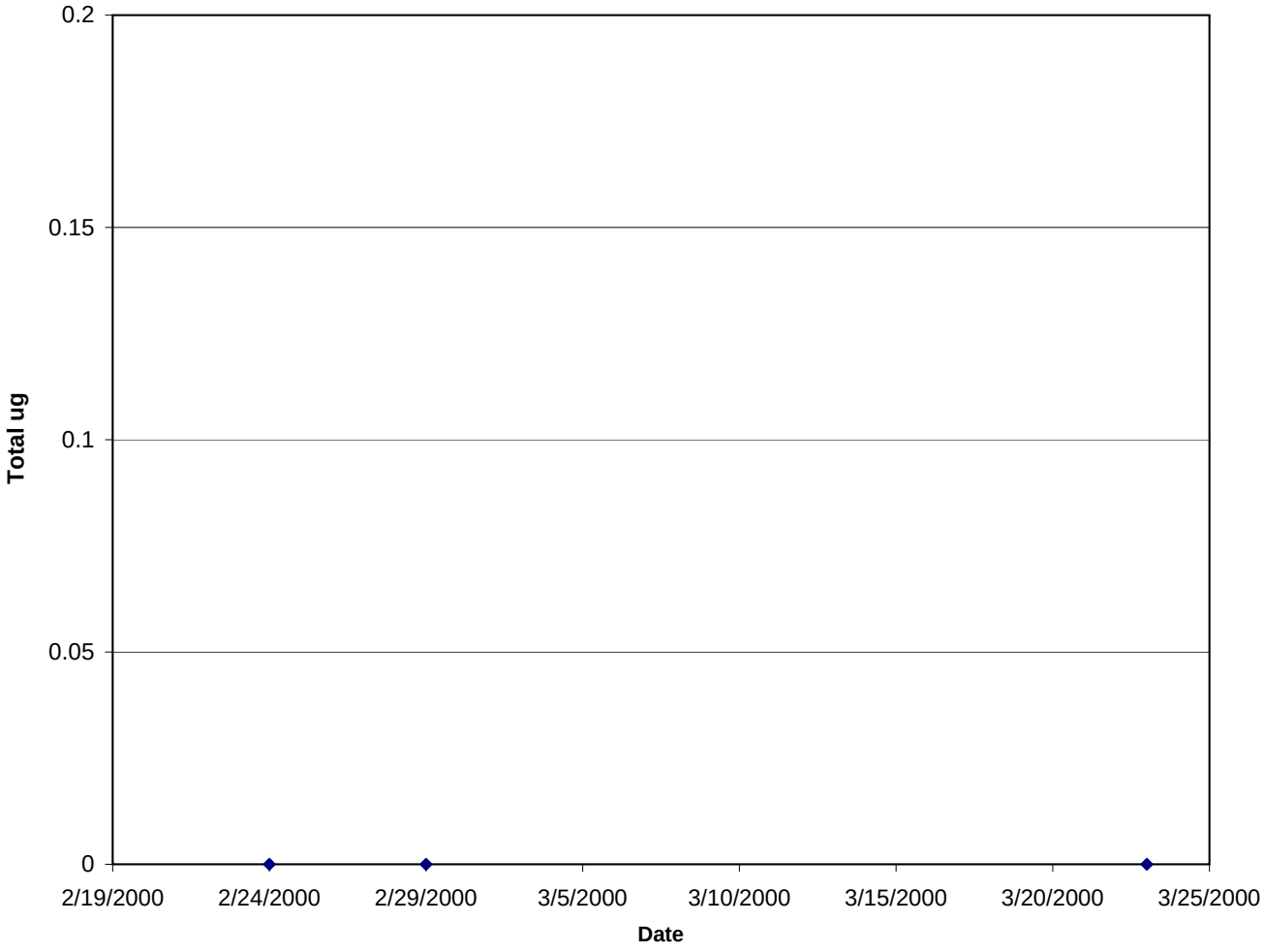


Figure 26e. IC DX-500. NH_4 in Deionized Water-Extracted Filter Blank
0.2
0.15
0.1
0.05
0
Total ug
2/14/00
2/24/00
3/5/00
3/15/00
3/25/00
Date

Figure 26f. IC DX-500. K in Deionized Water-Extracted Filter Blank



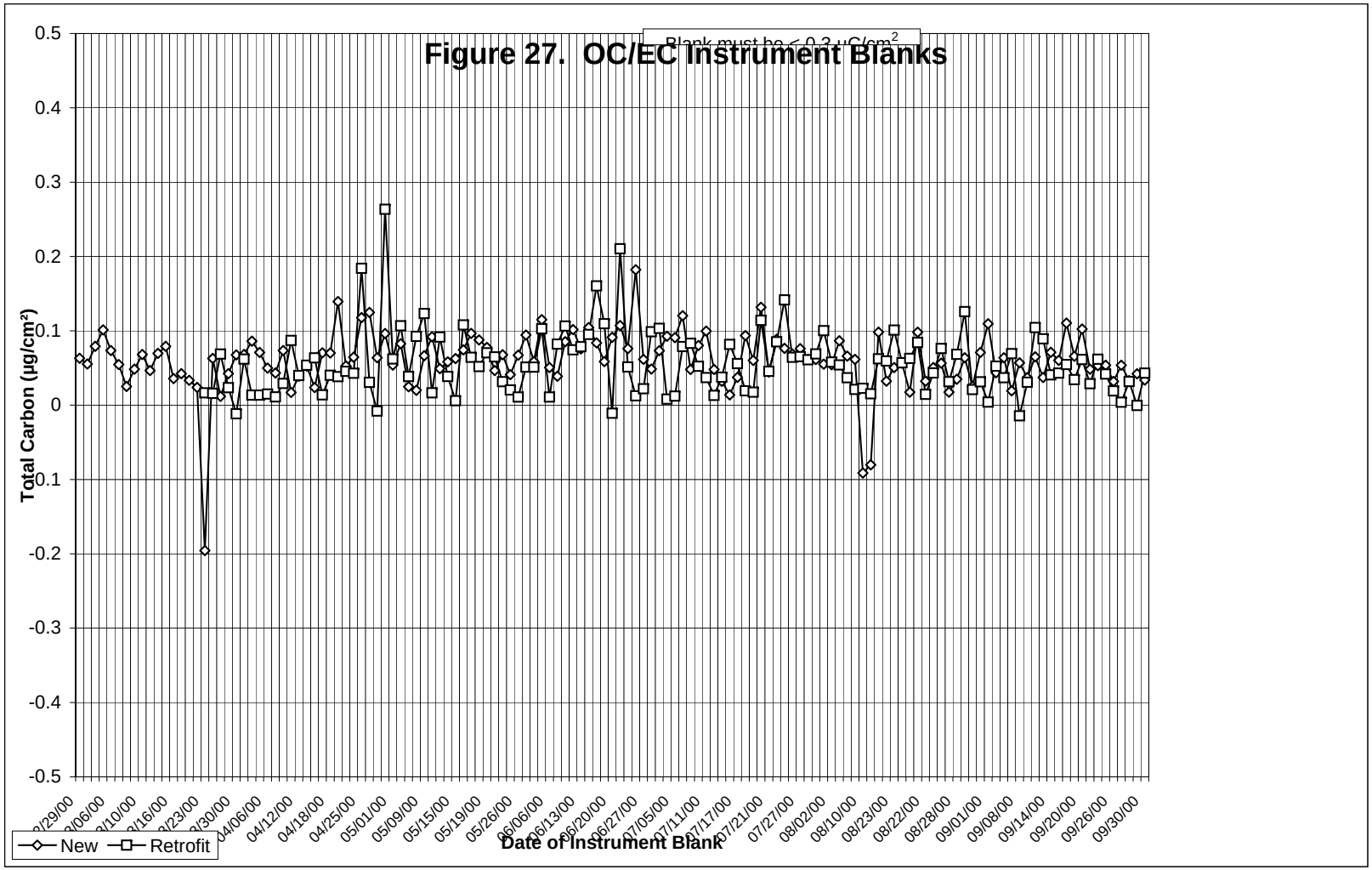


Figure 27. OC/EC Instrument Blanks
Blank must be < 0.3 uC/cm2
Total Carbon (µg/cm²)
0.5
0.4
0.3
0.2
0.1
0
-0.1
-0.2
-0.3
-0.4
-0.5
Date of Instrument Blank
New
Retrofit

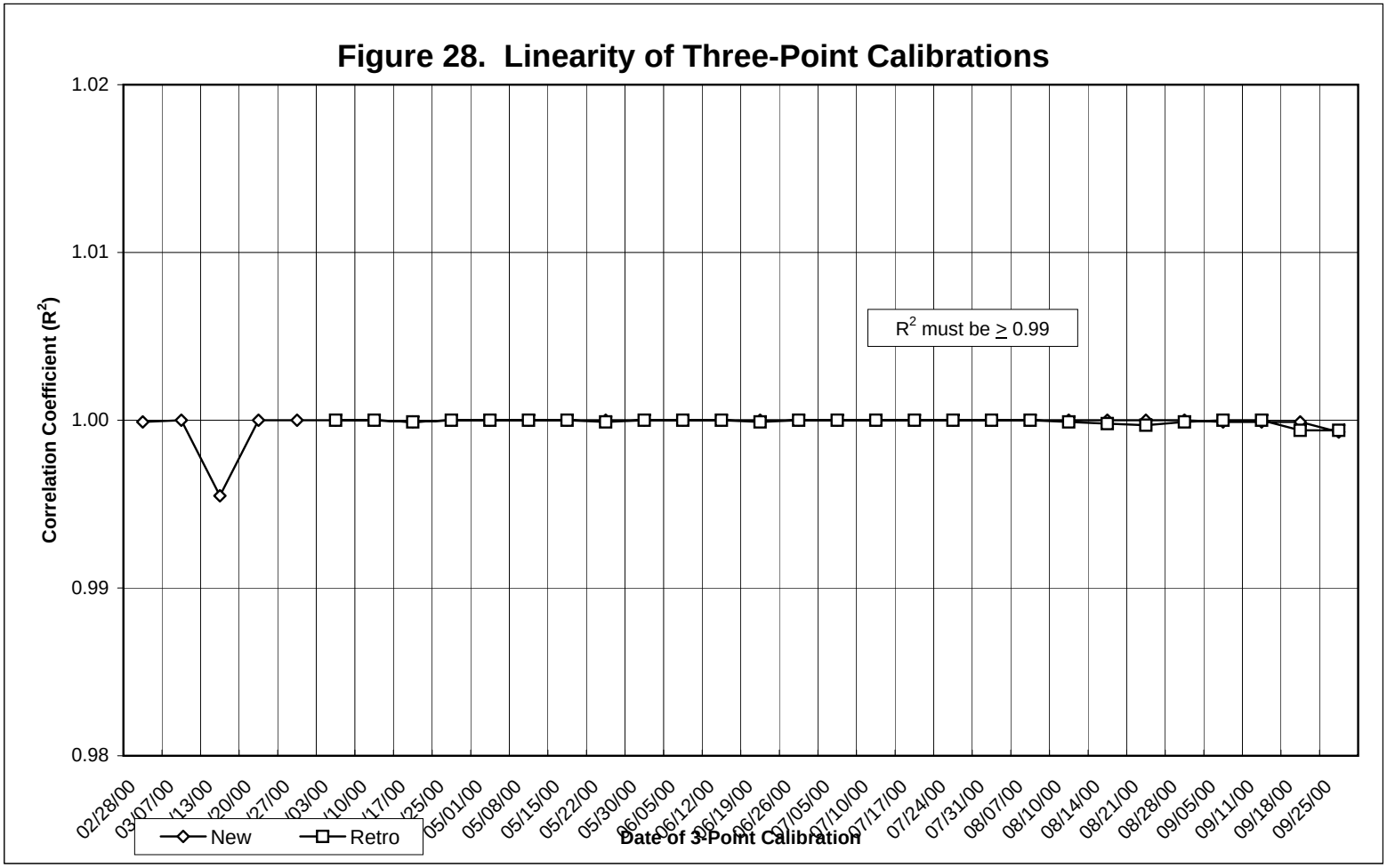


Figure 28. Linearity of Three-Point Calibrations
1.02
1.01
1.00
0.99
0.98
Correlation Coefficient (R^2)
R^2 must be ≥ 0.99
02/28/00
03/07/00
05/01/00
05/08/00
05/15/00
05/22/00
05/30/00
06/05/00
06/12/00
06/19/00
06/26/00
07/05/00
07/10/00
07/17/00
07/24/00
07/31/00
08/07/00
08/10/00
08/14/00
08/21/00
08/28/00
09/05/00
09/11/00
09/18/00
09/25/00
New
Retro
Date of 3-Point Calibration

Figure 29. Percent Recoveries for Three-Point Calibration Standards on the New OC/EC Analyzer

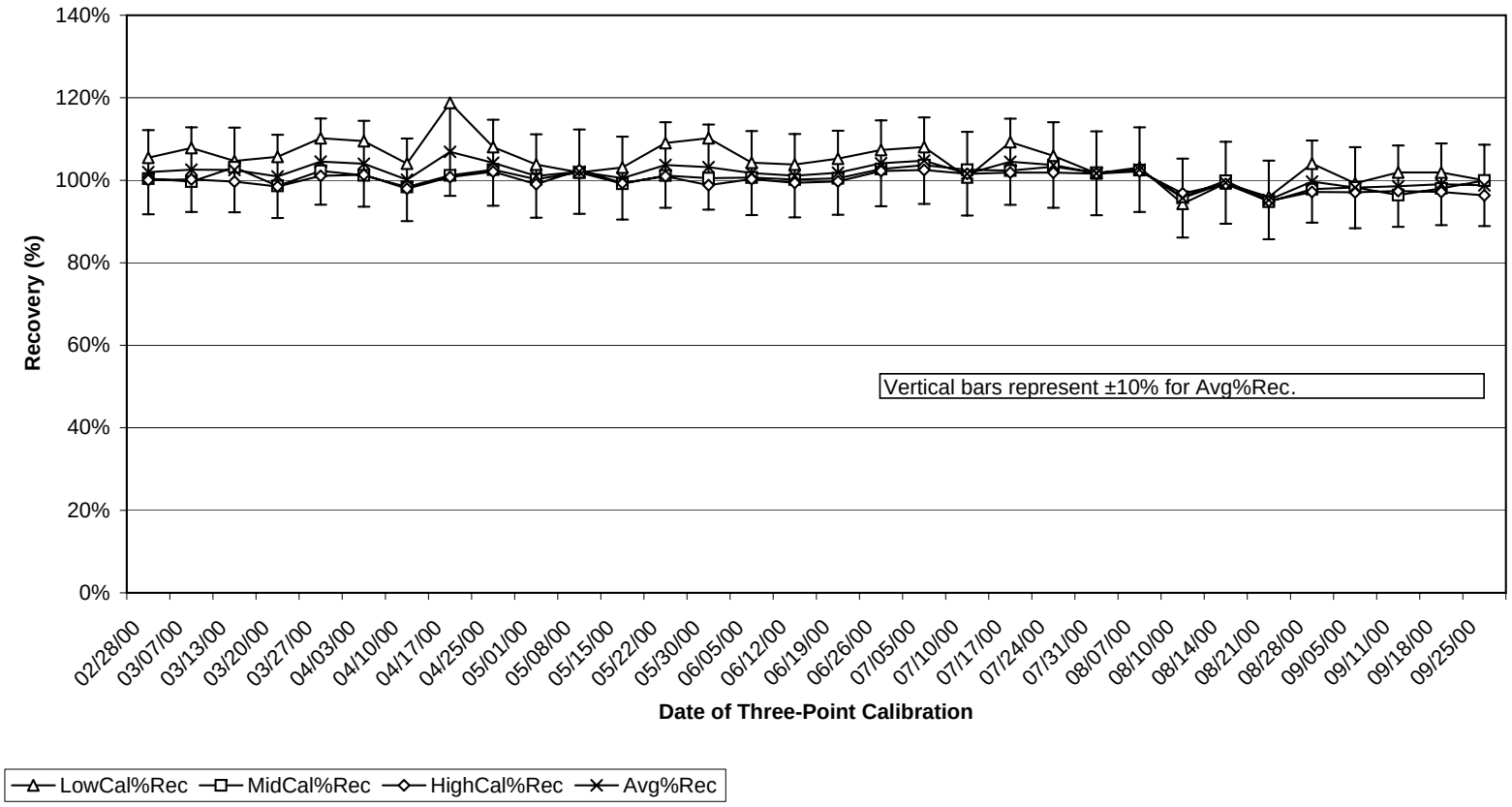
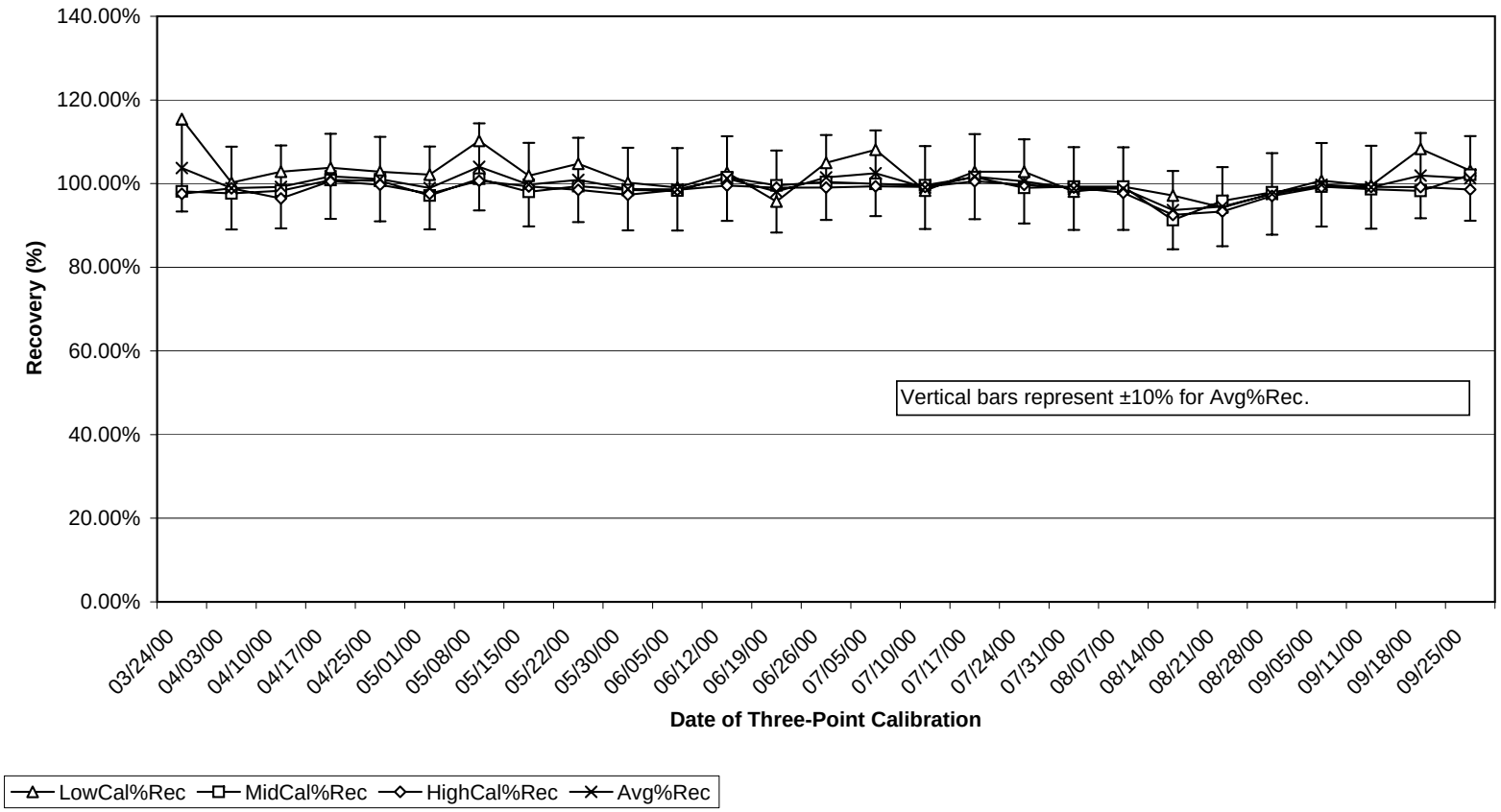


Figure 30. Percent Recoveries for Three-Point Calibration Standards on the Retrofit OC/EC Analyzer



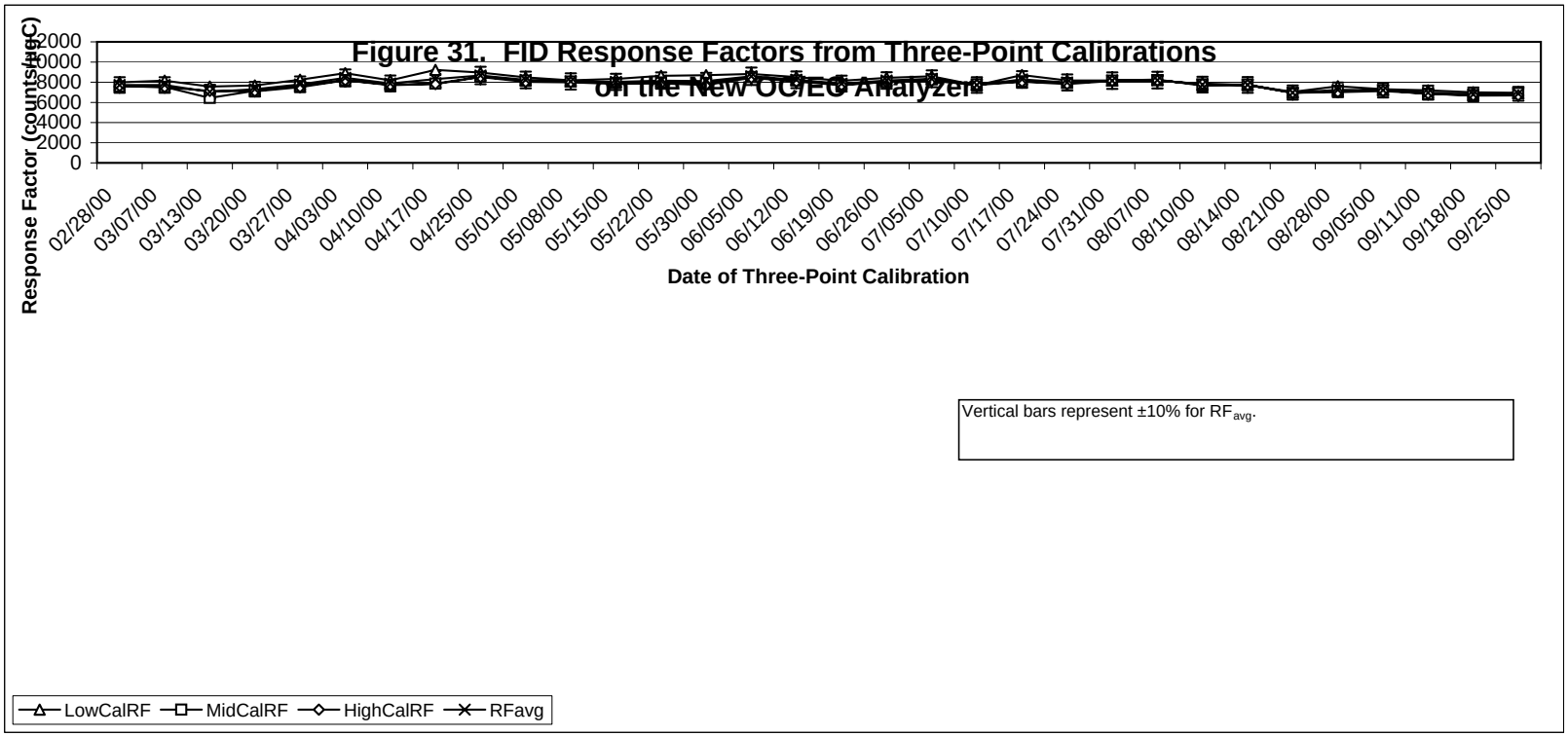


Figure 31. FID Response Factors from Three-Point Calibrations
on the New OC/EC Analyzer
Response Factor (counts/µgC)
12000
10000
8000
6000
4000
2000
0
02/28/00
03/07/00
03/13/00
03/20/00
03/27/00
04/03/00
04/10/00
04/17/00
04/25/00
05/01/00
05/08/00
05/15/00
05/22/00
05/30/00
06/05/00
06/12/00
06/19/00
06/26/00
07/05/00
07/10/00
07/17/00
07/24/00
07/31/00
08/07/00
08/10/00
08/14/00
08/21/00
08/28/00
09/05/00
09/11/00
09/18/00
09/25/00
Date of Three-Point Calibration
Vertical bars represent ±10% for RFavg.
LowCalRF
MidCalRF
HighCalRF
RFavg

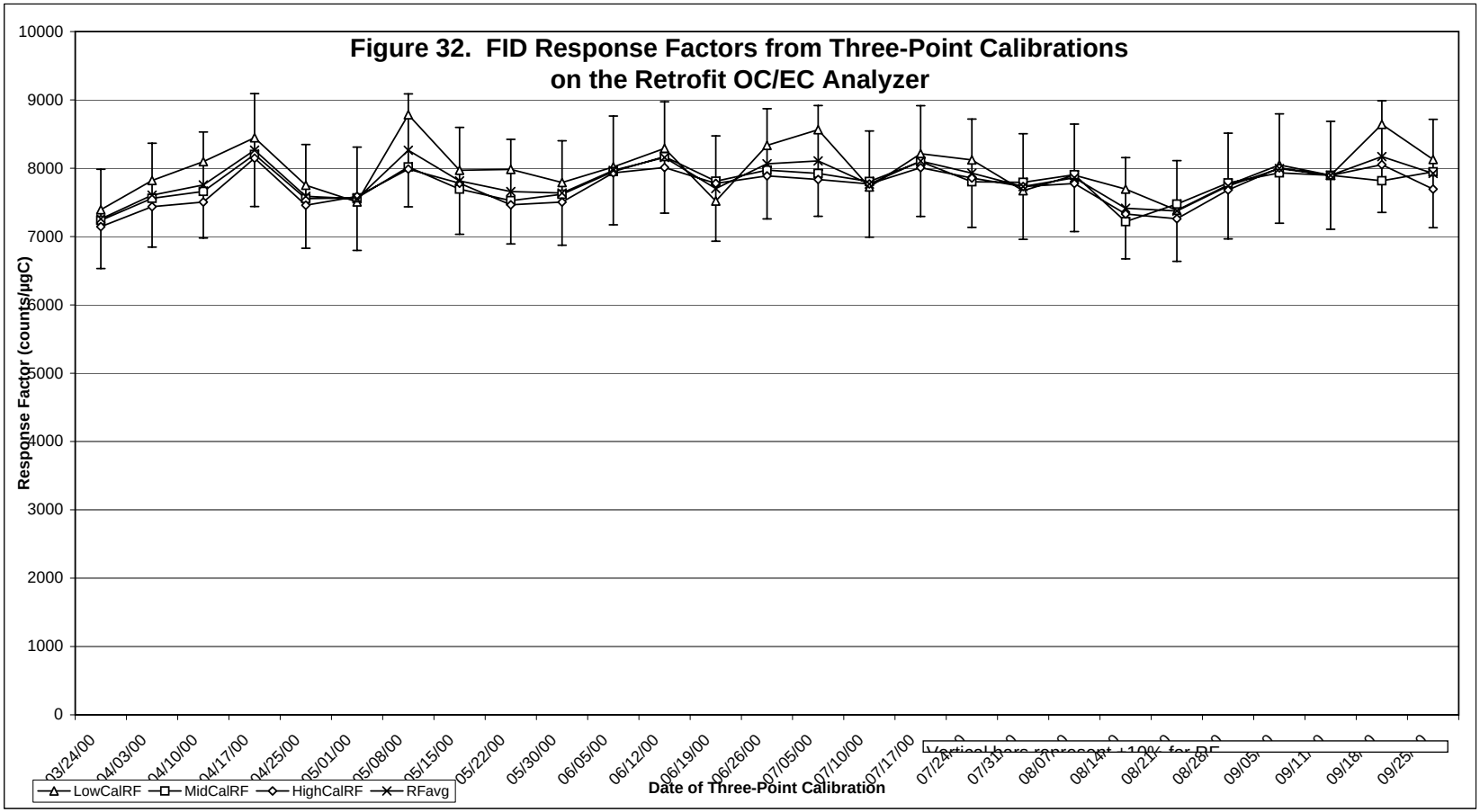


Figure 32. FID Response Factors from Three-Point Calibrations
on the Retrofit OC/EC Analyzer
Response Factor (counts/µgC)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
03/24/00
04/03/00
04/10/00
04/17/00
04/25/00
05/01/00
05/08/00
05/15/00
05/22/00
05/30/00
06/05/00
06/12/00
06/19/00
06/26/00
07/05/00
07/10/00
07/17/00
07/24/00
07/31/00
08/07/00
08/14/00
08/21/00
08/28/00
09/05/00
09/11/00
09/18/00
09/25/00
Date of Three-Point Calibration
LowCalRF
MidCalRF
HighCalRF
RFavg

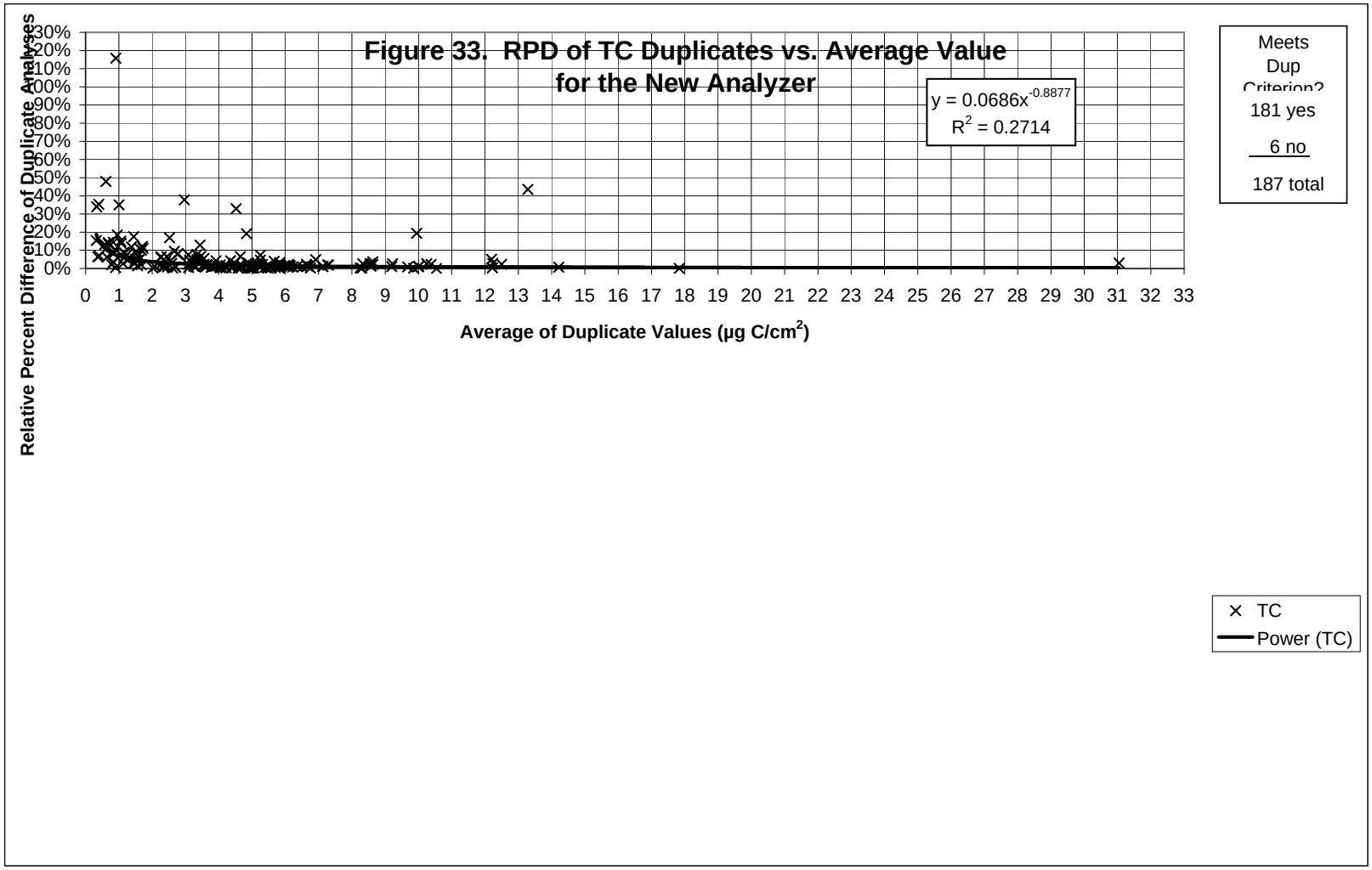
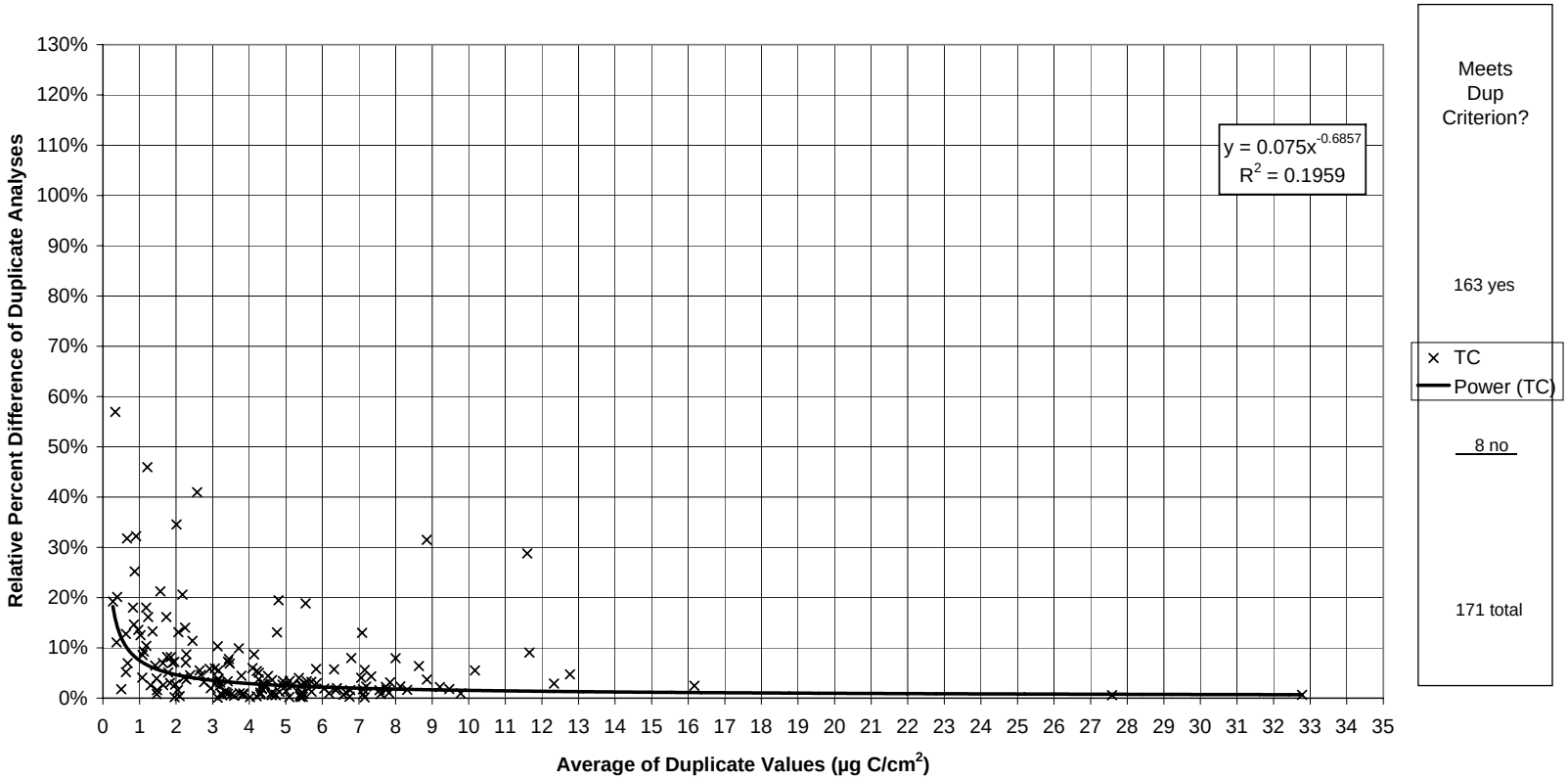


Figure 33. RPD of TC Duplicates vs. Average Value for the New Analyzer
y = 0.0686x^-0.8877
R^2 = 0.2714
Meets Dup Criterion?
181 yes
6 no
187 total
Relative Percent Difference of Duplicate Analyses
Average of Duplicate Values (µg C/cm^2)
TC
Power (TC)

Figure 34. RPD of TC Duplicates vs. Average Value for the Retrofit Analyzer



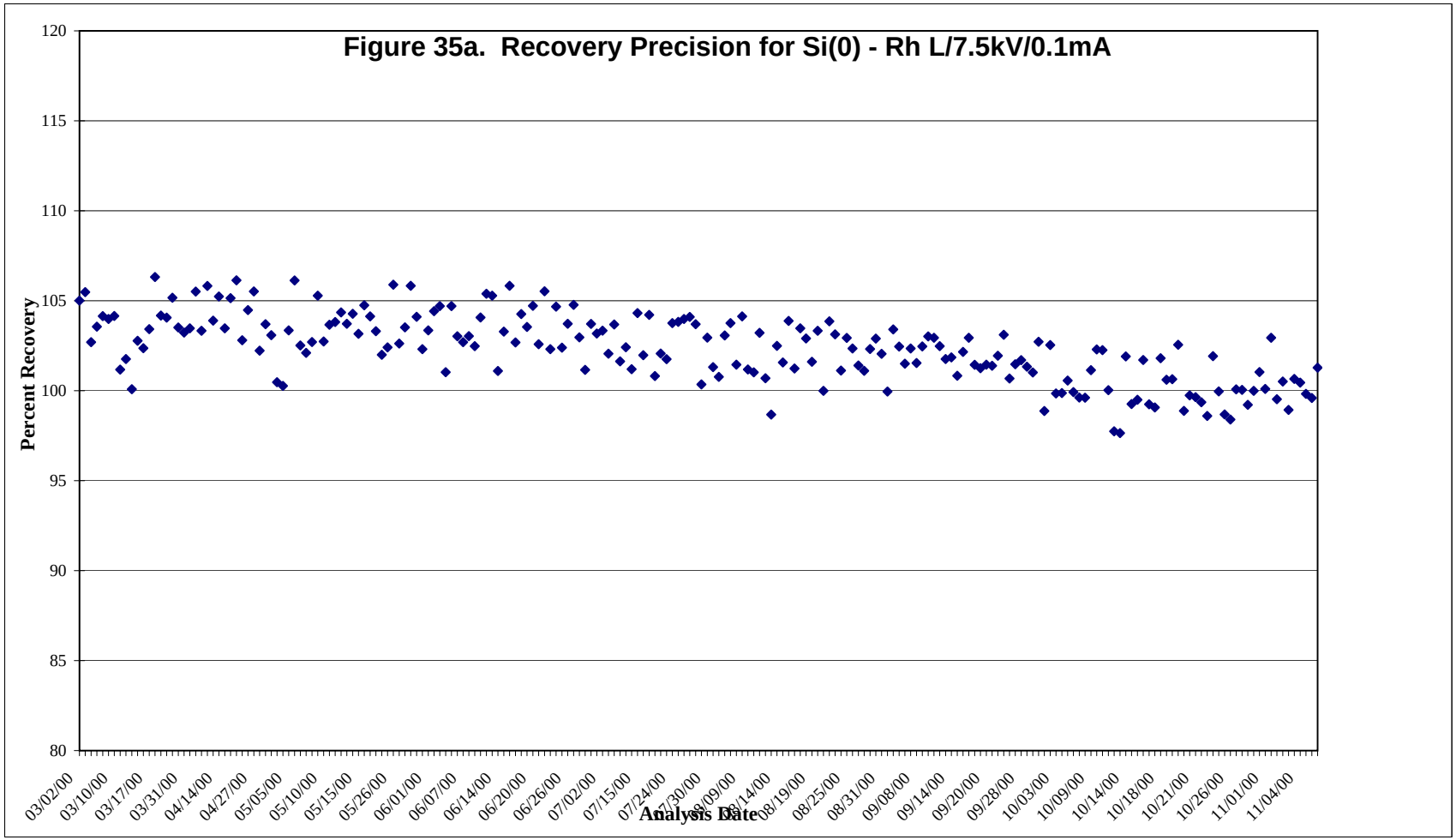


Figure 35a. Recovery Precision for Si(0) - Rh L/7.5kV/0.1mA
Percent Recovery
120
115
110
105
100
95
90
85
80
03/02/00
03/10/00
03/17/00
03/31/00
04/14/00
04/27/00
05/05/00
05/10/00
05/15/00
05/26/00
06/01/00
06/07/00
06/14/00
06/20/00
06/26/00
07/02/00
07/15/00
07/24/00
07/30/00
08/09/00
08/14/00
08/19/00
08/25/00
08/31/00
09/08/00
09/14/00
09/20/00
09/28/00
10/03/00
10/09/00
10/14/00
10/18/00
10/21/00
10/26/00
11/01/00
11/04/00
Analysis Date

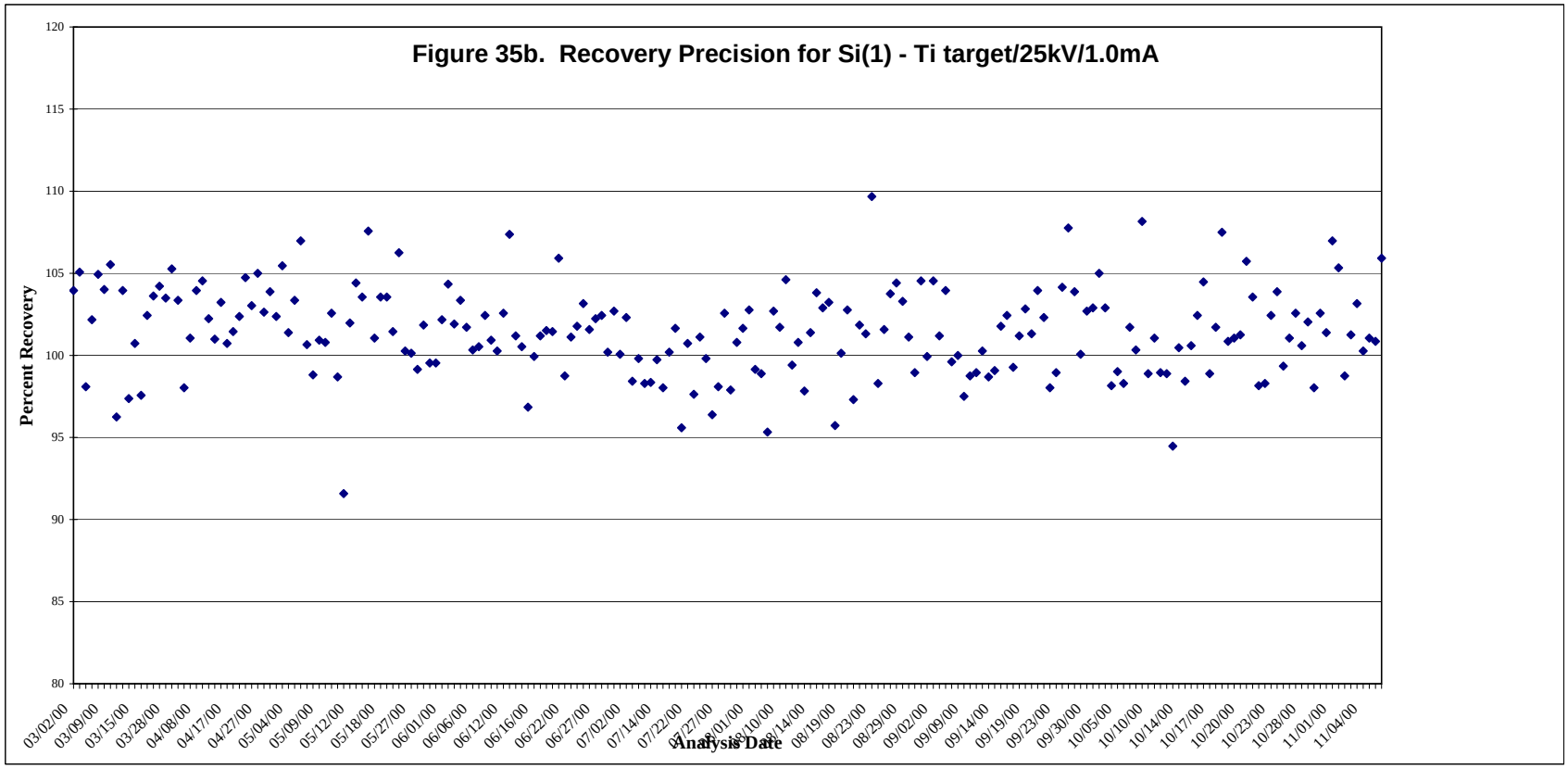


Figure 35b. Recovery Precision for Si(1) - Ti target/25kV/1.0mA
Percent Recovery
120
115
110
105
100
95
90
85
80
03/02/00
03/09/00
03/15/00
03/28/00
04/08/00
04/17/00
04/27/00
05/04/00
05/09/00
05/12/00
05/18/00
05/27/00
06/01/00
06/06/00
06/12/00
06/16/00
06/22/00
06/27/00
07/02/00
07/14/00
07/22/00
08/19/00
08/23/00
08/29/00
09/02/00
09/09/00
09/14/00
09/19/00
09/23/00
09/30/00
10/05/00
10/10/00
10/14/00
10/17/00
10/20/00
10/23/00
10/28/00
11/01/00
11/04/00
Analysis Date

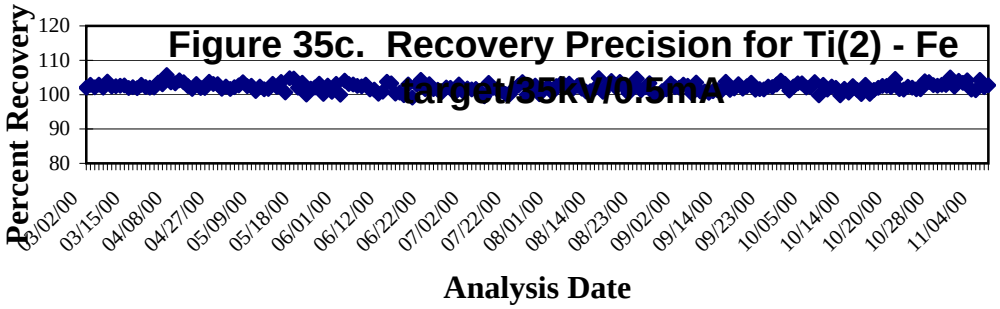


Figure 35c. Recovery Precision for Ti(2) - Fe target/35kV/0.5mA
Percent Recovery
120
110
100
90
80
03/02/00
03/15/00
04/08/00
04/27/00
05/09/00
05/18/00
06/01/00
06/12/00
06/22/00
07/02/00
07/22/00
08/01/00
08/14/00
08/23/00
09/02/00
09/14/00
09/23/00
10/05/00
10/14/00
10/20/00
10/28/00
11/04/00
Analysis Date

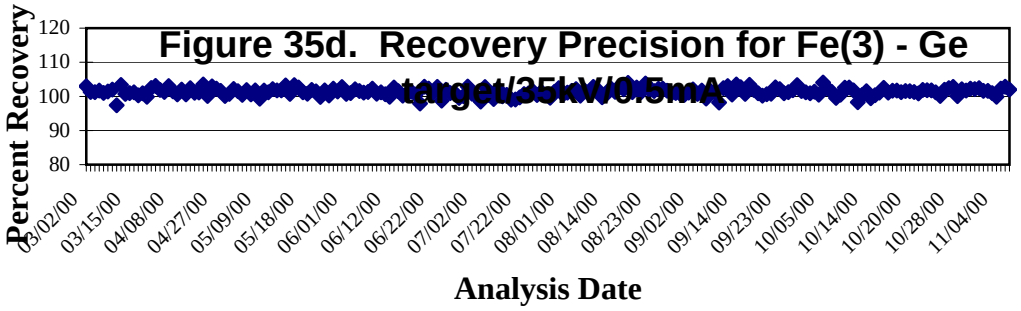


Figure 35d. Recovery Precision for Fe(3) - Ge target/35kV/0.5mA
Percent Recovery
120
110
100
90
80
03/02/00
03/15/00
04/08/00
04/27/00
05/09/00
05/18/00
06/01/00
06/12/00
06/22/00
07/02/00
07/22/00
08/01/00
08/14/00
08/23/00
09/02/00
09/14/00
09/23/00
10/05/00
10/14/00
10/20/00
10/28/00
11/04/00
Analysis Date

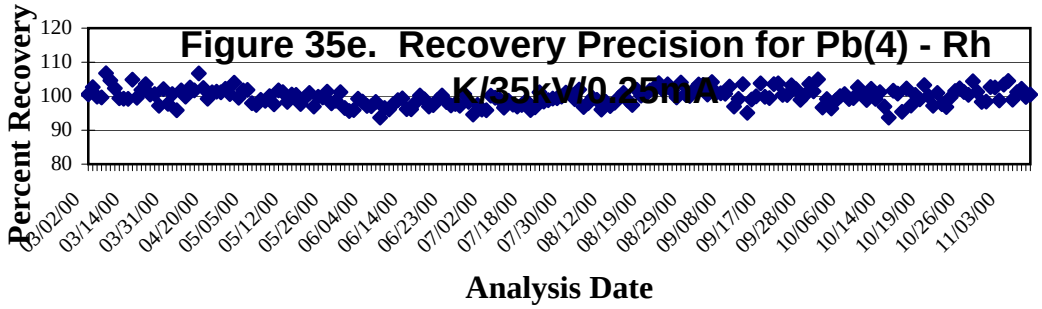


Figure 35e. Recovery Precision for Pb(4) - Rh K/35kV/0.25mA
Percent Recovery
120
110
100
90
80
03/02/00
03/14/00
03/31/00
04/20/00
05/05/00
05/12/00
05/26/00
06/04/00
06/14/00
06/23/00
07/02/00
07/18/00
07/30/00
08/12/00
08/19/00
08/29/00
09/08/00
09/17/00
09/28/00
10/06/00
10/14/00
10/19/00
10/26/00
11/03/00
Analysis Date

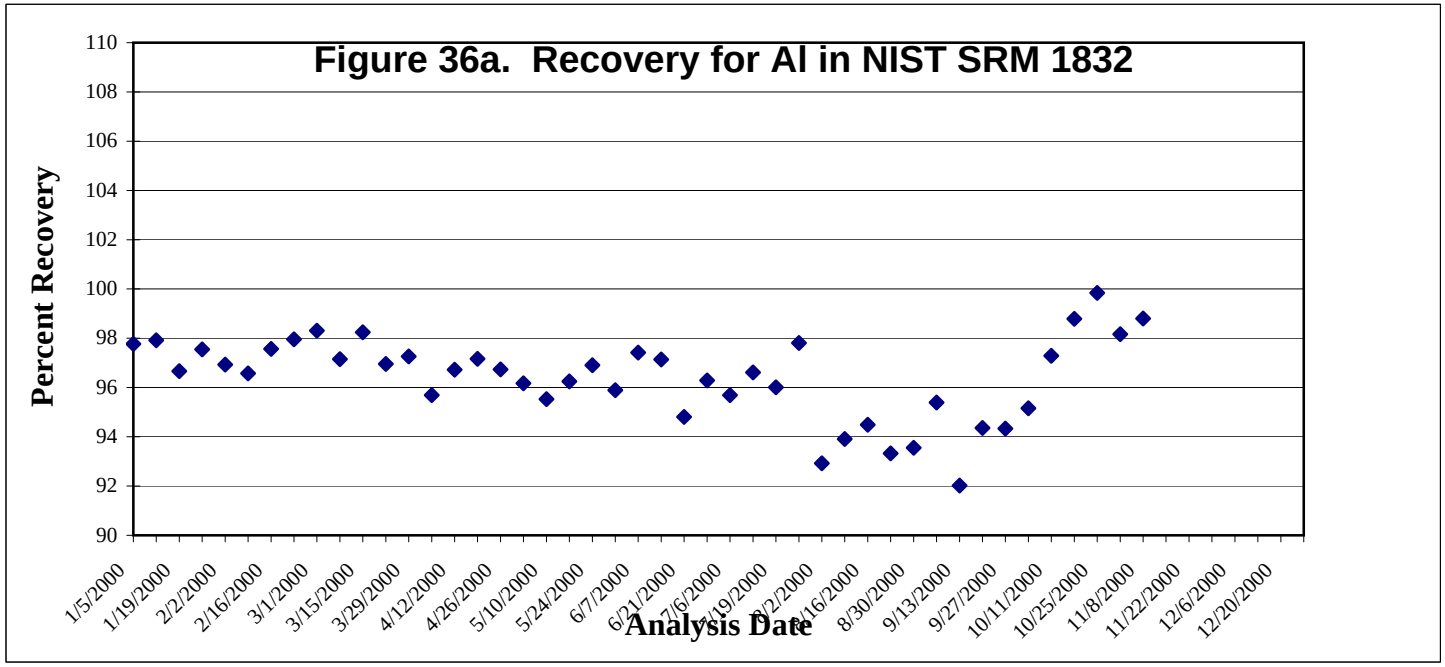


Figure 36a. Recovery for Al in NIST SRM 1832
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

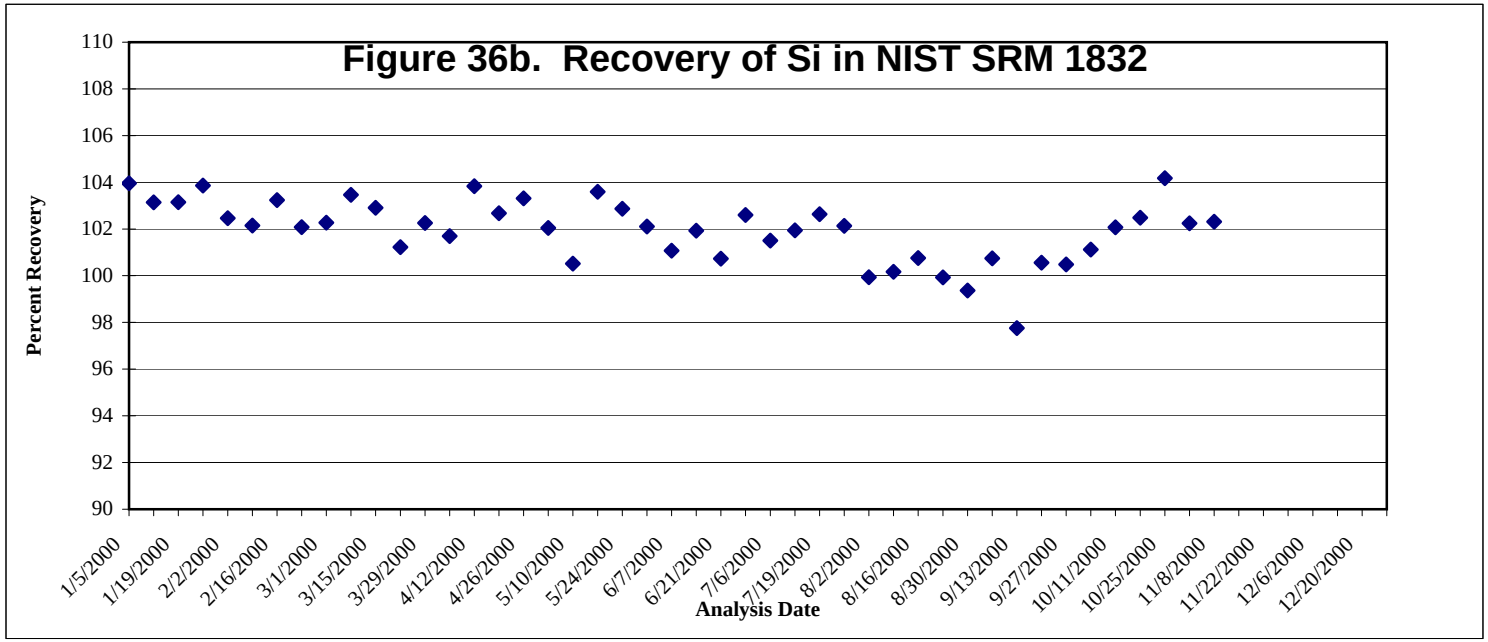


Figure 36b. Recovery of Si in NIST SRM 1832
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

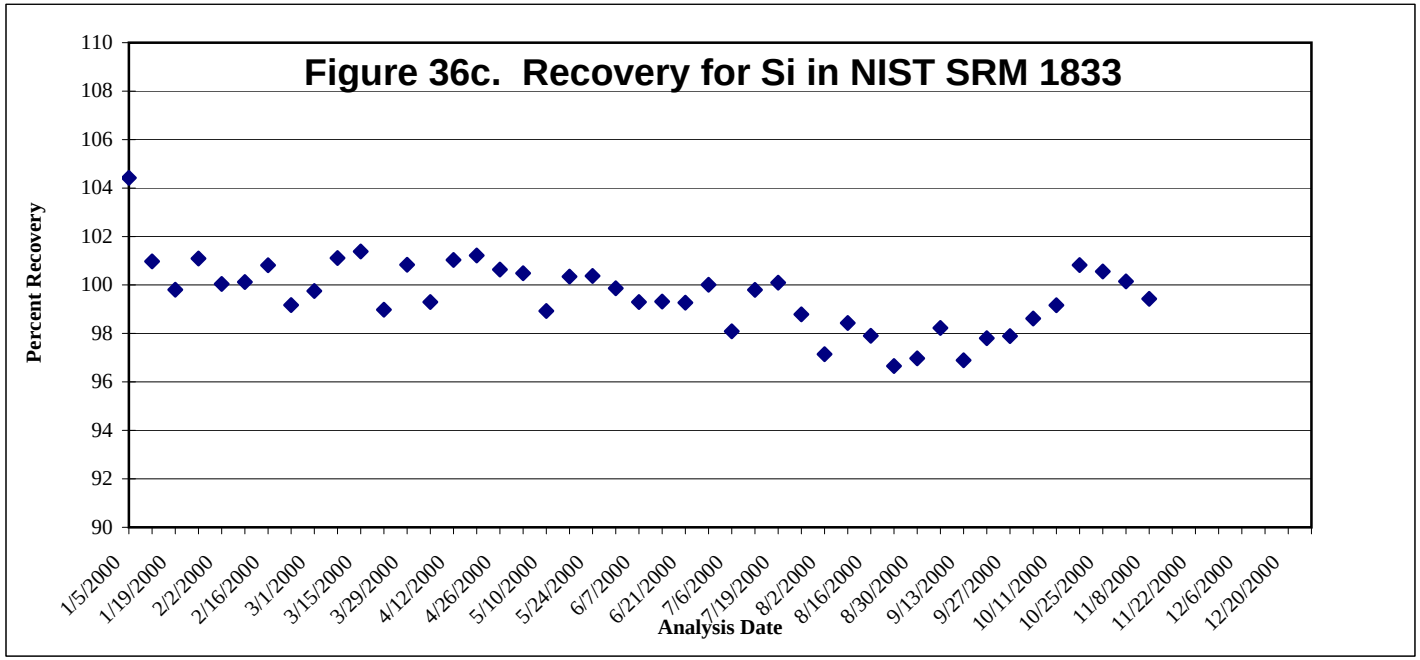


Figure 36c. Recovery for Si in NIST SRM 1833
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

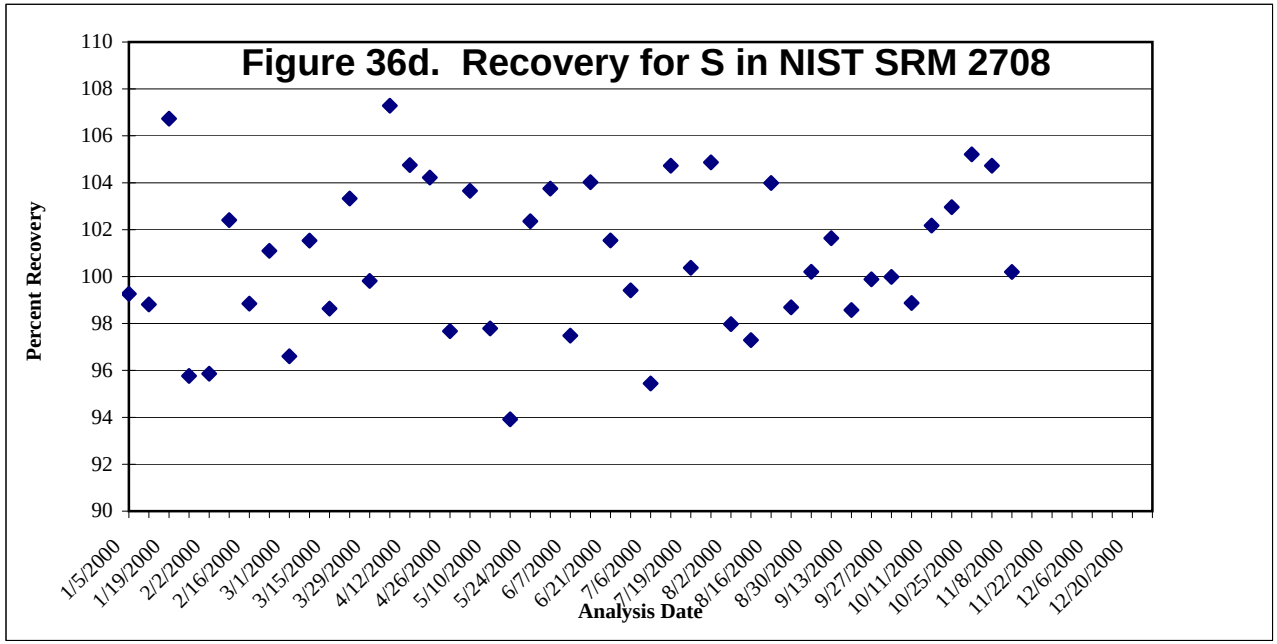


Figure 36d. Recovery for S in NIST SRM 2708
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

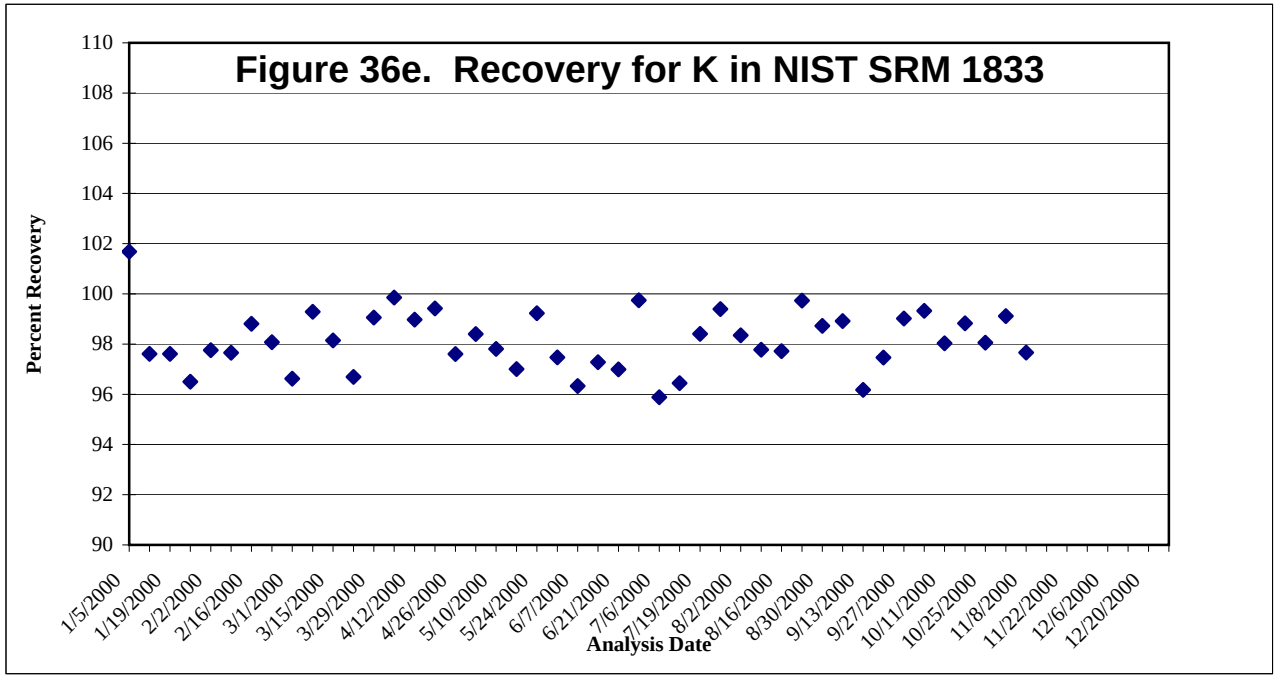


Figure 36e. Recovery for K in NIST SRM 1833
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

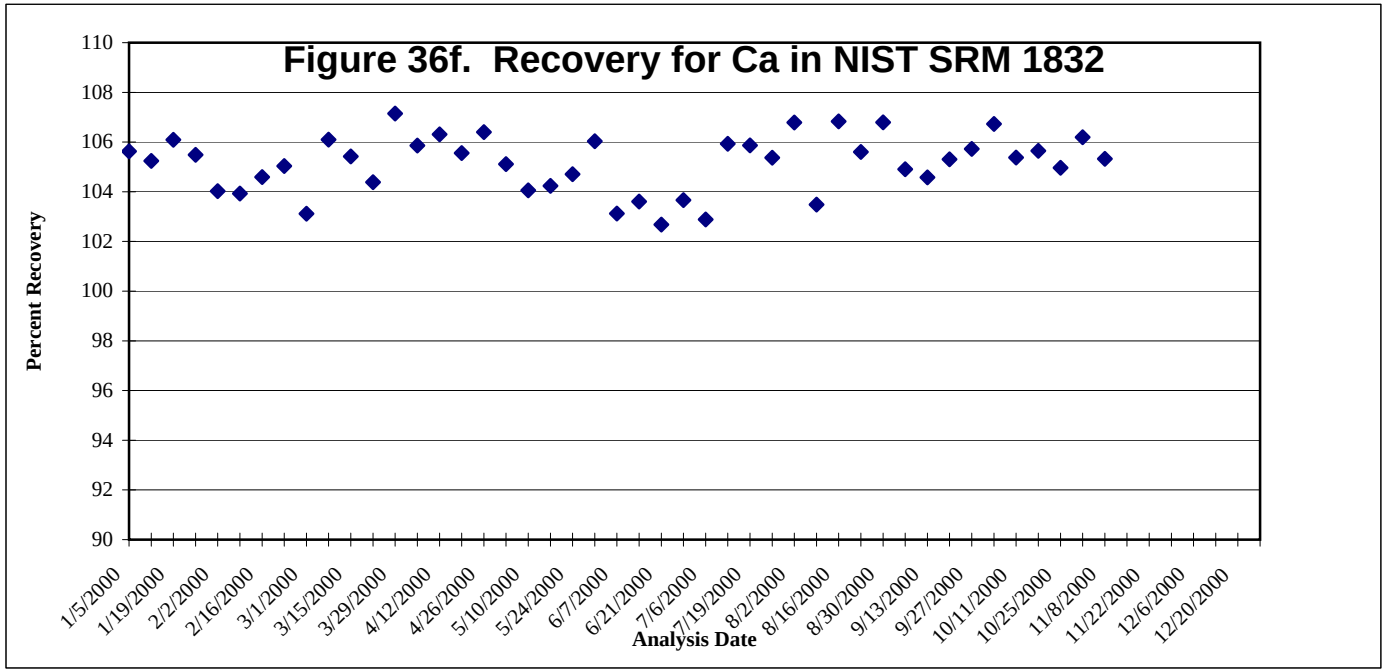


Figure 36f. Recovery for Ca in NIST SRM 1832
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

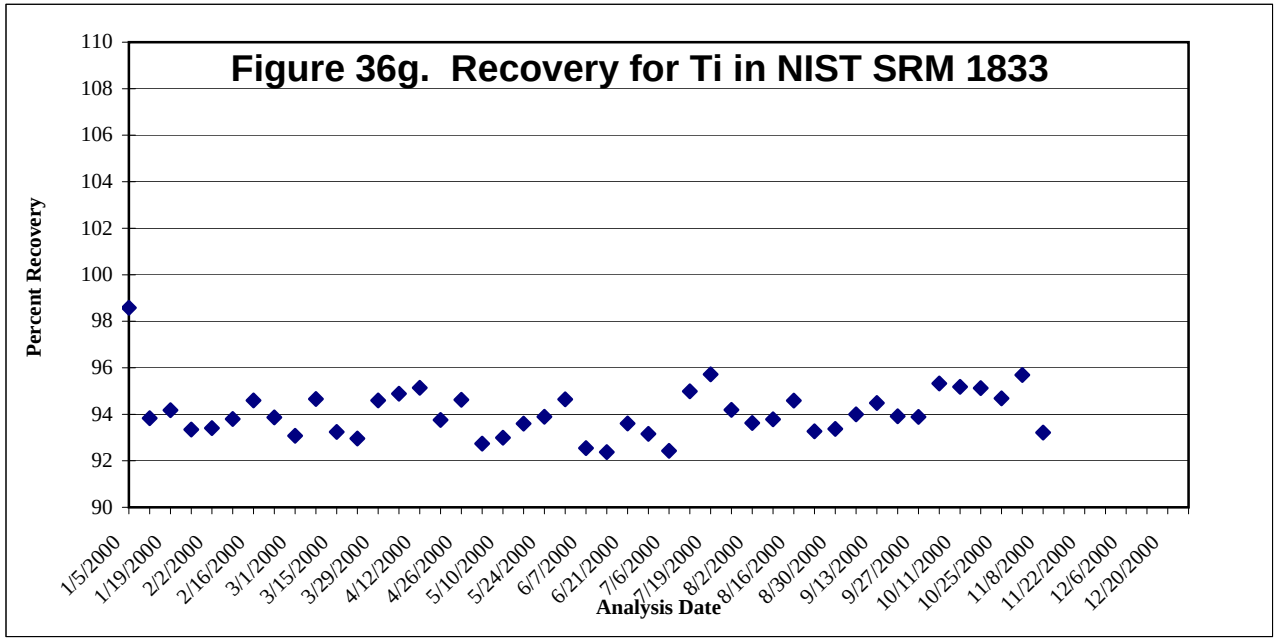


Figure 36g. Recovery for Ti in NIST SRM 1833
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

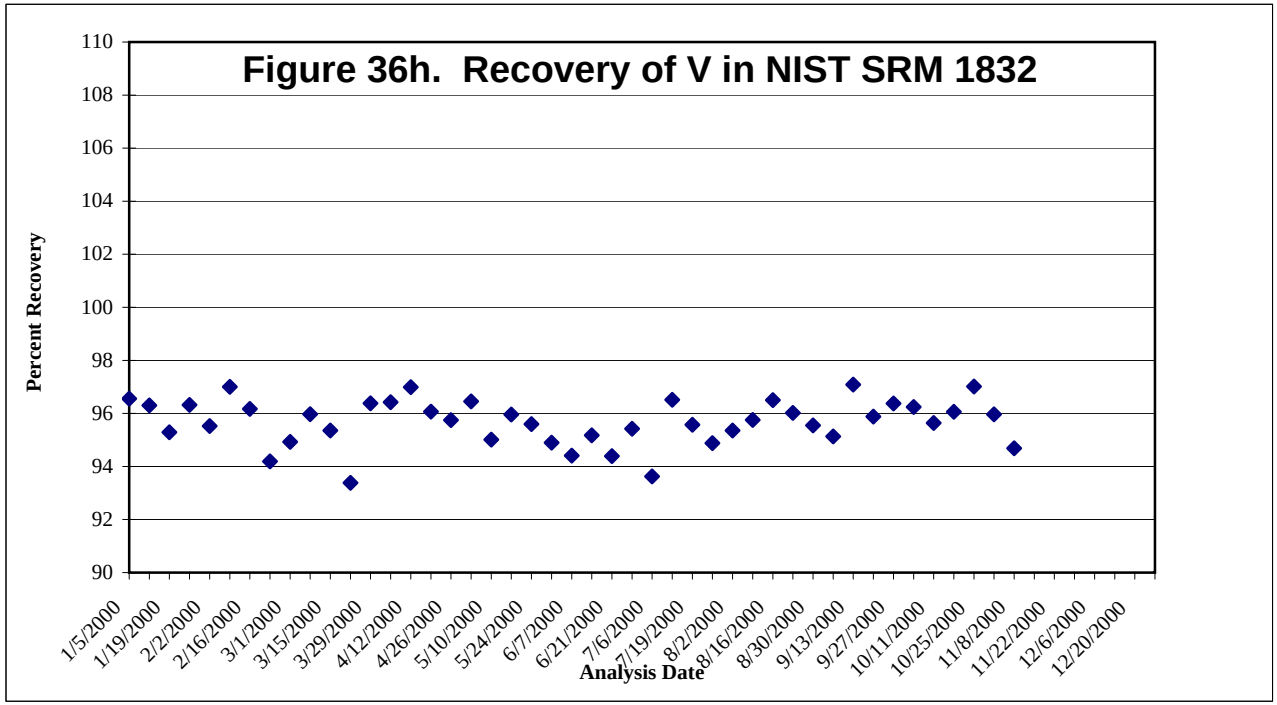


Figure 36h. Recovery of V in NIST SRM 1832
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

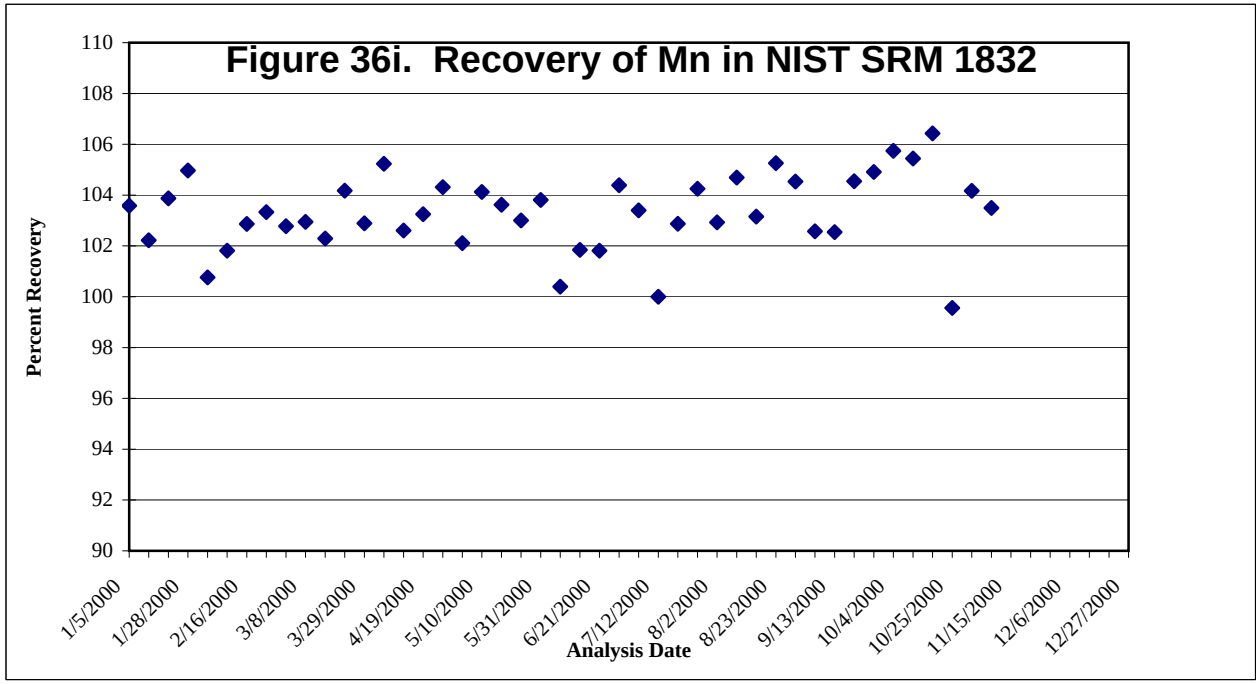


Figure 36i. Recovery of Mn in NIST SRM 1832
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/28/2000
2/16/2000
3/8/2000
3/29/2000
4/19/2000
5/10/2000
5/31/2000
6/21/2000
7/12/2000
8/2/2000
8/23/2000
9/13/2000
10/4/2000
10/25/2000
11/15/2000
12/6/2000
12/27/2000
Analysis Date

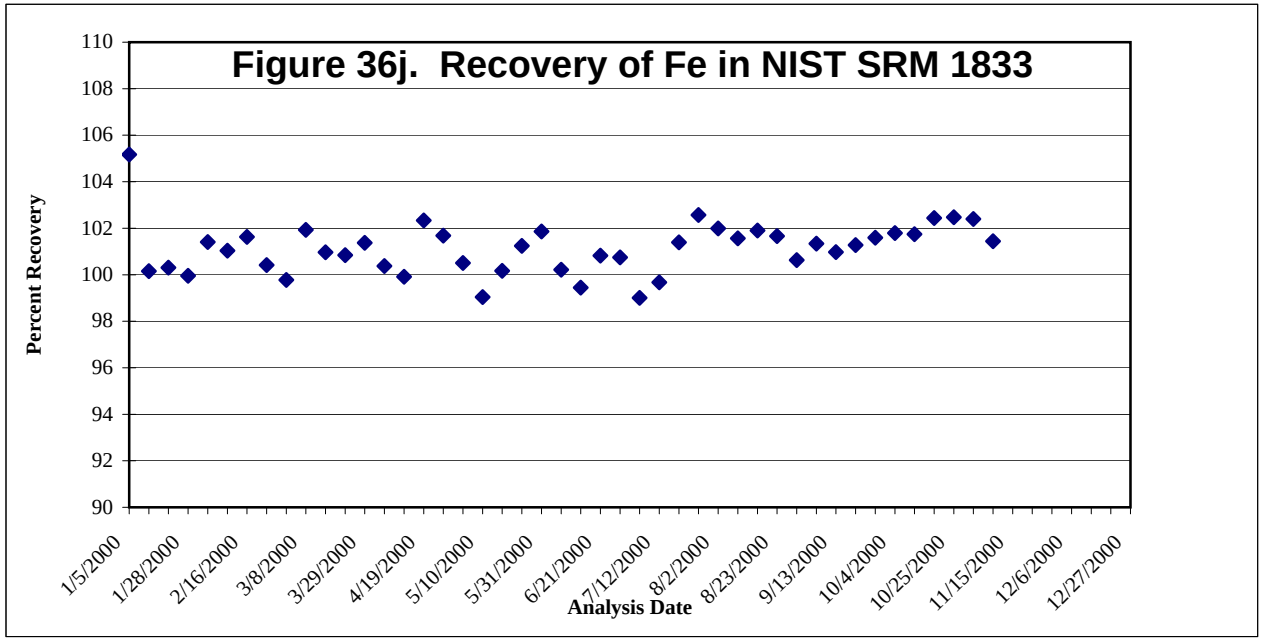


Figure 36j. Recovery of Fe in NIST SRM 1833
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/28/2000
2/16/2000
3/8/2000
3/29/2000
4/19/2000
5/10/2000
5/31/2000
6/21/2000
7/12/2000
8/2/2000
8/23/2000
9/13/2000
10/4/2000
10/25/2000
11/15/2000
12/6/2000
12/27/2000
Analysis Date

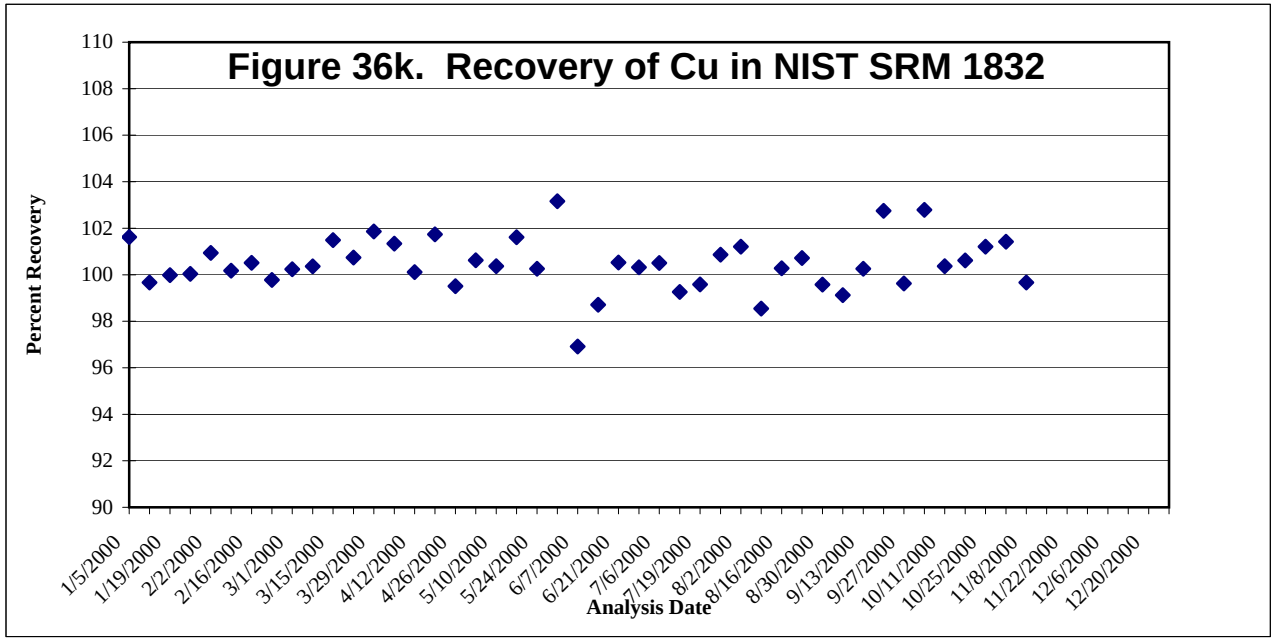


Figure 36k. Recovery of Cu in NIST SRM 1832
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

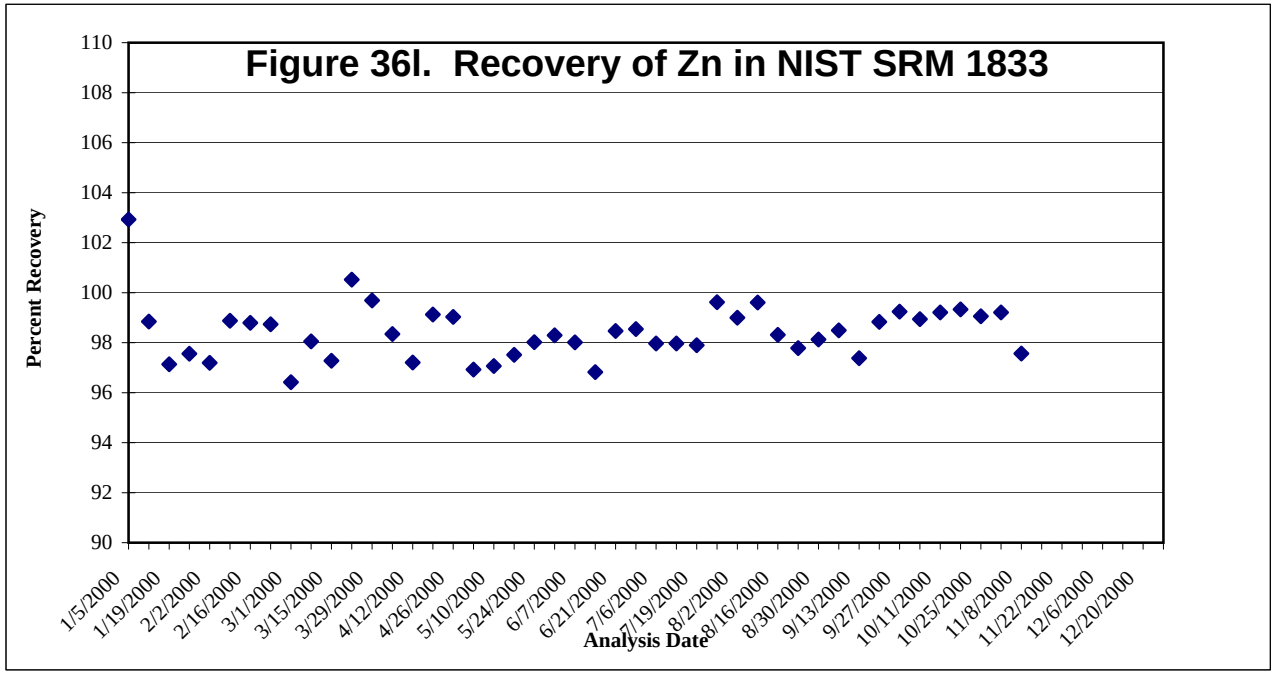


Figure 36l. Recovery of Zn in NIST SRM 1833
Percent Recovery
110
108
106
104
102
100
98
96
94
92
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

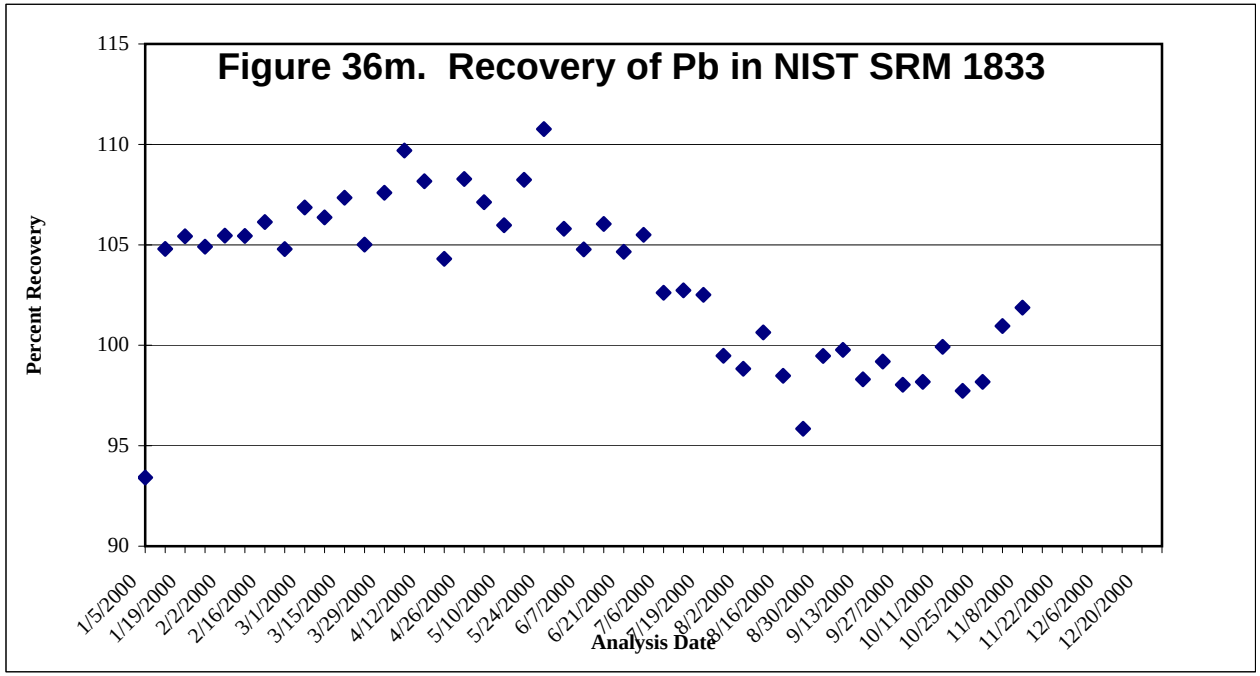


Figure 36m. Recovery of Pb in NIST SRM 1833
Percent Recovery
115
110
105
100
95
90
1/5/2000
1/19/2000
2/2/2000
2/16/2000
3/1/2000
3/15/2000
3/29/2000
4/12/2000
4/26/2000
5/10/2000
5/24/2000
6/7/2000
6/21/2000
7/6/2000
7/19/2000
8/2/2000
8/16/2000
8/30/2000
9/13/2000
9/27/2000
10/11/2000
10/25/2000
11/8/2000
11/22/2000
12/6/2000
12/20/2000
Analysis Date

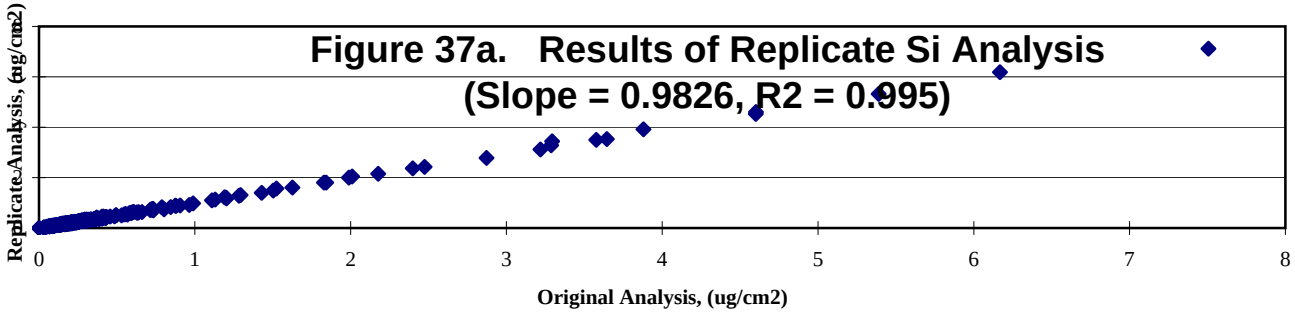


Figure 37a. Results of Replicate Si Analysis
(Slope = 0.9826, R2 = 0.995)
Replicate Analysis, (ug/cm2)
Original Analysis, (ug/cm2)
0
1
2
3
4
5
6
7
8

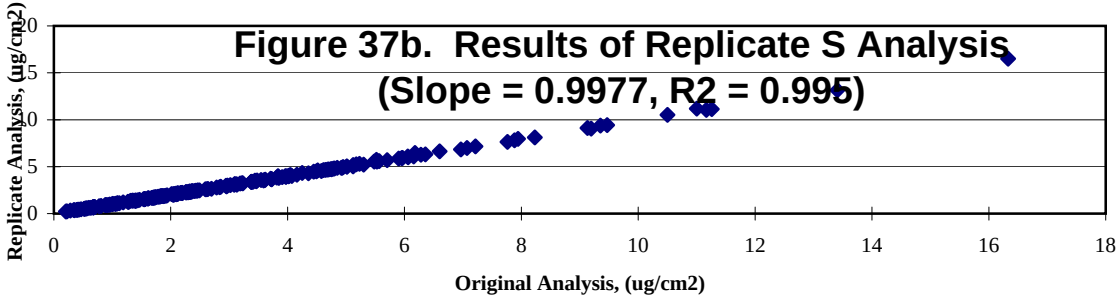


Figure 37b. Results of Replicate S Analysis
(Slope = 0.9977, R2 = 0.995)
Replicate Analysis, (ug/cm2)
Original Analysis, (ug/cm2)
0
2
4
6
8
10
12
14
16
18

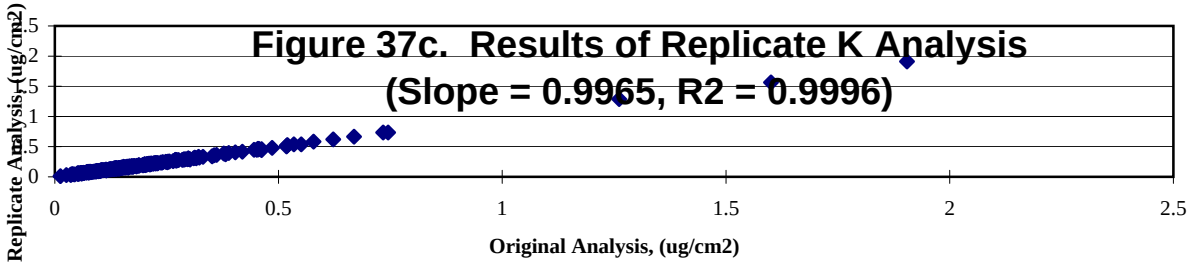


Figure 37c. Results of Replicate K Analysis
(Slope = 0.9965, R2 = 0.9996)
Replicate Analysis, (ug/cm2)
2.5
2
1.5
1
0.5
0
0
0.5
1
1.5
2
2.5
Original Analysis, (ug/cm2)

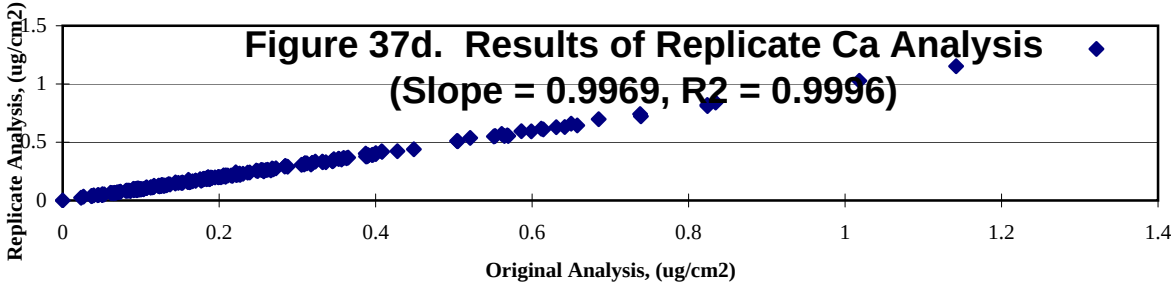


Figure 37d. Results of Replicate Ca Analysis
(Slope = 0.9969, R2 = 0.9996)
Replicate Analysis, (ug/cm2)
Original Analysis, (ug/cm2)
0
0.5
1
1.5
0
0.2
0.4
0.6
0.8
1
1.2
1.4

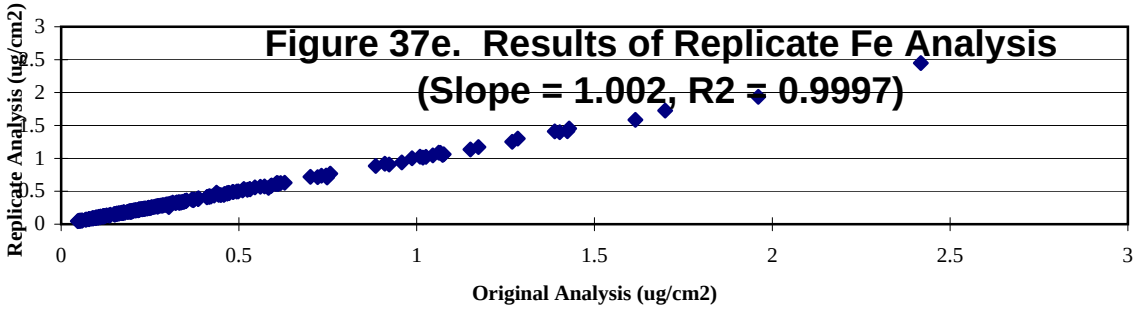


Figure 37e. Results of Replicate Fe Analysis
(Slope = 1.002, R2 = 0.9997)
Replicate Analysis (ug/cm2)
Original Analysis (ug/cm2)
0
0.5
1
1.5
2
2.5
3

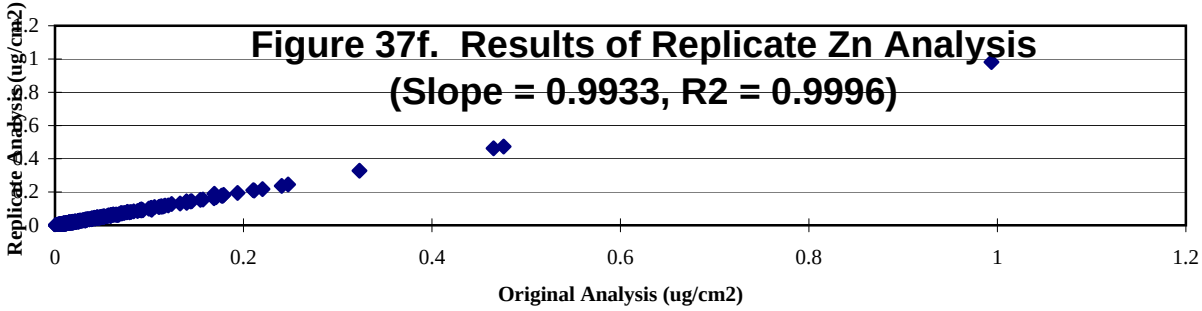


Figure 37f. Results of Replicate Zn Analysis
(Slope = 0.9933, R2 = 0.9996)
Replicate Analysis (ug/cm2)
1.2
1
0.8
0.6
0.4
0.2
0
0
0.2
0.4
0.6
0.8
1
1.2
Original Analysis (ug/cm2)

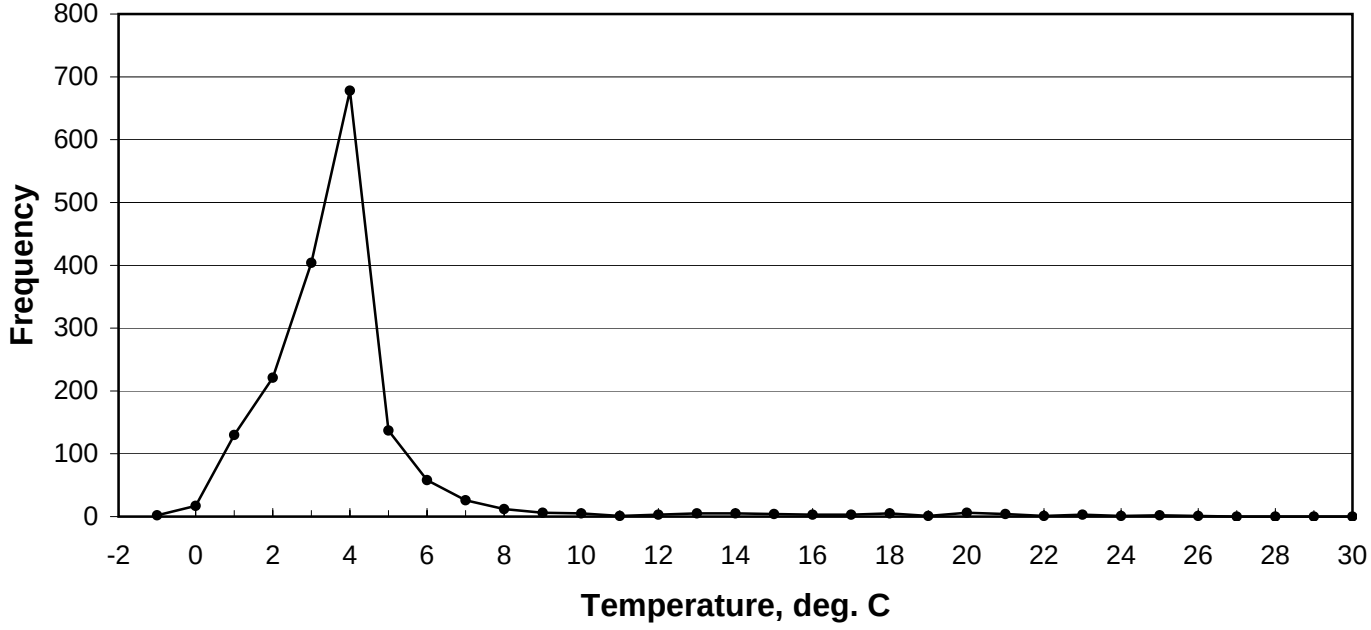



Figure 38. Distribution of Temperatures Measured While Unpacking Coolers