NADP QA Plan 2014-01

QUALITY ASSURANCE PLAN
CENTRAL ANALYTICAL LABORATORY

National Atmospheric Deposition Program

The NADP receives support from the U.S. Geological Survey; Environmental Protection Agency; National Park Service; National Oceanic and Atmospheric Administration; U.S. Department of Agriculture - Forest Service; U.S. Fish & Wildlife Service; Tennessee Valley Authority; Bureau of Land Management; and U.S. Department of Agriculture – National Institute of Food and Agriculture under agreement 2012-39138-20273. Additional support is provided by other federal, state, local, and tribal agencies, State Agricultural Experiment Stations, universities, and non-governmental organizations. Any opinions, findings, conclusions, or recommendations expressed in this publication are those of the authors and do not necessarily reflect the views of the U.S. Department of Agriculture or any other sponsor.

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Quality Assurance Plan

Version 7, May, 2014

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Gartman, Nina. 2014. *National Atmospheric Deposition Program Central Analytical Laboratory Quality Assurance Plan*, Illinois State Water Survey, Prairie Research Institute, University of Illinois. NADP QA Plan 2014-01. Champaign, IL.

Quality Assurance Plan Approval Form

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Acknowledgments

Thanks go to Pam Bedient, the CAL Office Administrator, without whose help this document could never have been completed. Thanks also go to all previous reviewers and contributors who have helped make this document as complete and accurate as possible. CAL contributors include: Tracy Dombek, Jane Rothert, Van Bowersox, Kathy Douglas, Karen Harlin, John Ingrum, Kenni James, Mark Peden, and Jackie Peden.

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Acronyms and Abbreviations

AIRMoN	Atmospheric Integrated Research Monitoring Network
AMoN	Ammonia Monitoring Network
ANSI	American National Standards Institute
ASQC	American Society for Quality Control
ASTM	ASTM, International
CAL	Central Analytical Laboratory
CPD	Conductance Percent Difference
DI	Deionized
DMAS	Data Management and Assessment Subcommittee
DQOs	Data Quality Objectives
ECPTP	Environment Canada Proficiency Testing Program
FIA	Flow Injection Analysis
FOF	Field Observer Form (AIRMoN)
FORF	Field Observer Report Form (NTN)
FR50	A synthetic rainwater solution formulated to approximate the 50^{th} percentile concentrations of the NADP/NTN
HDPE	High-Density Polyethylene
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
IPD	Ion Percent Difference
ISWS	Illinois State Water Survey
LABNO	Laboratory Number
LIMS	Laboratory Information Management System
MDL	Method Detection Limit
NADP	National Atmospheric Deposition Program
NILU	Norwegian Institute for Air Research
NIST	National Institute for Standards and Technology
NOS	Network Operations Subcommittee
NRSP-3	National Research Support Project

Acronyms and Abbreviations (concluded)

NTN	National Trends Network
PDS	Passive Diffusion Sampler
PO	Program Office
QA	Quality Assurance
QAAG	Quality Assurance Advisory Group
QA/R-5	EPA Requirements for QA Project Plans
QAP	Quality Assurance Plan
QC	Quality Control
QCS	Quality Control Sample
QMP	Quality Management Plan
Site ID	Station Identification code
SL	Screening Level
SOP	Standard Operating Procedure
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey
WMO/GAW	World Meteorological Organization/Global Atmospheric Watch

Quality Assurance Plan Document History

Approval Date:	August 21, 2002
Revisions:	2.0 June 1, 2006
B-2.0, Table 2	Revised sample dilution procedures implemented based on Network Operations Subcommittee (NOS) 2002 audit team recommendation
B-4.0	Revised procedure for calculating Method Detection Limits (MDLs)
B-4.0, Table 3	Inductively Coupled Plasma - Optical Emission Spectrometer (ICP-OES) replaced Atomic Adsorption Spectrometer as of January 2004. (Other changes were made throughout the document to reflect this change.)
B-4.0, Table 5	Table of historic MDLs updated
B-4.0, Table 6	Percentiles for concentration values updated
B-5.0	Records retention period changed to 2.5 years after date of analysis
C-1.0	Selection of samples for random reanalysis changed. Samples are selected automatically by the Laboratory Information Management System (LIMS) and not by the QA Specialist.
D	Chapter revised extensively with updated flowcharts and other information to reflect functionality of LIMS system implemented since last revision
All sections	Minor editorial changes were made throughout the document to clarify procedures.
Revisions:	3.0 April, 2008
B-3.0, Table 2	Revised sample volume threshold to 8.0 mL
B-3.0, Table 3	Deleted
B-4.0, Table 4	Deleted
B-4.0, Table 5	Deleted
B-4.0	Clarification of reanalysis procedures
B-7.0	Additional high concentration QCS for ICP
All sections	Minor editorial changes were made throughout the document to clarify procedures.

Revisions:		4.0 April 2009
	Section C	Tables 2 and 3 (IPC and CPD) removed
	Section D	Sections 2.0 and 3.0
	All sections	Minor editorial changes were made throughout the document to clarify procedures.
Revisions:		5.0 April 2010
	All Sections	Document reformatted to separate general provisions applicable to any project from those specific to NADP/NTN and NADP/AIRMoN. Editorial changes were made throughout the document Certain content that is in existing standard operating procedures (SOPs) was removed; where appropriate, the SOPs are referenced.
	B-3.0	New section on sample validation was added. The remainder of the section was renumbered.
	B-4.1, 4.2	Redefined bias as "Required Accuracy." Modified targets for pH to be precise within 0.04 pH units (formerly 0.03 pH units)
	B-4.3	Bias for reanalysis set at the same limits as that for blind samples stated in Section 4.1 (formerly 10%).
	B-9.1	Redefined duplicate analysis as analytical replicates for two percent of samples analyzed; redefined section 9.2 and renumber rest of section.
	B-12	Maintenance logs for instruments no longer reviewed by laboratory supervisor, as this supervisory position does not currently exist.
	C-5	Corrective action levels in 5.6.4, 5.6.5 redefined at the required accuracy levels (Section B-4.1).
	C-5.5	Calibration of instrument electronics is verified through use of QC standards on a daily basis; formerly calibrated annually.
	C-6.3	Clarified corrective actions based on control chart review.
	D-1	Removed reference to legacy R: Base system as all data are now stored in a SQL server LIMS system.

Revisions 6.0 July 2011

All Sections	Editorial changes were made throughout the document.
New Sections	Added description of Ammonia Monitoring Network (AMoN) in sections A-1.0, B-2.0, B-6.3, B-8.0, and B-10.4.
B-4.1	Clarification of bias at the MDL
B-7.0	Replicate sample added as acceptable QC solution.
B-10.1	Clarification of standard storage.
B-10.5	Clarification of external QA sample storage.
C-5.6.2	Clarification of standard expiration date.
C-5.6.3	Updated method for calculating control charts.

Revisions 7.0 May 2014

All Sections	Editorial changes were made throughout the document.
New Sections	Section A-4.5 was added in description of Personnel Qualifications and Training.
A-1.3 and B-2.1	Web address where CAL SOPs can be found was changed.
A-1.5	AIRMoN QAP was removed from the list of relevant source documents.
A-4.3, A-4.4 and A-4.5	The duties and responsibilities of CAL analytical, data and administrative stuff related to safety and technical training were clarified.
B-2.2.2	Bromide was added in description of the order for chemical analysis of AIRMoN samples.

B-2.2.3	Sample processing for AMoN samples was changed. Prepared samples should be placed in a freezer until they are shipped out (formerly they were refrigerated).
B-4.2	The method of determination of specific MDLs (for NTN, AIRMoN and AMoN samples) was added
B-4.2.1	AMoN was added in the list of projects.
B-6.1.4	The monitoring of the analytical and pan balances for proper operation and accuracy was clarified.
B-7.1	DI water was added to the list of quality control samples which are analyzed immediately after standardization.
B-9.2.1 and B-9.2.2	The procedure of preparing NTN and AIRMoN blind samples changed. Now two QC blind samples for AIRMoN and two QC blind samples for NTN are introduced weekly into the analytical queue (formerly one AIRMoN and three NTN blind samples).
B-10.1 and B-10.3	The general storage procedure and NADP/NTN storage procedures were clarified.
C-4.1	Quality assurance issues are discussed at monthly team meetings instead of at separate meetings.
D-2.4 and D-2.5	Data security procedures were clarified.

A. Project Management

1.0 Purpose of Plan

1.1 The Quality Assurance Plan (QAP) for the National Atmospheric Deposition Program (NADP) Central Analytical Laboratory (CAL) provides guidelines for producing quality assured and screened data for which NADP data quality objectives are quantified. Sample collection and transport, sample processing and chemical analysis, data validation and verification, and final transfer of data to the Program Office (PO) all require established protocols to ensure that data meet user needs. The QAP defines these quality indicators and specifies how they are to be monitored and quantified. This QAP is designed to cover all aspects of sample processing, sample analysis, instrument calibration, internal Quality Control (QC) checks, data handling, data screening, and final data processing prior to data transfer to the NADP PO.

1.2 The CAL provides site support, sample processing, chemical analysis, and data validation services for precipitation samples collected at the NADP/Atmospheric Integrated Research Monitoring Network component (NADP/AIRMoN), the NADP/National Trends Network (NADP/NTN), and passive type air samplers for the NADP/Ammonia Monitoring Network (NADP/AMoN). These sites must follow strict quality assurance (QA) and quality control (QC) procedures. The laboratory that has provided these services is located at the Illinois State Water Survey (ISWS), Prairie Research Institute (PRI), University of Illinois in Urbana-Champaign, Illinois. The CAL has been analyzing NADP/NTN samples since the network's inception in 1978, NADP/AIRMoN samples since 1992, and NADP/AMoN samples since 2007.

1.3 Quality assurance for the analytical measurement process at the CAL is a multi-tiered program that includes bench-level QC, laboratory management-level QA, and participation in external QA monitoring efforts. The laboratory continually strives to improve the current methods and to find new instrumentation that will achieve optimal detection limits, improve sample throughput, improve measurement precision, and reduce bias for analytical measurements. Documentation of these methods is updated annually in the laboratory QA report. Standard Operating Procedures (SOPs) for all support activities are maintained and updated annually. CAL SOPs can be found at http://nadp.isws.illinois.edu/cal/summary_of_procedures.html.

1.4 The NADP/CAL QAP follows the ISWS and the NADP Quality Management Plans (QMP), the "umbrella" QA documents that describe the processes and procedures for staff and management to follow in producing environmental data. The NADP/CAL QAP is patterned after a national consensus standard, American National Standards Institute/American Society for Quality Control (ANSI/ASQC) E4-1994, and U.S. Environmental Protection Agency (USEPA) Requirements for QA Project Plans (QA/R-5), a USEPA guidance document developed to assist each agency contractor in developing an agency-specific QAP.

1.5 The following is a list of relevant source documents:

ISWS Quality Management Plan
NADP Quality Management Plan
NADP Network QA Plan
CAL SOPs
CAL Work Plan[1]

1.6 The CAL QA Chemist and the CAL Director will review this plan annually and update as needed but no less frequently than once every three years. All revisions will be numbered and dated; previous versions will be kept in the CAL archives for reference.

2.0 Management and Organization

Figure 1 shows a current organizational chart for the NADP CAL.
The CAL Director reports to the NADP Program Coordinator and to the NADP Executive Committee. The NADP CAL Director is responsible for seeing that all laboratory activities follow the requirements defined in the CAL Work Plan.

- Quality Assurance
  - Laboratory Operations Quality Control
  - NADP Quality Assurance Advisory Group (QAAG) Participation
  - Annual QA Report
  - SOP Management
- Support Services
  - Site Supply Shipments
  - Sample Receipt
  - Supply Preparation
- Data Management
  - NTN Database
  - AIRMoN Database
  - AMoN Database
  - SAP Database
  - Laboratory Information Management System (LIMS)
  - Data Validation
  - Data Entry
  - Data Delivery

[1] The CAL Work Plan specifies CAL deliverables to the Program Office. For more information, contact the Program Coordinator or CAL Director.

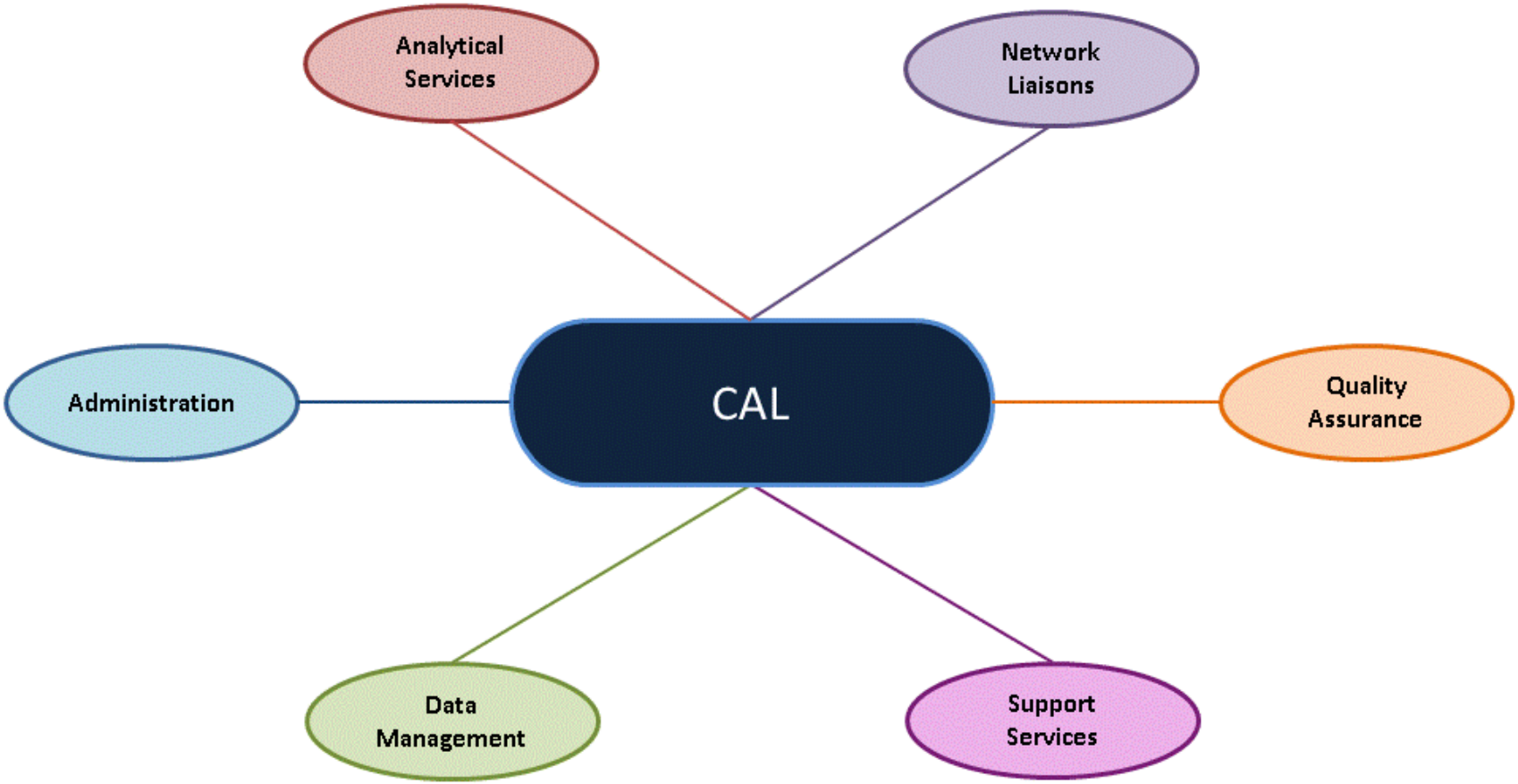

Figure 1. Central Analytical Laboratory Organizational Chart

- Administration
  - Personnel Management
  - Budget Management
  - Supply Database
  - SOP Database
- Analytical Services
  - Sample Preparation
  - Chemical Analysis
- Network Liaisons
  - Site Technical Support
  - Operator Training
  - Preliminary Data Reports

The NADP technical staff includes scientists, permanent support staff, and hourly staff. The NADP staff is committed to continued quality improvement of the network. As part of their routine responsibilities, the staff read and follows the CAL QAP, maintain and adhere to SOPs and participate in improving the overall quality of the CAL.

3.0 Elements of NADP CAL Quality System

3.1 This QAP describes the QA/QC procedures of the NADP CAL. The CAL QA Chemist is responsible for maintaining the CAL QAP. Standard Operating Procedures (SOPs) describe the detailed method for an operation, activity, or analysis so that the procedure can be performed consistently over a long time period.

3.2 Periodic on-site technical reviews are conducted to evaluate documents, activities, materials, data, and other work products that require technical verification for bias, precision, completeness, and representativeness. The NADP Committees, with the guidance of the Network Operations Subcommittee (NOS), Data Management and Analysis Subcommittee (DMAS), and the NADP QA Manager, conducts on-site CAL technical reviews every three years with a follow-up paper review one year following the on-site review per the NADP Quality Management Plan (NADP 2011).

3.3 Internal ISWS technical reviews may be conducted by ISWS staff with equivalent experience and training in the project discipline. These reviews may be requested at any time by the CAL Director or QA Chemist. The CAL Director is responsible for retaining records that document review findings and responses.

4.0 Personnel Qualifications and Training

4.1 Functions performed by CAL staff require different educational backgrounds. The specific requirements for each job are listed in the SOP for that task. All chemical measurements are performed by analysts who have at least a Bachelor of Science degree in a physical or life sciences discipline or who are under the direct supervision of a degreed scientific staff member.

4.2 As a minimum requirement, new staff must be trained for specific jobs by another CAL staff member familiar with that job and may need to attend structured courses that cover specific training in instrumentation, procedures, and other areas of specialized need. Analytical staff must be proficient in the operation of each instrument as proven by analysis of blind samples for which the chemistry is known to the QA Chemist but not to the analyst. Only when the analysis of the blind samples is completed within specific control limits is the new analyst allowed to begin routine analysis of NADP precipitation samples.

4.3 Training for CAL analytical and data staff is ongoing. The staff is encouraged to upgrade and expand their skills into new areas continually. Personal and professional development courses offered by the University of Illinois Office of Human Resources Development are available to all ISWS and CAL staff. Staff safety training also is provided though the University of Illinois Division of Research Safety. All CAL staff are required to participate in all safety training courses.

4.4 All safety and technical training certificates are kept on file by the CAL Administrative Coordinator.

4.5. Training is provided to CAL staff to ensure that critical areas are covered at all times by backup staff.

5.0 Laboratory Facilities

The CAL facilities are located on the campus of the University of Illinois at Urbana-Champaign. Total square footage for laboratories, shipping, and receiving at the CAL is approximately 4000 ft^2.

B. Laboratory Operations

1.0 Program Objectives

1.1 Program objectives include chemical analyses of wet deposition and related atmospheric samples and recording, verifying, screening, and reporting data. Integral parts of this program are QC of the sample analyses and QA of the data review and transfer.

1.2 Although this QA Plan addresses current NADP programs, new initiatives are periodically introduced in NADP resulting in new programs for the CAL to incorporate into this QA Plan. When new initiatives become approved programs, any specifics to those programs will be added to this QAP. When appropriate, procedures for ensuring quality data will be applied to any new programs. Unique procedures will be documented in SOPs for the new programs, with general information added to the QAP as needed.

2.0 Sample Processing and Chain of Custody

2.1 Detailed information on sample processing for the NADP/NTN, NADP/AIRMoN, and NADP/AMoN is contained in several different SOPs which can be found at http://nadp.isws.illinois.edu/cal/summary_of_procedures.html.

2.2 As samples are logged in, information from the field sample submission form is entered into a Laboratory Information Management System (LIMS) and from there into the data base. Each sample is identified by a unique laboratory number (LABNO) and station identification code (Site ID). These designators remain linked to the sample throughout data verification and transfer of final data to the PO. See the appropriate SOPs for sample receipt and processing for full details.

2.2.1 Sample processing for NTN

- Both pH and specific conductance must be measured for all samples of sufficient volume within three business days of sample login.
- All other analyses must be completed within three weeks of their arrival at the CAL.
- The order for chemical analyses is pH and conductivity first followed by the remaining analytes in no particular order.

2.2.2 Sample processing for AIRMoN

- Samples are refrigerated from the point of field collection until they are discarded.
- Both pH and specific conductance must be measured for all samples with sufficient volume within three business days of sample login.
- All other analyses must be completed within two weeks of their arrival at the CAL.

- The order for chemical analysis of AIRMoN samples is pH, specific conductance, ammonium, orthophosphate, chloride, nitrate, sulfate, bromide, calcium, magnesium, sodium, and potassium.

2.2.3 Sample processing for AMoN

- Prepared samples are placed in a freezer until they are shipped out.
- Returned samplers are placed in a freezer until extraction.
- Samples are prepared and extracted in a clean air bench.
- Sample extraction and analysis must be completed within 10 days of sample receipt.

2.3 Sample chain of custody is important at the CAL. All samples are kept in locked storage areas unless they are being used by the analysts in the laboratories. Barcoded identification is put on the samples which is traceable to the field sample submission forms. In general, chain of custody is maintained electronically in LIMS, with a record of receipt date, analysis date, and login identification of staff person handling the sample.

3.0 Site Resupply

The NADP ongoing long-term monitoring program requires specific equipment and established protocols to maintain data consistency throughout the networks. The CAL must supply materials of identical quality to those being replaced at the sites. The laboratory provides supplies and solutions for NTN, AIRMoN, and AMoN. For more detailed information, see SOPs relating to supplies preparation.

4.0 Sample Chemical Analysis

4.1 Precipitation samples are typically characterized by a low dissolved solids content (< 20 mg/L) resulting in a highly unbuffered system. The overall program objective is to produce analytical data for which precision and bias are quantified. Data Quality Objectives (DQOs) are defined to optimize data quality. Information relating to NADP analytes measured, instruments used, and the dates the instruments were purchased is available at http://nadp.isws.illinois.edu/cal/. Full documentation for all instrumentation is maintained.

4.2 Method Detection Limits (MDLs) are the minimum concentration of an analyte that can be reported with a 99 percent confidence that the value exceeds zero. The MDL is based on a standard deviation of greater than seven replicate measurements of the analyte in the matrix of concern at a concentration near the low standard (Code of Federal Regulations, Part 136, Vol. 49, No. 209). The MDLs are a data quality indicator and are reviewed annually and revised by the QA Chemist as warranted, e.g., when a new instrument is purchased, when a critical new part is installed on an existing instrument, or when analysts start using the instruments for the first time. The MDLs are calculated as described in SOP QA-0020. The specific MDLs for AIRMoN, NTN and AMoN are based on results of analysis of blind low concentrated samples which passed through all steps of processing NTN, AIRMoN and AMoN samples. The QA Chemist compiles the

results from the previous 12 months data and reviews them with the CAL Director. The MDLs are updated with the Program Office (PO) at least once a year.

4.2.1 Required accuracy for non-physical analytes

Accuracy (or Bias), as defined in the NADP QMP, is a systematic or persistent distortion of a measurement process that causes errors in one direction of the measured value from the true value. Accuracy for NTN, AIRMoN and AMoN is determined by the analysis of routine blind samples of known concentration.

4.2.2 The accuracy goals will depend on the concentration of the analyte

A maximum allowable bias of $\pm$ MDL at the MDL.
A $\pm$ 20 percent allowable bias at 10 times the MDL.
A $\pm$ 10 percent allowable bias at 100 times or greater the MDL

4.3 Required accuracy for physical parameters

The bias and precision targets for the pH and specific conductance of a sample are:

- Samples with pH less than 5.0 pH units, ± 0.1 pH units allowable bias and ± 0.04 pH units allowable precision.
- Samples with pH greater than 5.0 pH units, ± 0.3 pH units allowable bias and ± 0.1 pH units allowable precision.
- Samples with specific conductance of 10-100 µS/cm, ± 10 percent allowable bias and ± 3 percent allowable precision.
- Samples with specific conductance of greater than 100 µS/cm, ± 6 percent allowable bias and ± 2 percent allowable precision.

4.4 Bias for reanalysis

The difference allowed between the original sample analyses and replicate sample analyses or randomly selected reanalyzed sample analyses is the same as defined in Section 4.3.

4.5 Instrument standardization

4.5.1 Standardization is instrument specific. All instruments are standardized each day they are used. In addition, pH and specific conductance are standardized every 36 samples. The standard levels used are based on approximately the 5^{th} percentile to the 99^{th} percentile concentrations found in the NADP/NTN data set. All analytes with concentrations exceeding the highest standard must be diluted and analyzed in the diluted form unless a separate curve is generated for higher concentrations. This results in typically less than 1 percent of the precipitation samples requiring dilution. The calibration curves for other samples may need to be changed to address the concentration ranges of the samples. For specifics for each instrument refer to the instrument SOPs.

4.5.2 All primary standards must be confirmed using the following methods:

- Certified reference solutions or second source standards (standards made by and obtained from a different source from the primary standards) to compare with the new stock standards. NIST or NIST-traceable standards should be used when available.
- Prior standards to compare with the new standards.
- If other comparisons are done, they must be approved by the QA Chemist and documented in the laboratory log book and the SOP for that method. All primary standard solutions are remade or purchased a few days to a couple of weeks before the expiration date of the old solutions, allowing the analysts time to compare the new with the old and to verify the new standards are within tolerance levels. Instrument standardization procedures are documented for each analyte in the instrument SOP. The frequency of standardization may vary with the instrument but is not less than once per analysis day.

5.0 Record Archives

All CAL log books are kept permanently on file at the Illinois State Water Survey. Digital analytical records are maintained for a minimum of five years following date of analysis. Paper records for analyses not digitally saved must be retained for five years following date of analysis. For analytical methods with digital records and paper records, the paper records must be maintained for 2.5 years after date of analysis.

6.0 General Laboratory Procedures

Precipitation samples are typically characterized by low ionic strength (low ion concentrations) resulting in a highly unbuffered system. Because of this, a QA program for the chemical analysis of precipitation samples requires stringent laboratory conditions and careful control over all aspects of the analyses.

6.1 General laboratory supplies

6.1.1 High density polyethylene (HDPE) bottles are used for sample storage.

6.1.2 Borosilicate glass or HDPE containers are used for standard solution preparation and storage. All volumetric glassware is Class A under ASTM International Standards E287 for Burets, E288 for Volumetric Flasks, and E969 for Volumetric (transfer) Pipettes (*Annual Book of ASTM International Standards*, Vol. 14.02).

6.1.3 Deionized water used for solution preparation must have a resistivity of greater than or equal to 18 Mohms-cm, or ASTM International Type I water (ASTM International Standard Specification for Reagent Water, D1193, *Annual Book of ASTM International Standards*, Vol. 11.01).

6.1.4 The analytical and pan balances are monitored for proper operation and accuracy by using National Institute for Standards and Technology (NIST) Traceable Class S weights at each use or on a monthly basis, whichever occurs first. Analytical balances are

serviced yearly or when test weight values are not within the manufacturer's instrument specifications, whichever occurs first. CAL reference weights are submitted for external verification once every 5 years.

6.2 Laboratory supplies used for NTN samples

Polyethersulfone filters separate the dissolved and suspended fractions found in NTN precipitation samples. Because of the possible contamination of the NTN samples during filtration, whenever a new lot of filters is obtained, the filters are checked for contamination and approved by the QA Chemist providing the concentrations of the leachate check solutions are within established control limits.

6.3 Laboratory supplies used for AMoN samples

Only Passive-type Diffusion Samplers (PDS) approved by the NADP will be used for sampling. Diffusive bodies and cores will be tested following methods approved by the NADP.

7.0 Instrument Procedures

7.1 A high, a low quality control sample (QCS) and DI water are analyzed immediately after standardization to ensure that the system is in control. At a frequency of not less than one sample in 12, at least two of the following checks must be made:

- FR50
- High Standard (not used for calibration)
- Low Standard (not used for calibration)
- Replicate Sample

7.2 All QC data are recorded directly from the analytical instruments into the LIMS. Control charts of the data are automatically generated in the LIMS as soon as data transfer is complete. Analysts use the control charts to determine the condition of their analytical systems, i.e., in control, drifting, or biased.

8.0 Analytical Blanks

8.1 Supplies used at CAL are routinely checked for contamination. These supplies include, but are not limited to:

- Collection buckets and lids
- NTN and AIRMoN shipping bottles
- DI water
- Filter blanks
- Bucket and lid storage bags
- NTN sample bottles
- AMoN cartridge and hood preparation blanks

8.2 Procedures for these checks are described in SOP PR-0041. All new sources of laboratory glass and plastic ware are evaluated prior to use to ensure that ions of interest are neither adsorbed to nor leached from the surfaces in contact with the sample.

9.0 Sample Precision

9.1 Replicates

Analytical replicates are used in the laboratory as quality assurance for the analysts to determine precision of a known sample. Approximately two percent of the NADP/NTN and NADP/AIRMoN samples are reanalyzed following the original analysis of the sample on the same day by the same analyst. The analysts review the results before sending the value to LIMS only reporting one value as the official value for the sample.

9.2 Blinds

9.2.1 Blind QC samples are used to monitor the analytical procedures. Two QC samples per week are introduced into the analytical queue disguised as real precipitation samples for AIRMoN and two samples per week for NTN, for a total of four blind QC samples per week. Complete details are in SOP QA-0049.

9.2.2 Results of the measurements are compared with the target concentrations for each ion. Analytical bias is estimated from the mean differences between the measured and target values, and precision is estimated from the relative standard deviation of the measurements for each chemical matrix. The CAL QA Chemist reports results obtained from the blind samples for each network in the annual CAL QA report and summarizes and reviews the results monthly. The CAL QA Chemist also uses the data for calculating MDLs for all analytes.

10.0 Sample Storage

10.1 General storage procedures

As needed for stability, standards are stored at 4^{o}C. Reagent storage is dependent on the requirement of the reagent. Most are prepared the day they are used so storage is not necessary. If excess reagent is prepared, the reagent is stoppered or sealed and refrigerated at 4^{o}C if necessary or stored in the working hood. See individual SOPs for details.

10.2 NADP/AIRMoN storage procedures

All NADP/AIRMoN samples must be stored at 4^{o}C immediately after collection, during shipment, and upon arrival at the CAL. Samples are never allowed to warm to room temperature, but an aliquot is removed for pH and specific conductance and allowed to equilibrate to room temperature. For other analyses, the sample is poured at 4^{o}C and is warmed to the appropriate temperature during analyses or while in the autosamplers. AIRMoN samples are stored at 4^{o}C at the CAL for two years after finalized data have been published by the PO.

10.3 NADP/NTN storage procedures

Whenever there is sufficient NADP/NTN sample for 120 mL to be filtered, 60 mL is filtered into a round bottle and used for analyses. These bottles are kept at 4 °C until the data for those samples have been sent to the PO, then they are discarded. The second 60 mL is filtered into a square bottle and archived at 4^{o}C. Archived samples from three sites (NH02, NE15, and IL11) and every 100^{th} sample must be kept for the life of the program. All other archived samples must be stored for five years after data have been published by the PO. Samples can be discarded or sent to other researchers for independent studies after this time.

10.4 NADP/AMoN storage procedures

All AMoN PDS are prepared and extracted in a clean air bench that is monitored during preparation with blanks. AMoN PDS are stored at 4°C before deployment to sites. Returned PDS are stored in a freezer before extraction.

10.5 Other sample storage procedures

External interlaboratory comparison samples are stored at 4^{o}C when required by the specific study. These samples are treated as NADP/AIRMoN samples. When the results of the studies have been received by the CAL, these samples may be used as external QA samples or discarded.

11.0 Data Verification

11.1 Results for all analytes are captured directly by data acquisition software into the LIMS. Keyboard data entry is stroke-verified through double entry by a second person for NTN and AIRMoN field forms. For more information, see "Data Management Operations" (Section D).

11.2 Computer programs contain control checks for data entry. An ion percent difference (IPD) is calculated for each NTN and AIRMoN sample. The conductance percent difference (CPD) between calculated and measured specific conductance is tabulated (for information on the IPD and CPD see SOP #DA-0067). Samples are randomly selected for reanalysis for both AIRMoN and NTN to verify sample concentrations (see Section C "Laboratory QA/QC Procedures" for more information).

12.0 Preventive Maintenance/Service

A maintenance schedule is established for each instrument. See individual SOPs for specifics. A record log of all scheduled and unscheduled maintenance is kept. The record log includes, at a minimum, the date, name of service provider, and nature of the service. The CAL director or the CAL QA chemist will review the logs at least once a year.

C. Laboratory QA/QC Procedures

1.0 Performance and Systems Audits

1.1 The U.S. Geological Survey (USGS) is the external quality assurance laboratory for the NADP. One of its tasks is to administer the Interlaboratory Comparison Program for NADP/NTN. Refer to the following USGS webpage for more information: http://bqs.usgs.gov/precip/interlab_overview.php.

1.2 The CAL participates in several external interlaboratory comparison programs as well.

- The World Meteorological Organization/Global Atmospheric Watch (WMO/GAW) Quality Assurance Science Activity Centre – Americas (QASAC-Americas), http://qasac-americas.org/, sends three samples twice a year to participating laboratories. The CAL's identification number is 70003.
- The Environment Canada Proficiency Testing Program (ECPTP) National Water Research Institute (NWRI) has two studies a year made up of ten rain and soft water samples. There are studies for other constituents as well. The CAL's identification number is F053.
- The Norwegian Institute for Air Research (NILU), http://www.nilu.no/projects/ccc/intercomparison/index.html , sponsors one study annually of four precipitation samples. The CAL's identification number is 27.

1.3 On-site reviews of the CAL are conducted every three years by the NADP. For more information, see the NADP QMP.

2.0 Sample Reanalysis

Reanalysis of samples is a critical part of the QC at the CAL. Samples are selected for reanalysis if they exceed the predetermined control limits for ion balance and specific conductance differences. See SOP DA-0067 for the Ion Percent Difference (IPD) and Conductance Percent Difference (CPD) reanalysis criteria.

2.1 For NTN, one percent of the total number of samples analyzed is selected randomly for reanalysis.

2.2 For AIRMoN, two percent of the total number of samples analyzed is selected randomly for reanalysis

3.0 Sample Validation

3.1 Screening Level (SL)

SL codes identify samples that were compromised through contamination or mishandling. The default SL value is blank, but if a sample is contaminated or mishandled in either the field or laboratory, or does not meet other CAL defined criteria for "field blanks," it is assigned a non-blank SL code.

3.1.1 Single-letter SL codes are defined as follows:

[Null] Default flag (sample has not been compromised through mishandling or contamination.)

F Samples with gross handling violations of standard operating procedures (SOPs) at the field site, sample lost in transit to CAL, or sample otherwise compromised before receipt at the CAL.

L Samples that have been mishandled by the laboratory (e.g., spillage, contamination, etc.)

C Samples with observable extraneous contamination, which exhibit anomalous chemistry relative to site history.

3.1.2 SL codes are assigned in the following order of precedence: F > L > C

3.1.3 NTN and AIRMoN Specific SL Code Scoring

In the NTN and AIRMoN, all wet (W*) samples are assigned an SL score as follows based on the history of all valid data (SP = Null, SL = Null) at each individual site:

Concentration vs. Site History	pH & Conductivity	All Other Analytes
> Maximum	1	2
≥ 90th percentile & ≤ maximum	0.5	1
≤ 10th percentile & ≥ minimum	0.5	0
< Minimum	1	0

3.1.4 Trace Samples

Trace (NTN type = T, AIRMoN type = WI) samples may have only sufficient volume for pH and conductivity analyses. Thus, the criteria for point assignment differ from W and WD samples. Only samples with CAL-measured pH or conductivity undergo the SL coding process. The program assigns a score of 5.0 to any trace sample that is ≤ 10th or ≥ 90th percentile from site history for pH and conductivity. Trace samples are never assigned a score higher than 5.0 even if both measurements were at or beyond the 10th or 90th percentile.

3.2 Sampling Protocol (SP)

3.2.1 SP validation characterizes each sample as to the extent that the bucket contents represent wet-only deposition.

3.2.2 Single-letter SP codes are defined as follows:

[Null] Default flag for samples for which the wet-side bucket is exposed only during the occurrence of precipitation and nominally during times when no precipitation is occurring.

U A sample which was exposed to dry deposition for an extended period of time. In the NTN, the threshold is set at exposure greater than 6.0 hours. In the AIRMoN, the threshold is set at exposure greater than 1.0 hour, or more than 5 openings during which no precipitation occurred.

B A sample from the wet-side bucket of the collector which has been exposed to all deposition (i.e. wet and dry) for the entire sampling period. This SP code is used only in the NTN.

Q Internal or external QA samples, i.e. not environmental samples.

4.0 Screening and Reporting Noncompliance with Data Quality Objectives

4.1 Each month, CAL staff participates in team meetings. These meetings discuss the results and evaluation of internal QA program analyses and any current laboratory conditions. Use of control charts, improvement of analyses, and any proposed method changes also are discussed.

4.2 The CAL QA Chemist prepares an annual report that discusses and reports overall laboratory data quality as well as all CAL QA activities during the calendar year. Before publication, the CAL QA report is peer reviewed and sent to the ISWS editor.

4.3 Documents required to support the QC/QA activities of the analytical laboratory consist of log books, SOPs, and this CAL QAP.

- The analyst's log book, maintained by each analyst, contains a record of working standards preparation, reference sample results, and daily notes. The analyst's log book may be combined with the instrument log book and the standard solution log book.
- The instrument log book is maintained at the workstation for each instrument and contains the maintenance schedule, performance record of scheduled and unscheduled maintenance, daily instrument settings and calibration data, and observations. The instrument log book may be combined with the analyst's log book and the standard solution log book.
- The standard solution log book contains all information pertinent to preparation of stock standard solutions, including all weights and volumes, confirmatory analyses, and a shelf life table. The standard solution log book may be combined with the instrument log book and the analyst's log book.
- A copy of the CAL QAP (this document) must be available in each laboratory, either electronically or as a hard copy.

5.0 Corrective Actions

Depending on the analytical or CAL procedure, different corrective actions must be followed. For example, shipping and receiving is handled differently than the analytical processes in the laboratory. However, each process is important and has specific corrective actions for noncompliance. It is the QA Chemist's job to determine which processes are out of compliance and the CAL Director's responsibility to implement changes necessary to correct them.

5.1 Overview

Performance and systems audits are a routine part of the CAL operations. External reviews are mandatory every three years (see NADP Quality Management Plan). Internal reviews may be conducted at any time upon the request of the NADP QA Manager, the CAL Director, the CAL QA Chemist, or anyone within the CAL. The following sections cover some of the corrective actions required at the CAL.

5.2 Equipment and Supplies

5.2.1 When project specified equipment and supplies cannot be obtained, equivalent replacements must be located. The new equipment specifications must be the same as or similar enough to be indiscernible from the original. For any supplies with which the samples may come into contact, a series of blanks must be obtained after cleaning to confirm that there will be no sample contamination. For other supplies, tests may be needed to confirm that new supplies are as good as old supplies. If they are not similar, another source of supply must be found.

5.2.2 Routine confirmation of supply cleanliness is performed on supplies in the laboratory. If any buckets, lids, or bottles selected for random contamination checks are determined to be contaminated, the contaminated bucket, lid, or bottle is visually inspected to ascertain if the contamination is obvious as not being thoroughly cleaned. If the bucket, lid, or bottle is severely contaminated or the structural integrity of the supply is compromised, it is discarded.

5.3 General Laboratory

5.3.1 Good laboratory practices must be adhered to at all times. If analytical standards are not prepared in Class A glassware, standards are discarded and remade, and all samples analyzed using those standards are reanalyzed. If good laboratory practices are not kept or any unapproved supplies are used, the samples, standards, reagents and any other laboratory process affected must be flagged, discarded, and/or reviewed by the QA Chemist and/or the CAL Director and samples flagged accordingly.

5.3.2 If the pipettors used to measure liquid standards for dilution are not checked for precision and bias before use or are more than 10 percent above or below the expected values when checked with the analytical or semi-micro balance, then the standards made with these pipettors are discarded, and all samples analyzed using these standards are reanalyzed. New pipettors are purchased and checked and/or the old pipettors are returned to the manufacturer for recalibration and cleaning.

5.3.3 If DI water used for making the standards is less than 18.0 Mohm-cm (ASTM Type I water), the standard is discarded and any samples measured with this sample are reanalyzed when a new standard made with ASTM Type I water becomes available.

5.4 Sample Processing

Sample processing corrective actions are described in the SOPs specific to that process. The samples arrive at the CAL and are processed according to the SOPs. For details on the corrective actions needed, see the appropriate SOP.

5.5 Instrumentation

5.5.1 Service maintenance agreements, preferably with the instrument manufacturer, are purchased when possible. All recommended servicing of the instruments is done according to the manufacturer’s suggested time schedule.

5.5.2 For instruments without service maintenance agreements, calibration of the electronic components is verified daily using QC samples and standards; any problems are reported to the CAL QA Chemist. The chemists, the CAL QA Chemist and the CAL Director determine whether the instrument is still within manufacturer’s specifications. If not, troubleshooting procedures are implemented, and manufacturer’s technical support services are utilized.

5.5.3 Preventative maintenance/service keeps instruments operating within required accuracy limits. Instruments that are not maintained to perform within requirements cannot be used for sample analysis until they are operating properly. If routine maintenance by analysts does not correct instrument problems, the company service representative must be contacted. Instruments that are taken out of service for repairs must be clearly defined, appropriate users alerted, as the QA Chemist notified.

5.5.4 Major changes to analytical methods and instrumentation require approval by the NADP. Whenever new methods or instruments are introduced, there must be extensive comparisons with the old and the new to confirm that the two methods provide comparable results. The new method or instrument, to be accepted, must equal or exceed the old method in all aspects: bias, precision, and detection levels. It is the NADP policy to keep current with analytical techniques without sacrificing bias, precision, and detections limits. All changes in analytical techniques must be approved by the NADP following written procedures for new method validation protocols. See the NADP QMP for details.

5.6 Analytical Procedures

Instrumental analysis procedures determine whether the instruments are working correctly and that standardization or calibration of the instruments is correct.
SOPs are in place for all sample chemical analyses for AIRMoN, NTN and AMoN. These SOPs contain detailed information on analytical problems to avoid and suggestions for corrective actions when problems occur. Corrective actions specific to each SOP are included in that SOP.

5.6.1 Quality Control Samples

5.6.1.1 No analysis can be made if at least two Quality Control Samples (QCS) are not measured after calibration or standardization. If any single measurement of a reference sample measured to verify correct operation is outside the control limits (3σ), all analyses of samples ceases and corrective action is taken. If the instrument cannot be stopped because of programming constraints or other reasons, and analyses on that instrument must continue, the results from that run may not be reported until corrective action is taken and, when necessary, reanalysis of the samples with the system in control is complete. When instrument constraints allow, a second reference sample may be measured immediately following the out-of-control reference sample to confirm or negate whether the instrument was out of control. If this reference sample is also out of control, the instrument is restandardized and all samples since the instrument was in control, i.e., when the last reference sample measured was in control, must be reanalyzed. Any instrument adjustment made to bring the QA check sample into control requires complete restandardization or calibration verification. If a new solution of the check sample results in a reading within control, no further action needs to be taken.

5.6.1.2 When QCS analyses do not conform with the DQOs, the analysis method must be examined to determine if a change in procedure has caused this difference. If there is noncompliance with DQOs, the sample or samples in question must be reanalyzed. The QA Chemist contacts the analyst to check data for accuracy and for transcription errors. If this is not the problem, or if the system was out of control (analytical check samples were not within specified control limits) during the analytical process, the analyst is asked to reanalyze the samples. The CAL Director is notified of the problem and ensures that corrective action has been taken.

5.6.2 Standards

5.6.2.1 If the standards used have not been confirmed using both of the methods in Section B of this QAP, then all analysis must stop until the standards are confirmed. Any sample analyzed before confirmation of standard concentrations is completed must be reanalyzed after confirmation is obtained.

5.6.2.2 Standards should be given an expiration date and not used beyond this date.

5.6.3 Control Charts

5.6.3.1 Control charts are automatically generated for each QCS analyte in LIMS. The true or expected value of each analyte for each solution is determined before the sample is used as a QCS. The average concentration for each analyte in each QCS is determined by 7-10 replicate analyses of the solution. Warning limits are two times the standard deviation and the control limit is three times the standard deviation. The warning and control limits are plotted and form the basis of the control charts. The standard deviation is estimated using control charts and blind sample standard deviations from previous years. The LIMS updates the control charts each time the analysts send data to the LIMS. The date of the analysis is also recorded on the control. The LIMS maintains a record of the analyst operating the instruments each day.

5.6.3.2 Analysts have primary responsibility in reviewing QC charts. If it is determined that there is a potential bias for that analyte (more than 5% of measurements within the warning range), then the analyst must determine why this bias has developed. The analyst, with the help of the QA Chemist and the CAL Director, as needed, determines the corrective action to be taken.

5.6.3.3 Some possible checks that can be made to determine why the system appears to be out of control are:

- The reference solution must be checked for contamination.
- The reference solution must be checked against a certified standard.
- Note: currently the National Institute of Standards and Technology (NIST) does not make certified simulated rain standards. Other companies that make standards have proven to be unreliable in their target concentrations. Analysts may use

commercially available standards, but usually these need to be diluted to bring them within the concentration ranges of atmospheric deposition samples.

- A new bottle of reference solution must be measured to determine whether the same concentration is measured, in order to distinguish between a contaminated or improperly calibrated reference sample and instrument malfunction
- The instrument must be restandardized.
- New standards must be prepared or obtained for instrument standardization.

5.6.3.4 If none of the above procedures bring the instrument back into control, the instrument must be checked for mechanical, electrical, or optical problems.

5.6.4 Replicate Samples

If the difference between the replicate samples processed randomly during analysis or reanalysis exceed the required accuracy (Section B-4.1), the replicate and/or the original sample must be reanalyzed. If the original now matches the replicate, then the analyst must determine whether there was an error in the analysis, and if so, did that error affect any other samples. All samples affected must be reanalyzed and the data base corrected. If the replicate still exceeds the required accuracy, the analysts must try to determine if either the original or replicate sample was contaminated or was chemically changing. If no obvious error during analysis is found, the samples analyzed adjacent to the replicate may need to be reanalyzed to ensure that the problem was unique to the one sample.

5.6.5 Blind QCS

If the measured concentrations of the internal blind samples exceed the required accuracy (Section B-4.1), this bias in the laboratory analysis must be addressed. The reference sample values must be checked for bias and precision. Calibration or standardization of the instruments must be evaluated. If the problem persists, analysis must cease until the cause for the bias or precision problem is found and corrected.

5.6.6 Reanalysis Samples

For reanalysis samples (those identified for reanalysis due to IPD or CPD) if differences are found between the original analytical data and the reanalyzed sample, the difference must be investigated. Was the difference caused by an error in the laboratory? Was the difference caused by sample contamination? Was the difference caused by the sample changing over time? These questions must be addressed and answered before any edits can be made to the data base. If any cause for a difference other than chemical changes to the sample is found, samples adjacent to the sample must be checked and reanalyzed to ensure that the problem did not exist for adjacent samples. Targeted reanalysis samples must agree within the required accuracy (Section B-4.1), or an explanation of the difference must be sent to the QA Chemist.

5.6.7 Interlaboratory Comparison Samples

If the results from any interlaboratory comparison samples indicate a problem within the laboratory, those samples must be reanalyzed and the instrument and the calibration or standardization samples must be checked against a certified standard to verify that the instrument is operating properly and that the standardization or calibration is correct. Continual participation in and monitoring of interlaboratory studies ensures the CAL is producing analytical results comparable to the rest of the world.

D. Data Management Operations

1.0 Computer Hardware and Software

1.1 Computer hardware selection should be based on the project requirements for data storage, retrieval, and processing. Hardware purchased from approved vendors should have warranty periods consistent with industry norms. In selecting computers and peripherals, consideration also should be given to compatibility with existing hardware and software applications. The ISWS Information Management Committee and the Computer Services Coordinator should be consulted when selecting computer hardware.

1.2 Computer software should be purchased from an approved University of Illinois list or a list of authorized vendors, when possible. Software must be selected to ensure compatibility with the host hardware, existing applications and end of life support.

1.3 Internally developed software, including mathematical models, should be designed with input from all planned or potential users of the program(s). The software must contain adequate documentation clearly stating the purpose and limitations of the program and applications for which the software was developed. The author of the software must be identified, and a complete program listing of the source code must be available to users. All mathematical algorithms used in the software are described in a narrative description that accompanies the source code. Prior to use, newly developed software must be rigorously tested using predetermined acceptance criteria. Typically, this would involve running old and new versions of programs in parallel (where appropriate), to ensure that consistent results are obtained. Manual calculations must be conducted on test data sets to confirm the reliability of the software prior to routine use. Such calculations should be reviewed and/or replicated by a third person or party other than those involved in developing the programs.

1.4 Data management procedures are in place to ensure that data integrity is not compromised during data entry, electronic capture from automated instruments, or transfers between computers and databases. Written procedures to ensure the accuracy and reliability of computerized data products are described in task-specific SOPs developed for data verification purposes. Data verification methods shall include double entry of manually entered data and thorough data review procedures.

1.5 Data are compiled from sample receipt observations and measurements, sample submission forms, analytical measurements, and other information sources (e.g., telephone communications, e-mail, and faxes) to produce a reportable record for each sample. The CAL-maintained LIMS system consists of an SQL server relational database.

1.6 The CAL policy for record archives for data management is similar to the record archives policy for the laboratory. All field forms and raingage charts are sent to the PO once the data has been transferred. All correspondence with sites pertaining to data

updates or corrections is retained for two years after transmittal of final data to the PO and then discarded. Correspondence with sites concerning sampling gaps, site moves, siting variances, and subsampling is kept permanently.

2.0 Data Security

2.1 The CAL Database Manager ensures that all computers in regular use have full data backups performed at least once weekly. Backup media are used in rotation, with at least one copy maintained off site at all times. The integrity of backups is evaluated at least once each year to ensure the proper operation of hardware and software.

2.2 Paper records are maintained in a secure and environmentally controlled area during their required retention period. Records storage rooms are locked outside of normal business hours. All records must remain at the CAL, unless the Database Manager or CAL Director provide specific permission that they may be taken off site.

2.3 Each internally-developed LIMS program and algorithm is tested prior to deployment. The old and new systems are run in parallel, and data output is audited manually. Each module is tested to ensure expected response under the following data conditions:

- Acceptance of all valid sample data
- Rejection of all invalid sample data
- Rejection or flagging of all data outside of expected ranges
- Reporting of data with expected data notes and flags
- Resistance to inadvertent and unauthorized data changes

2.4 The Database Manager is the only staff member authorized to make direct changes once data are entered or uploaded in LIMS. Individual staff members edit data through data review interfaces. This automatically creates edits logs, which are structured as follows:

- Date of change
- Parameter
- Old value
- New value
- Reason for change
- Staff member submitting change
- Date change applied

2.5 All edits logs are periodically reviewed by the CAL Database Manager, the QA Specialist, or the CAL Director. All edits logs are stored and archived permanently in electronic format.

2.6 The ISWS maintains internal network security through network firewalls and campus-wide site licenses for virus protection software. The LIMS is only accessible through direct network wired access or virtual private networking (VPN) from authorized ISWS domain users. The LIMS data integrity is maintained through control of read/write/modification access for individual staff members, and automated logs of data changes. Individual computers for staff are secured through logins and passwords registered to the ISWS domain. ISWS computer security procedures are documented at http://www.isws.illinois.edu/staffonly/guidelines/compproc.asp.

E. Terms and Definitions

bias - a persistent positive or negative deviation of the measured value from the true value. In practice, bias is expressed as the difference between the value obtained from analysis of a homogeneous sample and the accepted true value.

data quality objectives (DQOs) - the qualitative and quantitative measures of data quality desired from a specific activity or program. DQOs may include characteristics of bias, precision, completeness, and representativeness.

environmental data - any measurements or information describing environmental processes, location, or conditions; ecological or health effects and consequences; or the performance of environmental technology. Environmental data include information collected directly from measurements, produced from models, and compiled from other sources such as databases or the literature.

management - those individuals directly responsible and accountable for planning, implementing, and assessing work.

management system - a structured, nontechnical system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for conducting work and producing items and services.

peer review - an in-depth assessment of the assumptions, calculations, extrapolations, alternate interpretations, methodology, acceptance criteria, and conclusions pertaining to specific work and of the supporting documentation by qualified individuals or an organization independent of those who performed the work.

quality assurance (QA) - an integrated system of management activities involving planning, implementation, documentation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

quality assurance plan (QAP) - a formal document describing in comprehensive detail the necessary QA, QC, and other technical activities that must be implemented to ensure that the results of the work performed will satisfy stated performance criteria.

quality control (QC) - the overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality.

quality management - that aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and other systematic activities (e.g., planning, implementation, documentation, and assessment) pertaining to the quality system.

quality management plan (QMP) - a document describing the quality system in terms of organizational structure, functional responsibilities of management and staff, lines of authority, and required interfaces for those planning, implementing, and assessing all activities conducted.

record - a completed document providing objective evidence of an item or process. Records may include photographs, drawings, magnetic tape, and other data recording media.

specifications - a document that states requirements and which refers to or includes drawings or other relevant documents. Specifications should indicate the means and the criteria for determining conformance.

standard operating procedure (SOP) - a written document detailing the method for an operation, analysis, or action with thoroughly prescribed techniques and steps; the officially approved method for performing certain routine or repetitive tasks.

technical review – an independent, in-depth analysis and evaluation that may include documents, activities, material, data, or items requiring technical verification or validation for applicability, correctness, adequacy, completeness, and assurance that established requirements are satisfied. It is also used to determine whether quality activities and related results comply with documented procedures such as SOPs and DQOs,