EN-CY-102

Laboratory Analytical Quality Control

Procedure Contains NMM ECH eB REFLIB Forms: YES ☐ NO ☒
Procedure Revision Type: New ☐ NON-Editorial ☒ Editorial ☐ TC ☐ Cancellation ☐

HQN Effective Date	**Procedure Owner:** **Title:** **Site:**	Michael Ponzo Manager, Chemistry RBS	**Governance Owner:** **Title:** **Site:**	Jeff Goldstein Fleet Chemistry and Environmental Manager HQN
03/26/2021				

Site	Site Procedure Champion	Title
ANO	Subrena Morris	Chemistry Manager
BRP	NA	NA
CNS	Guy Blankenbiller	Chemistry Manager
GGNS	Kevin Sanders	Chemistry Manager
IPEC	Timothy Higgins	Chemistry Manager
PLP	Michael Soja	Chemistry Manager
RBS	Michael Ponzo	Chemistry Manager
W3	Jeffrey Bourgeois	Chemistry Manager
HQN	Jeff Goldstein	Fleet Chemistry and Environmental Manager

For site implementation dates, see ECH eB REFLIB using site tree view (Navigation panel).

Site and NMM Procedures Canceled or Superseded By This Revision Rev 15
Process Applicability Exclusion: All Sites: ☐ Specific Sites: ANO ☐ BRP ☐ CNS ☐ GGNS ☒ IPEC ☒ PLP ☒ RBS ☒ W3 ☒

Associated PRHQN #: 2020-00467	**Procedure Writer:** Ryan Smothers
Contains Proprietary Information: YES ☐ NO ☒	

Revision Summary	
Location	**Description of Changes**
3.0.13	Added exceptions to use of statistical control chart limits. (NISP-201 rev 1)
4.0.3.g	Changed requirement to complete JFG in a timely manner to complete JFG in the time required by EN-CY-100 not to exceed 2 years (NISP-201 rev 1)
5.3.c	Removed requirement for shelf life extensions not to exceed 90 days (NISP-201 rev 1)
Attachment 5	Added attachment detailing list of NISP-201 exceptions.

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1.0 PURPOSE AND SCOPE

1. This document establishes consistent standards and practices for chemistry laboratory quality assurance and control. The paramount goal is to validate analytic data procedures for Entergy nuclear site chemistry laboratories.

2. Quality assurance (QA) of chemistry and radiochemistry results are necessary; the data generated by the chemistry laboratory are used to make decisions regarding public safety, reactivity management, nuclear safety, environmental safety and asset protection.

3. This procedure is applicable to all station or supplemental personnel that perform measurements of chemistry/radiochemistry key parameters in support of the safe and reliable operation of nuclear power plants. This procedure is not only applicable to personnel in traditional chemistry departments, it is also applicable to personnel involved in the analysis and reporting of key parameters that may be in other departments.

2.0 REFERENCES/COMMITMENTS

2.1 Performance References

1. Guidelines for Chemistry at Nuclear Power Stations, INPO-13-005, October 2013

2. Individual Site Off-Site Dose Calculation Manual (ODCMs)

2.2 Developmental References

1. The Chemical Analysis of Water, D.T.E. Hunt and A.L. Wilson, Royal Society of Chemistry, London, 1986

2. Quality Assurance of Chemical Measurements, J.K. Taylor, Lewis Publishers, 1987

3. Principles of Environmental Analysis, L.H. Keith, et.al., Analytical Chemistry 55:2210-2218

4. Handbook of Quality Assurance for the Analytical Laboratory, James P. Dux, Van Nostrand Reinhold Co., 1986

5. Statistical Techniques for Data Analysis, J.K. Taylor, Lewis Publishers, 1990

6. Manual on Presentation of Data and Control Chart Analysis, ASTM Manual MNL7, 6th Edition

7. Statistical Quality Control, E.L. Grant and R.S. Leavenworth, McGraw Hill, 1988

8. Quality Control and Industrial Statistics, Acheson J. Duncan, Richard D. Irwin, Inc.

9. Fundamentals of Analytical Chemistry, D. Skoog, D. West and F. Holler, Saunders College Publishing, 1969

10. Experimental Statistics, National Bureau of Standards Handbook 91, M.G. Natrella, United States Department of Commerce, 1963

11. Standard Methods for the Examination of Water and Wastewater

12. Federal Register, 40 CFR 136, Appendix B

13. Statistics for Analytical Chemistry, J.C. Miller, and J.N. Miller, 3rd Edition, Ellis Horwood PTR Prentice Hall, 1993

14. Limits for Qualitative Detection and Quantitative Determination, Lloyd A. Currie, Analytical Chemistry, Vol. 40, No. 3, March 1968

15. ASTM E-178, "Standard Practice for Dealing with Outlying Observations"

16. ASTM E-548, "Standard Guide for General Criteria Used for Evaluation Laboratory Competence"

17. NuclearIQ User's Manual, G.C.R.

18. INPO Draft Position Paper on Laboratory Quality Control

19. Reg. Guide 4.15 Rev. 1, "Quality Assurance for Radiological Monitoring Programs Effluent Streams and the Environment," February 1979

20. Reg. Guide 4.15 Rev. 2, "Quality Assurance for Radiological Monitoring Program Effluent Streams and the Environment", July 2007

21. ANI Information Bulletin 15-01, "Nuclear Liability Records Retention," May 2015

22. NRC Inspection Manual, Inspection Procedure 71124 Attachment 06, Radioactive Gaseous and Liquid Effluent Treatment (04/01/2016)

23. NRC Inspection Manual, Inspection Procedure 71124 Attachment 07, Radiological Environmental Monitoring Program (1/1/2016)

24. Chemistry Nuclear Industry Standard Process CY NISP 201, Chemistry Quality Assurance Program

25. Chemistry Nuclear Industry Standard Process CY NISP 101, Chemistry Standard Glossary of Terms

2.3 Commitments

1. None

2.4 Obligations

1. Per requirements ANI Information Bulletin 15-01, Rad Effluent and Rad Environmental records including calibration, interlab comparisons, and crosscheck results must be retained for the life of the facility form policy plus subsequent 10 years during which claims may be covered by the policy.

3.0 DEFINITIONS

1. Accuracy – A measure of the degree of conformity of a single test result, generated by a specific procedure, to the target value.

2. Administrative detection limit (ADL): A limit established by the Chemistry QC Program Owner (CYQCPO), higher than the Limit of Quantification (LOQ), for convenience in reporting that may be used as the LOQ.

3. Ascending / Descending points – 7 consecutive points ascending or descending.

4. Batch – A group of analyses (typically 10 or less samples) preceded or bracketed by QC checks. This may be bounded by the shift period.

5. Bias – Persistent positive or negative deviation of measured quantities from the accepted true value.

    - Persuasive Bias – A bias indicated by 7 successive points on one side of the control chart centerline greater than 1 standard deviation. Persuasive biases should be investigated and eliminated.
    - Non-persuasive Bias – A bias indicated by 7 successive points on one side of the control chart centerline less than or equal to one point above 1 standard deviation. This bias need not be investigated.
    - Significant Bias – A statistically significant deviation for measured quantities from the accepted true value. A significant bias is determined using a two-tailed Student's "t" test (5% significance), comparing the mean of the bias against the expected value.

6. Blank Matrix – A prepared sample that contains no added analyte and that matches the sample matrix as closely as practical.

7. Blank Sample – Actual sample to which no reagents have been added.

8. Calibration – The process of using standard reference material to establish a numerical relationship between an instrument response and the value of the parameter to be measured, also called standardization.

9. Calibration / Verification Checks – a combination of quality control (QC) activities performed on test stands or equipment to provide assurance of test or equipment operability between longer standardization intervals. Calibration checks typically utilize a known standard at a point near the system expected concentration, or a point mid-scale in calibration range. When validating methods two calibration checks may be used; one at a low concentration in the calibration range and one at a high concentration.

10. Calibration Standard – Primary or secondary standards of appropriate accuracy certified to a risk-informed pedigree (i.e. National Institute of Standards and Technology (NIST), American Chemical Society (ACS) grade, International Standards Organization (ISO) 17025, ISO 17034, ISO 9001). The bases for selection of a specific pedigree may be documented in the associated method development documentation or instrument notebook.

11. Chemistry Technician - Any analyst that perform sample preparation, chemistry/radiochemistry analysis, and reporting of a key parameter is considered a Chemistry Technician, regardless of title or department assignment.

12. Coefficient of Correlation/Linear Regression – Quality of fit of a linear regression equation to empirical data. An exact fit of empirical data would result in an r value of +/- 1.0000.

13. Control Limits – Limits shown on a control chart between which there is 99.7% probability (3 standard deviations) that a QC result will appear while the system is in a state of statistical control. On a control chart, they are the upper and lower limits used to signal that a procedure, method, or instrument is out of control.

    a. These are values at which action is required and data is no longer valid.

    b. Control limits may be manually or statistically derived.

        1) When they are statistically derived, they are the limit beyond which is improbable that a single point could lie while the system remains in a state of statistical control. Set at the centerline +/- 3 standard deviations.

        2) When manually derived, they are the limits that management has conservatively or conveniently predetermined to be the point that action is required and data is no longer valid.

        3) Control limits may be manually derived when control chart data is generated less than once per month, data is being collected to generate statistical limits (new methods), for balances, pH meters, conductivity meters, or when a method exhibits an unrealistically high degree of precision.

14. Duplicate Sample – A second sample obtained from the same source in a separate container to assist in the evaluation of sampling variance.

15. Double Failure - An occurrence where the submitted interlaboratory crosscheck result was outside of the provided limits and outside the +/- 3 standard deviations acceptance criteria.

16. Error – Difference between the true or expected value and the measured value of a quantity or parameter.

17. Expiration Date – Expiration dates are dates beyond which a chemical should not remain in use.

18. Instrument Response Parameter (IRP) – A parameter such as absorbance, peak area or height, blank reading, slope, etc. that is trended to ensure proper and consistent operation of test equipment.

19. Internal Standard – A material added to samples in known amounts to serve as a reference measurement, such as a spike or standard addition.

20. Interlaboratory – Analyses or processes performed by several laboratories. Used to ensure analytical accuracy for a "total" method (includes instrument, analyst, procedure).

21. Intralaboratory – Analyses performed within a single laboratory by several chemists. Used to ensure analytical accuracy of individual lab personnel.

22. Key Parameters - See EN-CY-102-01 Attachment 1.

23. LOD (Limit of Detection) – The lowest concentration reliably distinguished from an appropriate blank. LOD is calculated as three times the estimate of the standard deviation (Sx) of ten replicates of the blank.

24. LOQ (Limit of Quantification) – The lowest concentration that can be reliably detected at which some predefined goals for bias and precision are met. For example, concentrations at the LOQ might be expected to be within ±30% of the relative standard deviation 99 times out of 100. LOQ is calculated as ten times the estimate of the standard deviation (Sx) of ten replicates of the blank.

25. LLD (Lower Limit of Detection) – LLD is an *a-priori* measure of the detection capability of a radiometric measurement process based on instrument setup, calibration, background, decay time, and sample volume (NUREG/CR-4007). It is expressed as an activity concentration. Each site should refer to their ODCM for the appropriate determination of LLD.

26. Matrix - Known or unknown components of a sample, other than a target analyte, that may be in a given sample.

27. Matrix Matched - Preparation of standards with similar matrix materials that are contained in the unknown sample.

28. Mean - Arithmetic mean in Entergy laboratory QA/QC procedures. It may also be referred to as "average". The Arithmetic mean is the sum of a collection of a set of numbers divided by the number of members in the set.

29. Method Detection Limit (MDL) – MDL is the lowest concentration that can be obtained with 99% confidence the value is > 0 as defined in 40 CFR 136, Appendix B. MDL is calculated as 3.14 times the estimate of the standard deviation (Sx) of 7 replicates of the blank.

30. Minimum Detectable Activity (MDA) – For Radiometric Instruments, the MDA is the *a-posteriori* minimum concentration that a counting system detects.

31. Minimum Detectable Count Rate (MDCR) – LLD or MDA expressed as a count rate rather than counts.

32. Outlier - An observation or measurement that is significantly different than the other values in a sample set, as determined by conventional statistical method (e.g. Grubbs' Test).

33. Precision - Measure of the degree of agreement of replicate measurements under specified and repetitive conditions of the same parameter, expressed in multiples of a standard deviation.

34. Quality Control Check (QC Check) – A quality control measurement to ensure stable performance of an instrument using a material of known composition to evaluate a measurement process.

35. Quality Control Standard – Standards used to perform quality control measurements on instruments. The results of control standard analyses are plotted on control charts.

36. Radiometric Instruments – Instruments used to quantitatively measure the decay of radioactive material.

37. Recommended – Due to differences in site instrumentation, not all items in Attachment 1, Attachment 2, and Attachment 4 apply to all stations. Apply best practice approaches to completion of these recommendations.

38. Replicate - A technique where a single sample that is split into aliquots at the laboratory. Each aliquot is then analyzed for the same constituent(s) by the same method and the results compared. These replicates are useful in documenting the precision of the analysis process.

39. Shelf Life – The amount of time that a properly packaged, unopened, and stored standard will last without undergoing chemical or physical changes.

40. Spiked Samples – Spiking refers to addition of a known amount of standard analyte to a sample or a blank. The most common spiking technique involves analyzing a sample and then a duplicate of the sample with a known amount of standard added. Spiked samples are used to measure accuracy and interferences.

41. Split Sample – A replicate portion or sub-sample of a total sample obtained in such a manner that is not believed to differ significantly from other portions of the same sample.

42. Standardization – See Calibration.

43. Standardization Checks – See Calibration/ Verification Checks.

44. Standard Deviation – Analytical control limits will be calculated using this statistic. Standard deviation (Sx) is an estimate of “sigma” using a finite sample of analytical data population (where “sigma” is the standard deviation of the total population).

45. Stock Chemical – A commercially available laboratory chemical.

46. Warning Limits – The limits shown on a control chart within which most of the test results are expected to lie (within a 95% probability) while the system remains in a state of statistical control.

47. Verification/Validation – The process by which a sample, measurement method, or data are deemed useful for a specific purpose and meets some acceptance criteria.

4.0 RESPONSIBILITIES

1. Chemistry Manager is responsible for:

   a. Establishing the Quality Control Program

   b. Ensuring conformance to the Quality Control procedure

   c. Conducting performance monitoring to ensure the chemistry quality assurance program is effective.

   d. Chemistry Manager shall review all Quality Control Charts once per quarter.

2. Chemistry Supervisor is responsible for:

   a. Assisting in the implementation of the Laboratory QA/QC Program as delegated by the QA/QC Program Specialist

   b. A Chemistry Supervisor does not have the authority to override an analytical result voided by the QA/QC specialist.

3. Chemistry Specialist referred to as the QA/QC Specialist or designee(s), is responsible for overall implementation of the Quality Assurance/Quality Control Program including:

   a. Ensuring that analytic instruments and analytic methods are maintained, and their history documented

   b. Reviewing control charts and quality control data in a timely manner

   c. Ensuring that persuasive biases are investigated and eliminated

   d. Ensuring that when acceptance criteria are not met, or adverse trends occur that corrective actions are implemented

   e. Communicating QA/QC issues to the department on a periodic basis

   f. Coordinating activities to complete inter- and intralaboratory performance checks in a timely manner.

   g. Completion of the job familiarization workbook within the time required per EN-CY-100, not to exceed 2 years as required by NISP-201.

   h. Avoiding all involvement in any activity that would diminish the confidence of analytical results due to lack of competence or impartiality.

4. Chemistry Technicians/Radiochemists are responsible for:

    a. Performance of laboratory quality control program

        1) Instrumentation under control prior to reporting data.

        2) Performing corrective actions when acceptance criteria are not met

        3) Notifying Chemistry Supervision when:

            - Equipment is removed from service
            - Adverse trends are noted
            - Acceptance criteria are not met
            - Actions to correct problem fail

5.0 DETAILS

5.1 Precautions and Limitations

1. **IF** the analytical environment of the instrumentation is not properly maintained (excessive heat or cold, dust, high traffic times),
   THEN ENSURE instruments are in control before and after analysis. This must be performed prior to reporting or making operational decisions based upon analytical data.

5.2 Lab Technique

1. The following are general good practices for lab technique that should be considered in the implementation of the analytical quality control program at each station:

   - **RINSE** labware with reagent grade water a minimum of three (3) times prior to use
   - **FORBID** the introduction of spatulas, pipettes or other lab utensils into liquid stock reagent containers
   - **AVOID** using bottle caps for dispensing or pipetting stock solutions
   - **WEIGH** oven-dried or refrigerated reagents at room temperature
   - **AVOID** leaving stock reagents open to the atmosphere
   - **MEASURE** aqueous solutions at the bottom of the meniscus
   - **AVOID** siphoning fluids into the pipette bulb
   - **LOCATE** balances away from potential drafts, corrosive vapors, heat or vibration that could degrade performance (when possible)
   - **USE** volumetric glassware at room temperature only
   - **USE** Class 'A' glassware, calibrated glassware or calibrated micro-pipettes for volumetric measurements for sample dilutions or standard preparations except where leachate from glass can potentially affect methods
   - **CONSIDER** Class A glassware, calibrated glassware or calibrated micro-pipettes yet they are not required for gravimetric measurements
   - **CONTROL** dust in the laboratory to minimize the potential of sample contamination or possible instrument degradation
   - **MAINTAIN** room temperature stable as near as possible to 68°- 77° F for instrument reliability
   - It is permissible to **CREATE** a set of multi-point calibration standards by diluting a certified master standard.

Section 5.2(Continued)

- **PREPARE** standards from National Institute for Standards and Technology traceable standards, American Chemical Society reagent grade chemicals, or ASTM D1193, Standards Specifications for Reagent Water, International Standards Organization (ISO) or their equivalent. Other chemicals may be used if a written assessment based upon good industry practices is performed.
- **STORE** Sample containers intended for trace level analyses filled with deionized water or anticipated matrix (i.e. 2% nitric acid) to leach out potential contaminants.
- **USE** Gravimetric sample and standard preparation for ppb-level analyses unless otherwise allowed by procedure.
- Tightly **CAP** standards and reagents when not in use.
- When preparing matrix matched standards the matrix shall be analyzed to validate concentrations where possible based on aliquot required for validation and availability of analysis.

5.3 Control of Chemicals, Reagents, and Standards

1. Stock Chemicals

   a. **LABEL** and **STORE** commercially acquired laboratory chemicals according to procedure, preferably at room temperature or vendor recommendation.

   b. Chemical Receipt

   - **ENSURE** any new chemicals are approved per EN-EV-112.
   - **CONTACT** vendor if shelf life remaining is less than one year, as appropriate.
   - **ASSIGN** date of receipt
   - **ASSIGN** expiration date per vendor Section 5.3 Step 1.c
   - **LOCATE** appropriate storage location in accordance with the station's laboratory design and EN-EV-112 requirements
   - **DO NOT USE** standards or reagent if there is any sign of degradation

c. Chemical Expiration Surveillance

- **CHECK** chemicals that are used for analyses have not exceeded expiration dates.
- **ENSURE** chemicals past their expiration date are not used unless approved by the QC program owner, or Chemistry Manager / Supervisor with notification to the QC program owner.
  - Do not exceed the expiration date of a parent stock material for daughter standards.
  - Shelf-life extensions of reagents and standards shall be evaluated with current performance data, documented in the laboratory notebook, and labelled as approved.
- Chemical Expiration Assignment
  - **ASSIGN** expiration date based on manufacturer's shelf life for Stock Chemicals with manufacturer's shelf life
  - **ASSIGN** expiration date of five years from date of receipt for Stock Chemicals without manufacturer's shelf life unless the manufacturer states that an expiration date is not required, in which case it will be ten years.

2. Monthly Stock Chemical Expiration and Inventory Surveillance

   a. **INVENTORY** all stock chemicals.

   b. **REVIEW** stock chemical expiration dates.

   c. **DETERMINE** quantities remaining.

   d. **IF** chemicals are expired OR expire within 30 days, **THEN ORDER** new chemicals.

3. Weekly Prepared Chemical Expiration and Inventory Surveillances

   a. **INVENTORY** all prepared chemicals.

   b. **REVIEW** prepared chemical expiration dates.

   c. **DETERMINE** quantities remaining.

   d. **MAKE** new standards as required.

   e. **DETERMINE** if quantity remaining is sufficient.

NOTE

The following shelf life restrictions are derived from experience and are authorized by Chemistry Management:

- Prepared chemical control standards made daily are exempt from having to be marked with an expiration date
- Prepared chemical mixed standards will be marked to expire per the section below based on the expiration date of the most restrictive component

4. Prepared Standards

   a. **USE** the following guidance in the absence of suitable references or documented testing for setting the expiration dates of standards:

Standard Concentration	Time Before Expiration Date
100 ppm and above	12 months
> or = 10 ppm to < 100 ppm	3 months
> or = 1 ppm to < 10 ppm	1 month
> or = 100 ppb to < 1 ppm	1 week
less than 100 ppb	1 day

   b. **PREPARE** calibration standards from NIST, ACS, ASTM, ISO or equivalent.

   c. Clearly **LABEL** calibration standards and QC check standards.

   d. **PREPARE** any standard(s) made in house for performance checks or blind samples from a parent standard that has a different lot number than the traceable standard(s) used for the method calibration.

5.4 Laboratory Water

1. **MONITOR** quality of laboratory water used for analyses and reagent preparation on a regular basis.

2. **ENSURE** laboratory deionized water (i.e., reagent grade water) used in standard preparation has at least 18.0 megohms resistivity.

3. **DETECT** presence of ionic impurities using an ion chromatograph or equivalent.

4. **EVALUATE** reagent grade water for those parameters that may interfere with analyses performed by the laboratory. The following values are typical for reagent grade water:

Electrical conductivity	≤ 0.056 μS/cm at 25°C
Electrical resistivity	≥18.0 MΩ•cm at 25°C
Total organic carbon (BWR only)	<30 μg/L (ppb)
Sodium	<1 μg/L (ppb)
Chlorides and Sulfates	<1 μg/L (ppb)
Silica	<5 μg/L (ppb)

5.5 Instrument Calibration (Standardization)

Calibrations are performed to determine or set the relationship between an instrument's response and the concentration of an analyte. This section outlines the proper calibration of instruments and recommended frequencies.

5.5.1 Curve Based Analyses

1. **MAKE** calibration curves using of the calibration concentrations validated during method development and recorded in site procedures or instrument logbooks.

   a. **MAKE** calibration standards from a different lot or manufacturer than quality control/performance check standards, unless otherwise approved by Chemistry supervision as stated below.

   b. **IF** any of the following conditions apply:

   - Alternate lot not readily available from vendors
   - Alternate lot temporarily out of stock. An alternate lot should be used once available; calibrations should not have to be rerun.

   THEN separate container of the same lot may be used **AND** condition report generated.

2. **CONSTRUCT** linear curves with a minimum of three (3) points and have a linear regression value of > 0.995 unless limited by instrument software capabilities.

   a. A single point with an assumed zero may be appropriate for some non-critical analyses.

   b. Non-linear curves should be constructed with a minimum of five (5) points or follow vendor recommendations for special software requirements.

Section 5.5.1 Step 2 (Continued)

c. Exceptions relating to minimum number of points for the calibration curve are allowed where software limits the number of standards used or lacks non-linear algorithms.

d. Analyses such as titrations or conductivity measurements should be standardized per station specific procedures.

e. **IF** an analysis result exceeds the upper calibration point (ULQ, typically the highest concentration standard unless otherwise documented in method development)
THEN :

- **IF** another method exists that does not require dilutions to be performed,
THEN USE alternate method to analyze sample.
- **IF** no other method exists,
THEN dilutions may be performed but the dilution factor should be limited to minimize error introduction.
- **IF** dilutions are frequently required for plant analysis
THEN consideration should be given to developing an alternative method with appropriate calibration range.

5.5.2 Calibration and QC Check Frequencies

1. **PERFORM** Calibration and QC Checks in accordance with documented site specific frequencies or at the frequencies described in Attachment 1, Recommended Bench-Top Instrument Calibration and QC Check Frequencies and Attachment 2, Recommended On-Line Instrument Calibration and QC Check/Verification Frequencies.

   a. Frequencies may be modified if it can be shown that the change in frequency does not adversely affect the accuracy of the results but should be documented in the instrument logbook.

   b. Site specific calibration frequencies should be risk evaluated considering vendor, ASTM, or other method recommendation and the analyzed parameter's operational risk.

2. **ENTER** QC data in the appropriate quality control database.

3. **PERFORM** short term trending review for anomalies.

4. **USE** certified standards when calibrating instruments.

5. **ESTABLISH** a QC check standard concentration between ten times the LOD and the mid-point of the calibration range or within one order of magnitude of the expected sample concentration.

6. **PERFORM** a QC check and **ENSURE** the instrument is in control prior to use IAW site implementing procedure or bracket the analytical result with a closing QC check for non-radiometric benchtop analysis for key parameters.

5.6 Control Charts

This section addresses specific requirements for the use of control charts. Control charts will be used to monitor measurement processes.

5.6.1 Control Chart Use

1. **PLOT** results of analysis of standards or spikes used to ensure instrument accuracy on control charts as they are analyzed. Nuclear IQ may be used for this purpose.

2. **ANALYZE** QC/performance checks in accordance with documented site specific frequencies or Attachment 1, Recommended Bench-Top Instrument Calibration and QC Check Frequencies

3. An instrument not in use does not require a QC check.

5.6.2 Control Chart Evaluation Criteria

1. **EVALUATE** control charts for criteria indicating a review is needed as follows:

   - One point outside the Control Limit.
   - Two successive points outside the Warning Limit.
   - Persuasive bias is indicated.
   - Ascending / Descending points are indicated.

   a. **IF** evaluation criteria are not met
   THEN first analyst action should be to recheck all calculations including QC standard preparations.

   b. **IF** evaluation criteria are not met and the calculations / QC standards are correct
   THEN CONDUCT an investigation to determine the cause of the failure as follows:

      1) Such an investigation may require a review of previous QC results to determine when the trend started and if there was an action which could cause the anomaly (i.e. calibrations, maintenance, environmental conditions, etc.).

      2) **IF** failure was due to two consecutive checks outside warning limit or one check outside the control limit,
      THEN method should not be used for the failed analyte until cause can be identified and corrected.

      3) An out of service instrument should not be placed in service before QC check is passed within two sigma.

   c. **DOCUMENT** actions taken in the instrument logbook.

5.6.3 Evaluating Quality Control Responses

1. Attachment 3, Quality Control Check Data Entry Flow Chart contains a flow chart to assist with evaluating quality control responses.

2. **IF** chemistry instrument is out of control, not in service or otherwise unavailable, **THEN PEFORM** either of the following:

   a. **AFFIX** signage in an obvious manner to prevent inadvertent use.

   b. **USE** alternate approved means by plant policy (Nuclear IQ restrictions or Work Management process).

5.6.4 Control Chart Review

1. **PROVIDE** periodic independent review of all control charts in addition to the routine checks for anomalies and trends performed.

   - **IF** response data are produced daily, **THEN REVIEW** monthly.
   - **IF** response data are produced weekly, **THEN REVIEW** quarterly.
   - **IF** response data are produced at any greater frequency, **THEN REVIEW** when 20 points of data are available.

   a. Reviews should include whether anomalies have been correctly identified, investigated, and documented.

   b. Known causes from investigations should be considered for continuing training.

   c. Recurring anomalies should be investigated to determine whether the methods and/or procedure(s) need revision.

   d. Chemistry Manager shall review all Quality Control Charts once per quarter.

2. **PERFORM** periodic review of control charts to:

   a. **CONFIRM** statistical limits are still valid.

   b. **DETERMINE** if tolerance charts should be statistically based.

NOTE

Control charts should not be regenerated frequently as this may mask instrument performance issues.

3. **IF** Kolmogorov-Smirnov (K-S) Test indicates the periodic check distribution is not normal,
   THEN periodic check data cannot be used for statistical analysis.

4. **IF** K-S test indicates non-normal distribution,
   THEN tolerance limits should be considered.

5. **IF** student t-test indicates the mean of a periodic check is significantly different from the original mean,
THEN this should be trended, or the control chart regenerated.

6. **IF** Fisher F test indicates the standard deviation of a periodic check is significantly different than original standard deviation
THEN this should be trended, or the control chart regenerated, and new LOD/LOQ/MDL determinations performed.

5.7 Instrument Maintenance and Performance Trending

Instrument maintenance and performance trending helps ensure the operation of an instrument is correct and the values produced are consistent and accurate. Trending also helps detect problems before they have an adverse effect on daily laboratory operations.

5.7.1 Instrument Logbook

1. Instrument Logbooks should be maintained electronically for each instrument group, instrument, or method as appropriate. The maintenance record should include the following information:

   a. Documentation of any downtime, troubleshooting, repairs, upgrades, preventative maintenance, vendor maintenance, and electronic calibrations

   b. Documentation of downtime and repairs should include the following:

      - Time and date a problem was discovered
      - Who discovered the problem
      - Description of the problem
      - What maintenance activities were performed to fix the problem
      - Who performed the maintenance activities
      - Time and date instrument returned to service

   c. Low level contamination events are also to be recorded in the instrument logbook.

   d. Instrument logbooks should track preventive maintenance, reactive maintenance, out of service declarations, trouble shooting, etc.

5.7.2 Preventive Maintenance

1. Each instrument should be included in a manufacturer recommended preventive maintenance program to ensure that the instrument is functioning reliably.

2. The preventive maintenance program should include the following elements:

   a. A mechanism for scheduling preventive maintenance to ensure performance within the specified frequency

   b. Review of equipment deficiencies or failures to update preventive maintenance scheduling

   c. Documentation of preventative maintenance in the instrument logbook should include:

      - Time and date performed
      - Who performed the maintenance activities

   d. **WHEN** an instrument is removed from service for any reason, **THEN** an "Out of Service" or similar tag should be affixed to the instrument or placed in turnover log and the condition of the equipment to alert the user not to use the instrument

5.7.3 Performing Trending

1. See Attachment 4, Recommended Instrument Response Parameters, for recommended guidance on instrument parameters to monitor and their frequencies.

2. Each instrument's performance should be monitored via instrument performance parameters (IRP, i.e., millivolts, absorbance, blank response, peak area/height, etc.) in addition to QC check or verification data.

3. IRP's should be used as diagnostic tool to monitor instrument performance and not to validate or invalidate data results.

4. Once sufficient data has been generated, acceptance criteria may be determined that can be used to initiate troubleshooting or maintenance.

5. Any corrective action should be based on the judgment of the user and documented in the instrument logbook.

5.7.4 Instrument Selection

1. **SELECT, PURCHASE, INSTALL** and **PLACE IN SERVICE** instruments that meet needed *precision*, *sensitivity* and *accuracy* requirements, to analyze key parameters in the expected range of interest and in the expected *matrix* IAW *EN-CY-101,* Chemistry Activities.

5.8 Data Handling and Reporting

The purpose of chemistry data management is to ensure that chemistry data is properly calculated, recorded, reviewed, and controlled. This section addresses specific issues associated with data management.

5.8.1 Computer Calculations

1. Any computer program which converts raw data to a final value should have the appropriate documentation on file for verification.

5.8.2 Data Reporting

NOTE

Do not report method uncertainty with analytical results unless specifically required by report governance documents (i.e., Reg. Guide 1.21, etc.)

1. The number format for an analytical result will be defined in the station procedure/policy or chemistry data management system.

2. The number of significant figures may be derived as follows:

   a. **COMPLETE** all calculations without rounding then round the result to the number of significant figures in the least precise contributing factor

   b. Final zeros after a decimal point are significant

   c. Zeros before a decimal point with non-zero digits preceding them are significant

   d. **IF** all digits preceding a decimal point are zero,
   THEN the zeros after the decimal point but preceding other non-zero digits are not significant

   e. In rounding off values to the correct number of significant figures, add one to the last digit retained if the following digit is 5, or as indicated by software

3. Reporting of Low Level Data:

   a. **IF** a method intends to determine the presence or absence of an analyte, **THEN** a response less than the LOD is reported as absent and a response equal to or greater than the LOD is reported as present

   b. **IF** method intends to determine a quantitative result **AND** method produces a response less than the analyte' s LOQ or MDL
   THEN REPORT LOQ or the MDL as the result preceded by a "<".

   c. **IF** method intends to determine a quantitative result **AND** method produces a response equal to or greater than that analyte' s LOQ or MDL
   THEN REPORT the response to the correct significant figure.

Section 5.8.2 Step 3 (Continued)

d. For radiometric data counts less than or equal to the a priori LLD, MDA, or the MDCR, **RECORD** instrument LLD, MDA, or MDCR as the result preceded by a "<".

4. Reporting of Low Level Data for quality control parameters:

a. Values will be recorded as analyzed, whether negative, zero or positive with the appropriate coding.

b. All quality control results (unless it is immediately recognized as a mistake made by the Chemist such as wrong standard or wrong dilution in standard preparation) should be logged with appropriate comments for abnormal results.

c. Data from R&D and "information only" techniques will be designated as "Information Only." These analyses may not have established QC program requirements.

5. Data Recording

a. Raw data may be recorded on the appropriate analytical results form, an area logbook or directly into the computer database. An intermediate form can be used.

b. Data entered into the main database should be defensible by the QC program and can be edited if a data reviewer finds an error.

1) A comment on the edited point after validation should include the justification for the change.

2) After validation, the chemistry database will retain a record of changed values in a retrievable form.

5.9 Offsite Analysis

1. Laboratories performing analysis of samples from the nuclear stations should be on the Entergy Qualified Suppliers List (QSL) and have a laboratory quality control program commensurate with the station's program.

2. In cases where environmental regulatory required analyses are performed, the outside laboratory shall be certified by the federal or state agency which requires the analysis.

3. **MAINTAIN** a chain of custody (COC) when transferring a sample to an offsite laboratory.

4. Vendor Laboratories

   a. It is the responsibility of the Nuclear Independent Oversight (NIOS) Department to evaluate the effectiveness of the vendor laboratories' quality control program.

   b. In cases where analysis is to be performed by a vendor laboratory that does not have a satisfactory quality control program, authorization to utilize this laboratory must be approved by the station Chemistry Manager from which the sample is being obtained.

      1) Chemistry Manager shall then have the responsibility to ensure that tests performed by the vendor laboratory meet the quality standards as applicable.

      2) Direct, on location witness of the test(s) being performed by a member of Entergy chemistry staff is an acceptable method.

   c. Specific requirements for vendor laboratories include the following:

      1) For regulatory required analysis, vendor laboratories shall be certified for the specific analysis performed by the regulatory agency which requires the analysis

      2) Refer to ASTM E548 for guidelines to be used for evaluating vendor laboratories

      3) All vendor audit reports and responses should be documented and maintained by the NIOS. These records will be used to assess whether contracts with the laboratories should be suspended, continued, and/or renewed.

      4) Requirement to have Interlaboratory cross check samples analyzed by applicable vendor lab, for samples analyzed by vendor lab, may be met by one nuclear site in the fleet.

5.10 Program Oversight

1. **CONDUCT** a formal review meeting of the laboratory quality assurance program annually.

   a. **ENSURE** minimum quorum includes the QA program Specialist, a line supervisor, and Chemistry Manager or designee.

   b. **DISCUSS** as a minimum:

      - Trends of the last five interlaboratory crosscheck results,
      - Control charts for key parameters,
      - Status of intralaboratory crosscheck results,
      - Status of CY QA related actions.

c. **TRACK** actions from the laboratory quality assurance program review meeting. (Meeting minutes are not required).

2. **MAINTAIN** a replacement plan for hardware/software.

3. **ENSURE** offsite/vendor laboratories that analyze key parameters have a QA program that meets the requirements specified by the utility

5.11 Quality Controls for Laboratory Software

1. The Chemistry QC Program Owner must approve any software modifications that affect QC related calculations or affect an analytical method.

2. **ENSURE** laboratory software is included in cyber security controls as appropriate.

5.12 General Guidelines for Process Instruments

1. **IF** a process instrument does not meet performance criteria, not in service or otherwise unavailable,
THEN AFFIX signage in an obvious manner to prevent inadvertent use
OR use alternate approved means by plant policy (Nuclear IQ restrictions or Work Management process).

2. **MAINTAIN** sample stream temperatures at 25 +/- 5º C, for conductivity instruments and all instruments where temperature compensating firmware is not used.

3. **CHECK** all process instruments using a reference instrument or standard and compare to the larger of the acceptance criteria found in Attachment 2, annually.

6.0 INTERFACES

None

7.0 RECORDS

1. **MAINTAIN** records of radiometric instrumentation calibrations, source checks, instrument response parameters, and results.

2. **TRANSFER** critical data to permanent plant file per the Entergy Records Retention Policy.

3. **RETAIN** other records in predetermined locations for the appropriate retention time.

4. **RETAIN** records so they are not damaged, deteriorated or lost.

8.0 SITE SPECIFIC COMMITMENTS

Site	Document	Commitment Number or Reference	NMM Procedure Section/Step
ANO2	License Renewal Application, Appendix. B, Section B.1.30, 2CAN10032	P-17937 P-17938 P-17939	All

Attachment 1

Recommended Bench-Top Instrument Calibration and QC Check Frequencies

Method Type	Calibration Method	Calibration Frequency	QC Check Parameter	QC Check Frequency [Note 2]
Atomic Absorption and Plasma Emission/ICAP/ICP/ICP-MS	Multipoint curve or per vendor recommendation	Daily prior to use or per vendor recommendation	QC standard	Prior to use or with each batch or 1/shift
Balances	Verification with Class S weights or vendor calibration	Annually	Class 'S' weight measurements	At least monthly (2)
Conductivity – Dip cell	Single point KCl standard or comparison to certified reference cell	Quarterly or based on QC performance	QC standard or compared with reference cell	At least quarterly
Conductivity – flow cell	Compare to certified reference cell or prepared standard	Quarterly or as per vendor recommendation	QC standard or compared with reference cell	At least quarterly (1)
Gas Chromatograph	Multipoint curve or single point as appropriate.	Based on QC performance	QC standard	Daily prior to use or with each batch
Ion Chromatograph	Multi point curve	Quarterly or based on QC performance	QC standard	Daily when in use
Oil Content Analyzer	N/A	N/A	QC standard	Prior to use
pH Meter	At least 2 buffers	At least weekly or based on QC performance	QC buffer solution	Daily when in use
Pipette, mechanical	Gravimetric with deionized water	At least semiannually or based on QC performance	Gravimetric QC check with deionized water	Monthly or prior to use
Ion Selective Electrode	Multi-point curve	Quarterly or based on QC performance	QC standard	Daily when in use
Spectrophotometers	Multi-point curve	Based on QC performance	QC standard	Daily when in use or with each batch
Titration (auto)	1. At least 2-buffers for pH electrode 2. At least one standard for titer	1. At least weekly 2. At least monthly	QC standard	Daily when in use or with each batch
Titration (manual)	At least one standard for titer	Monthly when in use or based on QC performance	QC Standard	Daily when in use
Titration – Amperometric	Per vendor instruction	Based on QC performance	Total Residual Oxidant	Every other month

Attachment 1

Recommended Bench-Top Instrument Calibration and QC Check Frequencies

Method Type	Calibration Method	Calibration Frequency	QC Check Parameter	QC Check Frequency [Note 2]
Thermometers, bimetallic	Multi-point	Annual	Known reference temperature or reference thermometer	At least quarterly
Total Organic Carbon Analyzer	Multi-point	Annual	QC standard	Weekly or prior to use
Turbidity	Comparison to Formazin or equivalent primary standards.	Annually or based on QC performance	QC standard	Daily prior to use
X-Ray Fluorescence	Multi-point	Based on QC performance	QC standard	Prior to use
Flow Totalizer	Via reference totalizer or vendor performed	Once per cycle or as needed based on performance	Total flow check	At least annually
Gamma Spectrometer	Per EN-CY-110	Per EN-CY-110	Per EN-CY-110	Per EN-CY-110
Liquid Scintillation Counter	Quench Curve determination [Note 3]	Annually or based on QC performance	QC standard	Daily prior to use or each sample batch
Proportional Counter	Attenuation curve or voltage plateau	Based on QC performance	QC standard	Daily prior to use or each sample batch
Viscosity	Within range of viscometer	As needed	Kinematic Viscosity	As needed
Flash Point	Within range	As needed	Flashpoint performance check standard	As needed
Biodiesel Spectrometry	Within range	As needed	Biodiesel performance check standard	Prior to each batch sample

Notes:

1. For instruments with non-linear responses, the instrument vendor recommended correlation method or other standard methods should be used.
2. The frequencies listed in this attachment are required if a site specific frequency has not been established and documented. Site specific frequencies may be more or less restrictive provided they are risk evaluated and documented acceptable by the station chemistry supervision
3. IPEC paired observation can be utilized to satisfy this requirement.

Attachment 2 **Page 1 of 1**

Recommended On-Line Instrument Calibration and QC Check/Verification Frequencies

Instrument	Calibration Method	Calibration Frequency	QC check/ Verification Method	QC Check/ Verification Frequency	QC check/ Verification Acceptance[Note 1]
Conductivity	Calibration solution or reference cell	Based on QC performance	QC standard solution or reference cell	Weekly for process control, monthly for diagnostic	0.02 µS/cm or 10 percent
Hydrazine	Reference value or vendor recommendation	Based on QC performance	Grab comparison	Weekly	4 ppb or 10%
Hydrogen	Calibration standard introduction or reference probe	Quarterly or based on QC performance	QC standard introduction or reference probe	Monthly	10%
Ion Chromatograph	Multi-point calibration	Based on QC performance	QC standard introduction	Daily	Control charted
Oxygen	Vendor recommendation or vendor performed	Monthly or based on QC performance	Grab comparison or reference probe	Monthly	2 ppb or 10%
pH	Multi-point calibration	Based on QC performance	Grab comparison or reference probe	Weekly for process control, monthly for diagnostic	0.10 pH units
Silica	Blank and single-point calibration	Quarterly or based on QC performance	QC standard introduction	Weekly	Control charted or 1.0 ppb or 10%
Sodium	Calibration standard introduction or reference value	Monthly	QC standard introduction or reference value	Weekly	Control charted or 0.1 ppb or 10%
TOC	Vendor performed	Based on QC performance	Reference value or QC standard introduction	Weekly	Control charted or 5 ppb or 10 %
Gas Flow Totalizers/ Meters	Site process method or meter change out	Every 24 months (with allowance)	Daily check per procedure	Channel Check frequency each 24 hours	Per procedure

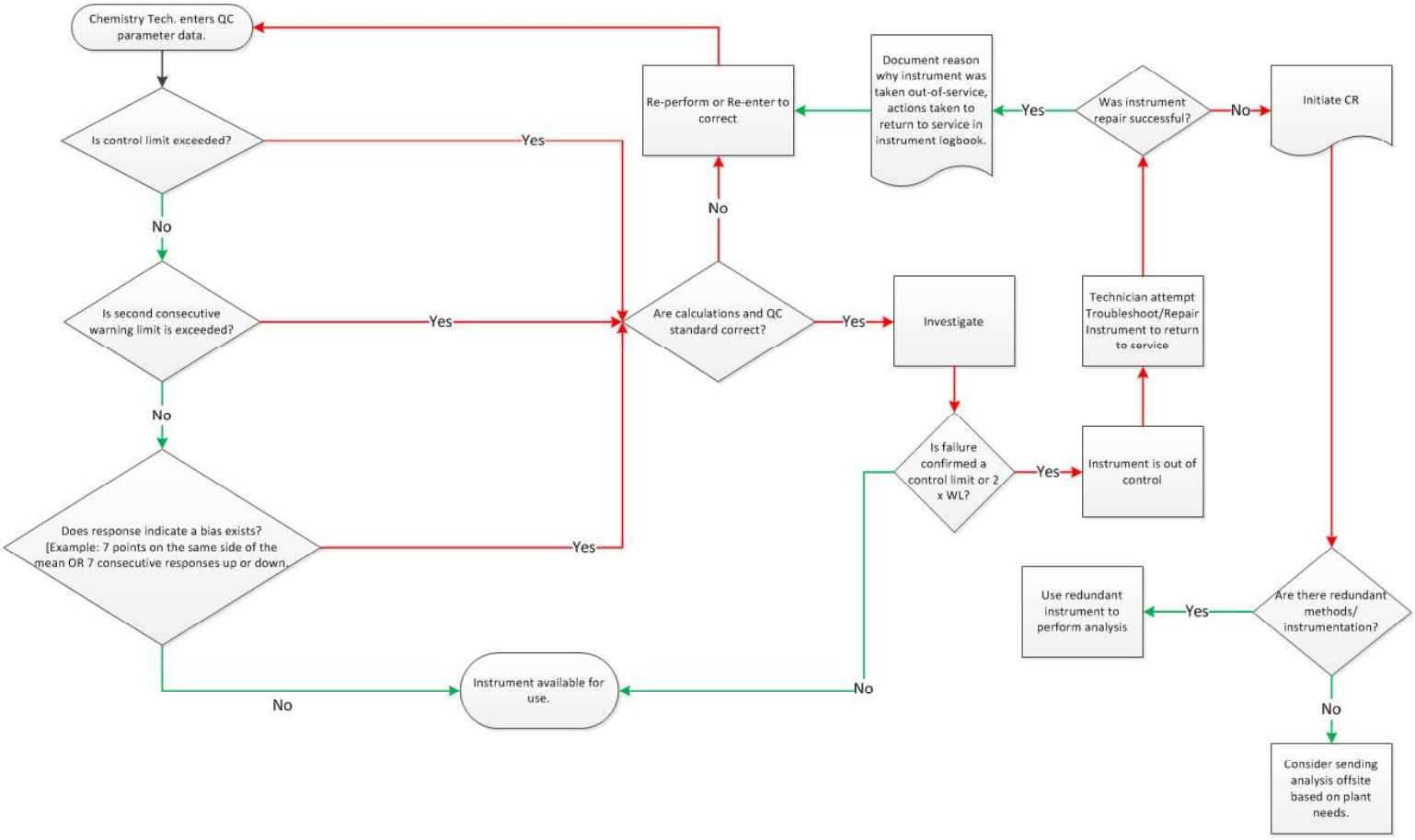
Notes:

1. Acceptance criteria is ppb range or %, whichever is larger
2. Indian Point ONLY – Radiological Environmental Controls, Part 1 of the Offsite Dose Calculation Manual Reference 2.1.2
3. The frequencies listed in this attachment are required if a site specific frequency has not been established and documented. Site specific frequencies may be more or less restrictive provided they are risk evaluated and documented acceptable by the station chemistry supervision

Attachment 3 **Page 1 of 1**

Quality Control Check Data Entry Flow Chart

Figure 1, Evaluation of Quality Control Response Data



Attachment 4

Recommended Instrument Response Parameters

Instrument	Response to Trend	Frequency
AA	Absorbance of QC standard	1/day when used or each QC check
Auto Titrator	Titrant normality or factor	Each calibration
Gas chromatograph	Peak height or area of QC standard and Retention Time	1/day when used or each QC check
ICP	Intensity of high calibration standard or alignment standard	Each calibration
Ion chromatograph	Peak height or area of QC standard and/or pump psi and background conductivity	1/day when used or each QC check
pH	Slope	Each calibration
Ion Selective Electrode	Millivolt reading of QC standard and Blank mV	1/day when used
Spectrophotometer	Absorbance of QC standard and blank absorbance for Silica	1/day when used
TOC	Millivolt reading of QC standard and Blank water	1/day when used
Gamma Spectrometer	FWHM, Channel, Energy, Background	Daily
Liquid Scintillation Counter	Quench parameter	1/day when used

Notes:

1. The frequencies listed in this attachment are required if a site specific frequency has not been established and documented. Site specific frequencies may be more or less restrictive provided they are risk evaluated and documented acceptable by the station chemistry supervision.

Attachment 5

NISP-201 Exceptions

NISP-201 Step	Exception Description	Justification
6.B.3	Step requires specific environmental controls in laboratories.	The QC program is adequate to discover environmental effects on instrumentation without the added burden of tracking laboratory environment conditions. This would add additional operational effort / cost and is expected to provide no benefit to laboratory quality. Individual site laboratories maintain environmental requirements necessary for instrumentation located therein.
6.C.2	Step requires establishment of hold times for all samples	Sites individually establish hold times based on governance (i.e. NPDES . SPDES) or other requirements (ASTM recommendations) in analytical procedures. Attempting to establish maximum hold times for all samples fleet wide would add additional operational effort / cost and is expected to provide no benefit to laboratory quality.
6.C.3	Step requires establishing guidance for sample preservation	Sites individually establish sample preservation requirements based on governance (i.e. NPDES . SPDES) or other requirements (ASTM recommendations) in analytical procedures. Attempting to establish sample preservation requirements fleet wide would add additional operational effort / cost and is expected to provide no benefit to laboratory quality.
6.C.5	Step requires all samples to be flushed at 3x the sample line volume.	Flush times are established at the site level. There are cases where studies have been performed to determine required flush times for representative samples which may conflict with this requirement. Establishing a fleet level requirement would override such justifications and provide no benefit to laboratory quality.
6.D.3	Step requires sites to analyze samples prior to their hold time.	See 6.C.2 exception justification.
6.M.6	Step requires no preparation of standards volumetrically if laboratory temperature is not maintained	See exception justification for 6.B.3

Attachment 5

NISP-201 Exceptions

6.N.1.b	Step requires use of significant figures	In use data management system does not provide a means to easily track the use of significant figures. It does allow establishment of maximum decimals which is currently employed. Compliance with this requirement would be difficult to enforce and provide no benefit to laboratory quality.
6.O.2	Step requires inline instrument sample stream temperatures to be maintained 25+/-3C	Historical evidence from Entergy plants shows that maintaining temperatures 25+/-5C as required by EN-CY-102 is adequate. Complying with this step may add additional operational effort with no benefit to laboratory quality.
7.C.II.6	Intralaboratory Acceptance criteria target for >7 participants will not target the consensus value defined as the interlaboratory mean by NISP-101. Entergy acceptance target with >7 participants will be the site's mean.	ISO 17025 and other industry peer-approved guidance (i.e. PPCAG Standard) suggest acceptance criteria based on data obtained within a site for intralaboratory proficiency testing. When sufficient data exists to use site statistics (>7 participants) then they will be used. When insufficient data exists the target of the interlaboratory mean is expected to be closer to the true value than the prepared value so that is used if available. Finally, if samples are prepared on site and insufficient data exists then the prepared value will be used.