Common Findings Related to Wet Chemistry Manual Method QC

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Common TNI Findings

- V1M2 4.13.2.3 Control of– Technical Records [NELAC Standard 5.4.12.2.3]

- When mistakes occur in records, each mistake shall be crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records shall be signed or initialed by the person making the correction. In the case of records stored electronically, equivalent measures shall be taken to avoid loss or change of original data.

- **The laboratory is obliterating errors in raw data records and the records do not include a reason for the change or the identity of the person responsible for making the changes.**

Common TNI Findings

- V1M1 5.1.1 Requirements for PT Sample Handling, Analysis and Reporting

- The laboratory shall analyze PT samples in the same manner as used for routine environmental samples using the same staff, sample tracking, sample preparation and analysis methods, standard operating procedures, calibration techniques, quality control procedures and acceptance criteria.

- **When PT sample testing is performed only the Quality Assurance Officer performs the analysis when the routine testing is consistently performed by the bench analyst.**

- **The laboratory reported PT Study results for USEPA Method 524.2 outside the calibration range. The values for several analytes were greater than the highest calibration standard. Results for routine environmental samples are not reported outside the calibration range.**

Common TNI Findings

- V1M2 4.13.2.3 Technical Records

- When mistakes occur in records, each mistake shall be crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records shall be signed or initialed by the person making the correction. In the case of records stored electronically, equivalent measures shall be taken to avoid loss or change of original data.

- **The laboratory raw data records contained mistakes in the records that were covered with liquid paper without any reason for the change noted.**

Common TNI Findings

- V1M4 1.6.3.1 and 1.6.3.2, Ongoing Demonstration of Capability (DOC)

- 1.6.3.1: The laboratory shall have a documented procedure describing ongoing DOC. The analyst(s) shall demonstrate on-going capability by meeting the quality control requirements of the method, laboratory SOP, client specifications, and/or this Standard. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.
- 1.6.3.2 This ongoing demonstration may be one of the following: acceptable performance of a blind sample (single blind to the analyst);
- another initial DOC; at least four (4) consecutive laboratory control samples with acceptable levels of precision and accuracy. The laboratory shall determine the acceptable limits for precision and accuracy prior to analysis. The laboratory shall tabulate or be able to readily retrieve four (4) consecutive passing LCSs for each method for each analyst each year;

The laboratory is not current with continuing DOCs for several wet chemistry analysts.

“Notes” in the TNI Standard

Example:

Note: Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624 or 5030/8260) would only require documentation for one of the tests.

CAN ASSESSORS ENFORCE THE NOTES????

Common TNI Findings

- V1M2, 4.2.8.4.i, Management

- The quality manual shall contain or reference: a list of all test methods under which the laboratory performs its accredited testing.

- **The laboratory's quality manual (or associated quality systems documentation) does not contain or make reference to a list of the wet chemistry test methods for which it is accredited.**

Common TNI Findings

- V1M2, 5.6.4.2, Documentation and Labeling of Standards, Reagents, and Reference Materials
- Documented procedures shall exist for the purchase, receipt and storage of consumable materials used for the technical operations of the laboratory.

- Records shall be maintained on standard, reference material, and reagent preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

- All containers of prepared standards, reference materials, and reagents shall bear a unique identifier and expiration date.

- **The laboratory is not documenting the preparation of the spiking solution used for wet chemistry analyses.**

Common TNI Findings

- V1M2 4.13.3 f.viii, Control of Records-Additional Requirements

- All information necessary for the historical reconstruction of data shall be maintained by the laboratory: analyst's or operator's initials/signature or electronic identification.

- **The laboratory is not consistently noting the initials of the analyst performing the analysis in the raw data records as required.**

Common TNI Findings

- V1M2 4.14 Internal Audits

- The laboratory shall periodically, and in accordance with a predetermined schedule and procedure, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the management system and this International Standard. The internal audit program shall address all elements of the management system, including the testing and/or calibration activities. It is the responsibility of the quality manager to plan and organize audits as required by the schedule and requested by management. Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited.

- **Although internal audits are being performed at the laboratory, the wet chemistry department has not been included in the annual schedule for the past four years. For the last internal audit performed of the wet chemistry department, there was no indication that any follow-up activities were performed.**

Common TNI Findings

- V1M2 4.13.1.1, Control of Records

- The laboratory shall establish and maintain procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. Quality records shall include reports from internal audits and management reviews as well as records of corrective and preventative actions.

- **The laboratory possessed various standard preparation logbooks and instrument maintenance logbooks. These logbooks were not uniquely identified and under the control of the Quality Assurance Officer (QAO).**

Common TNI Findings

- V1M2, 5.7.1 Collection of Samples

- The laboratory shall have a sampling plan and procedures for sampling when it carries out sampling of substances, materials or products for subsequent testing or calibration. The sampling plan as well as the sampling procedure shall be available at the location where sampling is undertaken. Sampling plans shall, whenever reasonable, be based on appropriate statistical methods. The sampling process shall address the factors to be controlled to ensure the validity of the test and calibration results.

- **The laboratory collects samples for client testing but the QM states that laboratory does not collect samples.**

Common TNI Findings

- V1M2, 5.8.7.1 Handling Samples and Test Items-Additional Requirements-Sample Receipt Protocols

- The laboratory shall implement procedures for verifying and documenting preservation.

- **The laboratory does not currently have a procedure in place for checking the pH of its aqueous samples upon receipt or for a check for the presence of (or the absence of) chlorine.**

Common TNI Findings

- V1M4, 1.7.2.c.i Technical Requirements- Continuing Calibration

- Instrument calibration verification shall be performed at the beginning and end of each batch.

- **The laboratory is not consistently ending the electrode batch testing for nitrates and chloride with a calibration verification standard as required.**

Common Method Findings

- V1M2 5.4.2, Selection of Methods
- The laboratory shall use test and/or calibration methods, including methods for sampling, which meet the needs of the customer and which are appropriate for the tests and/or calibrations it undertakes. Methods published in international, regional or national standards shall preferably be used. The laboratory shall ensure that it uses the latest valid edition of a standard unless it is not appropriate or possible to do so. When necessary, the standard shall be supplemented with additional details to ensure consistent application.

Common Method Findings

SM 4500-H B-11, Section 4 for pH; States that the pH meter shall be calibrated before each day of use.

- **The laboratory is not calibrating the rented multi-probe meter prior to use.**

SM 4500-CL G-11, Section 1, (Interferences) for chlorine: Compensate for color and turbidity by using sample to zero photometer.

- **The laboratory is using distilled water as a blank to zero the instrument prior to sample analysis.**

SM 4500-O G-11, Section 3.a (Procedure), requires that the laboratory calibrate dissolved oxygen instruments against air or air saturated water before each use.

- **The laboratory (i.e. field analyst) is not performing the required daily oxygen saturation test for the multi-probe meters used in the field. The laboratory must calibrate the dissolved oxygen instruments against air or air saturated water before each use.**

SM 2120 B Color by Visual Comparison Method, Section 6.c, Calculation: Report sample pH.

- **Raw data records show that the laboratory is not documenting the sample pH when performing color testing and therefore the pH of the sample is not reported with the results.**

Common Method Findings

- SW 846 Method 1010 (ASTM Flash Point) Ignitability: Section 6.5 (Apparatus) Barometer – with accuracy of ±0.5 kPa.

- Note 4- The barometric pressure used in this calculation is the ambient pressure for the laboratory at the time of test. Many aneroid barometers, such as those used at weather stations and airports, are pre-corrected to give sea level readings and would not give the correct readings for this test.

- **The laboratory did not have a barometer as part of the method-required apparatus. The laboratory must purchase a barometer and implement its use for ignitability testing.**

Common Method Findings

SM 2510B.2.a, 2011 editorial revisions, 22nd edition, Conductivity, Apparatus: Self-contained conductivity instruments: Use an instrument capable of measuring conductivity with an error not exceeding 1% or 1 µmhos/cm, and

SM 2510B.6, 2011 editorial revisions, 22nd edition, Conductivity, Precision and Bias: The precision of commercial conductivity meters is commonly between 0.1-1.0%.

- **The laboratory is using five (5) % for the acceptance criteria for the conductivity standard in use when the required precision is to be within 1% of the true value.**

Common Method Findings

SM 5210B-11: Examples

1. Not including 3 GGA bottles each batch
2. Not checking the initial temp. of the sample.
3. Not using 3 seed controls that meet the method.
4. Not following the formula in the method for final results.

Common Method Findings

- EPA Method 1664, N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry, Revision A and Revision B: Section 11.2.1 (Procedure) for oil and grease: Verify that the pH of the sample is less than 2 using the following procedure:

- 11.2.1.1 Dip a glass stirring rod into the well mixed sample.

- 11.2.1.2 Withdraw the stirring rod and allow a drop of the sample to fall on or touch the pH paper.

- NOTE: Do not dip the pH paper into the bottle or touch it to the sample on the lid.

- 11.2.1.3 Rinse the stirring rod with a small portion of n-hexane that will be used for extraction (to ensure that no extractable material is lost on the stirring rod). Collect the rinsate in the separatory funnel to be used for sample extraction.

- **The laboratory analyst stated that to determine the pH of oil and grease samples they are touching pH paper to the lid of the bottle.**

Common Method Findings

- EPA Method 1664 A, Oil and Grease, Section 9.5, Calibration Verification - Verify calibration of the balance per Section 10 before and after each analytical batch. If calibration is not verified after measurement of the analytical batch, recalibrate the balance and reweigh the batch.

- **The laboratory is not adhering to method requirements by verifying the calibration of the balance before and after each batch of samples.**