NUTRIENTS AND GENERAL CHEMISTRY: QUALITY ASSURANCE AND QUALITY CONTROL

1. SCOPE AND APPLICATION

1.1. The purpose of this Standard Operating Procedure (SOP) is to establish, maintain and document general quality control (QC) for the Nutrients and General Chemistry workgroups. The controls used are bias, precision, and detection limit statistics.

1.2. These procedures and calculations are used for all General Chemistry and Nutrients analyses: sediments, surface, ground and wastewaters. The procedures described in this SOP are general; more specific or stringent requirements are documented in the method and SOP for the relevant test.

2. QUALITY CONTROL

2.1. In general, for every batch of samples (max. 20 samples) digested and/or analyzed, the following quality control samples are analyzed:

A Laboratory Fortified Blank (LFB)

Digestion or Method Blank (MBLK)

One Matrix Spike

Matrix Spike Duplicate or Sample Duplicate

2.2. Accuracy.

2.2.1. Standardization:

2.2.1.1. Standards are prepared and documented in the Standard Preparation Tracker module of the Laboratory Information Management System (LIMS) program. Guidelines for the preparation of standards are documented in DEP SOP NU-042. Calibration curves are generated at the beginning of each analytical run or at the frequency specified in the method SOP. The curves are checked against certified or other independently prepared standards during each analytical run. Control standards are analyzed at least every 20 samples. A filtered blank is analyzed if any samples are filtered prior to analysis. The correlation coefficient for a standard curve should be 0.995 or greater and the recovery for each calibrant above the practical quantitation limit (PQL) should be 90 - 110 %. These recoveries verify the working calibration range with every calibration.

2.2.1.2 All analytical equipment must be calibrated on a regular basis (e.g. calibrate pipettes on a monthly basis). Guidelines for the calibration of auto-pipettes and dispensers are given in DEP SOP NU-039. The calibrations must be documented. All appliances

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used for storage or analysis must be monitored regularly and documentation must be maintained. See the supervisor for instructions on equipment calibration and monitoring. All glassware used to measure volumes for analytical purposes should be class A.

2.2.2. Spike Recoveries:

2.2.2.1. A representative sample is spiked with known quantities (preferably approximately 2 to 10 times the PQL) of the analyte before processing. Percent recoveries are calculated for the added analyte.

2.2.2.2. Matrix spike recoveries are indicators of sample matrix interference and contamination. The confidence range is set at 85 - 115 % for water matrices and 80 - 120 % recovery for soil matrices.

2.2.2.3. LFB recoveries are indicators of method failure and contamination. The confidence range is set at 85 - 115 % for water matrices and 80 - 120 % recovery for soil matrices.

2.2.3. Blanks:

2.2.3.1. Blank results are used to control contamination and to set minimum detection limits (MDL). Statistical calculations using historical data (at least 20 points) can be performed to establish the MDL for each analyte.

2.2.3.2. When samples are analyzed undigested, a blank is analyzed at the beginning, at least every 20 samples, and at the end of each run. Use deionized water for blanks and adjust the water to match the sample matrix (i.e. acidify, basify or add salt).

2.2.3.3. When samples are digested, use the digested blanks to obtain statistical information. The MDL is the concentration of analyte at which there is a greater than 99 % confidence that no analyte is present.

2.2.4. Practical Quantitation Limit:

2.2.4.1. Digested PQL standards are required with each tray of digested samples. The acceptance range for a PQL is 70 - 130 % recovery. Use the percent relative standard deviation (% RSD) of historical data (at least 20 points) to estimate the PQL. A 15 % RSD is optimum. If the % RSD is less than 15 then the PQL can be lowered.

2.2.4.2. If the % RSD is greater than 15 %, the PQL needs to be increased. With some chemistries there may be a method bias causing mean percent recoveries to vary consistently from 100 %. If the % RSD of 20 values remains 15 % or less (consistent bias), the PQL acceptance range can be adjusted to allow for the method bias.

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2.3. Precision.

2.3.1. Any time an instrument takes a replicate reading on a sample or if the sample is analyzed more than one time, the precision needs to be checked.

2.3.2. The relative percent difference (RPD) for two or more data points is measured either by the instrument or the analyst. When the average value of the concentration is above the PQL then the RPD must be less than 20 % in order to be acceptable.

3. QUALITY ASSURANCE

3.1. All data and quality control data are reviewed by a supervisor before being accepted as final. See DEP SOP NU-043.

3.2. All maintenance or analytical problems are reported to a supervisor. Document all instrument maintenance in the notebook assigned to the instrument. Also, document equipment calibrations (pipettes, balances etc.) in the assigned notebook or computer files. See the supervisor for instructions on how to document the instrument maintenance and equipment calibration.

3.3. Field sampling problems such as contamination, labeling, and holding times are reported to a supervisor and noted with the results using the NCR module of the LIMS program.

3.4. Results that are suspect for any reason are qualified and/or noted in the 'Ref. Method and Comment' line of the final report.

3.5. For methods that do not have electronic documentation of the time of analysis the start and end time of the analytical run should be documented by the analyst on the raw data. The end time of the run should be used as the time of analysis in the QC Manager. If samples are close to expiration the individual time of analysis should be noted and used for those samples.

3.6. Quality control results are documented electronically through the QC Manager module of the LIMS program.

4. CORRECTIVE ACTIONS

Contact a supervisor and report any non-compliant QC results. There are many different reasons for QC failures. In general for any failure associated with the initial calibration (failure of independent standard, correlation coefficient out-of-compliance etc.) the run should be aborted and the instrument should be recalibrated. For further information see DEP SOP NU-043.

5. REMARK CODES (SEE ALSO DEP SOP LB-027)

5.1. Sample results that are below the PQLs and/or MDLs have to be reported with a remark code (qualifier). Samples that are prepared and analyzed in duplicate are reported as an average and flagged with a qualifier. Samples that are analyzed

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after expiration must be reported with a qualifier. Samples associated with quality control failures may be qualified. Incorrectly preserved samples must be qualified. The QC Manager module of the LIMS program will assign most of these codes automatically (i.e. I, U, A). The Q qualifier and J qualifier for failed spike recoveries, RPDs, LFBs or CCVs are assigned automatically in the Data Review/Authorize module of the LIMS program. Appropriate comments for Q qualified data and non-automatically assigned J qualified data should be added in the QC Manager. There are default comments for the most common reasons for qualifying data.

5.1.1. Sample results that are below the MDL are reported as the MDL followed by the U qualifier.

5.1.2. Sample results that are below the PQL but at or above the MDL are reported with an I qualifier.

5.1.3. For samples that are poured out and analyzed in duplicate, QC manager will calculate the average and append the A qualifier.

5.1.4. Samples that are analyzed after expiration are reported with the Q qualifier.

5.1.5. For the QC sample in batches associated with failing QC, the result is reported with a J qualifier. All sample results in a batch with associated LFB failure are reported with a J qualifier. All sample results where the bracketing continuing calibration verification standard fails are reported with a J qualifier.

5.1.6. For samples that are incorrectly preserved, the result is reported with a Y qualifier. Make sure the preservation flag in LIMS is adjusted if the preservation issue was not detected at login but during analysis. Check with your supervisor before authorizing associated results.

5.2. Some conditions may require two or more qualifiers, use the list below to assign priorities (a is highest). Multiple codes may be assigned.

a U or I

b J

c Y

d A

6. REFERENCES

6.1. Quality Manual, Bureau of Laboratories, FDEP.

6.2. DEP SOP NU-043, Nutrients and General Chemistry Quality Assurance and Data Authorization Guidelines.

6.3. DEP SOP NU-042, Standard Preparation Procedure.

6.4. DEP SOP LB-027, Standard Operating Procedure for Reporting Qualified Data.

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6.5. DEP SOP NU-039, Calibration of Auto-Pipettes and Dispensers.

Appendix of Significant Changes

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