STANDARD PREPARATION PROCEDURE

1. SCOPE AND APPLICATION

This general procedure is followed when preparing standards used in the Nutrients workgroup. It describes labeling and rotation of purchased standards, standard preparation procedures, documentation of prepared standards, labeling of standard containers, rotation of prepared standards, and disposal of used and expired standards. Any specific instructions about standard preparation that appear in the relevant SOP for the method shall take precedence over these instructions. Also refer to DEP SOP LB-030 for instructions pertaining to purchasing and receiving laboratory chemicals and supplies.

2. DOCUMENTING AND LABELING OF PURCHASED STANDARDS

2.1. All shipping cartons containing standards for the Nutrients workgroup should be labeled with the date of receipt by the receiving/purchasing staff. The analyst is responsible for checking that the date of receipt is on the container and contacting their supervisor if it is not available.

2.2. Label all individual standard containers with the following information:

- Unique ID number
- Expiration date (see 2.3 for details)

All pertinent information about a standard is documented by entering it into the Standard Preparation Tracker module within the Laboratory Information Management System (LIMS). A label is also printed using this module.

2.3. If a chemical or reagent container and its certificate of analysis have no explicit expiration date, then,

(a) dry inorganic and organic solids are assigned an expiration date ten years from the day they were received in the lab;

(b) all concentrated inorganic acids and organic solvents are assigned an expiration date five years from the day they are received in the lab;

(c) all other liquid reagents are assigned an expiration date five years from the day they are received in the lab;

(d) shorter expiration dates than the ones listed above may be assigned based on historical experience and the discretion of the supervisor. They are documented in the applicable SOPs.

If an expiration date is provided by the manufacturer, it must be recorded on the container.

2.4 If available, a Certificate of Analysis for each purchased standard is maintained in the applicable analytical folder in the following directory:

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\\floridadep\data\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\RunData\MetaData for each analysis.

3. ROTATION OF PURCHASED STANDARDS

Use the oldest dated standard first and replace with a newer dated stock standard as necessary. Rotate all purchased standards within the specified shelf life.

4. STANDARD PREPARATION INSTRUCTIONS

Prepare standards as detailed in the relevant method SOP. The volumes prepared for the standards may be altered by the analyst as necessary. The final concentrations of the constituents of a standard must not be changed.

All standard preparations are documented in the reagent and standard logsheet (NU-RGTSTD). The logsheets are located at \\floridadep\data\DEAR\labs\lab3\Chem_data\Inorganic\Nutrients\RunData\MetaData for each analysis. Submit the updated logsheet as part of the analysis report folder. Make sure to use the latest version of the logsheet.

The work area and all glassware and equipment used for standard preparation should be scrupulously clean and organized. All containers should be appropriately labeled. The analyst should exercise utmost care to prevent contamination to standards. Pipets, spatulas, or any other objects should never be dipped directly into the stock containers. Instead an appropriate quantity of the stock should be poured into a clean intermediate container for further use. Standards should not remain uncovered for longer than necessary or left unattended without being properly secured.

Only the following volumetric measuring devices are allowed for use in standard preparation (see 2016 TNI Standard, EL-V1M2-2016 5.5.13.1):

- Class A glassware
- Calibrated mechanical devices (e.g. micropipettes, dispensers)
- Disposable or single-use volumetric equipment verified for each lot used

The analyst is responsible for ensuring that the above requirements are met, checking that all relevant calibration/verification records are correct and up to date.

If an oven is used for drying compounds, the analyst is responsible for checking that the oven thermometer calibration is up to date and that the temperature reading for the day(s) the oven is used is logged in correctly. When recording standard preparation in the statistic tracker, details of oven use should be provided under Additional Information. Standard ID, Oven unique ID, and time in and out of oven should be included, for example:

Additional Information: Standard 66125 dried in Oven 12. IN 9/25/20 4:50 PM, OUT 9/28/20 9:30 AM.

Only analytical balances are allowed for use in standard preparation. The analyst is responsible for ensuring that the balance is correctly calibrated on the day it is

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used and that the calibration range brackets all measurements taken. Measured weights should be entered to the Standard Preparation Tracker exactly as they appear on the balance readout, without rounding or truncation. The analyst is responsible for cleaning the balance after each use. Balance ID should be recorded in the Standard Preparation Tracker.

Results of all measurements should be recorded directly in the Standard Preparation Tracker. Recording weights/volumes elsewhere and later transferring them to the Standard Preparation Tracker is not allowed. The analyst should choose their work area so that a computer is within easy reach. If for any reason direct recording to Standard Preparation Tracker is impossible, this should be discussed with the supervisor and the records created during measurements permanently retained.

5. DOCUMENTING STANDARD PREPARATIONS

Standards are given a unique ID by logging them in to the Standard Preparation Tracker in the Laboratory Information Management System (LIMS). Labels can be printed from this module. Verification of stock and working standards is also documented in the LIMS Standard Preparation Tracker (see 8.3)

6. TRANSFERRING PREPARED STANDARDS

Discard old standard from container. Wash standard container and lid six times with deionized water and allow to dry. Rinse standard container with a small volume of prepared standard and transfer remaining prepared standard to container.

7. LABELING PREPARED STANDARD CONTAINERS

Label all prepared standard containers with the following information each time a standard is made:

- Unique ID number
- Identity
- Expiration date

Labels containing all pertinent information about a prepared standard are printed from the Standard Preparation Tracker in the LIMS after standard preparation is logged in.

8. ROTATION AND VERIFICATION OF PREPARED STANDARDS

8.1. The holding times of all prepared standards are documented in the applicable SOP.

8.2. Use the oldest dated working standard first and replace with a newer dated working standard as necessary. Rotate all working standards within the specified holding times.

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8.3 Make sure to prepare stock and working standards well before the ones in use expire. The newly prepared standards should be verified against the current ones and a standard reference material (SRM). For individual acceptance limits check the appropriate analysis SOP. Verification should be documented in the LIMS standard preparation tracker. Re-certification of standards needs approval from the supervisor.

8.3. Expired standards should be discarded as soon as possible after the date of expiration. Discarding standards should be recorded in the Standard Preparation Tracker. Any expired standards used for non-quantification purposes should be labeled as such.

8.4. If standards are prepared for method development purposes only they must be labeled "For Research Only" or equivalent.

9. DISPOSAL OF USED AND EXPIRED STANDARDS

9.1. Refer to the MSDS for information about chemicals being used.

9.2. Review the Laboratory Health and Safety Plan.

9.3. Refer to the individual chemistry SOPs for specific handling of standards. Some standards used may need to be treated as hazardous or treated prior to disposal.

REFERENCES

10.1. Laboratory Health and Safety Plan.

10.2. Quality Manual, Bureau of Laboratories, FDEP.

10.3 2016 TNI Standard.

10.4 DEP SOP LB-030, "Purchasing and Receiving Laboratory Chemicals and Supplies".

Appendix of Significant Changes

08/24/2010 Instructions for the verification of standards are added to section 5 and 8.

09/25/2018 Clarified storage location for certificates of analysis in section 2.

10/14/2020 Expanded Standard Preparation section.

12/20/2023 Section 2.3 was added to include instructions on assigning expiration dates in cases where none are provided by the manufacturer. The file paths are updated in section 2.4. and 4.. A reference to SOP LB-030 was added.

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