

APPENDIX C. PREPARATION OF QUALITY CONTROL SOLUTIONS

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C.1 Background

Quality control solutions (QCS) can be divided into two different categories (Clement, et al., 1997):

- 1) Synthetic QCS: Solutions prepared in deionized water using gravimetric measurements of reagent source materials or solutions prepared in natural precipitation with reagent source materials added to adjust the concentrations.
- 2) Natural Matrix QCS: Well-mixed natural precipitation that has been characterized by repeated testing over a specified length of time. Natural matrix QCS may be used as is or as the solute for synthetic QCS as in 1, above.

Using QCS to demonstrate that an analytical system is in control requires that these solutions be homogenous and stable. Chemical stability is essential so that a large data base of measurements is available from which to derive statistically stable results that are truly representative of the method.

Prepare QCS in volumes sufficient to last six months to a year. Some species such as carboxylic acids or orthophosphate are not stable and preparation of bulk QCS is not practical.

Synthetic QCS prepared in precipitation with reagent source material added has the advantage of allowing the laboratory to tailor specific species to the desired concentration range.

C.2 Preparation of Natural Matrix QCS

Collect and prepare precipitation in bulk quantities to use as a natural matrix QCS. Sterilization is critical to the long-term chemical stability of precipitation used for QC purposes.

- 1) Select a storage container large enough to hold a volume of solution intended to last one year. Large volume HDPE containers equipped with a stopcock are ideal.
- 2) Condition the storage container by filling it with deionized water and allowing it to stand for 24 hours. Empty the container, then rinse it three times with deionized water and shake out any excess water.
- 3) Collect and combine precipitation samples that have concentrations in the desired range. Refrigerate this bulk mixture at 4°C during this collection period.
- 4) When sufficient precipitation has been collected, follow these steps to stabilize the mixture:
 - (a) remove insoluble coarse material either by centrifugation or by allowing the material to settle to the bottom of the collection vessel and decanting the clear supernatant,

- (b) filter the supernatant through a 0.45 μm pore-size filter using a vacuum filtration device (Simser, 2016),
 - (c) vacuum filter the filtrate from (b) through a 0.20 μm pore-size filter, effectively stabilizing the filtrate by removing bacteria.
- 5) Add the stabilized filtrate to the pre-conditioned storage container, mix by inverting the container, and allow this natural matrix QCS to equilibrate for 24 hours before use.

C.3 Preparation of Synthetic QCS from Natural Precipitation Base

Choose one of two methods for preparation of synthetic QCS for the precipitation chemistry laboratory.

Method One: Prepare QCS that are specific to a particular instrument or analytical method.

Advantages:

- Control standards are tailored to better address the calibration range of a specific instrument or analytical method. For example, colorimetric methods tend to have a calibration range that is smaller than ion chromatographic methods.
- Standards can be prepared that avoid the potential for problems from reagents such as the acids that are added to stabilize metals in solution or for problems from other reagents that are not the target of the particular analytical method being tested.

Disadvantages:

- Each control standard requires a dedicated container, storage space, and labelling.
- Time is needed for preparation of each method-specific control standard.

Method Two: Prepare QCS that can be used for all analytical methods.

Advantages:

- Using a few solutions for all methods simplifies storage and ease of use.
- Use of a few solutions facilitates an evaluation of general laboratory quality indicators over time across all analytical instrumentation, methods, and personnel.

Disadvantage:

- If one chemical component deteriorates, the solution must be remade for all components and all methods must be reevaluated with the new control solution.
- Problems can arise from certain reagents, such as acids, that affect the calibration range.

In general, the sterilized natural matrix is tested for the species of interest and spiked with standard solutions to bring the concentrations into a target range. Standard solutions may be taken from standard stock solutions or may be purchased commercially. Suppliers are listed in [Appendix D](#). Control solutions using method one are described in each analytical method in the laboratory operations section. An example of method two is given in Table C.1 below.

Store control solutions in a container held at 4°C. If the container has a stopcock, draw and discard a small amount of solution to clear the stopcock, before drawing solution for use. It is advisable to draw solution from the storage container into a secondary container for use. This eliminates the need to repeatedly access the container, reducing the chance of contaminating its contents.

Table C.1: Example Control Solutions

| Component | Control Solution 1 | Control Solution 2 |
|---|--------------------|--------------------|
| SO₄²⁻ (mgL⁻¹) | 6.669 | 2.544 |
| NO₃⁻ (mgL⁻¹) | 3.811 | 1.744 |
| Cl⁻ (mgL⁻¹) | 1.157 | 0.463 |
| NH₄⁺ (mgL⁻¹) | 0.827 | 0.413 |
| Na⁺ (mgL⁻¹) | 0.751 | 0.300 |
| K⁺ (mgL⁻¹) | 0.611 | 0.204 |
| Ca²⁺ (mgL⁻¹) | 0.268 | 0.153 |
| Mg²⁺ (mgL⁻¹) | 0.310 | 0.186 |
| H⁺ (µL⁻¹) | 100 (pH 4.00) | 30 (pH 4.52) |
| Conductivity (25°C) (µS cm⁻¹) | 61.2 | 21.7 |

C.4 Calibration and Operational Tests and Procedures for Laboratory Apparatus and Tools

All laboratory apparatus and tools require calibration and operational tests. Standard Operating Procedures for each procedure are required. Documentation and retention of results is mandatory. The deionized water system requires calibration and service by a professional service representative. Other apparatus and tools can be calibrated by a laboratory technician. Several procedures for calibration and operational tests are described below.

Analytical Balance Calibration

Before weighing any chemicals, check the calibration and performance of the balance.

- i. Turn on the balance and allow it to warm up for a few minutes. Ensure that the area is free of drafts and the readings are stable before proceeding.
- ii. Check that the balance is level and clean. As needed,
 - a. level the balance by turning the feet of the balance until the leveling bubble is centered.
 - b. clean the balance of any residual chemicals using a soft brush and, if necessary, deionized water and a soft Kimwipe®.
- iii. Tare or zero the balance.
- iv. Use a NIST-referenced test weight to check the performance of the balance. Choose a test weight that is in the expected range of the substances to be weighed. Ensure that the test weights have been cleaned and verified by a certified provider.
- v. **Never touch the test weights.** Use tweezers to grasp and place the weight gently on the balance pan.
- vi. Allow the balance to stabilize. Record the weight in the laboratory logbook.
- vii. Remove the weight with tweezers and store it in its special container. NIST-referenced weights should come in a soft, dust-free box in which the weights cannot bump against one other. Store the set of weights in a clean and dry area.

Pipette Calibration

Pipette calibration procedures apply to hand-held or digital pipettes and also can be used for bottle-top dispensers. Use a calibrated analytical balance and Type 1 ultra-pure water with a resistivity >18.0 MΩ. Wear gloves.

- i. Place a weigh boat or small beaker on the balance pan. Tare the balance.
- ii. Draw and dispense an aliquot of deionized water into the boat or beaker.
- iii. Record the weight.
- iv. Repeat ten times **using a fresh pipette tip each time.**

- v. Establish standards for acceptable variability and deviation from the expected value. Use 1% in the absence of established limits.
- vi. Adjust or correct bottle-top dispensers by increasing or decreasing the stop line of the plunger. Ensure that the stop screw does not move around or slip.

Chemical residue must not be allowed to crust or cake on the glass plunger or in bottle-top dispensers. Use deionized water to clean the dispensers and rinse the plungers. If a reagent has crusted or caked onto the plunger, the plunger may break with the first use.

Pipette and Bottle-top Dispenser Leak Test

Leak tests apply to hand-held and digital pipettes.

- i. Use clean pipette tips.
- ii. Dispense the fluid for which the pipette is intended.
- iii. Draw up the fluid and observe the tip for ten seconds.
- iv. Repeat using ten different tips.
- v. **Tips must not leak.** Leaky tips may be attributed to a poor fit of the tip onto the pipette sleeve or faulty tips that may be deformed or poorly manufactured.
- vi. Document observations in the laboratory notebook. Take corrective action to eliminate leaks.

Check bottle-top dispensers for leaks.

- i. Depress the plunger and closely watch the tip for leakage. Solvents such as chloroform used for carboxylic acid analysis may draw back up in or drip from the dispenser tip.
- ii. Document observations in the laboratory notebook. Take corrective action to eliminate leaks.

Glassware Calibration

Glassware must be clean and dry prior to calibration. Use a calibrated analytical balance and Type 1 ultra-pure water with a resistivity >18.0 MΩ.

- i. Place the flask on the balance pan and tare the balance.
- ii. Carefully add deionized water to the flask until the weight exactly matches the expected volume of the flask.
- iii. Use an indelible marker to draw a fill line at the bottom of the meniscus. This is the line to which the flask must be filled to match the expected volume.

Glassware Storage

- i. Dedicate glassware to each respective analytical area and procedure. Glassware will become seasoned or conditioned with the chemicals used in that area or procedure.
- ii. Store glassware used during instrument calibration procedures at room temperature, stoppered and full of deionized water. Prior to use, empty and rinse three or more times with the calibration solution.

References

- Clement, R., et al. (1997). *Reference Materials for Environmental Analysis*. Boca Raton: Lewis Publishers.
- Simser, J. (2016, January 14). Sterilization Techniques for Water Matricies. (N. Lance, Interviewer).