

CALIBRATION AND STANDARDIZATION 6.6.2

Calibration is required for the pH instrument—follow instructions in NFM 6.4.

Alkalinity and ANC determinations require standardization if the 0.01639*N* sulfuric acid titrant (buret method) is used. In the procedure that follows (Fishman and Friedman, 1989), the reagent concentrations were selected so that the titrant volume would correspond exactly to the bicarbonate equivalent in milligrams per liter of sample.

Prepare fresh carbon dioxide-free deionized water (DIW):

1. Boil the DIW in a beaker for 15 minutes.
2. Cool the DIW to room temperature. Cover the beaker to minimize atmospheric contamination while the DIW cools.

An alternative to the method in steps 1 and 2 is to infuse the DIW with compressed helium or nitrogen gas for about 10 minutes.

Prepare a fresh standard solution of sodium carbonate (Na₂CO₃)(1.00 mL = 1.00 mg HCO₃):

1. Dry 1.0 g primary standard Na₂CO₃ at 150 to 160°C for 2 hours.
2. Cool in a desiccator; weigh out 0.8686 g Na₂CO₃; add to a 1-L volumetric flask.
3. Dilute with carbon dioxide-free DIW to the 1-L mark.

Prepare sulfuric acid titration solution:

1. Add 0.5 mL concentrated H₂SO₄ (specific gravity 1.84 g/mL) to 950 mL DIW.
2. Mix thoroughly.

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To standardize the sulfuric acid, follow steps 1 through 9 below. To check the normality of a sulfuric acid titration solution, follow steps 3 through 6 below.

1. Calibrate the pH system (follow the instructions in NFM 6.4).
2. Prepare a sodium carbonate standard solution as outlined above.
3. Pipet 25 mL of sodium carbonate standard solution into a 100-mL beaker.
4. Titrate with the sulfuric acid solution to pH 4.5.
5. Record the volume of titrant used.
6. Determine the normality of acid by use of the following equation:

$$N = (25) (0.01639) / mL_a$$

where

N = normality, and

mL_a = volume of sulfuric acid added in milliliters to reach pH 4.5.

7. Adjust the concentration of sulfuric acid to exactly 0.01639*N* by dilution with deionized water or by the addition of dilute acid, as indicated by the first titration.
8. Confirm the exact normality by restandardization.
9. Keep the solution in a tightly sealed 1-L glass bottle until used.
10. If the sulfuric acid titrant solution is not exactly 0.01639*N*, divide the actual normality of the H₂SO₄ titrant by 0.01639*N* to obtain the correction factor.

CAUTION: Wear appropriate safety gloves, glasses, and apron when working with corrosive and oxidizing solutions.