
EQUIPMENT AND SUPPLIES 6.6.1

Equipment and supplies for the electrometric method to determine alkalinity and ANC are listed in table 6.6-1. The equipment must be tested before each field trip and cleaned immediately after each use.

Buret, micrometer buret, digital titrator

The buret provides good accuracy and precision when used by a trained operator.

- ▶ Micrometer burets provide better accuracy and precision than burets—they can deliver acid increments to 0.0001 mL and are available commercially (for example, Gilmont™ micrometer burets).
- ▶ The digital titrator is not as accurate as a buret because it was designed for endpoint titrations. The digital titrator is popular because it is more convenient and less fragile than a buret and keeps the acid in a virtually closed system. (The Hach brand is used as an example in this document.) **Delivery tubes of clear (instead of red) plastic are recommended. Empty titrant cartridges are available.**

Volumetric pipet, graduated cylinder, digital balance

The volumetric pipet is used for dispensing the correct volume of filtered sample only.

- ▶ Use only class A “TD” pipets. TD is a calibration designation meaning “To Deliver” (TD is distinguished from “TC” or “To Contain” pipets).
- ▶ Class A pipets should not be used to aspirate or dispense solutions containing suspended particles—the small bore of this pipet tends to reject particles during aspiration and retain them during delivery (C.J. Patton, U.S. Geological Survey, written commun., 1995).

The graduated cylinder and digital balance are used for dispensing the correct volume of unfiltered (ANC) sample only. The digital balance yields higher precision than the graduated cylinder.

Table 6.6–1. Equipment and supplies used for alkalinity or ANC titrations¹

[mL, milliliters; ANC, acid neutralizing capacity; g, gram; $\mu\text{S}/\text{cm}$, microsiemens per centimeter at 25°Celsius; N, normal]

Equipment and supplies common for using either a digital titrator or a buret

- ✓ pH meter, preferably with automatic temperature compensator (see NFM 6.4 for selection and associated supplies)
- ✓ pH electrode, calibrated, combination or equivalent, and appropriate filling solution, if required
- ✓ Thermometer, calibrated (see NFM 6.1 for selection and calibration criteria)
- ✓ Stirrer, magnetic (battery operated) or glass stir rods
- ✓ Stirring bars, Teflon™ coated, smallest size (always carry spare bars)
- ✓ Volumetric pipets, class A—25 mL, 50 mL, and 100 mL (for alkalinity)
- ✓ Graduated cylinder (for ANC). For higher precision, use a digital balance, 0.1-g accuracy, 200-g capacity, pocket-sized (available from Acculab Company)
- ✓ Pipet squeeze bulb or pipet pump
- ✓ Sample bottle, 500 mL, acid rinsed or deionized-water rinsed
- ✓ Beakers, glass—50 mL, 100 mL, and 150 mL
- ✓ Beaker, Berzelius, 300 mL, tall form, spoutless, with two- or three-hole stopper
- ✓ Deionized water (DIW) (maximum conductivity of 1 $\mu\text{S}/\text{cm}$)
- ✓ Dispenser bottle, squeeze, for deionized water
- ✓ Filtration unit, in-line capsule or pressure unit with inert gas (for alkalinity)
- ✓ Sodium carbonate standard solution
- ✓ Safety gloves, glasses, acid spill kit, and apron
- ✓ Titrant solution, sulfuric acid solution, 0.1600*N*, 1.600*N*, and 0.01639*N* (pre-filled cartridges for use with the Hach system are available for 0.1600*N* and 1.600*N* solutions)
- ✓ Paper tissues, disposable, soft and lint free

Equipment and supplies for using a digital titrator

- ✓ Digital titrator and mounting assembly
- ✓ Delivery tubes, bent-stem, colorless, transparent
- ✓ Titrant cartridges, empty or prefilled according to study needs (used with Hach system)

Equipment and supplies for using a buret

- ✓ Calibrated buret, 25-mL capacity with 0.05-mL graduations and Teflon™ stopcock
- ✓ Calibrated buret, 10-mL capacity with 0.02-mL graduations and Teflon™ stopcock
- ✓ Micrometer buret (alternative to standard burets, for greater accuracy)
- ✓ Titrant solution, sulfuric acid solution, 0.01639*N*
- ✓ Buret stand and clamp
- ✓ Wire pen cleaner (for cleaning buret tip)
- ✓ Buret cap
- ✓ Buret meniscus reader
- ✓ Acid bottle, pump (for filling buret)

¹Modify this list to meet the specific needs of the field effort.

Sulfuric acid titrant

Sulfuric acid is the titrant used routinely by USGS. The normality of sulfuric acid titrant is subject to change with time.

- ▶ 0.1600*N* or 1.600*N* solutions in prefilled cartridges for the Hach digital titrator are available from the QWSU (USGS, Ocala, Fla.). Normality of sulfuric acid in prefilled Hach titrant cartridges is monitored by the QWSU for 1 year after date of purchase; the QWSU notifies USGS personnel if a change in normality occurs. The lot number indicates the date of preparation (QWSU will translate lot numbers upon request). **QWSU recommends that Hach titrant cartridges be used within 3 months of purchase.**
- ▶ 0.01639*N* solution for the buret is available from the QWSU. Normality of 0.01639*N* titrant is not monitored by the QWSU after preparation and standardization. **Check the normality of this titrant each month.**
- ▶ Acid solutions of other normalities may be needed, depending on the sample chemistry or the ionic strength. Prepare under a fume hood. Check the normality monthly.

TECHNICAL NOTE: For samples vulnerable to precipitation reactions, a Berzelius beaker can help minimize gas exchange. Select a size of Berzelius beaker that fits the sample volume and associated titrating equipment and yet minimizes headspace above the sample. Fit the Berzelius beaker with a two- or three-hole stopper to accommodate the electrode(s), the thermometer, and the digital or buret titrator. Another option is to work in a glove box filled with an inert gas atmosphere. Oceanographers use a closed cell with an expanding plunger to avoid gas exchange (Almgren and others, 1977).

CAUTION: Use the safety precautions outlined on the Material Safety Data Sheets (MSDS) when handling chemicals—wear safety glasses, gloves, and protective clothing.

6.6.1.A MAINTENANCE, CLEANING, AND STORAGE

Proper maintenance, cleaning, and storage of the pH instrument is critical for ensuring the accuracy of alkalinity or ANC determinations, and guidance is provided in NFM 6.4.

Clean the volumetric pipets, beakers, bottles, burets, and stirring bars with hot water and nonphosphate detergent; rinse them copiously with tap water followed by deionized water. If oily or chemical residues are difficult to remove, soak the glassware and nonmetal equipment in a mild (5 percent by volume) hydrochloric acid or nitric acid solution (see Horowitz and others, 1994), and repeat the detergent wash. Store cleaned equipment wrapped or bagged in plastic until ready for use.

Reagents must not exceed their shelf life. Store reagents, as appropriate, in a dust-free cabinet, desiccator, or refrigerator. When chemicals to be used for preparation of reagents are received, mark the dates of receipt and expiration on container. When a reagent is prepared, label the container with the contents, date of preparation, expiration date, and preparer's initials. Store the 0.01639*N* standard sulfuric acid solution and filled Hach titrant cartridges in a cool, dark place (a storage cabinet or frost-free refrigerator). Seal the filled cartridges in plastic bags to avoid moisture loss or gain.

Some of the procedures recommended herein for equipment operation may be out of date if the equipment being used is different from that described or incorporates more recent technological advances—follow the manufacturer's instructions.