Standard Preparation for NO_3, NO_2, PO_4, and NH_4 analysis (2/20/2015 DER)

Calibration standards and check standards should ideally be prepared from primary standard grade salts or other certified or traceable materials. The two should also come from entirely different sources to be able to use them to verify each other. Difficulties can arise in finding multiple independent sources of certified materials for some analytes. Occasionally, certified materials are not as accurate as would be expected. At Ecocore, a recently purchased "NIST traceable" nitrite standard solution was measured to be consistently 10% lower than the nominal value against three other materials. Because of these difficulties, ACF chemical reagent grade materials are sometimes used as check standards and more rarely as calibration standards.

Materials and Cleaning

All solid standard materials are oven dried at 105^{o} C overnight, then placed in a desiccators to cool to room temperature before weighing. All weighing is performed on a 4 place (0.0001 g) balance using a clean spatula and a disposable plastic weighing boat. Balances are calibrated by a certified balance technician annually and performance is verified using check weights periodically by the lab managers.

All solutions are made in Class A glass volumetric flasks and all dilutions are made using Class A glass volumetric pipettes and volumetric flasks. Standards are stored in Nalgene polyethylene or polypropylene bottles. All standards for water analysis are made in De-ionized water with an electrical conductance of less than 1 micro Siemens. This same grade water is used for all glassware cleaning.

All storage bottles, volumetric flasks and pipettes, funnels and beakers used for standard preparation are acid washed prior to use. The acid washing procedure is: items are individually rinsed 3 times with DI water. Items are then fully immersed in a 1.2 Molar Hydrochloric Acid bath (1 part concentrated HCl to 9 parts DI water) and left overnight. Items are then individually rinsed inside and out 6 times with DI water. Volumetric flasks are stored full of DI water and emptied just before use. Volumetric pipettes are oven dried at 105^{o} C. Beakers and funnels and storage bottles are air dried. All materials are stored in a designated space separate from other lab glassware.

Calibration Standards

A series of calibration standards are prepared and analyzed to create a standard curve which is used to calibrate the instrument. The concentrations of these standards are chosen to cover the expected range of the samples. 5-9 standards are typically run in a calibration curve. A significant volume (typically 500 ml) of a stock solution with a high concentration (eg. 1000 mg/l) is prepared. This allows for the amount of salt to be weighed to fall in the 2-5 gram range for most analytes. The salt is weighed to +/- 0.0001 grams of the nominal weight on a 4 place analytical balance. This allows for an extremely small weighing error.

Working standards are prepared by diluting the stock solution using volumetric pipettes and flasks. Very low concentration standards (less than 0.5 ppm) are prepared by a two step serial dilution. The stock solution is diluted to 10 or 1 ppm, then this solution is diluted to make the final working standards.

Check Standards

Check standards are prepared using the same technique as the working standards.

Storage

All calibration standards, check standards and stock solutions are stored in a refrigerator from day to day and frozen if storage is for more than a week.