

Laboratory Standards

With most of the instruments used in the lab, standards are necessary to translate the values provided by the instrument (e.g. absorbance) into usable information (e.g. concentration.) Analytic procedures and instruments do not measure the amount of an analyte directly but instead measure some property, quality or attribute of the analyte. The spectrophotometers measure the light absorbed by analyte¹. pH meters measure millivolts. In order for these instrument readings to related to concentration, solutions of known concentration (standards) must be used.

An analogy will help to illustrate the above idea. A person viewing a photograph of an unfamiliar object has no way of knowing its size unless a familiar object is placed nearby. However, if a ruler is place by the object, the observer can determine the objects size Standards perform the same function as the ruler. Standards are reference points by which the actual concentration of an analyte can be determined..

Standards serve another function aside from relating one parameter to another. Standards indicate how well a procedure or instrument is working. This is accomplished by graphing the values given by the instrument against the concentration of the standards. The line obtained is then judged with respect to optimum linearity, slope and y-intercept. The result is an equation with an r^2 value to show how well the data fits the equation. An r^2 of 1 indicates a perfect fit. An r^2 of .70 would indicate a poor fit.

Deciding the concentration of the standards to be used is an important part of an analytic procedure. Two is the minimum number of standards needed for an analysis. Using three or more standards improves accuracy and establishes the nature of the standard curve i.e. straight or curvilinear. Usually, three standards and a blank are sufficient for most procedures. The values of the standards should bracket the values expected from your samples.

Example: If you expect your sample solutions to have a Ca concentration between 110 and 140 ppm, then your standards should be 100ppm and 150 ppm Ca.

Samples that give readings greater than the standard values need to be diluted.

Equal in importance to the concentration of a standard, is its matrix². The matrix of a standard should match the matrix of the sample being analyzed as close as possible. For example, if samples are extracted with .5 N HNO₃ then the standards should be made in .5 N HNO₃. The only thing that should be different about samples and standards should be the concentration of the analyte.

Preparation of Standards

Working standards³ are usually made by diluting a solution of higher concentration with the sample matrix. This higher concentration solution is called a primary standard. Primary standards can be purchased from scientific supply houses or prepared in the laboratory. A primary standard should be prepared from oven dry reagent grade

chemicals. Use the following formula to calculate the amount of reagent needed to make solution.

Primary Standard Calculations

Grams of reagent needed = (MW of reagent / MW of analyte x moles of analyte in reagent) x grams of analyte needed.

Example: 1 gram of Na is needed from Na_2SO_4

$$\text{MW Na}_2\text{SO}_4 = 142 \text{ g / mole, MW Na} = 23, \text{ moles of Na per mole of Na}_2\text{SO}_4 = 2$$
$$(142\text{g Na}_2\text{SO}_4 / 23\text{g Na} \times 2) \times 1\text{g} = 3.09\text{g Na}_2\text{SO}_4$$

Example: Preparation of 1 liter of a primary standard of 1000 ppm* Sodium.

Calculations: $(1000 \text{ mg Na / L}) \times 1\text{L} \times (58.5 \text{ g NaCl / } 23\text{g Na})^{**} = 2.543\text{g NaCl}$

* ppm = mg/L

** M.W. NaCl = 58.5g / mole , M.W. Na = 23g / mole

1. Oven dry NaCl for at least 2 hours at 105C and place in desiccator to cool.
2. Weigh 2.543 g of NaCl and pour into 1L Volumetric. Fill flask half way with DI water and mix until NaCl is dissolved. Add DI water to 1 L mark. Seal top with Parafilm and invert 30 times to mix solution.
3. Pour solution into labeled bottle. (Solutions should not be stored in volumetric flasks.)

Secondary Standards

Standards prepared by diluting the primary standard are called secondary standards and are usually your working standards. A small portion or aliquot of the primary standard is diluted with the sample matrix to make secondary standards. Use the following formula to determine how much primary standard is needed to make a secondary standard.

$$C_1V_1 = C_2V_2$$

C_1 = Concentration of primary standard, V_1 = Volume of Primary standard

C_2 = Concentration secondary standard, V_2 = Volume of Secondary standard

Usually you need to calculate the volume of primary standard needed because you know the concentration of the primary and secondary standards and the volume of the primary standard. To solve for the volume of primary standard needed, use the following equation.

$$V_1 = C_2V_2/C_1$$

Example: Make a 100 ppm Ca secondary standard from a 1000 ppm primary standard.

First, you have to decide how much of the secondary standard to make. Secondary standards are usually prepared in volumetric flasks with defined volumes e.g. 1000ml, 500ml, 100ml, 50 ml etc.. Usually 50ml or 100ml is prepared. We will use a 100ml volumetric flask.

Calculations

$C_1 = 1000 \text{ ppm Ca}$, $C_2 = 100 \text{ ppm Ca}$, $V_1 = 100 \text{ ml}$

$C_2 V_2 / C_1 = V_1$, $(100 \text{ ppm} \times 100 \text{ ml}) / 1000 \text{ ppm} = 10 \text{ ml}$ of 1000 ppm standard.

Directions

1. Pipette 10 ml of 1000 ppm Ca into a 100ml Volumetric flask.
2. Fill volumetric flask to line with matrix solution.
3. Seal with Parafilm and invert 30 times.

Serial Dilutions

Often very low concentration standards are needed. These low concentrations are usually prepared from the secondary standards instead of the primary standard.

Example: Make 1 ppm and a .01 ppm Ca standards.

- a. Make a 100 ppm Ca secondary standard from the 1000 ppm Ca standard.
- b. Use the 100 ppm Ca solution to make the 100ml of 1 ppm Ca standard.
 $(1 \text{ ppm} \times 100 \text{ ml}) / 100 \text{ ppm} = 1 \text{ ml}$ of 100 ppm standard
- c. Use the 1 ppm Ca to make 100 ml of the .01 ppm Ca standard
 $(.01 \text{ ppm} \times 100 \text{ ml}) / 1 \text{ ppm} = 1 \text{ ml}$ of 1 ppm standard

Notes:

1. The analyte is the element or compound you are measuring.
2. The matrix is everything in the solution except the analyte.
3. Working standards are the standards used to calibrate an instrument or procedure. They are also called secondary standards