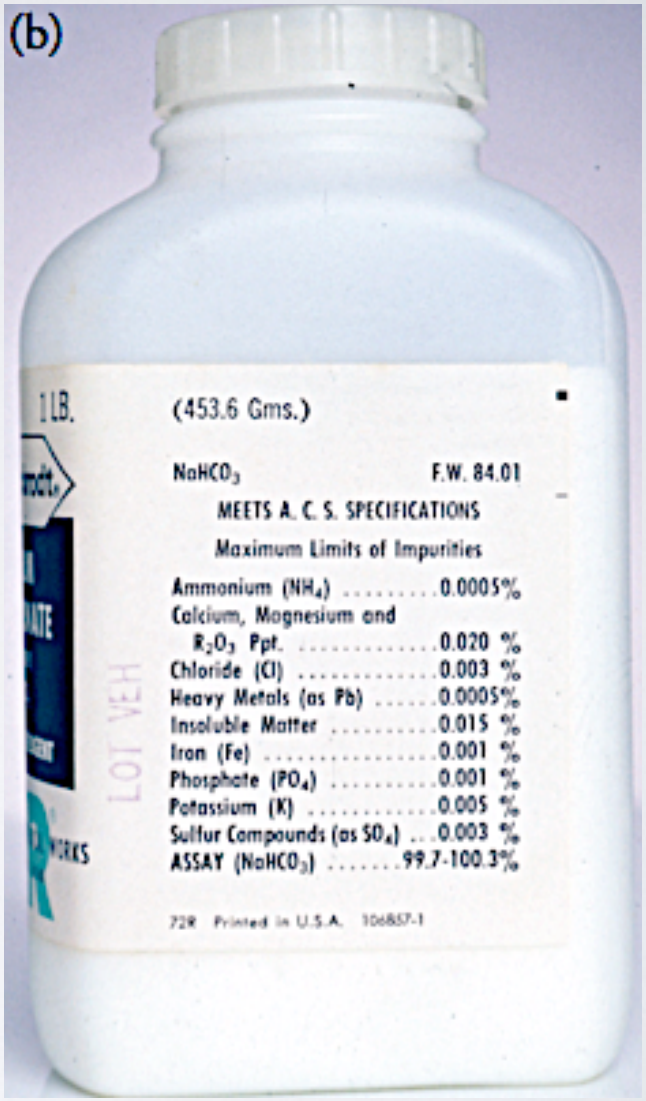
STANDARDS, CALIBRATIONS, & BLANKS

SDSU CHEM 251

TYPES OF STANDARDS

- **Primary Standards:** must have a known stoichiometry, a known purity, must be stable for long term storage.
- **Secondary Standards:** have their concentrations determined relative to a primary standard.
- It is crucial to know the amount of all other solution components when preparing standards (e.g. solvents, acids...)



STANDARD SOLUTIONS

- A single standard solution is typically insufficient for accurate calibration of the signal (S_{total}) - particularly if the relationship between signal and analyte is not linear.

- Two methods exist for creating a range of standards with different concentrations:

    - **Individual preparation:** weight standard for each solution to be prepared.

    - **Serial dilution:** A high concentration standard solution is prepared and a portion is taken to make a lower concentration standard. This process is repeated for each successive solution creating a range of solutions from one weighing of standard.

DETERMINING SENSITIVITY

- Determining the relationship between the **signal (S)** and the **amount (n_A or C_A)** of anlayte in the sample.
    - $S_{total} = k_A n_A + S_{reag}$ or $S_{total} = k_A C_A + S_{reag}$
- S_{total}: is the measured signal (voltage, mass, volume...)
- n_A(or C_A): is the number of moles, or concentration, of analyte
- S_{reag}: is the signal from components other than the analyte
- k_A: is the sensitivity of the method to the analyte

SIGNAL CALIBRATION

- Equipment used for analysis can yield a range of different signal types, depending on the tool: glassware, balances, pH meter, spectrophotometer...
- Calibration of the signal is necessary to ensure that there are no determinant (systematic) errors in the measurements to determine k_A or S_{reag}.
- This is similar to the calibration of your pipettes and buret.
- Calibration ensures that $S_{total} = S_{std}$ without calibration each measurement will have a systematic error (X): $S_{total} = S_{std} \pm \mathbf{X}$

STANDARDIZATION

- Single-point standardization: $S_{std} = k_A n_{std}$
- Using a known standard we can determine the sensitivity (k_A) of the technique.
- Poor choice for two reasons:
- Errors in k_A will carry over to all other measurements.
- We cannot accurately predict how the system will respond to other concentrations. May assume a linear relationship, but that may not be true.

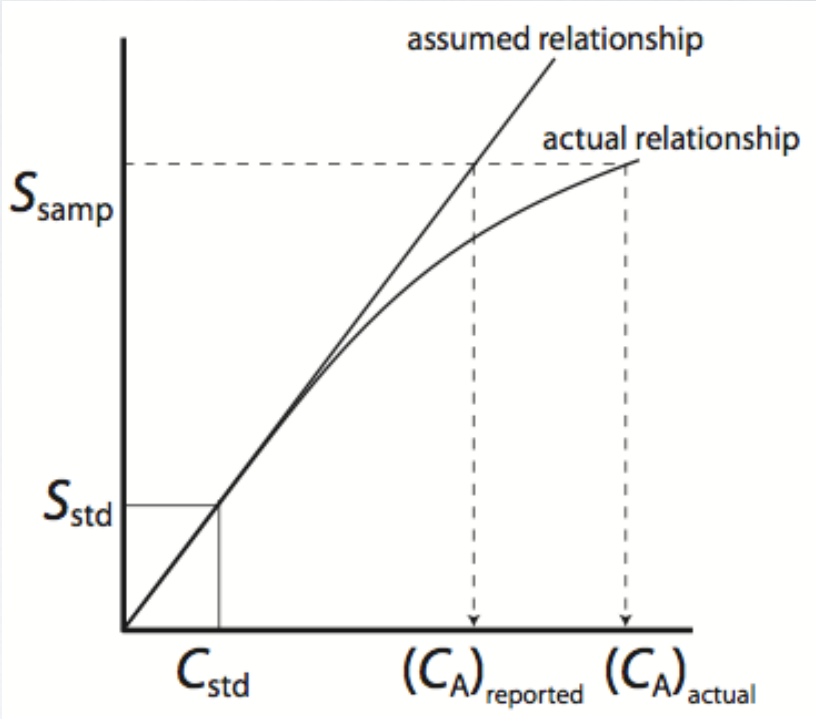
STANDARDIZATION

- Multiple-point standardization

- Involves multiple standard solutions, which ideally bracket the expected analyte ranges.

- This also minimizes the error of that may afflict a single-point calibration.

- Allows us to know the actual relationship between the signal and analyte concentration.



BLANK CORRECTIONS

- There is more than one type of blank that can be involved in chemical measurements.

- The "calibration blank" is a blank used to ensure that the measurement device gives a zero signal when there is no sample present. This can be an electronic adjustment of the signal intensity in the absence of sample.

- The "reagent blank" is a solution that contains all the same components (matrix) as the sample solution, but no known analyte materials. The reagent blank identifies the amount of the signal that is due to the reagents used in the preparation of the samples.

- Both blanks can, and should, be subtracted from the measured signal of a sample in order to determine the signal due to the analyte alone.