

PREPARATION OF AQUEOUS STANDARDS FOR ANALYSIS OF WATER SAMPLES

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PREPARATION OF AQUEOUS STANDARDS FOR ANALYSIS OF WATER SAMPLES

1. PURPOSE

The purpose of this procedure is to document the preparation of standards that are used to calibrate the analytical instrumentation used for characterization of water samples for the NNWSI waste isolation studies.

The response of most analytical instruments to the presence of an analyte is normally highly reproducible and proportional to the concentration of the analyte. However, the proportionality constant that defines the relationship between instrument response and analyte concentration is frequently difficult to calculate. To circumvent this problem, standards are employed to establish empirically the proportionality constant. Thus the quality of an analytical measurement will be no better than the quality of the standards used to calibrate the measurements.

2. SCOPE

This procedure is to be followed for the preparation of standards for analytical instrumentation including, but not limited to, plasma emission spectroscopy and ion chromatography.

3. PROCEDURE

Working standards are prepared by suitable volume dilutions of concentrated stock solutions of the species of interest. The concentrated stock solutions are prepared by weighing with an analytical balance an appropriate quantity of a dried metal or compound, dissolving the substance in a suitable solvent, and diluting the solution to a known volume. The compound or metal used must be:

- 1) of high purity - analytical reagent grade or better,
- 2) capable of being dried to a constant weight of known composition, and
- 3) soluble in a solvent that is compatible with the analytical procedure to be employed.

There are available commercially from many suppliers standard solutions, typically at a concentration level of 1 mg/mL, that are prepared from materials that meet the above listed criteria. These will also be called stock solutions. Examples of these are the solutions provided by the Alfa-Ventron Company; document TWS-CNC-11-11/78-154 attests to their accuracy.

3.1 Preparation of Stock Solutions

- 1) All reagents shall be analytical reagent grade or purer chemicals.
- 2) All water used for dilutions shall have a resistivity of at least $17\text{M}\Omega\text{-cm}$.
- 3) All volumetric glassware shall be type "A".
- 4) Dry a quantity of the standard reagent to a constant weight of known composition. If dried in an oven, allow to cool to room temperature in a desiccator over a suitable desiccant before weighing.
- 5) Weigh the dried material using a calibrated four- or five-place analytical balance.
- 6) Dissolve the material in an appropriate solvent, usually either water or a mineral acid; and dilute to a known volume.
- 7) Store this stock solution in a clean polyethylene or polypropylene bottle dated as to time of preparation and cross-referenced to a laboratory notebook by notebook number and page number.
- 8) The reagent used, its weight, and the final volume of solution shall be recorded in a laboratory notebook.
- 9) When removing aliquots of the stock solution, avoid introducing any foreign substance into the solution by always pouring a portion out into a clean container.

3.2 Preparation of Working Standards

Working standards are prepared from the concentrated stock solutions according to the following procedure for volumetric dilutions.

- 1) All water used for dilutions shall be at least $17\text{M}\Omega\text{-cm}$ resistivity.

- 2) All acids used to preserve standards shall be Baker Inc. Ultrex grade or equivalent.
- 3) All volumetric glassware shall be type "A".
- 4) The individual preparing the standard shall show in a laboratory notebook the volume of each stock solution used to prepare the standard, the final volume of the working standard, and the final concentration of each component of the standard.
- 5) Standards which contain trace metals shall be preserved by the addition of 1% of high purity concentrated nitric acid.
- 6) If non-adjustable positive displacement pipets are used, they shall be initially calibrated by weighing on an analytical balance five aliquots of deionized water to insure that they are dispensing an accurate quantity of solution; adjustments shall be made as necessary. These pipets shall be recalibrated every six months or after any maintenance has been performed on them.
- 7) If adjustable positive displacement pipets are used to prepare a new standard, each volume setting used must be verified by weighing five aliquots of deionized water with an analytical balance. The results of these weighings shall be recorded in the laboratory notebook, and a mean and standard deviation calculated for the measurements.
- 8) If a standard is being prepared to replace one that has been consumed, the new standard shall be run as a sample versus the old standard to verify the concentration of the components. This procedure will preclude the necessity of carrying out step 7.

4. QUALITY ASSURANCE

- 4.1 All procedures shall be carried out by qualified, certified staff members, or by technicians working under their supervision.

- 4.2 Calibration of equipment shall be performed by qualified personnel according to written procedures.
- 4.3 Data shall be documented in laboratory notebooks as described in section 3 above. The notebooks will be reviewed in accordance with the procedures in TWS-CMBQA-QP-03.