

The Use of Secondary Standards for Spectrophotometer/Colorimeter Calibration

Secondary standards (gel standards) are specifically designed to verify the instrument's calibration and to check the instrument's performance. They are not intended to be used to create calibration curves or to calibrate the instrument. Because the DPD reagent cannot be mixed with the gel standards, the quality and the reaction time of the reagent cannot be assessed. For this reason gel standards cannot take the place of primary standards.

The analyst is responsible for the following:

- Preparing the calibration curve for each instrument **once per month** at a minimum, with chlorine standards or potassium permanganate (see instructions below for KMnO₄) before the use of new DPD reagents, or the use of new gel standards
- Recording reagent lot #'s for reagents and standards
- Recording calibration concentrations
- Verified the calibration curve using a minimum of one blank and two gel standards that bracket the expected sample concentration
- Recording all verification data

STOCK STANDARD SOLUTION

0.891 grams of reagent grade KMnO₄ in 1000 mL vol. flask made to mark with deionized water. Deionized water must never be stored in plastic containers or exposed to airborne contamination. Store the stock solution in amber bottle in a cool area. The typical shelf life of the stock solution is six (6) months. If solids appear in the solution, **do not use**.

Avoid leaving the cap off for extended periods of time and avoid contamination.

INTERMEDIATE (WORKING) STANDARD SOLUTION (10 mg/L)

10 mL of *STOCK* made in 1000 mL vol. flask made to mark with deionized water. The flask should be labeled with the name, KMnO₄, date of preparation, initials of who made it.

This information should also be entered into a logbook.

The intermediate stock solution should be stable for approximately 5 days if kept cool and away from light.

Care should be taken that the pipette and glassware are clean and thoroughly rinsed with deionized water to avoid contamination. Store only in glass container (preferably amber glass) never in plastic containers. The working solution should be remade if solids appear in the bottom of the container.

CALIBRATION STANDARD SOLUTIONS

If using KMnO₄, four to five calibration standard solutions should be made, according to the table below, to create a calibration curve **once per month** at a minimum. The correlation coefficient (r) of the curve should be 0.995 or higher. This curve is then used to check or calibrate the instrument. Gel standards are run against the curve and must meet the manufacturer's published acceptance criteria for the specific instrument being used.

The working solution should be stable for approximately 2 hours if chlorine demand-free water is used.

A target value (e.g. permit value for a facility) should be known and three gel standards, 0.00 mg/L, blank, and two other standards (a low and a high standard) that bracket the target value should be chosen.

mL Working Standard Diluted w/Deionized water	Chlorine Equivalent mg/L
20 mL (vol. Pipette) to 100 mL (vol. flask)	2.0 mg/L
10 mL (vol. Pipette) to 100 mL (vol. flask)	1.0 mg/L
5 mL (vol. Pipette) to 100 mL (vol. flask)	0.5 mg/L
1 mL (vol. Pipette) to 100 mL (vol. flask)	0.1 mg/L
1 mL (vol. Pipette) to 200 mL (vol. flask)	0.05 mg/L
1 mL (vol. Pipette) to 500 mL (vol. flask)	0.02 mg/L
100 mL of deionized water	0.00 mg/L