Method 334: Creation of Calibration Check Standard Dilutions

Considerations:

- NIST certified stock standards must be stored according to manufacturer recommendations. Generally, they will need to be refrigerated to prevent degradation.
- It is critical that stock standards be used prior to the expiration date. Expired standards may have experienced degradation over time.
- When mixing calibration check standards, reagent grade water should be utilized to prevent issues related to chlorine demand. Distilled, deionized, organic-free, and chlorine demand-free water are all acceptable.
- Calibration standards must always be prepared immediately prior to analysis and should not be stored for use at a later time. The use of high quality, reagent grade water will minimize chlorine degradation through reaction with contaminants, but off-gassing of chlorine will still occur over time. While degradation this may not be appreciable at high concentrations, it may result in complications at concentrations towards the low end of the calibration curve.
- Potassium permanganate stock solutions can be used as a surrogate for free chlorine standards when performing aqueous calibration verifications for Method 334.0 provided the stock solution is NIST certified; however, the reaction of equivalent concentrations of potassium permanganate and free chlorine with DPD will produce differing degrees of color formation. If a potassium permanganate standard is being used, it is critical to note that a multiplier must be employed to determine the appropriate dilution ratio for a desired concentration. To mix a primary standard that is equivalent to 1.0 mg/L free chlorine, a potassium permanganate solution of 0.891 mg/L must be mixed.

Equipment for mixing standards:

Supplies:

- Class A variable volume pipette (range from 100 µL to 1,000 µL)
- Pipette tips
- Specimen cups (polystyrene specimen cups are relatively chemically inert and more durable than glass)
- Primary standard ampules
- Class A volumetric flasks
- Dilution water (reagent grade water includes distilled, deionized, organic-free or chlorine demand-free)
- Dry waste bag
- Liquid waste bottle

- Please note that volumetric flasks and containers used for dispensing check standard aliquots should be treated using a chlorine demand-free protocol to minimize degradation of the calibration check standard.
- Additionally, consider designating a set of labware specifically for the preparation of aqueous calibration check standards to minimize the likelihood of introducing contamination that may impact the concentration of the standards.

1. **Calculate the required volume of <u>certified stock solution</u> to mix the desired volume (e.g. 100 mL) of your chosen check standard concentration**

   Use the equation: $C_1V_1 = C_2V_2$

   Rearrange to solve for the volume of certified standard: $V_1 = (C_2V_2)/C_1$

   C_2 (mg/L) ☐ Desired concentration for calibration check standard that falls in your meter's DPD low range (typically 0.02 to 2.00 mg/L)

   V_2 (mL) ☐ Desired volume of the prepared check standard (Example: 100 mL for volumetric flask)

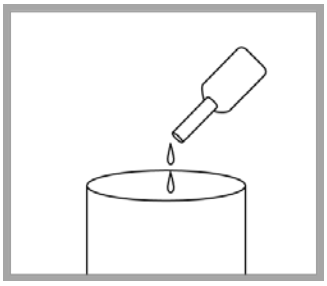
   C_1 (mg/L) ☐ Manufacturer's certified concentration of primary standard (Example: 66.5 mg/L)

   V_1 (mL) _____ **Calculated Volume of primary standard to mix desired volume (V2) of check standard**

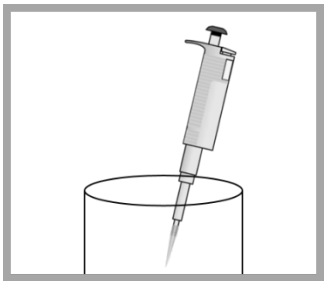
$$V_1 = \frac{C_2 \square \times V_2 \square}{C_1 \square}$$

2. **Mix aqueous calibration check standard at the desired diluted concentration.**

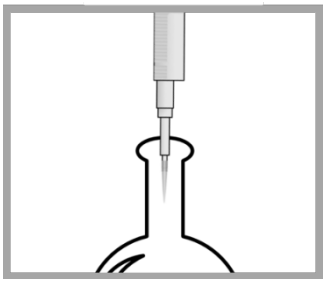
1. Add half the volume (that will be used as the final volume) of reagent grade water to the volumetric flask (e.g. 50 mL if the final volume will be 100 mL).*



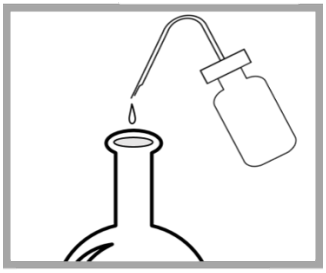
2. Break open the certified standard ampule (concentration is C_1 above) and pour into a chlorine demand free container, such as a specimen cup.



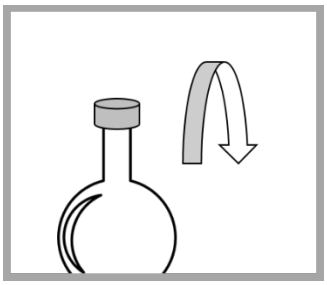
3. Adjust the pipet to the desired volume (V_1 above). Pipet the volume from the specimen cup… (First stop on pipette is for drawing solution)



4. ….to the volumetric flask (second stop is for expelling solution)



5. Add the reagent grade dilution water. Allow a small amount of room in the neck and then use a wash bottle of reagent grade water to fill it to the graduation mark.



6. Cap flask and invert gently to mix solution. Transfer the standard from the flask to a second container, such as a specimen cup for ease of pouring it into the sample cells.

***** Create enough solution standard solution to thoroughly rinse the sample cell between analysis. Remember, you will be running five duplicate analysis and you need to rinse in between. If you are running 10 mL samples, plan on 100 mL of solution to allow for rinsing.