

Instruction Sheet

CL17 Free and Total Chlorine Calibration/Verification

Guidelines for Use

Introduction

The CL17 Calibration/Verification Kit allows the user to perform a calibration or verification of the analyzer using a chlorine standard of known concentration.

Recommended use

Under normal circumstances, Hach Company does not recommend manual calibration of the CL17. Verification, however, is recommended following each change of analyzer reagents. This can be performed either by comparison with a laboratory, a portable instrument or by using the CL17 Calibration/Verification Kit.

Certain regulatory agencies and other organizations require manual calibration or verification of chlorine analyzers. Follow the guidelines set by those organizations to determine required frequency of calibration.

Guidelines for Accuracy

Making and using chlorine standards with the accuracy needed to calibrate or verify the CL17 requires precisely controlled reagents, clean hardware and careful laboratory practice. Use only the reagents specified in this instruction sheet and follow the instructions carefully. Failure to do so may result in incorrect calibration forcing the analyzer to read inaccurately. To confirm correct use of this kit, perform the statistical calculations described in Preparing the Chlorine Standard on page 6

Figure 1 shows the two styles of analyzers under the CL17 name.

Figure 1 CL17 Analyzers





Supplied Apparatus and Reagents

The Voluette contains a chlorine standard in the range 100–130 mg/L. Each Voluette is precisely analyzed by Hach Company prior to shipment and the actual chlorine concentration is printed on the tube container. When mixed with on of the bottles of organic Free Water, a Chlorine Standard in the range 4.5 to 5 mg/L is produced. The precise value of the standard is calculated as shown in Preparing the Chlorine Standard on page 6

Supplied F	Reagents and Apparatus Kit (Contains all items lister	Cat. No. 54490-00		
	3-Way Valve	3-Way Valve		
	Small Tube Clamp	Female Luer to ⁵ /32 inch	1	
	3-way Luer Lock Valve	 Male Luer to ⁵/32 inch 		
	Pressurization Apparatus Kit	Pressurization Apparatus Kit		
	60 cc Luer Lock Syringe (Cat. No. 22587-00)		-	
	One-Way Check Valve (Cat. No. 54495-00)			
	Calibration Standards Set	Calibration Standards Set		
	Two Bottles 500 mL ±5 ml	Two Bottles 500 mL ±5 mL Organic Free Water		
	One 20 mL Voluette® 100-130 ppm Chlorine Standard			

Installing the 3-Way Valve

- 1. Turn off the analyzer power.
- 2. Turn off flow to the analyzer.
- 3. Determine if your analyzer is "Old Style", or "New Style". Refer to Figure 1 on page 1.
- 4. If it is a new style analyzer, disconnect the sample in line by pushing the ring on the John-Guest fitting in and pulling the tubing out, see Figure 2 on page 4. If it is an old style analyzer, it will be connected with a plastic nut; disconnect the nut and remove the ferrule by cutting the tubing just above it. Allow the excess water to drain into a container or paper towel.
- 5. If this is an old style analyzer, skip this step. If it is a new style analyzer, cut about ½-inch off of the end of the tube just removed. Discard the ½-inch piece.
- 6. Cut a 2-inch piece off of the end of the tubing and insert the barbed end of the female luer fitting into this piece. Make sure the barbed end is pushed in until the tubing is snug against the luer end. Install the barbed end of the male luer into the end of the tubing that is attached to the sample line. See Figure 2 on page 4.
- 7. Attach the 3-Way luer lock valve to the male and female luer fittings. Hand-tighten, making sure the fittings are snug, but do not over-tighten. Do not use tools on luer-type fittings. Do not attach anything to the center port on the valve at this time. See Figure 3 on page 5.
- **8.** Push the end of the 2-inch long piece of tubing back into the John-Guest fitting, making sure that the tube goes through both o-rings in the fitting. If it is an old style analyzer, reinstall the tubing in the fitting and screw the fitting back onto the analyzer.

- **9.** Open the front of the analyzer and slide the tube clamp onto the bypass line. On old style analyzers, the bypass tubing will look similar, but the detector block is in front of it.
- **10.** Restore power to the analyzer. Make sure the 3-way luer lock valve is open so that process stream water is flowing into the analyzer. See Figure 4 on page 5.
- 11. Turn the sample flow back on and make sure there are no leaks.
- **12.** Allow the analyzer to complete a few measurements to clear the lines of air. If pump tubing or new reagents were installed, then perform a prime cycle (refer to the instrument manual).

Running the Zero Standard

- **1.** With sample flowing, reset the analyzer calibration to default values. From the Setup Menu, choose Default Setup.
- Note: To avoid contamination, rinse the outside of the calibration cap and flush the calibration cap tubing with deionized water before inserting it into the standard. This is particularly important when calibrating multiple instruments with a single calibration set—getting chlorine standard into the blank will cause a false positive result. Store the cap in the plastic shipping bag when not in use.
- 2. Open the front of the analyzer and pinch the drain bypass line shut with the clamp (if it is not already pinched shut) to force the standard to flow through the reaction cell instead of out to waste. Place the valve in Position B. See Figure 2 on page 4.
- 3. Make sure the calibration cap has been cleaned inside and out with deionized water (do not use Organic-free Water) and dried with a KimWipe[®]. Install the calibration cap onto the zero standard (Organic-free Water), making sure the internal draw line (long tube extending to bottom of bottle) reaches the bottom of the bottle and the vent line is not submerged in liquid, see Figure 5 on page 6. The cap should be tight so that it can hold pressure.
- **4.** Attach the end of the external portion of the draw line to the open port on the 3-way valve.
- 5. Remove the check valve from the syringe and pull the plunger to fill the syringe with air. (If the check valve is on the syringe when the plunger is pulled back, no air will be drawn in.)

Note: The syringe is used to pressurize the bottle so the contents will flow into the analyzer.

- **6.** Reinstall the check valve on the syringe. Install the syringe/check valve assembly onto the vent line of the standard bottle.
- **7.** Place the standard bottle in a stable location higher than the analyzer (on top of the CL17 analyzer is fine).
- **8.** Watch the pinch valve to observe when the sample line is pinched shut (far left tube).
 - **a.** Old Style Analyzer: Once it is pinched shut, wait 30 seconds and then inject all of the air from the syringe. Leave the syringe on the vent line at this time.
 - **b.** New Style Analyzer: Once it is pinched shut, wait one minute and then inject all of the air from the syringe. Leave the syringe on the vent line at this time.
- 9. As soon as the pinch valve begins to open to allow flow through the sample line, turn the 3-way valve to the "A" Position. You may be able to hear a rush of liquid going through the tube into the analyzer and see the liquid level of the standard begin to drop (on the old style analyzers it will drop more rapidly than on the new style analyzers).
- **10.** Immediately remove the check valve and syringe from the vent line.

Figure 2 Calibration/Verification Assembly Mounted on a New Style CL17

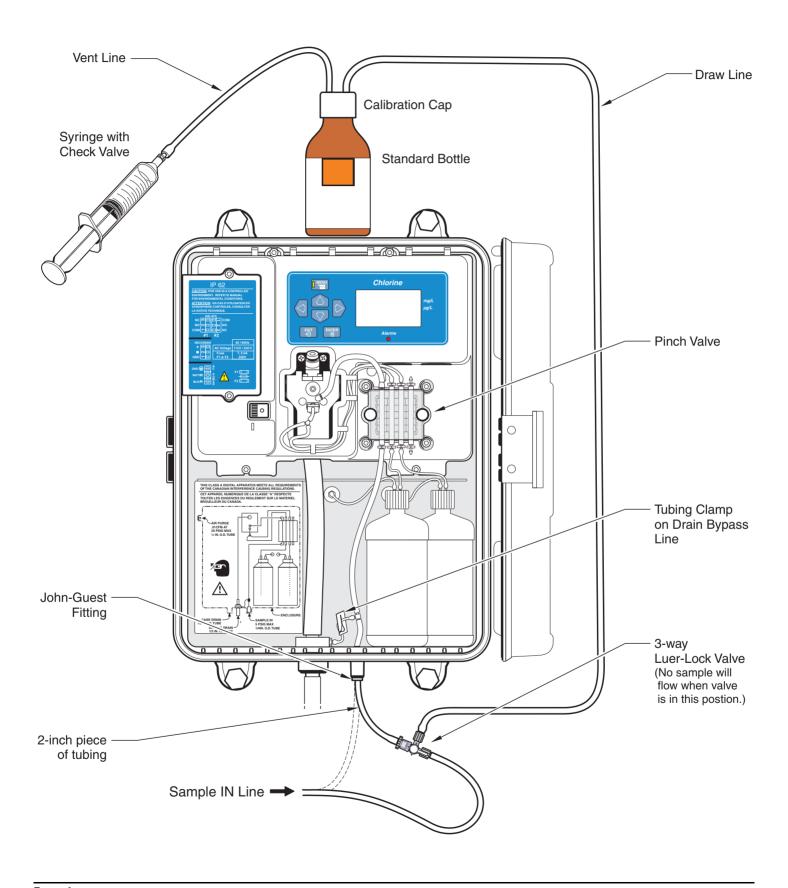


Figure 3 Assembling the 3-Way Valve and Tubing

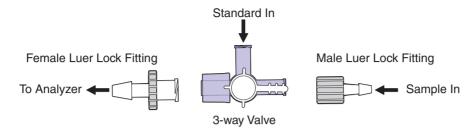
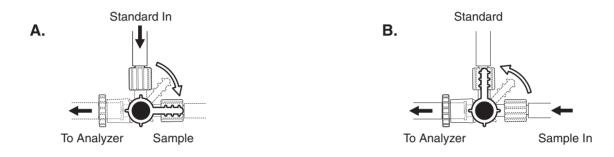


Figure 4 Valve Position



A. No sample flow in this position.

- B. Process sample flow position.
- **11.** If no flow was observed in step 9, turn the 3-way valve back to the "**B**" Position and repeat steps 4-10. See Figure 4.
- 12. Allow the analyzer to take four readings:

If Calibrating:

Allow the analyzer to make at least four measurements.

New Style Analyzer:

- a. After the four measurements are complete, press the MENU key.
- **b.** Using the up and down arrow keys, scroll to SETUP and press **ENTER**.
- c. Scroll to CAL ZERO and press ENTER.
- **d.** Using the up and down arrow keys, enter 0 for the expected value and press **ENTER**. Press **ENTER** again to set the measured value to be equal to 0.
- e. Exit to main display.

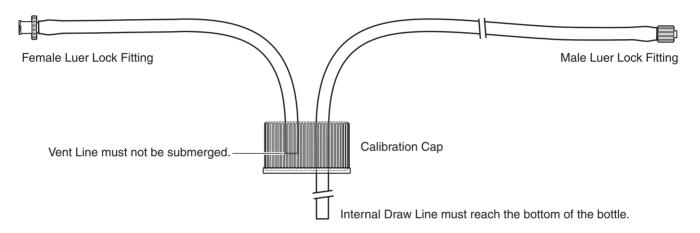
Old Style Analyzer:

- **a.** After the four measurements are complete, use the numeric keypad to enter **0**.
- b. Press the ZERO key.
- c. Exit to main display.

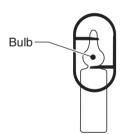
If Verifying:

- a. Allow the analyzer to make at least four measurements.
- **b.** Record each reading if the analyzer is not connected to a computer.
- **13.** Turn the lever on the 3-way valve to Position "**B**" to allow process water to be sampled (see Figure 4 on page 5).
- **14.** Move the Zero Standard (Organic-free Water) to a stable location below the level of the 3-way valve. Disconnect the draw line from the valve, remove the calibration cap from the bottle, and place the original cap on the bottle.
- **15.** Either discard the zero standard or immediately use it on the next analyzer being calibrated.

Figure 5 Calibration Cap Assembly



Preparing the Chlorine Standard



Important Note: Use the standard within two hours of preparation. After dilution, chlorine standards are not stable.

- 1. Make sure there is no Chlorine Standard in the bulb before breaking the ampule.
- 2. Break the top off the Voluette®* Chlorine Standard ampule.
- 3. Pour the contents of the ampule into a new 500-mL bottle of Organic-free Water. When the liquid starts to flow from the ampule, tilt the ampule and bottle to allow air into the ampule as it empties. Do not submerge any part of the ampule in the water. Make sure all the liquid is emptied into the bottle, and that none is left either in the ampule cap or body by tapping it side to side in the neck of the bottle.
- 4. Tightly cap the bottle with its original cap.
- **5.** Gently invert the bottle at least five times.

^{*} Voluette is a registered trademark of Hach Company.

Note: Take care when preparing the standard. An incorrectly prepared standard can skew the calibration curve and may result in a calibration failure.

6. Using the concentration of the ampule printed on the box, calculate the expected standard concentration using the following equation:

$$\frac{(Concentration of Ampule) \times (0.020 L)}{0.52 L} = Standard Concentration$$

7. Write the calculated concentration on the bottle.

Running the Chlorine Standard

1. Perform steps 1–15 of "Running the Zero Standard" using the prepared chlorine standard in place of the Zero Standard (Organic-free Water) and in step 12, use the exception below:

New Style Analyzer:

- a. After the four measurements are complete, press the MENU key.
- **b.** Record each reading if the analyzer is not connected to a computer.
- c. Using the up and down arrow keys, scroll to SETUP and press ENTER.
- d. Scroll to CAL STD and press ENTER.
- **e.** Using the up and down arrow keys, enter the calculated concentration and press **ENTER**.
- **f.** Press **ENTER** again to set the slope to its new value.
- g. Exit to main display.

Old Style Analyzer:

- After the four measurements are complete, use the numeric keypad to enter the calculated concentration.
- **b.** Press the **STD** kev.
- c. Exit to main display.

Calculating Average Concentration, %RSD and %Difference

1. Calculate the average concentration of the standard (omit the first replicate) by either manually calculating it or by using a spreadsheet program.

$$Avg = \frac{a+b+c}{3}$$

where a, b and c are the actual concentrations for the 2nd, 3rd and 4th readings of the standard.

2. Calculate the standard deviation (SD) and the % relative standard deviation (%RSD). The %RSD value should be less than 3%.

$$SD = \frac{((a - avg)^2 + (b - avg)^2 + (c - avg)^2)^{1/2}}{3}$$

$$%RSD = \frac{SD \times 100}{avg}$$

Important Note: If %RSD value is greater than 3%, rerun the test with fresh standard reagents.

3. Calculate the % difference from the calculated concentration.

This value should be within ± 5% (or between +5.0% and −5.0%).

$$\%Dif = \left(\frac{Avg}{Y} \times 100\right) - 100$$

where Y = calculated standard concentration written on the bottle, (step 6 of Preparing the Chlorine Standard).

Important Note: If %Dif is greater than 5%, rerun the test with fresh standard reagents.

Technical Notes

- Hach Company recommends performing a verification after each reagent change.
- Old-style CL17 analyzers without a bypass line should get 4–5 replicates from one bottle. Leaving the bypass line open will allow only 1–2 replicates. Since the first reading is generally discarded, 1–2 replicates is not enough. Always make sure the bypass line is closed.
- New-style CL17 analyzers should get 10-15 replicates from one bottle.
- Store ampules in their cardboard cylinders in the refrigerator to reduce any adverse effects from heat or light. Discard them if more than a year has passed from the date on the ampule.
- The stir bar in the old-style CL17 can be dislodged when the pressure in the bottle is released. Result: low response, Symptom: lack of color formation, or minimal color formation. You should see bright pink in the flow cell window of the old-style analyzer.
- Pressurize the bottle immediately before opening the 3-way valve. Although the cap is snug, it will not hold pressure for long periods of time.

Reorder Information

Descrip	otion	Catalog Number
CL17 Calibration/Verification Kit		54490-00
Reagent Refill Kit		28359-00
	Two Bottles 500 mL ±5 mL Organic Free Water One 20 mL Voluette® 100-130 ppm Chlorine Standard	