Chlorine, Total

DOC316.53.01156

USEPA[1] Amperometric Buret Titration Method[2]

Method 8168

(0.5 mg/L and above)

Buret Titration

Scope and Application: For water and wastewater.

[1] USEPA accepted (4500 Cl^- D).

[2] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test preparation

Before starting the test:

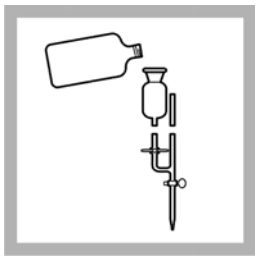
Chlorine can be lost from the sample during sample collection. Review the precautions in *Sample collection, preservation and storage* before the test is started.
Use only a 50-mm stir bar. The wrong size can cause the loss of chlorine, unstable readings and loss of method sensitivity, especially when measuring low level chlorine concentrations.
For added convenience when stirring, use the TitraStir® apparatus.
When a new probe is placed in service or when the probe has not been used recently, prepare it according to the Probe Stabilization instructions in the Amperometric Titrator Instruction Manual.

Collect the following items:

Description	Quantity
Phenylarsine Oxide Solution, 0.00564 N	1 bottle
Acetate Buffer Solution	1 mL
Potassium Iodide Powder Pillow	1
Beaker, 250-mL	1
Graduated cylinder, 250-mL	1

See *Consumables and replacement items* for reorder information.

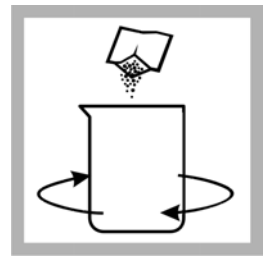
Buret titration



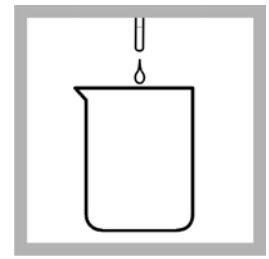
1. Fill the 5-mL automatic buret to the zero mark with 0.00564 N Phenylarsine Oxide (PAO) Solution.

2. Put a 50-mm stir bar into a 250-mL beaker.

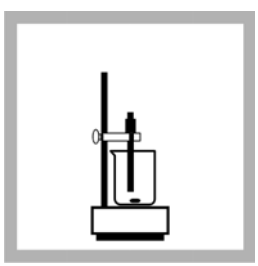
Use a graduated cylinder to measure 200 mL of sample. Add the sample to the beaker.



3. Add the contents of one Potassium Iodide Powder Pillow. Swirl to mix.

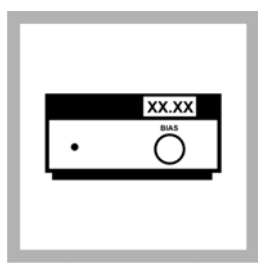


4. Add 1.0 mL of pH 4 Acetate Buffer Solution to make the prepared sample.



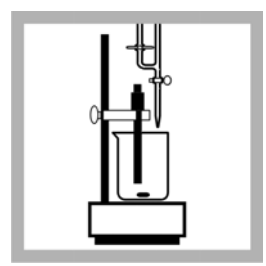
5. Place the beaker of prepared sample on the TitraStir titration stand and turn on the stirring motor. Put the tip of the probe fully into the prepared sample. The platinum wires must be submerged.

If a stir plate other than the TitraStir® is used, set the speed for moderate mixing. Do not adjust the speed after this point.

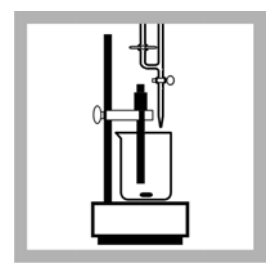


6. Turn the BIAS control knob to adjust the value on the display to approximately 1.00.

The BIAS adjustment controls the slope of the titration curve. The actual value is not important. Only the relative value as the titration continues is important. A precise adjustment is not necessary.

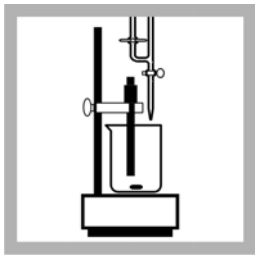


7. Dispense the titrant into the beaker in small increments while monitoring the values on the Amperometric Titrator. The values will decrease.

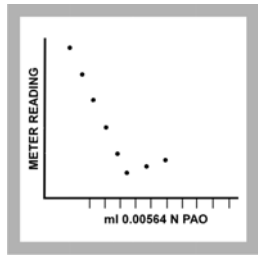


8. Continue dispensing slowly. Near the end point of the titration, write down the value on the display and the corresponding total volume of titrant that was added. Read the volume to the nearest 0.01 mL. Add a small amount of titrant and wait several seconds for a stable value. Write down the value.

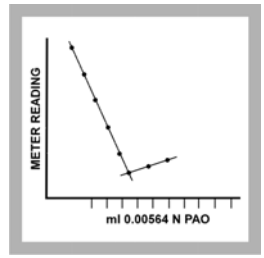
Buret titration (continued)



9. Continue the titration by recording at least three points on the downward sloping curve and at least three points after the end point has been reached. The value on the display will not change significantly after the end point.



10. Make a graph of the titration. Plot the values from the amperometric titrator on the vertical axis and the corresponding volume of titrant on the horizontal axis.



11. Draw the two best intersecting lines through the points as shown above. Find the volume of titrant to the nearest 0.01 mL at the intersection of the two lines. This is the mL titrant end point. This volume is equivalent to the total chlorine concentration in mg/L.

mL titrant =
mg/L total chlorine as Cl_2

Interferences

Refer to the Amperometric Titrator Instruction Manual for a discussion of sources of errors and interferences using the amperometric methods.

Sample collection, preservation and storage

Start the chlorine analysis immediately after the samples are collected. Chlorine is a strong oxidizing agent and is not stable in natural waters. Chlorine reacts quickly with various inorganic compounds and slowly oxidizes organic compounds. Many factors such as sample composition, sunlight, pH, temperature, and salinity can cause the decomposition of chlorine in water.

Do not use plastic containers because plastic can react with and consume chlorine. Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of demineralized water) for at least 1 hour. Rinse thoroughly with demineralized or distilled water. If sample containers are rinsed thoroughly with demineralized or distilled water after use, only occasional pre-treatment is necessary.

A common error in testing for chlorine is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes before sample collection. Let the sample container overflow with the sample several times, then cap the container so that there is no headspace (air) above the sample. Start the chlorine analysis immediately.

Summary of method

Total chlorine is measured after the addition of potassium iodide and acetate buffer by a titration at pH 4 with PAO solution to the amperometric end point. The amperometric titration method has greater sensitivity and accuracy when compared to colorimetric methods. Refer to the Amperometric Titrator Instruction Manual for more information.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Total Chlorine Reagent Set (approximately 200 tests), includes:			2460700
(2) Acetate Buffer Solution, pH 4	1 mL	100 mL MDB	1490932
(1) Phenylarsine Oxide Solution, 0.00564 N	varies	1 L	199953
(2) Potassium Iodide Powder Pillows	1	100/pkg	107799

Required apparatus

Description	Unit	Catalog number
Amperometric Buret Titrator System, 115 VAC	each	1930010
Amperometric Buret Titrator System, 230 VAC	each	1930012
Beaker, 250-mL	each	50046H
Graduated Cylinder, 250-mL	each	50846
Stir bar, 50 mm	each	2095355
TitraStir® apparatus, 115 VAC	each	1940000
TitraStir® apparatus, 230 VAC	each	1940010
pH paper, 0–14 pH range	100/pkg	2601300

Optional reagents and apparatus

Description	Unit	Catalog number
Chlorine Standard Solution, 10 mL Voluette® Ampules, 50–75 mg/L	16/pkg	1426810
Chlorine Standard Solution, 2 mL PourRite® Ampule, 50–75 mg/L	20/pkg	1426820
Voluette Ampule breaker, 10 mL	each	2196800
PourRite Ampule breaker, 2 mL	(each)	2484600
Chlorine Standard Solution, 2-mL PourRite® Ampule, 25–30 mg/L	20/pkg	2630020
Water, deionized	500 mL	27249

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