Determination of Chlorite—Quick 2-step method

DOC316.52.93149

Based on Standard Methods 4500-ClO_2 E for drinking water and wastewater

This application note covers the following applications:

Method	Range	Titrant	Buffer, KI and Acid	Sample volume
Chlorite (H) (Quick, 2-step)	0.100 to 5.00 mg ClO_2^-/L Cl_2 more than 0.100 mg/L	0.00564N PAO	1 mL pH 7 1g 2 mL 2.5N HCl	200 mL
Chlorite (L) (Quick, 2-step)	0.100 to 5.00 mg ClO_2^-/L Cl_2 less than 0.100 mg/L	0.00564N PAO	1 mL pH 7 1g 2 mL 2.5N HCl	200 mL

1. Important information

- The AT1000 is factory programmed to use the 10-mL syringe. The method uses a 5-mL syringe. Before analysis, make sure to change the syringe volume on the instrument. Refer to 10.1 Changing the syringe volume on the AT1000.
- Treat glassware to decrease chlorine demand before analysis. Soak glassware in dilute bleach solution for a minimum of 1 hour. Rinse thoroughly with deionized water. Use the glassware for this method only.
- Minimize agitation when measuring sample volumes. Remove sample portions with a volumetric pipette. Always put the tip of the pipette at the bottom of the sample container. If using 200 mL sample portions, use a 100-mL volumetric pipette to withdraw two portions of sample.
- Always use organic free water for sample dilution.
- Rinse the electrode and anti-diffusion tip with DI water before every titration.
- Purge the syringe each day before the analysis.
- Do not substitute buffers designed for calibrating pH meters. They contain dyes that can interfere with amperometric titration.
- Do not use buffers contaminated with mold or bacteria.
- Clean the electrode periodically Refer to 10.2 Cleaning the Electrode. Clean and correctly maintained electrodes are necessary to get sharp amperometric endpoints. Clean the electrode when noise in the titration curve interferes with detection of the endpoint. The electrode cleaning duration is approximately 10 minutes. Always clean new electrodes before the analysis.
- The electrode orientation is very important. Noise that occurs when the electrode is not correctly oriented can interfere with accurate detection of the equivalence point. Refer to 4.1 Position of the electrode and injection tips for information on electrode and delivery tip positioning for this method.
- A fast stirring can pull air into the sample and bubbles may get caught on the electrode tip. Air bubbles on the electrode tip have a negative effect on the analysis results. Adjust the stirring speed during a titration with the up and down arrows on the instrument. Alternatively, change the stirring speed in the method edit window.
- The method is programmed to measure **200 mL** of sample. **To use less than 200 mL of sample, change the sample volume at the method edit window**. Refer to 10 Appendix for more information.

2. Introduction

This application is based on the Standard Methods 4500-ClO_2 E, an amperometric method which distinguishes three different compounds: chlorine dioxide (ClO_2), free chlorine (Cl_2) and chlorite (ClO_2^-). **This method describes a quick 2-step method for chlorite only.** Both the **L** and **H** methods **cover the same range in chlorite**. The difference in the methods is in the range for Titration 1. The methods are as follows:

ClO_2^- (H)	0.100 – 5.00 mg ClO_2^- /L	Cl_2 **above** 0.100 mg (titration 1)
ClO_2^- (L)	0.100 – 5.00 mg ClO_2^- /L	Cl_2 **less than** 0.100mg (titration 1)

3. Principle

Two amperometric titrations are done on one titration sample. The results of each titration are stored and at the end of the sequence the concentration of chlorite (ClO_2^-) is displayed.

The sample pH is adjusted to 7 by addition of buffer, and the sample is then degassed with nitrogen to remove chlorine that might be present. An excess of KI is added and the titration is launched to neutralize any Cl_2 not volatized by the degassing. Next, concentrated hydrochloric acid is added to the cell to give the ClO_2^- determined during the second titration.

The table below gives details of the sequence:

Titration 1	Cl_2 not volatized by the nitrogen gas purge
Titration 2	ClO_2^-

4. Electrode and reagents

Electrode: Pt-Pt electrode with temperature sensor, IntelliCAL MTC695

Titrant: Phenyl Arsine Oxide (PAO) 0.00564 eq/L solution

Reagents: pH 7 phosphate buffer
Potassium iodide (KI) powder
Hydrochloric acid (HCl) 2.5 N solution

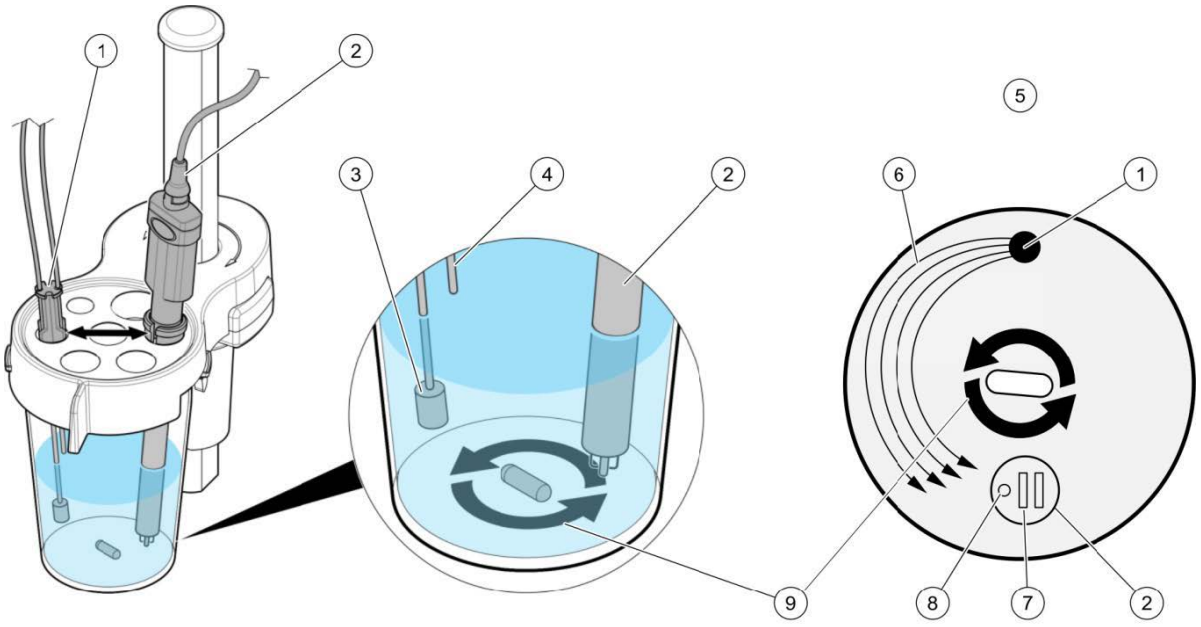
Deionized water

4.1. Position of the electrode and injection tips

The position of the electrode and injection tips in the titration cell is very important in this application. **If the electrode is incorrectly positioned, noise in the titration curve can adversely affect the results.**

Refer to the steps and the figure that follows to correctly position the electrode and injection tips.

1. Put the electrode in the opposite hole of the tubes in the sensor holder (items 1 and 2 in figure).
2. Turn the electrode so that the platinum wires are perpendicular to the sample flow and the temperature sensor is before the platinum wires (items 6 to 8 in figure).
3. Put the tube from the pump above the sample surface (item 4 in figure).
4. Make sure that the tube with the anti-diffusion tip is fully into the sample (item 3 in figure).



1. Tube holder	4. Tube from the pump	7. Platinum wires
2. Electrode	5. Top view	8. Temperature sensor
3. Anti-diffusion tip	6. Flow direction	9. Stirring direction

5. Settings

5.1. Chlorite determination

Two methods, Chlorine Dioxide L and Chlorine Dioxide H are available. The difference between **L** and **H** are the settings for **Titration 1**.

If the expected equivalent volume for titration 1 is very low (< 0.1 mL), use the **L** applications. If not, use the **H** applications. The ranges for Chlorine Dioxide, Chlorite, and Chlorine are the same for the two methods.

The settings below have been defined with:

- Sample volume: 200 mL
- Titrant concentration: 0.00564 eq/L Phenyl Arsine Oxide
- Continuous imposed voltage: 100 mV (reversed at each analysis)
- Syringe volume: **5 mL**
  The default syringe volume for the AT1000 is set to 10 mL. These applications need a 5-mL syringe. When loading an application, if the message syringe to replace is displayed, change the syringe volume in the Syringe management option of the Maintenance menu. Refer to 10 Appendix for more information.
- If a blank is tested, analyze this as a sample. The BLANK option is not compatible with this method.

Name	**Default parameter**		**Unit**
Application name	**Chlorite L**	**Chlorite H**	
Syringe			
Advisable	5 mL (Hamilton)		
Sample			
Name	Water ?[1]		
Amount	200		mL
QC			
Name	QC Sample		
Probe			
Recommended electrode	MTC695		
Titrant PAO 0.00564 N			
Real concentration	0.00564		eq/L
Sample preparation 1 (titration 1)			
Active	Yes		
Message	Sample amount: 200 mL in GWB and press OK		
Stirring speed	0		%
Sample preparation 2 (titration 1)			
Active	Yes		
Message	Add 1.0 mL of buffer pH 7 then start purge with N_2 and press OK		
Stirring speed	0		%
Purge (titration 1)			
Active	Yes		
Time	15		minutes
Stirring speed	0		%
Message	Purge in progress. Please wait…		
Sample preparation 3 (titration 1)			
Active	Yes		
Message	Pour the purged sample into a titration beaker – Add a stir bar and press OK		
Stirring speed	0		%
Manual addition 1 (titration 1)			
Active	Yes		
Message	Place the sample on the instrument - Add **1.0 g** of KI and press OK		
Stirring speed	0		%
Reagents mixing (titration 1)			
Active	Yes		
Time	5		seconds
Stirring speed	1		%
Message	Reagents mixing. Please wait…		
Dip electrode (titration 1)			

[1] **?** in the name, shows that the sample name will be automatically incremented with a number for each analysis

Name	Default parameter		Unit
Application name	**Chlorite L**	**Chlorite H**	
Active	Yes		
Message	Dip electrode in sample and press OK		
Stirring speed	1		%
Titration 1			
Active	Yes		
Stirring speed	1		%
Predose ordinate	0.1	0.4	µA
Predose speed	0.3	2.0	mL/min
Delay	20		seconds
Max. vol. stop point	5		mL
Stop on last EQP	Yes		
Increment size	0.001	0.010	mL
EQP min. ordinate	-0.03	-0.1	µA
EQP max. ordinate	0.03	0.2	µA
Result 1 name	Intermediate 3		mL
R1 hide	Yes		
R1 min	0		mL
R1 max	5		mL
R1 QC min	0		mL
R1 QC max	5		mL
Result 2 name	C		
R2 hide	Yes		
R2min	0		mL/mL
R2max	0.025		mL/mL
R2QC min	0		mL/mL
R2 QC max	0.025		mL/mL
R2 equation	FX*(R1/SA) = G3		
R2 user value	1		
Manual addition 2 (titration 2)			
Active	Yes		
Message	Add 2.0 mL of HCl 2.5 N then place the solution in the dark and press OK		
Stirring speed	1		%
Reaction (titration 2)			
Active	Yes		
Message	5		minutes
Stirring speed	0		%
Message	Dark reaction in progress. Please wait…		
Dip electrode (titration 2)			
Active	Yes		
Message	Place the sample on the instrument then dip the electrode in sample and press OK		
Stirring speed	0		%
Titration 2			
Active	Yes		
Stirring speed	1		%
Predose ordinate	1.0		µA
Predose speed	2.5		mL/min
Delay	20		seconds
Max. vol. stop point	15		mL
Stop on last EQP	Yes		
Increment size	0.010		mL
EQP min. ordinate	-0.1		µA
EQP max. ordinate	0.2		µA
Result 3 name	Intermediate 4		mL]
R3 min	0		mL/mL
R3 max	0.075		mL/mL
R3QC min	0		mL/mL
R3QC max	0.075		mL/mL
R3 equation	FX*(R1/SA) = G4		
R3 user value	1		
Result 3 name	D		

Name	Default parameter		Unit
Application name	Chlorite L	Chlorite H	
Result 3 hide	Yes		
Result 4 name	Chlorite		
R4 hide	No		
R4 min	0		mg ClO_2^-/L
R4 max	6		mg ClO_2^-/L
R4QC min	0		mg ClO_2^-/L
R4 QC max	6		mg ClO_2^-/L
R4 equation	V1/V1*FX*(G4+(F3/SA)-G3)*TC*16863		
R4 user value	1		

5.2. Recommendations for modification of the settings

Some parameters can be adjusted, but this is mainly for analysis time reduction. It should be noted that the impact of any adjustments can be a loss of precision on the results.

5.2.1. Sample preparation messages

Messages for sample preparation can be removed from the sequence by setting **No** in the field **Active** in the message section. In this way, the instrument will not give information about sample preparation during the analysis sequence. Methods which can be deactivated are the following:

- Automatic addition 1 (titration 1)
- Manual addition 1 (titration 1)
- Manual addition 2 (titration 1)
- Manual addition 3 (titration 2)
- Sample preparation 1 (titration 1)
- Sample preparation 2 (titration 1)
- Purge (titration 1)
- Sample preparation 3 (titration 1)
- Manual addition 1 (titration 1)
- Manual addition 2 (titration 2)
- Reaction (titration 2)

Note: It is not recommended to change the increment sizes because they have been optimized for the best equivalent point detection.

5.2.2. Pre-doses in ordinate

Predoses in ordinate are used to decrease the titration duration. They have been fixed for all titrations for chlorine dioxide and chlorite applications. Their parameters (**Predose ordinate** and **Predose speed**) have been set empirically and are system dependent. A titration starting with an ordinate under the target can happen but does not have an impact on the result. The table that follow shows some indications.

Observation	Resolution
The titration is still too long (too many points before inflection).	Decrease the predose ordinate in the ordinate section or increase the titrant addition speed (no more than 2.5 mL/min).
The initial point of the titration curve is too low (not enough points before inflection) and the EQP is not detected.	Decrease the titrant addition speed in the ordinate section or increase the predose ordinate.

6. Procedure

6.1. Before starting

NOTICE: All glassware must be treated for chlorine demand before any analysis.

- Soak all glassware in dilute bleach solution for at least 1 hour. Rinse thoroughly with deionized water. Use the glassware for this method only.
- Minimize agitation when measuring sample volumes. Remove sample portions with a volumetric pipette. Always put the tip at the bottom of the sample container. If using 200 mL sample increments, use a 100 mL pipette to withdraw two portions of sample.
- Always use organic free water for sample dilution.
- Do not substitute buffers designed for calibrating pH meters. They may contain dyes that interfere in amperometric titration
- Do not use buffers contaminated with mold or bacteria.

- Rinse the electrode and anti-diffusion tip with deionized water before every titration.

6.2. Sample analysis

1. Use a pipette to add 200 mL of sample into a Gas Washing Bottle (GWB).
2. Add 1.0 mL of phosphate buffer pH 7 and swirl to mix.
3. Insert the purge tube and dispersion tip into the GWB. Connect the GWB inlet to a tank of purified nitrogen.
4. Use a needle valve to adjust the flow of nitrogen to provide a steady stream of bubbles through the sample.
5. Purge the nitrogen gas through the sample for 15 minutes.
6. After the purge, transfer the purged sample to a 250-mL glass beaker. Add a magnetic stir bar and put in the instrument.
7. Add 1.0 g of KI.
8. The reagents are mixed. Dip the electrode and addition tip into the sample.
9. Titration 1 starts.
10. When titration 1 is finished, raise the electrode holder.
11. Add 2.0 mL of 2.5 N hydrochloric acid (HCl) and stir for a few seconds.
12. Carefully remove the sample from the stirrer and put the sample in a dark environment.
13. Wait 5 minutes for the reaction.
14. At the end of the 5 minutes, carefully put the sample back onto the instrument.
15. Dip the electrode and addition tip into the sample.
16. Titration 2 starts.
17. At the end of titration 2, the results are shown.

7. Results

7.1. Displayed Results

At the end of the analysis sequence the following result is displayed:

ClO_2^- in mg/L as ClO_2^-

7.2. Results calculation

ClO_2^- calculation:

$$ClO_2^- = D \times N \times 16863$$

Where:

D = Result of titration 2 (mL titrant at equivalent point/mL of sample)

N = Concentration of the titrant

8. Examples of chlorite determination

The results described below are indicative and obtained for a given sample in optimized conditions following good laboratory practices. These indicative values are sample-dependent, electrode-dependent and operating cell-dependent.

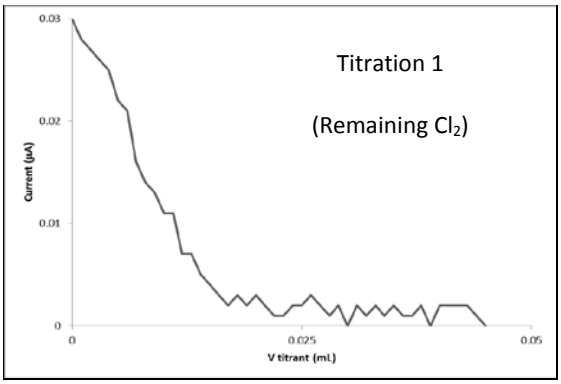
Results for four determinations of a synthetic mixture:

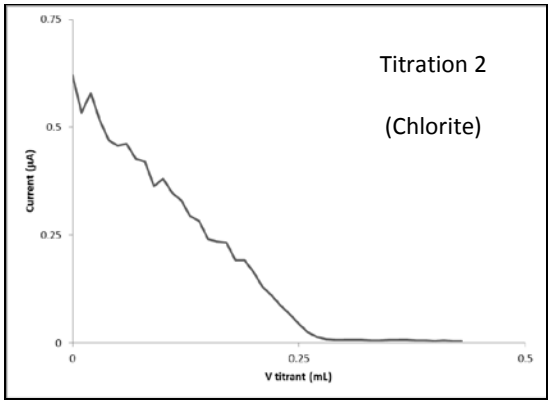
Sample: 200 mL of solution
Application: Chlorite **L**
Temperature of analysis: Room temperature
Mean values: ClO_2^-: 0.154 mg/L
Cl_2: -

Standard deviation: ClO_2^-: 0.008 mg/L
Cl_2: -

Relative standard deviation: ClO_2^-: 5.21%
Cl_2: -

Example of titration curves:





9. Bibliography

- *Standard Methods 4500-ClO_2 E*
- *AutoCAT 9000 Manual 50081 3rd edition*

10. Appendix

10.1. Changing the syringe volume on the AT1000.

The AT1000 instrument is delivered with the syringe volume set to 10 mL. The amperometric applications require a 5-mL syringe volume. The syringe volume must be changed before the applications can be started. Complete the steps that follow to change the syringe volume:

1. From the HOME screen select MAINTENANCE > SYRINGE MANAGEMENT > SYRINGE VOLUME CHANGE.

***Note**: If the AT1000 instrument has 2 syringes, select the syringe to edit.*

2. Use the arrow keys to select 5 ML (HAMILTON), then push **SELECT**. The display shows APPLYING 5ML (HAMILTON) SETTINGS followed by SYRINGE VOLUME UPDATED.
3. Push **OK**.
4. Push **HOME** to go back to the HOME screen.

10.2. Cleaning the Electrode

This procedure should be done before first use, after dry storage, and when the electrode response is slowed or equivalence points are missed.

1. Prepare a cleaning solution of 20-mL HNO_3/100 mL. Always add acid to water! Always wear personal protective equipment!
2. From the HOME screen, select MAINTENANCE > CLEAN PT-PT ELECTRODE
3. Pour enough solution in the beaker to cover the electrode.
4. Select OK

***Note**: If the stirrer does not start, push the up and down arrows.*

5. After five minutes, when prompted, rinse the electrode with DI water and fill the beaker with enough tap water to cover the electrode
6. Put the PtPt electrode in the water and select OK.
7. After five minutes, the cleaning is complete.

10.3. Hide or Show a Result

1. From the Home screen, select SETTINGS>APPLICATIONS.
2. Select EDIT from the list of actions.
3. Highlight the application and push **EDIT**.
4. Use the down arrow to go to METHOD>RESULTS.
5. Select YES or NO to show or hide results:
   - **Yes**—the result is not shown (hidden) at the end of the titration
   - **No**—the result is shown at the end of the titration

10.4. Titrant calibration

10.4.1. Set the Titer directly from the C.O.A.

Before the Titer is entered directly from the certificate of analysis (C.O.A.) refer to the customer laboratory standard operation procedure (S.O.P.) to determine if this is acceptable.

1. From the HOME screen, select SETTINGS>APPLICATIONS>EDIT

2. Select the application for the titer
3. Scroll down in the application to Titrant
4. Select REAL CONCENTRATION
5. Using the arrow keys or a keyboard, enter the titer value from the C.O.A.
6. Select OK
7. Go back to the Home screen.

   ***Note**: This step is needed only once for each syringe, even if there is more than one method associated with it. Enter the value from the C.O.A. using the up and down arrow keys or a USB Keyboard. Go back to the Home Screen.*

10.4.2. Calibration of the titrant with 0.0282N Iodine

The 0.000564N PAO titrant is calibrated against a standard solution of 0.0282 N Iodine

$$PhAsO\ (PAO) + I_3^- + 2H_2O \rightarrow PhAsO(OH)_2 + 3I^- + 2H^+ \qquad (Ph=phenyl)$$

The iodine solution can also be calibrated. The procedure is described in the in the Sulfite working procedures, which are included on the Amperometric titration method key.

If the standard iodine concentration given in the Certificate of Analysis (or obtained by calibration) is different from the default concentration of 0.0282 N, the real value must be manually entered as the concentration of the standard.

10.4.2.1. Procedure

Accurately pipette 0.5 mL of iodine standard solution 0.0282 N and dilute it to 200 mL with deionized water.

Calibrate the titrant using the titrant calibration option instead of the sample analysis. Add KI powder and pH 4 when required. On a titrator with 2 pumps, pH 4 buffer is pumped using Pump 2.

10.4.2.2. Results

The results described below are indicative and obtained following good laboratory practices. These indicative values are sample-dependent, electrode-dependent and operating cell–dependent.

The instrument calculates the titrant concentration directly in eq/L.

$$C_{(PAO)} = \frac{V_{(I2)} * C_{(I2)}}{V_{(PAO)}}$$

C(PAO) : Concentration of titrant: Phenylarsine Oxide (PAO) in eq/L,
C(I2) : Concentration of standard: Iodine (I2) in eq/L, currently 0.0282 eq/L
V(I2) : Volume of standard: Iodine (I2) in mL, currently 0.5 mL
V(PAO) : Volume of the titrant: Phenylarsine Oxide (PAO) in mL added to reach the equivalent point

Experimental conditions:

- **Burette volume**: 5 mL
- **Sample**: 200 mL of deionized water with 0.5 mL of standard solution iodine 0.0282 eq/L
- **Addition**: 0.1 g KI and 1 mL buffer pH 4
- **Titrant**: PAO 0.000564 eq/L
- **Acceptable Range**: 0.000564N +/- 10% (0.000508-0.000620N)

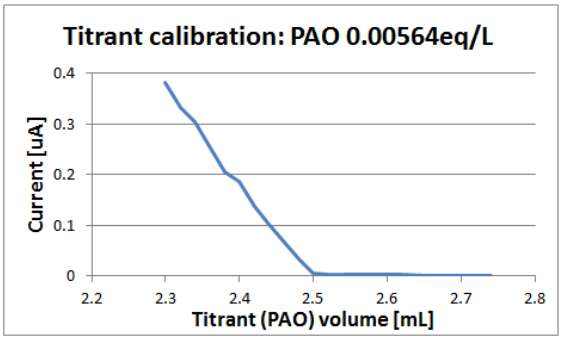
Results for 5 replicates of the titrant:

Average concentration	0.00561	eq/L
SD	0.00002	eq/L
RSD	0.4	%

Settings:

Titration curve: µA vs. volume of titrant

- **Settings**: Refer to 10.4.2.3 Titrant calibration settings (default parameters)
- **Number of determinations**: 5 samples
- **Temperature of analysis**: Room temperature



10.4.2.3. Titrant calibration settings (default parameters)

	Setting	Unit
Titrant name	PAO	
Nominal concentration	0.00564	eq/L
Calibration frequency	0	days
Stirring speed (%)	1	%
Predose volume	2.1	mL
Delay	20	seconds
Stop on last EQP	Yes	
Min increment size	0.02	mL
Max increment size	0.05	mL
EQP min. ordinate	-0.1	µA
EQP max. ordinate	0.2	µA
Titrant calibration result		
Min. titrant concentration	0.0055	eq/L
Max. titrant concentration	0.0058	eq/L
Standard		
Name	Iodine	
Amount	0.500	mL
Min amount	0.490	mL
Max amount	0.510	mL
Concentration	0.0282	eq/L

10.4.2.4. Modification of the parameters for the titrant calibration

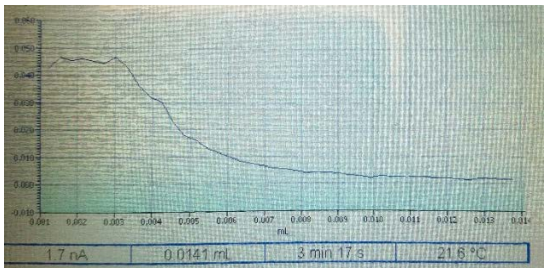
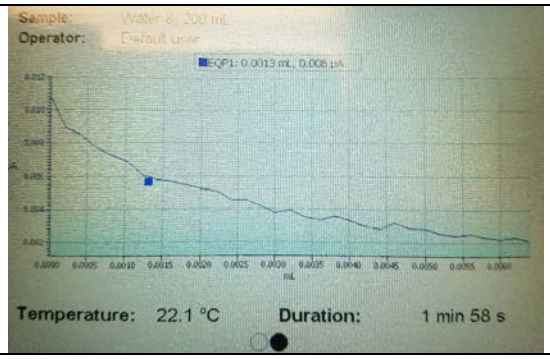
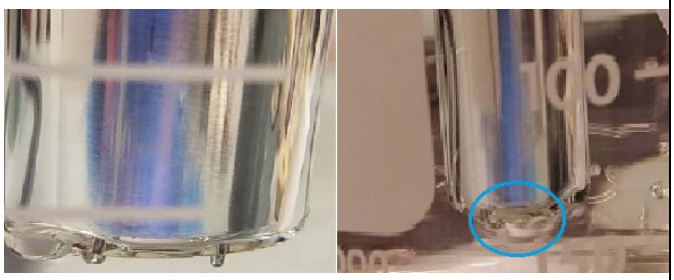
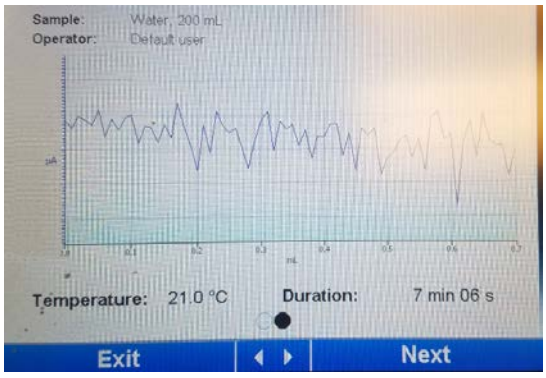
The titrant calibration application has been optimized for an amount of standard higher than 0.49mL, a standard concentration higher than 0.0270 eq/L and a titrant concentration between 0.0055 eq/L and 0.0058 eq/L.

Based on the concentration of the standard, the titrant volume needed for the equivalence will be affected by an amount or a concentration of the standard different to the default values. The predose volume can be adjusted in relation to this amount, to ensure about 0.2 mL of titrant before the equivalence point.

As an example, the table below shows the effect of the standard concentration on the equivalent volume and the optimum predose volume as a function of the equivalent volume expected.

Standard volume and concentration	Titrant concentration	Theoretical equivalent titrant volume	Number of addition points before equivalent point detection with default predose at 2.1mL	Optimized predose volume
0.50 mL at 0.0270 eq/L	0.0058 eq/L	2.33 mL	11	2.1 mL
0.50 mL at 0.0270 eq/L	0.0055 eq/L	2.45 mL	18	2.2 mL
0.50 mL at 0.0290 eq/L	0.0058 eq/L	2.50 mL	20	2.3 mL
0.50 mL at 0.0290 eq/L	0.0055 eq/L	2.64 mL	27	2.4 mL

11. Troubleshooting

Symptom	Probable cause	Solution
No clear equivalence point, equivalence point not found.		Concentration too low? Do a cleaning procedure After cleaning, analyze a mid-range standard to verify performance.
Titration curve is noisy. No or incorrect equivalence point found. Electrode responds slowly; titration takes longer than usual.		Concentration too low? Check for bubble caught on electrode Verify electrode is properly oriented Clean electrode
Predose exceeds the set ordinate. Electrode responds slowly; titration takes longer than usual.	Dirty or polarized electrode	The analyte level is too low to be detected. Clean the electrode.
There are bubbles on the electrode tip.		Picture on the left shows no bubbles. Picture on the right shows a bubble caught on the electrode. Adjust the stirring speed to 35-40% which will not normally cause bubbles to occur. If analyzing a standard, make sure that the volume of water used is sufficient.
Flat signal Noisy signal Electrode is dirty.		No analyte (blank) No buffer added No KI added Clean the electrode.

11.1. Waste management

The laboratory has the responsibility to follow all of the federal, state, and local regulations governing waste management (particularly the hazardous waste identification rules) and land disposal restrictions. The laboratory must minimize and control all releases from fume hoods and bench operation to protect the air, water and land. Compliance with all sewage discharge permits and regulations is also required.

For more information on waste management refer to the *Waste Management Manual for Laboratory Personnel* guide, available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N. W., Washington D. C. 20036, (202) 872-4477.

12. Parts List

Description	Quantity per test	Unit	Item no.
Required reagents			
Phenylarsine oxide (PAO) titrant, 0.00564 N	varies	1 L	199953
Buffer solution, pH 7 (automatic addition)	About 2 mL	1 L	2155353
Acetate buffer solution, pH 7 with dropper (manual addition)	About 2 mL	100 mL	2155332
Potassium iodide, ACS or better[2]	0.1 g	100 g	16726H
Swiftest™ dispenser, with refill vial	varies	each	2834100
Refill vial, Siwftest dispenser	varies	0.1 g	2105660
Nitric acid, 1 :1	20 mL	500 mL	254049
Required equipment			
0.1-g scoop for addition of KI to the sample	1	each	2657201
Beaker, low form Griffin, glass, 250 mL,	1	each	50046H
Beaker, low form Griffin, glass, 250 mL	1	12/pkg	50076H
Cylinder, graduated, 250 mL	1	each	50846
Magnetic stir bar, PTFE coated, 2 x 3/8 in.	1	each	5008500
Gas washing bottle, 1200 mL	1	each	2662200
Optional reagents			
Chlorine Standard Solution, Voluette® ampules, 50–75 mg/L 16, 10 mL ampules	varies	16/pkg	1426810
Chlorine standard solution, Pour Rite® ampules, 25–30 mg/L 20, 2 mL ampules	varies	20/pkg	2630020
Dilution Water, ASTM Type III organic-free	varies	500 mL	2641549

[2] The KI granules must be ground with a mortar and pestle for this method if the Swiftest is not used.

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