# Quaternary Ammonium Compounds DC

## **Direct Binary Complex Method**

## 0.2 to 5.0 mg/L as CTAB (cetyl-trimethylammonium bromide)

Method 8337 Powder Pillows

Scope and application: For cooling tower water and pool/spa water.

# ] Test preparation

## Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for specific instruments.

To use the table, select an instrument, then read across to find the applicable information for this test.

Instrument	Sample cell orientation	Sample cell
DR 6000	The fill line is to the right.	2495402
DR 3800		日
DR 2800		<u>10 mL</u>
DR 2700		
DR 1900		
DR 5000	The fill line is toward the user.	
DR 3900		

#### Table 1 Instrument-specific information

## **Before starting**

The sample cells can show a pink or purple film after several analyses. To remove the film, rinse with 1.0 N sodium hydroxide solution, then clean with a detergent and rinse with deionized water.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

## Items to collect

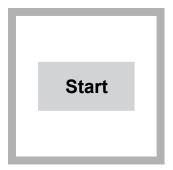
Description	Quantity
QAC Reagent 1 Powder Pillows	2
QAC Reagent 2 Powder Pillows	2
Bottle, square, with 25-mL mark	2
Clippers for plastic pillows	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument- specific information on page 1.)	2

Refer to Consumables and replacement items on page 5 for order information.

## Sample collection and storage

- Collect samples in glass bottles that have been rinsed several times with sample. Do not use plastic containers, which can adsorb quaternary ammonium compounds.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 28 days.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to ~7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

## **Test procedure**



1. Start program 401 QAC. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

**Note:** Although the program name can be different between instruments, the program number does not change.



- 2. Prepare the blank: Fill a marked mixing bottle to the 25-mL line with deionized water.
- **3. Prepare the sample:** Fill a second marked mixing bottle to the 25-mL line with sample.



**4.** Add the contents of one QAC Reagent 1 Powder Pillow to each bottle.



5. Swirl the bottles to dissolve the reagent. Do not shake! Shaking makes air bubbles that can cause incorrect results.



**6.** Add the contents of one QAC Reagent 2 Powder Pillow to each bottle.

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7. Swirl the bottles to dissolve the reagent. Do not shake.
A purple color forms if a quaternary ammonium compound is in the sample.



**8.** Start the instrument timer. A 2-minute reaction time starts.

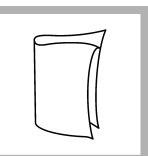


**9.** Pour 10 mL of the blank solution into a sample cell.

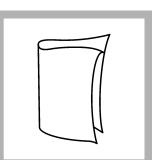


**13.** Pour 10 mL of the prepared sample into a sample cell.

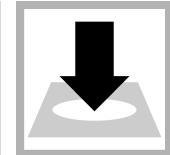
## Interferences



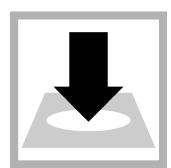
**10.** When the timer expires, clean the blank sample cell.



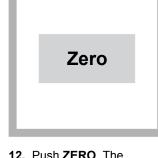
**14.** Clean the prepared sample cell.



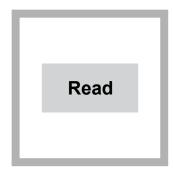
**11.** Insert the blank into the cell holder.



**15.** Insert the prepared sample into the cell holder.



**12.** Push **ZERO**. The display shows 0.0 mg/L CTAB.



**16.** Push **READ**. Results show in mg/L CTAB.

Interference studies were done with a CTAB standard solution of approximately 3 mg/L that contained the potential interfering substance. The substance was said to interfere when the resulting concentration changed by 10% or more. Table 2 shows interfering substances and levels. Table 3 shows substances that do not interfere up to the tested concentrations.

#### Table 2 Interfering substances

Interfering substance	Interference level
Calcium (as CaCO <sub>3</sub> )	Positive interference above 1350 mg/L
Chlorine, HOCI and CIO <sup>-</sup>	Positive interference above 7 mg/L
Cyanuric acid	Negative interference above 70 mg/L
Igepal <sup>™</sup> nonionic surfactant	Positive interference above 3 mg/L
lodine, I <sub>3</sub> -	Positive interference above 3 mg/L
Iron, Fe <sup>3+</sup>	Positive interference above 80 mg/L
Liquimine <sup>™</sup> 14-P, filming amine	Positive interference above 1825 mg/L
Magnesium, Mg <sup>2+</sup> (as CaCO <sub>3</sub> )	Positive interference above 1350 mg/L
Niaproof <sup>™</sup> anionic surfactant	Negative interference above 11 mg/L
Polyacrylic acid	Negative interference above 16 mg/L
Sodium lauryl sulfate	Negative interference above 8 mg/L
Sodium polyphosphate	Positive interference above 1325 mg/L
Tribenzylamine	Positive interference above 7 mg/L
Triton X-100 <sup>™</sup> nonionic surfactant	Positive interference above 4 mg/L

Interfering substance	Interference level		
Urea	Positive interference above 8 mg/L		
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary. Adjust the sample pH to between 3 and 5 with an acid or base such as 1.0 N sulfuric acid standard solution or 1.0 N sodium hydroxide standard solution. Correct the test result for the dilution caused by the volume additions.		

#### Table 2 Interfering substances (continued)

#### Table 3 Non-interfering substances

Non-interfering Substance	Highest Concentration Tested (mg/L)	
Silica, SiO <sub>2</sub>	400	
Potassium alum, AIKS <sub>2</sub> O <sub>8</sub>	500	
Sodium thiosulfate, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	30	

## Accuracy check

#### Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample. Items to collect:

- 100-mg/L CTAB Standard Solution
- Mixing bottles, 25-mL (3x)
- TenSette Pipet and pipet tips
- 1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
- 2. Go to the Standard Additions option in the instrument menu.
- 3. Select the values for standard concentration, sample volume and spike volumes.
- 4. Open the standard solution.
- Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 25-mL portions of fresh sample. Mix well.
- 6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
- 7. Select Graph to compare the expected results to the actual results.

**Note:** If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.

#### Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- 100-mg/L CTAB Standard Solution
- 100-mL volumetric flask, Class A
- 5.0-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

- 1. Prepare a 5.0-mg/L CTAB standard solution as follows:
  - **a.** Use a pipet to add 5.00 mL of a 100-mg/L CTAB standard solution into the volumetric flask.
  - **b.** Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
- **2.** Use the test procedure to measure the concentration of the prepared standard solution.
- 3. Compare the expected result to the actual result.

**Note:** The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.

## Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
401	3.0 mg/L CTAB	2.7–3.3 mg/L CTAB	0.04 mg/L CTAB

## **Summary of Method**

The reagents buffer the sample to an acidic pH and include a masking agent to remove potential interferences. The indicator reacts with quaternary ammonium compounds in the sample and forms a pale pink to vivid purple color. This test is applicable to monitor quaternary ammonium compounds in swimming pools and cooling towers. The measurement wavelength is 575 nm.

## **Consumables and replacement items**

#### **Required reagents**

Description	Quantity/Test	Unit	Item no.
Quaternary Ammonium Compounds Reagent Set (100 tests), includes:	_	_	2459200
(4) QAC Reagent 1 Powder Pillows	2 pillows	50/pkg	2401066
(8) QAC Reagent 2 Powder Pillows	2 pillows	25/pkg	2401268
Water, deionized	varies	4 L	27256

#### **Required apparatus**

Description	Quantity/test	Unit	ltem no.
Bottle, square, with 25-mL mark	1	each	1704200
Clippers for plastic pillows	1	each	96800

#### **Recommended standards**

Description	Unit	ltem no.
QAC Standard Solution, 100-mg/L as CTAB	100 mL	2415342

#### **Optional reagents and apparatus**

Description	Unit	Item no.
Alconox <sup>™</sup> detergent	1.8 kg	2088000
Brush, test tube	each	69000
Flask, volumetric, Class A, 100-mL	each	1457442
Pipet, TenSette <sup>®</sup> , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette <sup>®</sup> Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet, volumetric, Class A, 5.00-mL	each	1451537
Pipet filler, safety bulb	each	1465100
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
Sodium Hydroxide Solution, 5 N	50 mL	245026
Sulfuric Acid, ACS	500 mL	97949
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032

