Quaternary Ammonium Compounds

DOC316.53.01128

Direct Binary Complex Method

Method 8337

0.2 to 5.0 mg/L as CTAB

Powder Pillows

Scope and Application: For cooling tower water and pool/spa water



Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 372 Instrument-specific information

Instrument	Sample cell	Cell orientation
DR 6000	2495402	Fill line faces right
DR 5000	2495402	Fill line faces user
DR 3900	2495402	Fill line faces user
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right

Collect the following items:

Description	Quantity
QAC Reagent 1 Powder Pillows	2 pillows
QAC Reagent 2 Powder Pillows	2 pillows
Bottle, square, with 25 mL mark	2
Clippers, for opening powder pillows	1
Sample Cells, 1-inch, 10 mL	2

See Consumables and replacement items for reorder information.

Direct Binary Complex Method



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).



2. Blank Preparation: Fill one 25-mL mixing bottle with 25 mL of deionized water.



3. Prepared Sample: Fill another mixing bottle with 25 mL of 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 creates air bubbles that interfere with test results.



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



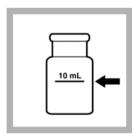
7. Swirl the bottles to dissolve the reagent. **Do not shake.**

A purple color will form if a quaternary ammonium compound is present



8. Start the instrument timer.

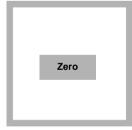
A two-minute reaction period will begin.



9. Pour at least 10 mL of the solutions from the bottles into the sample cells.



10. When the timer expires, insert the blank into the cell holder.



11. ZERO the instrument. The display will show: 0.0 mg/L CTAB



12. Insert the prepared sample into the cell holder.

READ the results in mg/L CTAB (cetyl-trimethylammonium bromide).

Interferences

Interference studies were conducted by preparing a CTAB standard solution of approximately 3 mg/L as well as a solution of the potential interference. The constituent was said to interfere when the resulting concentration changed by 10%. The *Interfering substances* table shows interfering substances and levels. The *Noninterfering substances* table shows substance that do not interfere up to the tested concentrations.

After several samples have been analyzed, the sample cells may exhibit a build-up of a pink or purple color. A rinse with 1.0 N Sodium Hydroxide Solution followed by an Alconox™ detergent wash and deionized water rinse will eliminate the build-up when it occurs.

Table 373 Interfering substances

Interfering substance	Interference level
Calcium (as CaCO ₃)	Positive interference above 1350 mg/L
Chlorine, HOCl and OCl-	Positive interference above 7 mg/L
Cyanuric acid	Negative interference above 70 mg/L
Igepal™ nonionic surfactant	Positive interference above 3 mg/L
Iodine, I ₃ ⁻	Positive interference above 3 mg/L
Iron, Fe ³⁺	Positive interference above 80 mg/L
Liquimine™ 14–P, filming amine	Positive interference above 1825 mg/L
Magnesium, Mg ²⁺	Positive interference above 1350 mg/L
Niaproof™ 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™ nonionic surfactant	Positive interference above 4 mg/L
Urea	Positive interference above 8 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment. Adjust the sample pH between 3 and 5 by using a pH meter or pH paper and adding dropwise an appropriate amount of acid or base such as 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standard Solution. If significant volumes of acid or base are used, a volume correction should be made.

Table 374 Noninterfering substances

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

Sample collection, preservation and storage

- Collect samples in glass bottles that have been rinsed several times with sample before final sample filling.
- · Do not use plastic containers; plastic adsorbs QACs.
- Acidify the sample to a pH of less than 2.
- Store at 4 ± 2 °C.

Accuracy check

Standard additions method (sample spike)

Required for accuracy check:

- QAC Standard Solution, 100-mg/L CTAB
- TenSette Pipet and tips
- Mixing bottles (3)
- 1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
- Select standard additions from the instrument menu: OPTIONS>MORE>STANDARD ADDITIONS.
- Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
- 4. Open the standard solution.
- Use the TenSette Pipet to prepare spiked samples: add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 25-mL portions of fresh sample.
- Follow the Direct Binary Complex Method test procedure for each of the spiked samples, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.
- Select GRAPH to view the results. Select IDEAL LINE (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard solution method

Note: Refer to the instrument user manual for specific software navigation instructions.

Required for accuracy check:

- QAC Standard, 100-mg/L as CTAB
- Deionized water
- 100-mL Class A volumetric flask
- Class A 5 mL volumetric pipet and pipet bulb
- 1. Prepare a 5.0 mg/L CTAB standard solution as follows:
 - a. Pipet 5.0 mL of QAC Standard, 100-mg/L as CTAB, into a 100-mL volumetric flask.
 - b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
- Use this solution in place of the sample. Follow the *Direct Binary Complex Method* test procedure.
- To adjust the calibration curve using the reading obtained with the standard solution, navigate to Standard Adjust in the software: OPTIONS>MORE>STANDARD ADJUST

4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

F	Program	Instrument	Standard	Precision—95% Confidence Limits of Distribution	Sensitivity—∆Concentration per 0.010 ∆Abs
	401	DR 5000	3.0 mg/L CTAB	2.7-3.3 mg/L CTAB	0.04 mg/L CTAB

Summary of method

The test method makes use of a colorimetric chemistry in which a quaternary ammonium compound reacts with an indicator to produce a color change from pale pink to vivid purple. The test is conducted in a stabilized, acid-buffered solution containing a masking agent to eliminate potential interferences. This test is applicable to the monitoring of QACs in swimming pools and cooling towers. Test results are measured at 575 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
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

Required apparatus (powder pillows)

Description	Quantity	Unit	Catalog number
Bottle, square, with 25 mL mark	2	each	1704200
Clippers, for opening powder pillows	1	each	96800
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402

Recommended standards

Description	Unit	Catalog number
QAC Standard Solution, 100-mg/L as CTAB	100 mL	2415342
Water, deionized	4 liters	27256

Optional reagents and apparatus

Description	Unit	Catalog number
Alconox [™] detergent	1.8 kg	2088000
Sodium Hydroxide Standard Solution, 1.0 N	100 mL	104532
Sodium Hydroxide Solution, 1.0 N	1000 mL	104553
Sulfuric Acid Standard Solution, 1.0 N	100 mL	127032
Thermometer, Non-Mercury, -10 to 225 °C	each	2635700
Pipet, TenSette®, Pipet, 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001 ¹	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001 ¹	1000/pkg	2185628
Flask, Class A volumetric, 100 mL	each	1457442
Pipet, Class A volumetric, 5 mL	each	1451537

¹ Other sizes are available

