



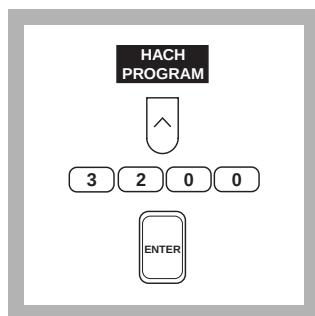
Method 8337

Direct Binary Complex Method

Powder Pillows

(0 to 5.00 mg/L as CTAB)

Scope and Application: For cooling tower and pool/spa water. The estimated detection limit for program number 3200 is 0.11 mg/L CTAB.



1. Press the soft key under **HACH PROGRAM**.

Select the stored program for quaternary ammonium compounds (QAC) by pressing **3200** with the numeric keys.

Press: **ENTER**

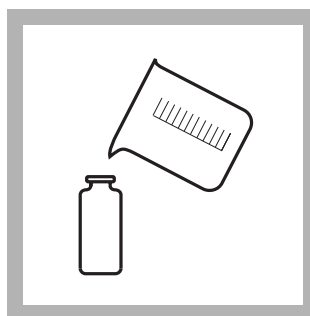
Note: If samples cannot be analyzed immediately, see *Sample Collection, Storage and Preservation* following these steps.

Note: The Flow Cell and Sipper Modules cannot be used with this procedure.

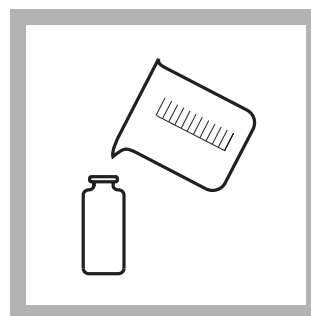


2. The display will show:
HACH PROGRAM: 3200 QAC

The wavelength (λ), **575 nm**, is automatically selected.



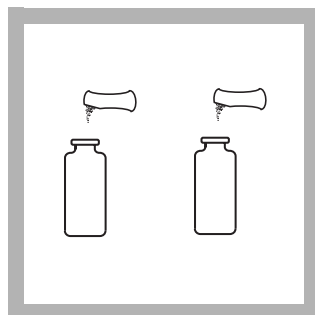
3. Fill a sample cell (the blank) with 25 mL of deionized water.



4. Fill another cell (the prepared sample) with 25 mL of sample.

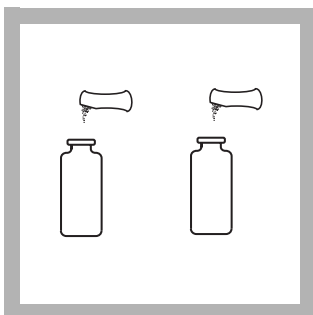
Note: For proof of accuracy, a 5.0-mg/L CTAB standard solution (see *Accuracy Check*) can be used in place of the sample.

QUATERNARY AMMONIUM COMPOUNDS, continued



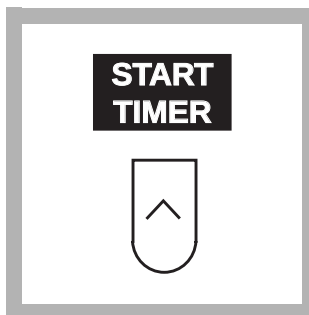
5. Add the contents of one Q.A.C. Reagent 1 Powder Pillow to each sample cell. Swirl (do not shake) the sample cells to dissolve the reagents.

Note: Shaking the sample cell causes air bubble turbidity that dissipates slowly, interfering with the test results.

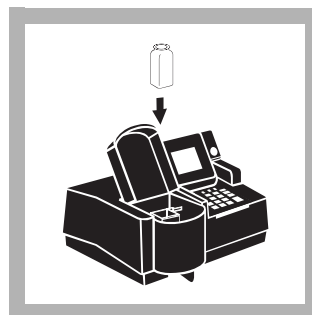


6. Add the contents of one Q.A.C. Reagent 2 Powder Pillow to each sample cell. Swirl (do not shake) the sample cells to dissolve the reagents.

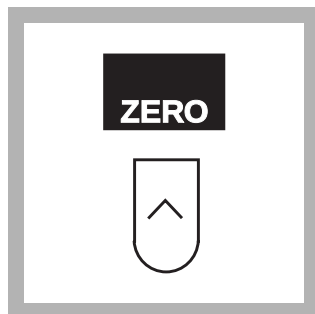
Note: A purple color will form if a quaternary ammonium compound is present.



7. Press the soft key under **START TIMER**. A 2-minute reaction period will begin.



8. When the timer beeps, place the blank into the cell holder. Close the light shield.



9. Press the soft key under **ZERO**.
The display will show:
0.00 mg/L CTAB



10. Place the prepared sample into the cell holder. Close the light shield. Results in mg/L (or chosen units) quaternary ammonium compounds as cetyl-trimethylammonium bromide will be displayed.

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%.

Table 1 Interfering Substances and Suggested Treatments

Interfering Substance	Interference Levels and Treatments
Calcium (as CaCO ₃)	Positive interference above 1,350 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 1,825 mg/L
Magnesium, Mg ²⁺	Positive interference above 1,350 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 1,325 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 ampules or 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. See Section 1.2.2 <i>Correcting for Volume Additions</i> .

No Interferences	Highest Concentration Tested (mg/L)
Silica, SiO ₂	400
Potassium alum, AlKSO ₈	500
Sodium thiosulfate, Na ₂ S ₂ O ₃	30

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 a Alconox™ detergent wash and deionized water rinse will eliminate the build-up when it occurs.

Sample Collection, Storage and Preservation

Collect samples in glass bottles that have been rinsed several times with sample before final sample filling. Do not use plastic containers as plastic adsorbs QACs.

Accuracy Check

Standard Additions Method

- a. Leave the unspiked sample in the sample compartment. Verify that the units displayed are in mg/L. Select standard additions mode by pressing the soft keys under **OPTIONS, (MORE)** and then **STD ADD**.
- b. Press **ENTER** to accept the default sample volume (mL), 25.0.
- c. Press **ENTER** to accept the default standard concentration (mg/L), 100.0.
- d. Press the soft key under **ENTRY DONE**.
- e. Open a Q.A.C. Standard Solution, 100-mg/L CTAB.
- f. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively to three 25-mL samples and mix each thoroughly.
- g. Analyze each standard addition sample as described above. Accept the standard additions reading by pressing the soft key under **READ** each time. Each addition should reflect approximately 100% recovery.
- h. After completing the sequence, the display will show the extrapolated concentration value and the “best-fit” line through the standard additions data points, accounting for matrix interferences.
- i. See Section 1.4.1 *Standard Additions* for more information.

Standard Solution Method

Prepare a 5.0-mg/L CTAB standard solution by pipetting 5.0 mL of Q.A.C Standard, 100-mg/L as CTAB, into a 100-mL volumetric flask. Dilute to the mark with deionized water. Mix well. Prepare this solution daily. Perform the quaternary ammonium compound procedure as described above.

Method Performance

Precision

Standard: 5.00 mg/L CTAB

Program	95% Confidence Limits
3200	4.92–5.08 mg/L CTAB

For more information on determining precision data and method detection limits, refer to Section 1.5.

Estimated Detection Limit

Program	EDL
3200	0.11 mg/L CTAB

For more information on derivation and use of Hach’s estimated detection limit, see Section 1.5.2. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, appendix B, see Section 1.5.1.

Sensitivity

Program Number: 3200

Portion of Curve	Δ Abs	Δ Concentration
Entire Range	0.010	0.046 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

Calibration Standard Preparation

To perform a quaternary ammonium compounds calibration using the Direct Binary Complex method, prepare calibration standards containing 1.00, 3.00, and 5.00 mg/L CTAB as follows:

- Into three different 100-mL Class A volumetric flasks, pipet 1.00, 3.00, and 5.00 mL of a 100-mg/L Q.A.C. Standard Solution (Cat. No 24153-42) using Class A glassware.
- Dilute to the mark with deionized water. Mix carefully to avoid foaming.
- Using the Direct Binary Complex method and the calibration procedure described in the *User-Entered Programs* section of the *DR/4000 Spectrophotometer Instrument Manual*, generate a calibration curve from the standards prepared above.

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.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheet* for information specific to the reagents used. For additional information, refer to *Section 1*.

Pollution Prevention and Waste Management

For information on pollution prevention and waste management, refer to *Section 1*.

QUATERNARY AMMONIUM COMPOUNDS, continued

REQUIRED REAGENTS AND STANDARDS

Quaternary Ammonium Compounds Reagent Set (100 tests)24592-00
Includes: (4) 24010-66, (8) 24012-68

Description	Quantity Required		Unit	Cat. No.
	per test			
Q.A.C. Reagent 1 Powder Pillows	2 pillows	50/pkg		24010-66
Q.A.C. Reagent 2 Powder Pillows	2 pillows	25/pkg		24012-68

REQUIRED EQUIPMENT AND SUPPLIES

Cylinder, graduated, 25-mL	1	each.....	508-40
Clippers, for opening powder pillows	1	each.....	968-00
DR/4000 1-Inch Cell Adapter	1	each.....	48190-00

OPTIONAL REAGENTS AND STANDARDS

Q.A.C. Standard Solution, 100-mg/L as CTAB	100 mL.....	24153-42
Sodium Hydroxide Standard Solution, 1.00 N.....	900 mL.....	1045-53
Sulfuric Acid Standard Solution, 1.0 N.....	50 mL SCDB.....	1270-26
Water, deionized.....	4 liters.....	272-56

OPTIONAL EQUIPMENT AND SUPPLIES

DR/4000 Carousel Module Kit	each.....	48070-02
Filter Paper, folded, 12.5-cm.....	100/pkg.....	1894-57
Flask, volumetric, Class A, 100-mL	each.....	14574-42
Funnel, poly, 65-mm	each.....	1083-67
Pipet Filler.....	each.....	12189-00
Pipet, volumetric, Class A, 1.00-mL	each.....	14515-35
Pipet, volumetric, Class A, 3.00-mL	each.....	14515-03
Pipet, volumetric, Class A, 5.00-mL	each.....	14515-37
Pipet, TenSette 0.1 to 1.0 mL	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg.....	21856-96



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932