



## Method 8015

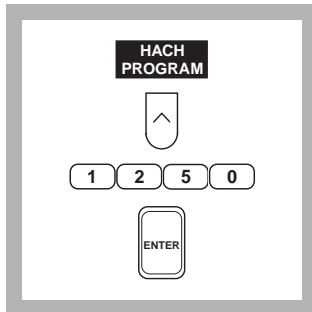
## Carminie Method\*

### Powder Pillows

(0 to 14.0 mg/L)

**Scope and Application:** For water and wastewater. The estimated detection limit for program number 1250 is 0.4 mg/L B.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.



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

Select the stored program number for Boron (B) by pressing **1250** with the numeric keys.

Press: **ENTER**

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

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



**2.** The display will show: **HACH PROGRAM: 1250 Boron**

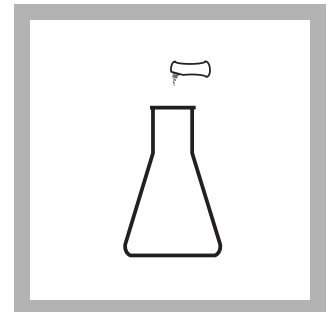
The wavelength ( $\lambda$ ), **605 nm**, is automatically selected.



**3.** Measure 75 mL of concentrated sulfuric acid, ACS, using a 100-mL graduated cylinder, into a 250-mL plastic erlenmeyer flask.

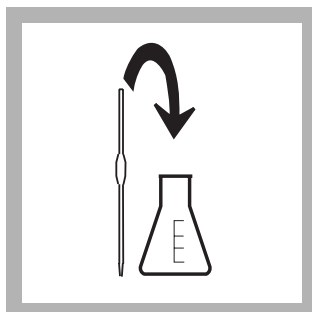
**Note:** All labware must be completely dry. Excess water will cause low results.

**Note:** For proof of accuracy, use a 4.0 mg/L boron standard solution in place of the sample (see *Accuracy Check*).

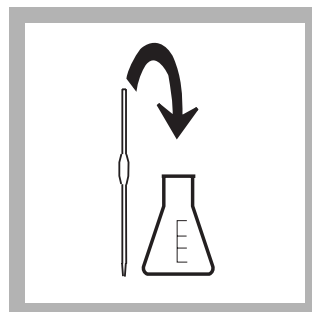


**4.** Add the contents of one BoroVer 3 Reagent Powder Pillow to the flask. Swirl to mix. Allow 5 minutes for the powder to dissolve completely.

**Note:** Use with adequate ventilation; see *Reagent Preparation* below.



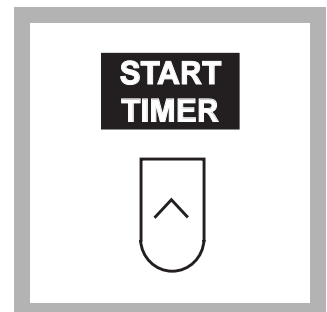
**5.** Accurately pipet 2.0 mL of deionized water into a 125-mL plastic erlenmeyer flask (the blank).



**6.** Accurately pipet 2.0 mL of sample into another 125-mL plastic erlenmeyer flask (the prepared sample).



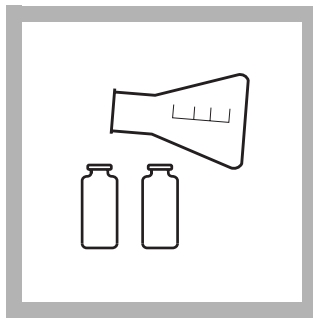
**7.** Add 35 mL of the BoroVer 3/Sulfuric Acid Reagent Solution to each erlenmeyer flask using a 50-mL graduated cylinder. Swirl to mix completely.



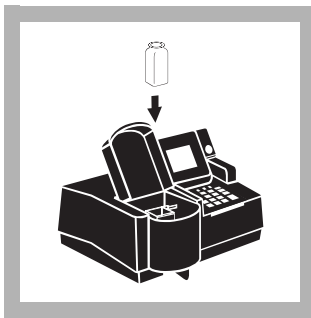
**8.** Press the soft key under **START TIMER**. A 25-minute reaction period will begin.

### Warning!

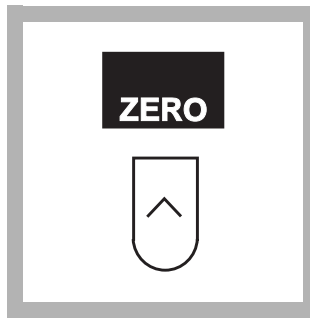
Do not use a stoppered or capped vessel to complete Steps 6 and 7.



**9.** When the timer beeps, pour at least 10 mL from each flask into separate 1-inch sample cells.



**10.** Place the blank into the cell holder. Close the light shield.

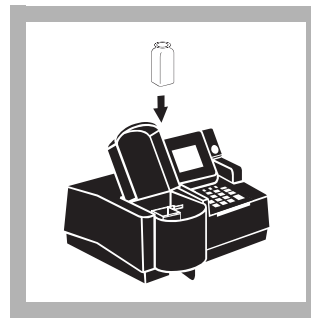


**11.** Press the soft key under **ZERO**.

The display will show:

**0.00 mg/L B**

*Note: For alternate concentration units, press the soft key under **OPTIONS**. Then press the soft key under **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.*



**12.** Place the prepared sample into the cell holder. Close the light shield. Results in mg/L boron (or chosen units) will be displayed.

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## Sample Collection, Preservation and Storage

Collect samples in clean polyethylene or polypropylene bottles.

## Reagent Preparation

To prepare additional BoroVer 3/Sulfuric Acid Solution, mix one BoroVer 3 Reagent Powder Pillow per 75 mL of concentrated sulfuric acid, adding the powder pillows individually while stirring. Preparation of this solution generates gaseous HCl when the indicator pillow is added to the sulfuric acid. Use of a fume hood or other well-ventilated lab area is strongly advised. This solution is stable up to 48 hours if stored in plastic containers. Prepare the reagent solution in a polypropylene flask.

## Accuracy Check

### Standard Additions Method

- 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**.
- Press **ENTER** to accept the default sample volume (mL), 25.
- Press **ENTER** to accept the default standard concentration (mg/L), 250.
- Press the soft key under **ENTRY DONE**.
- Snap the neck off a Boron Voluette Ampule Standard, 250-mg/L B.
- 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**

Check the accuracy of the test using Boron Standard Solution, 4 mg/L as B, listed under *OPTIONAL REAGENTS AND STANDARDS*.

Or, prepare this solution by pipetting 4.00 mL of the Boron Voluette Ampule Standard, 250-mg/L B, into a 250-mL volumetric flask. Dilute to volume with deionized water. Swirl to mix.

To adjust the calibration curve using the reading obtained with the 4.0-mg/L standard solution, press the soft keys under **OPTIONS, MORE** then **STD: OFF**. Press **ENTER** to accept the displayed concentration, the value of which depends on the selected units. If an alternate concentration is used, enter the actual concentration and press **ENTER** to return to the read screen. See Section 1.5.5 *Adjusting the Standard Curve* for more information.

**Method Performance**

**Precision**

Standard: 4.0 mg/L B

Program	95% Confidence Limits
1250	3.9–4.1 mg/L B

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

**Estimated Detection Limit**

Program	EDL
1250	0.4 mg/L B

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: 1250

Portion of Curve	ΔAbs	ΔConcentration
0.010 Abs	0.010	0.15 mg/L
7 mg/L	0.010	0.16 mg/L
12.6 mg/L	0.010	0.17 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

### Calibration Standard Preparation

To perform a boron calibration using the carmine method, prepare a 100.0-mg/L boron stock solution by pipetting 10.00 mL of 1000-mg/L Boron Standard Solution (Cat. No. 1914-42) into a 100-mL volumetric flask using Class A glassware. Dilute to the mark with deionized water and mix thoroughly. Prepare calibration standards containing 2.0, 4.0, 6.0, 8.0, 10.0, 12.0, and 14.0 mg/L B as follows:

- a. Into seven different 100-mL Class A volumetric flasks, pipet 2.00, 4.00, 6.00, 8.00, 10.00, 12.00 and 14.00 mL of the 100.0-mg/L B stock solution using Class A glassware.
- b. Dilute to the mark with deionized water and mix thoroughly.
- c. Using the carmine method and the calibration procedure described in the *User-Entered Programs* section in the *DR/4000 Spectrophotometer Instrument Manual*, generate a calibration curve from the standards prepared above.

### Summary of Method

Boron is determined by its reaction with carminic acid in the presence of sulfuric acid to produce a reddish to bluish color. The amount of color is directly proportional to the boron concentration.

### 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

The BoroVer 3/Sulfuric Acid Solution is highly acidic. Neutralize to pH 6–9 and flush down the drain for disposal. For more information on waste management, see *Section 1*.

**REQUIRED REAGENTS AND STANDARDS**

Description	Quantity Required Per Test	Unit Cat. No.
BoroVer 3 Boron Reagent Powder Pillows .....	1 pillow ..... 100/pkg.....	14170-99
Sulfuric Acid, ACS, concentrated .....	75 mL ..... 2.5 liters.....	979-09
Water, deionized .....	2.0 mL..... 4 liters.....	272-56

**REQUIRED EQUIPMENT AND SUPPLIES**

Cylinder, graduated, 50-mL .....	1 ..... each.....	508-41
Cylinder, graduated, 100-mL, plastic .....	1 ..... each.....	1081-42
DR/4000 1-Inch Cell Adapter .....	1 ..... each.....	48190-00
Flask, Erlenmeyer, 125-mL, plastic .....	2 ..... each.....	1082-43
Flask, Erlenmeyer, 250-mL, plastic .....	1 ..... each.....	1082-46
Pipet, volumetric, 2.0-mL .....	2 ..... each.....	14515-36

**OPTIONAL REAGENTS AND STANDARDS**

Boron Standard Solution, 4-mg/L B .....	500 mL.....	1963-49
Boron Standard Solution, 10-mL Voluette Ampule, 250-mg/L B .....	16/pkg.....	14249-10
Boron Standard Solution, 1000-mg/L B .....	100 mL.....	1914-42

**OPTIONAL EQUIPMENT AND SUPPLIES**

Cylinder, graduated, 500-mL .....	each.....	20885-49
DR/4000 Carousel Module Kit .....	each.....	48090-02
Flask, erlenmeyer, 1000-mL.....	each.....	505-53
Pipet, TenSette, 0.1 to 1.0 mL .....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet, volumetric, 4.00-mL .....	each.....	14515-04
Pipet Filler, safety bulb.....	each.....	14651-00
Sample cell, with 25-mL mark, 1-inch.....	2/pkg.....	13537-02



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