Boron

DOC316.53.01309

Carmine Method

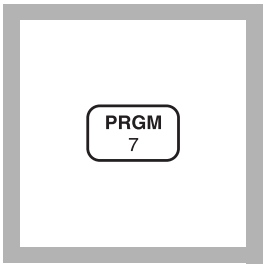
Method 10252

0 to 50 mg/L B

Powder Pillows

Scope and Application: For oil and gas field waters

Test procedure

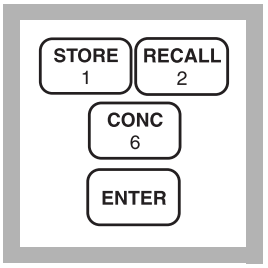


1. Push: **PRGM**

The display shows:

PRGM ?

Initial setup: go to “Instrument Setup” on page 3 to add the program to the instrument.



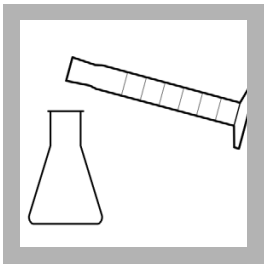
2. Push: 126 ENTER

The display shows **mg/L, B HR** and the **ZERO** icon.

To do an accuracy proof, use a 30 mg/L boron standard as an alternative to the sample.

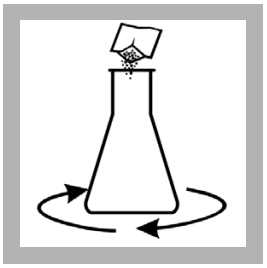
3. Put the COD/TNT vial adapter in the cell holder. Rotate the adapter until it goes in. Then push the adapter down until click is heard.

Note: A diffuser band covers the light path holes on the adapter. For best performance, do not remove the diffuser band.



4. Use a 100-mL graduated cylinder to measure 75 mL of concentrated sulfuric acid. Add the acid to a 250 mL plastic Erlenmeyer flask.

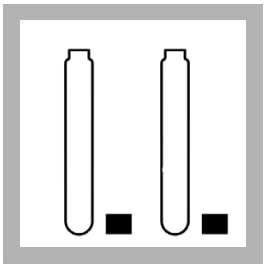
Notice! Prepare the solution in a well-ventilated area or use a fume hood to prevent injury.



5. Add the contents of one BoroVer 3 Reagent Powder Pillow to the flask. Swirl immediately to mix. Let the powder dissolve fully (no more than five minutes).

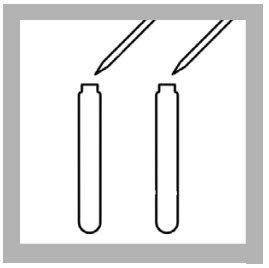
After the powder has dissolved fully, put a cap on the flask.

The solution can be used for 48 hours when it is stored at 20º C or below.



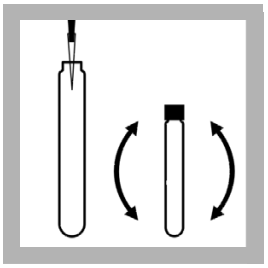
6. Remove the caps from two clean 16-mm screw-capped vials.

Refer to “How to use and clean glass vials” on page 2.



7. Add 0.2 mL of sample to one vial (the sample) and 0.2 mL of deionized water to the other vial (the blank).

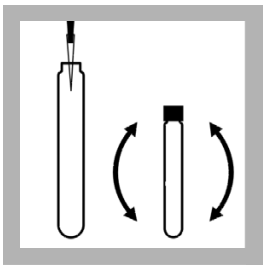
Note: As an alternative, use 0.4 mL of sample or DI water. Use 7.0 mL of BoroVer 3 in step 8.



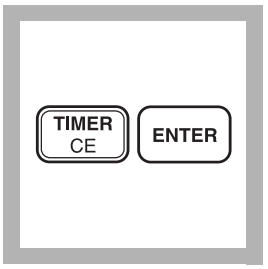
8. Add 3.5 mL of the BoroVer 3 solution prepared in step 5 to the sample vial. Tighten the cap and immediately invert to mix.

The solution in the vials will become warm.

Note: As an alternative, use 7.0 mL of BoroVer 3 solution with 0.4 mL of sample if a 3.5- mL pipet is not available.



9. Add 3.5 mL of the BoroVer 3 solution prepared in step 5 to the blank vial. Tighten the cap and invert to mix.

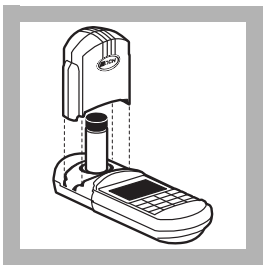


10. Push: **Timer Enter**

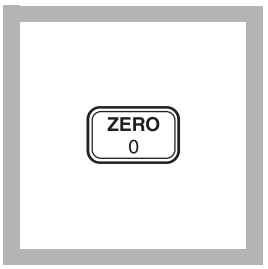
A 30-minute reaction period starts.

11. Clean the outer surface of the vial with a damp cloth. Dry the vial with a dry cloth to remove fingerprints and other marks.

12. After the timer beeps, put the **blank** into the adapter. Push straight down on the top of the vial until it is fully installed. The vial must not move, because this can cause reading errors.



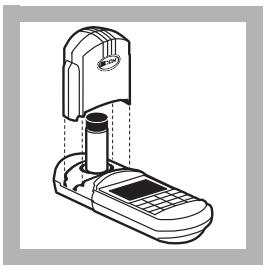
13. Put the instrument cap over the vial.



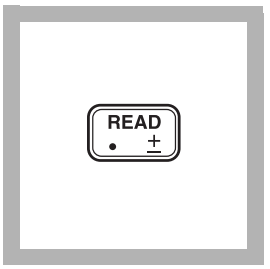
14. Push: **ZERO**

The cursor moves to the right, then the display shows:

0.00 mg/L B HR



15. Put the prepared **sample** in the adapter. Put the instrument cap over the vial. Push straight down on the top of the vial until it is fully installed. The vial must not move, because this can cause reading errors.



16. Push: **READ**

The cursor moves to the right, then the result in **mg/L B HR** is shown.

Note: For best results, use the Standard Adjust option. Refer to the procedures manual for more information about Standard Adjust.

Sample Collection, Preservation and Storage

Collect the samples in clean polyethylene bottles.

Reagent preparation

Mix one BoroVer 3 Reagent Powder Pillow per 75 mL of concentrated sulfuric acid. Add the powder pillow while stirring. The preparation of this solution generates gaseous HCl when the indicator pillow is added to the sulfuric acid. Prepare the solution in a well-ventilated area or use a fume hood to prevent injury. This solution is stable for up to 48 hours if stored in plastic containers. Do not store in borosilicate glassware (Pyrex® or Kimax®) for more than one hour. The solution may pull boron from these containers.

How to use and clean glass vials

The glass vials and caps can be used again. Discard the reacted BoroVer 3 Reagents correctly. Refer to the current material safety data sheets (MSDS) for safety protocols and disposal information. Flush the vials many times with deionized water and let them dry before use.

Note: *Let the empty vials drain fully before flushing with water.*

New vials can contain residual amounts of reactive boron from the glass manufacturing process. For best results, fill the vials with 3–4 mL of the prepared BoroVer 3 Reagent for 30 minutes. Discard the reacted solution. Flush and dry the vials for use in other analysis.

The BoroVer 3/Sulfuric Acid solution is very acidic. Make sure that the solution is neutral (pH 6–9) before disposal. Refer to the current material safety data sheets (MSDS) for safety protocols and disposal information.

Accuracy Check

Standard Solution Method

Use a 30 mg/L Boron Standard Solution to validate the test procedure, reagents and the instrument.

1. To prepare the standard solution, add 3.0 mL of a 1000 mg/L Boron Standard Solution to a 100-mL volumetric flask. Add deionized water to the mark, stopper and mix fully.
2. Use the 30 mg/L Boron Standard Solution as an alternative to the sample in step 7 of the procedure.

Standard Adjust

Use the reading with the 30 mg/L Boron Standard Solution to adjust the calibration curve.

1. Push the **SETUP** key and use the arrow keys to scroll to the STD option.
2. Push **ENTER**.
3. Push the numbers **30** to measure the value of the standard solution concentration.
4. Push **ENTER** to complete the adjustment. Refer to Section 1 *Standard Adjust* of the procedures manual for more information.

Method Performance

Precision

In a single laboratory, with a standard solution of 25 mg/L boron and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.8 mg/L boron.

Estimated Detection Limit

The estimated detection limit for program 126 is 2.2 mg/L B. For more information on derivation and use of the estimated detection limit, refer to Section 1 of the procedures manual.

Summary of Method

Boron reacts with a mixture of carminic acid and sulfuric acid and then shows a reddish to bluish color. The amount of color is directly proportional to the boron concentration.

Instrument Setup

This procedure adds the program 126 to a DR/850 or DR/890 instrument.

1. Push **ON** to turn on the instrument.
2. Push **SETUP**.
3. Push the down arrow until the prompt line shows USER and push **ENTER**.
4. Push the numbers **8138**, then push **ENTER**.
5. Refer to *Table 1*. Find the number from the Enter column that corresponds to Line Number 1 on the display. Push these numbers on the keypad, then push **ENTER**. Continue to add the numbers that correspond to each line number on the display.

Note: *Use the arrow keys to scroll and review or change numbers at any time.*

Table 1 Instrument setup

Line Number	Enter	Line Number	Enter
1	126	29	0
2	42	30	0
3	72	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	64	36	0
9	117	37	0
10	40	38	0
11	140	39	0
12	65	40	0
13	211	41	0
14	79	42	0
15	222	43	0
16	0	44	55
17	0	45	128
18	0	46	0
19	0	47	20
20	66	48	7
21	32	49	8
22	72	50	0
23	82	51	0
24	0	52	0
25	0	53	0
26	0	54	143
27	0	55	0
28	0	56	255

REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Item No.
BoroVer 3 Boron Reagent Powder Pillow	1pp/10 tests	100/pkg	1417099
Sulfuric Acid, ACS, concentrate	75mL/20 tests	500 mL	97949
Water, deionized	4.0 mL/20 tests	100 mL	27242
Vials, glass, 16 mm x 100 mm	2	6/pk	2275806
Caps, white,Teflon lining, for 16 mm vials	2	6/pk	2350206
Flask, polymethylpentene, screw cap, 250 mL	1	1	2089846
Cylinder, graduated, polypropylene, 100 mL	1	1	108142
Pipet, 0.2–1.0 mL	1	1	BBP078
Pipet Tip for BBP078	2	100/pk	BBP079
Pipet, 1.0–5.0 mL	1	1	BBP065
Pipet Tip for BBP065	1	75/pk	BBP068
OR			
Pipet, TenSette 0.1–1.0 mL	1	1	1970001
Pipet Tips, 0.1–1.0 mL for 1970001 Pipet	2	50/pk	2185696
Pipet, TenSette 1.0–10.0 mL	1	1	1970010
Pipet Tips, 1.0–10.0 mL for 1970010 Pipet	1	50/pk	2199796

OPTIONAL REAGENTS

Description	Unit	Item No.
Boron Standard Solution, 1000 mg/L as B	100 mL	191442
Sodium Hydroxide, 6 N	1000 mL	2332453

OPTIONAL APPARATUS

Description	Unit	Item No.
Pipet Tips, 0.1–1.0 mL for 1970001 TenSette Pipet	1000/pk	2185628
Pipet Tips, 1.0–10.0 mL for 1970010 TenSette Pipet	250/pk	2199725
Rack, Test Tube, for holding ten 16-mm vials	1	1864100
Pipets, includes one BBP078 and BBP065 pipet plus tips	1	LZP320
Goggles, safety, standard	1	2927902
Gloves, chemical-resistant, size 10	pair	2410105
Bottle, storage, write on, HDPE, 1000 mL	6/pk	2930004
Wipes, disposable 28 x 37 cm	188/pk	293280

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09/2011, Edition 1