ANALYTICAL PROCEDURES
For DR/2000 and DR/3000 Instruments

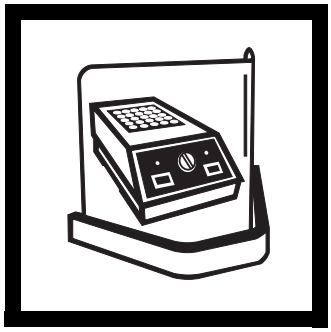
ORGANIC CARBON, TOTAL, Low Range

Method 10129

(0.0–20.0 mg/L C)

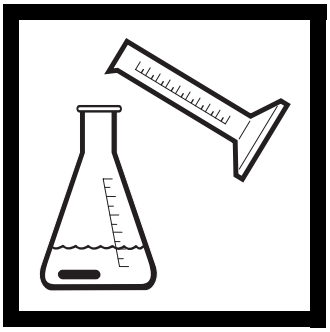
Direct Method*

Scope and Application: *For water, drinking water, and wastewater*



1. Turn on the COD reactor. Heat to 103-105 °C. Place the plastic shield in front of the reactor.

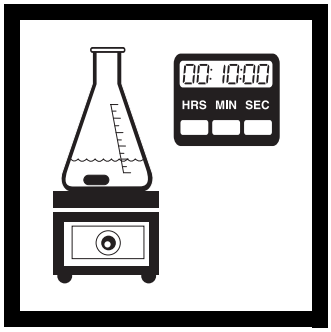
Note: *Ensure safety devices are in place to protect the analyst should leakage occur.*



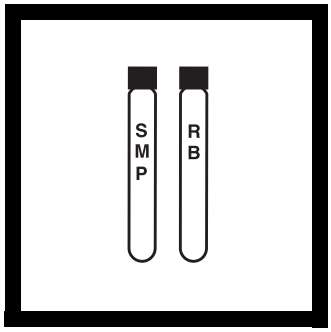
2. Use a graduated cylinder to add 10 mL of sample to a 50-mL Erlenmeyer flask containing a stir bar.

3. Add 0.4 mL of Buffer Solution, pH 2.0.

Note: *Use pH paper to make sure the sample pH is 2.*

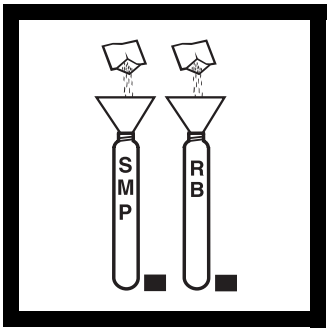


4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

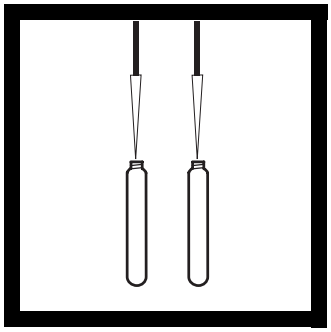


5. Label two Low Range Acid Digestion vials: **sample** and **reagent blank**.

Note: *A reagent blank is required for each series of samples.*



6. Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).

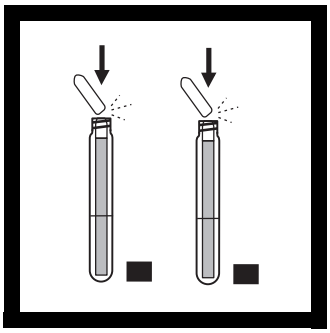


7. Use a TenSette® Pipet to add 3.0 mL of **organic-free water** to the **reagent blank** vial and 3.0 mL of **prepared sample** to the **sample** vial. Swirl to mix.

8. Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

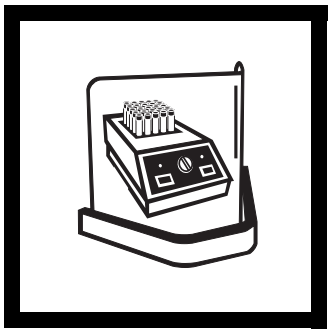
Note: Do not touch the ampules on the sides after wiping. Pick them up by the top.

* Patent pending

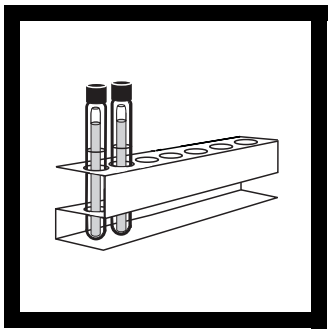


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Note: Do not invert or tilt the tube after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.

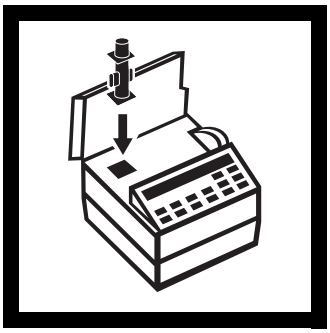


10. Cap the vial assemblies tightly and place them in the COD reactor for 2 hours at 103–105 °C.



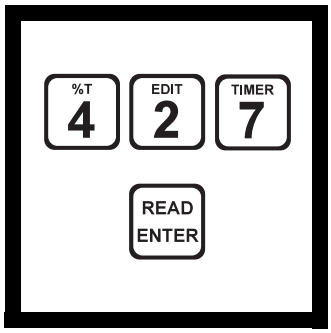
11. Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



12. Place the COD Vial Adapter into the cell holder with the marker to the right.

Note: *For DR/3000s, the groove in the adapter faces the front of the instrument.*



13. Enter the stored program for Low Range TOC.

Press: **427 READ/ENTER**

The display will show:

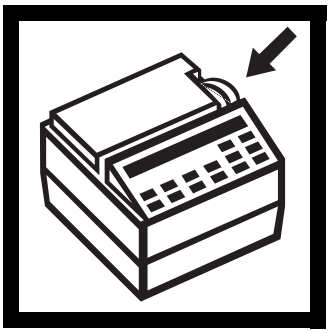
Dial nm to 600

Note: *See the Instrument Setup on page 7 to enter this method into the DR/2000.*

Note: *For DR/3000s:*
Press: **MANUAL PROGRAM**
Press: **-21.3 CONC FACTOR**
Press: **ZERO**
Press: **1 CONC**

Note: *DR/2000s with software versions 3.0 and greater will display* **P** *and the program number.*

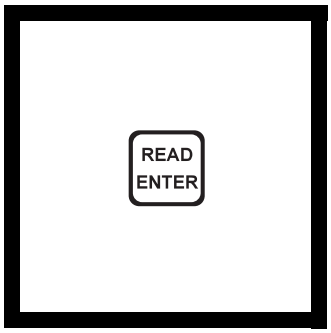
Note: *DR/2000s with software versions 3.0 and greater will not display* **Dial to** *if the wavelength is already set correctly. This display will show the message in step 15. Proceed to step 16.*



14. Rotate the wavelength dial until the display shows:

600 nm

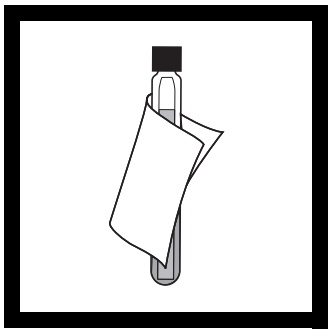
Note: *For DR/3000s, set the wavelength to 600 nm.*



15. Press: **READ/ENTER**

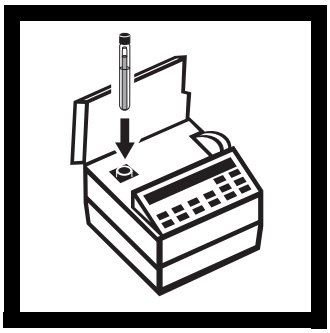
The display will show:

mg/l C

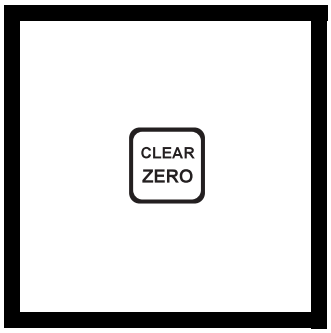


16. Wipe the reagent blank with a damp towel, followed by a dry one, to remove fingerprints or other marks.

Note: *The liquid in the reagent blank vial should be dark blue.*



17. Place the **reagent blank** vial assembly into the cell holder. Close the light shield.



18. Press: **ZERO**

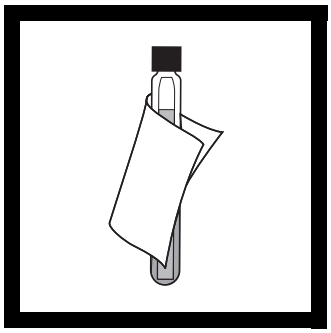
The display will show:

WAIT

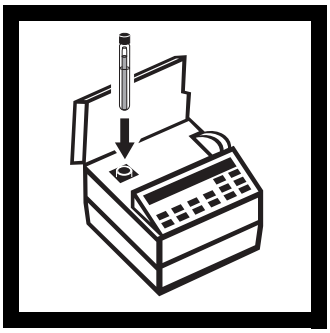
then:

0.0 mg/l C

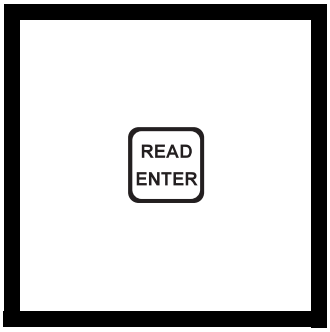
Note: *The DR/3000 will show* **0.0**.



19. Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.



20. Place the sample vial assembly into the cell holder. Close the light shield.



21. Press: **READ/ENTER**

the display will show:

WAIT

then the results in mg/L C will be displayed.

Note: *In the Constant-On mode, pressing* **READ/ENTER** *is not required.* **WAIT***will not appear. When the display stabilizes, read the result.*

Note: *For the DR/3000, insert the vial assembly and read the result.*

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle so there is no headspace. Cap. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Solutions Method

a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.

Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).

b. Prepare a 10.0 mg/L C standard by transferring 10.00 mL of the stock standard to a 1000-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh daily.

Standard Additions Method

a. Prepare a 150 mg/L C standard by transferring 15.00 mL of 1000 mg/L C stock solution to a 100-mL Class A volumetric flask. Dilute to volume with Organic-Free Water. Mix.

b. Use a TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 150 mg/L C standard to each of three Acid Digestion vials.

c. Add the contents of one TOC Persulfate Powder Pillow to each vial.

d. Add 3.0 mL of sample to each vial. Swirl to mix.

e. Proceed with the procedure starting at *step 8*.

f. The mg/L C concentration should increase by 5.0 mg/L for each 0.1 mL increment.

Method Performance

Precision
In a single laboratory, using a standard solution of 9.0 mg/L C and one lot of reagents, a single operator obtained a standard deviation of ±0.4 mg/L C.

Estimated Detection Limit
The estimated detection limit for Method 10129 is 0.3 mg/L C.

Sensitivity
At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 0.2 mg/L C.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Non-interfering Substances (Maximum Level Tested)

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	500 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN^-
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{3-}
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

If the sample contains greater than 600 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 50 NTU have been tested without interference.

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

Instrument Setup

DR/2000 with Software Version 3.0 and Above.

1. Turn the instrument on. Press **SHIFT METHOD** to enter configuration mode. The display will show: **MOMENTARY** or **CONSTANT ON**

2. Press the **UP ARROW** key twice to select **HACH UPDATE**. Press **READ/ENTER**. The display will show: **ENTER #:**

3. Press **427**. Then press **READ/ENTER**. The display will show: **P427 ENTER nm**

4. Press **600**. Press **READ/ENTER**.

Note: *If you make an error, press* **SHIFT CLEAR** *and re-enter the number. When the number is correct, press* **READ/ENTER**.

   The display will show: **P427 DECIMAL? 00.00**

5. Use the arrow keys to correctly position the decimal point. Press the down arrow key once. The display will show: **DECIMAL? 000.0**

   Press **READ/ENTER**. The display will show: **P427 UNITS?**

6. Use the arrow keys to select the appropriate unit of measure. Press the **DOWN ARROW** key twice. The display will show: **P427 mg/l**

7. Press **READ/ENTER** when the correct unit of measure is displayed. The display will show: **P427 mg/l _**

8. Construct the display to read the correct symbol. The symbol must be entered exactly as shown including dashes and spaces between characters.

   C TOC LR

   To enter the display symbol, press the following number sequences, following each number with an enter key to place the character then enter again to accept and move to the next position.

   To enter **C,** press **67 ENTER ENTER**

   To enter a space character, press **32 ENTER ENTER**

   To enter **T**, press **84 ENTER ENTER**

   To enter **O**, press **79 ENTER ENTER**

   To enter **C**, press **67 ENTER ENTER**

   To enter a space character, press **32 ENTER ENTER**

   To enter **L**, press **76 ENTER ENTER**

   To enter **R**, press **82 ENTER ENTER**

   Press **ENTER** to conclude the symbol entry.

   The instrument will exit Display entry mode.

Alternately, you can scroll to each of the proper characters with the arrow keys and use the following procedures:

a. Select letters by scrolling to the correct character with the arrow keys.

b. To make a letter uppercase, press the **SHIFT** key.

c. The space is the character displayed after one press of the **DOWN ARROW**.

d. Make sure to enter the display line EXACTLY as shown, including all spaces.

e. Accept each symbol by pressing **READ/ENTER.**

f. After accepting the last character of the symbol press **READ/ENTER** again. The instrument will exit Display entry mode.

9. The instrument will prompt for entry of timed steps: **P427 TIMER?**

10. This method has no timers, so press **READ/ENTER**. The display will show: **# 0 STANDARD**

11. Press **READ/ENTER** to display the zero data pair. The display will show: **0.000 Abs 000.0 mg/l.** Press **READ/ENTER**. The display will show: **# 1 STANDARD**

12. Press **READ/ENTER**. The display will prompt for entry of the first concentration point: **# 1 000.0 mg/l**

13. Enter concentration point #1 from the table below by pressing **0010** so that the display shows: **# 1 001.0 mg/l**

14. Press **READ/ENTER**. The display will prompt for entry of the first absorbance point: **# 1 0.000 Abs**

15. Enter absorbance point #1 from the table below by pressing **0037** so that the display shows: **# 1 0.037 Abs**

Since this is a negative slope method, change the absorbance value to negative by pressing the **SHIFT** key and the **MINUS** key to toggle between positive and negative slope. You only need to do this once. The remainder of the absorbance values will automatically be stored as negative values.

16. Press **READ/ENTER**. The display will show the first data pair: **-0.037 Abs 001.0 mg/l**

17. Press **READ/ENTER** to accept the first data pair.
The display will show: **# 2 STANDARD**

18. Following *steps 12* through *17* as above, enter the remaining data pair values from the table below:

Standard	Concentration	Absorbance
# 0	[0.0] mg/l	[0.000] Abs
# 1	[1.0] mg/l	[0.037] Abs
# 2	[3.0] mg/l	[0.114] Abs
# 3	[4.0] mg/l	[0.154] Abs
# 4	[5.0] mg/l	[0.195] Abs
# 5	[6.0] mg/l	[0.238] Abs
# 6	[7.0] mg/l	[0.281] Abs
# 7	[9.0] mg/l	[0.326] Abs
# 8	[10.0] mg/l	[0.419] Abs
# 9	[11.0] mg/l	[0.468] Abs
# 10	[13.0] mg/l	[0.569] Abs
# 11	[15.0] mg/l	[0.676] Abs
# 12	[17.0] mg/l	[0.787] Abs
# 13	[19.0] mg/l	[0.903] Abs
# 14	[20.0] mg/l	[0.962] Abs
# 15	[21.0] mg/l	[1.021] Abs

19. When the last point pair is entered the display will show: **#16 STANDARD**

20. Press **SHIFT READ/ENTER** to complete data point entry The display will show: **#:**

21. Enter the validation number: **10309** so that the display shows: **#: 10309**

22. Press **READ/ENTER**. The display will show **COMPLETED** then **P427 mg/l C TOC LR**

Note: *If the display shows:* **INCORRECT #** *then prompts again for the validation number you may have made an error during data entry. Make sure that the validation number is correct. If so then the error occurred during some other portion of the method entry. You must press* **METH** *and respond to the* **ABORT?** *message by pressing* **READ/ENTER** *then re-enter the method.*

The instrument is now ready for use with method 427.

ORGANIC CARBON, TOTAL, Low Range, continued

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Total Organic Carbon Direct Method Low Range			
Test 'N Tube™ Reagent Set		50 vials	27603-45
Includes:			
Acid Digestion Solution Vials, Low Range TOC	1	50/pkg	*
Funnel, micro	1	each	25843-35
Indicator Ampules, Low Range TOC	1	10/pkg	*
TOC Persulfate Powder Pillows	1	50/pkg	*
Water, organic-free**	3.0 mL	500 mL	26415-49

REQUIRED APPARATUS

COD Reactor, 115/230 V ac (U.S.A. and Canada)	1	each	45600-00
COD Reactor, 115/230 V ac (Europe)	1	each	45600-02
Cylinder, graduated, 10-mL	1	each	508-38
Flask, Erlenmeyer, 50-mL	1	each	505-41
Magnetic Stirrer	1	each	23436-00
Safety Shield, laboratory bench	1	each	23810-00
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette® Pipet	2	50/pkg	25589-96
Stir Bar, Magnetic	1	each	45315-00
Wipes, Disposable, Kimwipes	1	280/pkg	20970-00

OPTIONAL REAGENTS

TOC Standard Solution (KHP Standard, 1000 mg/L C)		5/pkg	27915-05
Potassium Acid Phthalate		500 g	315-34
Sulfuric Acid Reagent Solution, 5.25 N		100 mL MDB	2449-32

OPTIONAL APPARATUS

Analytical Balance		each	26103-00
Flask, volumetric, 1000-mL		each	14574-53
Flask, volumetric, 100-mL		each	14574-42
Pipet, Class A, 10.00-mL		each	14515-38
Pipet, Class A, 15.00-mL		each	14515-39

* These items are not sold separately.
** This item must be purchased separately.

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