



✓ Method 8034

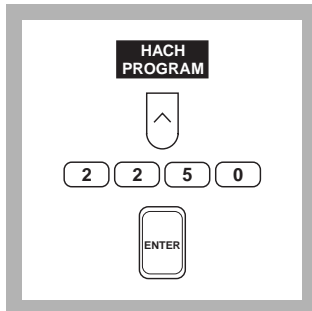
Periodate Oxidation Method*

HR (0 to 20.0 mg/L)

Scope and Application: For soluble manganese in water and wastewater; USEPA approved for reporting wastewater analyses (digestion is required)**. See Section 1 for digestion procedure. The estimated detection limit for program number 2250 is 0.1 mg/L as Mn.

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

** *Federal Register*, 44 (116) 34193 (June 14, 1979)



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

Select the stored program number for high range manganese (Mn) by pressing **2250** with the numeric keys.

Press: **ENTER**

Note: If samples cannot be analyzed immediately, see *Sample Collection, Storage and Preservation*, following these steps. Adjust pH of preserved samples before analysis.

Note: The Flow Cell and Sipper Modules can be used with this procedure. Use a 25-mL sample and reagents with the Flow Cell Module.



2. The display will show: **HACH PROGRAM: 2250 Manganese, HR**

The wavelength (λ), **525 nm**, is automatically selected.



3. Fill a cell with 10 mL of sample.

Note: For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 10, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under **ZERO**. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under **OPTIONS, (MORE)**, and then **BLANK:OFF**. Enter the reagent blank value and press **ENTER**. Repeat for each new lot of reagent.

Note: For proof of accuracy, use a 5.0 mg/L manganese standard solution (preparation given in the Accuracy Check section) in place of the sample.



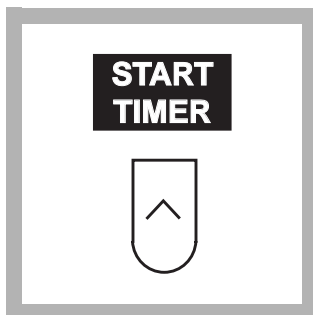
4. Add the contents of one Buffer Powder Pillow, Citrate Type for Manganese. Swirl to mix.

MANGANESE, continued



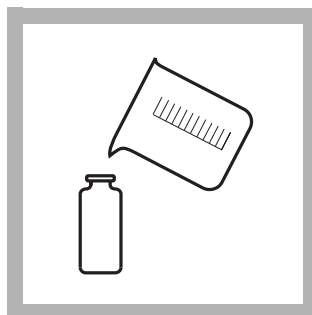
5. Add the contents of one Sodium Periodate Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

Note: A violet color will develop if manganese is present.

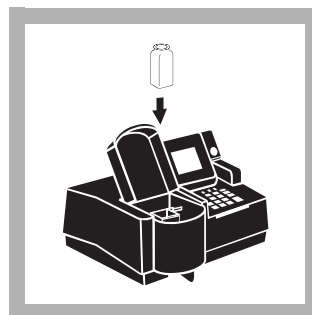


6. Press the soft key under **START TIMER**.

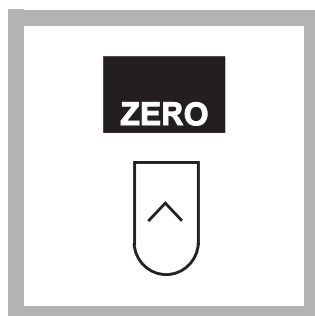
A 2-minute reaction period will begin.



7. Fill another sample cell (the blank) with 10 mL of sample.



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:

0.0 mg/L Mn

Note: If you are using a reagent blank correction, the display will show the correction.

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.



10. Within eight minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield. The result in mg/L Mn (or chosen units) will be displayed.

Note: Results may be expressed as permanganate (MnO_4^-) or as potassium permanganate ($KMnO_4$). Press the soft keys under **OPTIONS** and then **FORM**: to scroll through the available options.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Interfering Substance	Interference Levels and Treatments
Calcium	700 mg/L
Chloride	70,000 mg/L
Iron	5 mg/L
Magnesium	100,000 mg/L
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Section 1.3.1 <i>pH Interference</i> .

Sample Collection, Storage and Preservation

Collect samples in acid-washed plastic bottles. Do not use glass containers due to possible adsorption of Mn to glass. If samples are acidified, adjust the pH to 4–5 with 5.0 N Sodium Hydroxide before analysis. Do not exceed pH 5, as manganese may precipitate. Correct the test result for volume additions; see Section 1.2.2 *Correcting for Volume Additions*.

If only dissolved manganese is to be determined, filter the sample before acid addition.

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), 10.
- c. Press **ENTER** to accept the default standard concentration (mg/L), 100.
- d. Press the soft key under **ENTRY DONE**.
- e. Prepare a 10.0-mg/L manganese standard solution. (See *Calibration Standard Preparation*)
- f. Use the TenSette Pipet to add 0.1, 0.2 mL and 0.3 mL of standard, respectively to three 10-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 10.0-mg/L manganese standard solution by pipetting 10.0 mL of Manganese Standard Solution, 1000-mg/L, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the manganese periodate oxidation procedure as described above.

The calibration curve can be adjusted to account for variability in laboratory technique. To adjust the calibration curve using the reading obtained with the 10.0Hmg/L standard solution, press the soft keys under **OPTIONS, (MORE)** then **STD: OFF**. Press **ENTER** to accept the default concentration, the value of which will depend 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: 10.0 mg/L Mn

Program	95% Confidence Limits
2250	9.9–10.1 mg/L Mn

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

Estimated Detection Limit

Program	EDL
2250	0.1 mg/L Mn

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: 2250

Portion of Curve	Δ Abs	Δ Concentration
0.010 Abs	0.010	0.11 mg/L
10 mg/L	0.010	0.13 mg/L
18 mg/L	0.010	0.14 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

Calibration Standard Preparation

To perform a manganese calibration using the periodate oxidation method, prepare a 100-mg/L Mn stock solution by pipetting 10.00 mL of a 1000-mg/L Manganese Standard Solution (Cat. No. 12791-42) 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, 8.0, 12.0, 16.0 and 20.0 mg/L Mn as follows:

- a. Into six different 100-mL volumetric flasks, pipet 2.00, 4.00, 8.00, 12.00, 16.00 and 20.00 mL of the 100-mg/L Mn stock solution using Class A glassware.
- b. Dilute to the mark with deionized water. Mix thoroughly.
- c. Using the periodate oxidation method and the calibration procedure described above, generate a calibration curve from the standards prepared above.

Summary of Method

Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese 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

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

MANGANESE, continued

REQUIRED REAGENTS AND STANDARDS

High Range Manganese Reagent Set (100 Tests*)			Cat. No.
Includes: (1) 21076-69, (1) 21077-69			24300-00

Description	Quantity Required		Cat. No.
	per test	Unit	
Buffer Powder Pillows, citrate type for manganese	1 pillow	100/pkg	21076-69
Sodium Periodate Powder Pillows, for manganese	1 pillow	100/pkg	21077-69

REQUIRED EQUIPMENT AND SUPPLIES

DR/4000 1-Inch Cell Adapter	1	each	48190-00
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OPTIONAL REAGENTS AND STANDARDS

Hydrochloric Acid, 6.0 N	500 mL	884-49
Manganese Standard Solution, 1000-mg/L Mn	100 mL	12791-42
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Solution, 5.0 N	100 mL MDB	2450-32
Water, deionized	4 liters	272-56

OPTIONAL EQUIPMENT AND SUPPLIES

Ampule Breaker Kit	each	21968-00
Dropper, 0.5 and 1.0 mL marks	20/pkg	21247-20
DR/4000 Carousel Module Kit	each	48070-02
DR/4000 Flow Cell Module Kit, 1-inch	each	48070-04
DR/4000 Flow Cell Module Kit, 1-cm	each	48070-05
DR/4000 Sipper Module Kit, 1-inch	each	48090-03
Flask, Erlenmeyer, 250-mL	each	505-46
Flask, volumetric, Class A, 50-mL	each	14574-41
Flask, volumetric, Class A, 100-mL	each	14574-42
Flask, volumetric, Class A, 1000-mL	each	14574-53
pH Paper, pH 1.0 to 11.0	5 rolls/pkg	391-33
pH Meter, <i>sension</i> TM 1, portable	each	51700-00
Pipet, serological, 1-mL	each	532-35
Pipet, serological, 5-mL	each	532-37
Pipet, volumetric, Class A, 2.00-mL	each	14515-36
Pipet, volumetric, Class A, 4.00-mL	each	14515-04
Pipet, volumetric, Class A, 5.0-mL	each	14515-37
Pipet, volumetric, Class A, 6.0-mL	each	14515-06
Pipet, volumetric, Class A, 8.0-mL	each	14515-08
Pipet, volumetric, Class A, 10.0-mL	each	14515-38
Pipet, volumetric, Class A, 20.0-mL	each	14515-20
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Filler, safety bulb	each	14651-00

* 100 tests equal 100 samples and 100 blanks.



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