Manganese, High Range

DOC316.53.01460

USEPA[1] Periodate Oxidation Method[2]

Method 8034

0.1 to 20.0 mg/L Mn

Powder Pillows

Scope and application: For soluble manganese in water and wastewater.

[1] USEPA Approved for reporting wastewater analyses (digestion required). Federal Register, 44(116)34 193 (June 14, 1979).
[2] Adapted from Standard Methods for the Examination of Water and Wastewater.

Test preparation

Before starting

To make sure that all forms of the metal are measured, digest the sample with heat and acid. Use the mild or vigorous digestion. Refer to the *Water Analysis Guide* for more information.
Always do tests in sample cells. Do not put the instrument in the sample or pour the sample into the cell holder.
Make sure that the sample cells are clean and there are no scratches where the light passes through them.
Rinse the sample cell and cap with the sample three times before the sample cell is filled.
Make sure that there are no fingerprints or liquid on the external surface of the sample cells. Wipe with a lint-free cloth before measurement.
Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.
Install the instrument cap over the cell holder before ZERO or READ is pushed.
After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.
For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Description	Quantity
High Range Manganese Reagent Set, 10-mL	1
Sample cell, 25-mm (10 mL)	2

Refer to Consumables and replacement items on page 4 for order information.

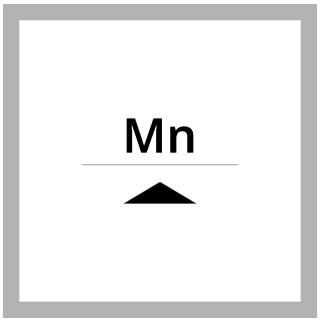
Sample collection and storage

- Collect samples in acid-washed plastic bottles. Do not use glass containers to prevent possible adsorption of manganese to glass.
- If only dissolved manganese is to be determined, filter the sample before acid addition.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (about 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at room temperature for a maximum of 6 months.

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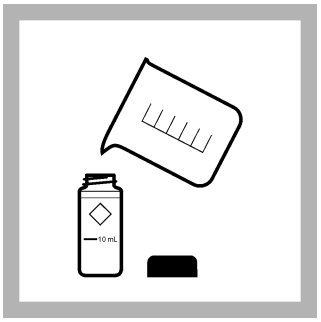
- Before analysis, adjust the pH to 4–5 with 5.0 N sodium hydroxide standard solution. Do not exceed pH 5 to prevent precipitation of the manganese.
- Correct the test result for the dilution caused by the volume additions.

Powder pillow procedure

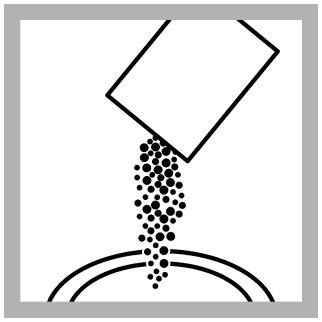


1. Set the instrument to manganese (Mn).

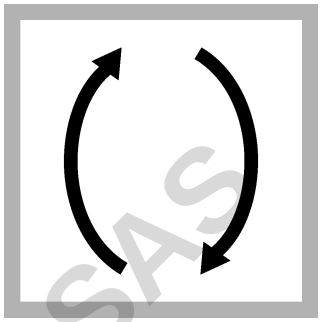
For DR300, push the up arrow button. For PCII, push the menu button, checkmark button, then the menu button again.



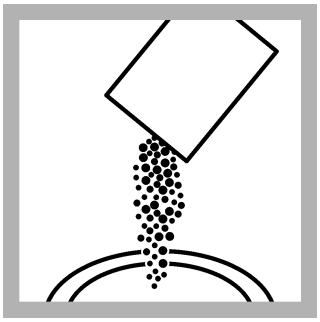
2. Prepare the sample: Rinse a sample cell and cap three times with sample. Fill the sample cell to the 10-mL mark with sample.



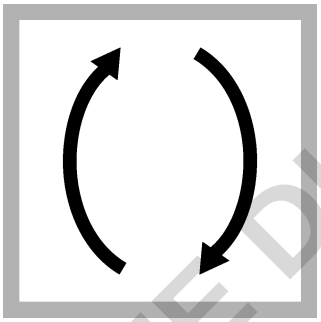
3. Add the contents of one Buffer Powder Pilow, Citrate Type for Manganese.



4. Put the stopper on the sample cell. Invert the sample cell to mix.



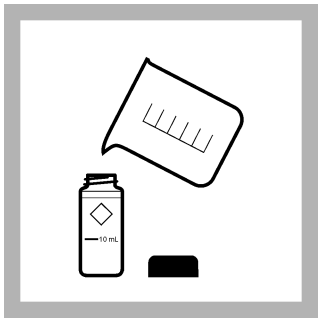
5. Add the contents of one Sodium Periodate Powder Pillow to the sample cell.



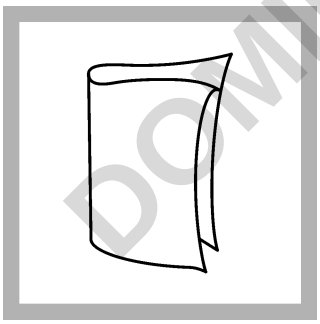
6. Put the stopper on the sample cell. Invert to mix. A violet color will show if manganese is in the sample.



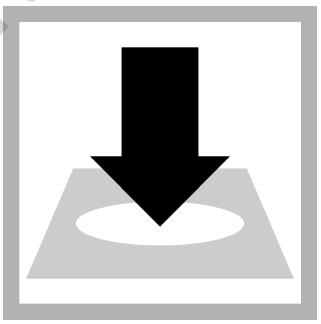
7. Set and start a timer for 2 minutes. A 2-minute reaction time starts.



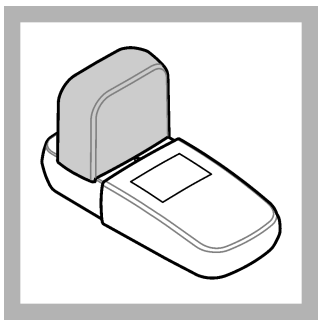
8. Prepare the blank: Rinse a sample cell and cap three times with sample. Fill the sample cell to the 10-mL mark with sample. Close the sample cell.



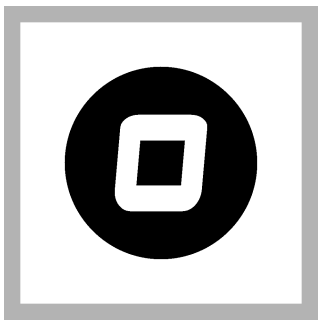
9. When the timer expires, clean the blank sample cell.



10. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.

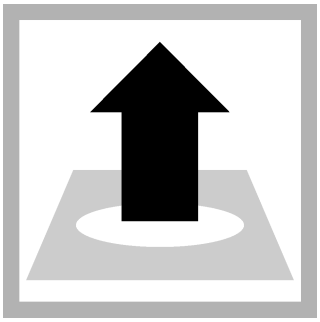


11. Install the instrument cap over the cell holder.

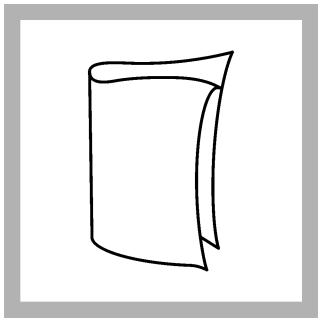


12. Push **ZERO**. The display shows “0.0”.

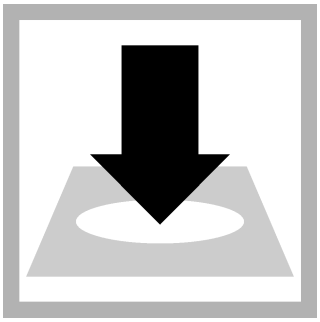
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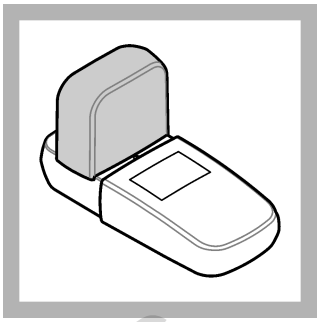
13. Remove the sample cell from the cell holder.



14. Clean the prepared sample cell.



15. Within 8 minutes after the timer expires, insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



16. Install the instrument cap over the cell holder.



17. Push **READ**. Results show in mg/L manganese (Mn). To change the result to MnO_4–, multiply the result by 2.16. To change the result to $KMnO_4$, multiply the result by 2.88.

Interferences

Interfering substance	Interference level
Calcium	700 mg/L
Chloride	70,000 mg/L
Iron	5 mg/L
Magnesium	100,000 mg/L
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment (of the sample) by the reagents. Sample pretreatment may be necessary.

Accuracy check

Standard additions method

Use the standard additions method to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Manganese Voluette® Ampule Standard, 25 mg/L Mn
- Ampule breaker

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- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Prepare three spiked samples: use the TenSette pipet to add 0.2 mL, 0.4 mL and 0.6 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.
2. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
3. Compare the expected result to the actual result. The expected manganese concentration increase is approximately 0.5, 1.0 and 1.4 mg/L for the 0.2, 0.4 and 0.6 mL of standard added, respectively.

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Manganese Standard Solution, 1000 mg/L
- 1-L volumetric flask, Class A
- 10-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 10.0 mg/L manganese standard solution as follows:
   a. Use a pipet to add 10.00 mL of 1000 mg/L manganese standard solution into the volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the prepared standard solution.
3. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard calibration adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a DR300 and a Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)
10.0 ± 0.2 mg/L Mn

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

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Manganese Reagent Set, High Range, 10-mL includes:	1	100/pkg	2430000
Buffer Powder Pillow, Citrate for Manganese, 10-mL	1	100/pkg	2107669
Sodium Periodate Powder Pillow for Manganese, 10 mL	1	100/pkg	2107769

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Required apparatus

Description	Quantity/test	Unit	Item no.
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606

Recommended standards and apparatus

Description	Unit	Item no.
Manganese Standard Solution, 25 mg/L Mn, 2-mL PourRite® Ampule	20/pkg	2112820
Manganese Standard Solution, 10 mg/L Mn, 2-mL PourRite® Ampule	20/pkg	2605820
Manganese Standard Solution, 1000-mg/L Mn	100 mL	1279142
PourRite® Ampule Breaker, 2-mL	each	2484600

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Flask, volumetric, Class A, 1000 mL glass	each	1457453
Paper, pH, 0–14 pH range	100/pkg	2601300
Pipet filler, safety bulb	each	1465100
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	1000/pkg	2185628
Pipet, TenSette®, 1.0–10.0 mL	each	1970010
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	250/pkg	2199725
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	50/pkg	2199796
Pipet, volumetric, Class A, 10 mL	each	1451538
Nitric Acid Solution, 1:1	500 mL	254049
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Water, deionized	4 L	27256

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