

## USEPA<sup>1</sup> Periodate Oxidation Method<sup>2</sup>

**Method 8034**
**0.1 to 20.0 mg/L Mn (HR)**
**Powder Pillows**

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

<sup>1</sup> USEPA Approved for reporting wastewater analyses (digestion required). Federal Register, 44(116)34 193 (June 14, 1979).

<sup>2</sup> Adapted from Standard Methods for the Examination of Water and Wastewater.



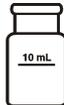
### Test preparation

### Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the applicable information for this test.

**Table 1 Instrument-specific information**

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

### Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

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.

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 automatically with the reagent blank adjust option.

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

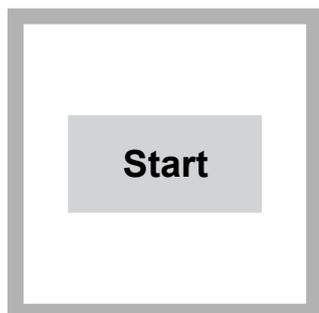
Description	Quantity
High Range Manganese Reagent Set, 10-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to <a href="#">Instrument-specific information</a> on page 1.)	2

Refer to [Consumables and replacement items](#) on page 5 for order information.

## Sample collection and storage

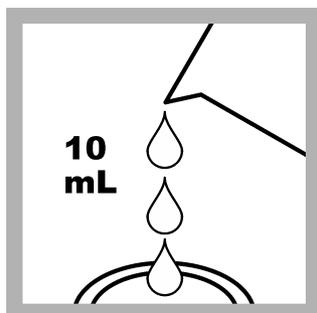
- Collect samples in acid-washed plastic bottles. Do not use glass containers to prevent possible adsorption of manganese to glass.
- If only dissolved manganese is to be determined, filter the sample before acid addition.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 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.
- Before analysis, adjust the pH to 4–5 with 5 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

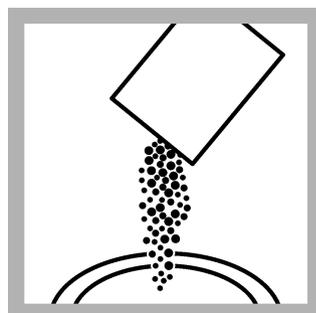


1. Start program **295 Manganese, HR**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

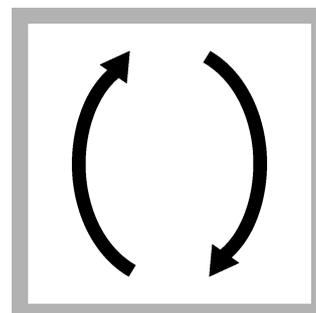
**Note:** Although the program name can be different between instruments, the program number does not change.



2. **Prepare the sample:** Fill a sample cell with 10 mL of sample.



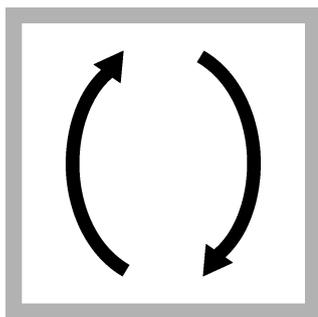
3. Add the contents of one Buffer Powder Pillow, Citrate Type for Manganese.



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



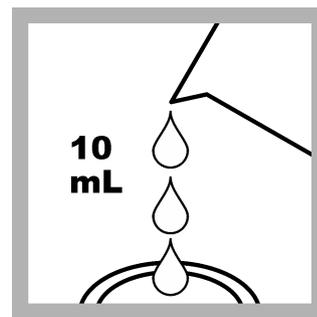
5. Add the contents of one Sodium Periodate Powder Pillow to the sample cell.



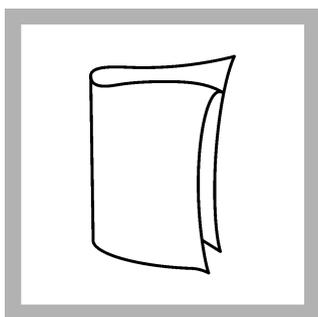
6. Put the stopper on the sample cell. Invert to mix. A violet color will show if manganese is present in the sample.



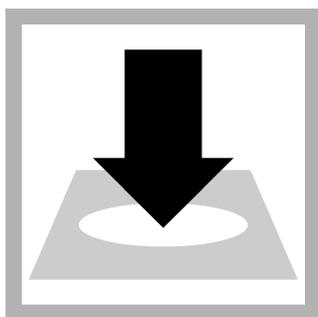
7. Start the instrument timer. A 2-minute reaction time starts.



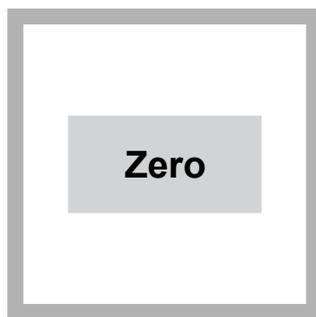
8. **Prepare the blank:** Fill a second sample cell with 10 mL of sample.



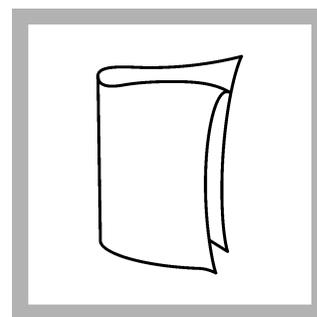
9. When the timer expires, clean the blank sample cell.



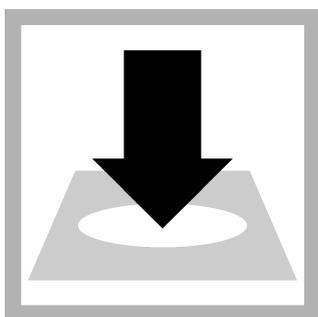
10. Insert the blank into the cell holder.



11. Push **ZERO**. The display shows 0.0 mg/L Mn.



12. Clean the prepared sample cell.



13. Within 8 minutes after the timer expires, insert the prepared sample into the cell holder.



14. Push **READ**. Results show in mg/L Mn.

## 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 pre-treatment may be necessary.

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## Accuracy check

### Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) 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, 250 mg/L Mn
  - Ampule breaker
  - Pipet, TenSette®, 0.1–1.0 mL and tips
1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
  2. Go to the Standard Additions option in the instrument menu.
  3. Select the values for standard concentration, sample volume and spike volumes.
  4. Open the standard solution.
  5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.
  6. 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.
  7. Select **Graph** to compare the expected results to the actual results.

***Note:** If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

### 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 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 slight variations in the reagents or instruments.*

## Method performance

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

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
295	10.0 mg/L Mn	9.6–10.4 mg/L Mn	0.1 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. The measurement wavelength is 525 nm for spectrophotometers or 520 nm for colorimeters.

## 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

### Required apparatus

Description	Quantity/test	Unit	Item no.
Sample cells, 10-mL square, matched pair	2	2/pkg	2495402
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

### Recommended standards and apparatus

Description	Unit	Item no.
Manganese Standard Solution, 1000-mg/L Mn	100 mL	1279142
Manganese Standard Solution, 250-mg/L Mn, 10-mL Voluette <sup>®</sup> Ampule	16/pkg	1425810
Water, deionized	4 L	27256
Ampule Breaker, 10-mL Voluette <sup>®</sup> Ampules	each	2196800

### Optional reagents and apparatus

Description	Unit	Item no.
Manganese Standard Solution, 2-mL PourRite <sup>®</sup> Ampule, 25 mg/L	20/pkg	2112820
Manganese Standard Solution, 10-mg/L Mn, 2 mL PourRite <sup>®</sup> Ampule	20/pkg	2605820
Paper, pH, 0–14 pH range	100/pkg	2601300
Pipet filler, safety bulb	each	1465100
Pipet, TenSette <sup>®</sup> , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette <sup>®</sup> Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette <sup>®</sup> Pipet, 0.1–1.0 mL	1000/pkg	2185628
Pipet, TenSette <sup>®</sup> , 1.0–10.0 mL	each	1970010
Pipet tips for TenSette <sup>®</sup> Pipet, 1.0–10.0 mL	250/pkg	2199725

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**Optional reagents and apparatus (continued)**

<b>Description</b>	<b>Unit</b>	<b>Item no.</b>
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	50/pkg	2199796
PourRite® Ampule Breaker, 2-mL	each	2484600
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Flask, volumetric, Class A, 1000-mL glass	each	1457453
Pipet, volumetric, Class A, 10-mL	each	1451538



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