Manganese, LR

DOC316.53.01057

1-(2-Pyridylazo)-2-Naphthol PAN Method[1] — Method 8149

0.006 to 0.700 mg/L Mn (LR) — Powder Pillows

Scope and application: For water and wastewater; digestion is necessary for total manganese determinations.

[1] Adapted from Goto, K., et al., Talanta, 24, 652-3 (1977).

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
DR6000 DR3800 DR2800 DR2700 DR1900	The fill line is to the right.	2495402 
DR5000 DR3900	The fill line is toward the user.	
DR900	The orientation mark is toward the user.	2401906 

Before starting

Install the instrument cap on the DR900 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.
Rinse all glassware with a 1:1 (50%) nitric acid solution. Rinse again with deionized water.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
The alkaline cyanide solution contains cyanide. Make sure to read the Safety Data Sheets and obey the safety precautions.
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.

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Items to collect

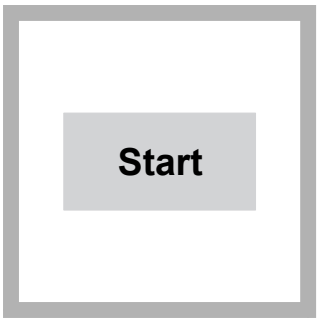
Description	Quantity
Alkaline Cyanide Reagent	12 drops
Water, deionized	10 mL
Ascorbic Acid Powder Pillow	2
PAN Indicator Solution, 0.1%	12 drops
Stoppers for 18-mm tubes and AccuVac Ampul	2
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 5 for order information.

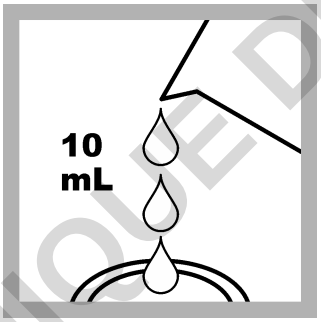
Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- 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 solution.
- Correct the test result for the dilution caused by the volume additions.

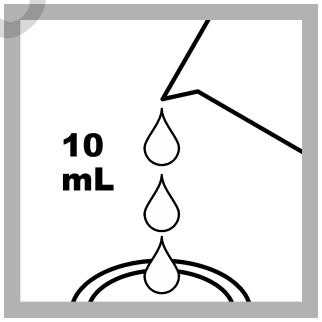
Powder pillow procedure



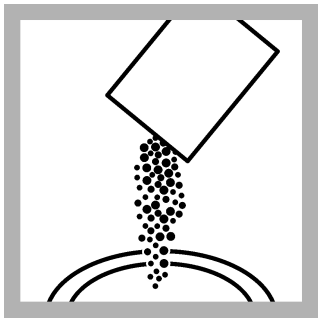
1. Start **Program 290 Manganese, LR PAN** . For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



2. Prepare the blank: Fill the sample cell with 10 mL of deionized water. Digestion is necessary for total manganese determination.

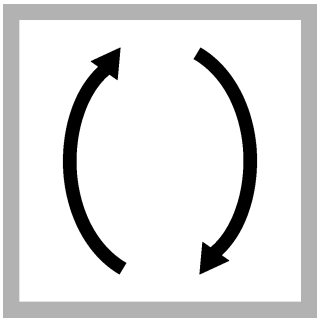


3. Prepare the sample: Fill a second sample cell with 10 mL of sample.

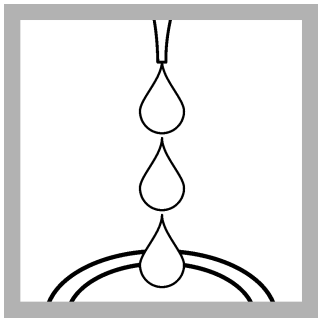


4. Add the contents of one Ascorbic Acid Powder Pillow to each sample cell.

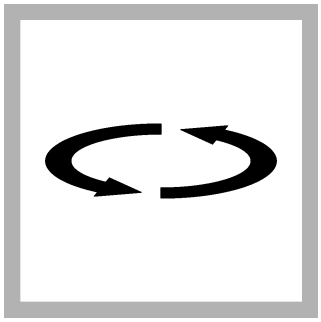
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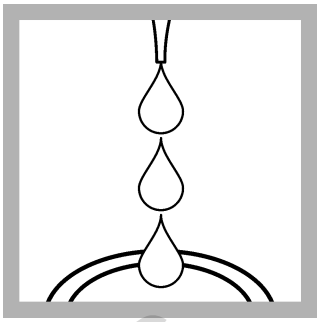
5. Put the stopper on both sample cells. Invert to dissolve the powder.



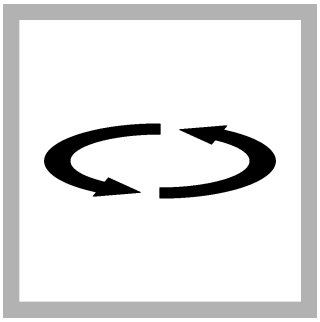
6. Add 12 drops of Alkaline Cyanide Reagent Solution to each cell.



7. Swirl to mix.
The solution may start to show turbidity. This should dissipate in the next step.



8. Add 12 drops of PAN Indicator Solution, 0.1% to each cell.



9. Swirl to mix.
The sample will show an orange color if manganese is present.



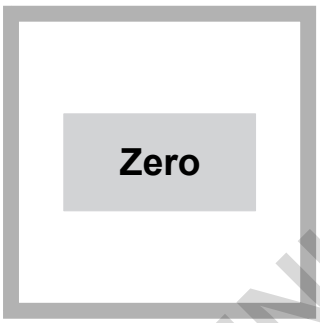
10. Start the instrument timer. A 2-minute reaction time starts.



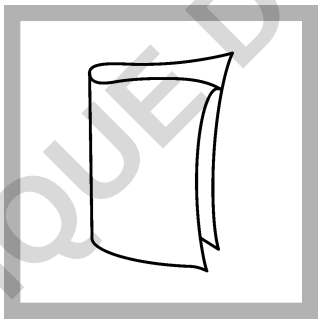
11. When the timer expires, clean the blank sample cell.



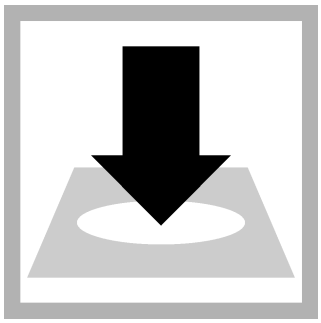
12. Insert the blank into the cell holder.



13. Push **ZERO**. The display shows 0.000 mg/L Mn.



14. Clean the prepared sample cell.



15. Insert the prepared sample into the cell holder.



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

Interferences

Interfering substance	Interference level
Aluminum	20 mg/L
Cadmium	10 mg/L
Calcium	1000 mg/L as $CaCO_3$
Cobalt	20 mg/L
Copper	50 mg/L
Hardness	For samples that contain more than 300 mg/L hardness as $CaCO_3$, add 4 drops of Rochelle Salt Solution to the sample and the blank after the Ascorbic Acid Powder Pillow is added.
Iron	25 mg/L (If the sample contains more than 5 mg/L iron, increase the reaction time to 10 minutes.)

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Interfering substance	Interference level
Lead	0.5 mg/L
Magnesium	300 mg/L as $CaCO_3$
Nickel	40 mg/L
Zinc	15 mg/L

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 Standard Solution, 10 mg/L Mn, PourRite Ampule
- 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, 25 mg/L Mn
- 100-mL volumetric flask, Class A
- 2.0-mL volumetric pipet, Class A and pipet filler
- Deionized water

1. Prepare a 0.5-mg/L manganese standard solution as follows:
   a. Use a pipet to add 2.0 mL of the 25-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. The result should be 0.5 mg/L Mn.

   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 small variations in the reagents or instruments.*

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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
290	0.500 mg/L Mn	0.491–0.509 mg/L Mn	0.006 mg/L Mn

Summary of method

The PAN method is a highly sensitive and rapid procedure to measure low levels of manganese. The ascorbic acid reagent reduces all oxidized forms of manganese to Mn^{2+}. The alkaline-cyanide reagent masks potential interferences. PAN Indicator then forms an orange-colored complex with Mn^{2+}. The measurement wavelength is 560 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Manganese Reagent Set, 10 mL, includes:	—	50 tests	2651700
Alkaline Cyanide Reagent	12 drops	50 mL SCDB	2122326
Ascorbic Acid Powder Pillow	2	100/pkg	1457799
PAN Indicator Solution, 0.1%	12 drops	50 mL SCDB	2122426
Water, deionized	varies	4 L	27256

Recommended standards and apparatus

Description	Unit	Item no.
Manganese Standard Solution, 10 mg/L Mn, 2-mL PourRite® Ampule	20/pkg	2605820
Manganese Standard Solution, 25 mg/L Mn, 2-mL PourRite® Ampule	20/pkg	2112820
PourRite® Ampule Breaker, 2 mL	each	2484600
Metals Drinking Water Standard, HR for Cu, Fe, Mn	500 mL	2833649

Optional reagents and apparatus

Description	Unit	Item no.
Mixing cylinder, graduated, 25 mL	each	2088640
Flask, volumetric, Class A, 100 mL, glass, Certified	each	2636642
Nitric Acid, concentrated	500 mL	15249
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, TenSette®, 1.0–10.0 mL	each	1970010
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 tips for TenSette® Pipet, 1.0–10.0 mL	50/pkg	2199796
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	250/pkg	2199725
Pipet, volumetric, Class A, 2 mL	each	1451536

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Optional reagents and apparatus (continued)

Description	Unit	Item no.
Rochelle Salt Solution	29 mL	172533
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032

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