Nickel

DOC316.53.01064

USEPA[1] Heptoxime Method[2]

Method 8037

0.02 to 1.80 mg/L Ni

Powder Pillows

Scope and application: For water, wastewater and seawater.

[1] USEPA accepted for reporting wastewater analysis (digestion required). Procedure is equivalent to Standard Method 3500-Ni D for wastewater.

[2] Adapted from *Chemie Analytique*, 36 43 (1954).

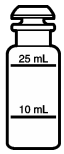
Test preparation

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.	2612602 
DR 5000 DR 3900	The fill line is toward the user.	

Before starting

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.

Make the cotton plug the size of a pea. A larger plug decreases the flow and a smaller plug can dislodge from the delivery tube of the funnel.

Do not pour chloroform solutions down the drain. Collect the water saturated with chloroform, chloroform solutions and the cotton plug used in the delivery tube of the separatory funnel for proper disposal. Refer to a current MSDS/SDS for safe handling and disposal instructions.

In bright light conditions (e.g., direct sunlight), close the cell compartment, if applicable, with the protective cover during measurements.

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

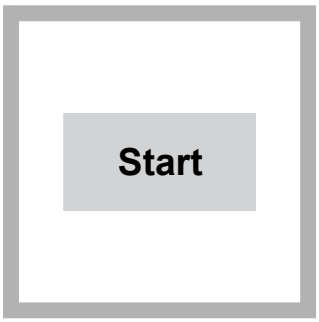
Description	Quantity
Chloroform, ACS	30 mL
Nickel 1 Reagent Powder Pillow	1
Nickel 2 Reagent Powder Pillow	1
Clippers to open powder pillows	1
Cotton balls	varies
Cylinder, graduated, 10-mL	1
Cylinder, graduated, 500-mL	1
Funnel, separatory with stand and stopper	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2
Water, deionized	varies

Refer to Consumables and replacement items on page 6 for order information.

Sample collection and storage

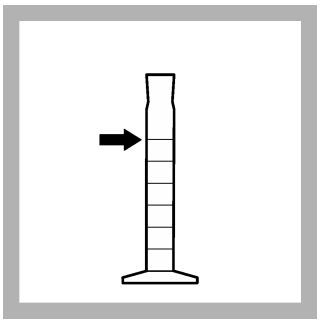
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is 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 3–8 with 5 N sodium hydroxide solution. Do not exceed pH 8 as this can cause some loss of nickel as a precipitate .
- Correct the test result for the dilution caused by the volume additions.

Test procedure

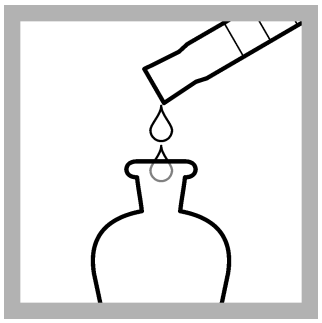


1. Start program **335 Nickel Heptoxime**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

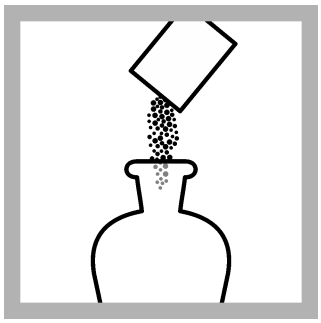
Note: *Although the program name can be different between instruments, the program number does not change.*



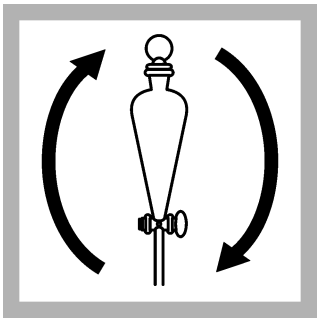
2. Measure 300 mL of the sample in a 500-mL graduated cylinder.



3. Pour the sample into a 500-mL separatory funnel.

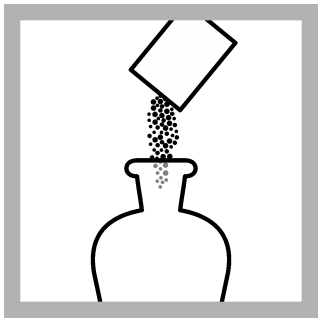


4. Add the contents of one Nickel 1 Reagent Powder Pillow to the funnel.

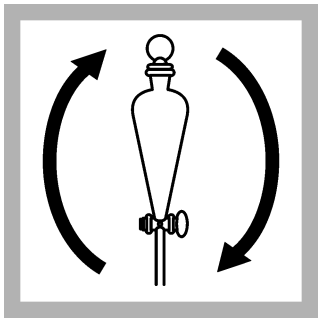


5. Put the stopper on the funnel. Invert to mix.

6. Start the instrument timer. The reaction time starts.

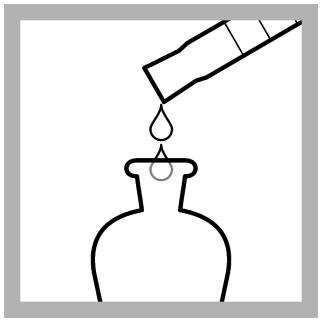


7. When the timer expires, add the contents of one Nickel 2 Reagent Powder Pillow to the funnel.

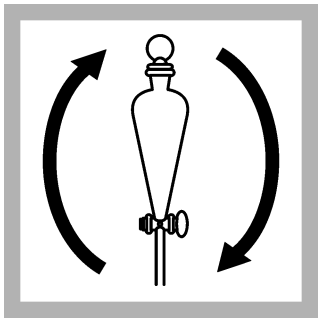


8. Put the stopper on the funnel. Invert to mix.

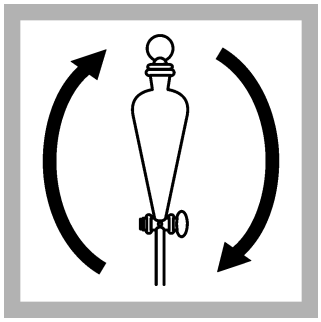
9. Start the instrument timer. The reaction time starts.



10. When the timer expires, add 10 mL of chloroform to the funnel.

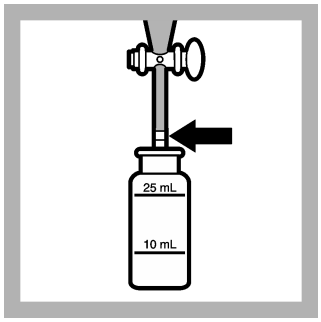


11. Put the stopper on the funnel. Carefully invert to mix. Make sure that the funnel tip is pointed up and away from people. Open the stopcock to release pressure.

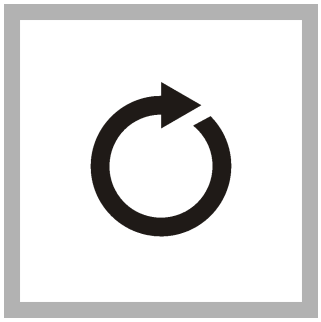


12. Close the stopcock. Invert for 30 seconds.

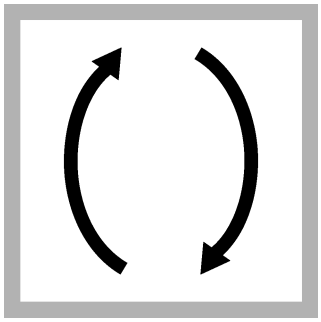
13. Start the instrument timer. The reaction time starts. Invert the funnel approximately 5 times during the 5-minute reaction period.



14. Prepared sample: When the timer expires, wait for the layers to separate. Put a cotton plug the size of a pea into the delivery tube of the funnel. Remove the stopper and drain the chloroform layer (the bottom layer) into a sample cell.

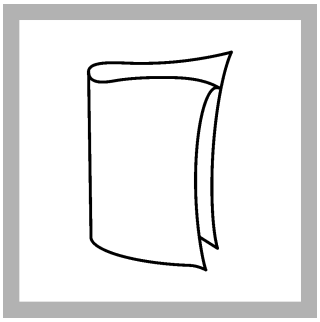


15. Do steps 10 to 14 two more times with 10-mL volumes of chloroform. The 5-minute reaction period is not necessary.

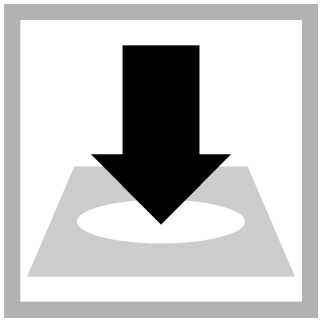


16. Put the cap on the sample cell. Invert to mix the extracts. The final volume is approximately 25 mL because of the slight solubility of chloroform in water.

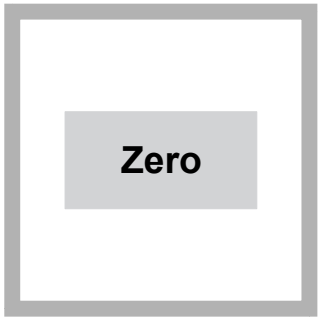
17. Prepare the blank: Fill a second sample cell with 10 mL of chloroform. Put the cap on the blank cell.



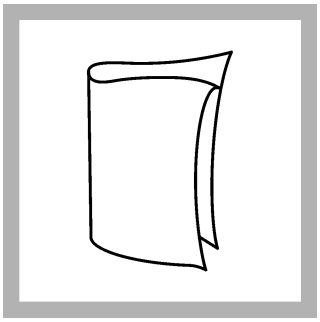
18. Clean the blank sample cell.



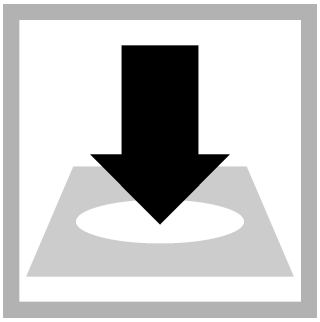
19. Insert the blank into the cell holder.



20. Push **ZERO**. The display shows 0.00 mg/L Ni.



21. Clean the prepared sample cell.



22. Insert the prepared sample into the cell holder.

23. Push **READ**. Results show in mg/L Ni.

Interferences

A preliminary acid digestion is necessary to determine suspended or precipitated nickel and to remove interference from organic matter. To determine total recoverable nickel or remove interference from organic matter, use the USEPA-approved acid digestion procedure.

To remove interference from cobalt, copper and iron, add additional Nickel 1 Reagent Powder Pillows in step 4 of the test procedure. The tolerance limit per powder pillow quantity is shown in Table 2.

Table 2 Tolerance limits vs. reagent quantity

Pillows of Nickel 1 Reagent	Tolerance Limit (mg/L)		
	Cobalt	Copper	Iron
1	1	10	20
2	7	16	65
3	13	22	110
4	18	28	155
5	25	35	200

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:

- Nickel Standard Solution, 1000-mg/L Ni

- Volumetric flask, 50-mL
- 15-mL volumetric pipet, Class A and pipet filler
- Deionized water
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Prepare a 300 mg/L nickel standard solution as follows:
   a. Use a pipet to add 15 mL of a 1000 mg/L nickel standard solution into a 50-mL 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 sample, then keep the (unspiked) sample in the instrument.
3. Go to the Standard Additions option in the instrument menu.
4. Select the values for standard concentration, sample volume and spike volumes.
5. Prepare three spiked samples: use the TenSette pipet to add 0.2 mL, 0.4 mL and 0.6 mL of the prepared standard solution, respectively, to three 300-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:

- Nickel Standard Solution, 1000-mg/L Ni
- 500 mL and 1000 mL volumetric flasks, Class A
- Volumetric pipets, 10 mL and 50 mL, Class A with pipet filler safety bulb
- Deionized water

1. Prepare a 10.00-mg/L nickel stock solution as follows:
   a. Use a pipet to add 10.00 mL of a 1000-mg/L nickel standard solution into a 1-L volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare the stock solution each day.
2. Prepare a 1.00-mg/L nickel standard solution as follows:
   a. Use a pipet to add 50.00 mL of the 10.00-mg/L nickel stock solution into a 500-mL volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare the standard solution each day.
3. Use the test procedure to measure the concentration of the prepared standard solution.
4. 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
335	1.00 mg/L Ni	0.93–1.07 mg/L Ni	0.02 mg/L Ni

Summary of Method

Nickel ion reacts with heptoxime to form a yellow complex that is extracted into chloroform, to concentrate the color and enable a more sensitive determination. Chelating agents are added to the sample to overcome the interferences caused by cobalt, copper and iron. The measurement wavelength is 430 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Nickel reagent Set (50 tests), includes:	—	—	2243500
Chloroform, ACS	30 mL	500 mL	1445849
Nickel 1 Reagent Powder Pillows	1	25/pkg	212368
Nickel 2 Reagent Powder Pillows	1	25/pkg	212468

Required apparatus

Description	Quantity/test	Unit	Item no.
Clippers	1	each	96800
Cotton balls, absorbent	1	100/pkg	257201
Cylinder, graduated, 10-mL	1	each	50838
Cylinder, graduated, 500-mL	1	each	50849
Funnel, separatory, 500-mL	1	each	52049
Support Ring, 4-inch	1	each	58001
Sample cells, 25 mL, matched, 1-inch square	2	2/pkg	2612602
Support, Ring Stand, 5-inch x 8-inch base	1	each	56300

Recommended standards

Description	Unit	Item no.
Nickel Standard Solution, 1000-mg/L Ni (NIST)	100 mL	1417642

Optional reagents and apparatus

Description	Unit	Item no.
Mixing cylinder, graduated, 25-mL	each	189640
Pipet, volumetric Class A, 15-mL	each	1451539
Pipet, volumetric, Class A, 50-mL	each	1451541
Flask, volumetric, Class A, 500-mL glass	each	1457449
Flask, volumetric, Class A, 1000-mL glass	each	1457453

Optional reagents and apparatus (continued)

Description	Unit	Item no.
Flask, volumetric, 50-mL	each	1457441
Pipet filler, safety bulb	each	1465100
Nitric Acid Solution, 1:1	500 mL	254049
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Pipet, volumetric, Class A, 10-mL	each	1451538
Water, deionized	4 L	27256

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