Nickel

DOC316.53.01064

USEPA[1] Heptoxime Method[2] **Method 8037**

0.02 to 1.8 mg/L Ni **Powder Pillows**

Scope and Application: For Water, Wastewater and Seawater

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

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

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 228 Instrument-specific information

Instrument	Sample cell	Cell orientation
DR 6000	2495402	Fill line faces right
DR 5000	2612602	Fill line faces user
DR 3900	2612602	Fill line faces user
DR 3800, DR 2800, DR 2700	2612602	Fill line faces right

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Make the cotton plug pea-size. A larger plug will restrict the flow; a smaller plug may become dislodged from the delivery tube of the funnel.
Chloroform (D022) solutions are regulated as hazardous waste. Do not pour these materials down the drain. Water saturated with chloroform, chloroform solutions and the cotton plug used in the delivery tube of the separatory funnel should be collected for proper disposal. Refer to a current MSDS for safe handling and disposal instructions.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the DR 3900, DR 3800, DR 2800 and DR 2700 cell compartment with the protective cover during measurements.

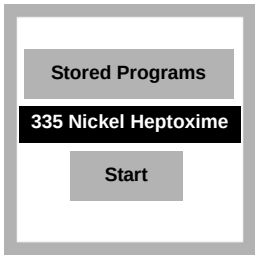
Collect the following items :

Description	Quantity
Chloroform, ACS	30 mL
Nickel 1 Reagent Powder Pillow	1
Nickel 2 Reagent Powder Pillow	1

Collect the following items (continued):

Description	Quantity
Clippers for Opening Pillows	1
Cotton Balls	varies
Cylinder, graduated. 10-mL	1
Cylinder, graduated, 500-mL	1
Funnel, separatory with stand and stopper	1
Sample Cells (see *Instrument-specific information*)	2
See *Consumables and replacement items* for reorder information.	

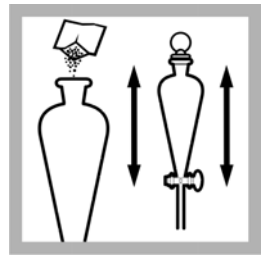
Heptoxime method for powder pillows



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

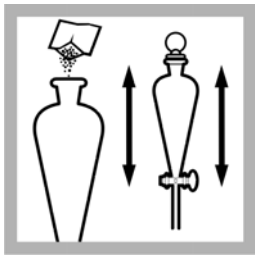
2. Measure 300 mL of sample in a 500-mL graduated cylinder. Pour into a 500-mL separatory funnel.



3. Add the contents of one Nickel 1 Reagent Powder Pillow to the funnel. Stopper and invert to mix.

4. Start the instrument timer.

A five-minute reaction time will begin.

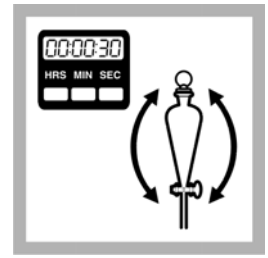


5. When the timer expires, add the contents of one Nickel 2 Reagent Powder Pillow to the funnel. Stopper and invert to mix.

6. Start the instrument timer.

A second five-minute reaction time will begin.

7. When the timer expires, add 10 mL of chloroform. Insert the stopper and invert gently. With the funnel inverted and the tip pointed away from people, open the stopcock to vent.



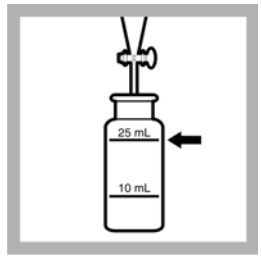
8. Close the stopcock and invert for 30 seconds.

Heptoxime method for powder pillows (continued)

9. Start the instrument timer.

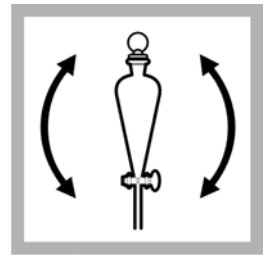
A third five-minute reaction period will begin.

Invert the funnel several times over the five minute period.

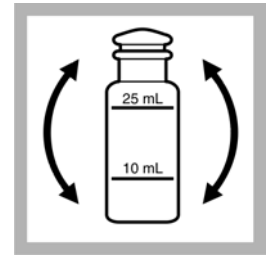


10. Prepared Sample: When the timer expires, wait for the layers to separate. Insert a pea-sized cotton plug into the delivery tube of the funnel. Remove the stopper and drain the chloroform layer (bottom layer) into a sample cell.

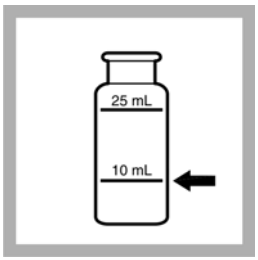
Insert the stopper into the funnel.



11. Repeat steps 7 through 10 two additional times with 10-mL volumes of chloroform. The five-minute reaction period is not necessary.



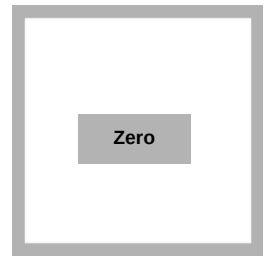
12. Cap the sample cell and invert to mix the extracts. The final volume will be about 25 mL due to the slight solubility of chloroform in water.



13. Blank Preparation: Fill a second sample cell with 10 mL of chloroform.

Cap the cell.

14. Wipe the blank and insert it into the cell holder.



15. ZERO the instrument.

The display will show:

0.00 mg/L Ni

16. Wipe the prepared sample and insert it into the cell holder.

READ the results in mg/L Ni.

Interferences

Cobalt, copper and iron interferences can be overcome by adding additional Nickel 1 Reagent Powder Pillows in step 3 of the *Heptoxime method for powder pillows*. The tolerance limits of these interferences are shown in the *Interfering substances* table.

A preliminary acid digestion is required to determine any suspended or precipitated nickel and to eliminate interference by organic matter. To eliminate this interference or to determine total recoverable nickel perform the USEPA approved digestion.

Table 229 Interfering substances

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

Sample collection, preservation and storage

- Collect samples in acid-washed plastic bottles.
- Adjust the sample pH to 2 or less with Nitric Acid*, about 5 mL per liter. Preserved samples can be stored up to six months at room temperature.
- Before analysis, adjust the sample pH to between 3–8 with 5.0 N Sodium Hydroxide Standard Solution*. Do not exceed pH 8 as this may cause some loss of nickel as a precipitate.
- Correct the test results for volume additions.

Accuracy check

Standard additions method (sample spike)
Prepare a 300 mg/L nickel standard by pipetting 15 mL of 1000 mg/L Nickel standard into a 50 ml volumetric flask. Dilute to volume and mix well.

Required for accuracy check:

- Nickel Standard Solution, 1000-mg/L Ni
- Volumetric Pipet, 15 mL
- Volumetric Flask, 50 mL
- TenSette Pipet and tips
- Pipet Filler

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select **Options>More>Standard Additions** from the instrument menu.
3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Use the TenSette Pipet to prepare spiked samples: add 0.2 mL, 0.4 mL and 0.6 mL of 300 mg/L standard to three 300-mL portions of fresh sample.
5. Follow the *Heptoxime method for powder pillows* test procedure for each of the spiked samples using the powder pillows, starting with the 0.2 mL sample spike. Measure each of the spiked samples in the instrument.
6. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

* See *Optional reagents and apparatus*.

Standard solution method

Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- Nickel Standard Solution, 1000-mg/L
- Deionized water
- Volumetric flasks, 500 mL and 1000 mL
- Volumetric pipets, 10 mL and 50 mL
- Pipet filler

1. Prepare a 10.0 mg/L nickel working solution as follows:
   a. Pipet 10.0 mL of a Nickel Standard Solution, 1000-mg/L, into a 1000-mL (1 liter) volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Prepare a 1.0-mg/L nickel standard solution by diluting 50.0 mL of the 10-mg/L working standard solution to 500 mL in a volumetric flask.
3. Use the standard solution in place of the sample. Follow the *Heptoxime method for powder pillows* test procedure.
4. To adjust the calibration curve using the reading obtained with the standard solution, select **Options>More>Standard Adjust** from the instrument menu.
5. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	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-colored complex which is then 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. Readings are taken at 430 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Nickel Reagent Set (50 Tests), includes:	—	—	2243500
(3) Chloroform, ACS	30 mL	500 mL	1445849
(2) Nickel 1 Reagent Powder Pillows	1	25/pkg	212368
(2) Nickel 2 Reagent Powder Pillows	1	25/pkg	212468

Required apparatus

Description	Quantity	Unit	Catalog number
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
Ring, support, 4-inch	1	each	58001
Sample Cell, 1-inch square, w/stopper, matched pair	2	2/pkg	2612602
Stand, support, 5 x 8-inch base	1	each	56300

Recommended standards

Description	Unit	Catalog number
Nickel Standard Solution, 1000-mg/L Ni (NIST)	100 mL	1417642
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Catalog number
Cylinder, mixing, 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	each	1457449
Flask, Volumetric, Class A, 1000 mL	each	1457453
Flask, Volumetric, Class A, 50 mL	each	1457441
Pipet Filler, Safety Bulb	each	1465100
Nitric Acid 1:1	500 mL	254049
Sodium Hydroxide Standard Solution, 5.0 N	100 mL	245053
Pipet, volumetric, 10 mL	each	1451538

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