



✓ Method 8037

Heptoxime Method*

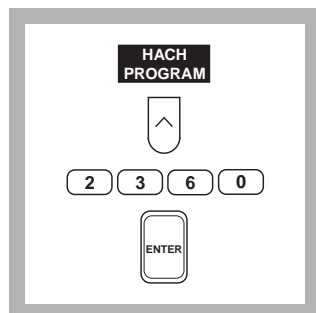
Powder Pillows

(0 to 1.80 mg/L Ni)

Scope and Application: For water, wastewater and seawater; USEPA accepted for reporting wastewater analyses (digestion required)**. See Section 1 for digestion procedure. The estimated detection limit for program number 2360 is 0.01 mg/L.

* Adapted from *Chimie Analytique*, 36 43 (1954)

** Procedure is equivalent to Standard Method 3500-Ni D for wastewater.



1. Press the soft key under **HACH PROGRAM**.

Select the stored program for nickel (Ni) by pressing **2360** with the numeric keys.

Press: **ENTER**

Note: If sample cannot be analyzed immediately, see *Sample Collection, Storage and Preservation* following these steps. Adjust the pH of preserved samples before analysis.

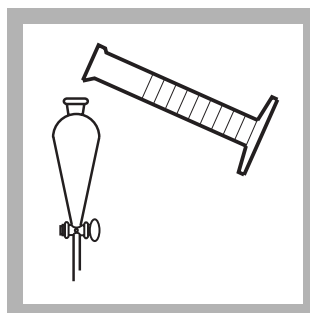
Note: The Flow Cell and Sipper Modules cannot be used with this procedure.



2. The display will show: **HACH PROGRAM: 2360 Nickel, Heptoxime**

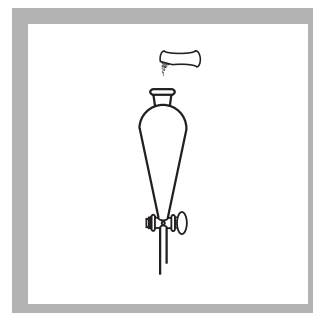
The wavelength (λ), **430 nm**, is automatically selected.

Note: For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 14, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under **ZERO**. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under **OPTIONS, (MORE)**, and then **BLANK:OFF**. Enter the reagent blank value and press **ENTER**. Repeat for each new lot of reagent.

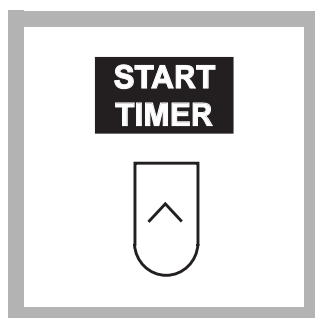


3. Measure 300 mL of sample in a 500-mL graduated cylinder. Pour into a 500-mL separatory funnel.

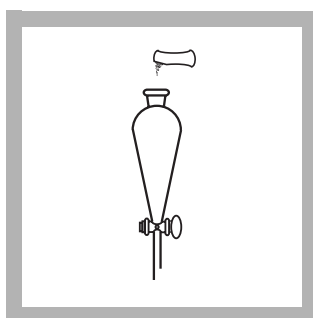
Note: For proof of accuracy, use a 1.0 mg/L nickel standard solution (preparation given in the Accuracy Check section) in place of the sample.



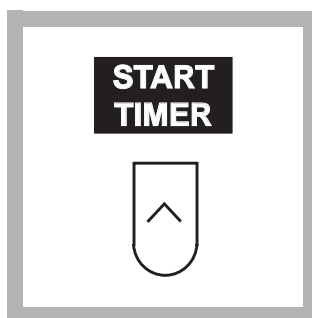
4. Add the contents of one Nickel 1 Reagent Powder Pillow to the funnel. Stopper. Shake to mix.



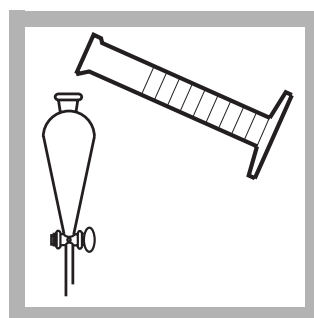
5. Press the soft key under **START TIMER**. A 5-minute reaction period will begin.



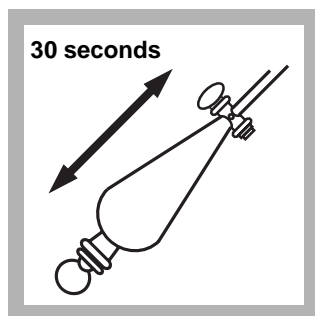
6. When the timer beeps, add the contents of one Nickel 2 Reagent Powder Pillow to the funnel. Stopper. Shake to mix.



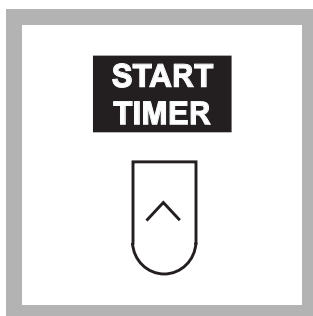
7. Press the soft key under **START TIMER**. A second 5-minute reaction period will begin.



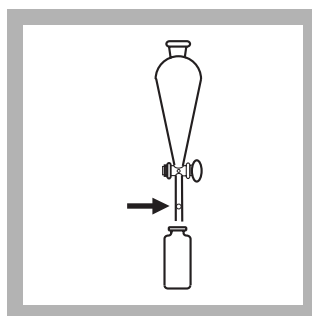
8. When the timer beeps, add 10 mL of chloroform. Stopper. Shake gently. Invert. With tip pointed up and away from people, open the stopcock to vent.



9. Close stopcock. Shake for 30 seconds.



10. Press the soft key under **START TIMER**. A third 5-minute reaction period will begin. Shake the funnel several times over the five minute period.



11. When the timer beeps wait for the layers to separate. Insert a pea-sized cotton plug into the delivery tube of the funnel. Drain the chloroform layer (bottom layer) into a sample cell (the prepared sample). Stopper.

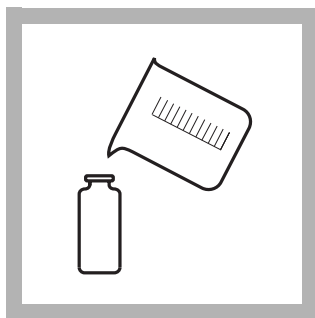


12. Repeat steps 8 to 11 two additional times with 10-mL portions of chloroform.

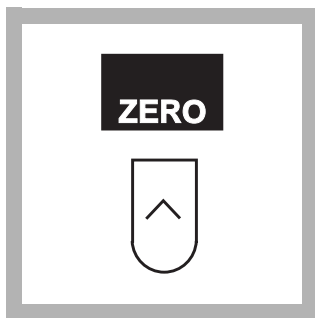
Note: The 5-minute reaction period is not necessary. Shake with chloroform, wait for layers to separate, then continue.

Note: The final volume of extract will be about 25 mL due to the slight solubility of chloroform in water.

Note: Swirl sample cell to mix extracts.



13. Fill a second cell (the blank) with 25 mL of chloroform. Stopper. Place the blank into the cell holder. Close the light shield.

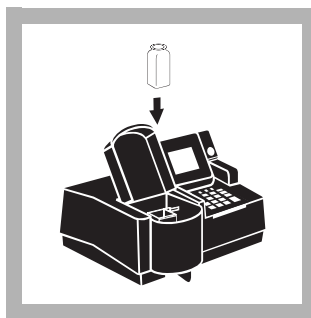


14. Press the soft key under **ZERO**.

The display will show:

0.00 mg/L Ni

Note: For alternate concentration units, press the soft key under **OPTIONS**. Then press the soft key under **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.



15. Place the prepared sample into the cell holder. Close the light shield. The result in mg/L nickel (or chosen units) will be displayed.

Interferences

Cobalt, copper and iron interferences can be overcome by adding additional Nickel 1 Reagent Powder Pillows in Step 4. The tolerance limits of these interferences are shown in the following table:

Table 1 Tolerance Limits vs. Number of Nickel 1 Reagent Powder Pillows

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

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 in Section 2.

Sample Collection, Storage and Preservation

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 and 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; see Section 1.2.2 *Correcting for Volume Additions*.

Accuracy Check

Standard Solutions Method

Prepare a 10.0-mg/L nickel working standard solution by pipetting 10.0 mL of a Nickel Standard Solution, 1000-mg/L, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. 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. Perform the heptoxime procedure as described above.

To adjust the calibration curve using the reading obtained with the 1.0-mg/L standard solution, press the soft keys under **OPTIONS, MORE** then **STD: OFF**. Press **ENTER** to accept the displayed concentration, the value of which depends on the selected units. If an alternate concentration is used, enter the actual concentration and press **ENTER** to return to the read screen. See Section 1.5.5 *Adjusting the Standard Curve* for more information.

Or, use the TenSette Pipet to add 1.0 mL of a Nickel Voluette Ampule Standard Solution, 300-mg/L Ni, into a 500-mL volumetric flask and dilute to volume with deionized water. This is a 0.60-mg/L nickel standard solution.

Method Performance

Precision

Standard: 1.00 mg/L Ni²⁺

Program	95% Confidence Limits
2360	0.99–1.01 mg/L Ni ²⁺

For more information on determining precision data and method detection limits, refer to Section 1.5.

Estimated Detection Limit

Program	EDL
2360	0.01 mg/L Ni ²⁺

For more information on derivation and use of Hach’s estimated detection limit, see Section 1.5.2. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, appendix B, see Section 1.5.1.

Sensitivity

Program Number: 2360

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	0.02 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

Calibration Standard Preparation

To perform a nickel calibration using the heptoxime method, prepare a 10-mg/L Ni stock solution by pipetting 10 mL of a 1000-mg/L Nickel Standard Solution (Cat. No. 23383-42) into a 1000-mL volumetric flask using Class A glassware. Dilute to the mark with deionized water and mix thoroughly.

Prepare calibration standards containing 0.10, 0.30, 0.60, 0.90, 1.20, 1.50 and 1.80 mg/L Ni as follows:

- a. Into seven different Class A 100-mL volumetric flasks, pipet 1, 3, 6, 9, 12, 15 and 18 mL of the 10-mg/L Ni stock solution using class A glassware.
- b. Dilute to the mark with deionized water. Mix thoroughly.
- c. Using the heptoxime method and the calibration procedure described in the *User-Entered Programs* section of the *DR/4000 Spectrophotometer Instrument Manual*, generate a calibration curve from the standards prepared above.

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.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheet* for information specific to the reagents used. For additional information, refer to Section 1.

Pollution Prevention and Waste Management

Chloroform (D022) solutions are regulated as hazardous waste by the Federal RCRA. 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 disposal with laboratory solvent wastes. See Section 1 for more information on proper disposal of these materials.

NICKEL, continued

REQUIRED REAGENTS AND STANDARDS

Nickel Reagent Set (50 Tests)	22435-00
Includes: (3) 14458-49, (2) 2123-68, (2) 2124-68	

Description	Quantity Required		Cat. No.
	per test	Unit	
Chloroform, ACS	55 mL	500 mL	14458-49
Nickel 1 Reagent Powder Pillows	1 pillow	25/pkg	2123-68
Nickel 2 Reagent Powder Pillows	1 pillow	25/pkg	2124-68

REQUIRED EQUIPMENT AND SUPPLIES

Clippers, for opening powder pillows	1	each	968-00
Cotton balls, absorbent	1	100/pkg	2572-01
Cylinder, graduated, 10-mL	1	each	508-38
Cylinder, graduated, 500-mL	1	each	508-49
DR/4000 1-Inch Cell Adapter	1	each	48190-00
Funnel, separatory, 500-mL	1	each	520-49
Ring, support, 4-inch	1	each	580-01
Sample Cells, matched pair, 1-inch, glass, with stoppers	2	pair	26126-02
Stand, support, 5" X 8" base	1	each	563-00
Stopper, hollow, Size 1	2	6/pkg	14480-00

OPTIONAL REAGENTS AND STANDARDS

Nickel Standard Solution, 1000-mg/L Ni	100 mL	14176-42
Nickel Standard Solution, 10-mL Voluette ampule, 300-mg/L Ni	16/pkg	14266-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Solution, 5.0 N	1 liter	2450-53
Water, deionized	4 liters	272-56

OPTIONAL EQUIPMENT AND SUPPLIES

DR/4000 Carousel Module Kit	each	48090-02
Flask, Erlenmeyer, 500-mL	each	505-49
Flask, volumetric, Class A, 500-mL	each	14574-49
Flask, volumetric, Class A, 1000-mL	each	14574-53
pH Paper, pH 1.0 to 11.0	5 rolls/pkg	391-33
Pipet, serological, 1-mL	each	532-35
Pipet, serological, 5-mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 1.0-mL	each	14515-35
Pipet, volumetric, Class A, 3.0-mL	each	14515-03
Pipet, volumetric, Class A, 6.0-mL	each	14515-06
Pipet, volumetric, Class A, 9.0-mL	each	14515-09
Pipet, volumetric, Class A, 10.00-mL	each	14515-38
Pipet, volumetric, Class A, 15.00-mL	each	14515-39
Pipet, volumetric, Class A, 50.00-mL	each	14515-41
Pipet Filler, safety bulb	each	14651-00



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