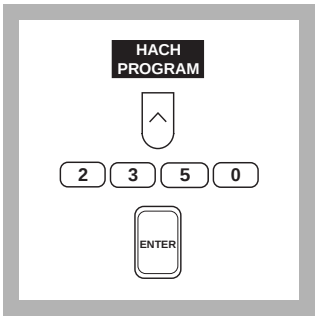
NICKEL, AUTOCATALYTIC

Method 8118 **Photometric Method**

(0 to 8.00 g/L)

Scope and Application: *For finishing baths. The estimated detection limit for program number 2350 is 0.05 g/L.*



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

Select the stored program for autocatalytic nickel by pressing **2350** with the numeric keys.

Press: **ENTER**

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

Note: *This method gives accurate results on most bath formulations. If the bath formulation in use responds differently, perform a new manual calibration. Prepare and store the calibration as directed under Calibration Standard Preparation.*

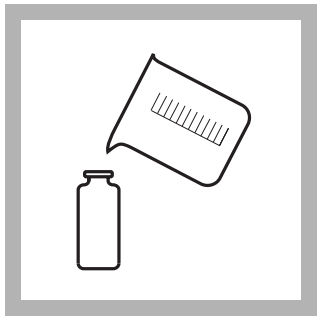
Note: *If samples cannot be analyzed immediately, see Sample Collection, Storage and Preservation following these steps.*

2. The display will show:

HACH PROGRAM: 2350 Nickel, Autocatal.

The wavelength (λ), **720 nm**, is automatically selected.

3. Fill a sample cell with 10 mL of deionized water (the blank).



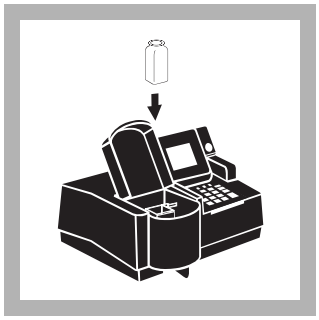
4. Fill a second cell with 10 mL of bath sample.

Note: *Filter highly turbid samples.*

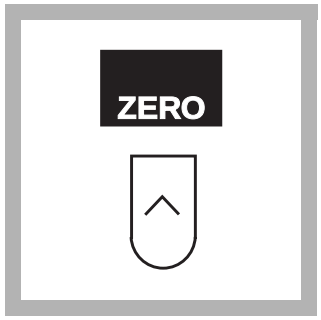
Note: *For proof of accuracy, use a 1000-mg/L (1.00-g/L) Nickel Standard Solution (see the OPTIONAL REAGENTS AND STANDARDS* section*) in place of the sample.*

5. Add the contents of one Potassium 1 Reagent Powder Pillow to the sample (the prepared sample). Stopper and shake to dissolve.

Note: *If a visible turbidity forms upon addition of Potassium 1 Reagent, dilute sample 1:1 with deionized water and repeat Step 5. Multiply results obtained in Step 8 by 2.*



6. Place the blank into the cell holder. Close the light shield.

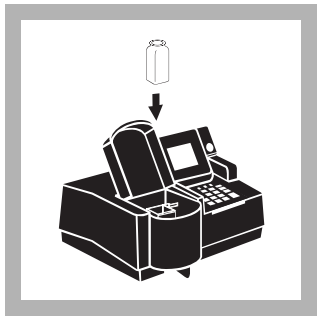


7. Press the soft key under ***ZERO***.

The display will show:

0.00 g/L Ni^{2+}

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



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

Interferences

Table 1 Interfering Substances That Cause a Negative Interference

Interfering Substance	Interference Levels and Treatments
Copper	All levels. Gives a similar blue color.

Sample Collection, Storage and Preservation

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower.

Accuracy Check

Standard Solutions Method

Use a 1000 mg/L Nickel Standard Solution in Step 4 in place of the sample. Perform the autocatalytic procedure as described above.

To adjust the calibration curve using the reading obtained with the 1000-mg/L Nickel 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.

Method Performance

Precision
Standard: 1.00 g/L Ni^{2+}

Program	95% Confidence Limits
2350	0.97–1.03 g/L Ni^{2+}

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

Estimated Detection Limit

Program	EDL
2350	0.05 g/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: 2350

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	0.12 g/L Ni^{2+}

See Section *1.5.3 Sensitivity Explained* for more information.

Calibration Standard Preparation

This method gives accurate results on most bath formulations. If the bath formulation in use responds differently, perform a new manual calibration.

a. Prepare a sample of the finishing bath of known correct strength (100%). Or, titrate a sample of the bath to determine its exact strength.

b. Dilute this bath sample 1:1 with deionized water to make a half-strength standard.

c. Into three separate cells, add 10-mL of the following: deionized water (reagent blank), half-strength standard, and full-strength standard (100%).

d. Using the colorimetric method for autocatalytic nickel 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. Use the following user-method selections:

Method name: Nickel, Autocatal.
Format: XX.XX
Units: g/L
Chemical form: Ni
Wavelength: 720 nm

e. Scroll to the Calibration table selection and press the soft key under ***CREATE TABLE***. If the program has been stored previously, the soft key will display ***EDIT TABLE*** instead.

f. Add the contents of one Potassium 1 Reagent Powder Pillow to each of the three cells as described in Step 5.

g. Enter the nickel concentration of the reagent blank. Zero the instrument using deionized water contained in a sample cell. Measure the absorbance of the reagent blank and accept the reading by pressing the soft key under ***READ***. Repeat this procedure for both the half and full-strength standards.

Note: *Some variations of the calibration procedure are possible. See the* DR/4000 Instrument Manual *for details.*

h. When repeating the calibration for new bath formulations, select the same user-assigned program number in the procedure above. Prepare a new calibration curve by editing only the calibration table each time. Use the same stored program number and method settings.

This procedure provides good readability on baths up to 8 g/L. If the bath in use is more concentrated, dilute the sample and apply an appropriate correction factor.

Summary of Method

A strong complexing agent chelates the nickel ions present in an "electroless" nickel bath to form a blue colored chelate. The blue color is then measured directly to give the g/L nickel present in the bath.

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 *3*.

Pollution Prevention and Waste Management

For information on pollution prevention and waste management, refer to Section *3*.

REQUIRED REAGENTS AND STANDARDS

Description	Quantity Required per test	Unit	Cat. No.
Potassium 1 Reagent Powder Pillows	1 pillow	25/pkg	14321-98
Water, deionized	10 mL	4 liters	272-56

REQUIRED EQUIPMENT AND SUPPLIES

DR/4000, 1-inch cell, Adapter	1	each	48190-00
Clippers, for opening powder pillows	1	each	968-00
Sample Cells, matched pair, 1-inch, glass, with stopper	2	pair	26126-02

OPTIONAL REAGENTS AND STANDARDS

Nickel Standard Solution, 1000-mg/L	100 mL	14176-42
Nickel Standard Solution, 1000-mg/L	500 mL	14176-49

OPTIONAL EQUIPMENT AND SUPPLIES

Filter Paper, folded, 12.5-cm	100/pkg	1894-57
Funnel, analytical poly, 65-mm	each	1083-67

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