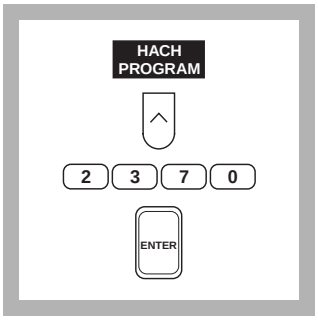
NICKEL

Method 8150 **1-(2 Pyridylazo)-2-Naphthol (PAN)* Method**

Powder Pillows **(0 to 1.000 mg/L)**

Scope and Application: *For water and wastewater; digestion is required for determining total nickel. See SECTION 2 for digestion procedure. The estimated detection limit for program number 2370 is 0.005 mg/L Ni.*

* Adapted from Watanabe, H., *Talanta*, 21 295 (1974)



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

Select the stored program for nickel (Ni) by pressing **2370** with the numeric keys.

Press: **ENTER**

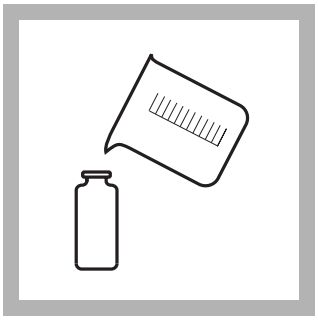
Note: *If sample cannot be analyzed immediately, see Sample Collection, Storage and Preservation following these steps. Adjust the pH of preserved samples before analysis.*

Note: *The Flow Cell and Sipper Modules can be used if rinsed well with deionized water between the blank and prepared sample.*

2. The display will show:

HACH PROGRAM: 2370
Nickel, PAN

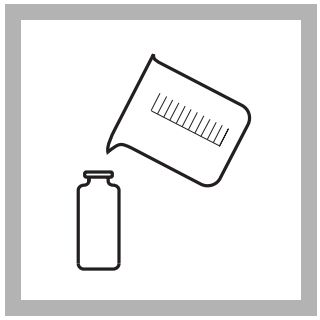
The wavelength (λ), **560 nm**, is automatically selected.



3. Fill a glass-stoppered cell to the 25-mL mark with sample (the prepared sample).

Note: *If sample is less than 10 °C (50 °F), warm to room temperature before analysis.*

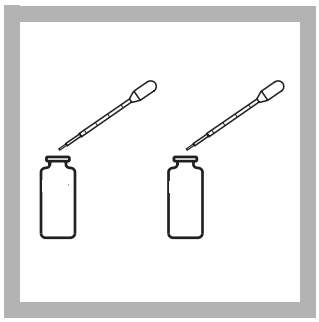
Note: *For proof of accuracy, use a 0.5-mg/L nickel standard solution (preparation given in the Accuracy Check* section*) in place of the sample.*



4. Fill another glass-stoppered cell to the 25-mL mark with deionized water (the blank).

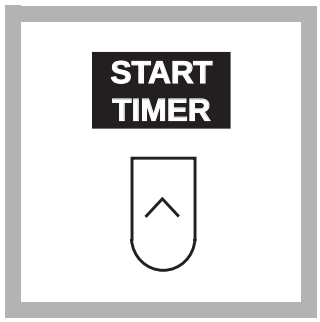
5. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cell. Stopper. Immediately shake to dissolve.

Note: *If sample contains iron (Fe^{3+}) it is important that all powder be dissolved completely before continuing with Step 6.*



6. Add 1.0 mL of 0.3% PAN Indicator Solution to each cell. Stopper. Invert several times to mix.

Note: *Use the plastic dropper provided.*

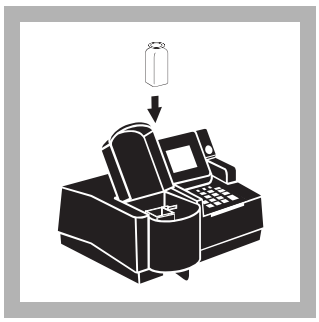


7. Press the soft key under ***START TIMER***.

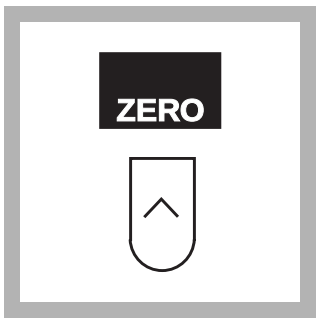
A 15-minute reaction period will begin.

Note: *During color development, the sample solution color may vary from yellowish-orange to dark red depending on the chemical make-up of the sample. The deionized water blank should be yellow.*

8. When the timer beeps add the contents of one EDTA Reagent Powder Pillow to each cell. Stopper. Shake to dissolve.



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



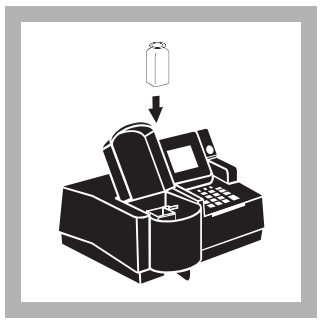
10. Press the soft key under ***ZERO***.

The display will show:

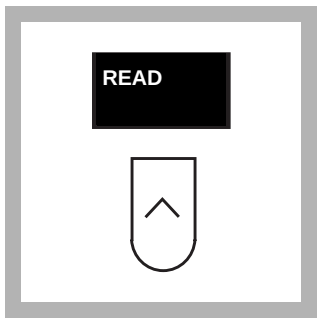
0.000 mg/L Ni

Note: *The instrument will zero at 560 nm and 620 nm.*

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.*



11. Place the sample into the cell holder. Close the light shield.



12. Press the soft key under ***READ***.

The instrument will read the sample at 560 nm and 620 nm. When finished, the result in mg/L nickel (or chosen units) will be displayed.

Note: *Determination of cobalt concentration may be made with the same prepared sample by using* ***HACH METHOD*** *program number* **1600***.*

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Table 1 Interfering Substances and Suggested Treatments

Interfering Substance	Interference Levels and Treatments
Al^{3+}	32 mg/L
Ca^{2+}	1000 mg/L as ($CaCO_3$)
Cd^{2+}	20 mg/L
Cl^-	8000 mg/L
Chelating agents	Interfere at all levels. Use either the Digesdahl or vigorous digestion to eliminate this interference (see *SECTION 2*).
Cr^{3+}	20 mg/L
Cr^{6+}	40 mg/L
Cu^{2+}	15 mg/L
F^-	20 mg/L
Fe^{3+}	10 mg/L
Fe^{2+}	Interferes directly and must not be present.
K^+	500 mg/L
Mg^{2+}	400 mg/L
Mn^{2+}	25 mg/L
Mo^{6+}	60 mg/L
Na^+	5000 mg/L
Pb^{2+}	20 mg/L
Zn^{2+}	30 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see Section *1.3.1 pH Interference*.

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 test results for volume additions, see Section *1.2.2 Correcting for Volume Additions*.

Accuracy Check

Standard Additions Method

a. Leave the unspiked sample in the sample compartment. Verify that the units displayed are in mg/L. Select standard additions mode by pressing the soft keys under ***OPTIONS, (MORE)*** and then ***STD ADD***.

b. Press **ENTER** to accept the default sample volume (mL), 25.

c. Press **ENTER** to accept the default standard concentration (mg/L), 50.

Note: *Alternative standard concentrations and additions volumes can also be used.*

d. Press the soft key under ***ENTRY DONE***.

e. Snap the neck off a Nickel Voluette Ampule Standard, 50-mg/L Ni.

f. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively to three 25-mL samples and mix each thoroughly.

g. Analyze each standard addition sample as described in the procedure. Accept the standard additions reading by pressing the soft key under ***READ*** each time. Each addition should reflect approximately 100% recovery.

h. After completing the sequence, the display will show the extrapolated concentration value and the "best-fit" line through the standard additions data points, accounting for matrix interferences.

i. See Section *1.4.1 Standard Additions* for more information.

Standard Solution Method
Prepare a 5.00-mg/L Nickel stock solution by pipetting 5.00 mL of Nickel Standard Solution, 1000-mg/L as Ni, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Prepare a 0.5-mg/L Ni working solution by pipetting 10.0 mL of the 5.00-mg/L nickel stock solution into a 100-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the nickel procedure as described above.

To adjust the calibration curve using the reading obtained with the 0.5-mg/L working 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: 0.500 mg/L Ni^{2+}

Program	95% Confidence Limits
2370	0.498–0.502 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
2370	0.005 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: 2370

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	0.006 Ni^{2+}

See Section *1.5.3 Sensitivity Explained* for more information.

Calibration Standard Preparation

To perform a nickel calibration using the PAN method, prepare a 5.0-mg/L nickel stock solution by pipetting 5.0 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 0.150, 0.300, 0.450, 0.600, 0.750, 0.900 and 1.000 mg/L Ni as follows:

a. Into seven different Class A 100-mL volumetric flasks, pipet 3, 6, 9, 12, 15, 18, and 20 mL of the 5.0 mg/L Ni stock solution using Class A glassware.

b. Dilute to the mark with deionized water. Mix thoroughly.

c. Using the PAN method and the calibration procedure described in *Creating and Running User Programs* in the *DR/4000 Instrument Manual*, generate a calibration curve from the standards prepared above.

Summary of Method

After buffering the sample and masking any Fe^{3+} with pyrophosphate, the nickel is reacted with 1-(2-Pyridylazo)-2-Naphthol indicator. The indicator forms complexes with most metals present. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt. The DR/4000 automatically adjusts for cobalt interference by measuring the absorbance of the sample at both 560 nm and 620 nm. This method is unique because both nickel and cobalt can be determined on the same sample.

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

For information on pollution prevention and waste management, refer to Section *1*.

REQUIRED REAGENTS AND STANDARDS

Nickel Reagent Set (100 Tests*) 22426-00
Includes: (2) 7005-99, (4) 21501-66, (2) 21502-32

Description	Quantity Required per test	Unit	Cat. No.
EDTA Reagent Powder Pillows	2 pillows	100/pkg	7005-99
Phthalate-Phosphate Reagent Powder Pillows	2 pillows	50/pkg	21501-66
PAN Indicator Solution, 0.3%	2 mL	100 mL MDB	21502-32
Water, deionized	25 mL	4 liters	272-56

REQUIRED EQUIPMENT AND SUPPLIES

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

OPTIONAL REAGENTS AND STANDARDS

Nickel Standard Solution, 1000-mg/L Ni	100 mL	14176-42
Nickel Standard Solution, 2-mL Voluette Ampule, 50-mg/L Ni	20/pkg	25576-20
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 Standard Solution, 5.0 N	100 mL MDB	2450-32

OPTIONAL EQUIPMENT AND SUPPLIES

DR/4000 Carousel Module Kit	each	48070-02
DR/4000 Flow Cell Module Kit, 1-inch	each	48070-04
DR/4000 Flow Cell Module Kit, 1-cm	each	48070-05
DR/4000 Sipper Module Kit, 1-inch	each	48090-03
Flask, volumetric, Class A, 100-mL	each	14574-42
Flask, volumetric, Class A, 1000-mL	each	14574-53
pH Paper, pH 1.0 to 11.0	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable	each	51700-00
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, 3.00-mL	each	14515-03
Pipet, volumetric, Class A, 5.00-mL	each	14515-37
Pipet, volumetric, Class A, 6.00-mL	each	14515-06
Pipet, volumetric, Class A, 9.00-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, 20.00-mL	each	14515-20
Pipet Filler, safety bulb	each	14651-00

* 100 Tests equals 50 sample and 50 blanks. Contact Hach for larger sizes.

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