

1-(2 Pyridylazo)-2-Naphthol (PAN) Method¹**Method 8150****0.01 to 1.00 mg/L Ni****Powder Pillows****Scope and application:** For water and wastewater.¹ Adapted from Watanabe, H., Talanta, 21 295 (1974).**Test preparation****Before starting**

To make sure that all forms of the metal are measured, digest the sample with heat and acid. Use the mild or vigorous digestion. Refer to the Water Analysis Guide for more information.

The recommended temperature for samples and reagents is 15–25 °C (59–77 °F).

Always do tests in sample cells. Do not put the instrument in the sample or pour the sample into the cell holder.

Make sure that the sample cells are clean and there are no scratches where the light passes through them.

Rinse the sample cell and cap with the sample three times before the sample cell is filled.

Make sure that there are no fingerprints or liquid on the external surface of the sample cells. Wipe with a lint-free cloth before measurement.

Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.

Install the instrument cap over the cell holder before ZERO or READ is pushed.

After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.

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.

This method will not differentiate between nickel and cobalt on single-wavelength instruments.

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
Cobalt/Nickel Reagent Set, PAN	1
Water, deionized	10 mL
Sample cells, 25 mm (10-mL)	2

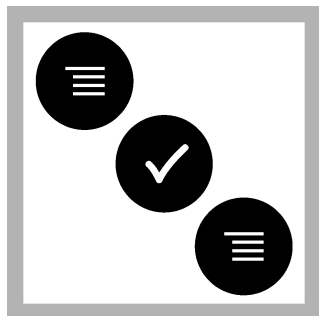
Refer to [Consumables and replacement items](#) on page 5 for order information.

Sample collection and storage

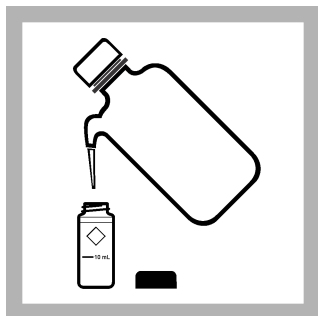
- Collect samples in plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.

- Keep the preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 3–5 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

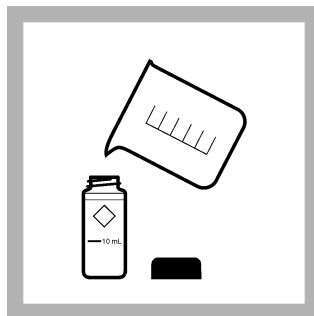
Powder pillow procedure



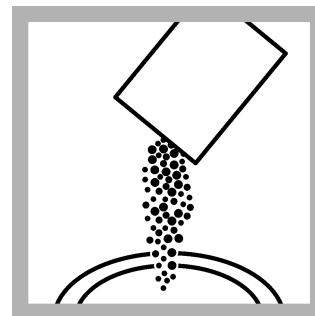
1. Set the instrument to nickel (Ni). Refer to the instrument documentation.



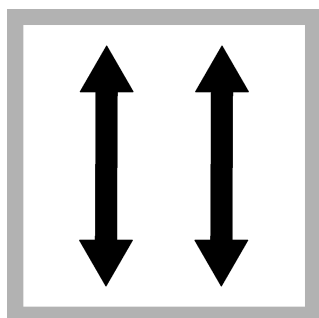
2. **Prepare the blank:** Fill a sample cell to the 10-mL mark with deionized water.



3. **Prepare the sample:** Fill a sample cell to the 10-mL mark with sample.

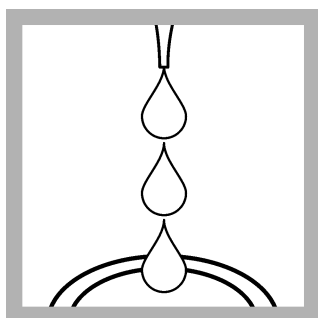


4. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each sample cell.

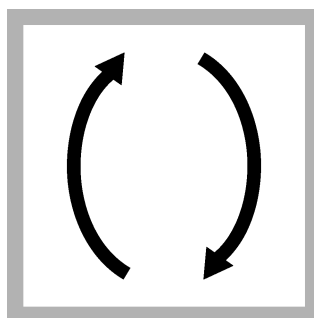


5. Close the sample cells. Immediately shake to dissolve the reagent powder.

If the sample contains iron, make sure that all the powder is dissolved before the PAN Indicator is added.



6. Add 0.5 mL of 0.3% PAN Indicator Solution to each cell. Use the plastic dropper supplied.

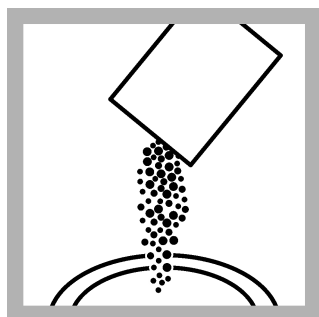


7. Put the stopper on the sample cells. Invert several times to mix.

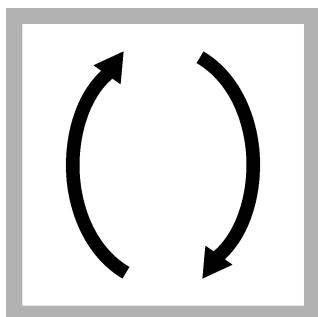


8. Set and start a timer for 15 minutes. A 15-minute reaction time starts.

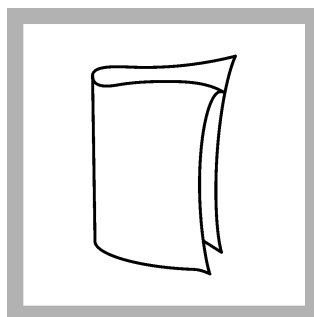
During color development the sample solution color can become yellowish-orange to dark red, based on the chemical makeup of the sample. The blank will be yellow.



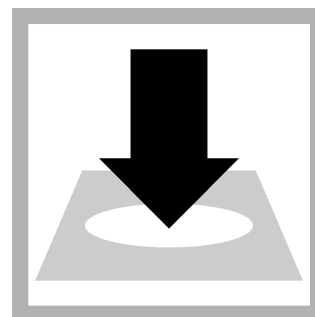
9. When the timer expires, add the contents of one EDTA Reagent Powder Pillow to each cell.



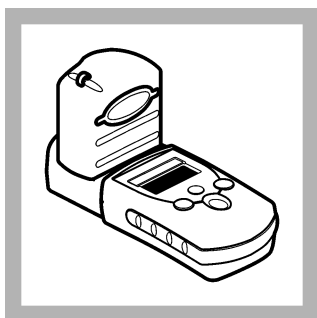
10. Close the sample cells. Gently invert to dissolve the reagent. Let the bubbles exit the solution before measurement.



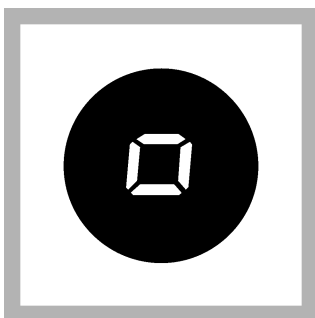
11. Clean the blank sample cell.



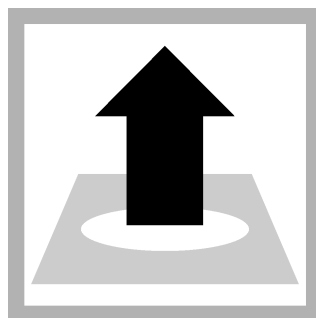
12. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



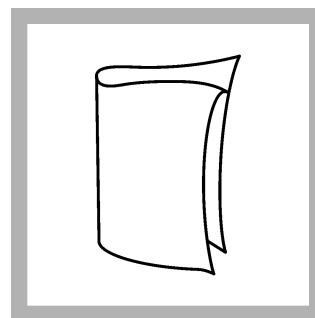
13. Install the instrument cap over the cell holder.



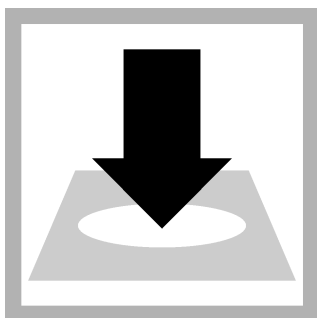
14. Push **ZERO**. The display shows "0.00".



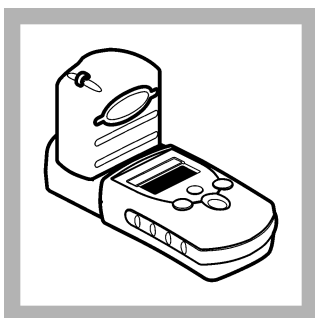
15. Remove the sample cell from the cell holder.



16. Clean the prepared sample cell.



17. Insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



18. Install the instrument cap over the cell holder.



19. Push **READ**. Results show in mg/L nickel (Ni).

Interferences

Interfering substance	Interference level
Al ³⁺	32 mg/L
Ca ²⁺	1000 mg/L as (CaCO ₃)
Cd ²⁺	20 mg/L
Cl ⁻	8000 mg/L
Chelating agents (e.g., EDTA)	Interfere at all levels. Use either the Digesdahl or vigorous digestion to remove this interference.
Cr ³⁺	20 mg/L
Cr ⁶⁺	40 mg/L
Cu ²⁺	15 mg/L
F ⁻	20 mg/L
Fe ³⁺	10 mg/L. If the sample contains iron, make sure that all the powder is dissolved before the PAN Indicator is added.
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

Interfering substance	Interference level
Pb ²⁺	20 mg/L
Zn ²⁺	30 mg/L
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary.

Accuracy check

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- 1000-mg/L Nickel Standard Solution
 - 1-L volumetric flask, Class A
 - 100-mL volumetric flask, Class A
 - 10-mL volumetric pipet, Class A and pipet filler
 - 5-mL volumetric pipet, Class A and pipet filler
 - Deionized water
1. Prepare a 5.00-mg/L nickel stock solution as follows:
 - a. Use a pipet to add 5.00 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 0.5 mg/L nickel standard solution as follows:
 - a. Use a pipet to add 10.00 mL of the 5.00-mg/L nickel stock solution into a 100-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 calibration 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 Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)
0.50 ± 0.02 mg/L Ni

Summary of method

After the sample is buffered and pyrophosphate is added to mask any Fe³⁺, the cobalt reacts with 1-(2-Pyridylazo)-2-Naphthol indicator. The indicator forms complexes with most metals. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt. This method is unique because both nickel and cobalt can be determined on the same sample. Single-wavelength instruments, such as the Pocket Colorimeter, cannot differentiate between nickel and cobalt.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Cobalt/Nickel Reagent Set, PAN, 10-mL, includes:	—	100/pkg	2651600
EDTA Reagent Powder Pillow	2	100/pkg	700599
Phthalate-Phosphate Reagent Powder Pillow, 10-mL	2	100/pkg	2615199
PAN Indicator Solution, 0.3%	1 mL	100 mL MDB	2150232
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606

Recommended standard

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

Optional reagents and apparatus

Description	Unit	Item no.
Flask, volumetric, Class A, 100-mL glass	each	1457442
Flask, volumetric, Class A, 1000-mL glass	each	1457453
Pipet, volumetric, Class A, 10-mL	each	1451538
Pipet filler, safety bulb	each	1465100
Nitric Acid, concentrated	500 mL	15249
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032



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