Nickel

DOC316.53.01063

1-(2 Pyridylazo)-2-Napthol (PAN) Method[1]

Method 8150

0.006 to 1.000 mg/L Ni

Powder Pillows

Scope and application: For water and wastewater.

[1] Adapted from Watanabe, H., Talanta, 21 295 (1974).

Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
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.
For spectrophotometers, this method can measure the cobalt concentration on the same sample with Program Number 110.
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
EDTA Powder Pillow	2
Phthalate-Phosphate Reagent Powder Pillow	2

Items to collect (continued)

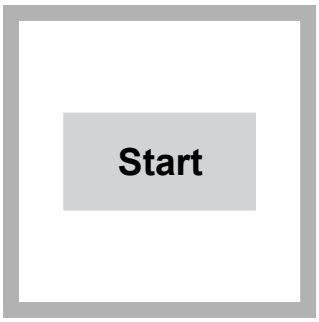
Description	Quantity
PAN Indicator Solution 0.3%	1 mL
Deionized water	25 mL
Stoppers	2
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 6 for order information.

Sample collection and storage

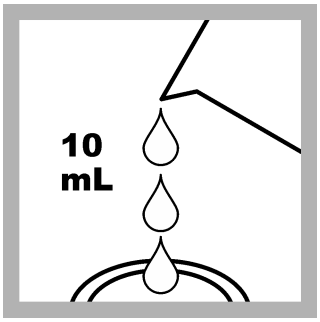
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- If the sample temperature is less than 10 °C (50 °F), warm the sample to room temperature before analysis.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (about 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 3–8 with 5.0 N sodium hydroxide standard solution. Do not exceed pH 8 to prevent precipitation of the metal.
- Correct the test result for the dilution caused by the volume additions.

Test procedure

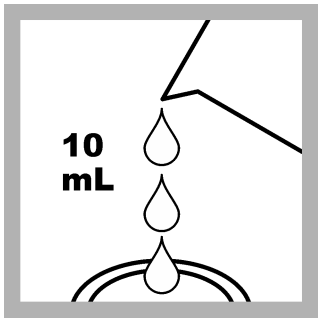


1. Start program **340 Nickel, PAN**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

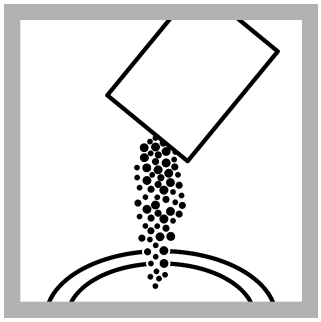
Note: *Although the program name can be different between instruments, the program number does not change.*



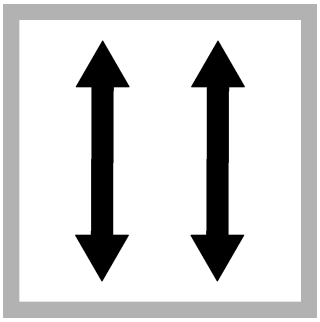
2. Prepare the blank: Fill a sample cell with 10 mL of deionized water.



3. Prepare the sample: Fill a second sample cell with 10 mL of sample.

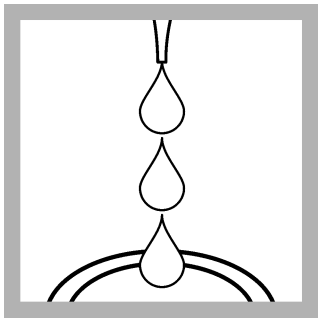


4. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cell.

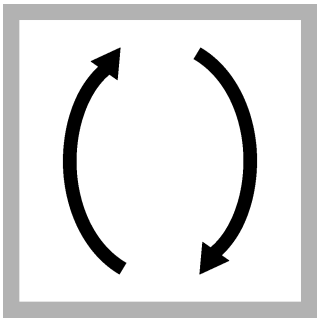


5. Close the sample cells. Immediately shake to dissolve the reagent.

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



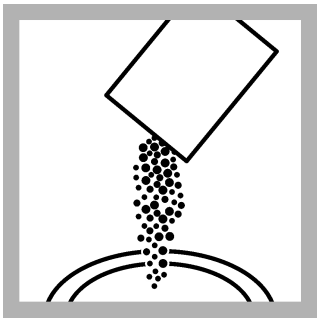
6. Add 0.5 mL of 0.3% PAN Indicator Solution to each cell.



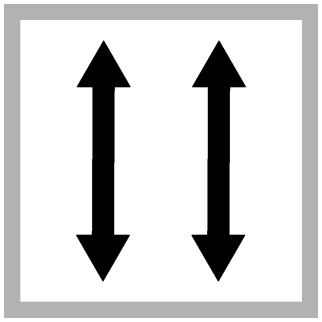
7. Put the stopper on the sample cells. Invert several times to mix.

8. Start the instrument timer. A 15-minute reaction time starts.

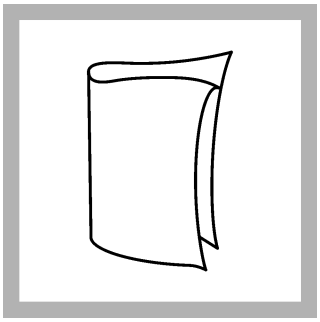
During color development the sample solution color may vary from yellow-orange to dark red, based on the chemical makeup of the sample. The blank will be yellow.



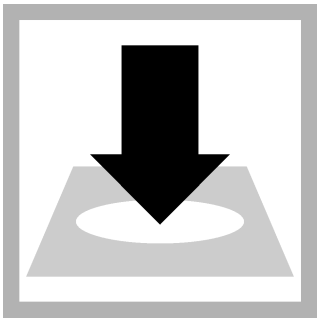
9. When the timer expires, add the contents of one EDTA Reagent Powder Pillow to each cell.



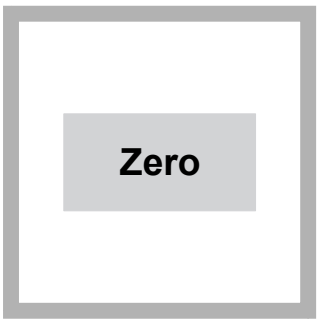
10. Close the sample cells. Shake to dissolve the reagent powder.



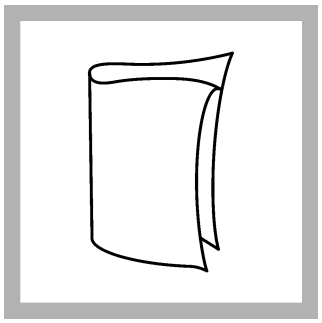
11. Clean the blank sample cell.



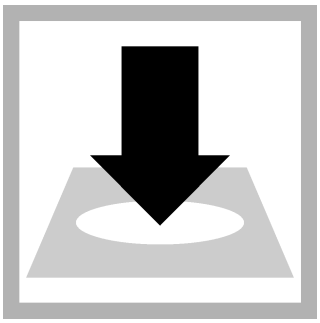
12. Insert the blank into the cell holder.



13. Push **ZERO**. The display shows 0.00 mg/L Ni and Co (spectrophotometers) or mg/L Ni (colorimeters) *.



14. Clean the prepared sample cell.



15. Insert the prepared sample into the cell holder.

16. Push **READ**. Results show in mg/L Ni and Co (spectrophotometers) or mg/L Ni (colorimeters).

* Spectrophotometers "zero" at 560 nm and 620 nm. Colorimeters "zero" at 560 nm.

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
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 additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- 1000-mg/L Nickel Standard Solution
- 100-mL volumetric flask, Class A
- 5-mL volumetric pipet, Class A and pipet filler
- Deionized water
- Pipet, TenSette®, 0.1–1.0 mL and tips
- Mixing cylinders, 25-mL (3)

1. Prepare a 50 mg/L nickel standard solution as follows:
   a. Use a pipet to add 5.00 mL of a 1000 mg/L nickel standard solution into a 100-mL volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
3. Go to the Standard Additions option in the instrument menu.
4. Select the values for standard concentration, sample volume and spike volumes.

5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the prepared standard solution, respectively, to three 25-mL portions of fresh sample. Mix well.
6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

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 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 spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
340	0.500 mg/L Ni	0.492–0.508 mg/L Ni	0.006 mg/L Ni

Summary of method

After pyrophosphate is added to buffer the sample and mask any Fe^{3+}, the nickel reacts 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. Spectrophotometers automatically adjust 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 with a spectrophotometer. The measurement wavelength is 560 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Cobalt/Nickel Reagent Set, PAN, 10-mL, includes:	—	100/pkg	2651600
Includes:			
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.
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

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

Optional reagents and apparatus

Description	Unit	Item no.
Mixing cylinder, graduated, 25-mL	each	189640
Flask, volumetric, Class A, 100-mL glass	each	1457442
Pipet, volumetric 5.00-mL	each	1451537
Pipet filler, safety bulb	each	1465100
Pipet, volumetric, Class A, 10-mL	each	1451538
Flask, volumetric, Class A, 1000-mL glass	each	1457453
Water, deionized	4 L	27256
Nitric Acid Solution, 1:1	500 mL	254049
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032

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01/2014, Edition 9