Cobalt

DOC316.53.01036

1-(2-Pyridylazo)-2-Napthol (PAN) Method[1] — Method 8078

0.01 to 2.00 mg/L Co — Powder Pillows

Scope and application: For water and wastewater. Digestion is required to determine total recoverable cobalt. If EDTA is in the sample, use the vigorous digestion.

[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 specific instruments.

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.	

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).
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 automatically with the reagent blank adjust option.
This method can measure the nickel concentration on the same sample with Program Number 340. Make sure to use a reagent blank for the nickel procedure.
The Pour-Thru Cell can only be used (for applicable instruments) with reagents for 25-mL samples.
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

Items to collect (continued)

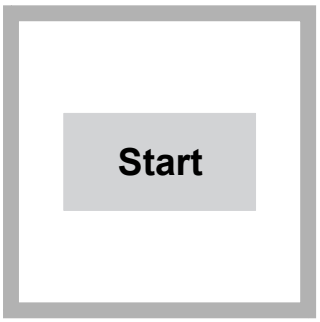
Description	Quantity
Stopper, Neoprene, solid, size #1	2
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 5 for order information.

Sample collection and storage

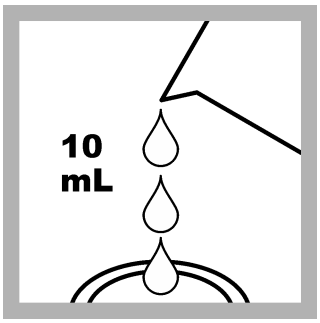
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is 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 3–8 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

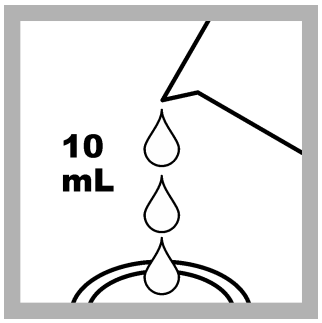


1. Start program **110 Cobalt**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

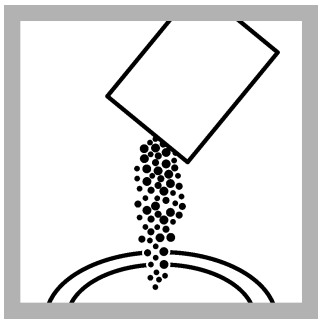
Note: *Although the program name can be different between instruments, the program number does not change.*



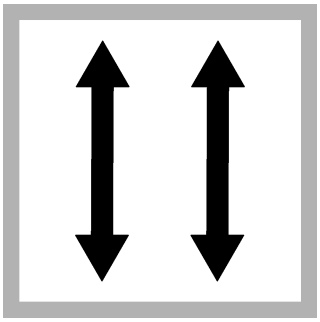
2. Prepare the blank: Fill a sample cell with 10 mL of deionized water.



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

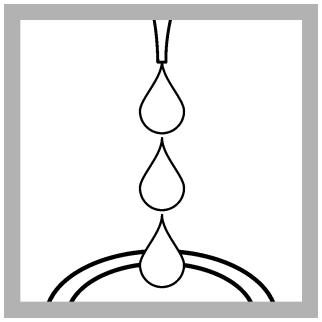


4. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cell.

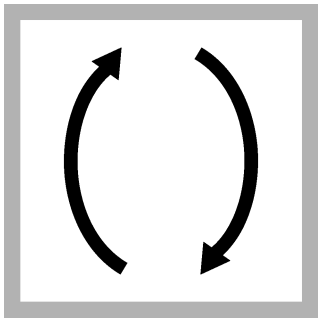


5. Close the sample cells. Immediately shake to dissolve the reagent.

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



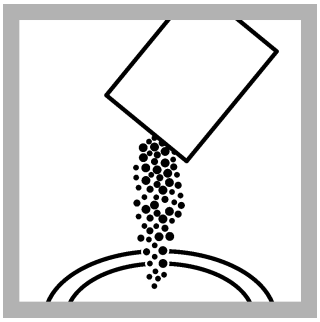
6. Add 0.5 mL of 0.3% PAN Indicator Solution to each cell.



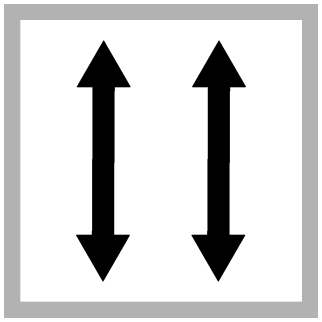
7. Put the stopper on the sample cells. Invert several times to mix.

8. Start the instrument timer. A 3-minute reaction time starts.

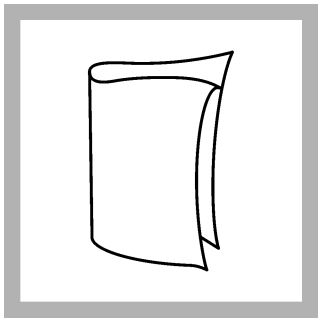
During color development the sample solution color can change from green to dark red, based on the chemical composition of the sample. The blank will be yellow.



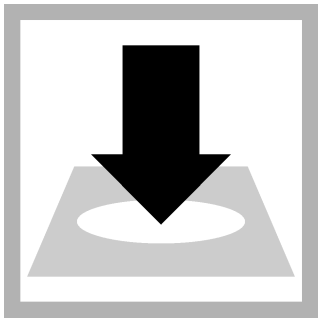
9. When the timer expires, add the contents of one EDTA Reagent Powder Pillow to each cell.



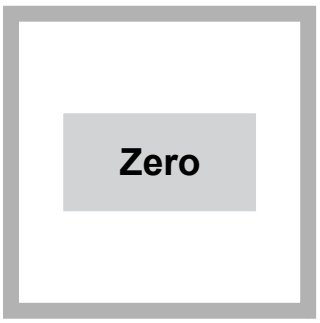
10. Close the sample cells. Shake to dissolve the reagent powder.



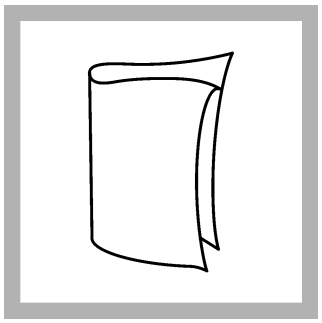
11. Clean the blank sample cell.



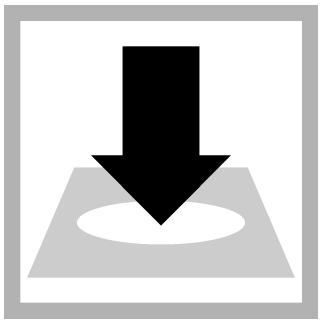
12. Insert the blank into the cell holder.



13. Push **ZERO**. The display shows 0.00 mg/L Co.



14. Clean the prepared sample cell.



15. Insert the prepared sample into the cell holder.

16. Push **READ**. Results show in mg/L Co.

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

Interfering substance	Interference level
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 solution method

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

Items to collect:

- 1000-mg/L Cobalt Standard Solution
- 1-L volumetric flask, Class A
- 100-mL volumetric flask, Class A
- 10-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 10.00-mg/L cobalt stock solution as follows:
   a. Use a pipet to add 10.00 mL of a 1000-mg/L cobalt 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 1.00-mg/L cobalt standard solution as follows:
   a. Use a pipet to add 10.00 mL of the 10.00-mg/L cobalt 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
110	1.00 mg/L Co	0.99–1.01 mg/L Co	0.01 mg/L Co

Summary of Method

After the sample is buffered and pyrophosphate is added to mask any Fe^{3+}, 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 which can be determined with the same sample preparation. The measurement wavelength is 620 nm.

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.
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

Description	Unit	Item no.
Cobalt Standard Solution, 1000-mg/L Co	100 mL	2150342

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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03/2014, Edition 8