Spectroquant®

Nickel Test



1. Method

Nickel(II) ions are oxidized by iodine and then transformed with dimethylglyoxime in an ammoniacal solution into a red-brown complex that is determined photometrically.

2. Measuring range and number of determinations

Cell mm	Measuring range mg/l Ni	Number of determinations
50	0.02 - 1.00	
20	0.05 - 2.50	250
10	0.10 - 5.00	

For programming data for selected photometers / spectrophotometers see www.service-test-kits.com.

3. Applications

This test measures only nickel(II) ions. Samples must be decomposed by digestion before undissolved or complex-bound nickel can be measured (see

Sample material:

Groundwater and surface water

Drinking water

Industrial water

Wastewater and percolating water

This test is **not suited** for seawater.

4. Influence of foreign substances

This was checked in solutions containing 1.5 and 0 mg/l Ni. The determination is not yet interfered with up to the concentrations of foreign substances given in the table.

Concentrations of foreign substances in mg/l or %							
Al ³⁺	1000	F-	1000	Pb ²⁺	1000	EDTA	1
Ca ²⁺	1000	Fe ³⁺	10	PO ₄ 3-	1000	Surfactants1)	5 %
Cd ²⁺	100	Hg ²⁺	100	S ²⁻	10	Na-acetate	10 %
CN-	10	Mg ²⁺	500	SO ₃ 2-	1000	NaCl	20 %
Cr ³⁺	1	Mn ²⁺	1	Zn ²⁺	1000	NaNO₃	20 %
Cr ₂ O ₇ ²⁻	10	NH ₄ ⁺	1000			Na ₂ SO ₄	20 %
Cu ²⁺	10	NO ₂ -	1000				

Reducing agents interfere with the determination.

5. Reagents and auxiliaries

Please note the warnings on the packaging materials!

The test reagents are stable up to the date stated on the pack when stored closed at +15 to +25 °C.

Package contents:

- 1 bottle of reagent Ni-1
- 1 bottle of reagent Ni-2
- 1 bottle of reagent Ni-3
- 1 AutoSelector

Other reagents and accessories:

Nitric acid 65 % for analysis EMSURE®, Cat. No. 100456

Spectroquant® Crack Set 10C, Cat. No. 114688

+ thermoreactor

Spectroquant® Crack Set 10, Cat. No. 114687

+ empty cells 16 mm with screw caps (25 pcs), Cat. No. 114724

+ thermoreactor

Charcoal activated GR for analysis, Cat. No. 102186

Ammonia solution 25 % for analysis EMSURE®, Cat. No. 105432 MQuant™ Nickel Test, Cat. No.110006,

measuring range 10 - 500 mg/l Ni²⁺
MColorpHast™ Universal indicator strips pH 0 - 14, Cat. No. 109535

MColorpHast™ pH-indicator strips pH 7.5 - 14, Cat. No. 109532

Sodium hydroxide solution 1 mol/l TitriPUR®, Cat. No. 109137 Sulfuric acid 0.5 mol/l TitriPUR®, Cat. No. 109072

Spectroquant® CombiCheck 40, Cat. No. 114692

Pipette for a pipetting volume of 5.0 ml

Rectangular cells 10, 20, and 50 mm (2 of each), Cat. Nos. 114946, 114947, and 114944

Semi-microcells 50 mm (2 pcs), Cat. No. 173502

6. Preparation

- Analyze immediately after sampling. Otherwise preserve with nitric acid 65 % (1 ml nitric acid per 1 l of sample solution).
- Undissolved or complex-bound nickel can be determined after pretreatment of the sample using one of the Spectroquant® Crack Sets.
- Decolorize any yellow stained samples by filtering through activated charcoal at pH 4. In the event that iron is the cause of the coloration, precipitate this with ammonia solution as iron hydroxide and separate from the solution.
- Check the nickel content with the MQuant™ Nickel Test. Samples containing more than 5.00 mg/l Ni must be diluted with distilled water prior to digestion.
- The pH must be within the range 3 8.

Adjust, if necessary, with sodium hydroxide solution or sulfuric acid.

• Filter turbid samples.

7. Procedure

Pretreated sample (10 - 40 °C)	5.0 ml	Pipette into a test tube.		
Reagent Ni-1	1 drop ¹⁾	Add and mix. A slight yellow coloration must persist. If necessary, add reagent Ni-1 dropwise until the color remains stable.		
Leave to stand for 1 min (reaction time A).				
Reagent Ni-2	2 drops ¹⁾	Add and mix. The pH must be within the range 10 - 12. Check with MColorpHast™ pH-indicator strips. Adjust the pH, if necessary, with sodium hydroxide solution or sulfuric acid.		
Reagent Ni-3	2 drops ¹⁾	Add and mix.		
Leave to stand for 2 min (reaction time B), then fill the sample into the cell, and measure in the photometer.				

¹⁾ Hold the bottle vertically while adding the reagent!

For measurement in the 50-mm cell both the sample volume as well as the quantities of reagents Ni-1, Ni-2, and Ni-3 must be doubled. Alternatively, the semi-microcell Cat. No. 173502 can be used.

Notes on the measurement:

- Certain photometers may require a blank (preparation as per measurement sample, but with distilled water instead of sample).
- For photometric measurement the cells must be clean. Wipe, if necessary, with a clean dry cloth.
- Measurement of turbid solutions yields false-high readings.
- The pH of the measurement solution must be within the range 9.5 11.0.
- The color of the measurement solution remains stable for 30 min after the end of the reaction time B stated above. (After 60 min the measurement value would have diminished by 5 %.)
- A red precipitate forms when nickel concentrations are too high.

8. Analytical quality assurance

recommended before each measurement series

To check the photometric measurement system (test reagents, measurement device, handling) and the mode of working, Spectroquant® CombiCheck 40 can be used. Besides a standard solution with 2.00 mg/l Ni²⁺, this article also contains an addition solution for determining sample-dependent interferences (matrix effects).

Additional notes see under www.qa-test-kits.com.

Characteristic quality data:

In the production control, the following data were determined in accordance with ISO 8466-1 and DIN 38402 A51 (10-mm cell):

Standard deviation of the method (mg/l Ni)	± 0.028
Coefficient of variation of the method (%)	±1.1
Confidence interval (mg/l Ni)	± 0.06
Number of lots	31

Characteristic data of the procedure:

		Measuring range mg/l Ni	
	0.02 - 1.00	0.10 - 5.00	
Sensitivity: Absorbance 0,010 A corresponds to (mg/l Ni)	0.01	0.05	
Accuracy of a measurement value (mg/l Ni)	max. ± 0.03	max. ± 0.16	

For quality and batch certificates for Spectroquant® test kits see the website.

- Reclose the reagent bottles immediately after use.
- Information on disposal can be obtained at www.disposal-test-kits.com.

Merck KGaA, 64271 Darmstadt, Germany, Tel. +49(0)6151 72-2440 www.analytical-test-kits.com EMD Millipore Corporation, 290 Concord Road, Billerica, MA 01821, USA, Tel. +1-978-715-4321

¹⁾ tested with nonionic, cationic, and anionic surfactants