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EZ2304 Total Nickel & Nickel Analyser

Method and reagent sheets

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1. Legal information

Manufacturer: AppliTek NV/SA

Distributor: Hach Lange GmbH

The translation of the manual is approved by the manufacturer.

2. Analytical specifications

Please refer also to the respective technical datasheet at Hach Support Online.

Nickel - All specifications			
Analysis method	Colorimetric measurement using DMG colour solution		
Parameter	Ni Total, Ni		
Cycle time	Standard measurement cycle time: 30 minutes Internal dilution: + 5 min. External dilution: + 5 – 10 min.		
Limit of detection (LOD)	≤ 10 µg/L		
Precision/Repeatability	Better than 2% full scale range for standard test solutions		
Cleaning	Automatic; frequency freely programmable		
Calibration	Automatic, 2-point; frequency freely programmable		
Validation	Automatic; frequency freely programmable		
Interferences	Metal ions like Aluminium (III) [(Al) ³⁺], Bismuth (II) [(Bi) ²⁺], Cadmium (II) [(Cd) ²⁺], Chromium (III) [(Cr) ³⁺], Cobalt (II) [(Co) ²⁺], Copper (II) [(Cu) ²⁺], Iron (II) [(Fe) ²⁺], Iron (III) [(Fe) ³⁺], Lead (II) [(Pb) ²⁺], Manganese (II) [(Mn) ²⁺], Magnesium (II) [(Mg) ²⁺], Mercury (II) [(Hg) ²⁺], Palladium (II) [(Pd) ²⁺], Platinum (II) [(Pt) ²⁺], Silver (II) [(Ag) ⁺], Tin (II) [(Sn) ²⁺] & Zinc (II) [(Zn) ²⁺]. Large amounts of color and turbidity interferes. Fats, oil, proteins, surfactants and tar.		
Measuring ranges	% of range - Dilution	Low range (µg/L)	High range (µg/L)
	B 25% of standard range	10	125
	C 50% of standard range	10	250
	0 standard range	20	500
	1 internal MP dilution (factor 4)	160	2000
3 internal MP dilution (factor 10)	320	5000	

3. Analysis method

Summary

The determination Nickel and total Nickel is based on two methods, combined in one analyser.

The Nickel concentration is determined in the 'Ni' method. The total Nickel concentration is determined in the 'Total Ni' method. The concentration of all parameters is determined alternately in the 'Main'-method.

The calibration for Nickel is performed in the 'Ni' method. The calibration for total Nickel is performed in the 'Total Ni' method.

Remark

The methods cannot be started at the same time.

3.1 Nickel

Summary

Nickel reacts with dimethylglyoxime (DMG) in the presence of an oxidizing agent in an alkaline medium to form an orange-brown coloured complex. The absorption is measured at a wavelength of 450 nm.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling and addition of the buffer and oxidizing reagent the initial absorbance value is measured at 450 nm. Next, the colour solution is added and after respecting a stirring period – performed to obtain complete colour development – the final absorbance value is determined. With the obtained absorbance values, the nickel concentration can be calculated according to Beer's law

Calibration

The calibration procedure measures a REF1 Ni solution (channel 9, REF1 valve) and a REF2 Ni solution (channel 10, REF2 valve) to adapt the slope and offset factors by means of a two point calibration.

3.2 Total Nickel

Summary

The nickel ions react with dimethylglyoxime (DMG) in an alkaline medium and in the presence of an oxidizing agent to form an orange-brown coloured complex. The absorption is measured at a wavelength of 450 nm. Prior to the total nickel analysis, the sample is digested by use of an acid solution.


Analysis steps


The sample is mixed with the acid solution and heated to 120 °C (or up to 150 °C – programmable) in an oven during several minutes (standard 10 minutes; programmable up to 60 minutes). After digestion, the sample is cooled and transferred into the analysis vessel. The buffer solution and oxidizing agent are added to the sample. After respecting a stirring period - the initial absorbance value is measured. Next, the colour solution is added. The final absorbance value is determined. With the obtained absorbance values, the nickel concentration can be calculated according to Beer's law.

Calibration

The calibration procedure measures a REF1 Ni solution (channel 9, REF1 valve) and a REF2 Ni solution (channel 10, REF2 valve) to adapt the slope and offset factors by means of a two point calibration.

4. Reagents

⚠ CAUTION	
	Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Read the safety data sheet from the supplier before bottles are filled or reagents are prepared. For laboratory use only. Make the hazard information known in accordance with the local regulations of the user.

⚠ CAUTION	
	Chemical exposure hazard. Dispose of chemicals and wastes in accordance with local, regional and national regulations.

4.1 Reagent overview and consumption

In the tables below, the products that are needed to prepare the reagents are listed. The product name, the formula, the molecular weight, the CAS No. and the amount needed to prepare 1 litre of the reagents is given. Check the consumption of the reagents (28 days) to adapt the volumes needed.

Product	Consumption	Consumption/28 days A rata 1 analysis/40 min	Recommended containers
Acid solution (Total Ni)	~ 1.0 mL / analysis	~ 1.4L	Plastic – 2.5 L
Buffer solution (Total Ni)	~ 2.0 mL / analysis	~ 4.0 L	Plastic – 5.0 L
Buffer solution (Ni)	~ 2.0 mL / analysis		
Colour solution (Total Ni)	~ 0.5 mL / analysis	~ 1.0 L	Plastic – 2.5 L
Colour solution (Ni)	~ 0.5 mL / analysis		
Oxidizing agent (Total Ni)	~ 1.0 mL / analysis	~ 2.0 L	Plastic – 2.5 L
Oxidizing agent (Ni)	~ 1.0 mL / analysis		
REF1 solution (Total Ni + Ni)	~ 1 L / calibration	/	Plastic – 1 L
REF2 solution (Total Ni + Ni)	~ 1 L / calibration	/	Plastic – 1 L

4.2 DI-water overview and consumption

	Rinse water (mL/analysis) Type I		Dilution water (mL/analysis) Type I		Total (mL/analysis)	Consumption/28 days A rata 1 analysis/30 min
	Total Ni	Ni (II)	Total Ni	Ni (II)	Total Ni + Ni (II)	Total Ni + Ni (II)
B	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
C	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
0	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
1	55 mL	55 mL	20 mL	10 mL	140 mL	190 L
3	55 mL	55 mL	20 mL	10 mL	140 mL	190 L

Remark

The indicated volumes are an estimation of the consumption for rinse and dilution water, based on a standard operating procedure, as defined in the specifications of the EZ analyser. Please be aware that, depending on the sample matrix, the rinse water volumes might increase.

4.3 Storage and quality of chemicals

Quality of chemicals

All chemicals should be of Reagent grade, ACS grade or better (*). The use of pro analysis chemicals is recommended. Poor quality of the reagents can affect the analyser performance.

(* Analytical Reagent (AR), Guaranteed Reagent (GR), UNIVAR, AnalaR, Premium Reagent (PR), ReagentCertified ACS reagent, ACS Plus reagent, puriss p.a. ACS reagent, ReagentPlus®, TraceCERT®, Suprapur®, Ultrapur®, or better are also possible.

Quality of DI-water

All EZ analysers are tested with standard solutions, reagents and dilution water prepared using type I water or better as defined by ASTM D1193-91.

To achieve the specifications as stated on the data sheet, method and reagents sheet and acceptance test reports, the same water quality (or better) must be used for the preparation of the standard solutions, reagents and dilution water.

Additionally the water used for the preparation of the standard solutions for an EZ analyser must be free of the parameter or any of the interferences for the method of that EZ analyser.

Storage of Reagents

While operating the instrument, keep in mind the reagent requirements as stated in the reagent overview, the chapters below and/or in the data sheet of the instrument.

CAUTION



For longer-term storage: Store the reagents cold; Store the reagents in the dark;
If applicable: Store the reagents in a fridge during operation

CAUTION



Refresh the reagents after one month (unless stated differently in the chapters below).
Do not mix old reagents with freshly prepared reagents. Remove old reagents from the container before adding freshly prepared reagents.

4.4 Acid solution (1M)

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Nitric acid (65%)	HNO ₃	63.01	7697-37-2	69 mL

Preparation

Dilute 69 mL nitric acid (HNO₃, 65%) in 500 mL de-ionized water. Fill up to 1 litre with de-ionized water.

4.5 Buffer solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Citric acid monohydrate	C ₆ H ₈ O ₇ * H ₂ O	210.14	5949-29-1	54.5 g
Ammonium hydroxide solution (25%)*	NH ₄ OH	35.05	1336-21-6	300 mL

* Density: 0.91 g/ml (20°C)

Preparation

Dissolve 54.5 g citric acid monohydrate (C₆H₈O₇ * H₂O) in 500 mL de-ionized water. Next, add carefully 300 mL ammonium hydroxide solution (NH₄OH, 25%). Cool down and fill up to 1 litre with de-ionized water.

4.6 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Dimethylglyoxime disodium salt octahydrate	C ₄ H ₆ N ₂ Na ₂ O ₂ * 8H ₂ O	304.20	75006-64-3	33 g

Preparation

Dissolve 33 g of dimethylglyoxime disodium salt octahydrate (C₄H₆N₂Na₂O₂ * 8H₂O) in 500 mL de-ionized water. Mix and fill up to 1 litre with de-ionized water.

4.7 Oxidizing reagent

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium peroxodisulfate	Na ₂ S ₂ O ₈	238.10	7775-27-1	100 g

Preparation

Dissolve 100 g of sodium peroxodisulfate (Na₂S₂O₈) in 500 mL de-ionized water. Mix and fill up to 1 litre with de-ionized water.

4.8 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Nickel(II)chloride hexahydrate	NiCl ₂ * 6H ₂ O	237.69	7791-20-0	4.049 g
Nitric acid (65%)	HNO ₃	63.01	7697-37-2	/

Preparation

1000 mg/L Ni stock solution

Prepare a stock solution of 1000 mg/L Ni: Dissolve accurately 4.049 g nickel(II) chloride hexahydrate (NiCl₂ * 6H₂O) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Adjust the pH to 4 with nitric acid (HNO₃, 65%). Add de-ionized water up to the mark grade.

Ni standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 1000 mg/L Ni stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

	Measuring range	Concentration REF2	Amount of stock solution to add to 1 litre
B	125 µg/L Ni	125 µg/L Ni	0.125 mL
C	250 µg/L Ni	250 µg/L Ni	0.25 mL
0	500 µg/L Ni	500 µg/L Ni	0.50 mL
1	2000 µg/L Ni	2000 µg/L Ni	2.0 mL
3	5000 µg/L Ni	5000 µg/L Ni	5.0 mL

Ni standard solution – REF1

Prepare a standard solution of 0 µg/L Ni. Use de-ionized water.

4.9 Cleaning solution (facultative)

The cleaning procedure should prevent any build-up of chemicals in the analyser. To obtain an effective cleaning procedure one has to test the cleaning solution and the cleaning interval for each application. Perform the selected cleaning solution and interval for a trial period, check then the effectiveness of the procedure and change if necessary.

Change Information	
Date: 25/05/2022	Previous version: Edition 1.01 to Edition 1.02
Reason for Change	
- Correction of CAS number of Nitric Acid	
Description of Change	
- Correction of CAS number of Nitric Acid from 7697-32-2 to 7697-37-2 (chapter 4.4 and 4.8)	