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## **EZ2004 Total 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.

Total Nickel - All specifications				
<b>Analysis method</b>	Colorimetric measurement using DMG colour solution			
<b>Parameter</b>	Ni Total			
<b>Cycle time</b>	Standard measurement cycle time: 20 minutes Internal dilution: + 5 min. External dilution: + 5 – 10 min.			
<b>Limit of detection (LOD)</b>	≤ 10 µg/L			
<b>Precision</b>	Better than 2% full scale range for standard test solutions			
<b>Cleaning</b>	Automatic; frequency freely programmable			
<b>Calibration</b>	Automatic, 2-point; frequency freely programmable			
<b>Validation</b>	Automatic; frequency freely programmable			
<b>Interferences</b>	Metal ions like Aluminium (III) [(Al) <sup>3+</sup> ], Bismuth (II) [(Bi) <sup>2+</sup> ], Cadmium (II) [(Cd) <sup>2+</sup> ], Chromium (III) [(Cr) <sup>3+</sup> ], Cobalt (II) [(Co) <sup>2+</sup> ], Copper (II) [(Cu) <sup>2+</sup> ], Iron (II) [(Fe) <sup>2+</sup> ], Iron (III) [(Fe) <sup>3+</sup> ], Lead (II) [(Pb) <sup>2+</sup> ], Manganese (II) [(Mn) <sup>2+</sup> ], Magnesium (II) [(Mg) <sup>2+</sup> ], Mercury (II) [(Hg) <sup>2+</sup> ], Palladium (II) [(Pd) <sup>2+</sup> ], Platinum (II) [(Pt) <sup>2+</sup> ], Silver (II) [(Ag) <sup>+</sup> ], Tin (II) [(Sn) <sup>2+</sup> ] & Zinc (II) [(Zn) <sup>2+</sup> ].			
<b>Measuring ranges</b>	<b>% of range - Dilution</b>		<b>Low range (µg/L)</b>	<b>High range (µg/L)</b>
	B	25% of standard range	10	125
	C	50% of standard range	10	250
	<b>0</b>	<b>standard range</b>	<b>20</b>	<b>500</b>
	1	internal MP dilution (factor 4)	160	2000
	3	internal MP dilution (factor 10)	320	5000
	4	internal MP dilution (factor 20)	1000	10000

### 3. Analysis method

#### 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.


The calibration is performed in the MAIN method.

#### Remark

The methods cannot be started at the same time.

## 4. Reagents

<b>⚠ CAUTION</b>	
	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.

<b>⚠ CAUTION</b>	
	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 liter 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/20 min	Recommended containers
Acid solution	~ 1.0 mL / analysis	~ 2.0 L	Plastic – 2.5 L
Buffer solution	~ 2.0 mL / analysis	~ 4.0 L	Plastic – 5.0 L
Colour solution	~ 0.5 mL / analysis	~ 1.0 L	Plastic – 2.5 L
Oxidizing agent	~ 1.0 mL / analysis	~ 2.0 L	Plastic – 2.5 L
REF1 solution	~ 0.5 L / calibration	/	Plastic – 1 L
REF2 solution	~ 0.5 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/20 min
B	N.A.	N.A.	N.A.	N.A.
C	N.A.	N.A.	N.A.	N.A.
0	N.A.	N.A.	N.A.	N.A.
1	55 mL	20 mL	75 mL	152 L
3	55 mL	20 mL	75 mL	152 L
4	55 mL	20 mL	75 mL	152 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 <sub>3</sub>	63.01	7697-37-2	69 mL

##### Preparation

Dilute 69 mL nitric acid (HNO<sub>3</sub>, 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 <sub>6</sub> H <sub>8</sub> O <sub>7</sub> * H <sub>2</sub> O	210.14	5949-29-1	54.5 g
Ammonium hydroxide solution (25%)*	NH <sub>4</sub> OH	35.05	1336-21-6	300 mL

\* Density: 0.91 g/ml (20°C)

##### Preparation

Dissolve 54.5 g citric acid monohydrate (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub> \* H<sub>2</sub>O) in 500 mL de-ionized water. Next, add carefully 300 mL ammonium hydroxide solution (NH<sub>4</sub>OH, 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 <sub>4</sub> H <sub>6</sub> N <sub>2</sub> Na <sub>2</sub> O <sub>2</sub> * 8H <sub>2</sub> O	304.20	75006-64-3	33 g

##### Preparation

Dissolve 33 g of dimethylglyoxime disodium salt octahydrate (C<sub>4</sub>H<sub>6</sub>N<sub>2</sub>Na<sub>2</sub>O<sub>2</sub> \* 8H<sub>2</sub>O) 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 <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	238.10	7775-27-1	100 g

### Preparation

Dissolve 100 g of sodium peroxodisulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub>) 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 <sub>2</sub> * 6H <sub>2</sub> O	237.69	7791-20-0	4.049 g
Nitric acid (65%)	HNO <sub>3</sub>	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 (NiCl<sub>2</sub> \* 6H<sub>2</sub>O) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Adjust the pH to 4 with nitric acid (HNO<sub>3</sub>, 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
<b>0</b>	<b>500 µg/L Ni</b>	<b>500 µg/L Ni</b>	<b>0.50 mL</b>
1	2000 µg/L Ni	2000 µg/L Ni	2.0 mL
3	5000 µg/L Ni	5000 µg/L Ni	5.0 mL
4	10000 µg/L Ni	10000 µg/L Ni	10 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.

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