EZ2729sc Nitrogen, Total Analyzer

DOC312.53.94565

2.5 to 100 mg/L Total Nitrogen

Method

EZ2729sc

Scope and application: For industrial and municipal water.

Test preparation

Before starting

Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Refer to the current safety data sheets (MSDS/SDS) for safety protocols.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment. Dispose of chemicals and wastes in accordance with local, regional and national regulations.
Review the Safety Data Sheets (MSDS/SDS) before the bottles are filled or the reagents are prepared.
All chemicals must be of reagent grade, ACS grade or better[1]. The use of pro-analysis chemicals is recommended. Use of reagents that are not of sufficient quality can have a negative effect on the analyzer performance.
All EZ analyzers are put through long tests with standard solutions, reagents and dilution water prepared with Type I water or better water as specified in ASTM D1193-91. To get the specifications shown on the data sheet, method and reagents sheet and acceptance test reports, the same water quality (or better) must be used to prepare the standard solutions, reagents and dilution water. In addition, prepare the standard solutions for an EZ analyzer with water that does not contain the parameter to be measured or interferences for the method.
When operating the device, always make sure to follow the reagent recommendations given in Reagent consumption on page 2.
For longer-term storage, keep the reagents in a cold and dark place. Do not keep reagents longer than recommended. If applicable, keep the reagents in a refrigerator during measurements. Refer to Reagent consumption on page 2 for the reagent temperature.
The manufacturer recommends to replace the reagents, stock and standard solution at 28-day intervals unless specified differently in the sections that follow. Do not mix used reagents with freshly prepared reagents. If reagents, standards or DI water in the containers are replaced, discard all of the container contents in accordance with local, regional and national regulations. Rinse out all of the containers and then fill each container with freshly prepared new reagent.

Specifications

Specifications are subject to change without notice.

Specification	Details
Analysis method	NEDD colorimetric method after reduction with hydrazine
Measurement wavelength	546 nm
Parameter	Long description: Nitrogen, Total Short description (default): Total N Options: None
Unit	mg/L (default); ppm, ppb, µg/L
Precision	The precision value is found on the full-scale range for standard test solutions. Refer to Table 1.
Cleaning	Automatic or manual; frequency is freely programmable

[1] 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.

Specification	Details
Calibration	Automatic or manual; 2-point, offset or slope; frequency is freely programmable ***Note:*** *The manufacturer recommends that a calibration is done when the reagents are replaced.*
Validation	Automatic or manual; frequency is freely programmable
Grab	Manual
Interferences	Ions, e.g., antimony (III) (Sb^{3+}), bismuth (III) (Bi^{3+}), chloroplatinate ($PtCl_6^{2-}$), gold (III) (Au^{3+}), iron (III) (Fe^{3+}), lead (II) (Pb^{2+}), mercury (II) (Hg^{2+}), metavanadate (VO_3^-) and silver (I) (Ag^+) can cause precipitation with nitrate. Copper (Cu^{2+}) ions can decompose the diazonium salt and cause a low result. NCl_3 can cause a red color. Strong oxidizing agents interfere. Large quantities of color and turbidity interfere. Fats, oil, proteins, surfactants and tar interfere.

Table 1 Measuring ranges

Range code	Description	LOD (mg/L)	Range (mg/L)	Precision (%)	Cycle time (minutes)	
					Continuous	Default
V	internal dispenser dilution (factor 5)	0.25	10	4	36	45
W	internal dispenser dilution (factor 10)	0.5	20	4	36	45
X	internal dispenser dilution (factor 25)	1.25	50	4	36	45
Y	internal dispenser dilution (factor 50)	2.5	100	4	36	45

Summary of method

Summary

Nitrate in the water sample is reduced to nitrite by a reducing agent. The nitrite reacts with the color reagent in an acidic solution to form a violet color. The absorbance is measured at a wavelength of 546 nm. The sample is first digested with heat and persulfate to make sure that all forms of nitrogen are measured.

Analysis steps

The analyzer mixes the sample with persulfate and NaOH in the digester vessel and heats the solution to 130 °C (266 °F) for 10 minutes (default digestion temperature and time). The undissolved and complexed forms of nitrogen break apart into a reactive form. After digestion, the sample temperature is decreased until sufficiently cool.

The digested sample is then moved into the analysis vessel. The copper, buffer and reducing agent reagents are added and the initial absorbance value is measured. The color reagent is then added and a stir period starts.

After the stir period, the color is fully developed and the final absorbance value is measured. The analyzer uses the absorbance values and Beer's Law to calculate the concentration of total nitrogen in the sample.

Calibration

The calibration procedure measures the REF1 solution (Channel 9, REF1 valve) and the REF2 solution (Channel 10, REF2 valve).

Validation

The validation procedure measures the REF2 solution (Channel 10, REF2 valve).

Reagent consumption

Table 2, Table 3 and Table 4 show the consumption rate of the reagents and calibration standards. Examine the consumption of the reagents after 28 days to adjust the

quantities prepared. Refer to Necessary reagents on page 4 to collect the necessary items to prepare the reagents.

Table 2 Reagent consumption

Product information			Consumption		Recommendation		
Code	**Label**	**Product**	**Each analysis**	**Per 28 days, rate of 1 analysis/45 minutes**	**Use life**	**Containers**	**Operation temperature**
Red	Reagent 1	Copper	~ 0.6 mL	~ 0.6 L	28 days	Plastic; 2.5 L	10 to 30 °C (50 to 86 °F)
Blue	Reagent 2	Buffer	~ 1.0 mL	~ 0.9 L	28 days	Plastic; 2.5 L	10 to 30 °C (50 to 86 °F)
Green	Reagent 3	Reducing agent	~ 1.0 mL	~ 0.9 L	28 days	Plastic; 5 L	Refrigerated 10 to 15 °C (50 to 59 °F)
Yellow	Reagent 4	Color	~ 1.0 mL	~ 0.9 L	28 days	Glass amber; 2.5 L	Refrigerated 10 to 15 °C (50 to 59 °F)
White	Reagent 5	Persulfate	~ 0.5 mL	~ 0.5 L	28 days	Plastic - 2.5 L	10 to 30 °C (50 to 86 °F)

Table 3 Calibration standards

Product information		Consumption	Recommendation	
Label	**Product**	**Per calibration**	**Use life**	**Containers**
REF1	REF1 standard	~ 0.2 L	28 days	Plastic, 1 L (align with recommendation)
REF2	REF2 standard	~ 0.2 L	28 days	Plastic, 1 L (align with recommendation)

Table 4 Calibration recommendations

Calibration	Time (minutes)		Recommended frequency	Solutions
	No dilution	**With dilution**		
Offset	—	110	Daily	REF1
2-point (recommended)	—	215	Reagent replacement (28 days)	REF1 and REF2

DI water consumption

The volumes shown in Table 5 are an estimation of the consumption for rinse and dilution water based on a standard operating procedure as given in the specifications of the EZ analyzer.

Note: *Rinse water volumes can increase because of the sample matrix.*

Table 5 DI water consumption

Range code	Rinse water Type I (mL/analysis)	Dilution water Type I (mL/analysis)	REF1 Type I (mL/analysis)	Total (mL/analysis)	Per 28 days, rate of 1 analysis each 45 minutes
V - W - X - Y	156 mL	27.5 mL	8 mL	191.5 mL	172 L

Rinse water

If the analyzer does a dilution, a deionized water rinse must be used. If no dilution is done, use the sample to rinse. If there is a filter panel in front of the analyzer, make sure that the rinse water also flows through the filter.

Necessary reagents

The full list of reagents is shown in Table 6. The product name, formula, molecular weight, CAS number and the necessary quantity to prepare 1 L of the reagents are given.

Table 6 Reagent list

Solutions	Products	Formula	MW (g/mol)	CAS number	For each 1 L solution
Reagent 1: Copper Code: Red	Hydrochloric acid (36%)	HCl	36.46	7647-01-0	Refer to Reagent 1: Copper on page 4.
	Copper(II) sulfate pentahydrate	$CuSO_4 * 5H_2O$	249.69	7758-99-8	Refer to Reagent 1: Copper on page 4.
Reagent 2: Buffer Code: Blue	Sodium hydroxide	NaOH	40.00	1310-73-2	20 g
Reagent 3: Reducing agent Code: Green	Hydrazine sulfate	$N_2H_4 * H_2SO_4$	130.12	10034-93-2	1.5 g
Reagent 4: Color Code: Yellow	Phosphoric acid 85%	H_3PO_4	98.00	7664-38-2	100 mL
	Sulfanilamide	$H_2NC_6H_4SO_2NH_2$	172.20	63-74-1	10 g
	N-(1-Naphthyl) ethylenediamine dihydrochloride	$C_{12}H_{16}Cl_2N_2$	259.17	1465-25-4	0.5 g
Reagent 5: Persulfate Code: White	Sodium peroxodisulfate[2]	$Na_2S_2O_8$	238.11	7775-27-1	40 g
Stock solution	Sodium nitrate	$NaNO_3$	84.99	7631-99-4	60.68 g
REF1 calibration standard	Deionized water (Type I or better)	—	—	—	—
REF2 calibration standard	10000 mg/L NO_3-N stock solution	—	—	—	Refer to Table 7 on page 6.
Validation standard (optional)	REF2 calibration standard	—	—	—	Refer to Validation standard on page 6.
Cleaning solution (optional)	Hydrochloric acid (36%)	HCl	36.46	7647-01-0	41.5 mL

Reagent preparation

Prepare the reagents as follows. Refer to Table 6 on page 4 to collect the applicable items. To calculate the correct reagent quantity, refer to Reagent consumption on page 2. Make sure to discard the remaining solution from the analyzer bottles before new reagents are added.

Reagent 1: Copper

1. Add 50 mL of deionized water to a beaker.
2. Slowly mix in 4.15 mL of concentrated hydrochloric acid (HCl, 36%).
3. Add 0.5 g of copper sulfate pentahydrate ($CuSO_4 * 5H_2O$).
4. Mix until fully dissolved.
5. Pour the solution into a 100-mL volumetric flask.
6. Add deionized water to the mark.
7. Fully mix the solution.

[2] Hach BCZ822 recommended

8. Pipet 4 mL of the 0.5 g/100 mL copper sulfate solution into a 1000-mL volumetric flask.
9. Add deionized water to the mark.
10. Fully mix the solution.
11. Discard the remaining 0.5 g/100 mL copper sulfate solution.

    Note: *The 0.5 g/100 mL copper sulfate solution has a short shelf life and must be prepared each time the Reagent 1: Copper is prepared.*

Reagent 2: Buffer

1. Add 500 mL of deionized water to a beaker.
2. Slowly mix in 20 g of sodium hydroxide (NaOH).
3. Pour the solution into a 1000-mL volumetric flask.
4. Add deionized water to the mark.
5. Fully mix the solution.

Reagent 3: Reducing agent

1. Add 500 mL of deionized water to a beaker.
2. Add 1.5 g of hydrazine sulfate ($N_2H_4.H_2SO_4$).
3. Mix until fully dissolved.
4. Pour the solution into a 1000-mL volumetric flask.
5. Add deionized water to the mark.
6. Fully mix the solution.

Reagent 4: Color

1. Add 500 mL of deionized water to a beaker.
2. Slowly mix in 100 mL of phosphoric acid (H_3PO_4, 85%).
3. Add 10 g of sulfanilamide ($H_2NC_6H_4SO_2NH_2$).
4. Mix until fully dissolved.
5. Add 0.5 g of N-(1-naphthyl) ethylenediamine dihydrochloride ($C_{12}H_{16}Cl_2N_2$).
6. Mix until fully dissolved.
7. Pour the solution into a 1000-mL volumetric flask.
8. Add deionized water to the mark.
9. Fully mix the solution.

   Make sure that the solution has no color. Prepare a new color reagent if the solution color becomes pink/brown,

Reagent 5: Persulfate

1. Add 500 mL of deionized water to a beaker.
2. Add 40 g of sodium peroxodisulfate ($Na_2S_2O_8$).
3. Mix until fully dissolved.
4. Pour the solution into a 1000-mL volumetric flask.
5. Add deionized water to the mark.
6. Fully mix the solution.

Calibration standards

Calibrations are completed with two standards: a REF1 calibration standard and a REF2 calibration standard. The REF2 calibration standard is a dilution of a stock solution.

Stock solution

Prepare a 10000-mg/L NO_3-N stock solution as follows. Refer to Necessary reagents on page 4 to collect the applicable items.

1. Add 500 mL of deionized water to a beaker.
2. Add 60.68 g of sodium nitrate ($NaNO_3$).
3. Mix until fully dissolved.
4. Pour the solution into a 1000-mL volumetric flask.
5. Add deionized water to the mark.
6. Fully mix the solution.

REF1 calibration standard

Use deionized water for the REF1 calibration standard.

REF2 calibration standard

Dilute the stock solution to prepare the REF2 calibration standard.

1. Use a pipet to add the applicable quantity (mL) of the stock solution into a 1000-mL volumetric flask. Refer to Table 7.
2. Add deionized water to the mark.
3. Fully mix the solution.

Table 7 Calibration standard preparation

Range code	REF2 concentration (mg/L NO_3—N)	Quantity (mL) of stock solution
V	10	1
W	25	2.5
X	50	5
Y	100	10

Validation standard

By default, the automatic validation procedure is not enabled. When enabled, the default validation standard is the REF2 calibration standard. For best results, do not use the same solution that was used for calibration. Use a different standard solution from a different source for the validation standard. The concentration of the validation standard must be within the measuring range of the analyzer.

Before validation, connect the REF2 sample line to the validation standard. After validation, connect the REF2 sample line to the REF2 calibration standard again. For multi-channel setups, a different channel can be used.

Cleaning solution

By default, the automatic cleaning procedure is not enabled. When enabled, the default volume of cleaning solution that is used during each cleaning cycle is 30 mL.

The cleaning procedure must prevent the collection of chemicals in the analyzer. For an accurate cleaning procedure, examine the cleaning solution and the cleaning interval for each application. Make sure that the cleaning procedure is sufficient. Change the cleaning procedure if necessary.

The manufacturer recommends to use a 0.5 M hydrochloric acid (HCl) solution. Refer to Necessary reagents on page 4. Prepare the solution as given in the steps that follow or use a commercially available solution.

1. Add 500 mL of deionized water to a beaker.
2. Slowly mix in 41.5 mL of concentrated hydrochloric acid (HCl, 36%).
3. Pour the solution into a 1000-mL volumetric flask.

4. Add deionized water to the mark.
5. Fully mix the solution.

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