DOC312.53.94555

EZ1102 Ammonia Analyser

Method and reagent sheets

02/2023, Edition 1.01

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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.

Ammonia - All specifications				
Analysis method	Colorimetric measurement at 630 nm Adapted method, based on standard method APHA4500-NH3 F			
Parameter	NH_4-N			
Cycle time	30 minutes Internal dilution: + 5 min. External dilution: + 10 min.			
Limit of detection (LOD)	≤ 5 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	Amino acids, hydrazine [H_4N_2] and urea [H_2NCONH_2]. Large amounts of color and turbidity interfere. Fats, oil, proteins, surfactants and tar. Possible negative interference on Alkalinity > 800 mg/L (as $CaCO_3$, Mg > 10 mg/L			
Measuring ranges	**% of range - Dilution**		**Low range (mg/L)**	**High range (mg/L)**
	A	10% of standard range	0.005	0.1
	B	25% of standard range	0.010	0.25
	C	50% of standard range	0.010	0.5
	0	standard range	**0.025**	1
	1	internal MP dilution (factor 4)	0.20	4
	2	Internal MP dilution (factor 8)	0.40	8
	W	internal dispenser dilution (factor 10)	0.25	10
	X	internal dispenser dilution (factor 25)	1.00	25
	Y	internal dispenser dilution (factor 50)	1.25	50
	Z	internal dispenser dilution (factor 75)	2.00	75
	5	internal dispenser dilution (factor 100)	2.50	100

3. Analysis method

Summary

The determination of the ammonia concentration in water is based on the reaction of ammonium ions with hypochlorite and a thymol component in a strong alkaline medium (pH>10) to form a green complex. The absorption is measured at 630 nm. Sodium nitroprusside is added as a catalyst to accelerate the reaction. Sodium tartrate is added to prevent metal-hydroxide precipitation.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling, the initial absorbance value is measured at 630 nm. Next, reagent B, reagent A and reagent C are 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 ammonia concentration can be calculated according to Beer's Law.

Calibration

The calibration procedure measures a REF1 N-NH_4 solution (channel 9, REF1 valve) and a REF2 N-NH_4 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

⚠ 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 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/30 min	Recommended containers
Buffer	~ 0.5 mL / analysis	~ 0.725 L	Glass – 2.5 L
Chlorine	~ 0.5 mL / analysis	~ 0.725 L	Glass – 2.5 L
Color	~ 0.5 mL / analysis	~ 0.725 L	Glass – 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 / 30 min
A	N.A.	N.A.	N.A.	N.A.
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	60 mL	15 mL	75 mL	110 L
2	60 mL	15 mL	75 mL	110 L
W	60 mL	15 mL	75 mL	110 L
X	60 mL	15 mL	75 mL	110 L
Y	60 mL	15 mL	75 mL	110 L
Z	60 mL	15 mL	75 mL	110 L
5	60 mL	15 mL	75 mL	110 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.

Contamination due to vapors

Contamination due to atmospheric nitrogen (example: ammonia vapors) can significantly influence the analysis results.

4.4 Buffer

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide	NaOH	40.00	1310-73-2	10 g
Di-sodium tartrate dihydrate	$C_4H_4Na_2O_6$ * $2H_2O$	230.08	6106-24-7	80 g

Preparation

Dissolve 10 g sodium hydroxide (NaOH) and 80 g di-sodium tartrate dihydrate ($Na_2C_4H_4O_6$ * $2H_2O$) in 700 mL de-ionized water. Mix and fill up to 1 litre with de-ionized water.

4.5 Chlorine

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide	NaOH	40.00	1310-73-2	15 g
Sodium hypochlorite solution (6 – 14%)	NaOCl	74.44	7681-52-9	20 mL

Preparation

Add carefully 15 g of the NaOH pellets to 700 mL de-ionized water. Add 20 mL sodium hypochlorite solution (NaOCl with 6 – 14 % active chlorine). Mix and fill up to 1 litre with de-ionized water.

Procedure for determination of the exact chlorine concentration:

a. Dilute the sodium hypochlorite (6-14% free chlorine) solution with factor 100 (1 ml solution in 100 ml de-ionized water)
b. Take 20 ml of this sample into a 100 ml beaker
c. Add approx. 3 g of Potassium Iodide to this sample. The sample will turn in to a yellow-brown colour.
d. Add 1 mL of a 2 N Acid solution
e. Titrate with a 0.05 M Thiosulfate solution until titter turns colourless again.
f. Calculate amount of active Cl_2 component:

$$a\ g/\mathrm{L}\ Cl_2 = \frac{ml\ thiosulphate\ added * M\ thiosulphate * 71 * 100}{2 * 20}$$

Note: The minimum amount of active component of bleach should be > than 6% (60 g/l)

4.6 Color

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium salicylate	HOC_6H_4COONa	160.00	54-21-7	80 g
Sodium nitroprusside dihydrate	$Na_2[Fe(CN)_5NO] * 2H_2O$	297.96	13755-38-9	5.0 g

Preparation

Dissolve 80 g sodium salicylate (HOC_6H_4COONa) and 5 g sodium nitroprusside dihydrate ($Na_2[Fe(CN)_5NO] * 2H_2O$) in 700 mL de-ionized water. Mix and fill up to 1 litre with de-ionized water.

4.7 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Ammonium chloride	NH_4Cl	53.49	12125-02-9	3.821 g

Preparation

1000 mg/L NH_4-N stock solution

Prepare a stock solution of 1000 mg/L NH_4-N: Dissolve accurately 3.821 g Ammonium chloride (NH_4Cl) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

NH_4-N standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 1000 mg/L NH_4-N 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
A	0.10 mg/L NH_4-N	0.10 mg/L	0.10 mL
B	0.25 mg/L NH_4-N	0.25 mg/L	0.25 mL
C	0.50 mg/L NH_4-N	0.50 mg/L	0.50 mL
0	**1.0 mg/L NH_4-N**	**1.0 mg/L**	**1.0 mL**
1	4.0 mg/L NH_4-N	4.0 mg/L	4.0 mL
2	8.0 mg/L NH_4-N	8.0 mg/L	8.0 mL
W	10 mg/L NH_4-N	10 mg/L	10 mL
X	25 mg/L NH_4-N	25 mg/L	25 mL
Y	50 mg/L NH_4-N	50 mg/L	50 mL
Z	75 mg/L NH_4-N	75 mg/L	75 mL
5	100 mg/L NH_4-N	100 mg/L	100 mL

NH_4-N standard solution – REF1

Prepare a standard solution of 0 mg/L NH_4-N. Use de-ionized water.

4.8 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: 02/02/2023	Previous version: Edition 1.00 to Edition 1.01

Reason for Change

- Preparation Buffer wrong volume NaOH in description

Description of Change

- Change preparation Buffer volume NaOH in description to 10g instead of 40g