DOC313.53.94203

EZ2500 Total Cyanide Analyser

Method and reagent sheets

08/2021, Edition 1.01

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Table of Contents

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 Cyanide - All specifications				
Analysis method	Colorimetric measurement at 578 nm using chloramine T method			
Parameter	CN Total			
Cycle time	Standard measurement cycle time: 75 minutes Internal dilution: + 5 min.			
Limit of detection (LOD)	≤ 1 µg/L			
Precision/Repeatability	Better than 3% full scale range for standard test solutions			
Cleaning	N.A.			
Calibration	Automatic, 2-point; frequency freely programmable			
Validation	Automatic; frequency freely programmable			
Interferences	Ions like nitrite [(NO_2)] > 5 mg/L, sulphide [$(S)^{2-}$] > 100 mg/L and sulphite[$(SO_3)^{2-}$]. Thiocyanate will cause high results. Large amounts of color and turbidity interfere. Fats, oil, proteins, surfactants and tar.			
Measuring ranges	**% of range - Dilution**		**Low range (µg/L)**	**High range (µg/L)**
	0	**standard range**	**1**	**100**
	3	internal MP dilution (factor 10)	100	1000

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3. Analysis method

Summary

Cyanide reacts with chloramine-T in a medium with pH<8 and addition of isonicotinic-barbituric acid to form a red-blue coloured complex. This colour is determined at a wavelength of 578 nm.

Analysis steps

The sample is mixed with the acid solution and catalyst and heated to 130 °C (or up to 150 °C – programmable) in an oven (standard 40 minutes; programmable up to 60 minutes). During this process the cyanide compounds are converted to HCN and stripped from the sample with a continuous air flow and then absorbed in a sodium hydroxide solution. CN^- is converted to CNCl by reaction with chloramines-T at pH<8 without hydrolysing to CNO^-. When the reaction is complete, CNCl forms a red-blue colour on addition of isonicotinic-barbituric acid. The absorbance value is measured at a wavelength of 578 nm. With these obtained absorbance values, the cyanide concentration is calculated according to Beer's Law.

Calibration

The calibration procedure measures a REF1 CN solution (channel 9, REF1 valve) and a REF2 CN 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.

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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/75 min.	Recommended containers
Acid solution	~ 2.0 mL / analysis	~ 1.08 L	Plastic – 2.5 L
NaOH solution	~ 3.0 mL / analysis	~ 1.6 L	Plastic – 2.5 L
Buffer solution	~ 2.0 mL / analysis	~ 1.08 L	Plastic – 2.5 L
Chloramine-T solution	~ 1.0 mL / analysis	~ 1.04 L	Plastic – 2.5 L
Colour solution	~ 2.0 mL / analysis	~ 1.08 L	Glass – 2.5 L
REF1 solution	~ 0.5 L / calibration	/	Plastic – 1 L
REF2 solution	~ 0.5 L / calibration	/	Plastic – 1 L

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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 / 50 min.
0	50 mL	N.A.	50 mL	40.32 L
3	60 mL	15 mL	75 mL	34.6 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.

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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	
	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.

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4.4 Acid solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sulfuric acid (96%)	H_2SO_4	96.08	7664-93-9	556 g
Magnesium chloride hexahydrate	$MgCl_2 * 6H_2O$	203.30	7791-18-6	128.8 g

Preparation

Dissolve 128.8 g magnesium chloride hexahydrate ($MgCl_2 * 6H_2O$) in 400 mL de-ionized water. Next, add carefully in small amounts 556 g sulfuric acid (H_2SO_4, 96%) and dissolve completely. Fill up to 1 litre with de-ionized water.

4.5 NaOH solution (0.02M)

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide	NaOH	40.00	1310-73-2	0.8 g

Preparation

Dissolve 0.8 g sodium hydroxide (NaOH) in 500 mL de-ionized water. Fill up to 1 litre with de-ionized water.

4.6 Buffer solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide	NaOH	40.00	1310-73-2	2.3 g
Sodium hydroxide solution (1M)	/	/	/	/
Potassium hydrogen phthalate	$C_8H_5KO_4$	204.22	877-24-7	20.5 g

Preparation

Dissolve 2.3 g sodium hydroxide (NaOH) in 500 mL de-ionized water. Add 20.5 g potassium hydrogen phthalate ($C_8H_5KO_4$) and fill up to 950 mL with de-ionized water. Adjust the pH with sodium hydroxide (NaOH, 1M) until pH 5.2. Fill up to 1 litre with de-ionized water.

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4.7 Chloramine-T solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Chloramine T trihydrate	$C_7H_7ClNaNO_2S * 3H_2O$	281.69	7080-50-4	2 g

Preparation

Dissolve 2 g of chloramine T trihydrate ($C_7H_7ClNaNO_2S * 3H_2O$) in 500 mL de-ionized water and fill up to 1 litre with de-ionized water.

4.8 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide	NaOH	40.00	1310-73-2	7 g
Isonicotinic acid	$C_6H_5NO_2$	123.11	55-22-1	13.6 g
Sodium barbiturate	$C_4H_3N_2NaO_3$	150.06	4390-16-3	16.3 g
Hydrochloric acid (37%)	HCl	36.46	7647-01-0	~ 5 mL

Preparation

Dissolve 7 g of sodium hydroxide (NaOH) in 500 mL de-ionized water. Add 13.6 g of isonicotinic acid ($C_6H_5NO_2$) and dissolved completely. Next, add 16.3 g of sodium barbiturate ($C_4H_3N_2NaO_3$). Fill up to 900 mL with de-ionized water. Gently heat the solution to when it's just not boiling while stirring for at least 1 hour. The reagents should now be dissolved. Cool down. Add hydrochloric acid (HCl 37%) until pH 5.2 (approximately 3 to 5 mL) and fill up to 1000 mL with de-ionized water. Put the solution in the fridge for at least 12 hours. Filter the solution before use.

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4.9 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide solution (1M)	NaOH	40.00	1310-73-2	40.0 g
Potassium cyanide	KCN	65.12	151-50-8	0.250 g

Preparation

100 mg/L CN stock solution

Prepare a stock solution of 100 mg/L CN: Take 10 mL sodium hydroxide (NaOH, 1M) in 500 mL de-ionized water using a volumetric flask of 1000 mL. Dissolve 0.250 g potassium cyanide (KCN). Add de-ionized water up to the mark grade.

CN standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 100 mg/L CN stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

	Measuring range	Amount to add to 1 litre
0	**100 µg/L CN**	**1.0 mL**
3	1000 µg/L CN	10 mL

CN standard solution – REF1

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

4.10 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.

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4.11 Warning letter

RE: Hach EZ2500 Total Cyanide analyzer

Dear Valued Customer,

Please forward this notice to your plant and laboratory operators who work with and near the above-referenced instrument and its chemical reagents.

WHAT
This notification is to make the customer aware on a possible health challenge with the EZ2500 (Total Cyanide Analyzer) if not following the standard procedures for operation and maintenance.

WHY
The analysis method of the EZ2500 (Total Cyanide Analyzer) involves the formation of HCN which is a toxic gas harming human health.

HOW
For this reason, we want customers to pay extra attention to the correct connection of the vent of the analyzer and ensure to follow the installation instructions of the analyzer described in the Basic User Manual DOC023.xx.90633, like well-ventilated room.

Hach has experienced no reports of HCN-caused issues for customers globally for an EZ2500 analyzer yet, but we want to emphasize once more the possible risk for human health, if not following the standard procedure for operation and maintenance.

Best regards,

Hach

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Change Information	
Date: 19/08/2021	Previous version: Edition 6 to Edition 1.01

Reason for Change

- Compliance request
- Addition of water consumption
- Addition of information reagents

Description of Change

- Addition of warning letter (chapter 4.11)
- Addition of estimated consumption of water for rinse and dilution (chapter 4.2)
- Addition of extra information regarding storage and quality of reagents (chapter 4.3)

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