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EZ1012 Cyanide Analyser

Method and reagent sheets

08/2021, 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.

Cyanide - All specifications				
Analysis method	Colorimetric measurement at 578 nm using chloramine T method, conform with standard method APHA 4500-CN (E)			
Parameter	Cyanide			
Cycle time	Standard measurement cycle time: 20 minutes Internal dilution: + 5 min. External dilution: + 5 – 10 min.			
Limit of detection (LOD)	≤ 1 µg/L			
Precision/Repeatability	Better than 5% 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	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)**
	A	10% of standard range	1	20
	B	25% of standard range	2	50
	C	50% of standard range	2	100
	0	**standard range**	**5**	**200**
	1	internal MP dilution (factor 4)	40	800
	2	internal MP dilution (factor 8)	80	1600
	W	internal dispenser dilution (factor 10)	50	2000
	X	internal dispenser dilution (factor 25)	125	5000
	Y	internal dispenser dilution (factor 50)	250	10000
	Z	internal dispenser dilution (factor 75)	375	15000
	5	internal dispenser dilution (factor 100)	500	20000

3. Analysis method

Summary

Cyanide is converted to CNCl by reaction with chloramine T . After the reaction is finished the CNCl will form an coloured complex with isonicotinic acid barbituric acid. The absorption is measured at a wavelength of 578 nm

Analysis steps

The analysis vessel is cleaned with fresh sample. After addition of the buffer solution and the chloramine-T solution, the initial absorbance measurement is performed at 578 nm. This measurement is done to correct for any colour contribution of the sample. Next, the colour solution is dosed and after respecting a stirring period, the final absorbance measurement is performed. With the obtained absorbance values, the free cyanide concentration can be 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.

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/20 min	Recommended containers
Buffer solution	~ 1 mL / analysis	~ 2 L	Plastic – 2.5 L
Chloramine-T solution	~ 1 mL / analysis	~ 2 L	Plastic – 2.5 L
Colour solution	~ 2 mL / analysis	~ 4 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 / 20 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	151 L
2	60 mL	15 mL	75 mL	151 L
W	60 mL	15 mL	75 mL	151 L
X	60 mL	15 mL	75 mL	151 L
Y	60 mL	15 mL	75 mL	151 L
Z	60 mL	15 mL	75 mL	151 L
5	60 mL	15 mL	75 mL	151 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.

Reagent preparation

Use 0.1N sodium hydroxide to clean all glassware and laboratory equipment to prevent any cyanide from being released.

4.4 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 1M solution	NaOH	40.00	1310-73-2	Dependent on pH
Potassium hydrogen phtalate	$C_8H_5KO_4$	204.22	877-24-7	20.5 g

Preparation

Dissolve 2.3 g of sodium hydroxide (NaOH) in 500 mL of de-ionized water. Add 20.5 g of potassium hydrogen phthalate ($C_8H_5KO_4$) and fill up to 950 mL with de-ionized water. Adjust the pH with sodium hydroxide (1M) until pH 5.2. Fill up to 1000 mL with de-ionized water.

4.5 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 of de-ionized water and dilute to 1 litre with de-ionized water.

4.6 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	Dependent on pH

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. Then, 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.

4.7 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide solution (1M)	NaOH	40.00	1310-73-2	10 mL
Potassium cyanide	KCN	65.12	151-50-8	0.125 g

Preparation

50 mg/L cyanide stock solution

Prepare a stock solution of 50 mg/L CN: Dilute 10 mL of the 1 M sodium hydroxide solution (NaOH) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Dissolve carefully 0.125 g of potassium cyanide (KCN). Add de-ionized water up to the mark grade.

Cyanide standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 50 mg/L cyanide 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	20 µg/L CN	20 µg/L CN	0.4 mL
B	50 µg/L CN	50 µg/L CN	1 mL
C	100 µg/L CN	100 µg/L CN	2 mL
0	**200 µg/L CN**	**200 µg/L CN**	**4 mL**
1	800 µg/L CN	800 µg/L CN	16 mL
2	1600 µg/L CN	1600 µg/L CN	32 mL
W	2000 µg/L CN	2000 µg/L CN	40 mL
X	5000 µg/L CN	5000 µg/L CN	100 mL
Y	10000 µg/L CN	10000 µg/L CN	200 mL
Z	15000 µg/L CN	15000 µg/L CN	300 mL
5	20000 µg/L CN	20000 µg/L CN	400 mL

Cyanide standard solution – REF1

Prepare a standard solution of 0 mg/L CN. 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.

4.9 Warning letter

RE: Hach EZ1012 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 EZ1012 (Cyanide Analyzer) if not following the standard procedures for operation and maintenance.

WHY
Since the sample contains cyanide it is not allowed to use acid as a cleaning reagent which becomes a problem when pH-level drops to acid levels in the reaction vessel in the analyzer. The reason is that it forms HCN, which is a toxic gas harming human health.

HOW
For this reason, we want to avoid customers to apply any kind of acid (as a cleaning reagent) to the analyzer to prevent HCN-formation and thus ensuring safe operation.

Hach has experienced no reports of HCN formation in an EZ1012 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

Change Information	
Date: 19/08/2021	Previous version: Edition 4 to Edition 1.01

Reason for Change

- Compliance request
- Addition of extra ranges to the portfolio of EZ1012
- Addition of water consumption
- Addition of information reagents

Description of Change

- Addition of warning letter (chapter 4.9)
- Addition of extra ranges for internal dispenser dilution: 10x, 25x, 50x, 75x (chapter 4.7)
- 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)