EZ1012 Cyanide Analyzer

DOC313.53.94178

Standard range: 0.05 to 0.2 mg/L CN

Method EZ1012

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 3.
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 3 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	Chloramine T colorimetric method, conforms with Standard Method APHA 4500-CN (E)
Measurement wavelength	578 nm
Parameter	Long description: Cyanide Short description (default): CN 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
Interferences	Ions, e.g., nitrite (NO_2^-) > 5 mg/L, sulfide (S^{2-}) > 100 mg/L and sulfite(SO_3^{2-}) interfere. Thiocyanate causes high results. 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
A	10% of standard range	0.001	0.02	2	28	30
B	25% of standard range	0.002	0.05	2	28	30
C	50% of standard range	0.002	0.1	2	28	30
0	**Standard range**	**0.05**	**0.2**	**2**	**28**	**30**
V	internal dispenser dilution (factor 5)	0.025	1	2	31	45
W	internal dispenser dilution (factor 10)	0.05	2	2	31	45
X	internal dispenser dilution (factor 25)	0.125	5	2	31	45
Y	internal dispenser dilution (factor 50)	0.25	10	2	31	45
Z	internal dispenser dilution (factor 75)	0.375	15	2	31	45
5	internal dispenser dilution (factor 100)	0.5	20	2	31	45

Summary of method

Summary

Cyanide reacts with chloramine T to form cyanogen chloride (CNCl). The cyanogen chloride then reacts with isonicotinic acid-barbituric acid to form a violet-blue color. The absorbance is measured at a wavelength of 578 nm.

Analysis steps

The analysis vessel is rinsed and filled with new sample. The buffer and chloramine-T 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 cyanide 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/20 minutes**	**Use life**	**Containers**	**Operation temperature**
Red	Reagent 1	Buffer	~ 1 mL	~ 2.1 L	28 days	Plastic; 2.5 L	10 to 30 °C (50 to 86 °F)
Blue	Reagent 2	Chloramine-T	~ 1 mL	~ 2.1 L	28 days	Plastic; 2.5 L	10 to 30 °C (50 to 86 °F)
Green	Reagent 3	Color	~ 2 mL	~ 4.1 L	28 days	Glass; 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	85	95	As necessary	REF1
2-point (recommended)	170	185	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.*

Note: *The range codes A, B, C, 0 are configured as default without the use of rinse and dilution water.*

Table 5 DI water consumption

Range code	Rinse water Type I (mL/analysis)	Dilution water Type I (mL/analysis)	Total (mL/analysis)	Per 28 days, rate of 1 analysis each 45 minutes
A - B - C - 0 (no dilution)	—	—	—	—
V - W - X - Y - Z - 5 (with dilution)	51 mL	16 mL	67 mL	60 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: Buffer Code: Red	Sodium hydroxide	NaOH	40.00	1310-73-2	2.3 g
	Sodium hydroxide solution (1 M)	NaOH	40.00	1310-73-2	Dependent on the pH
	Potassium hydrogen phthalate	$C_8H_5KO_4$	204.22	877-24-7	20.5 g
Reagent 2: Chloramine T Code: Blue	Chloramine T trihydrate	$C_7H_7ClNaNO_2S * 3H_2O$	281.69	7080-50-4	2 g
Reagent 3: Color Code: Green	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 the pH
Stock solution	Sodium hydroxide solution (1 M)	NaOH	40.00	1310-73-2	10 mL
	Potassium cyanide	KCN	65.12	151-50-8	0.125 g
REF1 calibration standard	Deionized water (Type I or better)	—	—	—	—
REF2 calibration standard	50 mg/L cyanide stock solution	—	—	—	Refer to Table 7 on page 6.
	Sodium hydroxide (1 M)	NaOH	40.00	1310-73-2	1 mL
Validation standard (optional)	REF2 calibration standard	—	—	—	Refer to Validation standard on page 6.
Cleaning solution (optional)	EDTA disodium salt dihydrate	$C_{10}H_{14}N_2Na_2O_8 * 2H_2O$	372.24	6381-92-6	18.6 g
	Sodium hydroxide	NaOH	40.00	1310-73-2	20 g

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 3. Make sure to discard the remaining solution from the analyzer bottles before new reagents are added.

Reagent 1: Buffer

1. Add 500 mL of deionized water to a beaker.
2. Add 2.3 g of sodium hydroxide (NaOH).
3. Mix until fully dissolved.
4. Add 20.5 g of potassium hydrogen phthalate ($C_8H_5KO_4$) and fully mix.
5. Pour the solution into a 1000-mL volumetric flask.
6. Add deionized water to approximately 950 mL and fully mix.
7. Adjust the pH to 5.2 with 1 M NaOH.

8. Add deionized water to the mark.
9. Fully mix the solution.

Reagent 2: Chloramine-T

1. Add 500 mL of deionized water to a beaker.
2. Slowly mix in 2 g of chloramine T trihydrate ($C_7H_7ClNaNO_2S * 3H_2O$).
3. Pour the solution into a 1000-mL volumetric flask.
4. Add deionized water to the mark.
5. Fully mix the solution.

Reagent 3: Color

1. Add 500 mL of deionized water to a beaker.
2. Slowly mix in 7 g of sodium hydroxide (NaOH).
3. Add 13.6 g of isonicotinic acid ($C_6H_5NO_2$).
4. Mix until fully dissolved.
5. Add 16.3 g of sodium barbiturate ($C_4H_3N_2NaO_3$).
6. Add deionized water to approximately 900 mL.
7. Slowly heat and stir the solution to just below the boiling point for a minimum of 1 hour. The reagents should now be dissolved.
8. Let the temperature of the solution decrease until sufficiently cool.
9. Slowly add approximately 3 to 5 mL of hydrochloric acid (HCl 37%) until the pH is 5.2.
10. Pour the solution into a 1000-mL volumetric flask.
11. Add deionized water to the mark.
12. Fully mix the solution.
13. Put the solution in a refrigerator for a minimum 12 hours. Filter the solution before use.

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 50 mg/L CN stock solution as follows. Refer to Necessary reagents on page 4 to collect the applicable items.

1. Add 300 mL of deionized water to a beaker.
2. Add 10 mL of 1 M of NaOH.
3. Fully mix the solution.
4. Carefully add 0.125 g of potassium cyanide (KCN).
5. Fully mix the solution.
6. Pour the solution into a 1000-mL volumetric flask.
7. Add deionized water to the mark.
8. 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 plastic 1000-mL volumetric flask. Refer to Table 7.
2. Use a pipet to add the applicable quantity (mL) of 1 M sodium hydroxide from Table 7 to the volumetric flask.
3. Add deionized water to the mark.
4. Fully mix the solution.

Table 7 Calibration standard preparation

Range code	REF2 concentration (mg/L CN)	Quantity (mL) of stock solution	Quantity (mL) of 1 M NaOH
A	0.02	0.4	1
B	0.05	1	1
C	0.1	2	1
0	0.2	4	1
V	1	20	1
W	2	40	1
X	5	100	1
Y	10	200	1
Z	15	300	1
5	20	400	1

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, 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

⚠ DANGER

Chemical exposure hazard. Do not use an acidic solution for cleaning to prevent the possible formation of toxic hydrogen cyanide gas.

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 EDTA in 0.5 M NaOH 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. Mix in 20 g of sodium hydroxide (NaOH).

3. Add 18.6 g of ethylenediaminetetraacetic acid disodium salt dihydrate ($C_{10}H_{14}N_2Na_2O_8 * 2H_2O$).
4. Mix until fully dissolved.
5. Pour the solution into a 1000-mL volumetric flask.
6. Add deionized water to the mark.
7. Fully mix the solution.

Warning letter

Subject:

With reference to: 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

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