DOC312.53.94455

EZ1007 Total Chlorine Analyser

Method and reagent sheets

09/2020, 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.

Total Chlorine - All specifications				
Analysis method	Colorimetric measurement using DPD colour solution			
Parameter	Total Chlorine (Cl_2)			
Cycle time	5 minutes (dilution +5 min.) Internal dilution: + 5 min. External dilution: + 5 – 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	High concentrations of monochloramine and oxidizes forms of manganese. Large amounts of colour and turbidity interferes. Fats, oil, proteins, surfactants and tar.			
Measuring ranges	**% of range - Dilution**		**Low range (mg/L)**	**High range (mg/L)**
	A	10% of standard range	0.005	0.150
	B	25% of standard range	0.010	0.375
	C	50% of standard range	0.010	0.750
	0	standard range	0.025	1.5
	1	internal MP dilution (factor 4)	0.2	6.0
	2	internal MP dilution (factor 8)	0.4	12
	W	internal dispenser dilution (factor 10)	0.25	15
	X	internal dispenser dilution (factor 25)	0.625	37.5
	Y	internal dispenser dilution (factor 50)	1.25	75
	Z	internal dispenser dilution (factor 75)	1.875	112.5
	5	internal dispenser dilution (factor 100)	2.5	150

3. Analysis method

Summary

Total chlorine reacts with the potassium iodide solution and oxidizes the iodide to iodine. Both the iodine and the free chlorine react with DPD (N,N-diethyl-p-phenylenediamine)-colour solution to form a highly intensive pink coloured complex. The absorption is measured at a wavelength of 510. nm.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling the initial absorbance value is measured at 510 nm. Next, the potassium iodide, the colour solution and buffer solution 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 total chlorine concentration can be calculated according to Beer's law.

Calibration

The calibration procedure measures a REF1 Cl_2 solution (channel 9, REF1 valve) and a REF2 Cl_2 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 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/5 min	Recommended containers
Buffer solution	~ 0.5 mL / analysis	~ 4.0 L	Plastic – 5 L
Colour solution	~ 0.5 mL / analysis	~ 4.0 L * (~ 2.0 L / 14 days)	Glass, Amber – 2.5 L
KI solution	~ 0.5 mL / analysis	~ 4.0 L	Glass, Amber – 2.5 L
REF1 solution	~ 0.5 L / calibration	/	Plastic – 1 L
REF2 solution	~ 0.5 L / calibration	/	Plastic – 1 L

*This solution is stable for maximum 2 weeks

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 / 10 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	604 L
2	60 mL	15 mL	75 mL	604 L
W	60 mL	15 mL	75 mL	604 L
X	60 mL	15 mL	75 mL	604 L
Y	60 mL	15 mL	75 mL	604 L
Z	60 mL	15 mL	75 mL	604 L
5	60 mL	15 mL	75 mL	604 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.

4.4 KI solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Potassium iodide	KI	166.00	7681-11-0	10.0 g

Preparation

Dissolve 10 g potassium iodide (KI) in 800 mL de-ionized water. Fill up to 1 litre with de-ionized water.

4.5 Buffer solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Disodium hydrogen phosphate dihydrate	$Na_2HPO_4 * 2H_2O$	177.99	10028-24-7	30 g
Potassium dihydrogen phosphate	KH_2PO_4	136.09	7778-77-0	46 g
EDTA	$C_{10}H_{14}N_2Na_2O_8 * 2H_2O$	372.2	6381-92-6	0.8 g

Preparation

Dissolve 30 g disodium hydrogen phosphate dihydrate ($Na_2HPO_4 * 2H_2O$) and 46 g potassium dihydrogen phosphate (KH_2PO_4) in 800 mL de-ionized water. Next, dissolve 0.8 g EDTA in 100 mL de-ionized water. Combined the 2 solutions and dilute to 1 litre with de-ionized water.

4.6 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
EDTA	$C_{10}H_{14}N_2Na_2O_8 * 2H_2O$	372.2	6381-92-6	0.2 g
Sulfuric acid 96%	H_2SO_4	98.08	7664-93-9	2 mL
N,N-diethyl-p-phenylenediamine sulfate	$(C_2H_5)_2NC_6H_4NH_2 * H_2SO_4$	262.33	6283-63-2	1.5 g

Preparation

Dissolve 0.2 g of EDTA in 500 mL de-ionized water. Next, add 2 mL sulfuric acid (H_2SO_4, 96%). Finally dissolve 1.5 g N,N-diethyl-p-phenylenediamine sulfate ($(C_2H_5)_2NC_6H_4NH_2 * H_2SO_4$) and dilute to 1 litre with de-ionized water.

This solution is stable for maximum 2 weeks. Store the reagent in a closed (brown) bottle and in a fridge during operation to prolong stability up to one month Avoid contact of the colour solution with ambient air. The color solution should be colourless. If the colour solution turns brownish, please replace to guarantee good results.

We recommend to use N,N-diethyl-p-phenylenediamine sulfate with following specifications:

Product	Brand	Product No.	Specification
N,N-diethyl-p-phenylenediamine sulfate	Fluka	07670	Puriss. p.a. for spectrophotometric detection of S_2, Cl_2

4.7 Calibration solution

The analyzer is factory calibrated. The standard solutions, used for calibration, should be prepared by use of potassium permanganate (expressed as a chlorine equivalent).

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Potassium permanganate	$KMnO_4$	158.03	7722-64-7	0.891 g

Preparation

1000 mg/L Cl_2 stock solution

Prepare a stock solution of 1000 mg/L Cl_2: Dissolve accurately 0.891 g potassium permanganate ($KMnO_4$) in 500 mL de-ionized water using a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

This solution is stable for 1 day. Always prepare fresh calibration standards before calibration.

Cl_2 standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 1000 mg/L Cl_2 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	0.15 mg/L Cl_2	0.15 mg/L Cl_2	0.15 mL
B	0.375 mg/L Cl_2	0.375 mg/L Cl_2	0.375 mL
C	0.75 mg/L Cl_2	0.75 mg/L Cl_2	0.75 mL
0	**1.5 mg/L Cl_2**	**1.5 mg/L Cl_2**	**1.5 mL**
1	6.0 mg/L Cl_2	6.0 mg/L Cl_2	6.0 mL
2	12.0 mg/L Cl_2	12.0 mg/L Cl_2	12.0 mL
W	15.0 mg/L Cl_2	15.0 mg/L Cl_2	15.0 mL
X	37.5 mg/L Cl_2	37.5 mg/L Cl_2	37.5 mL
Y	75.0 mg/L Cl_2	75.0 mg/L Cl_2	75.0 mL
Z	112.5 mg/L Cl_2	112.5 mg/L Cl_2	112.5 mL
5	150.0 mg/L Cl_2	150.0 mg/L Cl_2	150.0 mL

Cl_2 standard solution – REF1

Prepare a standard solution of 0 mg/L Cl_2. Use de-ionized water.

4.8 Validation solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hypochlorite (6-14% free chlorine)	NaOCl	262.33	7681-52-9	/

Preparation

1000 mg/L Cl_2 stock solution

Prepare a stock solution of 1000 mg/L Cl_2.

1) Determine the exact chlorine concentration by titration with sodium thiosulfate $Na_2S_2O_5$ 0.01 M.

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 (paragraph 4.4)
e. Titrate with a 0.05 M Thiosulfate solution (paragraph 4.5) until titter turns colourless again.
f. Calculate amount of active Cl_2 component:

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

2) To prepare the stock solution of 1000 mg/L Cl_2, take accurately 'b' mL of the sodium hypochlorite (6-14% free chlorine) with a concentration of 'a' g/L Cl_2 and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

Concentration active Cl_2	Amount (mL) of sodium hypochlorite (6-14% free chlorine) to add to 1 litre	Calculation
a g Cl_2/L	b mL	$b = \frac{1000}{a}$ mL

4.9 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: 17/09/2021	Previous version: Edition 3 to Edition 1.01

Reason for Change

- Addition of water consumption
- Addition of information reagents
- Correction CAS

Description of Change

- 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)
- Correction CAS-number Sodium hypochlorite solution (6 – 14%) (chapter 4.8)