DOC313.53.94281

EZ7002 COD Cr Analyser

Method and reagent sheets

02/2020, 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.

COD Cr - All specifications				
Analysis method	Redox titration after oxidation by acid-potassium dichromate solution			
Parameter	Chemical Oxygen Demand			
Cycle time	Standard measurement cycle time: 40 minutes			
Limit of detection (LOD)	≤ 60 mg/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	Chloride [$(Cl)^-$] > 1 g/L, inorganic reducing agents such as nitrites [$(NO_2)^-$], sulphides [$(S)^{2-}$] and iron(II) [$(Fe)^{2+}$] will increase the result, aromatic hydrocarbons and pyridine are not oxidized to any appreciable extent. Some very volatile organic substances may escape the oxidation by evaporation. Straight chain aliphatic compounds are effectively oxidized by the silver sulphate/sulphuric acid solution. Fats, oil, proteins, surfactants and tar.			
Measuring ranges	**% of range - Dilution**		**Low range (mg/L)**	**High range (mg/L)**
	0	**standard range**	**60**	**1000**

3. Analysis method

Summary

The determination of the COD concentration in water is based upon the titration with ammonium iron (II) sulphate solution (Mohr's salt). During the oxidation phase the organic compounds are oxidized and converted to carbon dioxide, nitrates and water, while potassium dichromate is converted to chrome (III). The amount of ammonium iron (II) sulphate solution titrated is used to calculate the COD concentration of the sample.

Analysis steps

The sample is diluted and mixed with dichromate solution ($K_2Cr_2O_7$) and the sulfuric acid solution (H_2SO_4). During the digestion process, the organic material is oxidized. After digestion, a titration with ammonium iron(II) sulphate solution (Mohr's salt) is performed. The amount of Mohr's salt titrated is used to calculate the COD concentration of the sample.

Calibration

The calibration procedure measures a REF1 O_2 solution (channel 9, REF1 valve) and a REF2 O_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 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/40 min	Recommended containers
Dichromate solution	~ 3 mL / analysis	~ 4.0 L	Plastic – 5 L
Acid solution	~ 6 mL / analysis	~ 6.0 L	Plastic – 5 L
Mohr's salt solution	~ Depending on COD concentration	3.0 L < Volume < 10 L	Plastic – 10 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 rate 1 analysis / 40min
0	**~ 150 mL**	**~ 3 mL**	**~ 153 mL**	**~ 155 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 Dichromate solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Potassium dichromate	$K_2Cr_2O_7$	294.19	7778-50-9	3.2 g
Sulfuric acid 96%	H_2SO_4	101.19	7664-93-9	100 mL
Mercury(II) sulfate	$HgSO_4$	296.65	7783-35-9	20 g

Preparation

Dissolve 20 g mercury (II) sulphate in 500 mL de-ionized water. Add 100 mL sulfuric acid (H_2SO_4 96%). Allow to cool down and add 3.2 g potassium dichromate ($K_2Cr_2O_7$) dried at 105 °C for 2 hours. Fill up to 1 litre with de-ionized water. The final concentration of this solution is 0.0652 N.

4.5 Acid solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sulfuric acid 96%	H_2SO_4	101.19	7664-93-9	965 mL
Silver sulphate	Ag_2SO_4	311.8	10294-26-5	10 g

Preparation

Add 10 g of silver sulphate (Ag_2SO_4) to 35 mL de-ionized water. Dissolve carefully 965 mL sulfuric acid (H_2SO_4 96%).

4.6 Mohr’s salt solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sulfuric acid 96%	H_2SO_4	101.19	7664-93-9	20 mL
Ammonium iron(II) sulphate hexahydrate	$(NH_4)_2Fe(SO_4)_2 * 6H_2O$	392.14	7783-85-9	7.8428 g

Preparation

Dissolve 7.8428 g ammonium iron (II) sulphate hexahydrate ($(NH_4)_2Fe(SO_4)_2 * 6H_2O$) in 500 mL de-ionized water. Add carefully 20 mL sulfuric acid (H_2SO_4 96%) and fill up to 1 litre with de-ionized water. The final concentration of this solution is 0.02 M.

4.7 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Potassium hydrogen phthalate	$KC_8H_5O_4$	204.22	877-24-7	8.5 g

Preparation

O_2 stock solution

Prepare a stock solution of 10000 mg O_2/L : Dissolve accurately 8.5 g potassium hydrogen phthalate ($KC_8H_5O_4$) in 500 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

O_2 standard solution – REF2

Prepare a standard solution of 1000 mg O_2/L: Take accurately 100 mL of the 10000 mg O_2/L stock solution in 500 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

O_2 standard solution – REF1

Prepare a standard solution of 0 mg O_2/L. 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: 18/02/2022	Previous version: Edition 4 to Edition 1.01
Reason for Change	
- Addition of water consumption - Addition of reagents information	
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)	