DOC313.53.94284

EZ7050 COD Mn Analyser

Method and reagent sheets

09/2020, Edition 5

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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 Mn - All specifications			
Analysis method	Redox titration after oxidation by potassium permanganate solution		
Parameter	Chemical Oxygen Demand		
Cycle time	40 minutes, including oxidation time of 30 minutes		
Limit of detection (LOD)	≤ 0.2 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 [$(Cl)^-$] > 3 g/L, inorganic reducing agents such as nitrites [$(NO_2)^-$], sulphides [$(S)^{2-}$] and iron(II) [$(Fe)^{2+}$] will increase the result. Fats, oil, proteins, surfactants and tar.		
Measuring ranges	**% of range - Dilution**	**Low range (mg/L)**	**High range (mg/L)**
	standard range	0.2	20

3. Analysis method

Summary

The determination of the COD concentration in water is based upon the titration of remaining sodium oxalate with potassium permanganate. During the oxidation phase the COD reacts with the potassium permanganate. The amount of permanganate titrated is used to calculate the COD concentration of the sample.

Analysis steps

The sample is mixed with the permanganate solution ($KMnO_4$), the sulfuric acid solution (H_2SO_4) and the silver nitrate solution ($AgNO_3$). The solution is heated to 100°C for 30 minutes in order to oxidize the organic material with the permanganate. After the oxidation, an excess of oxalate solution ($Na_2C_2O_4$) is added. A titration with permanganate is performed. The amount of permanganate titrated is used to calculate the COD concentration of the sample.

Calibration

The calibration procedure measures a REF1 O solution (channel 9, REF1 valve) and a REF2 O 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
$KMnO_4$ solution	~ 6.5 ml + x ml Depending on the COD concentration	10.8 L < Volume < 14.2 L	Dark Plastic – 10 L
Acid solution	~ 3.0 mL / analysis	~ 3.2 L	Plastic – 5 L
Oxalate solution	~ 6.0 mL / analysis	~ 6.0 L	Plastic – 10 L
REF1 Solution	~ 1 L / calibration	/	Plastic – 2.5 L
REF2 Solution	~ 1 L / calibration	/	Plastic – 2.5 L

4.2 Storage and quality of chemicals

Quality of chemicals

All chemicals should be of ACS grade or better. We recommend the use of pro analysis chemicals.

Quality of water

Reagent grade, de-ionized water must be used to prepare the chemical solutions and for rinse purposes.

Storage of Reagents

While operating the instrument, keep in mind the ambient temperature conditions as stated in the data sheet of the instrument.

Store the reagents cold; Store the reagents in the dark; Refresh the reagents after one month (unless stated differently in the chapters below).

4.3 $KMnO_4$ solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Potassium permanganate	$KMnO_4$	158.03	7722-64-7	0.32 g

Preparation

Dissolve 0.32 g potassium permanganate ($KMnO_4$) in 500 mL de-ionized water. Fill up to 1 litre with de-ionized water. Heat and filter the solution.

4.4 Acid solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sulfuric acid 96%	H_2SO_4	101.19	7664-93-9	333 mL

Preparation

Add 333 mL of sulfuric acid (H_2SO_4 96%) in 500 mL de-ionized water. Fill up to 1 litre with de-ionized water. Add drops of the KMnO4 (section 4.3) on the hot acid solution until a faint red color remains about 60s, then cool the acid solution and complete until the mark of 1 litre.

4.5 Oxalate solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium oxalate	$C_2Na_2O_4$	134.00	62-76-0	1.675 g

Preparation

Dissolve 1.675 g sodium oxalate ($C_2Na_2O_4$) in 500 mL de-ionized water. Fill up to 1 litre with de-ionized water.

4.6 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium oxalate	$C_2Na_2O_4$	134.00	62-76-0	1.675 g

Preparation

200 mg/L O stock solution

Prepare a stock solution of 200 mg O/L : Dissolve accurately 1.675 g sodium oxalate (C_2NaO_4) in 500 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

O standard solution – REF2

Prepare a standard solution of 16 mg O/L: Take accurately 80 mL of the 200 mg O/L stock solution and transfer in a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

O standard solution – REF1

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

4.7 Validation solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
D(+)-glucose	$C_6H_{12}O_6$	180.16	50-99-7	1.676 g

Preparation

1000 mg O/L stock solution

Prepare a stock solution of 1000 mg O/L. Dissolve 1.676 g the D(+)-Glucose stock solution in a 1000 ml measuring flask and add de-ionized water up to the marked line. The COD value of this solution is equivalent to 1000 mg O/L.

10 mg O/L validation solution

Prepare a standard solution of 10 mg O/L. Take accurately 10 ml of 1000 mg O/L stock solution and transfer into a volumetric flask of 1000 ml. Add de-ionized water up to the mark grade.

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.