DOC313.53.94198

EZ2303 Total Manganese & Manganese (II) Analyser

Method and reagent sheets

05/2022, Edition 1.02

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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.

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3. Analysis method

Summary

The determination of Manganese (II) and total Manganese is based on two methods, combined in one analyser.

The Manganese (II) concentration is determined in the 'Mn' method. The total Manganese concentration is determined in the 'Total Mn' method. The concentration of all parameters is determined alternately in the 'Main'-method.

The calibration for Manganese (II) is performed in the 'Mn' method. The calibration for total Manganese is performed in the 'Total Mn' method.

Remark

The methods cannot be started at the same time.

3.1 Manganese (II)

Summary

The determination of the manganese concentration in water is based on the reaction of formaldoxime with ammonium hydroxide in an alkaline solution to an intense coloured orange-red complex. The absorption is measured at 450 nm.

EDTA and hydroxylamine hydrochloride (reducing reagent) are added to minimize the interference of iron (Fe^{2+} and Fe^{3+}).

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling the initial absorbance value is measured at 450 nm. This measurement is performed to correct for any colour contribution of the sample itself. Next, the colour solution and buffer solution are added and after respecting a stirring period – performed to obtain complete colour development – the EDTA and reducing agent are added. The final absorbance value is determined. With the obtained absorbance values, the manganese concentration can be calculated according to Beer's law.

Calibration

The calibration procedure measures a REF1 Mn solution (channel 9, REF1 valve) and a REF2 Mn solution (channel 10, REF2 valve) to adapt the slope and offset factors by means of a two point calibration.

3.2 Total Manganese

Summary

The determination of the manganese concentration in water is based on the reaction of formaldoxime with ammonium hydroxide in an alkaline solution to an intense coloured orange-red complex. The absorption is measured at 450 nm. Prior to the total manganese analysis, the sample is digested by use of an acid solution.

EDTA and hydroxylamine hydrochloride (reducing reagent) are added to minimize the interference of iron (Fe^{2+} and Fe^{3+}).

Analysis steps

The sample is mixed with the acid solution and heated to 120 °C (or up to 150 °C – programmable) in an oven during several minutes (standard 10 minutes; programmable up to 60 minutes). After digestion, the sample is cooled and transferred into the analysis vessel. The initial absorbance value is measured. Next, the buffer solution and the colour solution are added. After respecting a stirring period, reducing reagent and EDTA are added and stirred. The final absorbance value is determined. With the obtained absorbance values, the manganese concentration can be calculated according to Beer’s law.

Calibration

The calibration procedure measures a REF1 Mn solution (channel 9, REF1 valve) and a REF2 Mn solution (channel 10, REF2 valve) to adapt the slope and offset factors by means of a two point calibration.

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/30 min	Recommended containers
Acid solution (Total Mn)	~ 1.0 mL / analysis	~ 1.5 L	Plastic – 2.5 L
Buffer solution (Total Mn)	~ 1.5 mL / analysis	~ 2.7 L	Plastic – 5 L
Buffer solution (Mn)	~ 0.5 mL / analysis		
Colour solution (Total Mn)	~ 0.5 mL / analysis	~ 1.5 L	Plastic – 2.5 L
Colour solution (Mn)	~ 0.5 mL / analysis		
Reducing reagent (Total Mn)	~ 0.5 mL / analysis	~ 1.5 L	Plastic – 2.5 L
Reducing reagent (Mn)	~ 0.5 mL / analysis		
EDTA solution (Total Mn)	~ 0.5 mL / analysis	~ 1.5 L	Plastic – 2.5 L
EDTA solution (Mn)	~ 0.5 mL / analysis		
REF1 solution (Total Mn & Mn)	~ 1 L / calibration	/	Plastic – 1 L
REF2 solution (Total Mn & Mn)	~ 1 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/30 min
	Total Mn	**Mn (II)**	**Total Mn**	**Mn (II)**	**Total Mn + Mn (II)**	**Total Mn + Mn (II)**
A	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
B	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
C	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
0	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
1	55 mL	55 mL	20 mL	10 mL	140 mL	190 L
3	55 mL	55 mL	20 mL	10 mL	140 mL	190 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 Acid solution (1 M)

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Nitric acid (65%)	HNO_3	63.01	7697-37-2	69 mL

Preparation

Dilute 69 mL nitric acid (HNO_3, 65%) in 500 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
Ammonium hydroxide solution (25%)*	NH_4OH	35.05	1336-21-6	100 mL

* Density: 0.91 g/ml (20°C)

Preparation

Take 100 mL concentrated ammonium hydroxide (NH_4OH 25%) and dilute to 1 litre with de-ionized water. This solution is stable for 1 month.

4.6 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Hydroxylamine hydrochloride	H_3NOHCl	69.49	5470-11-1	40 g
Formaldehyde (37%)	CH_2O	30.03	50-00-0	20 mL

Preparation

Dissolve 40 g hydroxylamine hydrochloride (H_3NOCl) in 500 mL de-ionized water. Next, add 20 mL of formaldehyde (CH_2O 37%) solution. Fill up to 1 litre with de-ionized water. This solution is stable for 2 weeks.

4.7 Reducing reagent (1%)

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Hydroxylamine hydrochloride	H_3NOHCl	69.49	5470-11-1	10 g

Preparation

Dissolve 10 g of hydroxylamine hydrochloride (H_3NOHCl) in 500 mL de-ionized water and dissolve completely. Fill up to 1 litre with de-ionized water.

4.8 EDTA solution (0.1M)

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	37.22 g

*ethylenediaminetetraacetic acid disodium salt dihydrate

Preparation

Dissolve 37.22 g of ethylenediaminetetraacetic acid disodium salt in 500 mL de-ionized water and dissolve completely. Fill up to 1 litre with de-ionized water.

4.9 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Manganese(II) nitrate tetrahydrate	$MnN_2O_6 * 4H_2O$	251.01	20694-39-7	4.5688 g
Nitric acid (65%)	HNO_3	63.01	7697-37-2	35 mL

Preparation

1000 mg/L Mn stock solution

Prepare a stock solution of 1000 mg/L Mn: Dissolve accurately 4.5688 g manganese(II)nitrate tetrahydrate ($MnN_2O_6 * 4H_2O$) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Add 35 mL of concentrated nitric acid (HNO_3 65%). This addition is done to keep the solution stable. Fill up to 1 litre with de-ionized water.

Mn standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 1000 mg/L Mn 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	0.10 mg/L Mn	0.10 mg/L Mn	0.10 mL
B	0.25 mg/L Mn	0.25 mg/L Mn	0.25 mL
C	0.50 mg/L Mn	0.50 mg/L Mn	0.50 mL
0	**1.0 mg/L Mn**	**1.0 mg/L Mn**	**1.0 mL**
1	4.0 mg/L Mn	4.0 mg/L Mn	4.0 mL
3	10 mg/L Mn	10 mg/L Mn	10 mL

Mn standard solution – REF1

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

4.10 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: 25/05/2022	Previous version: Edition 1.01 to Edition 1.02
Reason for Change	
- Correction of CAS number of Nitric Acid	
Description of Change	
- Correction of CAS number of Nitric Acid from 7697-32-2 to 7697-37-2 (chapter 4.4 and 4.9)	