DOC313.53.94194

EZ2005 Total Iron 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.

Total Iron - All specifications				
Analysis method	Colorimetric measurement using TPTZ colour solution			
Parameter	Fe Total			
Cycle time	Standard measurement cycle time: 20 minutes Internal dilution: + 5 min. External dilution: + 5 – 10 min.			
Limit of detection (LOD)	≤ 5 µg/L			
Precision	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	Metal ions like cobalt [$(Co)^{2+}$)] > 0,05 mg/l, nickel [$(Ni)^{2+}$] > 2 mg/L. Strong oxidizing agents, cyanide [$(CN)^{-}$], nitrite [$(NO_2)^{-}$], chromium [Cr], Manganese [Mn], Molybdenum [Mo]. Copper [Cu] in excess of 0,6 mg/l, nickel [Ni] in excess of 2 mg/l. Cadmium [Cd], mercury [Hg] Large amounts of color and turbidity interferes. Fats, oil, proteins, surfactants and tar.			
Measuring ranges	**% of range - Dilution**		**Low range (mg/L)**	**High range (mg/L)**
	B	25% of standard range	0.005	0.25
	C	50% of standard range	0.005	0.50
	0	**standard range**	**0.01**	**1.0**
	1	internal MP dilution (factor 4)	0.08	4.0
	3	internal MP dilution (factor 10)	0.16	10
	4	internal MP dilution (factor 20)	2	20

3. Analysis method

Summary

Ferrous iron reacts with 2,4,6-tripyridyl-1,3,5-triazine in a mildly acidic medium (pH 4.5±0.2) to form a blue coloured complex. Ferric iron is reduced to ferrous iron using a reducing agent. The absorption is measured at a wavelength of 578 nm. Prior to the total iron analysis, the sample is digested by use of a reducing reagent.

Analysis steps

The sample is mixed with the reducing reagent 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). During the digestion process, the undissolved iron is converted to soluble iron. 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 is added. The final absorbance value is determined. With the obtained absorbance values, the iron concentration can be calculated according to Beer's law.

Calibration

The calibration procedure measures a REF1 Fe solution (channel 9, REF1 valve) and a REF2 Fe 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/20 min	Recommended containers
Buffer solution	~ 1.0 mL / analysis	~ 2.1 L	Plastic – 2.5 L
Colour solution	~ 0.8 mL / analysis	~ 1.7 L	Glass – 2.5 L
Reducing reagent	~ 0.8 mL / analysis	~ 1.7 L	Plastic – 2.5 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 rata 1 analysis/20 min
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	55 mL	20 mL	75 mL	152 L
3	55 mL	20 mL	75 mL	152 L
4	55 mL	20 mL	75 mL	152 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 Buffer solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Ammonium acetate	$C_2H_7NO_2$	77.08	631-61-8	250 g
Acetic acid	$C_2H_4O_2$	60.05	64-19-7	350 mL

Preparation

Dissolve 250 g ammonium acetate ($C_2H_7NO_2$) in 500 mL de-ionized water. Next, add 350 mL acetic acid ($C_2H_4O_2$), mix and fill up to 1 litre with de-ionized water. The pH value of the buffer solution should be 4.5±0.2.

4.5 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
2,4,6-tripyridyl-1,3,5-triazine	$C_{18}H_{12}N_6$	312.33	3682-35-7	0.750 g
Hydrochloric acid (1M)	HCl	34.46	7647-01-0	100 mL

Preparation

Dissolve 0.750 g of TPTZ (2,4,6-tripyridyl-1,3,5-triazine) in 500 mL de-ionized water. Next, add 100 mL of the Hydrochloric acid solution (HCl, 1M), mix and fill up to 1 litre with de-ionized water.

4.6 Reducing reagent

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Hydroxylamine hydrochloride	H_3NO * HCl	69.49	5470-11-1	100 g
Hydrochloric acid (1M)	HCl	34.46	7647-01-0	200 mL

Preparation

Dissolve 100 g of hydroxylamine hydrochloride (H_3NO * HCl) in 300 mL de-ionized water and dissolve completely. Add 200 mL of the hydrochloric acid solution (HCl, 1M). Mix and fill up to 1 litre with de-ionized water. The addition of hydrochloric acid is done to prevent the formation of iron hydroxides.

4.7 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Ammonium iron (II) sulphate hexahydrate	$(NH_4)_2Fe(SO_4)_2 * 6H_2O$	392.14	7783-85-9	7.021 g
Sulfuric acid (96%)	H_2SO_4	101.19	7664-93-9	5 mL

Preparation

1000 mg/L Fe stock solution

Prepare a stock solution of 1000 mg/L Fe: Dissolve accurately 7.021 g ammonium iron (II) sulphate hexahydrate [$(NH_4)_2Fe(SO_4)_2 * 6H_2O$] in 300 mL de-ionized water using a volumetric flask of 1000 mL. Add carefully 5 mL concentrated sulfuric acid (H_2SO_4, 96%). Add de-ionized water up to the mark grade. The addition of sulfuric acid is done to prevent the formation of iron hydroxides.

Fe standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 1000 mg/L Fe 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
B	0.25 mg/L Fe	0.25 mg/L Fe	0.25 mL
C	0.50 mg/L Fe	0.50 mg/L Fe	0.50 mL
0	**1.0 mg/L Fe**	**1.0 mg/L Fe**	**1.0 mL**
1	4.0 mg/L Fe	4.0 mg/L Fe	4.0 mL
3	10 mg/L Fe	10 mg/L Fe	10 mL
4	20 mg/L Fe	20 mg/L Fe	20 mL

Fe standard solution – REF1

Prepare a standard solution of 0 mg/L Fe. 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: 25/05/2022	Previous version: Edition 1.01 to Edition 1.02

Reason for Change

- Correction of LOD value

Description of Change

- Due to obsolescence of the 10% Range, the LOD value had to be corrected to 5 ppb instead of 2 ppb. This was not corrected in the previous version of the Method and Reagent sheet. (chapter 2)