DOC313.53.94408

EZ1035 Silica Analyser

Method and reagent sheets

09/2021, 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.

Silica - All specifications				
Analysis method	Colorimetric measurement using Molybdate colour solution at 630 nm			
Parameter	Si			
Cycle time	10 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	Tannin, large amounts of Iron, color, turbidity, Sulphide [$(S)^{2-}$] and Phospate [$(PO_4)^{2-}$] interfere. 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	0.5
	0	standard range	0.01	1.0
	1	internal MP dilution (factor 4)	0.08	4.0
	2	Internal MP dilution (factor 8)	0.16	8.0
	W	internal dispenser dilution (factor 10)	0.10	10
	X	internal dispenser dilution (factor 25)	0.25	25
	Y	internal dispenser dilution (factor 50)	0.50	50
	Z	internal dispenser dilution (factor 75)	0.75	75
	5	internal dispenser dilution (factor 100)	1.00	100

3. Analysis method

Summary

Silica reacts with molybdate in an acidic medium to form a green-yellow coloured complex that in its turn is converted to a blue complex by ammonium iron(II) sulphate hexahydrate. Oxalic acid is added to minimize the phosphate interference. The absorption is measured at a wavelength of 630 nm.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling and addition of the colour and oxalic acid solution the initial absorbance value is measured at 630 nm. Next, the reducing solution is 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 silica concentration can be calculated according to Beer's law.

Calibration

The calibration procedure measures a REF1 Si solution (channel 9, REF1 valve) and a REF2 Si 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/10 min	Recommended containers
Colour solution	~ 0.5 mL / analysis	~ 2.0 L	Plastic – 2.5 L
Oxalic acid solution	~ 0.5 mL / analysis	~ 2.0 L	Plastic – 2.5 L
Reducing reagent	~ 0.5 mL / analysis	~ 2.0 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 / 10 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	N.A.	N.A.	N.A.	N.A.
2	N.A.	N.A.	N.A.	N.A.
W	60 mL	15 mL	75 mL	302 L
X	60 mL	15 mL	75 mL	302 L
Y	60 mL	15 mL	75 mL	302 L
Z	60 mL	15 mL	75 mL	302 L
5	60 mL	15 mL	75 mL	302 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.

Reagent preparation

Since the measurement of silica is very sensitive to 'pollution' of the reagents by silica, it is advisable to use dedicated plasticware and plastic reagent containers to prepare and store the mentioned reagents. Always use the same plasticware and reagent containers. If necessary, rinse the plasticware and the reagent containers with a diluted acid solution. Afterwards, rinse with de-ionized water.

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 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Ammonium-hepta-molybdate-tetrahydrate	$(NH_4)_6Mo_7O_{24} * 4H_2O$	1235.86	12054-85-2	50 g
Sulfuric acid 96%	H_2SO_4	98.08	7664-93-9	50 mL

Preparation

Dissolve 50 g ammonium-hepta-molybdate tetrahydrate ($(NH_4)_6Mo_7O_{24} * 4H_2O$) in 500 mL de-ionized water. Next, add carefully 50 mL sulfuric acid (H_2SO_4, 96%), mix and fill up to 1 litre with de-ionized water. Store this solution in a plastic container.

4.5 Oxalic acid solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Oxalic acid dihydrate	$C_2H_2O_4 * 2H_2O$	126.07	6153-56-6	100 g

Preparation

Dissolve 100 g of oxalic acid dihydrate ($C_2H_2O_4 * 2H_2O$) in 500 mL de-ionized water. Mix and fill up to 1 litre with de-ionized water. Store this solution in a plastic container.

4.6 Reducing reagent

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	20 g
Sulfuric acid 96%	H_2SO_4	98.08	7664-93-9	12.5 mL

Preparation

Dissolve 20 g of ammonium iron(II) sulphate hexahydrate ($(NH_4)_2Fe(SO_4)_2 * 6H_2O$) in 500 mL de-ionized water and dissolve completely. Add carefully 12.5 mL sulfuric acid (H_2SO_4, 96%). Mix and fill up to 1 litre with de-ionized water. Store this solution in a plastic container.

4.7 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium metasilicate pentahydrate	$Na_2SiO_3 * 5H_2O$	212.14	10213-79-3	7.5534 g

Preparation

1000 mg/L Si stock solution

Prepare a stock solution of 1000 mg/L Si: Dissolve accurately 7.5534 g sodium metasilicate pentahydrate ($Na_2SiO_3 * 5H_2O$) in 500 mL de-ionized water using a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade. Store this solution in a plastic container.

Si standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 1000 mg/L Si stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade. Store this solution in a plastic container.

	Measuring range	Concentration REF2	Amount of stock solution to add to 1 litre
B	0.25 mg/L Si	0.25 mg/L Si	0.25 ml
C	0.50 mg/L Si	0.50 mg/L Si	0.50 ml
0	**1.0 mg/L Si**	**1.0 mg/L Si**	**1.0 ml**
1	4.0 mg/L Si	4.0 mg/L Si	4.0 ml
2	8.0 mg/L Si	8.0 mg/L Si	8.0 ml
W	10 mg/L Si	10 mg/L Si	10 mL
X	25 mg/L Si	25 mg/L Si	25 mL
Y	50 mg/L Si	50 mg/L Si	50 mL
Z	75 mg/L Si	75 mg/L Si	75 mL
5	100 mg/L Si	100 mg/L Si	100 mL

Si standard solution – REF1

Prepare a standard solution of 0 mg/L Si. Use de-ionized water. Store this solution in a plastic container.

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: 22/09/2021	Previous version: Edition 4 to Edition 1.01

Reason for Change

- Addition of extra ranges to the portfolio of EZ1035
- Addition of water consumption
- Addition of information reagents

Description of Change

- Addition of extra ranges for internal dispenser dilution: 10x, 25x, 50x, 75x (chapter 4.7)
- 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)