DOC313.53.94186

EZ1036 Sulphate Analyser

Method and reagent sheets

10/2022, Edition 1.02

Table of Contents

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.

Sulphate - All specifications				
Analysis method	Colorimetric measurement of turbidity at 450 nm after barium precipitation.			
Parameter	SO_4			
Cycle time	Standard measurement cycle time: 10 minutes Internal dilution: + 5 min. External dilution: + 10 min.			
Limit of detection (LOD)	≤ 10 mg/L			
Precision/Repeatability	Better than 3% 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	Other metals that form complexes with EDTA. Silica [SiO_2] > 500 mg/L, organic matter in water. Suspended or colloidal organic matter also may interfere with the endpoint. Large amounts of color and turbidity interfere. Fats, oil, proteins, surfactants and tar.			
Measuring ranges	**% of range - Dilution**		**Low range (mg/L)**	**High range (mg/L)**
	0	**standard range**	**10**	**40**
	1	internal MP dilution (factor 4)	40	160
	2	internal MP dilution (factor 8)	80	320
	W	internal dispenser dilution (factor 10)	100	400
	X	internal dispenser dilution (factor 25)	250	1000
	Y	internal dispenser dilution (factor 50)	500	2000
	Z	internal dispenser dilution (factor 75)	750	3000
	5	internal dispenser dilution (factor 100)	1000	4000

3. Analysis method

Summary

The determination of the sulphate concentration in water is based on a turbidimetric method. The sulphate ions is precipitated in an acidified medium with barium chloride so as to form barium sulphate crystals of uniform size. Light absorbance of the $BaSO_4$ suspension is measured at 450 nm.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling, the solution is acidified to pH 1.5 with the buffer solution. After a stabilisation period, the initial absorbance value is measured at 450 nm. Next, the barium chloride solution is added to the sample solution and after respecting a stirring period, the final absorbance value is measured. With the obtained absorbance values, the sulphate concentration is calculated according to Beer's Law. When the analysis is finished, the analysis vessel is drained and rinsed with de-ionized water and an EDTA solution is added in order to remove the white precipitation.

Calibration

The calibration procedure measures a REF1 SO_4 solution (channel 9, REF1 valve) and a REF2 SO_4 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
Buffer solution	~ 1 mL / analysis	~ 4.0 L	Plastic – 5 L
$BaCl_2$ solution	~ 1 mL / analysis	~ 4.0 L	Plastic – 5 L
EDTA solution	~ 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
0	N.A.	N.A.	N.A.	N.A.
1	60 mL	15 mL	75 mL	302 L
2	60 mL	15 mL	75 mL	302 L
U	60 mL	15 mL	75 mL	302 L
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.

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
Hydrochloric acid (37%)	HCl	36.46	7647-01-0	41.5 mL
Sodium chloride	NaCl	58.44	7647-14-5	50 g

Preparation

Dissolve 50 g of sodium chloride (NaCl) in 400 mL of de-ionized water and dissolve completely. Add 41.5 mL hydrochloric acid (HCl 37%) and dilute the solution to 1 litre with de-ionized water.

4.5 $BaCl_2$ solution (0.1M)

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Barium chloride dihydrate	$BaCl_2 * 2H_2O$	244.28	10326-27-9	24.428 g
Tween 80	/	/	9005-65-6	2.0 g
Ethanol, Pure	CH_3CH_2OH	46.07	64-17-5	20 mL

Preparation

1) Dissolve 24.428 g of barium chloride dihydrate ($BaCl_2 * 2H_2O$) in 500 mL de-ionized water using a volumetric flask of 1000 mL.
2) Dissolve 2.0 g of Tween 80 in 20 mL of ethanol.

Add solution (2) to solution (1). Mix and dissolve completely. Dilute to 1 litre with de-ionized water.

We recommend to use of Barium chloride with following specifications:

Product	Brand	CAS No.	Product No.
Barium chloride dihydrate	Sigma aldrich	10326-27-9	1.01719

4.6 EDTA solution (0.2M)

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
EDTA disodium salt dihydrate	$C_{10}H_{14}N_2Na_2O_8 * 2H_2O$	372.24	6381-92-6	74.4 g
Sodium hydroxide	NaOH	40.00	1310-73-2	40 g

Preparation

Dissolve 74.4 g of ethylenediaminetetraacetic acid disodium salt dihydrate ($C_{10}H_{14}N_2Na_2O_8 * 2H_2O$) in 500 mL de-ionized water using a volumetric flask of 1000 mL. Add carefully 40 g sodium hydroxide (NaOH). Dissolve completely and dilute to 1 litre with de-ionized water.

4.7 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium sulphate	Na_2SO_4	142.04	7757-82-6	29.57 g

Preparation

20000 mg/L SO_4 stock solution

Prepare a stock solution of 20000 mg/L SO_4. Dissolve 29.5716 g of sodium sulphate (Na_2SO_4) in 900 mL de-ionized water and dilute to 1 litre with de-ionized water

SO_4 standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 20000 mg/L SO_4. 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
0	**40 mg/L SO_4-**	**40 mg/L SO_4-**	**2 mL**
1	160 mg/L SO_4-	160 mg/L SO_4-	8 mL
2	320 mg/L SO_4-	320 mg/L SO_4-	16 mL
W	400 mg/L SO_4-	400 mg/L SO_4-	20 mL
X	1000 mg/L SO_4-	1000 mg/L SO_4-	50 mL
Y	2000 mg/L SO_4-	2000 mg/L SO_4-	100 mL
Z	3000 mg/L SO_4-	3000 mg/L SO_4-	150 mL
5	4000 mg/L SO_4-	4000 mg/L SO_4-	200 mL

SO_4 standard solution – REF1

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 20000 mg/L SO_4. stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

	Measuring range	Concentration REF1	Amount of stock solution to add to 1 litre
0	**40 mg/L SO_4-**	**15 mg/L SO_4-**	**0.75 mL**
1	160 mg/L SO_4-	60 mg/L SO_4-	3.00 mL
2	320 mg/L SO_4-	120 mg/L SO_4-	6.00 mL
W	400 mg/L SO_4-	150 mg/L SO_4-	7.50 mL
X	1000 mg/L SO_4-	375 mg/L SO_4-	18.75 mL
Y	2000 mg/L SO_4-	750 mg/L SO_4-	37.50 mL
Z	3000 mg/L SO_4-	1125 mg/L SO_4-	56.25 mL
5	4000 mg/L SO_4-	1500 mg/L SO_4-	75.0 mL

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.

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Hydrochloric Acid (36%)	HCl	36.46	7647-01-0	41.5 mL

Preparation

Prepare a 0.5 M hydrochloric acid (HCl) solution. Dilute 41.5 mL of hydrochloric acid (HCl 36%) in 500 mL de-ionized water using a volumetric flask of 1000 mL. Mix and fill up to the grade mark with de-ionized water.

Change Information	
Date: 04/10/2022	Previous version: Edition 1.01 to Edition 1.02
Reason for Change	
- Addition of information Barium chloride	
Description of Change	
- Addition of recommendation of supplier for Barium chloride (chapter 4.5)	