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EZ1037 Sulphide Analyser

Method and reagent sheets

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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.

Sulphide - All specifications				
Analysis method	Colorimetric measurement 670 nm			
Parameter	S ²⁻			
Cycle time	Standard measurement cycle time: 15 minutes Internal dilution: + 5 min. External dilution: + 5 – 10 min.			
Limit of detection (LOD)	≤ 50 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	Strong reducing agents like Hydrosulphide [(S ₂ O ₄) ²⁻], Nitrite [(NO ₂)] > 0,2 mg/l, Sulphite [(SO ₃) ²⁻] > 200 mg/l and Thiosulphate [(S ₂ O ₃) ²⁻] > 5 mg/l interfering by reducing the blue color. Oxygen reacts with Sulphide and must be avoided. Large amounts of color and turbidity interferes. Fats, Oil, Proteins, Surfactants and Tar.			
Measuring ranges	% of range - Dilution		Low range (µg/L)	High range (µg/L)
	0	standard range	50	500
	1	internal MP dilution (factor 4)	200	2000
	2	internal MP dilution (factor 8)	400	4000
	W	internal dispenser dilution (factor 10)	500	5000
	X	internal dispenser dilution (factor 25)	1250	12500
	Y	internal dispenser dilution (factor 50)	2500	25000
	Z	internal dispenser dilution (factor 75)	3750	37500
5	internal dispenser dilution (factor 100)	5000	50000	

3. Analysis method

Summary

The determination of the sulphide concentration in water is based on hydrogen sulphide and acid-soluble metal sulphide that react with N,N-dimethyl-p-phenylenediamine to form a blue complex. The absorption is measured at 670 nm.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling, the initial absorbance value is measured at 670 nm. Next, the buffer solution and colour solution are added to the sample solution and after respecting a stirring period, the final absorbance value is measured. With the obtained absorbance values, the sulphide concentration is calculated according to Beer's Law.

Calibration


The calibration procedure measures a REF1 S²⁻ solution (channel 9, REF1 valve) and a REF2 S²⁻ 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/20 min	Recommended containers
Buffer solution	~ 1.4 mL / analysis	~ 2.8 L	Glass - Amber – 2.5 L
Colour solution	~ 1.4 mL / analysis	~ 2.8 L	Glass - Amber – 2.5 L
Dilution solution (if applicable)	~ 7.5 mL / analysis	~ 15.2 L	Plastic – 10 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	Consumption/28 days A rata 1 analysis / 20 min
0	N.A.	N.A.
1	60 mL	121 L
2	60 mL	121 L
W	60 mL	121 L
X	60 mL	121 L
Y	60 mL	121 L
Z	60 mL	121 L
5	60 mL	121 L

	Dilution solution (mL/analysis) (chapter 4.6)	Consumption/28 days A rata 1 analysis / 20 min
0	N.A.	N.A.
1	6 mL	15.2 L
2	6 mL	15.2 L
W	7.5 mL	15.2 L
X	7.5 mL	15.2 L
Y	7.5 mL	15.2 L
Z	7.5 mL	15.2 L
5	7.5 mL	15.2 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
Iron (III) chloride hexahydrate	$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$	270.30	10025-77-1	4.866 g
Hydrochloric acid (37%)	HCl	36.46	7647-01-0	100 mL

Preparation

Dissolve 4.866 g of iron (III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in 400 mL of de-ionized water and dissolve completely. Add 100 mL hydrochloric acid (HCl, 37%) and dilute the solution to 1 litre with de-ionized water.

4.5 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
N,N-dimethyl-p-phenylenediamine	$(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{NH}_2$	136.19	99-98-9	4.1824 g
Hydrochloric acid (37%)	HCl	36.46	7647-01-0	600 mL

Preparation

Dissolve 4.1824 g of N,N-dimethyl-p-phenylenediamine ($(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{NH}_2$) in 100 mL de-ionized water using a volumetric flask of 1000 mL. Add 600 mL hydrochloric acid (HCl, 37%) and dilute to 1 litre with de-ionized water.

4.6 Dilution solution

Products	Formula	MW (g/mol)	CAS No.	
Zinc acetate dihydrate	$(\text{CH}_3\text{COO})_2\text{Zn} \cdot 2\text{H}_2\text{O}$	219.49	5970-45-6	8.7 g

Preparation

Dissolve 8.7 g zinc acetate dihydrate ($(\text{CH}_3\text{COO})_2\text{Zn} \cdot 2\text{H}_2\text{O}$) in 50 mL HPLC water using a volumetric flask of 100 mL and dilute to 100 mL with de-ionized water.

Take 0.5 mL of the zinc acetate solution and dilute to 1 litre with de-ionized water.

This solution is stable for maximum 2 weeks. Store the reagent in a fridge during operation to prolong stability.

We recommend to use of HPLC water with following specifications:

Product	Brand	CAS No.	Product No.	Specification
Water, HiPerSov CHROMANORM® for HPLC	VWR	7732-18-5	23595.400	Filtered through a 0.2 µm filter, packaged under nitrogen.

4.7 Calibration solution

Due to limited stability of sulphide in aqueous solutions, it is recommended to use freshly prepared stock and standard solutions for each calibration. The standard solutions (REF1 and REF2) must be used immediately after preparation. The stock solution is stable for at least one day after preparation when stored on a cool ($\pm 4^{\circ}\text{C}$), dark place.

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide	NaOH	40.00	1310-73-2	0.4 g
Sodium sulfide nonahydrate	Na ₂ S * 9H ₂ O	240.18	1313-84-4	3.745 g

Preparation

500 mg/L S²⁻ stock solution

Prepare a stock solution of 500 mg/L S²⁻. Dissolve 0.4 g of sodium hydroxide (NaOH) in 500 mL HPLC water and allow the solution to cool down. Add carefully 3.745 g sodium sulphide nonahydrate (Na₂S * 9H₂O) and dissolve completely. Fill up to 1 litre by use of (freshly boiled) de-ionized water.

S²⁻ standard solution – REF2

Prepare a standard solution for calibration according to the following table: take 0.5 mL of zinc acetate dehydrate solution (§4.5) and dissolve completely in 500 mL de-ionized water. Next, add accurately x mL of the 500 mg/L S²⁻ stock solution. Fill up to 1 litre with de-ionized water.

	Measuring range	Concentration REF2	Amount of stock solution to add to 1 litre
0	500 µg/L S ²⁻	500 µg/L S ²⁻	1 mL
1	2000 µg/L S ²⁻	2000 µg/L S ²⁻	4 mL
2	4000 µg/L S ²⁻	4000 µg/L S ²⁻	8 mL
W	5000 µg/L S ²⁻	5000 µg/L S ²⁻	10 mL
X	12500 µg/L S ²⁻	12500 µg/L S ²⁻	25 mL
Y	25000 µg/L S ²⁻	25000 µg/L S ²⁻	50 mL
Z	37500 µg/L S ²⁻	37500 µg/L S ²⁻	75 mL
5	50000 µg/L S ²⁻	50000 µg/L S ²⁻	100 mL

S²⁻ standard solution – REF1

Prepare a standard solution for calibration according to the following table: take 0.5 mL of zinc acetate dehydrate solution (§4.5) and dissolve completely in 500 mL de-ionized water. Next, add accurately x mL of the 500 mg/L S²⁻ stock solution. Fill up to 1 litre with de-ionized water.

	Measuring range	Concentration REF2	Amount of stock solution to add to 1 litre
0	50 µg/L S ²⁻	50 µg/L S ²⁻	0.10 mL
1	200 µg/L S ²⁻	200 µg/L S ²⁻	0.40 mL
2	400 µg/L S ²⁻	400 µg/L S ²⁻	0.80 mL
W	500 µg/L S ²⁻	500 µg/L S ²⁻	1.0 mL
X	1250 µg/L S ²⁻	1250 µg/L S ²⁻	2.5 mL
Y	2500 µg/L S ²⁻	2500 µg/L S ²⁻	5.0 mL
Z	3750 µg/L S ²⁻	3750 µg/L S ²⁻	7.5 mL
5	5000 µg/L S ²⁻	5000 µg/L S ²⁻	10 mL

We recommend to use of HPLC water with following specifications:

Product	Brand	CAS No.	Product No.	Specification
Water, HiPerSov CHROMANORM® for HPLC	VWR	7732-18-5	23595.400	Filtered through a 0.2 µm filter, packaged under nitrogen.

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: 30/09/2021	Previous version: Edition 5 to Edition 1.01
Reason for Change	
<ul style="list-style-type: none">- Addition of extra ranges to the portfolio of EZ1037- Addition of water consumption- Addition of information reagents	
Description of Change	
<ul style="list-style-type: none">- 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)- Addition of water specifications for preparation of calibration standards (chapter 4.7)	