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EZ4032 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.

Nitrite - All specifications			
Analysis method	Iodometric titration (I ₂)		
Parameter	S ²⁻		
Cycle time	10 minutes (No internal or external dilution possible)		
Limit of detection (LOD)	≤ 2 mg/L		
Precision/Repeatability	Better than 2% full scale range for standard test solutions		
Cleaning	Automatic; frequency freely programmable		
Calibration	Automatic; one point; frequency freely programmable		
Validation	Automatic; frequency freely programmable		
Interferences	Reducing agents such as sulphites, thiosulphate and heavy-metal ions interfere. They react with iodine, which contributes to positive errors. Oxidizable (organic) compounds can cause high results. Fats, Oil, Proteins, Surfactants and Tar.		
Measuring ranges	% of range - Dilution	Low range (mg/L)	High range (mg/L)
	A 10% of standard range	2	100
	B 25% of standard range	5	250
	C 50% of standard range	10	500
	0 standard range	20	1000

3. Analysis method

Summary

The sulphide (S^{2-}) concentration is determined by an iodometric back titration using a platinum electrode. An excess amount of iodine is dosed. Then the excess of iodine is titrated with a sodium thiosulphate solution. After determination of the end point, the sulphide concentration is calculated.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling the iodine solution is added, followed by the buffer solution. The iodometric titration with a sodium thiosulphate solution is performed until it reaches the endpoint of 220 mV. After the determination of the end point, the sulphide concentration in the sample is calculated.

Calibration


The calibration procedure measures a REF1 (channel 9, REF1) and a REF2 solution (channel 10, REF2 valve) to adapt the slope factor 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	~ 2.0 ml	~4.1 L	Plastic – 5 L
Iodine solution	~ 4.0 ml	~ 8.1 L	Plastic Dark – 10 L
Sodium thiosulphate solution	Depending on concentration*	40.3 L > Volume > 12.1 L	Plastic – 10 L
REF1 solution	~ 1 L / calibration	/	Plastic – 2.5 L
REF2 solution	~ 1 L / calibration	/	Plastic – 2.5 L

* Due to back titration: The higher the sulphide concentration, the lower the consumption.

4.2 Storage and quality of chemicals

Quality of chemicals

All chemicals should be of ACS grade or better. We recommend the use of pro analysis chemicals.

Quality of water

Reagent grade, sulphide- and oxygen-free de-ionized water must be used to prepare the chemical solutions and for rinse purposes.

Storage of Reagents

While operating the instrument, keep in mind the ambient temperature conditions as stated in the data sheet of the instrument.

Store the reagents cold; Store the reagents in the dark; Refresh the reagents after one month (unless stated differently in the chapters below).

4.3 Buffer solution (2N)

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sulfuric acid (96%)	H ₂ SO ₄	98.07	7664-93-9	56 ml

Preparation

Carefully dilute 56 ml of sulfuric acid (H₂SO₄, 96 %) in 500 ml of de-ionized water using a volumetric flask of 1000 ml. Full up to 1 litre with de-ionized water.

4.4 Iodine solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Potassium iodide	KI	166.00	7681-11-0	x g
Potassium iodate	KIO ₃	214.00	7758-05-6	x g
Sodium bicarbonate	NaHCO ₃	84.01	144-55-8	0.5 g

Preparation

Prepare a x M Iodine (I₂). Dissolve accurately x g potassium iodide (KI), x g potassium iodate (KIO₃) and 0.5 g sodium bicarbonate (NaHCO₃) in 500 mL de-ionized water and fill up to 1 litre.

	Measuring range	Concentration I ₂ solution	Amount KI to add to 1 litre	Amount KIO ₃ to add to 1 litre
A	100 mg/L S ²⁻	0.05 M	20 g	1.784 g
B	250 mg/L S ²⁻	0.125 M	50 g	4.459 g
C	500 mg/L S ²⁻	0.25 M	100 g	8.917 g
0	1000 mg/L S ²⁻	0.5 M	200 g	17.835 g

4.5 Sodium thiosulphate solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium thiosulfate pentahydrate	Na ₂ S ₂ O ₃ ·5H ₂ O	248.18	10102-17-7	x g

Preparation

Prepare a x M thiosulphate (Na₂S₂O₃). Dissolve accurately x g Sodium thiosulphate (Na₂S₂O₃) in 500 mL de-ionized water and fill up to 1 litre.

	Measuring range	Concentration Na ₂ S ₂ O ₃ solution	Amount Na ₂ S ₂ O ₃ to add to 1 litre
A	100 mg/L S ²⁻	0.025 M	6.20 g
B	250 mg/L S ²⁻	0.05 M	15.51 g
C	500 mg/L S ²⁻	0.125 M	31.02 g
0	1000 mg/L S ²⁻	0.25 M	62.05 g

4.6 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium thiosulfate pentahydrate	$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$	248.18	10102-17-7	154.82 g

Preparation

10000 mg/L S^{2-} stock solution

Prepare a stock solution of 10000 mg/L S^{2-} . Dissolve accurately 154.82 g sodium thiosulphate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 500 ml de-ionized water using a volumetric flask of 1000 ml. Fill up to 1 litre with de-ionized water.

Sulphide standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 10000 mg/L S^{2-} stock solution and transfer into a volumetric flask of 1 litre. Add de-ionized water up to the mark grade.

	Measuring range	Concentration REF2	Amount to add to 1 litre
A	100 mg/l S^{2-}	100 mg/l	10 ml
B	250 mg/l S^{2-}	250 mg/l	25 ml
C	500 mg/l S^{2-}	500 mg/l	50 ml
0	1000 mg/l S^{2-}	1000 mg/l	100 ml

4.7 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.