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## **EZ3519 Sulphide analyser**

Method and reagent sheets

01/2023, 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.

| Sulphide - All specifications   |  |                       |                         |                          |
|---------------------------------|--|-----------------------|-------------------------|--------------------------|
| <b>Analysis method</b>          | Discontinuous measurement by combined ion-selective electrode with standard addition   |                       |                         |                          |
| <b>Parameter</b>                | Sulphide   |                       |                         |                          |
| <b>Cycle time</b>               | Standard measurement cycle time: 10 minutes  |                       |                         |                          |
| <b>Limit of detection (LOD)</b> | ≤ 1.0 mg/L   |                       |                         |                          |
| <b>Precision/Repeatability</b>  | Better than 3% full scale range for standard test solutions  |                       |                         |                          |
| <b>Cleaning</b>                 | Automatic; frequency freely programmable   |                       |                         |                          |
| <b>Calibration</b>              | Automatic, 2-point; frequency freely programmable  |                       |                         |                          |
| <b>Validation</b>               | Automatic; frequency freely programmable   |                       |                         |                          |
| <b>Interferences</b>            | Silver [(Ag) <sup>+</sup> ] and mercury [(Hg) <sup>2+</sup> ] have very high interference and can only be tolerated in very low concentrations relative to the sulphide - ideally they should be absent. Fats, oil, proteins, surfactants and tar. |                       |                         |                          |
| <b>Measuring ranges</b>         | <b>% of range - Dilution</b>   |                       | <b>Low range (mg/L)</b> | <b>High range (mg/L)</b> |
|                                 | B  | 25% of standard range | 1.0                     | 25                       |
|                                 | C  | 50% of standard range | 2.0                     | 50                       |
|                                 | 0  | standard range        | 5.0                     | 100                      |

### 3. Analysis method

#### Summary

The Sulphide ( $S^{2-}$ ) concentration is determined by a standard addition using an ionselective electrode.

#### Analysis steps

The analysis vessel is drained and rinsed with fresh sample. A specific amount of sample is dosed into the analysis vessel. A TISAB [T(otal) I(onic) S(trength) A(djustment) B(uffer)] solution is added to the sample to adjust the pH and to assure the total ionic strength of the sample. The potential is measured using an ion selective electrode. Next a known volume of standard solution is added. The solutions are mixed and a second reading is taken. The analyzer calculates the initial ion concentration in the sample.

#### Calibration

The calibration procedure measures a REF1 Blank solution (channel 9, REF1 valve) to adapt the slope by means of a one point calibration.

The calibration is performed in the MAIN method.

#### Remark

The methods cannot be started at the same time.

## 4. Reagents

| <b>⚠ CAUTION</b>  |  |
|---|--|
|  | 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. |

| <b>⚠ CAUTION</b>  |  |
|---|--|
|  | 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<br>A rata 1 analysis/10 min | Recommended<br>containers |
|---|---|---|---------------------------|
| Buffer solution                           | ~ 2 mL / analysis                                 | ~ 8.1 L*  | Glass – 2.5 L             |
| Standard solution<br>Sulphide (dispenser) | ~ 1.0 mL / analysis<br>~ 2 à 4.5 mL / calibration | ~ 4.1 L*  | Glass – 2.5 L             |
| Blank solution (REF1)                     | ~ 1.0 L / calibration                             | /   | Plastic – 2.5 L           |

\*This solution is stable for maximum 2 weeks – It is advisable to replace the solution every week to guarantee the continuous functioning of the analyzer – store the solution in a dark glass bottle for optimum analysis results

### 4.2 DI-water overview and consumption

|   | Rinse water<br>(mL/analysis) Type I | Dilution water<br>(mL/analysis) Type I | Total<br>(mL/analysis) | Consumption/28 days<br>A rata 1 analysis / 5 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.   |

## 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 |
|------------------|--|------------|-----------|------------------|
| Sodium Hydroxide | NaOH   | 40.00      | 1310-73-2 | 200 g            |
| Ascorbic acid    | C <sub>6</sub> H <sub>8</sub> O <sub>6</sub> | 176.12     | 50-81-7   | 30 g             |
| Water, HPLC      | /  | /          | /         | /                |

### Preparation

Solution A: add 200 g sodium hydroxide (NaOH) in 500 mL HPLC water and dissolve completely. Be careful: this solution becomes hot. Let the solution cool down completely.

Solution B: add 30 g ascorbic acid (C<sub>6</sub>H<sub>8</sub>O<sub>6</sub>) in 250 mL HPLC water and dissolve completely.

Add solution B to solution A. Mix and fill up to 1 litre with HPLC water.

**The buffer solution should be colourless to yellow. If the colour of the solution is brown, it cannot be used. Replace the solution every week to guarantee optimal analysis results. Store this solution in a dark bottle.**

We recommend to use of HPLC water with following specifications:

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

## 4.5 Standard solution sulphide

| Products                    | Formula                                      | MW (g/mol) | CAS No.   | 1 litre solution |
|-----------------------------|--|------------|-----------|------------------|
| Sodium Hydroxide            | NaOH   | 40.00      | 1310-73-2 | 100 g            |
| Ascorbic acid               | C <sub>6</sub> H <sub>8</sub> O <sub>6</sub> | 176.12     | 50-81-7   | 18 g             |
| Sodium salicylate           | HOC <sub>6</sub> H <sub>4</sub> COONa        | 160.00     | 54-21-7   | 80 g             |
| Sodium sulphide nonahydrate | Na <sub>2</sub> S * 9H <sub>2</sub> O        | 240.18     | 1313-84-4 | x g              |
| Water, HPLC                 | /  | /          | /         | /                |

### Preparation

#### x mg/L Sulphide standard solution (dispenser)

Prepare a stock solution of x mg/L Sulphide:

Solution A: add 100 g sodium hydroxide (NaOH) in in 400 mL HPLC water and dissolve completely. Be careful: this solution becomes hot. Let the solution cool down completely before mixing the solution in the next steps.

Solution B: add 18 g ascorbic acid (C<sub>6</sub>H<sub>8</sub>O<sub>6</sub>) in 200 mL HPLC water and dissolve completely.

Solution C: add 80 g sodium salicylate (HOC<sub>6</sub>H<sub>4</sub>COONa) in 200 mL HPLC water and dissolve completely.

Solution D: add x g sodium sulphide nonahydrate (Na<sub>2</sub>S \* 9H<sub>2</sub>O) in 100 mL HPLC water and dissolve completely. Follow instructions in table below for the exact amount of sodium sulphide nonahydrate (Na<sub>2</sub>S \* 9H<sub>2</sub>O)

| Measuring range                | Concentration Standard          | Amount Na <sub>2</sub> S * 9H <sub>2</sub> O to add |
|--------------------------------|---------------------------------|---|
| 25 mg/L S <sup>2-</sup>        | 250 mg/L S <sup>2-</sup>        | 1.873 g   |
| 50 mg/L S <sup>2-</sup>        | 500 mg/L S <sup>2-</sup>        | 3.745 g   |
| <b>100 mg/L S<sup>2-</sup></b> | <b>1000 mg/L S<sup>2-</sup></b> | 7.489 g   |

Add solution C to solution A and dissolve completely. Next add solution B and dissolve completely. At last add solution D and dissolve completely.

**The standard solution should be colourless to yellow. If the colour of the solution is brown, it cannot be used. Replace the solution every week to guarantee optimal analysis results. Store this solution in a dark bottle. Don't insert tubing completely to the bottom of the container due to possible precipitation.**

We recommend to use of HPLC water with following specifications:

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

## 4.6 Calibration solution

### Blank solution - REF1

Use HPLC water.

We recommend to use of HPLC water with following specifications:

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

## 4.7 Validation solution

### Sulphide validation solution

Prepare a standard solution according to the following table: take accurately x mL of the 1000 mg/L Sulphide standard solution and transfer into a volumetric flask of 1000 mL. Add HPLC water up to the mark grade.

| Measuring range                | Concentration Standard         | Amount of stock solution to add to 1 litre |
|--------------------------------|--------------------------------|--|
| 25 mg/L S <sup>2-</sup>        | 25 mg/L S <sup>2-</sup>        | 25 mL                                      |
| 50 mg/L S <sup>2-</sup>        | 50 mg/L S <sup>2-</sup>        | 50 mL                                      |
| <b>100 mg/L S<sup>2-</sup></b> | <b>100 mg/L S<sup>2-</sup></b> | <b>100 mL</b>                              |

### Preparation

#### 1000 mg/L Sulphide standard solution

| Products                    | Formula                                      | MW (g/mol) | CAS No.   | 1 litre solution |
|-----------------------------|--|------------|-----------|------------------|
| Sodium Hydroxide            | NaOH   | 40.00      | 1310-73-2 | 100 g            |
| Ascorbic acid               | C <sub>6</sub> H <sub>8</sub> O <sub>6</sub> | 176.12     | 50-81-7   | 18 g             |
| Sodium salicylate           | HOC <sub>6</sub> H <sub>4</sub> COONa        | 160.00     | 54-21-7   | 80 g             |
| Sodium sulphide nonahydrate | Na <sub>2</sub> S * 9H <sub>2</sub> O        | 240.18     | 1313-84-4 | 7.489 g          |

Prepare a stock solution of 1000 mg/L Sulphide:

Solution A: add 100 g sodium hydroxide (NaOH) in in 400 mL HPLC water and dissolve completely. Be careful: this solution becomes hot. Let the solution cool down completely before mixing the solution in the next steps.

Solution B: add 18 g ascorbic acid (C<sub>6</sub>H<sub>8</sub>O<sub>6</sub>) in 200 mL HPLC water and dissolve completely.

Solution C: add 80 g sodium salicylate (HOC<sub>6</sub>H<sub>4</sub>COONa) in 200 mL HPLC water and dissolve completely.

Solution D: add 7.489 g sodium sulphide nonahydrate ( $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ ) in 100 mL HPLC water and dissolve completely.

Add solution C to solution A and dissolve completely. Next add solution B and dissolve completely. At last add solution D and dissolve completely.

**The standard solution should be colourless to yellow. If the colour of the solution is brown, it cannot be used. Replace the solution every week to guarantee optimal analysis results. Store this solution in a dark bottle.**

We recommend to use of HPLC water with following specifications:

| Product                                 | Brand | CAS No.   | Product No. | Specification  |
|---|-------|-----------|-------------|--|
| Water, HiPerSov<br>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.

| <b>Change Information</b>  |   |
|--|---|
| Date: 17/01/2023   | Previous version: Edition 4 to Edition 1.01 |
| <b>Reason for Change</b>   |   |
| <ul style="list-style-type: none"><li>- Addition of water consumption</li><li>- Addition of information reagents</li><li>- Correction in preparation of validation solution</li></ul>  |   |
| <b>Description of Change</b>   |   |
| <ul style="list-style-type: none"><li>- Addition of estimated consumption of water for rinse and dilution (chapter 4.2)</li><li>- Addition of extra information regarding storage and quality of reagents (chapter 4.3)</li><li>- Correction in preparation of validation solution, typo in description: 100 mg/L corrected to 1000 mg/L</li></ul> |   |