



## DETERMINATION OF ZINC CONTENT IN WATER SAMPLES

### INTRODUCTION

The method is designed to measure mass concentration of zinc in samples of natural, drinking and waste water by fluorometric method using the FLUORAT®-02 analyzer.

### MEASUREMENT METHOD

Fluorescence method of measuring zinc mass concentration is based on determination of a coordination compound of zinc ions with 8-mercapto-quinoline in the acetate buffer medium (pH 4.6–4.9). The coordination compound is extracted by chloroform, and the change in the fluorescence intensity of the extract using the FLUORAT®-02 analyzer. The 8,8'-diquinolyl disulfide is used to eliminate the interfering influence of copper, and 1,10-phenanthroline is used to eliminate the interference of iron.



### CONCENTRATION RANGES

The range of measured zinc concentrations is **0.005–2.0 mg/l**.

The table gives acceptable contents of other ions.

Ions	Maximum content
Nitrates	Up to 1 g/l
Sulfates	Up to 1 g/l
Chlorides	Up to 1 g/l
Alkaline, alkaline-earth elements	Up to 1 g/l
Aluminium	Up to 1 g/l
Copper	Up to 20 mg/l
Lead	Up to 2 mg/l
Iron	Up to 20 mg/l
Magnesium	Up to 1 mg/l
Cobalt	Up to 1 mg/l
Nickel	Up to 1 mg/l

In the case of larger content of interfering agents, it may be necessary to use another method.

### EQUIPMENT AND REAGENTS

The following equipment and reagents are required for measurements:

- FLUORAT®-02 Analyzer
- Certified zinc ion solution with a mass concentration of 1 g/l
- Double distilled water
- 8-Mercapto-quinoline sodium salt, dihydrate UPG
- Ascorbic acid
- Sodium acetate, trihydrate UPG
- 66% Nitric acid UPG
- Acetic acid UPG
- 30% Hydrogen peroxide solution UPG
- 1,10-Phenanthroline
- Chloroform AG
- Ammonium hydroxide solution UPG
- 8,8'-Qiquinolyl disulfide UPG
- Ethyl alcohol



## PREOPERATIONAL PROCEDURES

Preoperational procedures include the following: sample collection and conservation, calibration of the FLUORAT<sup>®</sup>-02 analyzer, and control of the calibration characteristic linearity.

### Sample collection

Samples of natural, drinking or waste water are collected according to ISO 5667. Volume of the taken sample should be not less than 250 ml, with the expected zinc concentration at a level of not less than 0.2 mg/l. In the case of higher expected concentrations, the volume of the taken sample should be not less than 50 ml. For transportation and storage of the samples, PE or PTFE containers should be used. To conserve the samples, concentrated nitric acid is added in proportion of 3 ml for one liter of the sample. The conserved sample can be stored for 3 days. A non-conserved sample should be analyzed within 4 hours after collection. If the sample was not conserved, it has to be acidified prior to the analysis by 0.7% nitric acid in the proportion of 3 ml for one liter of the sample.

### Device calibration

The analyzer is calibrated by registration of fluorescence of the aqueous extract complex solution with known content of zinc ions (0.0 mg/l and 1.0 mg/l). 5 ml of zinc solution are transferred into a 50 mL separation funnel, and 1 mL of ascorbic acid solution (2 g/l), 5 ml of acetate buffer solution (pH 4.6–4.9), 1 ml of phenantroline solution (5 g/l) and 1 ml of sodium 8-mercapto-quinolate solution (1 g/l) should be added. After two minutes, 5 ml of chloroform are added and within one minute, zinc 8-mercapto-quinolate should be extracted. After the layers separate, the lower layer may be filtered through the “red ribbon” filter directly to the cuvette of the FLUORAT<sup>®</sup>-02 analyzer. For analyzer calibration and all measurements light filter No 11 is used in the activation channel, and the light filter No 5 is used in the registration channel.

### Sample treatment

The volume of the analyzed sample is 5–50 ml. The sample is treated similar to the treatment of the calibration solution.

## MEASUREMENTS

The measurement of the mass concentration of zinc in samples by fluorescence method is performed after destruction of organic substances, masking interfering agents, and the preparation of sample extractions.

## EXAMPLE OF REAL ANALYSIS

### Calibration diagram:

Zinc (FLUORAT<sup>®</sup>-02-3M)

C	J
0.00	0.0115
1.00	0.3197

### Measurement results:

Sample	Content, mg/l		
	Sample	Additive	Sample with additive
Drinking water, St Petersburg	0.784	1.0	1.65
The Sestra river	0.045	0.25	0.298
The Sestra river	0.041	0.75	0.799

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