INTERNATIONAL STANDARD

**ISO 16264**

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# Water quality — Determination of soluble silicates by flow analysis (FIA and CFA) and photometric detection

*Qualité de l'eau — Dosage des silicates solubles par analyse en flux (FIA et CFA) et détection photométrique*

Reference number
ISO 16264:2002(E)

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# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16264 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical, biochemical methods*.

# Introduction

Further investigation will be necessary to determine whether and to what extent particular problems will require the specification of additional minor conditions.

It is absolutely essential that tests conducted according to this International Standard be carried out by suitably qualified staff.

Differentiation is required between flow injection analysis (FIA)[1], [2], and continuous flow analysis (CFA) [3]. Both methods share the feature of an automatic dosage of the sample into a flow system (manifold) where the analytes in the sample react with the reagent solutions on their way through the manifold. The sample preparation may be integrated into the manifold. The reaction product is determined in a flow detector (e.g. photometer). This detector produces a signal from which the concentration of the parameter can be calculated.

Methods using flow analysis automate wet chemical procedures and are particularly suitable for processing many analytes in water in large sample series at a high analysis frequency.

# Water quality — Determination of soluble silicates by flow analysis (FIA and CFA) and photometric detection

**WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.**

## 1 Scope

This International Standard specifies two methods, i.e. flow injection analysis (FIA) and continuous flow analysis (CFA), for the determination of soluble silicate ions in various types of water (such as ground, drinking, surface, leachate and waste water). Both methods are applicable to the determination of a mass concentration of silicate ($SiO_2$) ranging from 0,2 mg/l to 20 mg/l (with working ranges 0,2 mg/l to 2,0 mg/l and 2 mg/l to 20 mg/l). Other mass concentration ranges are applicable, provided they cover exactly one decade of concentration units (e.g. 0,02 mg/l to 0,2 mg/l in $SiO_2$).

These methods can be made applicable to seawater by changing the sensitivity and by adapting the reagent and calibration solutions to the salinity of the samples.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 8466-1:1990, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

## 3 General interferences

Tannin, large amounts of iron, sulfide and phosphate interfere. The treatment with oxalic acid reduces the interference from tannin and eliminates interference from phosphate provided the mass concentrations are $< 20$ mg/l. If this value is exceeded, the amount of oxalic acid may not be sufficient to destroy all molybdophosphoric acid being formed. In this case, dilute the sample with water (5.1).

When aluminium ions are present in the water sample, the addition of oxalic acid is not completely effective in eliminating phosphate interference. In this case, add 0,2 ml of a 0,1 mol/l sodium cyanide solution per litre of sample prior to analysis.

If the sample contains fluoride in concentrations $> 150$ mg/l, treat the samples with boric acid.

High concentrations of oxidizing agents are destroyed by adding 1 mol/l of sodium sulfite solution prior to analysis.

If the sample contains large concentrations of sulfide, e.g. $> 5$ mg/l, special provisions are necessary.

# 4 Principle

## 4.1 FIA method

The sample is injected into a carrier stream of water by means of a valve. An acidic solution of heptamolybdate is added to the sample and reacts with silicates and phosphates to form molybdosilicate and molybdophosphoric acid. The latter is destroyed by oxalic acid. The molybdosilicate is then reduced to molybdenum blue using a tin(II) chloride solution. The result is expressed in milligrams of $SiO_2$ per litre of water.

## 4.2 CFA method

An acidic solution of heptamolybdate is added to the sample and reacts with silicates and phosphates to form molybdosilicate and molybdophosphoric acid. The latter is destroyed by oxalic acid. The molybdosilicate is then reduced to molybdenum blue using ascorbic acid. The result is expressed in milligrams of $SiO_2$ per litre of water.

# 5 Reagents

Use analytical grade chemicals unless otherwise specified. Check the silicate blank value of the water and of the reagents (8.2.1). Carefully degas all reagent solutions, e.g. by vacuum filtration or by purging with helium (about 1 min).

Use only polyethene or polypropene laboratory ware (flasks, beakers and pipettes) for the preparation and storage of the samples (see clause 7), water (5.1) and the solutions 5.8, 5.9 and 5.10.

**5.1 Water**, complying with purity grade 1 of ISO 3696.

**5.2 Sulfuric acid** ($H_2SO_4$), $\rho(H_2SO_4)$ = 1,84 g/ml.

**5.3 Molybdate solution**, (see R1 in Figures 1 and 2).

In a 1 000 ml volumetric flask, dissolve (30 ± 0,3) g of hexaammonium heptamolybdate tetrahydrate, $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$, in about 800 ml of water (5.1). Add 15 ml of sulfuric acid (5.2) and dilute to volume with water.

The solution may be stored for up to 3 months at room temperature. Discard the solution if precipitation or coloration occurs.

**5.4 Oxalic acid solution**, (see R2 in Figures 1 and 2).

In a 1 000 ml volumetric flask, dissolve (44 ± 0,4) g of oxalic acid dihydrate, $C_2H_2O_4{\cdot}2H_2O$, in about 800 ml of water (5.1) and dilute to volume with water.

The solution may be stored up to 3 months at room temperature.

**5.5 Tin(II) chloride reagent**, for the determination of silicate by FIA (see R3 in Figure 1).

Slowly add 28 ml of sulfuric acid (5.2) to 800 ml water (5.1) in a 1 000 ml volumetric flask. Dissolve (240 ± 2) mg of tin(II) chloride dihydrate, $SnCl_2{\cdot}2H_2O$, and (2 ± 0,02) g of hydrazine sulfate, $N_2H_6SO_4$, in this solution. Cool to room temperature and dilute to volume with water.

The solution is stable for 1 month if stored at (4 ± 2) °C.

**5.6 Ascorbic acid solution**, for the determination of silicate using CFA (see R3 in Figure 2).

Dissolve (4 ± 0,04) g of ascorbic acid, $C_6H_8O_6$, in about 80 ml of water (5.1) and dilute to 100 ml with water.

Prepare the solution freshly before use.

**5.7 Surfactant solutions for CFA systems:**

**5.7.1 Surfactant solution I**, for a working range I (0,2 mg/l to 2,0 mg/l; see C in Figure 2).

In a 1 000 ml volumetric flask, dissolve (10 ± 0,1) g of sodium dodecyl sulfate, $NaC_{12}H_{25}O_4S$, in about 800 ml of water (5.1) and dilute to volume with water.

The solution may be stored for up to 6 months at room temperature.

**5.7.2 Surfactant solution II**, for working range II (2 mg/l to 20 mg/l; see B in Figure 2).

In a 1 000 ml volumetric flask, dissolve (1 ± 0,01) g of sodium dodecyl sulfate, $NaC_{12}H_{25}O_4S$, in about 800 ml of water (5.1) and dilute to volume with water.

The solution may be stored for up to 6 months at room temperature.

**5.8 Alkaline silicate solution**, containing 0,500 0 g/l, expressed as $SiO_2$ (e.g. $SiCl_4$ in 0,1 mol/l of sodium hydroxide solution, NaOH, or sodium metasilicate nonahydrate, $Na_2SiO_3 \cdot 9H_2O$).

The solution is stable for 6 months if stored at room temperature.

NOTE A typical procedure for preparing the alkaline silicate solution from silicium tetrachloride, $SiCl_4$ is the following.

While cooling intensively, dissolve (1,415 ± 0,001) g of $SiCl_4$ for synthesis (commercially available). In small portions in about 900 ml of 0,1 mol/l sodium hydroxide solution, NaOH. Dilute to volume with 0,1 mol/l sodium hydroxide solution.

**5.9 Silicate stock solution**, $\rho$ = 50 mg/l (expressed as $SiO_2$).

In a 1 000 ml volumetric flask, dilute 100 ml of alkaline silicate solution (5.8) with water (5.1) and dilute to volume with water.

The solution is stable for 6 months if stored at room temperature.

**5.10 Calibration solutions.**

Prepare at least five calibration solutions by diluting the silicate stock solution (5.9) with water (5.1) for the required range. The concentrations of the solutions shall be distributed equidistantly over the corresponding working range (see ISO 8466-1).

Tables 1 and 2 give examples for the preparation of ten calibration solutions for the working ranges I (0,2 mg/l to 2,0 mg/l) and II (2 mg/l to 20 mg/l $SiO_2$).

**Table 1 — Typical preparation scheme of silicate calibration solutions for working range I (0,2 mg/l to 2,0 mg/l $SiO_2$)**

| Volume (ml) of silicate stock solution (5.9) diluted to 1 000 ml | 4 | 8 | 12 | 16 | 20 | 24 | 28 | 32 | 36 | 40 |
|---|---|---|---|---|---|---|---|---|---|---|
| Mass concentration of calibration solutions (mg/l of $SiO_2$) | 0,2 | 0,4 | 0,6 | 0,8 | 1,0 | 1,2 | 1,4 | 1,6 | 1,8 | 2,0 |
| These calibration solutions are stable for 1 month if stored at 4 °C to 10 °C. | | | | | | | | | | |

**Table 2 — Typical preparation scheme of silicate calibration solutions for working range II (2 mg/l to 20 mg/l $SiO_2$)**

| Volume (ml) of silicate stock solution (5.9) diluted to 100 ml | 4 | 8 | 12 | 16 | 20 | 24 | 28 | 32 | 36 | 40 |
|---|---|---|---|---|---|---|---|---|---|---|
| Mass concentration of calibration solutions (mg/l of $SiO_2$) | 2 | 4 | 6 | 8 | 10 | 12 | 14 | 16 | 18 | 20 |
| These calibration solutions are stable for 6 months if stored at 4 °C to 10 °C. | | | | | | | | | | |

**5.11 Rinsing solution.**

In a 1 000 ml volumetric flask, dissolve 65 g of sodium hydroxide (NaOH) in about 500 ml of water (5.1). Add 6 g of ethylenediaminetetraacetic acid disodium salt dihydrate, $Na_2EDTA$, $C_{10}H_{12}O_8N_2Na_2{\cdot}2H_2O$ and shake to dissolve. Dilute to volume with water.

This solution is stable for 6 months if stored at room temperature.

## 6 Apparatus

**6.1 Flow injection analysis system (FIA),** usually consisting of the following components. A typical schema of the system is shown in Figure 1.

**6.1.1 Reagent containers**.

**6.1.2 Low pulse pump**.

**6.1.3 Calibrated pump tubes**, if required.

**6.1.4 Sample injection system**, with an injection volume of, for example, 40 µl and 360 µl.

**6.1.5 Transport tubes**, with an internal diameter of 0,5 mm to 0,8 mm, **tube connections** and **T-connections**, made of inert material and having minimum dead volumes.

**6.1.6 Photometric detector with flow cell**, with a wavelength range of 720 nm ± 10 nm.

The optical path length of the flow cell shall be chosen so that the linearity of the calibration curve is not limited by the maximum measurable absorbance.

**6.1.7 Recorder unit** (e.g. strip chart recorder, integrator or printer/plotter), generally used to evaluate the peak height signals.

**6.1.8 Autosampler**, if required.

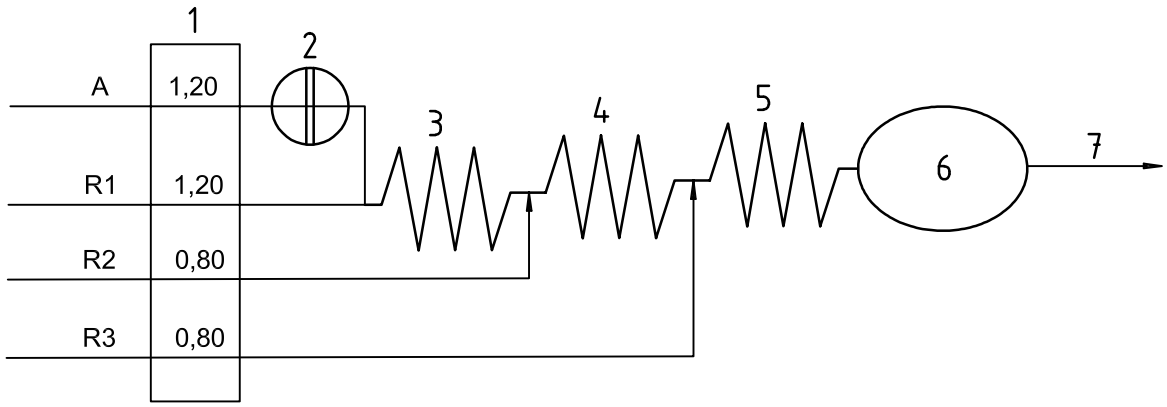
**6.2 Continuous flow analysis system (CFA)**, usually consisting of the following components. A typical schema of the system is shown in Figure 2.

**6.2.1 Automatic sampler**, which allows reproducible sample introduction and sample transport.

**6.2.2 Reagent containers**.

**6.2.3 Low pulse pump**, with calibrated chemical-resistant pump tubing.

**6.2.4 Inlet connector**, made of glass or a chemically resistant material, with reproducible air-, sample- and reagent segmentation and with calibrated transport tubing.




**Key**

1 Pump (flow rates in ml/min)
2 Injector (sample volume of 360 µl for working range I)
(sample volume of 40 µl for working range II)
3 Reaction coil (internal diameter 0,7 mm, length 140 cm, maintained at 70 °C)
4 Reaction coil (internal diameter 0,7 mm, length 180 cm)
5 Reaction coil (internal diameter 0,7 mm, length 60 cm)
6 Photometric detector (wavelength 720 nm ± 10 nm, optical path length 10 mm)
7 Waste
A Water (5.1)
R1 Molybdate solution (5.3)
R2 Oxalic acid solution (5.4)
R3 Tin(II) chloride reagent (5.5)

Working range I: 0,2 mg/l to 2,0 mg/l of $SiO_2$
Working range II: 2 mg/l to 20 mg/l of $SiO_2$

**Figure 1 — Typical schema of an FIA system used for the determination of silicate ($SiO_2$) within working ranges I and II**

**6.2.5 Transport tubing**, **reaction coils**, **tube connections** and **T-connections,** made of inert material and with minimum dead volumes.

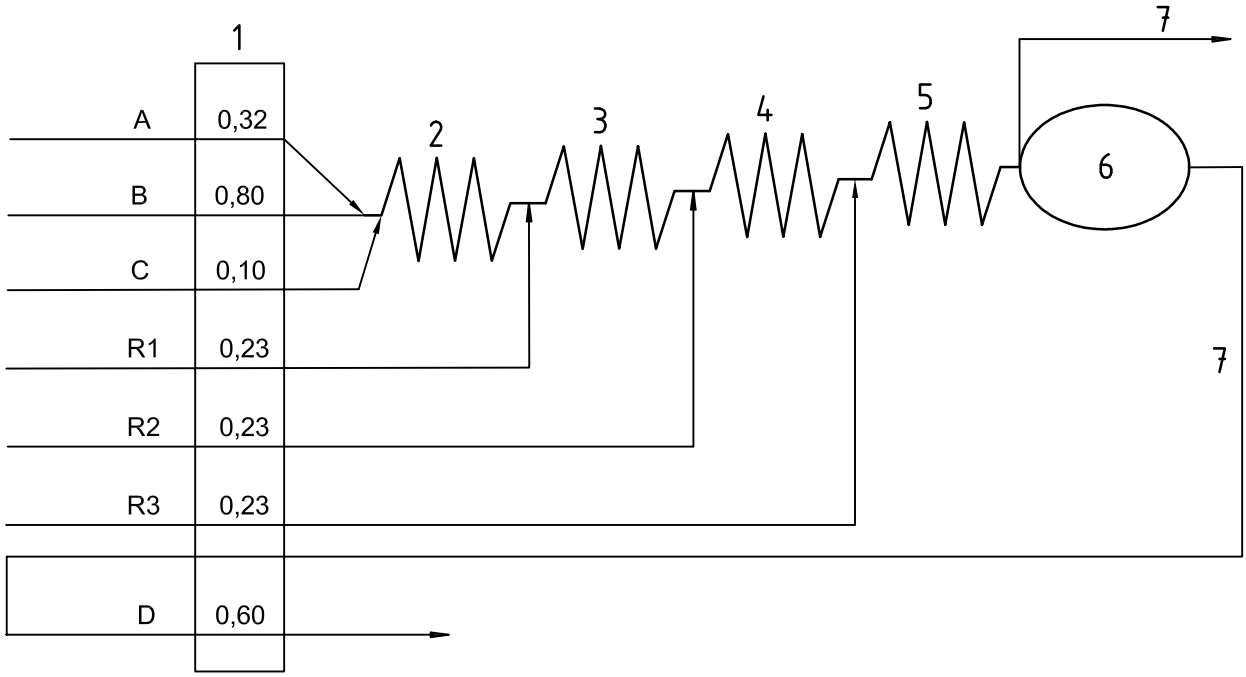
Figure 2 shows a CFA system with 2 mm internal diameter tubing. Other tubing diameters (e.g. approximately 1 mm) may be used as long as the flow rates are in the same proportion.

**6.2.6 Thermostat** (optional), for maintaining the test system at a temperature of 37 °C.

**6.2.7 Photometric detector with flow cell,** with a wavelength range of 810 nm ± 10 nm.

The optical path length of the flow cell shall be chosen so that the linearity of the calibration curve is not limited by the maximum measurable absorbance.

**6.2.8 Recorder unit,** (e.g. strip chart recorder, integrator or printer/plotter) generally used to evaluate peak height signals.




**Key**

1 Pump (flow rates in ml/min)
2 Reaction coil (internal diameter 2 mm, length 10 cm)
3 Reaction coil (internal diameter 2 mm, length 30 cm)
4 Reaction coil (internal diameter 2 mm, length 30 cm)
5 Reaction coil (internal diameter 2 mm, length 90 cm, thermostatted at 37 °C)
6 Detector (wavelength 810 nm ± 10 nm, optical path length 10 mm)
7 Waste
A Air
B Sample for working range I; surfactant solution II (5.7.2) for working range II
C Surfactant solution I (5.7.1) for working range I; sample for working range II
D Degassed waste
R1 Molybdate solution (5.3)
R2 Oxalic acid solution (5.4)
R3 Ascorbic acid solution (5.6)

Working range I: 0,2 mg/l to 2,0 mg/l $SiO_2$
Working range II: 2 mg/l to 20 mg/l $SiO_2$

**Figure 2 — Typical schema of a CFA system for the determination silicate ($SiO_2$) in working ranges I and II**

### 6.3 Additional apparatus:

**6.3.1 Polyethene** or **polypropene laboratory ware** (flasks, beakers and pipettes) for the preparation and storage of water (5.1) and the solutions 5.8, 5.9 and 5.10 and consisting of the following.

**6.3.1.1 Graduated flasks**, of 100 ml, 200 ml, 500 ml and 1 000 ml nominal capacities.

**6.3.1.2 Pipettes**, of 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 10 ml, 20 ml and 25 ml nominal capacities.

**6.3.1.3 Beakers**, of 25 ml, 100 ml and 1 000 ml nominal capacities.

**6.3.2 Laboratory glassware**, for all other solutions and reagents than those mentioned in 6.3.1.

If a working range below 0,2 mg/l $SiO_2$ is applied, it is recommended to use only polyethene or polypropene material specified in 6.3.1 for the preparation and storage of all reagents and solutions.

**6.3.3 Membrane filter assembly** with **membrane filters**, having a pore size of 0,45 µm.

# 7 Sampling and sample preparation

Prior to use, rinse all containers thoroughly with water (5.1).

Take the samples without adding any reagents using plastic containers for all samples.

Filter the samples through a membrane filter (6.3.3) immediately after sampling. Filtration prevents the interference from sulfides as well as clogging the analyser tubing (in the case of particles larger than 100 µm).

Analyse the sample as soon as possible. If necessary, store the samples at temperatures between 4 °C to 25 °C. They should not be stored below 4 °C so as to avoid the formation of polysilicates. A maximum preservation time of 24 h is recommended.

# 8 Procedure

## 8.1 Pretreatment

Set up the flow analysis system for the desired procedure (for FIA, see 6.1 and Figure 1, for CFA, see 6.2 and Figure 2).

In the case of FIA, pump the reagents 5.1, 5.3, 5.4 and 5.5 through the system for 10 min and set the baseline to zero. In the case of CFA, pump the reagents 5.3, 5.4, 5.6 and 5.7.1, respectively 5.7.2 through the system for 10 min and set the baseline to zero.

The flow analysis system is ready for use once the baseline becomes stable.

Proceed with steps 8.2 to 8.5.

## 8.2 Quality requirements for the measuring system

### 8.2.1 Reagent blank check

In the case of FIA, replace the solutions 5.3, 5.4 and 5.5 with water (5.1). Wait for the baseline to stabilize and note the absorbance.

In the case of CFA, replace the solutions 5.3, 5.4 and 5.6 with surfactant solution (5.7). Wait for the baseline to stabilize and note the absorbance.

Set the flow analysis system to its original state. Watch the change (increase) in absorbance.

If the absorbance per centimetre of path length increases by more than 0,1 $cm^{-1}$ for the FIA method, or 0,02 $cm^{-1}$ for the CFA method respectively, the reagents or the water entering the sampler may be contaminated. Suitable measures to eliminate the interference shall be undertaken before starting the analysis.

If absorbance readings cannot be made using the photometric detector (6.1.6 or 6.2.7), the absorbance may be determined by comparing an external absorbance using a measuring photometer.

### 8.2.2 Daily sensitivity test and noise test

A calibration solution (5.10) with a $SiO_2$ content of 1 mg/l in working range I (0,2 mg/l to 2 mg/l) or of 10 mg/l in working range II (2 mg/l to 20 mg/l) shall exhibit an absorbance per centimetre of at least 0,05 $cm^{-1}$ for the FIA method, or at least 0,2 $cm^{-1}$ for the CFA method, respectively. See the last paragraph in 8.2.1.

A calibration solution (5.10) with the lowest possible $SiO_2$ concentration (0,2 mg/l within working range I, 2 mg/l within working range II) shall exhibit a signal to noise ratio of at least 3:1.

If a recorder is used for the evaluation, transfer the sample probe into a calibration solution (5.10) having an $SiO_2$ concentration in the middle of the applied working range. When there is a positive response at the registration unit due to the colour produced from the calibration solution, adjust the response to read about 45 % of full-scale deflection.

## 8.3 Calibration

Select the desired working range and the appropriate calibration solutions (5.10).

Perform a separate calibration with at least five calibration solutions for each working range.

Before starting the analysis, set the baseline as recommended by the instrument manufacturer, or as appropriate.

Calibrate the analyser with the calibration solutions and a blank.

Determine the sample concentrations using the procedure specified by the analyser manufacturer as long as it is not in contradiction with this International Standard.

Use the same analytical conditions for both calibration solutions and samples (see 8.4). The output signal is proportional to the silicate and the $SiO_2$ concentration.

Determine the calibration curve in accordance with ISO 8466-1.

Use the following equation:

$$y = b\rho + a \tag{1}$$

where

$y$ is the measured value, expressed in system-related-units;

$b$ is the slope of the calibration curve, expressed in system-related-units $\times$ litres per milligram;

$\rho$ is the mass concentration, expressed in milligrams per litre, of $SiO_2$;

$a$ is the calibration curve intercept, expressed in system-related-units.

## 8.4 Measurement

Analyse the samples, prepared according to clause 7, using the same conditions as the calibration solutions (see 6.1 for FIA and 6.2 for CFA).

If the sample concentration is higher than the selected working range, analyse the sample in a different range or dilute it.

After each group of sample measurements, or at most every 20 measurements, check the calibration with calibration solutions in the lower and upper third of the working range (8.3). If necessary, re-calibrate the system.

## 8.5 Purification and closing down of the system

Purify the flow system as follows in order to remove any precipitates.

At the end of a run, rinse the system for about 5 min with the rinsing solution (5.11), then for another 5 min with water (5.1).

## 9 Calculation

Calculate the mass concentration, $\rho$, of the samples using equation (2):

$$\rho = (y - a)/b \qquad (2)$$

For an explanation of the symbols, see equation (1).

Calculate the concentration of silicate ($SiO_2$) in the samples with respect to the calibration range within which they fall.

Do not extrapolate a concentration from the calibration curve if it is higher than the highest calibration solution.

Report the results to 2 significant figures.

EXAMPLES

soluble silicate (expressed as $SiO_2$): 0,42 mg/l

soluble silicate (expressed as $SiO_2$): 6,3 mg/l

## 10 Precision

Statistical data obtained in an interlaboratory trial carried out in Germany in 2000 in accordance with ISO 5725-2 (after elimination of outliers) are given in Tables 3 to 5.

**Table 3 — Statistical data for the determination of silicate (as $SiO_2$) using FIA**

| **Sample** | **Matrix** | $l$ | $n$ | $o$ | $x_{corr}$ | $X$ | **Recovery** | $s_R$ | **Reproducibility** CV | $s_r$ | **Repeatability** CV |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | % | mg/l | mg/l | % | mg/l | % | mg/l | % |
| Si-1 | Drinking water | 12 | 45 | 6,25 | 17 | 12,8 | 75 | 0,413 | 2,43 | 0,12 | 0,708 |
| Si-2 | Surface water | 12 | 45 | 6,25 | 2,9 | 2,82 | 97 | 0,204 | 7,03 | 0,02 | 0,687 |
| Si-3 | Waste water | 11 | 37 | 15,9 | 20 | 19,4 | 97 | 0,710 | 3,55 | 0,103 | 0,514 |

Samples:

Si-1 Drinking water (city of Wiesbaden), original

Si-2 Surface water (river Rhine), original

Si-3 Domestic waste water (city of Wiesbaden), original

Symbols:

$l$ is the number of received laboratory sets (including outliers)

$n$ is the number of outlier-free individual analytical values

$o$ is the relative portion of the outliers

$x_{corr}$ is the correct value by convention

$X$ is the total mean, depending on outlier-free values

$s_R$ is the reproducibility standard deviation

CV is the coefficient of variation

$s_r$ is the repeatability standard deviation

**Table 4 — Statistical data for the determination of silicate (as $SiO_2$) by CFA**

| Sample | Matrix | $l$ | $n$ | $o$ | $x_{corr}$ | $X$ | Recovery | $s_R$ | Reproducibility CV | $s_r$ | Repeatability CV |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | % | mg/l | mg/l | % | mg/l | % | mg/l | % |
| Si-1 | Drinking water | 10 | 28 | 30 | 17 | 15,1 | 89 | 1,26 | 7,42 | 0,051 | 0,301 |
| Si-2 | Surface water | 10 | 40 | 0 | 2,9 | 2,19 | 76 | 0,754 | 26,0 | 0,013 | 0,444 |
| Si-3 | Waste water | 10 | 40 | 0 | 20 | 21,1 | 105 | 1,65 | 8,25 | 0,226 | 1,13 |
| For an explanation of the samples and symbols, see Table 3. | | | | | | | | | | | |

**Table 5 — Statistical data for the determination of silicate (as $SiO_2$) by flow analysis — Combined evaluation (CFA and FIA)**

| Sample | Matrix | $l$ | $n$ | $o$ | $x_{corr}$ | $X$ | Recovery | $s_R$ | Reproducibility CV | $s_r$ | Repeatability CV |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | % | mg/l | mg/l | % | mg/l | % | mg/l | % |
| Si-1 | Drinking water | 22 | 81 | 7,95 | 17 | 13,8 | 81 | 1,40 | 8,25 | 0,119 | 0,701 |
| Si-2 | Surface water | 22 | 85 | 3,41 | 2,9 | 2,52 | 87 | 0,614 | 21,2 | 0,017 | 0,584 |
| Si-3 | Waste water | 21 | 77 | 8,33 | 20 | 20,0 | 100 | 1,00 | 5,01 | 0,201 | 1,01 |
| For an explanation of the samples and symbols, see Table 3. | | | | | | | | | | | |

## 11 Test report

The test report shall include the following information:

a) all information necessary for the identification of the sample tested;

b) a reference to this International Standard, i.e. ISO 16264;

c) a description of the type of analytical procedure used [i.e. CFA or FIA (6.1 or 6.2)] and the flow analysis conditions;

d) the details of sample preparation, if appropriate;

e) the results of the tests, calculated in accordance with clause 9;

f) any deviations from the procedure specified or any circumstances which may have affected the result.

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