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ISO 29441

Water quality — Determination of total nitrogen after UV digestion — Method using flow analysis (CFA and FIA) and spectrometric detection

Qualité de l'eau — Détermination de l'azote totaux après UV digestion — Méthode par analyse en flux (CFA et FIA) et détection spectrométrique

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Introduction

Methods using flow analysis enable wet chemistry procedures to be automatized and are particularly suitable for the processing of many analytes in water in large series of samples at a high analysis frequency (up to 100 samples per hour).

A differentiation is made 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 will react with the reagent solutions on their way through the manifold. The sample preparation may be integrated in the manifold. The reaction product is measured in a flow detector (e.g. flow photometer).

Water quality — Determination of total nitrogen after UV digestion — Method using flow analysis (CFA and FIA) and spectrometric detection

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This 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.

Waste containing cadmium in liquid or solid form shall be disposed of appropriately.

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

1 Scope

This International Standard specifies a method for the determination of total nitrogen after inline UV digestion, in various types of waters, such as ground, drinking, surface, and waste waters in mass concentrations ranging from 2 mg/l to 20 mg/l for total nitrogen, all in the undiluted sample.

Other mass concentration ranges are possible, provided they cover exactly one decade of concentration (e.g. 0,2 mg/l to 2,0 mg/l). The range of application may be changed by varying the operating conditions.

NOTE Sea water can be analysed with changes in respect to sensitivity and adaptation of the carrier solution and calibration solutions to the salinity of the samples.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

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

ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

ISO 13395, *Water quality — Determination of nitrite nitrogen and nitrate nitrogen by flow analysis — Spectrometric method*

3 Interferences

3.1 General Interferences

Samples with extreme pH values and samples with a high buffering capacity are prone to interferences. For checking, it is advisable to analyse several dilutions of the sample and to check the results for consistency.

High concentrations of organic substances can cause problems as the oxidation capacity may not be sufficient. In case of samples containing more than 100 mg/l of total organic carbon (TOC), reduced yields in nitrogen determination may arise. Therefore the sample should be run with several dilutions in order to check for consistency or standard addition techniques should be applied.

In sea water samples, high concentrations of calcium and magnesium may occur. In alkaline medium, magnesium hydroxide or other hydroxides or calcium carbonate may be formed which will interfere with the UV digestion.

3.2 Additional interferences for FIA

Turbid and coloured samples may need pretreatment prior to analysis: To check for turbidity, all samples may be run without colour reagent. The eventually found peak heights can be subtracted from the originally found peak heights.

4 Principle

The sample is pretreated with a buffered peroxodisulfate solution and thermal UV radiation. Nitrate will be formed and is determined either by flow injection analysis (FIA) or by continuous flow analysis (CFA). With flow injection analysis (FIA) the sample is fed into a continuously flowing buffer solution (carrier stream) by means of an injection valve, or, when applying continuous flow analysis (CFA), it is continuously mixed with this buffer solution. Nitrate in the sample is reduced with metallic cadmium to nitrite [4]. Subsequently a phosphoric acid reagent solution also flowing continuously is admixed. Nitrite resulting from the reduction of nitrate will diazotize sulfanilamide in acidic solution to the diazonium salt which is then coupled with *N*-(1-naphthyl)ethylenediamine to form a red soluble dye (see ISO 13395) [5] [6] [7].

5 Reagents

If not stated otherwise, use only reagents of recognized analytical grade. Check the blank value of the reagents regularly (see 10.3).

5.1 General reagents

5.1.1 Water, complying with grade 1 as defined in ISO 3696.

5.1.2 Phosphoric acid, H_3PO_4, ρ = 1,86 g/ml.

5.1.3 Potassium peroxodisulfate, $K_2S_2O_8$.

5.1.4 Sodium hydroxide, NaOH.

5.1.5 Disodium tetraborate decahydrate, $Na_2B_4O_7 \cdot 10\ H_2O$.

5.1.6 Sulfanilamide, 4-aminobenzenesulfonamide, $C_6H_8N_2O_2S$.

5.1.7 N-(1-naphthyl)ethylenediaminedihydrochloride,
[N-(1–naphthyl)-1,2-diaminoethanedihydrochloride, $C_{12}H_{16}N_2CI_2$].

5.1.8 Sodium nitrite, $NaNO_2$, dried to constant mass at 150 °C.

5.1.9 Potassium nitrate, KNO_3, dried to constant mass at 150 °C.

5.1.10 Imidazole, $C_3H_4N_2$.

5.1.11 Hydrochloric acid

5.1.11.1 Hydrochloric acid I, HCl, concentrated, w = 37 %.

5.1.11.2 Hydrochloric acid II, c(HCl) = 4 mol/l.

5.1.11.3 Hydrochloric acid III, c(HCl) ≈ 0,1 mol/l.

5.1.12 Dichloromethane, CH_2Cl_2.

5.1.13 Copper sulfate solution I, ρ ($CuSO_4 \cdot 5\ H_2O$) = 2,5 g/l.

5.1.14 Copper sulfate solution II, ρ ($CuSO_4 \cdot 5\ H_2O$) = 20 g/l.

5.1.15 Boric acid, H_3BO_3.

5.1.16 Sulfuric acid I, H_2SO_4, ρ = 1,84 g/ml.

5.1.17 Sulfuric acid II, c(H_2SO_4) = 1 mol/l.

5.1.18 Urea, $CO(NH_2)_2$.

5.1.19 Cadmium granulate (Cd), grain size, for example 0,3 mm to 1,5 mm (a minimum reduction capacity of 90 % shall be reached; see 6.1 and 6.2).

5.1.20 Imidazole stock solution, c = 1 mol/l (R1, Figure A.1).

Dissolve 68,1 g of imidazole (5.1.10) in approximately 800 ml of water (5.1.1) in a 1 l beaker.

While stirring with a magnetic stirrer, add hydrochloric acid I (5.1.11.1) and adjust the pH to 7,5 using a pH electrode (6.3.2).

Transfer to a 1 000 ml volumetric flask and dilute to volume with water (5.1.1).

The solution is stable for 4 weeks if kept in a brown glass bottle at room temperature.

5.1.21 Urea stock solution, ρ(N) = 1 000 mg/l.

Dissolve in a 500 ml volumetric flask 1,071 7 g of urea (5.1.18). Add 0,5 ml of dichloromethane (5.1.12) for preservation.

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

5.1.22 Urea working solution, ρ(N) = 20 mg/l.

Dilute 5 ml of urea stock solution (5.1.21) in a 250 ml volumetric flask with water (5.1.1). Acidify the solution with sulfuric acid II (5.1.17) to pH ≤ 2.

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

5.1.23 Buffered copper sulfate solution

Mix 20 ml of the copper sulfate solution II (5.1.14) and 20 ml of the imidazole stock solution (5.1.20) in a 50 ml beaker.

Prepare the solution freshly before use.

5.1.24 Reagent solution, (R2, in Figure A.1 and R1 in figure A.2; see also ISO 13395).

In a 500 ml volumetric flask, dissolve 5 g of sulfanilamide (5.1.6) and 0,5 g of N-(1-naphthyl)ethylenediaminedihydrochloride (5.1.7) in water (5.1.1), add 75 ml of phosphoric acid (5.1.2), and dilute to volume with water (5.1.1).

Stored in a brown glass bottle, the solution is stable for 1 month.

NOTE The solutions of sulfanilamide (5.1.6) and N-(1-naphthyl)ethylenediaminedihydrochloride (5.1.7) can as well be prepared separately and dosed into the equipment by different lines.

Prior to use, degas solution R2 for FIA , for example by membrane filtration (vacuum).

5.1.25 Nitrite(N) stock solution, ρ(N) = 100 mg/l.

In a 1 000 ml volumetric flask, dissolve 492,6 mg of sodium nitrite (5.1.8) in water (5.1.1) and dilute to volume with water (5.1.1).

This solution is stable for at least 2 weeks if kept in a stoppered glass bottle at (4 ± 2) °C.

5.1.26 Nitrite(N) solution, ρ(N) = 20 mg/l.

Pipette 20 ml of the nitrite(N) stock solution (5.1.25) into a 100 ml volumetric flask and dilute to volume with water (5.1.1).

Prepare the solution freshly before use.

5.1.27 Nitrate(N) solution, ρ(N) = 200 mg/l.

In a 100 ml volumetric flask, dissolve 144,4 mg of potassium nitrate (5.1.9) in water (5.1.1) and dilute to volume with water (5.1.1). Acidify the standard solutions with sulfuric acid II (5.1.16) to pH<2.

The solution is stable for at least 1 month.

5.1.28 Calibration solutions

Prepare calibration solutions by diluting the nitrate(N) solution (5.1.27). At least five calibration solutions are recommended.

For example, proceed according to Table 1 for the preparation of 10 calibration solutions.

Table 1 — Preparation of the calibration solutions for total nitrogen(N)

Nitrate(N) concentration mg/l	Volume of nitrate(N) solution (5.1.27) diluted with water (5.1.1) to 100 ml ml
2	1
4	2
6	3
8	4
10	5
12	6
14	7
16	8
18	9
20	10

Prepare all calibration solutions immediately before measurement.

5.1.29 Rinsing solution, for rinsing the CFA sampler and diluting the samples (Clauses 7 and 8).

Acidify water to pH ≤ 2 with sulfuric acid II (5.1.17).

5.2 Additional reagents for FIA (5.1)

5.2.1 FIA Oxidising reagent I (R3, Figure A.1).

In a 250 ml volumetric flask, dilute 6,8 ml of sulfuric acid II (5.1.17) in about 100 ml of water (5.1.1). Dissolve 7,2 g of boric acid (5.1.15) and 10,1 g of potassium peroxodisulfate (5.1.3) in about 100 ml of water (5.1.1) and bring to volume with water (5.1.1).

The solution is stable for 1 d if kept at 2 °C to 8 °C. Do not close the reagent container airtight.

5.2.2 FIA Oxidizing reagent II (R4, Figure A.1).

In a 250 ml volumetric flask, dissolve 6,0 g of sodium hydroxide (5.1.4) and 10,1 g of potassium peroxodisulfate (5.1.3) in about 200 ml of water (5.1.1) and bring to volume with water (5.1.1).

The solution is stable for one week if kept at 2 °C to 8 °C. Do not close the container airtight.

5.2.3 FIA Carrier solution (C, Figure A.1).

Mix 1 000 ml of water (5.1.1) with 100 µl of copper sulfate solution I (5.1.13). Prepare the solution freshly before use.

Prior to use degas the solution, for example by membrane filtration (vacuum).

5.3 Additional reagents for CFA (5.2)

5.3.1 Polyethyleneglycol dodecyl ether, [HO - $(CH_2CH_2 - O)_n$ - $C_{12}H_{21}$], surfactant, F = 33 °C to 41 °C, solution, w = 30 %.

5.3.2 CFA Oxidising reagent (R5, Figure A.2).

Using a 1 000 ml volumetric flask dilute 45,0 g of potassium peroxodisulfate (5.1.3) in about 900 ml of water (5.1.1). Add 12,0 g of sodium hydroxide (5.1.3) and dissolve. Dilute to volume with water (5.1.1). Do not heat the solution otherwise the peroxodisulfate will loose oxygen spontaneously.

The solution is stable for 2 weeks if stored at room temperature.

NOTE A Titanium catalyst can be used to improve digestion of N-compounds. In this case, it is possible to dilute 5 ml of a stock $TiCl_4$ solution, to 200 ml, and add 0,25 ml of this solution to the CFA oxidising reagent.

5.3.3 CFA Buffer solution I (R4, Figure A.2).

Dissolve 24,0 g of boric acid (5.1.14) in a 1 000 ml volumetric flask in water (5.1.1) and bring to volume with water (5.1.1).

The solution is stable for 1 month if stored at room temperature.

5.3.4 CFA buffer solution II (R3, Figure A.2).

In a 1 000 ml volumetric flask add 5,0 g of Na_2EDTA (5.3.7) to 500 ml of the imidazole stock solution (5.1.20) and dissolve. Dilute to volume with water (5.1.1), add 1ml of polyethyleneglycol dodecylether (5.3.1) and mix.

The solution is stable for 1 month if stored in a brown bottle at room temperature.

5.3.5 CFA buffer solution III (R2, Figure A.2).

Dilute 250 ml of the imidazole stock solution (5.1.20) to 1 000 ml with water (5.1.1). Add 1 ml of polyethyleneglycol dodecylether (5.3.1) and mix.

5.3.6 Ethylenedinitrilotetraacetic acid, disodium salt, dihydrate, Na_2EDTA, $C_{10}H_{14}N_2Na_2O_8 \cdot 2\ H_2O$.

6 Apparatus

Usual laboratory apparatus and the following.

6.1 Flow injection system (FIA), usually comprising of the following (see Figure A.1).

6.1.1 Reagent reservoirs

6.1.2 Low pulsation pump, with suitable, chemically inert pump tubes.

6.1.3 Sample injector, injection volume 30 µl (or 400 µl for smaller concentrations).

6.1.4 Cadmium reductor, minimum reduction efficiency 90 %, e.g. cadmium tube [5] of internal diameter 1,1 mm or cadmium column (5.1.19).

NOTE Other reductor types are possible as long as the reduction capacity supercedes 90 %. This is to encourage other techniques to be made available (e.g. enzymatic reductors).

6.1.5 Transport tubes and reaction coils, internal diameter 0,5 mm to 0,8 mm, with tube connections and T-connections of chemically inert plastics.

6.1.6 Photometric detector, with flow cell, wavelength range 520 nm to 560 nm.

6.1.7 Recording unit, e.g. strip chart recorder, integrator or printer/plotter. In general, peak height signals are evaluated.

6.1.8 Autosampler, if required.

6.1.9 UV reactor/digester

6.2 Continuous flow analysis (CFA), usually comprising the following components (see Figure B.1).

6.2.1 Autosampler, or any other device allowing a reproducible application of the sample.

6.2.2 Reagent reservoirs

6.2.3 Low pulsation pump, with suitable, chemically inert pump tubes.

6.2.4 Cadmium reductor, with a minimum reduction efficiency of 90 %, e.g. packed cadmium column with granulate (5.1.19), of internal diameter 4,0 mm, for example, and minimum length 5 cm (see 6.1).

See note in 6.1.4.

6.2.5 Manifold, with highly reproducible gas-bubble feeding (nitrogen is recommended), sample and reagent feeding, with appropriate transport systems and connection assemblies of chemically inert plastics or metal. The application of the cadmium reductor requires oxygen-free gas. Before entering the cadmium column, degas the flow stream, if air is used for segmenting the flow stream.

6.2.6 Dialysis cell, if required, with, for example, a cellulose membrane, suitable for the predilution of the sample or the elimination of interfering compounds.

6.2.7 Photometric detector, with flow cell, wavelength 520 nm to 560 nm.

6.2.8 Recording unit, e.g. strip chart recorder, integrator or printer/plotter. In general, peak height signals are evaluated.

6.2.9 UV reactor/digester

NOTE Figure A.2 describes a flow system (CFA) with an internal diameter of approximately 2 mm. It is possible to use other diameters (e.g. 1 mm) as well.

6.3 Additional apparatus used for FIA and CFA

6.3.1 Membrane filter assembly, with membrane filters of pore size 0,45 µm. Cellulose acetate filters should be used.

6.3.2 pH electrode

6.3.3 Homogenization device (e.g. ultraturrax), if needed.

7 Sampling and sample preparation

Before use, clean all containers coming into contact with the sample thoroughly with water (5.1.1) (see ISO 5667-3).

For total nitrogen, collect the sample in a glass or polyethene bottle. Acidify these samples with sulfuric acid II (5.1.17) to approximately pH 2. Store the samples for at least 12 h to dissolve and predigest particulate nitrogen.

The maximum storage time is 1 month at 2 °C to 8 °C.

The amount of particles shall not exceed 30 mg/l. In case of higher amounts dilute the sample after homogenization.

As an exception, the samples may be stored in the freezer at approximately –20 °C for 8 d, provided the applicability of this preservation has been checked.

NOTE If the sample contains larger particles, a homogenization device (6.3.3) is necessary.

Prior to measurement, dilute samples with a total salt concentration of >30 g/l with rinsing solution (5.3.6).

8 Procedure

8.1 Preparation, activation and checking of the cadmium reductor

8.1.1 Cadmium column with granulate

Place a sufficient quantity of the cadmium granulate (5.1.19) to fill the column (see 6.1) in a 25 ml beaker. Stir with hydrochloric acid II (5.1.11.2) until the surface of the granulates shows a metallic shine.

NOTE 1 Activated cadmium granules and columns are commercially available.

Remove the acid by rinsing several times with water.

Decant the water and stir the granulate twice for approximately 2 min with copper sulfate solution II (5.1.14). The surface of the granulate will turn black.

Decant and carefully rinse with water (5.1.1).

Fill the column with the granulate, avoiding air bubbles and large cavities, and stopper the ends of the column (e.g. with glass wool).

Assemble the column in the flow system and activate the reductor by applying the highest calibration solution (5.1.28) three times.

Repeatedly measure a calibration solution (5.1.28) with a nitrate(N) concentration of 20 mg/l, until stable results are obtained.

NOTE 2 The cadmium column can be stored, free from air bubbles, in the imidazole stock solution (5.1.19). Prior to re-use, stabilize and activate the column as described above.

8.1.2 Cadmium tube

Using the syringe (6.6), aspirate approximately 5 ml of the buffered copper sulfate solution (5.1.23) into the cadmium tube (see 6.1.4) and allow to react for 5 min. Repeat the procedure, avoiding air bubbles.

Using the syringe, aspirate approximately 20 ml of imidazole stock solution (5.1.20) through the tube and allow to react, avoiding air bubbles.

Assemble the column in the flow system, activate and stabilize as described in 8.1.1.

NOTE The cadmium tube can be stored, free from air bubbles, in the imidazole stock solution (5.1.20). Prior to measurement, stabilize or treat, if required, (see 8.1.3) with buffered copper sulfate solution (5.1.23).

8.1.3 Checking the flow system

8.1.3.1 Sensitivity, check of the digestion process

A calibration solution (5.1.28) with a nitrate(N) concentration of 10 mg/l, measured in the system (FIA or CFA respectively) should result in an absorbance of at least 0,04 per 10 mm path length.

NOTE If the photometric detector (see 6.1.6 or 6.2.7) does not allow any absorbance readings, the absorbance can be determined by comparing with an external absorbance-measuring photometer.

Sequentially analyse a nitrate calibration solution (5.1.28) and a urea solution (5.1.21) with a nitrogen mass concentration of 20 mg/l each, and compare the measured values obtained.

If the measured value for urea is less than 85 % of the measured nitrate value, take appropriate measures by cleaning the system with hydrochloric acid III (5.1.11.3) or the UV lamp should be changed.

Check the UV digestion process weekly.

8.1.3.2 Checking the reduction capacity

By replacing the oxidizing reagents (5.3.2 for CFA; 5.2.1 and 5.2.2 for FIA) with water and with the UV reactor switched off, the reduction capacity of the cadmium reductor (6.1.4, 6.2.4) may be checked.

Sequentially analyse a nitrate calibration solution (5.1.28) and a nitrite standard solution (5.1.26) with a nitrogen mass concentration of 20 mg/l each, and compare the measured values obtained.

If the measured value for nitrate is less than 90 % of the measured nitrite value, take appropriate measures according to 8.1.1 and 8.1.2 in order to obtain a reduction capacity of at least 90 %.

Check the reduction capacity again, prior to the analysis of each series of samples.

8.2 Preparation for measurement

Assemble the flow system according to the method of determination desired (CFA or FIA).

Prior to measurement of total nitrogen, continuously run the reagent solutions through the system for approximately 10 min without the cadmium reductor and subsequently for approximately 20 min with the cadmium reductor in operation. Record and zero the base absorbance.

The system is in operation condition when the baseline is stable. A satisfactory signal-to-noise relation of 3:1 shall be obtained. Perform the reaction steps in the sequence of 8.3 to 8.5.

8.3 Monitoring the blank of the reagents

Allow the baseline to stabilize.

Instead of the buffer solution (R2) and the reagent solution (R1), transport water (5.1.1) for 2 min and record changes in the measuring signal.

If the absorbance changes by more than 0.200 AU per 10 mm path length, either the water being used or the reagent solutions may be contaminated. Take appropriate measures to eliminate the interference.

Then transport the reagent solutions again.

8.4 Calibration

Prepare the calibration solutions (5.1.28). Calibrate by sequentially adding the calibration solutions and the blank solution.

Prior to calibration, zero the instrument, if necessary following the manufacturer's instructions.

Determine the measured values from the calibration solutions used while following the manufacturer's instructions, as long as they do not contradict the specifications of this International Standard.

The test conditions for the calibration and the measurement of samples (8.5) are the same. The magnitude of the measuring signal is proportional to the mass concentration of nitrogen(N). Establish the regression line for the measuring series obtained.

Calculate the calibration curve as specified in ISO 8466-1.

Apply the following general Equation (1):

$$y = b \cdot \rho_N + a \quad (1)$$

where

y is the measured value, in instrument-related units;

b is the slope of the calibration function, in instrument-related units · litre per milligram;

ρ_N is the mass concentration, in milligrams per litre, mg/l of nitrogen(N);

a is the ordinate intercept of the calibration function, in terms of instrument-related units.

If the linearity test described in ISO 8466-1 shows that the calibration curve is not linear, calculate the calibration curve as specified in ISO 8466-2.

8.5 Sample measurement

Analyse the samples, pretreated according to Clause 7, in the same way as the calibration solutions (5.1.28), with the flow system (FIA, see 6.1 and Figure A.1; CFA, see 6.2 and Figure B.1).

If the mass concentrations to be determined exceed the validity range of the selected working range, dilute the sample or analyse using another working range.

Check the validity of the calibration function after each sample series, but at latest after the measurement of 10 to 20 samples, using one calibration solution each for the lower and upper third of the respective working range. Check the reduction capacity (8.1.3.2) again or set up a new calibration (8.4), if necessary.

After measurement, store the cadmium reductor in an oxygen-fee imidazole solution (see notes in 8.1 and 8.1.2).

9 Evaluation

Determine the mass concentration of the determinand in the measuring solution using the measured value obtained as described in 8.5, from the calibration function [Equation (1), 8.4].

For the evaluation, use the appropriate function. Do not extrapolate beyond the working range selected.

Calculate ρ_N using Equation (2):

$$\rho_N = \frac{y - a}{b} \quad (2)$$

where

ρ_N is the mass concentration, in milligrams per litre, mg/l, of nitrogen(N) in the sample;

y, a and b are as defined in Equation (1).

For the calculation of results in case of a non-linear calibration curve see ISO 8466-2.

10 Expression of results

Report the results to a maximum of two significant figures.

EXAMPLE

Total nitrogen(N): 2,9 mg/l

11 Precision

The statistical data in Annex B were established in an interlaboratory trial, carried out in <date to be inserted>.

12 Test report

The test report shall refer to this International Standard. It may contain the following additional information:

a) identity of the water sample;

b) specification of the procedure applied (CFA or FIA);

c) description of the sample pretreatment;

d) description of the type of instrument or of the flow conditions;

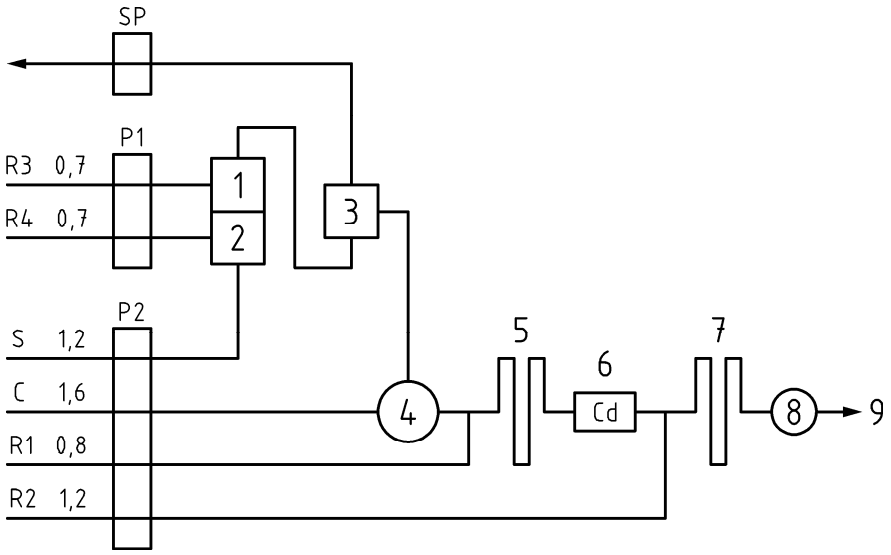
e) expression of the results according to Clause 10;

f) precision and trueness of the results, if available;

g) any deviation from this method and all events which may have influenced the result.

Annex A
(informative)

Examples of flow systems (6.1 or 6.2) for the determination of total nitrogen after inline UV digestion (2 mg/l to 20 mg/l)



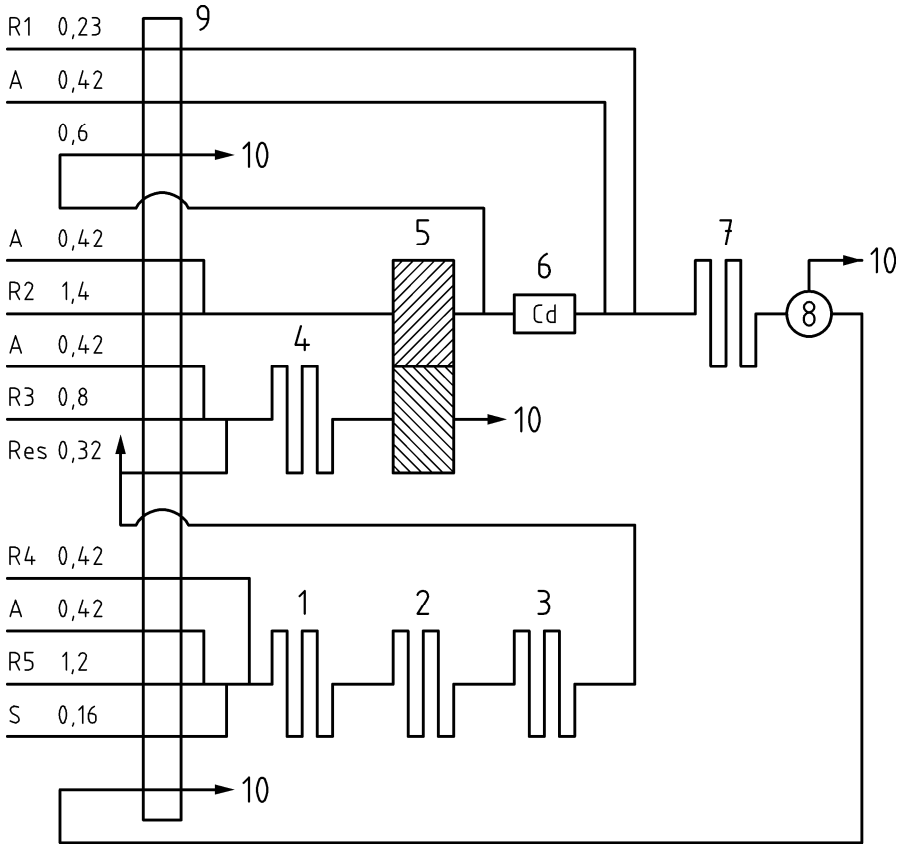
Key

SP sample pump
P1 pump, flow rate in ml/min (6.1.2)
P2 pump, flow rate in ml/min (6.1.2)
C carrier solution (5.2.3), 1,60 ml/min
R2 reagent solution (5.1.24), 1,20 ml/min
R1 imidazole Stock solution (5.1.20), 0,80 ml/min
R3 FIA Oxidising reagent I (5.2.1), 0,70 ml/min
R4 FIA Oxidising reagent II (5.2.2)0,70 ml/min
S sample, 1,20 ml/min

1 UV reactor (6.1.9)
2 thermo reactor
3 degassing station
4 injector 30 µl
5 reaction coil, length 50 cm, internal diameter 0,5 mm
6 cadmium reductor (6.1.4)
7 reaction coil, length 150 cm, internal diameter 0,5 mm
8 Detector, optical length 1 cm, wave length 520 to 560 nm (6.1.6)
9 waste

Figure A.1 — Example of a flow injection system (FIA) for the determination of total nitrogen in the range 2 mg/l to 20 mg/l according to 6.1

NOTE An injector volume of 400 µl is suitable for the determination of total nitrogen in the range of 0,2 mg/l to 2 mg/l.



Key

R1	reagent solution (5.1.24), flow rate 0,23 ml/min	1	reaction coil, 70 °C, length: 270 cm, internal diameter: 2,0 mm
R2	CFA buffer solution III (5.3.5), flow rate 1,40 ml/min	2	UV digester with UV lamp (8 W) and quartz coil 332 cm, internal diameter: 1,8 mm (6.2.9)
R3	CFA buffer solution II (5.3.4), flow rate 0,80 ml/min	3	reaction coil, 70 °C, length 270 cm, internal diameter 2,0 mm
R4	CFA buffer solution I (5.3.3), flow rate 0,42 ml/min	4	reaction coil, length: 35 cm, internal diameter: 1,5 mm
R5	CFA oxidising reagent (5.3.2), flow rate 1,20 ml/min	5	dialyser, 15 cm (6.2.6)
A	air, flow rate 0,42 ml/min	6	cadmium reductor (6.2.4)
S	sample, flow rate 0,16 ml/min	7	reaction coil, length: 200 cm, internal diameter: 1,5 mm
Res	resample, flow rate 0,32 ml/min	8	detector, wavelength: 520 nm to 560 nm, optical length 1 cm (6.2.7)
		9	pump, flowrates in ml/min (6.2.3)
		10	waste

Figure A.2 — Example of a continuous flow system (CFA) for the determination of total nitrogen in the range 2 mg/l to 20 mg/l according to 6.2

NOTE The addition of $TiCl_4$ to the destruction mixture to a final concentration of 0,001 % catalyses the decomposition.

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