



BSI Standards Publication

**Water quality — Performance requirements and conformity test procedures for water monitoring equipment — Automated sampling devices (samplers) for water and waste water**

**National foreword**

This British Standard is the UK implementation of EN 16479:2014.

The UK participation in its preparation was entrusted to Technical Committee EH/3, Water quality.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 75401 2

ICS 13.060.45

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 July 2014.

**Amendments issued since publication**

Date	Text affected
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EUROPEAN STANDARD

**EN 16479**

NORME EUROPÉENNE

EUROPÄISCHE NORM

July 2014

ICS 13.060.45

English Version

## Water quality - Performance requirements and conformity test procedures for water monitoring equipment - Automated sampling devices (samplers) for water and waste water

Qualité de l'eau - Exigences de performance et modes opératoires d'essai de conformité pour les équipements de surveillance de l'eau - Dispositifs d'échantillonnage automatiques (échantillonneurs) pour l'eau et les eaux usées

Wasserbeschaffenheit - Leistungsanforderungen und Konformitätsprüfungen für Geräte zum Wassermonitoring - Automatische Probenahmegeräte für Wasser und Abwasser

This European Standard was approved by CEN on 22 May 2014.

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## Foreword

This document (EN 16479:2014) has been prepared by Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2015, and conflicting national standards shall be withdrawn at the latest by January 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document was submitted to the Formal Vote with the reference FprEN 16479-1.

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## Introduction

This European Standard is a product standard for automated sampling devices (samplers) for water and waste water. It defines general requirements, performance requirements, and procedures for the conformity testing of samplers. Samplers that are shown, by means of the tests, to conform with the specified requirements are considered to be fit for purpose. However, this European Standard does not cover the installation and on-going use of samplers.

The requirements of this European Standard are intended to be independent of measurement technology and applicable to all automated sampling devices.

Water monitoring equipment is widely used for compliance monitoring purposes under national and European regulations. This European standard supports the requirements of the following EU Directives:

- Industrial Emissions Directive (2010/75/EU) [6].
- Urban Waste Water Treatment Directive (UWWTD) (91/271/EEC and 98/15/EEC) [7].
- Water Framework Directive (2000/60/EC) [8].
- Marine Strategy Framework Directive (2008/56/EC) [9].

## 1 Scope

This European Standard defines general requirements, performance requirements and conformity test procedures for automated sampling devices (samplers) for water and waste water that:

- sample water and waste water from non-pressurized (i. e. open to atmosphere) channels or vessels;
- sample over extended periods to collect discrete or composite samples based on time, event or flow proportional sampling.

Specific sample integrity requirements are defined for samplers to be used for the collection of samples of final effluent or influent for the purpose of monitoring the performance of waste water treatment works, as required under the Urban Waste Water Treatment Directive (UWWTD). Samplers to be used for other industrial applications do not have to be assessed against these specific sample integrity requirements.

This European Standard does not cover the installation and on-going use of samplers.

## 2 Normative References

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

EN ISO 5667-3:2012, *Water quality - Sampling - Part 3: Preservation and handling of water samples (ISO 5667-3:2012)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025)*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 3.1

#### **automated sampling device for water and waste water**

#### **automated sampler**

equipment for collecting and storing samples of water or waste water for subsequent analysis

### 3.2

#### **bias**

estimate of a systematic measurement error

Note 1 to entry: The systematic measurement error is a component of measurement error that in replicate measurements remains constant or varies in a predictable manner.

[SOURCE: ISO/IEC Guide 99:2007, 2.18, modified — Note 1 to entry has been added.]

### 3.3

#### **composite sample**

two or more samples or sub-samples, mixed together in appropriate known proportions (either discretely or continuously), from which the average result of a desired requirement may be obtained

Note 1 to entry: The proportions are usually based on time or flow measurements.

[SOURCE: ISO 6107-2:2006/AMD, 1:2012, 29]

**3.4**  
**constant volume variable time sampling**  
**C.V.V.T**  
flow proportional sampling based on collecting equal volumes of sample at frequencies proportional to flow

**3.5**  
**constant time variable volume sampling**  
**C.T.V.V**  
flow proportional sampling based on collecting samples at fixed time intervals but where the volume of sample is varied in proportion to the flow

**3.6**  
**constant time constant volume sampling**  
**C.T.C.V**  
equal volumes of sample or sub-sample collected at equal increments of time

**3.7**  
**determinand**  
property/substance that is required to be measured and to be reflected by/present in a calibration solution

[SOURCE: EN ISO 15839:2006, 3.13]

**3.8**  
**discrete sample**  
single sample taken from a body of water

[SOURCE: ISO 6107-2:2006, 40, modified – “process, whereby” deleted]

**3.9**  
**measurement error**  
**error of measurement**  
**error**  
measured quantity value minus a reference quantity value

Note 1 to entry: The concept of “measurement error” can be used both:

- a) when there is a single reference quantity value to refer to, which occurs if a calibration is made by means of a measurement standard with a measured quantity value having a negligible measurement uncertainty or if a conventional quantity value is given, in which case the measurement error is known, and
- b) if a measurand is supposed to be represented by a unique true quantity value or a set of true quantity values of negligible range, in which case the measurement error is not known.

Note 2 to entry: Measurement error is not be confused with production error or mistake.

[SOURCE: ISO/IEC Guide 99:2007, 2.16]

**3.10**  
**rated operating conditions**  
minimum to maximum values of any environmental, fluid or electrical parameter within which the sampler is designed to operate without adjustment and with errors within performance limits

**3.11**  
**lift height**  
vertical distance between the surface of the fluid being sampled and the highest point to which the sample is lifted

Note 1 to entry: Sometimes called “sampling head” or “suction height”.



Note 2 to entry: The maximum lift height for samplers using vacuum pumps (e.g. pneumatic samplers and peristaltic samplers) is set to an atmospheric pressure of 1 000 mbar. At low atmospheric pressure the maximum lift height will be consequentially lower.

### 3.12

#### **precision**

closeness of agreement between indications or measured quantity values obtained by replicate measurements on the same or similar objects under specified conditions

Note 1 to entry: Measurement precision is usually expressed numerically by measures of imprecision, such as standard deviation, variance, or coefficient of variation under specified conditions of measurement.

Note 2 to entry: The “specified conditions” can be, for example, repeatability conditions of measurement, intermediate precision conditions of measurement, or reproducibility conditions of measurement (see ISO 5725-3:1994).

Note 3 to entry: Measurement precision is used to define measurement repeatability, intermediate measurement precision, and measurement reproducibility.

Note 4 to entry: Sometimes “measurement precision” is erroneously used to mean measurement accuracy.

[SOURCE: ISO/IEC Guide 99:2007, 2.15]

### 3.13

#### **sampling interval**

time between successive sampling events

### 3.14

#### **sampling line**

conduit from intake point to inlet of dosing system

[SOURCE: ISO 6107-2:2006/AMD, 1:2012, 115, modified – “sampling probe” was replaced by “intake point” and delivery point was replaced by “inlet of dosing system”]

## **4 General requirements for samplers**

See 6.3 for details on verification by inspection.

A sampler shall:

- a) have an unique designation that unambiguously identifies it (e.g. model, serial number);
- b) be designed (including its operating methodology) and constructed to ensure that the composition of the sample is, as far as is practicable, not altered by the sampling procedure;

It can be impracticable to prevent the loss of volatile substances during sampling with vacuum and peristaltic samplers.

- c) have a rated maximum lift height at which all of the performance requirements of this standard are fulfilled. The rated maximum lift height shall be inscribed on the sampler or declared in the operating manual published by the manufacturer;

Conformity testing of the sampler shall be based on a range of lift heights up to and including the sampler's rated maximum lift height.

- d) have provision for the user to set the volume of a discrete sample;
- e) have rated minimum and maximum sample volumes of a discrete sample inscribed on the sampler or declared in the operating manual published by the manufacturer;

Unless otherwise stated conformity testing of the sampler shall be based on a sample volume of 250 ml or the rated maximum sample volume, if smaller.

- f) have the stated capacities, for any integrated sample storage, both by number(s) and volume(s) of individual bottles and of a composite container, inscribed on the sampler or declared in the operating manual published by the manufacturer;
- g) be capable of collecting a series of samples, on a timed, event and/or a flow proportional basis. Samples can be collected and stored in individual bottles or a single composite sample bottle;
- h) have its possible sampling intervals inscribed on the sampler or declared in the operating manual published by the manufacturer;
- i) have provision for the user to set the sample interval as a minimum in the range 5 min to 1 h with increments of 1 min, for time proportional samplers;
- j) have provision for the sample interval (in the case of C.V.V.T. sampling) or the sample volume (in the case of C.T.V.V. sampling) to be set on the basis of a flow signal (e.g. pulse or analogue) from a flow meter. For pulse inputs, the relationship between pulse input and sample interval or volume should be adjustable as a minimum over the range 1 pulse to 999 pulses in increments of 1 pulse;
- k) have a control unit capable of recording sample collection failures;
- l) have a control unit capable of recording any low battery alarm during sample collection;
- m) be designed to minimise the possibility of clogging of the sample line by suspended solids in the waste water. The nominal internal diameter of the sample line shall be not less than 9 mm and the average sample line velocity shall not be less than 0,5 m/s. The sampler shall be capable of achieving this average sample line velocity at all lift heights up to and including its maximum rated lift height.

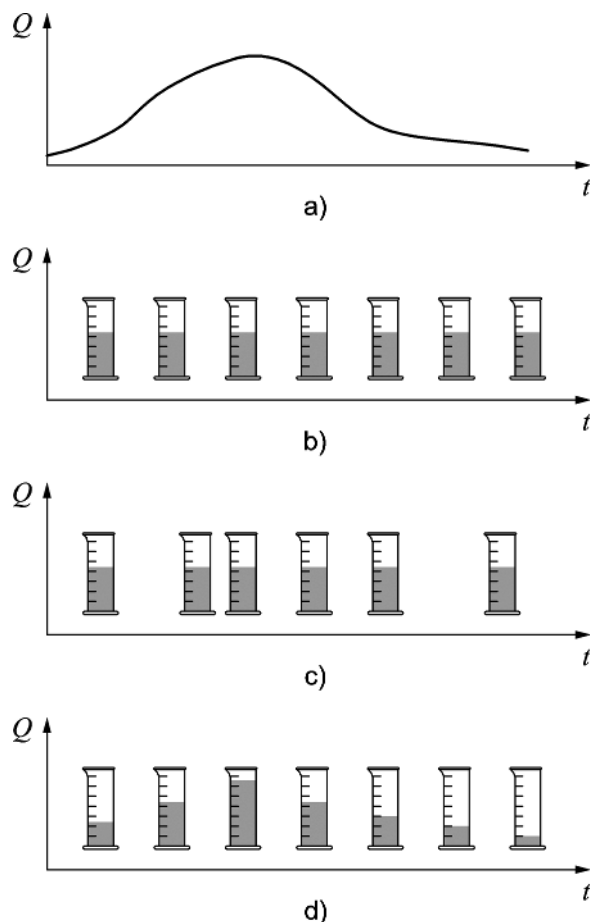
These requirements on sample line diameter exclude pipe restriction caused by the normal operation of pinch valves and peristaltic pumps.

National legal requirements can specify different minimum values for internal sample line diameter and average sample line velocity. These may need to be taken into account.

- n) be capable of purging the contents of the sampling line between each sampling event;
- o) have stated ingress protection (IP) rating inscribed on the sampler or stated in the operating manual.

Requirements for ingress protection are detailed in EN 60529:1991 [1]

The possible sampling options are illustrated in Figure 1.



**Key**

- $Q$  discharge
- $t$  time
- a) Flow rate curve
- b) C.T.C.V. Time proportional sampling
- c) C.V.V.T. Flow proportional sampling
- d) C.T.V.V. Flow proportional sampling

**Figure 1 — Sampling options**

## 5 Performance requirements

### 5.1 Sample volume

See conformity test in 6.4.1.1 and 6.4.1.2.

The bias of the sample volume and precision at the 95 % confidence limit shall each not be greater than 5 % of the set volume over the tested range for lift height.

NOTE Details of how to calculate bias and precision and worked example calculations are given in A.1.

### 5.2 Sampling principles

See conformity tests in 6.4.2.2, 6.4.2.3, 6.4.2.4 and 6.4.2.5.

The performance of the sampling principle shall be tested and the results reported. The timing error for each operating principle shall not be greater than 1 %.

### 5.3 Sample line velocity

See conformity test in 6.4.3.

The average velocity of the sample as it passes through the sample line during the sampling event shall not be less than 0,5 m/s at each tested lift height and at the rated voltage for the power supply.

NOTE 1 A worked example calculation of sample line velocity is given in A.2.

NOTE 2 National legal requirements can specify different minimum values for internal sample line diameter and average sample line velocity and these may need to be taken into account.

### 5.4 Power supply

See conformity test in 6.4.4.

The average velocity of the sample as it passes through the sample line during the sampling event shall not be less than 0,5 m/s between the minimum and maximum rated voltages for the power supply.

NOTE A worked example calculation of sample line velocity is given in A.2.

### 5.5 Sample integrity

See conformity test in 6.4.5 and the example procedure at Annex B.

Analyses for BOD (biochemical oxygen demand), COD (chemical oxygen demand), total nitrogen, and total phosphorus in samples taken by the sampler and in samples taken manually from a test fluid in accordance with the conformity test detailed in 6.4.5 shall show no significant statistical difference based on an analysis of variance.

NOTE Details of how to calculate sample integrity using analysis of variance and a worked example calculation are given in A.3.

The design of a sample integrity test for other applications shall be based on EN ISO 5667-3 which describes the precautions to be taken to preserve and transport water samples. The same pass criteria should be applied when using a test fluid for an application other than the UWWTD and for which relevant determinands have been identified.

### 5.6 Sample timing error

See conformity test in 6.4.6.

The error of the sampler interval timing mechanism shall be no greater than  $\pm 10$  s per 24 h.

NOTE A worked example calculation of sample timing error is given in A.4.

### 5.7 Effect of ambient air temperature

#### 5.7.1 Samplers not incorporating sample temperature control

See conformity tests in 6.4.7.

Samplers which do not incorporate a means for maintaining the temperature of the sample within pre-set limits shall conform to the sample volume error requirements in 5.1 when operated within one of the following sets of rated operating conditions with regards to ambient temperature:

a) from +5 °C to +40 °C; or

b) from  $-10\text{ }^{\circ}\text{C}$  to  $+40\text{ }^{\circ}\text{C}$ .

Temperature range a) should be used for samplers designed without integral frost protection and for use only indoors where the building provides protection from frost. Temperature range b) should be used for samplers designed for use outdoors and which have integral frost protection.

NOTE Details of how to calculate bias and precision and worked example calculations are given in A.1.

### 5.7.2 Samplers incorporating sample temperature control

See conformity tests in 6.4.7.

Samplers which incorporate a means for maintaining the temperature of the sample within pre-set limits shall conform to the sample volume error requirements in 5.1 when operated within an ambient temperature range from  $-10\text{ }^{\circ}\text{C}$  to  $+40\text{ }^{\circ}\text{C}$ .

The mean temperature of the sample shall be maintained within the range from  $0\text{ }^{\circ}\text{C}$  to  $+5\text{ }^{\circ}\text{C}$  during the sampling period, when the sampler is operated within the rated operating conditions for ambient temperature and process fluid temperature. The design of the sample temperature control shall ensure that ice does not form in the sample.

In the case of mains powered samplers, after completion of the sampling period, the sample temperature shall remain within the range from  $0\text{ }^{\circ}\text{C}$  to  $+5\text{ }^{\circ}\text{C}$  for a minimum period of 24 h. The design of the sample temperature control shall ensure that ice does not form in the sample.

In the case of portable samplers which are not powered directly from a mains electricity supply, the minimum period of time, from the end of the sampling period, over which the temperature of the sample remains within the range from  $0\text{ }^{\circ}\text{C}$  to  $+5\text{ }^{\circ}\text{C}$  with no formation of ice shall be 12 h.

NOTE Details of how to calculate bias and precision and worked example calculations are given in A.1.

## 6 Conformity testing

### 6.1 General requirements

The sampler shall be installed in accordance with any instructions provided by the manufacturer.

Each performance requirement for the sampler shall be considered on its own when performing the conformity tests.

Conformity testing of the sampler shall be carried out in accordance with the requirements of EN ISO/IEC 17025 or other equivalent standards accepted at international level.

Sampler conformance shall be determined by processing the data from the tests in accordance with the calculation methods summarised in Annex A.

Results from the conformity testing should be reported using the proposed format for the report given in Annex C.

### 6.2 Test conditions

The sample line shall be arranged so that no part of the test fluid is retained within the sample line.

Prior to carrying out any series of consecutive tests, the sampler shall be operated, for a total operating period of 2 000 sampling cycles, under the following conditions:

— the rated maximum lift height;

- sampling interval 5 min;
- sample volume 250 ml, (or the stated maximum volume if less than 250 ml);
- samples shall be discarded.

During tests that require the sampler to collect a sample of fluid, the vessel containing the fluid to be sampled shall be open to the atmosphere.

In the case of a battery powered sampler, the battery shall be fully charged at the start of each conformity test unless stated otherwise in the test conditions.

The sampler should be maintained, cleaned or recalibrated in accordance with the manufacturer's instructions prior to any test, but adjustments shall not be carried out during the course of the test.

Table 1 gives the reference conditions for possible influence quantities. The sampler shall be tested with all influence quantities at their reference values, including tolerances, unless where specifically varied in any one test.

**Table 1 — Test reference conditions**

Influence quantity	Reference value	Tolerance
Ambient temperature	20 °C	±2,5 °C
Ambient humidity at 20 °C	< 60 %	—
Sample temperature	20 °C	±2,5 °C
Supply voltage (AC)	230 V <sup>a</sup> (or 110 V)	±6 % <sup>a</sup>
Supply voltage (DC)	Rated voltage	Rated voltage
<sup>a</sup> see EN 60038:2011 [2]		

### 6.3 Verification by inspection

The sampler shall be set up, calibrated and adjusted in accordance with the manufacturer's instructions.

The sampler (or statements in the manufacturer's operating manual) shall be inspected to verify conformance to the general requirements listed in Clause 4, as appropriate to the sampler under test. The means by which each requirement is fulfilled shall be reported.

### 6.4 Performance tests

#### 6.4.1 Sample volume

##### 6.4.1.1 C.T.C.V. Time proportional sampling principle

Determine the sample volume errors in accordance with the following procedure.

- 1) Set the sampler to operate on time proportional sampling.
- 2) Programme the sampler to collect 24 samples with a fixed 10-min-interval and a sample volume of 250 ml (or the maximum sample volume if less than 250 ml).
- 3) Operate the programme at a lift height of 1 m.
- 4) Measure and record the volume of each sample.

- 5) Repeat the sampling programme at the rated maximum lift height and at half the rated maximum lift height. In each case, measure and record the volume of each sample.
- 6) Calculate and report in accordance with A.1.

This test may be combined with 6.4.2.2 by recording the time at which the first six samples are taken.

#### **6.4.1.2 C.T.V.V. flow proportional sampling principle**

Determine the sample volume errors in accordance with the following procedure.

For samplers capable of operating with different input signals (analogue, pulse, digital) each input should be tested separately.

- 1) Programme the sampler for the collection of C.T.V.V. flow proportional samples at a rate of 4 samples per hour and a sample volume of 25 ml/ m<sup>3</sup>. If possible programme the input to range such that 100 % equals 100 m<sup>3</sup>/h.
- 2) Operate the sampler at a lift height of 1 m.
- 3) Apply a simulated flow signal representing 25 % of the maximum input signal until 6 individual samples have been collected.
- 4) Sequentially increase the flow signal to represent flow rates of 50 %, 75 % and 100 % and in each case collect 6 individual samples.
- 5) Determine the volume of each sample and the simulated flow volume increment between each sample event.
- 6) Repeat the procedure from step 2 for lift heights at the rated maximum lift height and at half the rated maximum lift height. In each case, measure and record the volume of each sample.
- 7) Calculate and report in accordance with A.1.

#### **6.4.2 Testing of sampling principles**

##### **6.4.2.1 General**

For samplers capable of operation under more than one sampling principle, i. e. C.T.C.V. time proportional, C.V.V.T. flow proportional impulse, C.V.V.T. flow proportional analogue, and event triggered, all available sampling principle options shall be tested.

##### **6.4.2.2 C.T.C.V**

This test may be combined with 6.4.1.

Samplers operating on the C.T.C.V. time proportional sampling principle shall be tested as follows:

- 1) Programme the sampler for the collection of time related samples based on 10 min per one 250 ml sample;
- 2) Operate the sampler at a sampling head of 1 m;
- 3) Run the sampler with this setup for 60 min;
- 4) Determine and report how many, and at what time, samples have been taken during this test period, by manually monitoring during the test.

#### 6.4.2.3 C.V.V.T (impulse flow signal)

Samplers operating on the C.V.V.T. flow proportional sampling principle controlled by an impulse signal shall be tested as follows:

- 1) Connect the sampler to a suitable flow impulse generator;
- 2) Programme the sampler for the collection of flow related samples based on 10 impulses per one 250 ml sample;
- 3) Operate the sampler at a sampling head of 1 m;
- 4) Apply a simulated flow impulse at a fixed frequency of 1 impulse per minute;
- 5) Run the sampler with this setup for 60 min;
- 6) Determine and report how many, and at what time, samples have been taken during this test period, by manually monitoring during the test.

#### 6.4.2.4 C.V.V.T (analogue flow signal)

Samplers operating on the C.V.V.T flow proportional sampling principle controlled by an analogue signal shall be tested as follows:

- 1) Connect the sampler to a suitable analogue flow signal generator;
- 2) Programme the sampler for the collection of flow related samples based on the analogue input with 1 sample to be taken every 5 min with the maximum input flow rate signal;
- 3) Operate the sampler at a sampling head of 1 m;
- 4) Apply a simulated flow rate signal representing 50 % of the maximum flow;
- 5) Run the sampler with this setup for 60 min;
- 6) Determine and report how many, and at what time, samples have been taken during this test period, by manually monitoring during the test.

#### 6.4.2.5 Event triggered sampling

Samplers operating on the event triggered sampling principle shall be tested as follows:

- 1) Connect the sampler to a suitable event trigger generator;
- 2) Programme the sampler for the collection of event triggered samples based on 1 trigger per one 250 ml sample;
- 3) Operate the sampler at a sampling head of 1 m;
- 4) Apply a simulated event trigger at a fixed frequency of 1 trigger per 10 min;
- 5) Run the sampler with this setup for 60 min;
- 6) Determine and report how many, and at what time, samples have been taken during this test period, by manually monitoring during the test.



### 6.4.3 Sample line velocity

The sampler shall be operated at lift heights between 1 m and the maximum rated lift height in increments of 1 m. Determine the mean sample line velocity at each lift height by the following procedure:

- 1) Set up the sampler with a vertical sampling line of total length equal to the sampler's maximum rated lift height plus 1 m. Calibrate the sampling line with marks at 1 m divisions from a reference point at the surface of the liquid being sampled to the inlet port of the sampler;
- 2) Operate the sampler and determine the time taken from the instant that the leading edge of the sample fluid passes the reference point to when the leading edge passes the 1 m mark;
- 3) Repeat step 2 two more times to provide three determinations at the 1 m lift height;
- 4) Increase the lift height by 1 m intervals up to the sampler's maximum rated lift height and repeat steps 2 and 3 at each metre interval mark;
- 5) Calculate the mean sample line velocity in metres per second (m/s) at each lift height by dividing the height in metres by the time taken in seconds;
- 6) Report the individual test results, the mean sample velocity and the associated lift height.

Prior to undertaking the sample line velocity test, battery powered samplers shall be conditioned as follows to demonstrate the battery capacity and to ensure that the sampler can operate for more than 24 h. To condition the sampler, fully charge the battery then using the sampler collect 24 samples under the following conditions:

- sample interval 1 h;
- sample volume 250 ml (or the maximum sample volume if less than 250 ml);
- the rated maximum lift height;
- carry out steps 1 to 6 of the sample line velocity test within a period of 2 h after the completion of the sampling operation without recharging the battery.

### 6.4.4 Power supply test

#### 6.4.4.1 Mains supply samplers

For mains supply samplers, the following power supply test shall be carried out:

- 1) Operate the sampler at the rated maximum lift height;
- 2) Set the supply voltage to the maximum rated voltage for the sampler;
- 3) Carry out the sample line velocity test detailed in 6.4.3;
- 4) Report:
  - a) the voltage at which the low power alarm is displayed or the sampler ceases to operate;
  - b) the sample line velocity.
- 5) Repeat steps 1 to 4 with the supply voltage set to the minimum rated voltage for the sampler.

#### 6.4.4.2 DC and battery powered samplers

For both DC and battery powered samplers the following power supply test shall be carried out:

- 1) Remove the batteries from the sampler and substitute a variable DC power supply;
- 2) Set the supply voltage to the rated voltage for the sampler;
- 3) Operate the sampler at the rated maximum lift height;
- 4) Carry out the sample line velocity test detailed in 6.4.3;
- 5) Increase the supply voltage by 10 % and repeat the sample line velocity test detailed in 6.4.3;
- 6) Return the voltage to the rated voltage for the sampler;
- 7) Reduce the voltage by 0,5 V and repeat the sample line velocity test detailed in 6.4.3;
- 8) Repeat step 7 until a low power alarm is displayed or the sampler ceases to operate;
- 9) Report:
  - a) the voltage at which the low power alarm is displayed or the sampler ceases to operate;
  - b) the mean sample velocity and the associated supply voltage;
  - c) identify the maximum change in sample velocity caused by changes to the supply voltage.

#### 6.4.5 Sample integrity

The test for sample integrity is carried out at the rated maximum lift height and at half the rated maximum lift height. The sampler shall be set up in manual grab sample mode and set up to sample a test fluid which is representative of waste water from an urban waste water treatment plant regulated under the UWWTD.

NOTE Annex B provides information on concentration ranges for biochemical oxygen demand (BOD), chemical oxygen demand (COD), total nitrogen, and total phosphorus in waste waters.

During the test, the test fluid shall be maintained in a homogenous state, this can be achieved by the use of a mechanical stirrer or a small submersible pump. The method of stirring shall avoid drawing air into the test fluid.

- 1) Obtain 50 l of test fluid and a supply of demineralized water;

If the sampler works with a conductivity sensor to monitor the presence of sample liquid a dilute solution of sodium chloride with a conductivity appropriate to the sampler may be used.

- 2) Install the sampler at a lift height of half the rated maximum;
- 3) Insert the sample line in a container of fresh demineralized water;
- 4) Operate the sampler and collect 1 l of demineralized water as four discrete samples of 250 ml each. Discard each sample;
- 5) Relocate the sample line into the container of test fluid, operate the sampler and collect a composite 1 l sample of test fluid by combining four discrete samples of 250 ml each;

- 6) Obtain 1 l of test fluid manually from the container of test fluid in such a way as to ensure that this sample is representative, in terms of the determinands of interest, of the test fluid;
- 7) Label each sample with a unique code. Record and report the date, time, sampling height and sample codes;
- 8) Place the sample line into a container of fresh demineralized water and repeat steps 4 to 7, then proceed to step 9;
- 9) Relocate the sampler at the rated maximum lift height. The sampler can be calibrated to the new lift height before re-starting the sampling programme;
- 10) Repeat steps 3 to 8 inclusive;
- 11) Determine the concentration of BOD (biochemical oxygen demand), COD (chemical oxygen demand), total nitrogen, and total phosphorus in the eight samples of test fluid (4 manual samples and 4 composite samples obtained using the sampler) using standard reference analytical methods;
- 12) Undertake an analysis of variance in accordance with A.3. Report the analysis of variance calculation.

#### **6.4.6 Sampler timing error**

The sampler shall be tested for sample timing error by the following procedure.

Programme the sampler to collect 24 samples with a sampling interval of 60 min and a sample volume of 250 ml (or the maximum sample volume if less than 250 ml).

- 1) Operate the sampler at any convenient lift height;
- 2) Determine the elapsed time from the instant the sampler activates for the first sample to the instant the sampler starts to collect the 24th sample;
- 3) Calculate the timing error as the elapsed time between the first and last sampling action minus 23 h, such that if the timing function overruns the error is reported as positive;
- 4) Normalise the reported error to seconds per 24 h by dividing by 23 and multiplying by 24.

#### **6.4.7 Ambient air temperature effects**

##### **6.4.7.1 Test settings**

The sampler shall be tested for sensitivity to ambient air temperature by the following volumetric test and sample temperature control test procedures.

Install the sampler within a temperature controlled test chamber. Maintain the temperature of the air surrounding the sampler at the values specified in 5.7.1 and 5.7.2 with a tolerance of  $\pm 2$  °C. Allow sufficient time to ensure that the equipment under test has reached a stable operating temperature (at least 5 h) before the sampling programme is initiated. During each test, maintain the temperature of the fluid being sampled at a value of  $20$  °C  $\pm 2,5$  °C.

##### **6.4.7.2 Volumetric test**

Undertake the following test once at each of the rated lower and upper operating ambient temperature limits for the sampler (see 5.7):

- 1) Programme the sampler to collect 24 samples with a sampling interval of 60 min and a sample volume of 250 ml (or the maximum sample volume if less than 250 ml, in which case state the actual volume used);

NOTE In the case of samplers with less than 24 bottles then the samples collected will be distributed equally between the available number of bottles.

- 2) Operate the sampler at its rated lower operating ambient temperature for 24 h. Measure the sample volume in each of the sample bottles and examine the sample fault log for indication of malfunction;
- 3) Operate the sampler at its rated upper operating ambient temperature for 24 h. Measure the volume of sample in each of the sample bottles and examine the sample fault log for indication of malfunction;
- 4) Calculate and report in accordance with A.1;
- 5) Report the details of any malfunction identified by the sample fault log.

#### 6.4.7.3 Sample temperature control test

This test applies only to samplers with temperature control. Test the sampler at ambient temperatures of +10 °C, +20 °C and +40 °C.

- 1) Programme the sampler to collect 24 samples with a sampling interval of 60 min and a sample volume of 250 ml (or the maximum sample volume if less than 250 ml; state the volume used). Collect the samples as a composite into a single container. Install calibrated thermocouples into the middle of the composite sample container, the source fluid container and the test chamber and away from interfering airflow. High performance thermocouples with repeatability ( $\pm 0,1$  °C) and tolerance ( $\pm 0,5$  °C) over the range from -40 °C to +125 °C should be used. Record the output from each thermocouple independently taking a reading at least every 5 s. Report these readings as a series of averages calculated at 5 min intervals throughout the test period.
- 2) Operate the sampler at each ambient test temperature for 24 h.
- 3) At the end of the sampling period, leave the sampler in the test chamber for a further 24 h at the ambient test temperature.
- 4) Examine the sample collected and observe and report whether there is any formation of ice within the sample.
- 5) For each ambient test temperature calculate and report in accordance with A.5:
  - the maximum, minimum and mean sample temperature during the 24 h sampling period;
  - the maximum, minimum and mean sample temperature during the 24 h post sampling period. If the sample temperature exceeds the range 0 °C to 5 °C during the post sampling period, identify the time at which this condition occurred and the time period over which the sample temperature remained outside the specified temperature values;
  - the maximum, minimum and mean ambient test temperature within the test chamber during the 48 h test period;
  - the maximum, minimum and mean temperature of the fluid being sampled during the 24 h sampling period.
- 6) Report, in graphical form, the recorded temperature within the sample from the start of the test to the completion of the 24 h post-sampling period.

## Annex A (normative)

### Evaluation of conformity test data

#### A.1 Sample volume error

Calculate:

- 1) The mean sample volume at each tested lift height;
- 2) The bias (in %), as the mean volume minus the set volume divided by the mean volume, for each test height according to Formula A.1:

$$b = \left( \frac{V_1 - V_2}{V_1} \right) \times 100 \quad (\text{A.1})$$

where

- $b$  is the bias in percent (%);
- $V_1$  is the mean sample volume in millilitres (ml);
- $V_2$  is the set sample volume in millilitres (ml).

- 3) The precision (in %), as 1,96 times the sample standard deviation of the measured volumes divided by the mean volume at the 95 % confidence limit according to Formula A.2:

$$S = \left( \frac{s}{V_1} \times 100 \right) \times 1,96 \quad (\text{A.2})$$

where

- $S$  is the precision in percent (%);
- $s$  standard deviation of the measured volume as a percentage of mean sample volume;
- $V_1$  is the mean sample volume in millilitres (ml).

In practice a factor of 2 rather than 1,96 is used. The worked example given in Table A.1 for an automated sampler tested at 1 m, 3,5 m and 7 m lift heights uses a factor of 2.

Note that the bias and precision sample volume error requirements are specified at 5.1.

Table A.1 — Sample volume error (worked example according to 6.4.1)

Lift height	7 m	3,5 m	1 m	7 m	3,5 m	1 m	7 m	3,5 m	1 m
	Run Numbers								
	1A	2A	3A	1B	2B	3B	1C	2C	3C
Sample number	Volumes collected ml								
1	247	234	247	246	247	250	238	240	240
2	249	256	251	249	250	248	246	259	247
3	248	245	250	249	251	254	248	258	248
4	248	245	248	253	250	249	248	249	247
5	248	244	244	250	242	249	249	250	252
6	247	244	245	254	248	248	254	255	258
7	254	245	245	248	250	248	250	249	252
8	248	245	245	249	252	250	256	256	253
9	246	245	246	242	250	243	253	250	252
10	249	248	244	245	248	247	244	247	241
11	248	244	249	249	252	250	240	241	246
12	246	256	244	248	254	248	248	249	248
13	250	245	246	244	246	250	248	250	250
14	247	244	245	246	249	250	249	243	254
15	249	245	245	242	250	241	240	250	240
16	249	246	248	244	248	242	249	250	254
17	249	245	247	248	249	250	248	254	250
18	247	245	248	247	248	249	248	249	250
19	249	246	246	246	250	248	247	249	248
20	249	245	246	250	250	254	256	250	249
21	248	246	246	250	249	248	248	249	249
22	248	246	246	248	249	253	256	249	250
23	249	245	248	242	243	250	248	250	248
24	248	248	245	249	248	250	249	250	247
Set volume (ml)	250	250	250	250	250	250	250	250	250
Mean sample volume (ml)	248,33	245,71	246,42	247,42	248,88	248,71	248,33	249,83	248,88
Standard deviation (ml)	1,58	4,05	1,89	3,17	2,61	3,17	4,70	4,51	4,32
Standard deviation, %	0,64	1,65	0,77	1,28	1,05	1,27	1,89	1,80	1,73
Precision, %	1,27	3,30	1,53	2,57	2,10	2,55	3,78	3,61	3,47
Bias, %	-0,67	-1,75	-1,45	-1,04	-0,45	-0,52	-0,67	-0,07	-0,45

Average	1 m	3,5 m	7 m
Precision, %	2,52	3,00	2,54
Bias, %	-0,81	-0,76	-0,80

## A.2 Sample line velocity

Test method is given in 6.4.3.

A worked example is given in Table A.2 for an automated sampler with an internal sample line diameter of 9 mm tested up to 7 m lift height.

**Table A.2 — Test of sample line velocity (worked example according to 6.4.3)**

Lift height m	Run	Measured time s	Velocity m/s	Average flow velocity m/s
2	a	3,49	0,57	0,57
	b	3,50	0,57	
	c	3,47	0,58	
3	a	5,31	0,56	0,57
	b	5,26	0,57	
	c	5,22	0,57	
4	a	7,10	0,56	0,56
	b	7,15	0,56	
	c	6,97	0,57	
5	a	8,88	0,56	0,57
	b	8,55	0,58	
	c	8,70	0,57	
6	a	14,00	0,43	0,42
	b	14,45	0,42	
	c	14,21	0,42	
7	a	20,25	0,35	0,34
	b	21,21	0,33	
	c	20,10	0,35	

Note that values for average flow velocity of < 0,5m/s do not meet the requirement specified in 5.2.

## A.3 Sample integrity

### A.3.1 Calculation of results based on the analysis of variance

The analysis of variance technique is a statistical tool used to test the hypothesis that means from two or more samples are drawn from populations with the same mean, i.e. whether the variations seen in two sets of data are statistically significant. The formulae and numbers presented here are correct for this application, with 2 replications, 2 depths and 2 'treatments' (sample/reference).

### A.3.2 Notation

The observed (measured) values are denoted by  $A_{i,j,k}$ , where the suffixes are given in Table A.3.

**Table A.3 — Notation**

Suffix	Value = 1	Value = 2
i	Replicate 1	Replicate 2
j	Lift height = 3,5 m	Lift height = 7 m
k	Reference	Sample

The same suffix notation is also used for any derived values.

Averages are denoted by a bar, e.g.

$\bar{A}_{1,2}$  is the average over the replications at lift height 3,5 m ( $j = 1$ ) for the sample ( $k = 2$ ).

In symbols:

$$\bar{A}_{1,2} = (A_{1,1,2} + A_{2,1,2}) / 2$$

Similarly:

$\bar{A}_{..k}$  is the average over all replications and lift heights, and  $\bar{A}_{...}$  is the overall average.

$N$  is the total number of observations (= 8).

### A.3.3 Calculations

The main effects are estimated using a two factor analysis as:

$$\text{Effect of lift height} \quad (E_d)_j = \bar{A}_{.j} - \bar{A}_{...}$$

$$\text{Effect of treatment} \quad (E_t)_k = \bar{A}_{..k} - \bar{A}_{...}$$

For each effect,  $E$ , there are two values which are equal in magnitude but of opposite sign, i.e.

$$(E_d)_1 + (E_d)_2 = 0 \text{ and}$$

$$(E_t)_1 + (E_t)_2 = 0$$

The interactions,  $I$ , (i.e. whether the effect of lift height is different between reference and sample, or the effect of reference/sample is different at different lift heights) are estimated as:

$$I_{j,k} = \bar{A}_{j,k} - \bar{A}_{.j} - \bar{A}_{..k} + \bar{A}_{...}$$

Although there are 4 different interactions, there is only one independent value, and the other 3 are either equal to it or equal but of opposite sign.

Predicted values for each lift height and for sample/reference are calculated as the averages  $\bar{A}_{j,k}$ .

The Analysis of Variance (ANOVA) is then calculated as shown in Table A.4. Many commercial spreadsheets incorporate this analysis.



**Table A.4 — Analysis of Variance Calculations**

Source of Variation	Sum of Squares $S$	Degrees of Freedom $D$	Mean Square $M$	$F$	P-value	$F_{crit}^a$
Lift height <sup>b</sup>	$S_d = N \times (E_d)^2$	$D_d = 1$	$M_d = S_d/D_d$	$M_d/M_r$	<sup>c</sup>	7,71
Treatment <sup>b</sup>	$S_t = N \times (E_t)^2$	$D_t = 1$	$M_s = S_t/D_t$	$M_s/M_r$	<sup>c</sup>	7,71
Interaction	$S_l = N \times I^2$	$D_l = 1$	$M_l = S_l/D_l$	$M_l/M_r$	<sup>c</sup>	7,71
Residual	$S_r = \Sigma(A_{i,j,k} - \bar{A}_{j,k})^2$	$D_r = 4$	$M_r = S_r/D_r$			
Check <sup>d</sup>	$S_d+S_s+S_l+S_r$					
Total <sup>d</sup>	$\Sigma(A_{i,j,k} - \bar{A}_{...})^2$	7				

<sup>a</sup> This value is obtained from the F distribution (tables or in software), with degrees of freedom corresponding to (i) each row and (ii) the residual and with a level of significance of 5 %.

<sup>b</sup> Since  $(E_d)_1 = - (E_d)_2$  and  $(E_t)_1 = - (E_t)_2$  either value may be used in each case.

<sup>c</sup> This value is provided by some spreadsheets. It is the probability of the observed differences, or more extreme differences, being obtained by chance, on a scale of 0 to 1. The lower it is, the more definite the evidence for a real difference.

<sup>d</sup> When calculated, the values in these rows for the sum of squares column should be equal.

### A.3.4 Interpretation of the results

When the values in Table A.4 are calculated, if  $F > F_{crit}$  on any line, then there is evidence of a significant difference.

A worked example is given below in Table A.5 and Table A.6 and an example ANOVA calculation is given at Table A.7

**Table A.5 — Data set (worked example)**

Treatment	Lift Height m	Total Nitrogen mg/l	Symbols
Reference	3,5	10,5	$A_{1,1,1}$
Reference	3,5	10,3	$A_{2,1,1}$
Reference	7	10,2	$A_{1,2,1}$
Reference	7	10,3	$A_{2,2,1}$
Sample	3,5	10,2	$A_{1,1,2}$
Sample	3,5	10,1	$A_{2,1,2}$
Sample	7	9,8	$A_{1,2,2}$
Sample	7	10	$A_{2,2,2}$

**Table A.6 — Intermediate calculation (worked example)**

Overall mean	$A_{...}$	10,175	$N$	8
Effect of Head	$A_{.1.}$	10,275	$(E_d)_1$	0,1
	$A_{.2.}$	10,075	$(E_d)_2$	-0,1
Effect of Treatment	$A_{..1}$	10,325	$(E_t)_1$	0,15
	$A_{..2}$	10,025	$(E_t)_2$	-0,15
Interactions	$A_{.11}$	10,4	$I_{.11}$	-0,025
	$A_{.12}$	10,15	$I_{.12}$	0,025
	$A_{.21}$	10,25	$I_{.21}$	0,025
	$A_{.22}$	9,9	$I_{.22}$	-0,025
	$S_r = \sum(A_{ijk} - \bar{A}_{jk})^2$	0,05		
	$\sum(A_{ijk} - \bar{A}_{...})^2$	0,315		

**Table A.7 — ANOVA Table (worked example)**

Source of Variation	Sum of Squares $S$	Degrees of Freedom $D$	Mean Square $M$	$F$	P-value <sup>c</sup>	$F_{crit}$ <sup>a</sup>
Sampling head <sup>b</sup>	0,08	1	0,08	6,4	<sup>c</sup>	7,71
Treatment <sup>b</sup>	0,18	1	0,18	14,4	<sup>c</sup>	7,71
Interaction	0,005	1	0,005	0,4	<sup>c</sup>	7,71
Residual	0,05	4	0,0125			
Check <sup>d</sup>	0,315					
Total <sup>d</sup>	0,315	7				

<sup>a</sup> This value is obtained from the F distribution (tables or in software), with degrees of freedom corresponding to (i) each row and (ii) the residual and with a level of significance of 5 %.

<sup>b</sup> Since  $(E_d)_1 = - (E_d)_2$  and  $(E_t)_1 = - (E_t)_2$  either value may be used in each case.

<sup>c</sup> This value is provided by some spreadsheets. It is the probability of the observed differences, or more extreme differences, being obtained by chance, on a scale of 0 to 1. The lower it is, the more definite the evidence for a real difference.

<sup>d</sup> When calculated, the values in these rows for the sum of squares column should be equal.

Conclusion : The treatment factor has a significant influence on the results as  $F$  value is greater than 7,71.

#### **A.4 Sampler timing error**

Test method is given in 6.4.6.

Data should be presented as shown in the worked example at Table A.8.

**Table A.8 — Sample timing error (worked example according to 6.4.6)**

	hh:mm:ss
Activation time for 1st sample	12:59:20
Activation time for 24th sample	11:59:15
Elapsed time for 24 samples	22:59:55
Timing error	-00:00:05
Normalised timing error	5,2s

#### **A.5 Ambient air temperature effects**

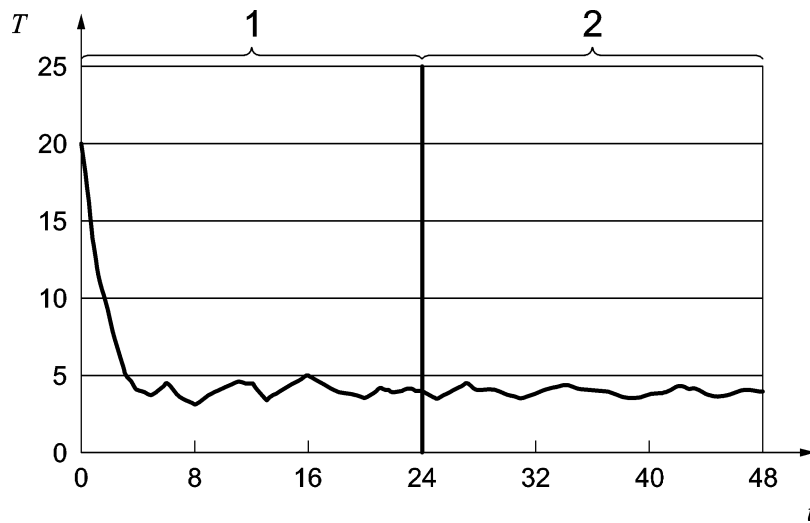
Test method is given in 6.4.7.

Data to be presented as in the example below:

EXAMPLE Automated sampler ambient temperature effects at 20 °C.

Table A.9 — Ambient air temperature effects (example according to 6.4.7)

	Maximum temperature °C	Minimum temperature °C	Mean temperature °C
Environmental chamber temperature	22,8	20,3	20,8
Temperature of water being sampled	21	19,5	20,1
Temperature of sample (over 24 h sampling)	20,1	3,1	5,3
Temperature of sample (over 24 h post sampling)	4,5	3,6	4,0



**Key**

- $t$  Time in h
- $T$  Temperature in °C
- 1 Sampling period (24 h)
- 2 Post-sampling period (24 h)

Figure A.1 — Ambient temperature effects at 20 °C on the sample temperature (see 6.4.7)

## Annex B (informative)

### Example procedure for demonstrating sample integrity for samplers to be used for Urban Waste Water Treatment Directive (UWWTD) sampling

#### B.1 General

The objective of setting sample integrity requirements is to ensure that the chemical composition of samples is not altered by the sampler's sampling system. This annex describes a procedure for determining conformance with the sample integrity requirements specified in 5.5 in accordance with the conformity test specified in 6.4.5.

#### B.2 Test fluid

Carry out the sample integrity test using a single test fluid where the values for all the determinands in Table B.1 are within the specified ranges, or with a number of test fluids each of which has one or more of the determinands in Table B.1 within the specified ranges. In the latter case, the test shall be repeated until the integrity of all four determinands listed below has been tested.

**Table B.1 — Determinand value ranges**

Determinand	Lower limit mg/l	Upper limit mg/l
Suspended solids	17,5	52,5
COD	62,0	187
BOD	12,5	37,5
Total nitrogen	7,5	22,5
Total phosphorus	1,0	3,0

NOTE 1 Total nitrogen "means sum of total Kjeldahl nitrogen (organic and ammoniacal nitrogen), nitrate – nitrogen and nitrite – nitrogen. See note to Table 2 in Commission Directive 98/15/EC of 27 February 1998.

NOTE 2 The UWWTD does not define total phosphorus but specifies the "reference method as molecular absorption spectrophotometry". See Table 2 in Commission Directive 98/15/EC of 27 February 1998.

NOTE 3 The range for BOD, COD, total nitrogen and total phosphorous is the permitted concentration value  $\pm 50\%$  of that value given in Tables 1 and 2 of the Urban Waste Water Treatment Directive (91/271/EEC). Where Table 2 includes two ranges, the higher range has been used.

NOTE 4 Suspended solids are included in the matrix as it is an optional requirement in the Urban Waste Water Treatment Directive (91/271/EEC). The lower range for suspended solids from Table B.1 has been used. However, the integrity of solids collection is ensured through the requirements and tests for sample line velocity specified at 5.3 and 6.4.3 respectively.

#### B.3 Sample collection

Set up the sampler to take a minimum of 24 discrete samples without changing the sample containers .

Set up the sampler to maintain the temperature of the samples within pre-set limits of  $(3 \pm 2) ^\circ\text{C}$ .

#### **B.4 Sample volume**

Set up the sampler to:

- a) take discrete samples of not less than 200 ml, and
- b) collect not less than 2,5 l of sample in a composite or in individual bottles.

#### **B.5 Sample integrity**

In each of the eight samples (4 manual samples and 4 samples obtained using the sampler) collected determine the

- biochemical oxygen demand (BOD);
- chemical oxygen demand (COD);
- total nitrogen;
- total phosphorus.

#### **B.6 Determination of conformance**

Calculate the statistical differences for each determinand for the sets of samples collected by the sampler and manually using the analysis of variance.

## Annex C (informative)

### Example format for the report

Test results should be reported in a tabular format as shown below. In addition, numerical results from performance tests should also be presented graphically, where appropriate.

<b>Report format for performance evaluations of automated sampling devices (samplers) for water and waste water</b>		
Name of Test Organization		
Report type:		
Sampler tested:		
Manufacturer:		
Test period, from to		
Date of report:		
Report number:		
Scope of report:		
<b>Contents</b>		
<b>1</b>	<b>Synopsis</b>	
1.1	Summary of test results	<i>The report should include a brief summary of the performance of the sampler, stating the capabilities of the sampler with respect to the tested range(s).</i>
1.2	Sampler details	<i>The report should include the following information:</i> <ul style="list-style-type: none"> <li>- <i>specific sampler identity;</i></li> <li>- <i>field of application.</i></li> </ul> <i>Any restrictions. Such limitations should be recorded if testing shows that the sampler does not cover the full scope of possible application fields.</i> <i>Attention should be drawn to any equipment peculiarities.</i>
1.3	Previous test reports	<i>In cases of supplementary or extended testing, reference should be made to all preceding test reports and include the name of the test organization and the test report number and date of compilation.</i>
<b>2</b>	<b>Task definition</b>	
2.1	Nature of the tests	<i>First test or supplementary testing</i>
2.2	Objectives	<i>Specification of which performance specifications were tested.</i> <i>Bibliography.</i> <i>Scope of any supplementary tests.</i>
<b>3</b>	<b>Description of sampler tested</b>	
3.2	Sampler scope and set-up	<i>Description of all components covered in the scope of testing.</i> <i>Statement of technical specifications, if appropriate in tabular form.</i>

<b>Report format for performance evaluations of automated sampling devices (samplers) for water and waste water</b>		
<b>4</b>	<b>Test programme</b>	
4.1	Laboratory test / laboratory inspection	<i>Details should be provided on the test programme, in relation to the sampler under test. In the case of supplementary or extended testing, the additional scope of testing should be detailed and substantiated. Statement of all test steps involved.</i>
<b>5</b>	<b>Methods of reference measurements</b>	
5.1	Reference methods	<i>State the sample matrix used. State the reference method used for sample analysis.</i>
<b>6</b>	<b>Test procedure</b>	
6.1	Citation of performance specifications	<i>The version of the performance standard.</i>
6.2	Equipment	<i>The report should outline the type of equipment used for the test.</i>
6.3	Method	<i>There should be a brief description of the test.</i>
6.4	Maintenance	<i>Details of the necessary maintenance work and remedial actions necessary to ensure that the sampler met the performance standards should be reported.</i>
<b>7</b>	<b>Test results</b>	
7.1	Summary table of test results	<i>The test results should be summarized in a table and include the following information: - reference of the performance specification; - the performance specification in abbreviated form; - the test result; - compliance with the specification (yes/no); - a reference to the relevant page of the test report; - relevant comments and restrictions.</i>
7.2	Graphical presentation of results	
	<b>Appendices</b>	
A	Appendix A: Raw data from the tests	
B	Appendix B: Operating instructions	<i>Instructions and manuals for the sampler should be appended to the report.</i>



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- [6] Directive 2010/75/EU of the European parliament and the Council of 23 October 2010 on the limitation of industrial emissions (integrated pollution prevention and control)
- [7] Council Directive 91/271/EEC as amended by Commission Directive 98/15/EC concerning urban waste water treatment
- [8] Directive 2000/60/EC of the European Parliament and of the Council establishing a framework for the Community action in the field of water policy
- [9] Directive 2008/56/EC of the European Parliament and of the Council establishing a framework for the Community action in the field of marine environmental policy





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