



INZRAK

Enhanced environmental protection inspection for efficient control of air quality monitoring and of all entities under obligation within system of greenhouse gas emission allowance trading, in order to achieve better quality of air in Republic of Croatia



REPUBLIKA HRVATSKA

MINISTARSTVO ZAŠTITE
OKOLIŠA I ENERGETIKE



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FINANCIARANJE I UGOVARANJE



This project is funded by the European Union



EKOENERG

Energy research and Environmental Protection Institute



TOPIC 9: Normative regulation

9.1 HRN EN ISO/IEC 17025

LABORATORIES

A body performing one or more of the following activities: examination, calibration, sampling related to incoming examination or calibration.

Air quality testing laboratories conduct pollutant concentration measurements in the air.

The calibration laboratories provide the measuring traceability of the analyzers that are conducting the measurements.

In what way any laboratory can prove its ability, impartiality, expertise and consistent work?

ANSWER: by applying the requirements of HRN EN ISO/IEC 17025.

9.1 HRN EN ISO/IEC 17025

IMPORTANCE OF THE STANDARD

HRN EN ISO/IEC 17025 is a standard that provides requirements for laboratory qualification.

International standard HRN EN ISO/IEC 17025 promotes confidence in the laboratory work by setting requirements by fulfilling which laboratories can prove that their activities are conducted impartially, professionally and correctly in order to provide a quality results to their customers.

Laboratories operating in accordance with the requirements of HRN EN ISO/IEC 17025 are considered as skilled laboratories, and this ability can demonstrate to the accreditation body (in the RH: HAA) which, once it is found that all requirements are fulfilled, it can issue a certificate of accreditation, which is a formal proof of such qualification issued by a third independent party.

9.1 HRN EN ISO/IEC 17025

HISTORICAL BACKGROUND

ISO/IEC 17025 is an internationally recognized laboratory qualification standard. Through the accreditation system, the results of accredited laboratories around the world are mutually recognized without further verification.

ISO/IEC 17025 is the basis for laboratory accreditation. It is used by all accreditation bodies in the world as a fundamental document by which the labs prove their own competence.

ISO/IEC 17025 encompasses management requirements (in compliance with ISO 9001) and technical requirements related to specific laboratory activities.

9.1 HRN EN ISO/IEC 17025

HISTORICAL BACKGROUND

The European standard EN 45001: 1989, which is the forerunner of international standard HRN EN ISO/IEC 17025, was published in **1989**.

When the accreditation system started to develop in Croatia, this standard was accepted as HRN EN 45001:1996.

In **1998**, the first certificates of accreditation of laboratories in Croatia were issued.

The International Standardization Organization, ISO, publishes an improved version of EN 45001 as ISO/IEC 17025: 1999, as the first edition of the International Standard for laboratory competence.

9.1 HRN EN ISO/IEC 17025

HISTORICAL BACKGROUND

The **first edition of ISO/IEC 17025: 1999** was adopted next year as the Croatian Publication of HRN EN ISO/IEC 17025:2000.

The **second edition of ISO/IEC 17025: 2005** published in 2005 is aligned with the latest issue of ISO 9001.

The Croatian editions are not accompanied by ISO/IEC publications, such as the second edition of ISO/IEC 17025: 2006 + AC: 2006 in the Republic of Croatia published in Croatian language as the fifth edition of HRN EN ISO/IEC 17025:2007.

The **third edition of ISO/IEC 17025: 2017** was recently published (1.12.2017), and is in the process of being adopted worldwide. It brings a lot of news. Laboratories shall be complied with a new release until 1.12.2020.

9.1 HRN EN ISO/IEC 17025

ACCREDITATION

Laboratories operating in accordance with HRN EN ISO/IEC 17025 can be accredited. Accreditation is a third independent party that confirms the laboratory competence.

The accreditation system is developing in each country. Each country has ONE accreditation body, as the umbrella organization of quality and acting independently.

The accreditation body evaluates the laboratory and if the laboratory proves its qualification according to ISO/IEC 17025, it obtains the accreditation certificate, with the exact area for which the laboratory is capable of carrying out its activities.

In Croatia, the Croatian Accreditation Agency (HAA) is the accreditation body.

9.1 HRN EN ISO/IEC 17025

STANDARD STRUCTURE

HRN EN ISO/IEC 17025	
Management requirements	Chapter 4 4.1-4.15
Technical requirements	Chapter 5 5.1-5.10

9.1 HRN EN ISO/IEC 17025

	Standard item
Organization	4.1
Management system	4.2
Document control	4.3
Review of requests, tenders and contracts	4.4
Subcontracting of tests and calibrations	4.5
Purchasing services and supplies	4.6
Service to the customer	4.7
Complaints	4.8
Control of nonconforming testing	4.9
Improvement	4.10
Corrective action	4.11
Preventive action	4.12
Control of records	4.13
Internal audits	4.14
Management reviews	4.15

9.1 HRN EN ISO/IEC 17025

	Standard item
General	5.1
PERSONNEL	5.2
ACCOMMODATION AND ENVIRONMENTAL CONDITIONS	5.3
TEST AND CALIBRATION METHODS	5.4
EQUIPMENT	5.5
MEASUREMENT TRACEABILITY	5.6
SAMPLING	5.7
HANDLING OF TEST AND CALIBRATION ITEMS	5.8
ASSURING THE QUALITY OF RESULTS	5.9
REPORTING THE RESULTS	5.10

9.1 HRN EN ISO/IEC 17025

4.1 ORGANIZATION

Laboratories must be a legally recognizable body (or organization within which the laboratory works)

It must be responsible for all its activities in any way (on a permanent lab locations, temporary locations, mobile or stationary)

It must work independently, professionally, consistently and impartially - it must not exist conflict of interest

The powers and responsibilities must be defined

An effective organization must be provided

There must be policies and procedures for protecting confidential information

There must be policies and procedures to ensure expertise, impartiality, judgments and consistency in work

Inter-relations within the laboratory have to be defined

9.1 HRN EN ISO/IEC 17025

4.1 ORGANIZATION

There must be a responsible staff (Laboratory Manager)

Staff supervision must be carried out

There must be a person responsible for the management system (Laboratory Quality Representative)

Staff must be aware of the importance of their processes

They need to develop and maintain appropriate communication processes

This can be achieved by describing how the requirements are met *in the manual or documented procedure*.

9.1 HRN EN ISO/IEC 17025

4.2 MANAGEMENT SYSTEM

Laboratories must establish, apply and maintain a management system in accordance with HRN EN ISO/IEC 17025

There must be:

- Quality policy
- Handbook and documentation with a defined hierarchy
- Quality objectives

The board must decide on:

- the development and implementation of the management system and its continuous improvement of its effectiveness
- communication to the organization the importance of customer requests, regulations and others
- ensuring the integrity when changes are made

9.1 HRN EN ISO/IEC 17025

4.3 DOCUMENT CONTROL

Laboratories must have a documented process for control of documents, internal and external

There must be a list of documents that the lab uses

The procedure must ensure:

- availability of valid documents
- periodical review of documents
- distribution of documents
- invalid document control
- keeping of old documents
- uniqueness of document marking

9.1 HRN EN ISO/IEC 17025

4.3 DOCUMENT CONTROL

- implementation of changes in documents
- procedures for modifying, reviewing and approving the document
- the procedure for issuing the document
- control of electronic documents

This can be achieved by describing how the requirements are met in appropriate ***documented procedure***.

9.1 HRN EN ISO/IEC 17025

4.4 REVIEW OF REQUESTS/TENDERS/CONTRACTS

Laboratories must have a documented procedure for reviewing the requests, tenders and contracts

The procedure must ensure:

- that the laboratory at its disposal has the necessary resources
- to properly select the method (testing, calibration, sampling)
- to have the ability to fulfil the customer's request
- to fully arrange with the customer what does he look for
- to conclude the job through the offer - by order or contract (everything must be defined; e.g. the method must be listed in the offer, order or contract)
- to keep records of everything, all subsequent agreements and modifications.

This can be achieved by describing how the requirements are met in appropriate ***documented procedure***.

9.1 HRN EN ISO/IEC 17025

4.5 SUBCONTRACTING

Laboratories can subcontract part of the service, and this must be a documented procedure.

Subcontracting in the laboratory:

- due to unforeseen circumstances (lack of resources, equipment failure, temporary disability, ...)
- permanent subcontracting (mediation and franchise agreements)

The laboratory may, under certain conditions, outsource part of the business to the subcontractor, if the customer agrees with it

Laboratory is responsible for subcontracting to its customer

The laboratory must have selection criteria and list of subcontractors

9.1 HRN EN ISO/IEC 17025

4.6 PROCUREMENT

Laboratories must have documented policies and procurement procedures

The procedure must ensure:

- procurement overview (services/equipment/chemicals...)
- keep records of inspection and check after procurement
- defining procurement criteria (purchasing items must include specifications)
- to have the technical content of the procurement documents reviewed and approved prior to the release
- to evaluate and rank the suppliers
- to keep a list of suppliers

This can be achieved by describing how the requirements are met in appropriate ***documented procedure.***

9.1 HRN EN ISO/IEC 17025

4.7 SERVICE TO THE CUSTOMER

Laboratories must be willing to cooperate with the customer

In case the customer wishes to participate in the activities that the laboratory conducts, it must respect the confidentiality of other customers

Laboratories must collect and analyze feedback information from the customer (e.g. by polling the customer)

Feedback information should be used to improve efficiency

This can be achieved by describing how the requirements are met in appropriate *documented procedure*.

9.1 HRN EN ISO/IEC 17025

4.8 COMPLAINTS

Laboratories must have documented policy and complaint handling procedure

The procedure must ensure a consistent investigation of the causes of the complaint

The records shall be kept on everything

This can be achieved by describing how the requirements are met in appropriate *documented procedure*.

9.1 HRN EN ISO/IEC 17025

4.9 CONTROL OF NONCONFORMING TESTING

Laboratories must have documented policies and procedures for occurrence of nonconforming work

In case of occurrence of nonconforming work

- responsibilities must be assigned, e.g. for termination of work, approval of continuation and similar
- corrections must be made, if necessary inform the customer
- evaluate the importance of nonconforming work
- assess the need to initiate a corrective action

This can be achieved by describing how the requirements are met in appropriate ***documented procedure***.

9.1 HRN EN ISO/IEC 17025

4.10 IMPROVEMENT

The laboratory must continually improve the efficiency of its management system using:

- quality policies,
- quality objectives,
- evaluation results,
- data analysis,
- corrective and preventive actions and
- management reviews.

9.1 HRN EN ISO/IEC 17025

4.11 CORRECTIVE ACTIONS

Laboratories must have documented policies and procedures for their application of corrective actions

When performing a corrective action, it must be:

- analyzed the cause of nonconformity/problem
- selected and applied the appropriate action
- monitored the implementation and evaluation of the effectiveness of the action
- in the toughest cases, conducted the internal audit

The application of the corrective action can be described in the appropriate ***documented procedure***.

9.1 HRN EN ISO/IEC 17025

4.12 PREVENTIVE ACTIONS

The laboratory must identify the necessary improvements and possible sources of nonconformity, either of a technical nature or of a management system.

When performing a preventive action, it must be :

- analyzed the cause of **potential** nonconformity/problem
- applied and monitored the action plans
- conducted the evaluation of the effectiveness of the action

The application of the preventive action can be described in the appropriate *documented procedure*.

9.1 HRN EN ISO/IEC 17025

4.12 PREVENTIVE ACTIONS

The laboratory must identify the necessary improvements and possible sources of nonconformity, either of a technical nature or of a management system.

When performing a preventive action, it must be :

- analyzed the cause of **potential** nonconformity/problem
- applied and monitored the action plans
- conducted the evaluation of the effectiveness of the action

The application of the preventive action can be described in the appropriate *documented procedure*.

9.1 HRN EN ISO/IEC 17025

4.13 CONTROL OF RECORDS

documents vs. records

INFORMATION – A set of meaningful data.

DOCUMENT - **Information** together with the media on which it was written (media: printed, electronic - paper, computer, CD, DVD, USB, server...).

RECORD - A **document** that indicates the obtained *results* or provides evidence of *performed actions*.

9.1 HRN EN ISO/IEC 17025

4.13 CONTROL OF RECORDS

The standard distinguishes:

a) *Quality system records*

- *internal audit reports*
- *management report*
- *report on preventive or corrective actions...*

a) *Technical records*

- *records on testing*
- *staff records*
- *testing report ...*

9.1 HRN EN ISO/IEC 17025

4.13 CONTROL OF RECORDS

The laboratory must establish and maintain procedures for identifying, collecting, indexing, accessing, filling, storing, maintaining and deleting records

Records must be:

- readable
- safely stored
- protected from access (confidentiality!)
- with a defined retention period
- with defined usage mode (use, copy...)

The application of the preventive action can be described in the appropriate ***documented procedure***.

9.1 HRN EN ISO/IEC 17025

4.13 CONTROL OF RECORDS

In addition, laboratories must define how to correct errors in records.

Observations, data and calculations must be recorded at the time they are performed and must be able to connect with these actions.

Records - proof of application of all requirements of the standard HRN EN ISO/IEC 17025!

The assessment of the requirements of HRN EN ISO/IEC 17025 is largely covered by the review and verification of the records.

9.1 HRN EN ISO/IEC 17025

4.14 INTERNAL AUDIT

The laboratory must periodically and in accordance with a pre-determined plan and procedure conduct internal audits of its work to see if this work is permanently in conformity with the requirements of the standard - at least ONCE A YEAR

The following should be done:

- carry out audits according to the established audit programme
- organize by quality representative
- ensure that audits are carried out by qualified and independent persons
- take the necessary action when the audit findings point to the deviation
- record the audit record

The application of internal audit can be described in the appropriate ***documented procedure***.

9.1 HRN EN ISO/IEC 17025

4.15 MANAGEMENT REVIEW

In accordance with the pre-established plan and procedure, the laboratory management shall periodically carry out the assessment of the laboratory management system to ensure its permanent suitability and efficiency - at least ONCE A YEAR

Management review includes:

- suitability of policies and procedures
- reports of management and supervisory staff
- the results of the latest internal audits
- corrective and preventive actions
- assessments carried out by external bodies
- results of interlaboratory comparisons or testing of abilities
- changes in scope and type of work

9.1 HRN EN ISO/IEC 17025

4.15 MANAGEMENT REVIEW

- feedback from the customer
- complaints
- recommendations for improvement
- other important factors, such as quality management, resources and staff training.

The application of management review can be described in the appropriate *documented procedure*.

9.1 HRN EN ISO/IEC 17025

5.1 GENERAL

Many factors determine the accuracy and reliability of testing and/or calibration conducted by the laboratory

These factors include contributions of:

- human factors (5.2)

- accommodation and environment conditions (5.3)

- testing and calibration methods and their validation (5.4)

- equipment (5.5)

- measurement traceability (5.6)

- sampling (5.7)

- handling objects for testing and calibration (5.8).

9.1 HRN EN ISO/IEC 17025

5.2 PERSONNEL

Personnel – one of the most important pillars of quality.

The criteria, powers and responsibilities of the functions must be defined

Education and training:

- initial training (new person, new function)
- continuous education and training (training programme!)

Management must indicate the needs and goals of staff training

9.1 HRN EN ISO/IEC 17025

5.2 PERSONNEL

Training:

- training program (which everyone must master)
- evaluation of effectiveness (confirmation that the program is mastered and the person is trained)

Records on personnel:

- certificates of education
- certificates of training
- records of permanent education
- all records that confirm the abilities, knowledge and skills

9.1 HRN EN ISO/IEC 17025

5.2 PERSONNEL

The laboratory management must ensure the training of all who:

- work with special equipment
- conduct tests and/or calibrations
- evaluate the results and
- sign test reports and calibration certificates

The following shall be defined:

- job descriptions
- personnel authority
- those who approve the reports

Application of personnel requests can be described in the appropriate ***documented procedure***.

9.1 HRN EN ISO/IEC 17025

5.3 ACCOMMODATION AND ENVIRONMENTAL CONDITIONS

Laboratory spaces must be correct for use

Fieldwork must be carried out in accordance with working conditions

Environmental conditions need to be recorded

Neighboring areas where nonconformity activities are carried out must be effectively separated

Measures should be taken to prevent mutually damaging effects

Measures should be taken to maintain the rooms in a laboratory

The application of internal audit can be described in the appropriate ***documented procedure***.

9.1 HRN EN ISO/IEC 17025

5.4 TESTING AND CALIBRATION METHODS

The laboratory must use appropriate methods and procedures for all testing (testing laboratory) or calibration (calibration laboratory) in the area of its operation.

Methods:

- a) Standardized (ISO, IEC, EN, HRN...)
- b) Non-standardized:
 - developed by the laboratory
 - customized standardized methods
 - published in any way (journals, scientific texts...)

9.1 HRN EN ISO/IEC 17025

5.4 TESTING AND CALIBRATION METHODS

All instructions, standards, manuals and reference data essential to the laboratory operation must be modernized and easily accessible to the personnel

The deviation from the testing and calibration method can only happen if the deviation is documented, technically justified and has been accepted by the customer

Validation methods - non-standardized methods must be validated.

Validation is a testimony by examining and gathering objective evidence that requirements for special intended use have been fulfilled.

9.1 HRN EN ISO/IEC 17025

5.4 TESTING AND CALIBRATION METHODS

Estimation of measurement uncertainty

Measurement uncertainty: parameter assigned to the result of the measurements describing the dispersion values that could reasonably be attributed to the measured value.

When measuring uncertainty, all components of uncertainty must be taken into account which are important in that case, using appropriate analytical methods.

The components are divided into components of:

- a) type A - statistical (repeatability)
- b) type B – non-statistical (resolution, impact assessment of appropriate parameters, from technical data etc.)

9.1 HRN EN ISO/IEC 17025

5.4 TESTING AND CALIBRATION METHODS

Budgeting and data transfers must be subjected to a systematic appropriate verifications.

Use of computers and automated equipment:

- a) the computer programs developed by the laboratory must be documented;
- b) appropriate validation of these programs must be undertaken;
- c) data protection procedures must be applied;
- d) computers and automated equipment must be maintained.

9.1 HRN EN ISO/IEC 17025

5.5 EQUIPMENT

The laboratory shall be **equipped with all equipment** for sampling, measurement and testing required for the proper conduct of testing or calibration

Equipment and its program support used for testing, calibration and sampling must be capable of achieving the **required accuracy** and must meet the specifications relating to the tests concerned or calibration

Equipment must be operated by authorized personnel.

Equipment must be maintained, serviced if necessary, calibrated...

9.1 HRN EN ISO/IEC 17025

5.5 EQUIPMENT

Equipment shall be:

- uniquely recognizable (tag!)
- calibrated according to the calibration program (calibrated equipment!)
- contain information on the calibration (when it is calibrated, as long as it is valid)
- supported by appropriate records (instructions, equipment datasheet...)
- withdrawn from use if it is defective
- protected against failure, overloading etc.
- checked occasionally

Equipment requirements may be described in the appropriate ***documented procedure***

9.1 HRN EN ISO/IEC 17025

5.6 MEASUREMENT TRACEABILITY

Traceability: The characteristic of the measurement result or the value of the standard to which it is derived can be related to the reference standards that are realized by the SI unit (usually state or international) unbroken by a line of comparisons that have established uncertainties.

Measurement traceability requires a **calibration hierarchy** established!

Calibration: An action by which under certain conditions in the first step is established a relationship between the value with the measurement uncertainties provided by measurement standards and corresponding indicators to which the measurement uncertainties are assigned. In the second step, these data are used to establish relationship to obtain the measurement result

9.1 HRN EN ISO/IEC 17025

5.6 MEASUREMENT TRACEABILITY

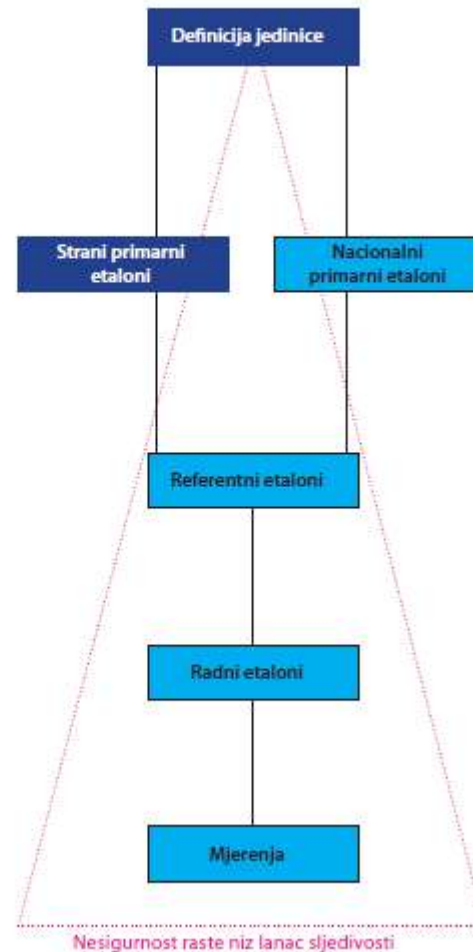
BIPM
(Bureau International des
Poids et Mesures)

Primarni laboratoriji
nacionalne mjeriteljske
ustanove ili imenovani
nacionalni laboratoriji

Umjerni laboratoriji, često
akreditirani

Poduzeća, fakulteti,
zakonodavstvo, bolnice

Krajnji korisnici



9.1 HRN EN ISO/IEC 17025

5.6 MEASUREMENT TRACEABILITY

Measurement traceability is provided by calibration laboratories!

Calibration is performed by:

- a) **Measurement standards** (according to traceability chain: primary standard – national standard - reference standard - working standard – meter)
- b) **Certified Reference Materials** ("CRM")

Measurement traceability must be described in the laboratory management system documentation.

9.1 HRN EN ISO/IEC 17025

5.7 SAMPLING

The laboratory must have a *sampling plan and sampling procedures* when conducting the sampling of substances, materials or products **which are then tested or calibrated**

The laboratory must have procedures for recording essential data and procedures that refer to the sampling that forms part of the testing or calibration.

All sampling records must be properly managed.

9.1 HRN EN ISO/IEC 17025

5.8 HANDLING OF TEST AND CALIBRATION ITEMS

The laboratory must have procedures for *transportation, reception, handling, protection, storing, storing or disposing of items* being tested or calibrated, including all the provisions necessary to protect the integrity of the subjects being tested or calibrated and protect the interests of a laboratory or customer

The laboratory must have a system for recognizing the items being tested or calibrated.

Recognition must be retained throughout the course of the laboratory

The system must be planned and implemented to ensure that items cannot to be replaced physically or when referred to in records or other documents

9.1 HRN EN ISO/IEC 17025

5.8 HANDLING OF TEST AND CALIBRATION ITEMS

Upon receipt of the item that will be tested or calibrated, the laboratory must record peculiarities or deviations from the usual or established conditions

Laboratory must have procedures and appropriate facilities and equipment to avoid the spoiling of the feature, loss or damage of the item being examined or calibrated

When items need to be stored or adjusted under established environmentally conditions, these conditions must be maintained, monitored and recorded

9.1 HRN EN ISO/IEC 17025

5.9 ASSURING THE QUALITY OF RESULTS

The laboratory must have quality management procedures that serve as supervision of validity of the conducted tests and calibration

Quality control are work techniques and actions that are being undertaken to meet quality requirements.

Quality assurance covers the overall quality system measures that ensure the proper trust that the quality requirements will be fulfilled.

The laboratory must develop a quality control strategy.

9.1 HRN EN ISO/IEC 17025

5.9 ASSURING THE QUALITY OF RESULTS

The quality control strategy encompasses the application of appropriate measures which prove the quality of the results.

These measures may be internal or external.

The laboratory must develop a quality control program that includes applicable external and internal quality control measures.

Quality control data must be analyzed and when it is found out they are out of pre-determined criteria, the planned corrective action must be taken to prevent the display of invalid results.

9.1 HRN EN ISO/IEC 17025

5.9 ASSURING THE QUALITY OF RESULTS

Internal quality control measures are e.g.:

- a) use of reference materials or quality control materials
- b) use of traceable substitute instruments
- c) functional inspection of measuring and test equipment
- d) use of control or working standards with control charts
- e) inter-inspection of measuring equipment
- f) repeat testing or calibration using the same or different method
- g) repeated testing or calibration of retained items
- h) correlation of results for different properties of an item
- i) comparisons within the laboratory
- k) blind sample testing...

9.1 HRN EN ISO/IEC 17025

5.9 ASSURING THE QUALITY OF RESULTS

External quality control measures are:

- a) interlaboratory comparison
- b) proficiency testing

Interlaboratory comparison is an organization, implementation and evaluation of the measurement or testing of the same or similar items in two or more laboratories *under predetermined conditions*.

Proficiency testing (PT) is *evaluation* of the participants *according to predefined criteria* by interlaboratory comparison.

9.1 HRN EN ISO/IEC 17025

5.10 REPORTING THE RESULTS

The results of each testing or calibration conducted by the laboratory must be displayed accurately, clearly, unambiguously and objectively and in accordance with all special instructions in testing and calibration methods.

The results are given in the form:

- a) **test report** (test results, issued by the testing laboratory)
- b) **calibration certificates** (calibration results; issued by the calibration laboratory)
- c) **sampling reports** (sampling results; issued by the testing or calibration laboratory)

9.1 HRN EN ISO/IEC 17025

5.10 REPORTING THE RESULTS

Test reports or calibration certificates must include:

- a) **title** (e.g. "Test report" or "Calibration certificate")
- b) **name and address of the laboratory** and the place where the activities were carried out
- c) **unique label**
- d) **name and address of the customer;**
- e) **mark of the method used**
- f) **description and status of item** being tested or calibrated
- g) **date of receipt of item** being tested or calibrated where it is critical to validity and application of results and date(s) of testing or calibration

9.1 HRN EN ISO/IEC 17025

5.10 REPORTING THE RESULTS

- h) **designation of the sampling plan and the procedures** used by the laboratory or other bodies where they are important for validity or application of results;
- i) **testing or calibration results**, where appropriate with the measuring units;
- j) name, function and signature or equivalent label of the person approving the test report or calibration certificate;
- k) where relevant, **a statement that the results refer only to the items being tested or calibrated.**

9.1 HRN EN ISO/IEC 17025

5.10 REPORTING THE RESULTS

Opinions and Interpretations

When opinions and interpretations are involved, the laboratory must document the basis on which these opinions and interpretations are given.

In test report, opinions and interpretations must be clearly marked as such.

Opinions and interpretations do not accede to accreditation! (accreditation policy!)

9.1 HRN EN ISO/IEC 17025

5.10 REPORTING THE RESULTS

Electronic transmission of results

In case of transmission of test results or calibration results by telephone, telex, fax or other electronic or electromagnetic means, the requirements of this international standard must be fulfilled

Report and certificate form

The report must be formatted in such a way that it is adapted to any type of testing or calibrations carried out and to minimize the possibility of misunderstanding or misuse

9.1 HRN EN ISO/IEC 17025

ADDITIONAL REQUIREMENTS IN LABORATORY ACCREDITATION

Rules for accreditation

Although the basic document for laboratories accreditation is standard HRN EN ISO/IEC 17025, it is not the only one

Each accreditation body has rules that point to additional elements that refer to the accreditation process, as well as additional requirements for laboratories supplementing the needs for accreditation according to HRN EN ISO/IEC 17025

The accreditation rules also provide the obligations and responsibilities of accredited laboratories, and which laboratories are obliged to respect since from the moment of accreditation, accreditation body supervises the work of the accredited laboratory

9.1 HRN EN ISO/IEC 17025

ADDITIONAL REQUIREMENTS IN LABORATORY ACCREDITATION

Calling for accredited laboratory status

Calling for accredited laboratory status is regulated in two ways:

- a) Declarative sentence
- b) Accreditation symbol

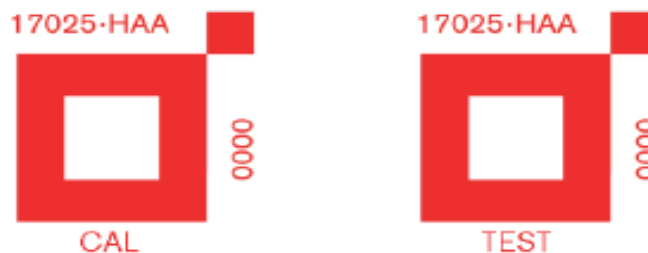
Declarative sentence:

The *organization/laboratory* is accredited (testing calibration) laboratory according to the standard HRN EN ISO/IEC 17025 by the Croatian Accreditation Agency in the field described in the attachment to the accreditation certificate No. XXXX.

9.1 HRN EN ISO/IEC 17025

ADDITIONAL REQUIREMENTS IN LABORATORY ACCREDITATION

Accreditation symbol



The testing laboratories have the number of accreditation certificate and abbreviation ,TEST' on accreditation symbol;

The calibration laboratories have the number of accreditation certificate and abbreviation ,CAL' on accreditation symbol;

It is important to emphasize:

If the laboratory does not invoke the status of accreditation in its report (by sentence or symbol), such a report **is not considered accredited!**

9.1 HRN EN ISO/IEC 17025

ADDITIONAL REQUIREMENTS IN LABORATORY ACCREDITATION

In the accreditation there are some special features that are further elaborated and to which in particular, places attention such as:

- interlaboratory comparisons
- traceability of results
- measurement uncertainty estimation
- presentation of the declaration of conformity

etc...

These additional details are elaborated in the rules and instructions of the accreditation body which the accredited laboratory must adhere to.

9.1 HRN EN ISO/IEC 17025

ADDITIONAL REQUIREMENTS IN LABORATORY ACCREDITATION

Interlaboratory comparisons

Interlaboratory comparisons serve for comparisons of laboratory work

The results are compared with each other in order to conclude the laboratory's abilities in the pursuit of their activities

These results are most often processed statistically.

Statistical indicators have been developed that concern the abilities of the laboratory (e.g. z-value, En-number)

9.1 HRN EN ISO/IEC 17025

ADDITIONAL REQUIREMENTS IN LABORATORY ACCREDITATION

Capability testing

When interlaboratory comparisons are used to control the quality of the testing results according to predefined criteria, then such an evaluation is called **capability testing!**

Capability testing is carried out by trained organizers.

The organizers are accredited according to HRN EN ISO/IEC 17043

Capability testing provided by accredited organizers serves laboratories as proof of external quality control as required by HRN EN ISO/IEC 17025

9.1 HRN EN ISO/IEC 17025

ADDITIONAL REQUIREMENTS IN LABORATORY ACCREDITATION

Capability testing

According to the *COMMISSION DIRECTIVE (EU) 2015/1480 of 28 August 2015 on the amendment of certain annexes to Directives 2004/107/EC and 2008/50/EC of the European Parliament and the Council on the establishment of rules for reference methods, data validation and location of sampling points for air quality assessment*, Annex I, Section C:

“National reference laboratory that organizes an interlaboratory comparison at national level should be accredited in accordance with the relevant harmonized standard for capability testing.”

9.1 HRN EN ISO/IEC 17025

SHORT OVERVIEW ON NEW EDITION ISO/IEC 17025:2017

Structure of standard

The standard is no longer divided into 2 main chapters, but the requirements are distributed into five chapters:

- a) general requirements (Chapter 4)
- b) structural requirements (Chapter 5)
- c) resource requirements (Chapter 6)
- d) process requirements (Chapter 7)
- e) requirements of the management system (Chapter 8).

9.1 HRN EN ISO/IEC 17025

SHORT OVERVIEW ON NEW EDITION ISO/IEC 17025:2017

Process approach

Chapter 7 – Process requirements – contains requirements from customer inquiries to report issuance:

- 7.1 Review of requests, tenders and contracts ("4.4")
- 7.2 Selection, verification and validation of methods ("5.4")
- 7.3 Sampling ("5.7")
- 7.4 Handling of test and calibration items ("5.8")
- 7.5 Technical records ("4.13.2")
- 7.6 Assessment of measurement uncertainty ("5.4.6")

9.1 HRN EN ISO/IEC 17025

SHORT OVERVIEW ON NEW EDITION ISO/IEC 17025:2017

- 7.7 Assuring the quality of results ("5.9")
- 7.8 Reporting the results ("5.10")
- 7.9 Complaints ("4.8")
- 7.10 Nonconforming testing ("4.9")
- 7.11 Data control and information management (new + "5.4.7")

9.1 HRN EN ISO/IEC 17025

SHORT OVERVIEW ON NEW EDITION ISO/IEC 17025:2017

Enhanced requirements

The following is asked from the laboratory :

- management of impartiality
- increased confidentiality requirements
- more detailed reports
- information management (*laboratory information management system*)
- increased measures for the results validity (no more quality assurance of results but assurance of results validity)
- risk management

9.1 HRN EN ISO/IEC 17025

SHORT OVERVIEW ON NEW EDITION ISO/IEC 17025:2017

Simplified requirements

What is facilitated in the new release of the standard is:

- application of management system (given the possibility of a management system build on ISO 9001)
- less documentation requirements
- no application of preventive actions is required, this is covered by risk management

9.1 HRN EN ISO/IEC 17025

SHORT OVERVIEW ON NEW EDITION ISO/IEC 17025:2017

Transfer to a new edition

At present laboratories are accredited according to HRN EN ISO/IEC 17025:2007 (ISO/IEC 17025:2005).

New standard was published on December 1, 2017, and from that moment initiates the THREE-YEAR PERIOD of transferring to a new edition.

Until 1.12.2020. all accredited laboratories will have to align with the new release standard, ISO/IEC 17025: 2017, or their accreditations cease to be valid.

9.2 REFERENCE AND EQUIVALENT METHODS

REFERENCE METHODS

Reference measurement methods for determining the concentration of SO₂, NO₂, CO, O₃, benzene, PM₁₀, PM_{2.5} and total gaseous mercury are defined in the Ordinance on the Air Quality Monitoring (OG 3/13), Annex 7 (Methods of Measurement), Part I,

Principle of

Pollutant

*measurement/analytical
method*

Method of measurement

SO₂

UV fluorescence

HRN EN 14212:2012 – Standard method for the measurement of the concentration of sulphur dioxide by ultraviolet fluorescence (EN 14212:2012)

NO/NO₂

Chemiluminescence

HRN EN 14211:2012 – Standard method for the measurement of the concentration of nitrogen dioxide and nitrogen monoxide by chemiluminescence (EN 14211:2012)

CO

IR spectroscopy

HRN EN 14626:2012 – Standard method for the measurement of the concentration of carbon monoxide by non-dispersive infrared spectroscopy (EN 14626:2011)

O₃

UV absorption

HRN EN 14625:2012 – Standard method for the measurement of the concentration of ozone by ultraviolet photometry (EN 14625:2012)

9.2 REFERENCE AND EQUIVALENT METHODS

REFERENCE METHODS

<i>Pollutant</i>	<i>Principle of method</i>	<i>Method of measurement</i>
Benzene	GC-PID or GC-FID	<p>HRN EN 14662-1:2007 – Standard method for measurement of benzene concentrations - Part 1: Pumped sampling followed by thermal desorption and gas chromatography (EN 14662-1:2005)</p> <p>HRN EN 14662-2:2007 – Part 2: Pumped sampling followed by solvent desorption and gas chromatography (EN 14662-1:2005),</p> <p>HRN EN 14662-3:2007 – Part 3: Automated pumped sampling with in situ gas chromatography (EN 14662-1:2005)</p>
PM₁₀	Gravimetric	<p>HRN EN 12341:2006 – Determination of the PM 10 fraction of suspended particulate matter - Reference method and field test procedure to demonstrate reference equivalence of measurement methods (EN 12341:1998)</p>
PM_{2.5}	Gravimetric	<p>HRN EN 14907:2006 – Standard gravimetric measurement method for the determination of the PM_{2,5} mass fraction of suspended particulate matter (EN 14907:2005)</p>
Total gaseous mercury	CV AAS or CV AFS	<p>HRN EN 15852:2010 – Standard method for the determination of total gaseous mercury</p>

9.2 REFERENCE AND EQUIVALENT METHODS

REFERENCE METHODS

According to the *DIRECTIVE 2008/50/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 21 May 2008 on air quality and clean air for Europe, ANNEX VI, Reference methods for estimation of the concentration of sulphur dioxide, nitrogen dioxide and nitrogen oxides, particulate matter (PM 10 and PM 2,5), lead, benzene, carbon monoxide and ground-level ozone :*

Reference method for the measurement of sulphur dioxide

Reference method for the measurement of sulphur dioxide is described in EN 14212:2012, Ambient air – Standard method for the measurement of the concentration of sulphur dioxide by ultraviolet fluorescence.

9.2 REFERENCE AND EQUIVALENT METHODS

REFERENCE METHODS

Reference method for the measurement of nitrogen dioxide and nitrogen oxides

Reference method for the measurement of nitrogen dioxide and nitrogen oxides is described in EN 14211:2012, Ambient air – Standard method for the measurement of the concentration of nitrogen dioxide and nitrogen monoxide by chemiluminescence.

Reference method for lead sampling and measurement

Reference method for lead sampling and measurement is described in Part A (4) of this Appendix. Reference method for lead measurement is described in EN 14902:2005 „Standard method for the measurement of Pb, Cd, As and Ni in the PM10 fraction of suspended particulate matter”.

9.2 REFERENCE AND EQUIVALENT METHODS

REFERENCE METHODS

Reference method for sampling and measurement of PM 10

Reference method for sampling and measurement of PM10 is described in EN 12341:2014, Ambient air – Standard gravimetric measurement method for the determination of the PM10 or PM2,5 mass concentration of suspended particulate matter’.

Reference method for sampling and measurement of PM 2,5

Reference method for sampling and measurement of PM2,5 is described in EN 12341:2014, Ambient air – Standard gravimetric measurement method for the determination of the PM10 or PM2,5 mass concentration of suspended particulate matter’.

9.2 REFERENCE AND EQUIVALENT METHODS

REFERENCE METHODS

Reference method for sampling and measurement of benzene

Reference method for sampling and measurement of benzene is described in EN 14662:2005, parts 1, 2 and 3 „Ambient air quality - Standard method for measurement of benzene concentrations”.

Reference method for measurement of carbon monoxide

Reference method for measurement of carbon monoxide is described in EN 14626:2012, Ambient air – Standard method for the measurement of the concentration of carbon monoxide by non-dispersive infrared spectroscopy’.

9.2 REFERENCE AND EQUIVALENT METHODS

REFERENCE METHODS

Reference method for measurement of ground-level ozone

Reference method for measurement of ground-level ozone is described in EN normi 14625:2012, Ambient air – Standard method for the measurement of the concentration of ozone by ultraviolet photometry’.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

Ambient air – Standard method for the measurement of the concentration of sulphur dioxide by ultraviolet fluorescence

This standard has a correction:

HRN EN 14212:2012/Cor.1:2014 (EN 14212:2012/AC:2014)

SO₂

This European standard establishes a method for continuous measurement for determination of concentrations of sulphur dioxide in the ambient air on the measurement principle of **ultraviolet fluorescence**.

The standard describes the performance characteristics and sets the minimum criteria required for selecting the appropriate analyzer.

It also includes an assessment of the suitability of the analyzer for use on the monitoring station to meet the data quality requirements in accordance with the Directive 2008/50/EC.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

MEASUREMENT AREA

0 - 1000 $\mu\text{g}/\text{m}^3$
(0 - 376 nmol/mol)

MEASUREMENT PRINCIPLE

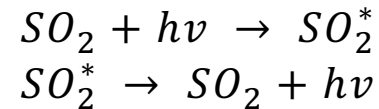
UV FLUORESCENCE



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

The measurement principle is based on the fluorescence radiation of the SO₂ molecules when they are exposed to ultraviolet (UV) radiation. UV fluorescence occurs when the molecule SO₂ due to exposure to UV radiation goes from normal to excited state and then returns to normal state with radiation emission.



The intensity of the emitted radiation is proportional to the number of SO₂ molecules in a given volume, i.e. concentration of SO₂. Dependency of SO₂ concentration on the intensity of radiation can be described by the following equation :

$$F = k \cdot c_{SO_2}$$

where:

F – intensity of radiation

k – coefficient of proportionality

c_{SO_2} – SO₂ concentration

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

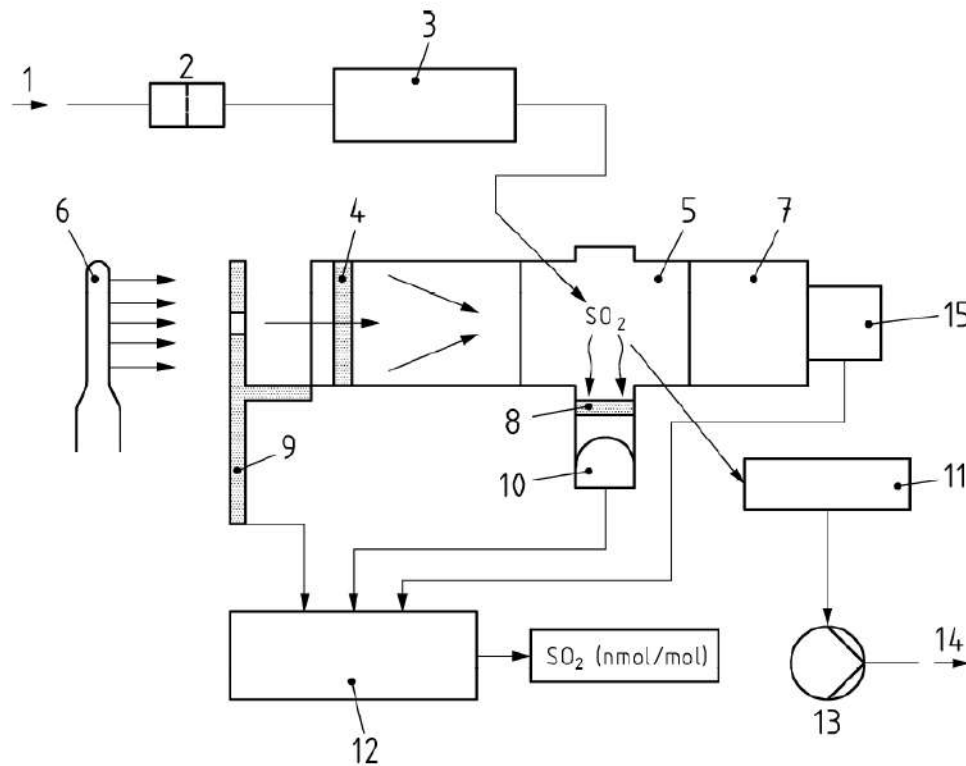
Before entering the reaction chamber, the air is filtered and passed through a hydrocarbon scrubber that would otherwise cause interference. Then the air enters the reaction chamber where it is, under standardized conditions, exposed to UV rays in the wavelength area between 200 and 220 nm. When returning from excited to the normal state, the SO₂ molecules emit radiation in the range of wavelengths between 240 and 420 nm. This emission is, by using selective optical filters, transformed into an electric signal by a photoconductor or a photodiode. The electrical signal is measured and its intensity is proportional to the number of molecules, i.e. at standardized conditions of SO₂ concentration which entered the reaction.

Concentrations of SO₂ are measured directly into volume/volume units (ppb) since the absorption in the IR spectrum is proportional to the concentration of SO₂ in volume/volume units. After obtaining concentration in ppb values, the result is converted to µg/m³ using standard conversion factors for a temperature of 20 °C and a pressure of 101.3 kPa whereat:

$$1 \text{ ppb (nmol/mol) SO}_2 = 2,66 \text{ µg/m}^3 \text{ SO}_2$$

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012



- 1 sample
- 2 sampling inlet filter
- 3 selective traps for interfering agents
- 4 optical filter
- 5 reaction chamber
- 6 UV lamp
- 7 optical trap
- 8 optical outlet filter
- 9 modulator
- 10 photomultiplier tube
- 11 compensation pressure flow rate
- 12 synchronous electronic amplification
- 13 pump
- 14 exhaust
- 15 reference detector

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

EQUIPMENT

HORIBA – APSA 370



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

EQUIPMENT

THERMO SCIENTIFIC

– MODEL 43i



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

EQUIPMENT

TELEDYNE

– API T100



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

EQUIPMENT

ENVIRONNEMENT

– AF 22



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

Ambient air – Standard method for the measurement of the concentration of sulphur dioxide by ultraviolet fluorescence

This standard has a correction:

HRN EN 14212:2012/Cor.1:2014 (EN 14212:2012/AC:2014)



This European standard establishes a method for continuous measurement for determination of concentrations of sulphur dioxide in the ambient air on the measurement principle of **ultraviolet fluorescence**.

EQUIVALENT METHOD

Hydrogen sulphide is converted into **sulphur dioxide** which determination is based on the principle of UV fluorescence.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012 (H₂S)

When entering the analyzer, the air is filtered and passes the scrubber of sulphur dioxide that completely adsorbs SO₂ from the sampled air, which is necessary to avoid SO₂ measurement which would then be displayed as H₂S. Then the air passes through a converter in which hydrogen sulphide is oxidized to sulphur dioxide in moles per mol. After this procedure, further measurement is identical to measurement of SO₂.

Before entering the reaction chamber, the air is filtered and passed through a hydrocarbon scrubber that would otherwise cause interference. Then the air enters the reaction chamber where it is, under standardized conditions, exposed to UV rays in the wavelength area between 200 and 220 nm.

When returning from excited to the normal state, the SO₂ molecules emit radiation in the range of wavelengths between 240 and 420 nm. This emission is, by using selective optical filters, transformed into an electric signal by a photoconductor or a photodiode. The electrical signal is measured and its intensity is proportional to the number of molecules, i.e. at standardized conditions of SO₂ concentration which entered the reaction.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012 (H₂S)

Concentrations of SO₂ are measured directly into volume/volume units (ppb) since the absorption in the IR spectrum is proportional to the concentration of SO₂ in volume/volume units. After obtaining concentration in ppb values, the result is converted to µg/m³ using standard conversion factors for a temperature of 20 °C and a pressure of 101.3 kPa whereat :

$$1 \text{ ppb (nmol/mol) SO}_2 \text{ (H}_2\text{S)} = 1,41 \text{ µg/m}^3 \text{ H}_2\text{S}$$

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

EQUIPMENT

HORIBA

– APSA 370 + CU



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

EQUIPMENT

THERMO SCIENTIFIC

– MODEL 450i



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

EQUIPMENT

TELEDYNE

– API T101E



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14212:2012

EQUIPMENT

ENVIRONNEMENT

– AF 22



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14211:2012

Ambient air - Standard method for the measurement of the concentration of nitrogen dioxide and nitrogen monoxide by chemiluminescence

NO/NO_x

This European standard establishes a method for continuous measurement for determination of concentration of nitrogen dioxide and nitrogen monoxide in the ambient air at the **chemiluminescence** principle.

The standard describes the performance characteristics and sets the minimum criteria required for selecting the appropriate analyzer.

It also includes an assessment of the suitability of the analyzer for use on the meter station to meet the data quality requirements in accordance with the Directive 2008/50/EC.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14211:2012

MEASUREMENT AREA

NO₂	0 - 500 $\mu\text{g}/\text{m}^3$ (0 - 261 nmol/mol)
NO	0 - 1200 $\mu\text{g}/\text{m}^3$ (0 - 962 nmol/mol)

MEASUREMENT PRINCIPLE

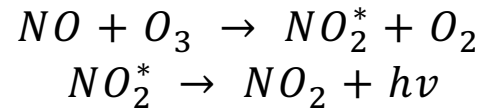
CHEMILUMINESCENCE



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14211:2012

The measurement principle is based on the chemiluminescence of the NO₂ molecules when switching from excited to normal state. Excitation occurs when NO in the presence of O₃ oxidizes to NO₂.



The radiation intensity (chemiluminescence) is proportional to the number of NO molecules which are reacted in a given volume or NO concentration. Radiation intensity is measured using a photodiode.

After entering the analyzer, the air is divided into two of which one leads air directly into reaction chamber and thereby the NO concentration is measured in the air sample. The second leads the air through the catalytic converter where all NO₂ from air is reduced to NO and then introduced into the reaction chamber where the processes described in equations above are conducted. In this way, the concentration of NO_x is measured. NO₂ is calculated from the difference in concentrations of NO_x and NO.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14211:2012

NO₂ concentrations are measured directly in volume/volume units (ppb) if the calibration is carried out with a 'vol/vol' ethalon. After obtaining concentration in ppb the result is converted to µg/m³ using standard conversion factors for temperature 20 ° C and a pressure of 101.3 kPa.

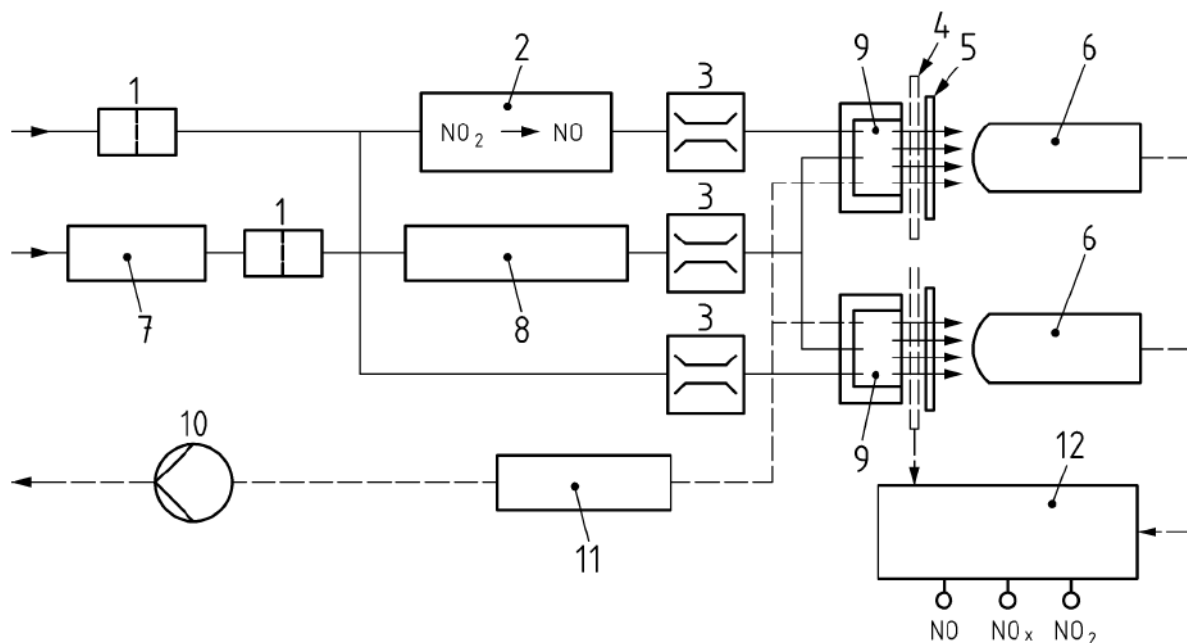
$$1 \text{ ppb (nmol/mol) NO}_2 = 1,91 \text{ µg/m}^3 \text{ NO}_2$$

$$1 \text{ ppb (nmol/mol) NO} = 1,25 \text{ µg/m}^3 \text{ NO}$$

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14211:2012

- 1 particle filter
- 2 converter
- 3 flow rate controller
- 4 chopper
- 5 optical filter
- 6 photo multiplier tube
- 7 drier
- 8 ozone generator
- 9 reaction chamber
- 10 sampling pump
- 11 ozone filter
- 12 synchroniser output



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14211:2012

EQUIPMENT

HORIBA – APNA 370



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14211:2012

EQUIPMENT

THERMO SCIENTIFIC

– MODEL 42i



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14211:2012

EQUIPMENT

TELEDYNE
– API T200



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14211:2012

EQUIPMENT

ENVIRONNEMENT^T

– ACM 32



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14625:2012

Ambient air – Standard method for the measurement of the concentration of ozone by ultraviolet photometry



This European Standard establishes a method for continuous measurement for determination of concentrations of ozone in the ambient air by **ultraviolet photometry**.

The standard describes the performance characteristics and sets the minimum criteria required for selecting the appropriate analyzer.

It also includes an assessment of the suitability of the analyzer for use on the meter station to meet the data quality requirements in accordance with the Directive 2008/50/EC.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14625:2012

MEASUREMENT AREA

0 - 500 $\mu\text{g}/\text{m}^3$
(0 – 250 nmol/mol)

MEASUREMENT PRINCIPLE

UV PHOTOMETRY



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14625:2012

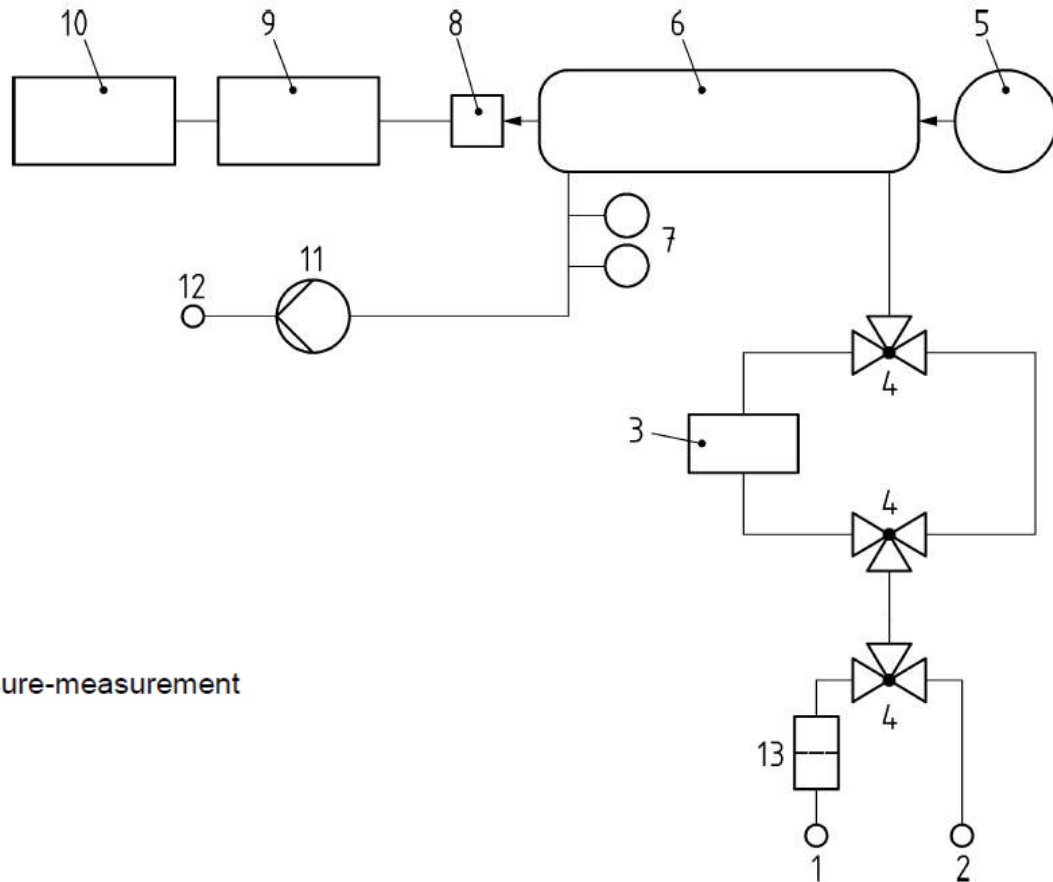
The method is based on the ability of ozone to absorb UV radiation.

Sampled air is filtered at the inlet into the analyzer, and with constant and known flow flows to the reaction cell in which the conditions of constant pressure and temperature are maintained. In that conditions the air is exposed to constant radiation of low pressure mercury lamps with peak values of 253.7 nm. Sensitive photodiodes or photomultiplier detectors measure the intensity of the radiation which passed through the reaction cell. Ozone in the air sample absorbs a certain amount of radiation that is at constant conditions in proportion to the concentration of ozone in the sample.

There are two basic ways of quantifying this absorption. One is to get through the same reaction cell the alternating air and ozone-free air sample, and the other is that there are two identical cells, and through one there is a continuous passage of air sample, and through the other the air without ozone. In both modes, ozone-free air is obtained by catalytically conducting air ozone convector. The difference in radiation intensity measured in the air with or without ozone, represents the absorption of radiation. Sampling of air containing a known concentration of ozone the analyzer is tuned, and it is possible to calculate the absorption from the calibration direction ozone concentration.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14625:2012



- 1 sample in
- 2 reference in
- 3 ozone scrubber
- 4 solenoid valve
- 5 source 254 nm
- 6 absorption cell
- 7 temperature- and pressure-measurement
- 8 detector
- 9 digital electronics
- 10 display
- 11 pump
- 12 exhaust
- 13 particle filter

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14625:2012

EQUIPMENT

HORIBA – APOA 370



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14625:2012

EQUIPMENT

THERMO SCIENTIFIC

– MODEL 49i



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14625:2012

EQUIPMENT

TELEDYNE

– API T400



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14625:2012

EQUIPMENT

ENVIRONNEMENT

– O342



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

Ambient air – Standard method for the measurement of the concentration of carbon monoxide by non-dispersive infrared spectroscopy

CO

This European standard establishes a method for continuous measurement for determination of concentrations of carbon monoxide in the ambient air on the measurement principle of **non-dispersive infrared spectroscopy**.

The standard describes the performance characteristics and sets the minimum criteria required for selecting the appropriate analyzer.

It also includes an assessment of the suitability of the analyzer for use on the meter station to meet the data quality requirements in accordance with the Directive 2008/50/EC.

9.2 REFERENCE AND EQUIVALENT METHODS

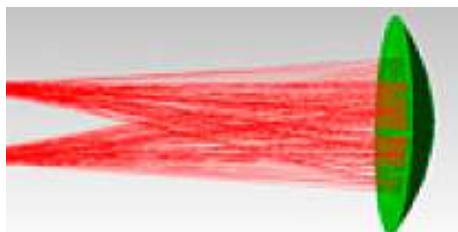
HRN EN 14626:2012

MEASUREMENT AREA

0 - 100 mg/m³
(0 – 86 µmol/mol)

MEASUREMENT PRINCIPLE

NON-DISPERSIVE INFRARED SPECTROSCOPY



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

The measurement principle is based on non-dispersive infrared spectroscopy since CO molecules have the ability to absorb infrared radiation. Spectroscopic analysis is based on applying the Beer - Lambert law. CO has the highest absorption at 4.67 μm and the absorption degree of IR radiation depends on the length of the absorption chamber, the absorption coefficient and CO concentration in the sample introduced into the absorption chamber according to the following equation:

$$T = I/I_0 = e^{-axc}$$

where:

T - radiation transmission through the sample to detector

I - radiation intensity after absorption in the CO sample

I_0 - radiation intensity after passing through the sample without CO

a - molar absorption coefficient of CO

x - length of the absorption chamber

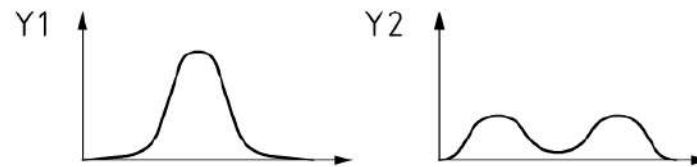
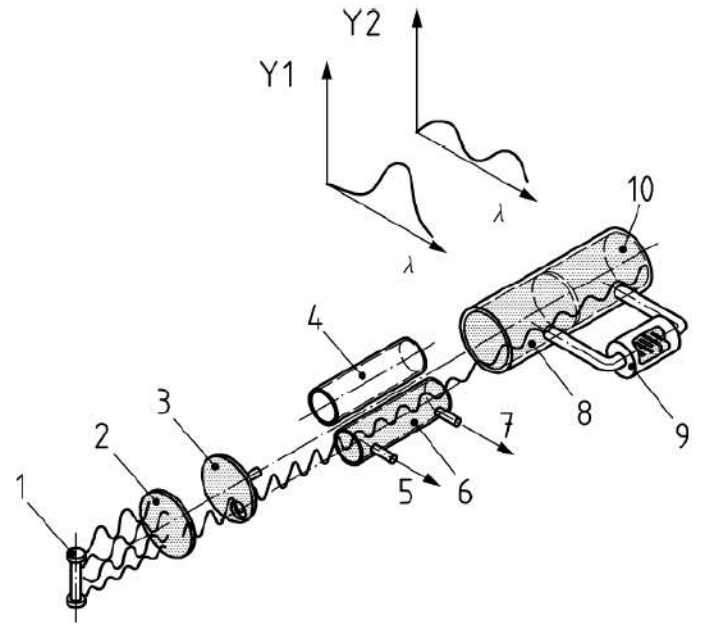
c - concentration of CO

Interferences from other gases that absorb at close wavelengths are removed using a gas filter called the correlation wheel.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

- 1 IR lamp
- 2 light filter
- 3 chopper
- 4 reference cell
- 5 gas out
- 6 sample cell
- 7 gas out
- 8 first chamber
- 9 micro flow sensor
- 10 second chamber
- Y1 light absorption in first chamber
- Y2 light absorption in second chamber
- λ wavelength



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

EQUIPMENT

HORIBA – APMA 370



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

EQUIPMENT

THERMO SCIENTIFIC

– MODEL 48i



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

EQUIPMENT

TELEDYNE

– API T300



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

EQUIPMENT

ENVIRONNEMENT

– CO 12



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

Ambient air – Standard method for measurement of benzene concentrations – Part 3: Automated pumped sampling with in situ gas chromatography



This European standard establishes a method for continuous measurement for determination of concentrations of benzene in the ambient air based on automated sampling by **gas chromatography**.

The standard describes the performance characteristics and sets the minimum criteria required for selecting the appropriate analyzer.

It also includes an assessment of the suitability of the analyzer for use on the meter station to meet the data quality requirements in accordance with the Directive 2008/50/EC.

9.2 REFERENCE AND EQUIVALENT METHODS

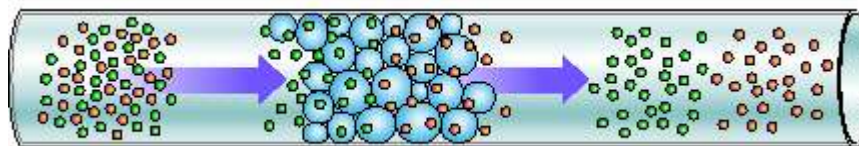
HRN EN 14626:2012

MEASUREMENT AREA

0 - 50 $\mu\text{g}/\text{m}^3$
(0 – 15,4 nmol/mol)

MEASUREMENT PRINCIPLE

GAS CHROMATOGRAPHY



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

The metering principle for benzene measurement involves sampling by diluting with heat desorption and simultaneous analysis by gas chromatography.

The method according to HRN EN 14662-3 is based on classical gas chromatography and detection using a **flame (FID) or photoionisation (PID)** detector. Also, it is allowed to use and any other suitable detector.

Measured air volume is pumped by using a pump on the adsorption pre - column which binds the benzene from the sample, and the air without benzene exits the analyzer. Thereafter by heating the benzene vapor free from the adsorbent and by using inert gas (most often nitrogen of high purity) are transferred to a chromatographic column where separation of benzene occurs from other easily volatile hydrocarbons (xylene, toluene). Benzene injected with inert gas after passing through the column at exactly the same time as retention time (for the same chemical compound, same system and same conditions, retention time is always the same) enters the detector that registers the passage of benzene to the proportional mass of benzene.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

By comparing this signal with zero air signal (benzene-free air) and signal obtained by passing the air with a known benzene concentration can be calculated the concentration of benzene in the taken sample using the following equation:

$$c_m = \frac{m_{sam}}{V_{sam}}$$

where:

c_m = concentration of benzene in the air sample;

m_{sam} = mass of benzene in the sample;

V_{sam} = sample volume.

Subsequently, the concentration is converted to $\mu\text{g}/\text{m}^3$ using standard conversion factors for a temperature of 20°C and an atmospheric pressure of 101.3 kPa.

9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

EQUIPMENT

CHROMATOTEC

– GC 866



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

EQUIPMENT

SYNTECH
– GC 955



9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

EQUIPMENT

AMA INSTRUMENTS

– GC 5000



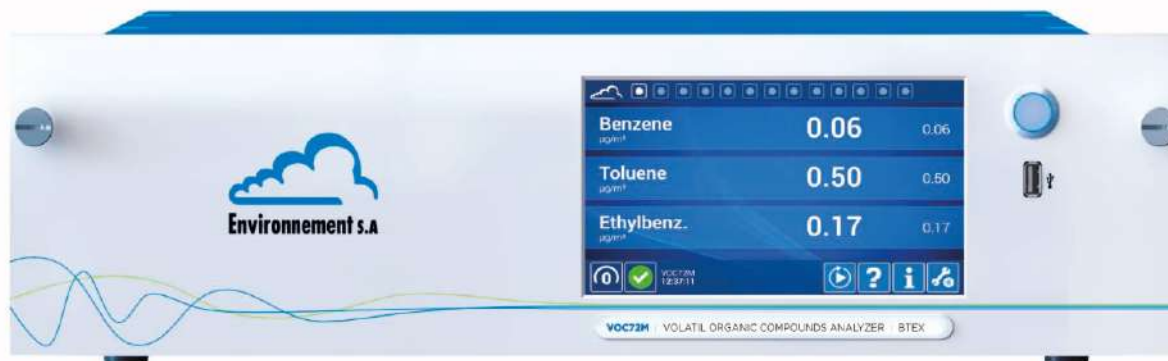
9.2 REFERENCE AND EQUIVALENT METHODS

HRN EN 14626:2012

EQUIPMENT

ENVIRONMENT

– VOC 72



9.2 REFERENCE AND EQUIVALENT METHODS

MEASUREMENT SYSTEM

The measurement system consists of:

- Gas analyzers
- Null and Range Response Check System
- Sampling system
- Thermostated isothermal stations
- Data transfer and data collection systems



9.2 REFERENCE AND EQUIVALENT METHODS

MEASUREMENT SYSTEM

GAS ANALYZERS

Gas analyzers serve to measure concentrations of individual gases.

Before they are placed in the isothermal shelter (measuring station) they must be calibrated as well check the operating characteristics.

After setting up the station, the response to the zero position and position of the range with the analyzer response tester on the zero and the range at the station are checked.

9.2 REFERENCE AND EQUIVALENT METHODS

MEASUREMENT SYSTEM

ZERO AND SPAN RESPONSE SYSTEM

The system consists of the following components:

- Dilution unit
- Certified reference gas (gas mixture)
- Zero gas generator

The system enables automatic checking of the response to zero and span for the purpose of measuring quality control as well as long-term monitoring of analyzer deviation.

Mixing of zero gas and certified reference gas a desired gas concentration is obtained by which the analyzer span is checked.

The dilution unit is supplied with zero gas from the gas generator and reference gas from a certified gas mixture.

9.2 REFERENCE AND EQUIVALENT METHODS

MEASUREMENT SYSTEM

ZERO AND SPAN RESPONSE SYSTEM

The dilution unit is supplied with zero gas from the *gas generator* and reference gas from a *certified gas mixture*.



9.2 REFERENCE AND EQUIVALENT METHODS

MEASUREMENT SYSTEM

SAMPLING SYSTEM

The sampling system consists of steel (inox) head and sample tube designed so that they prevent getting wet, both in the system and in the station interior. Through the tube of the sampler, the tube of silicone glass is passed which binds to the sample separator in the station which is also of the boron of silicate glass.

At the end of the system there is a sampler fan that ensures a constant sample flow with negligible difference in the pressures between the station and manifold. The system is constructed so that the sampled air comes into contact with only silicate boron glass and to ensure constant flow and air pressure in the manifold from which the instruments take the sample.

9.2 REFERENCE AND EQUIVALENT METHODS

MEASUREMENT SYSTEM

SAMPLING SYSTEM



9.2 REFERENCE AND EQUIVALENT METHODS

MEASUREMENT SYSTEM

THERMOSTATIC ISOTHERMAL STATION

Isothermal stations are designed to provide a constant temperature of $22 \text{ }^{\circ}\text{C} \pm 4 \text{ }^{\circ}\text{C}$ and constant power supply of a stable voltage of 220V. Gas analyzers are located inside.



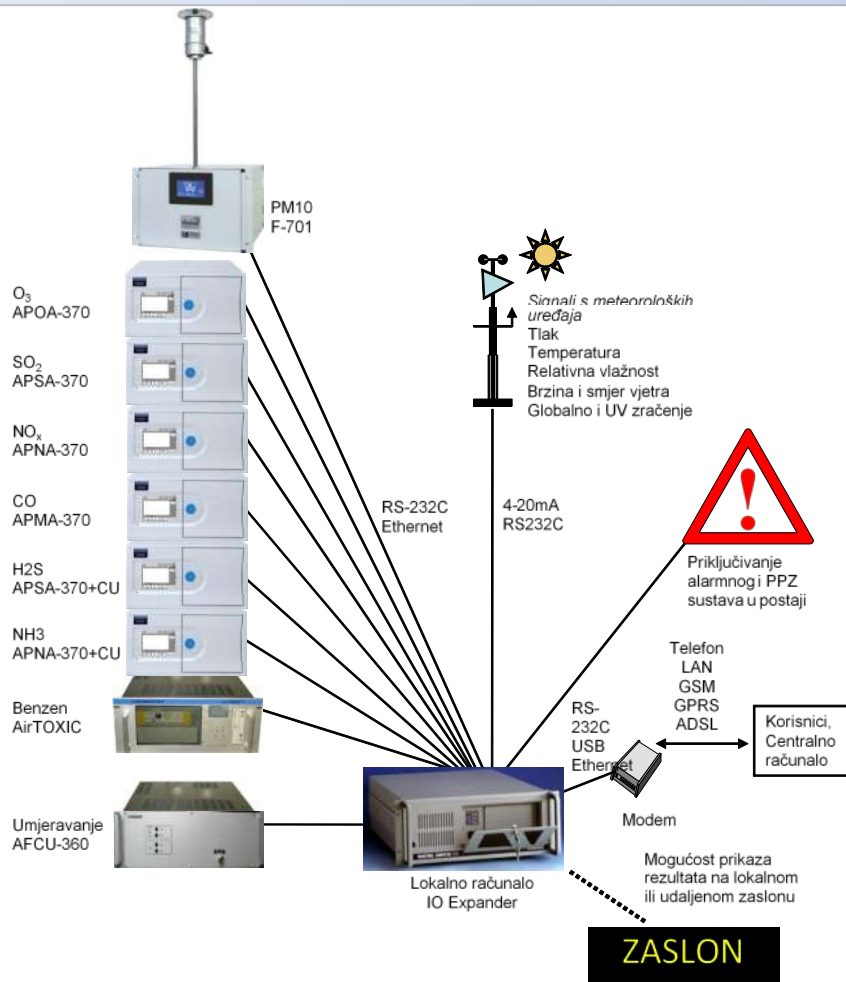
9.2 REFERENCE AND EQUIVALENT METHODS

MEASUREMENT SYSTEM

DATA TRANSMISSION AND COLLECTION SYSTEM

The data transfer and data collection system enables correct receiving, storing, processing and transferring of data from instruments to the datalogger (or computer) and from datalogger to a supervisory computer in the Testing Laboratory.

9.2 REFERENCE AND EQUIVALENT METHODS



9.3 EXAMPLES OF USING HRN EN STANDARDS

SUITABILITY ASSESSMENT

A suitability assessment is performed to define the metering point, the conditions that exist at the metering point and how these conditions affect the operation of the gas analyzer.

The assessment is carried out for each individual measurement. Criteria to be assessed are defined in the standards :

HRN EN 14211:2012 – for NO/Nox measurement

HRN EN 14212:2012 – for SO₂ and H₂S measurement

HRN EN 14625:2012 – for O₃ measurement

HRN EN 14626:2012 – for CO measurement

HRN EN 14662-3:2012 – for C₆H₆ measurement

9.3 EXAMPLES OF USING HRN EN STANDARDS

SUITABILITY ASSESSMENT

Example of suitability assessment for CO measurement according to HRN EN 14626:2012

Variations in sample pressure:

- First, check the manifold*
- impact of the pressure drop due to the sampler pump and the efficiency of sampling*
- if the checks are satisfactory and the sampling system is "standard,, it is estimated that the maximum pressure variation throughout the year does not exceed that of the type approvals, so the component of the type approval is taken into account in the calculation of uncertainty*

9.3 EXAMPLES OF USING HRN EN STANDARDS

SUITABILITY ASSESSMENT

Variations in sample temperature:

- if the system for sampling, shelter and air conditioning are "standard,, it is estimated that the maximum variation of sample temperature over the year does not exceed the one from the type approval and then the component of the type approval is taken into account in the calculation of uncertainty*
- temperature of sampled air is monitored throughout the year and after the first year, the results are corrected*
- if the system for sampling, shelter and air conditioning are "standard,, it is estimated that the maximum variation of station temperature over the year does not exceed the one from the type approval and then the component of the type approval is taken into account in the calculation of uncertainty*
- the station temperature is monitored throughout the year and after the first year, the results are corrected*

9.3 EXAMPLES OF USING HRN EN STANDARDS SUITABILITY ASSESSMENT

Variations in the voltage of the power grid at the station :

- if the instrument is powered over the UPS, it is estimated that the maximum voltage variation in the station throughout the year does not exceed that of the type approval and so the component of type approval is taken in the calculation of the measurement uncertainty*
- the voltage in station is monitored throughout the year and after the first year, the results are corrected*

Range of H₂O concentration in the sample:

- if the system for sampling, shelter and air conditioning is "standard,, it is estimated that the maximum variation of moisture in the sample throughout the year does not exceed that from the type approval, so the calculation of uncertainty takes the component from type approval*

9.3 EXAMPLES OF USING HRN EN STANDARDS

SUITABILITY ASSESSMENT

Range of CO₂ concentration in the sample:

- if it is a typical urban traffic station it is estimated that the maximum concentrations of CO₂ do not exceed 500 $\mu\text{mol/mol}$ so the calculation of uncertainty takes the component from type approval

Range of NO concentration in the sample:

- if it is a typical urban traffic station it is estimated that the maximum concentrations of NO do not exceed 1 $\mu\text{mol/mol}$ so the calculation of uncertainty takes the component from type approval

9.3 EXAMPLES OF USING HRN EN STANDARDS

SUITABILITY ASSESSMENT

Range of N₂O concentration in the sample:

- if it is a typical urban traffic station it is estimated that the maximum concentrations of N₂O do not exceed 50 nmol/mol so the calculation of uncertainty takes the component from type approval

Extended measurement uncertainty of calibration gas:

- it is calculated and included in the extended measurement uncertainty

Frequency of calibration and checking of analyzer response:

- it is calculated, and the response frequency is taken into account

9.3 EXAMPLES OF USING HRN EN STANDARDS

INITIAL INSTALLATION

Initial procedures provide and prove that the type approved analyzer is in satisfactory condition, that it is mounted in the correct manner and in conditions that provide measurements that will satisfy the quality of the data provided by the regulations of the Republic of Croatia and EU.

Records of initial installation include:

1. reports on the suitability of individual analyzers
2. certificates of calibration of the analyzer
3. reports on test performance of the analyzer
4. analyzer installation logs
5. reports on the data receiving and collection system

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

Ensuring the quality of the test results includes the following elements:

- preventive maintenance
- quality control at the station
- calibration
- performance check
- corrective actions
- external quality control.

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

PREVENTIVE MAINTENANCE

Procedures for preventive maintenance of the analyzer include: checking of technical correctness, regular service inspection of the measuring station, regular maintenance of the system for sampling, regular maintenance of the air conditioning and fire alarm system and regular annual service.

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

PREVENTIVE MAINTENANCE

Procedure	Frequency of performance
Verification of technical validity of instruments	three times a week
Check the analyzer response on the measuring station	every 25 hours
Regular service inspection of the station	every 15 days
Regular maintenance of the sampling system	every 6 months or as required
Regular maintenance of air conditioning and fire alarm system	every 6 months or as required
Regular annual service of the analyzer	once a year

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

QUALITY CONTROL AT THE STATION

The quality control procedures at the station are used to control everyday operation of the measuring system. In this way, the functionality of the instruments is regularly maintained and it enables a timely reaction to possible irregularities in the instruments operation that might otherwise remain unnoticed for a long time.

If checking of the response to zero gas and gas range shows that the eligibility criteria are not satisfied, then the metrics are validated again for the period of the time when the respondents were last satisfied with the criteria.

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

CALIBRATION

Calibration is carried out in a calibration laboratory that can provide the measurement traceability according to HRN EN ISO/IEC 17025.

It is conducted:

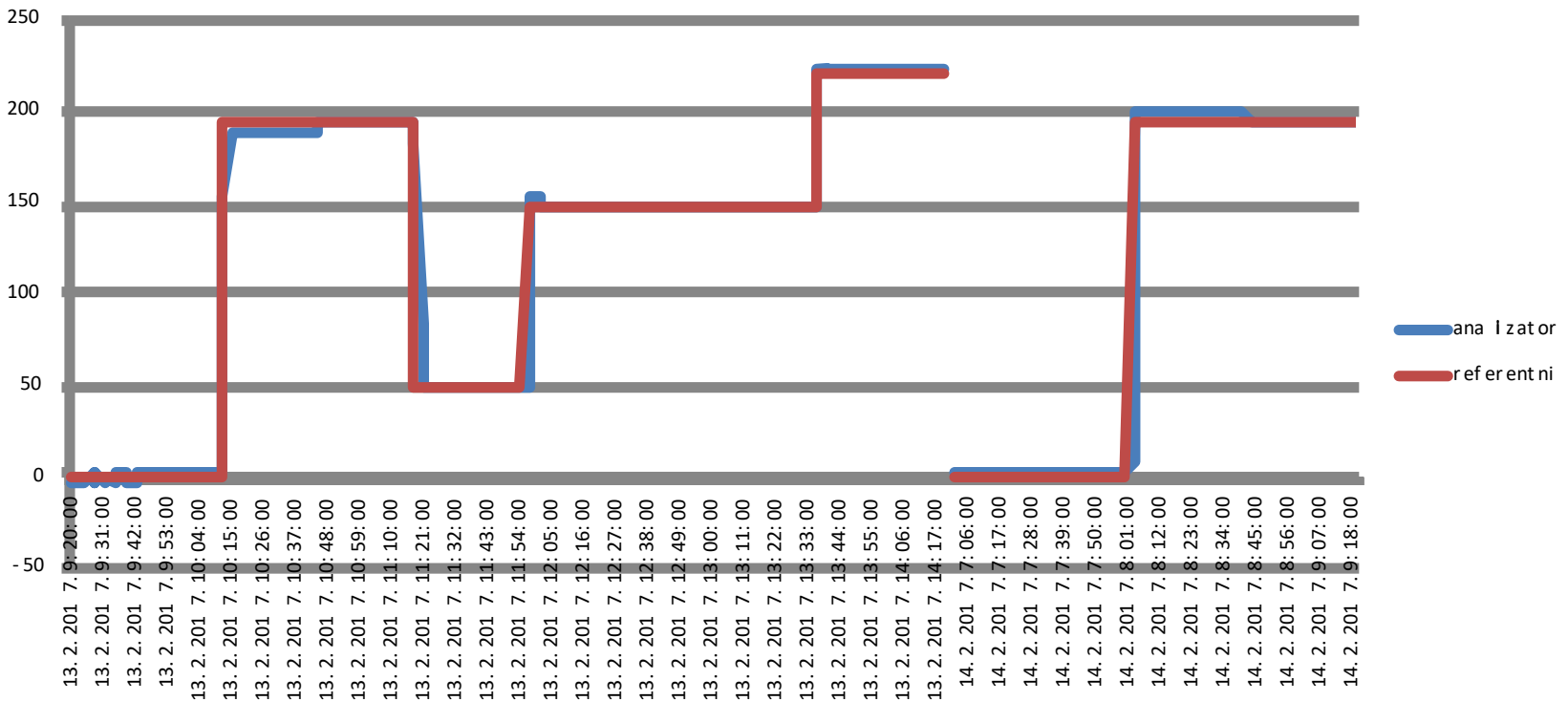
- after each annual service
- in the case of passing the acceptance limits to check the zero position or position range
- after major servicing procedures on the analyzer.

The calibration result is a Certificate or Calibration Certificate, and it is kept in the analyzer folder.

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

CALIBRATION



9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

PERFORMANCE TEST

In addition to the calibration, a test of the performance of the analyzer is carried out which includes:

- lack of fit
- check the standard deviation of three-point and zero repeatability and
- short-time check to zero and range
- checking the efficiency of the converter (NO/NO_x analyzers).

The analyzer must meet the values within the admissibility limits prescribed for each individual analyzer prescribed by the corresponding HRN EN standard.

The performance check is carried out once a year (usually during calibration).

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

Performance test - SO₂ analyzers

Broj prema Tablici 6 ili 1 norme HRN EN 14212 Number according to Table 6 or 1 of the EN 14212	Oznaka iz norme HRN EN 14212 Symbol according to EN 14212	Karakteristika Characteristic	Rezultat provjere (nmol/mol ili %) Result of the check (nmol/mol or %)	Granice prihvatljivosti Acceptance limits	Sukladnost Compliance
Tablica 6/2a Table 6/2a	Sr,z	ponovljivost na nultom plinu repeatability at zero	0,09	< 1,0 nmol/mol	Zadovoljava Complies
Tablica 6/2b Table 6/2b	Sr	ponovljivost na rasponu (80% mjernog područja) repeatability at span (80% of measurement range)	0,16%	< 1,5%	Zadovoljava Complies
Tablica 6/5b Table 6/5b	r _{max}	nelinearnost za točke različite od 0 lack of fit at concentrations higher than zero	0,78%	< 4,0%	Zadovoljava Complies
Tablica 6/5a Table 6/5a	x _{l,z}	nelinearnost na 0 lack of fit at zero	-0,73	< 5,0 nmol/mol	Zadovoljava Complies
Tablica 1/13 Table 1/13	Ds,z	kratkotrajni odmak na nultom plinu short-term drift at zero level	-0,40	< 2,0 nmol/mol kroz 12 sati / over 12 hours	Zadovoljava Complies
Tablica 1/14 Table 1/14	Ds,s	kratkotrajni odmak na rasponu short-term drift at span level	1,65	< 6,0 nmol/mol kroz 12 sati / over 12 hours	Zadovoljava Complies

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

Performance test - CO analyzers

Broj prema Tablici 6 ili 1 norme HRN EN 14626 Number according to Table 6 or 1 of the EN 14626	Oznaka iz norme HRN EN 14626 Symbol according to EN 14626	Karakteristika Characteristic	Rezultat provjere (μmol/mol ili %) Result of the check (μmol/mol or %)	Granice prihvatljivosti Acceptance limits	Sukladnost Compliance
Tablica 6/2a Table 6/2a	Sr,z	ponovljivost na nultom plinu repeatability at zero	0,00	< 0,5 μmol/mol	Zadovoljava Complies
Tablica 6/2b Table 6/2b	Sr	ponovljivost na rasponu (80% mjernog područja) repeatability at span (80% of measurement range)	0,02%	< 3,0 %	Zadovoljava Complies
Tablica 6/5b Table 6/5b	r _{max}	nelinearnost za točke različite od 0 lack of fit at concentrations higher than zero	0,36%	< 4,0%	Zadovoljava Complies
Tablica 6/5a Table 6/5a	x _{l,z}	nelinearnost na 0 lack of fit at zero	0,12	< 0,5 μmol/mol	Zadovoljava Complies
Tablica 1/13 Table 1/13	Ds,z	kratkotrajni odmak na nultom plinu short-term drift at zero level	0,01	< 0,1 μmol/mol kroz 12 sati / over 12 hours	Zadovoljava Complies
Tablica 1/14 Table 1/14	Ds,s	kratkotrajni odmak na rasponu short-term drift at span level	0,07	< 0,6 μmol/mol kroz 12 sati / over 12 hours	Zadovoljava Complies

9.3 EXAMPLES OF USING HRN EN STANDARDS RESULTS QUALITY ASSURANCE

Performance test - NO/NO_x analyzers

Broj prema Tablici 6 ili 1 norme HRN EN 14211 Number according to Table 6 or 1 of the EN 14211	Oznaka iz norme HRN EN 14211 Symbol according to EN 14211	Karakteristika Characteristic	Rezultat provjere (nmol/mol ili %) Result of the check (nmol/mol or %)	Granice prihvatljivosti Acceptance limits	Sukladnost Compliance
Tablica 6/2a Table 6/2a	Sr,z	ponovljivost na nultom plinu repeatability at zero	0,76	< 1,0 nmol/mol	Zadovoljava Complies
Tablica 6/2b Table 6/2b	Sr	ponovljivost na rasponu (80% mjernog područja) repeatability at span (80% of measurement range)	0,04%	< 0,75%	Zadovoljava Complies
Tablica 6/5b Table 6/5b	r _{max}	nelinearnost za točke različite od 0 lack of fit at concentrations higher than zero	0,97%	< 4,0%	Zadovoljava Complies
Tablica 6/5a Table 6/5a	xl,z	nelinearnost na 0 lack of fit at zero	-2,20	< 5,0 nmol/mol	Zadovoljava Complies
Tablica 6/6 Table 6/6	Ec	učinkovitost konvertera converter efficiency	98,8%	> 95%	Zadovoljava Complies
Tablica 1/13 Table 1/13	Ds,z	kratkotrajni odmak na nultom plinu short-term drift at zero level	-1,05	< 2,0 nmol/mol kroz 12 sati / over 12 hours	Zadovoljava Complies
Tablica 1/14	Ds,s	kratkotrajni odmak na rasponu short-term drift at span	2,96	< 6,0 nmol/mol kroz 12 sati / over 12 hours	Zadovoljava Complies

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

Performance test - O₃ analyzers

Broj prema Tablici 6 ili 1 norme HRN EN 14625 Number according to Table 6 or 1 of the EN 14625	Oznaka iz norme HRN EN 14625 Symbol according to EN 14625	Karakteristika Characteristic	Rezultat provjere (nmol/mol ili %) Result of the check (nmol/mol or %)	Granice prihvatljivosti Acceptance limits	Sukladnost Compliance
Tablica 6/2a Table 6/2a	Sr,z	ponovljivost na nultom plinu repeatability at zero	0,08	< 1,5 nmol/mol	Zadovoljava Complies
Tablica 6/2b Table 6/2b	Sr	ponovljivost na rasponu (80% mjernog područja) repeatability at span (80% of measurement range)	0,12%	< 2,0%	Zadovoljava Complies
Tablica 6/5b Table 6/5b	r _{max}	nelinearnost za točke različite od 0 lack of fit at concentrations higher than zero	0,16%	< 4,0%	Zadovoljava Complies
Tablica 6/5a Table 6/5a	xl,z	nelinearnost na 0 lack of fit at zero	-0,06	< 5,0 nmol/mol	Zadovoljava Complies
Tablica 1/13 Table 1/13	DS,z	kratkotrajni odmak na nultom plinu short-term drift at zero level	0,55	< 2,0 nmol/mol kroz 12 sati / over 12 hours	Zadovoljava Complies
Tablica 1/14 Table 1/14	DS,s	kratkotrajni odmak na rasponu short-term drift at span level	2,61	< 6,0 nmol/mol kroz 12 sati / over 12 hours	Zadovoljava Complies

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

Performance test - C₆H₆ analyzers

Tablica u normi HRN EN 14662-3 Table in the EN 14662-3	Oznaka iz norme HRN EN 14662-3 Symbol according to EN 14662-3	Karakteristika Characteristic	Rezultat provjere ($\mu\text{g}/\text{m}^3$ ili %) Result of the check ($\mu\text{g}/\text{m}^3$ or %)	Granice prihvatljivosti Acceptance limits	Sukladnost Compliance
9	S _{r,ct}	ponovljivost na rasponu repeatability at span	0,12	< 0,25 $\mu\text{g}/\text{m}^3$	Zadovoljava Complies
9	S _{rz}	ponovljivost na 10%-tnoj razini godišnje vrijednosti repeatability at at 10 % of the level of the annual limit	0,06	< 0,2 $\mu\text{g}/\text{m}^3$	Zadovoljava Complies
9	r _{max}	nelinearnost, najveće odstupanje lack of fit, largest residual	2,57%	< 5,0%	Zadovoljava Complies
formula (3)	l _{det}	granica detekcije detection limit	0,06	N/A	
1	D _{s,s}	kratkotrajni odmak na rasponu short term drift at span level	0,01	< 2 $\mu\text{g}/\text{m}^3$	Zadovoljava Complies

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

CORRECTIVE ACTIONS

In case of exceeding the limits of acceptability of working characteristics and other major problems with the tests, the analyzer is considered unsuitable for further measurements and it is provided for servicing.

After servicing, it is necessary to calibrate and check its operating characteristics.

If calibration and checking show that the analyzer is suitable for further measurement, it is returned to the measuring station.

If checking the response to zero gas and gas range shows that the eligibility criteria are not satisfied, then the metrics are validated again for the period of the time when the respondents were last satisfied with the criteria.

9.3 EXAMPLES OF USING HRN EN STANDARDS

RESULTS QUALITY ASSURANCE

EXTERNAL QUALITY CONTROL

External quality control is performed by participating in ability testing schemes.

Ability Testing Schemes are organized by the organizer of Ability Testing in compliance with the requirements of international standard HRN EN ISO/IEC 17043.

9.3 EXAMPLES OF USING HRN EN STANDARDS

MEASUREMENT UNCERTAINTY

Based on data from the type approval, calibration and measurement at the station the measurement uncertainty for the data from the reporting period is calculated and reported in annual report in the form of expanded measurement uncertainty.

Measurement uncertainties are calculated according to the following standards:

HRN EN 14211:2012 – for NO/NO_x measurement

HRN EN 14212:2012 – for SO₂ and H₂S measurement

HRN EN 14625:2012 – for O₃ measurement

HRN EN 14626:2012 – for CO measurement

HRN EN 14662-3:2012 – for C₆H₆ measurement

and document JCGM 100 (GUM method).

9.3 EXAMPLES OF USING HRN EN STANDARDS

MEASUREMENT UNCERTAINTY

An example of a measurement uncertainty for **CO measurements in accordance with HRN EN 14626:2012**:

Measurement uncertainty is calculated in the form of extended measurement uncertainty for hourly averaging time at the *limit value for the maximum daily 8-hour sliding average* u_c , absolute extended uncertainty for hourly averaging time at the limit value for 8-hour sliding average U_c and relative uncertainty $U_{c,rel}$.

Measurement uncertainty is calculated for a specific analyzer (e.g. HORIBA APMA 370 below) using the data from the type approval and the calibration certificate.

9.3 EXAMPLES OF USING HRN EN STANDARDS

MEASUREMENT UNCERTAINTY

HRN EN 14626	Code from standard	Component of measurement uncertainty	Results from	$u(p)$ ($\mu\text{mol/mol}$)	$u^2(p)$
1	$U_{r,z}$	repeatability at zero gas	Calibration certificates	x	y
2	$U_{r,lv}$	repeatability at limit value for 8h GV	Calibration certificates	x	y
3a	U_l	nonlinear analyzer (lack of fit test) for 8h GV	Calibration certificates	x	y
4	U_{gp}	impact of pressure variability of sampled gas	Typ. approval Table 38	0,0120	0,0001
5	U_{gt}	impact of temperature variability of sampled gas	Typ. approval Table 38	0,0240	0,0006
6	U_{st}	impact of temperature variability of ambient air	Typ. approval Table 38	-0,1823	0,0332
7	U_v	impact of voltage variability of electricity	Typ. approval Table 38	0,0280	0,0008
8a	U_{H_2O}	presence of water steam at limit value for 8h GV	Typ. approval Table 38	0,3261	0,1063
8b,c,d	U_{int}	interfering substances (positive int. – negative int.)	Typ. approval Table 38	0,4051	0,1641

9.3 EXAMPLES OF USING HRN EN STANDARDS

MEASUREMENT UNCERTAINTY

9	u_{av}	averaging effect	Typ. approval Table 38	0,0621	0,0039
10	$u_{r,f}$	reproducibility	Typ. approval Table 38	0,0052	0,00003
11	$u_{d,l,z}$	long-term shift to zero	Typ. approval Table 38	0,0993	0,0099
12	u_{d,l,l_v}	long-term shift to limit value for 8h GV	Typ. approval Table 38	0,0268	0,0007
21	u_{cg}	calibration gas at 8h GV	Calibration certificates	x	y
Square of the assembled measurement uncertainty $u^2 = \sum u^2(p)$					
Compound measurement uncertainty at 8h GV $u_{GV} = \sqrt{u^2}$				($\mu\text{mol/mol}$)	
Expanded uncertainty at 8h GV $U_{GV} = u \cdot k$ ($k=2$)				($\mu\text{mol/mol}$)	
Relative uncertainty at 8h GV $U_{GV,rel} = (U_{GV}/8,6) \cdot 100$				(%)	
Relative uncertainty at 8h GV required by regulations				(%)	15

9.3 EXAMPLES OF USING HRN EN STANDARDS

MAINTENANCE

HRN EN standards also describe and require maintenance, which includes:

- particle filter changes (at least every 3 months)
- modification of sampling lines (at least every 6 months)
- Changes in consumables (according to manufacturer's requirements)
- preventive/regular maintenance of parts of the analyzer (according to manufacturer's requirements)

9.4 TYPE APPROVAL OF ANALYZER

TYPE APPROVAL

A type approval is a procedure which determines and confirms after the equipment testing is carried out, that this type of equipment complies with the conditions of relevant directives.

The type approval standard is the standard that provides the requirements which some type of equipment must meet to comply with the relevant directive.

This standard also defines which tests should be carried out to determine the value of the parameters being observed and evaluated.

9.4 TYPE APPROVAL OF ANALYZER

Type approvals for individual analyzers are described in HRN EN standards:

HRN EN 14211:2012 – for NO/NO_x analyzers

HRN EN 14212:2012 – for analyzers

HRN EN 14625:2012 – for O₃ analyzers

HRN EN 14626:2012 – for CO analyzers

HRN EN 14662-3:2012 – for C₆H₆ analyzers

9.4 TYPE APPROVAL OF ANALYZER

Type approvals for NO/NO_x analyzers – HRN EN 14211:2012, chapter 8

Determination of NO and NO₂ concentrations in the ambient air must be consistent with the requirement of the highest measurement uncertainty of measurement values as described in Directive 2008/50/EC.

In order to achieve that the measurement uncertainty is less than or equal to the required, the chemiluminescent analyzer must meet the criteria for a large number of operating characteristics as defined in Chapter 8 of the HRN EN 14211: 2012 standard (table 1).

The standard provides the criteria, test methods and formulas for calculations based on which it can determine if the analyzer can be approved and placed on the market.

9.4 TYPE APPROVAL OF ANALYZER

Table 1 — Relevant performance characteristics and criteria

nr	Performance characteristic	Symbol	Section	Lab. test		Field test		Performance criterion for NO and/or NO ₂
				NO	NO ₂	NO	NO ₂	
1	Repeatability standard deviation at zero	$s_{r,z}$	8.4.5	x				$\leq 1,0$ nmol/mol
2	Repeatability standard deviation at concentration c_t	$s_{r,ct}$	8.4.5	x				$\leq 3,0$ nmol/mol
3	Lack of fit (residual from the linear regression function)		8.4.6					
3a	Largest residual from the linear regression function at concentrations higher than zero	r_{max}		x				$\leq 4,0$ % of the measured value
3b	Residual at zero	r_z		x				$\leq 5,0$ nmol/mol
4	Sensitivity coefficient of sample gas pressure	b_{gp}	8.4.7	x				$\leq 8,0$ nmol/mol/kPa
5	Sensitivity coefficient of sample gas temperature	b_{gt}	8.4.8	x				$\leq 3,0$ nmol/mol/K

9.4 TYPE APPROVAL OF ANALYZER

6	Sensitivity coefficient of surrounding temperature	b_{st}	8.4.9	x				$\leq 3,0$ nmol/mol/K
7	Sensitivity coefficient of electrical voltage	b_V	8.4.10	x				$\leq 0,30$ nmol/mol/V
8	Interferents at zero and at concentration c_t^a		8.4.11					
8a	H ₂ O with concentration 19 mmol/mol ^b	$X_{H_2O,z.ct}$		x				$\leq 5,0$ nmol/mol
8b	CO ₂ with concentration 500 μ mol/mol	$X_{CO_2,z.ct}$		x				$\leq 5,0$ nmol/mol
8c	NH ₃ with concentration 200 nmol/mol ^c	$X_{NH_3,z.ct}$			x^c			$\leq 5,0$ nmol/mol
9	Averaging effect	E_{av}	8.4.12	x	x			$\leq 7,0$ % of the measured value
10	Reproducibility standard deviation under field conditions	$s_{r,f}$	8.5.5				x	$\leq 5,0$ % of the average of a three month period
11	Long term drift at zero level	$D_{l,z}$	8.5.4				x	$\leq 5,0$ nmol/mol
12	Long term drift at span level	$D_{l,s}$	8.5.4				x	$\leq 5,0$ % of maximum of certification range
13	Short-term drift at zero level	$D_{s,z}$	8.4.4	x				$\leq 2,0$ nmol/mol over 12 h
14	Short-term drift at span level	$D_{s,s}$	8.4.4	x				$\leq 6,0$ nmol/mol over 12 h

9.4 TYPE APPROVAL OF ANALYZER

nr	Performance characteristic	Symbol	Section	Lab. test		Field test		Performance criterion for NO and/or NO ₂
				NO	NO ₂	NO	NO ₂	
15	Response time (rise)	t_r	8.4.3	x	x			≤ 180 s
16	Response time (fall)	t_f	8.4.3	x	x			≤ 180 s
17	Difference between rise time and fall time	t_d	8.4.3	x	x			≤ 10 s
18	Difference sample/calibration port ^d	Δx_{sc}	8.4.13	x				≤ 1,0 %
19	Period of unattended operation		8.5.6			x		3,0 months or less if manufacturer indicates a shorter period
20	Availability of the analyser	A_a	8.5.7			x		≥ 90 %
21	Converter efficiency ^{e,f}	E_c	8.4.14		x			≥ 98 %
22	Residence time in the analyser		8.4.15		x			≤ 3,0 s

9.4 TYPE APPROVAL OF ANALYZER

EXAMPLE: Type approvals for
NO/NO_x analyzers

– HORIBA APNA 370

The certificate is issued by the Umwelt Bundesamt (German Federal Environment Agency) and TÜV Rheinland. It certifies the APNA 370 for NO_x analyzer from HORIBA, Ltd. The certificate number is 0000028755_03. The test institute is TÜV Rheinland Energie und Umwelt GmbH. The certificate is valid until 25 January 2021. It references standards VDI 4202-1 (2002), VDI 4203-3 (2004), EN 14211 (2012), EN 15267-1 (2009), and EN 15267-2 (2009). The certificate is awarded in respect of the conditions stated in this certificate (This certificate contains 10 pages).

Certified AMS:	APNA 370 for NO _x
Manufacturer:	HORIBA, Ltd. 2 Miyano Higashi Kisshoin Minami-ku Kyoto 610-8510 Japan
Test Institute:	TÜV Rheinland Energie und Umwelt GmbH

This is to certify that the AMS has been tested and certified according to the standards

VDI 4202-1 (2002), VDI 4203-3 (2004), EN 14211 (2012),
EN 15267-1 (2009) and EN 15267-2 (2009)

Certification is awarded in respect of the conditions stated in this certificate
(This certificate contains 10 pages).

	Suitability Tested Complying with 2008/50/EC EN 15267 Regular Surveillance www.tuv.com ID 0000028755
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Publication in the German Federal Gazette (BAnz.) of 14 October 2006	This certificate will expire on: 25 January 2021
German Federal Environment Agency Dessau, 21 January 2016 <i>Marcel Langner</i> i. A. Dr. Marcel Langner	TÜV Rheinland Energie und Umwelt GmbH Cologne, 20 January 2016 <i>Peter Wilbring</i> ppa. Dr. Peter Wilbring

9.4 TYPE APPROVAL OF ANALYZER

AMS name:

APNA 370

Manufacturer:

HORIBA, Ltd., Kyoto, Japan

Distributor:

HORIBA Europe GmbH, Leichlingen

Approval:

For continuous monitoring of NO, NO₂ and NO_x (stationary operation).

Measuring ranges during the suitability test:

NO₂ 0 - 400 µg/m³

NO₂ 0 - 500 µg/m³

NO 0 - 1200 µg/m³

Software version:

P1000878001C

Test institute:

TÜV Immissionsschutz und Energiesysteme GmbH, Cologne
TÜV Rheinland Group

Test report:

No. 936/21204643/C of 7 July 2006

9.4 TYPE APPROVAL OF ANALYZER

Certified product

This certificate applies to automated measurement systems conforming to the following description:

The APNA 370 is based on the measuring principle of chemiluminescence.

This method allows continuous measurement of the nitrogen oxides (NO , NO_2 and NO_x ($\text{NO} + \text{NO}_2$)) within the atmosphere. The concentration of NO_2 is calculated from the concentrations of NO and NO_x . The measuring principle complies with the reference measuring method described in section 5.2 of Standard EN 14211 (2012).

The sample gas is split into two streams within the APNA 370 measuring system. One stream is used for measuring the concentration of NO_x ($\text{NO} + \text{NO}_2$) by reducing NO_2 to NO via a NO_x converter. The other stream is used for direct determination of the NO concentration. The NO , NO_x and span gas tubes are switched every 0.5 s by using a solenoid valve and led into the reaction chamber.

Outside air is drawn through a separate filter, dried by a self-regenerative silica gel dehumidifier and passed through the ozonizer by generating the required ozone. The ozone is passed into the reaction chamber. The sample gas then reacts with the ozone and the emitted light is detected using a photo diode.

The device calculates the concentrations of NO , NO_2 and NO_x from the signal of the photo diode, which is proportional to the NO_x and NO concentrations, and displays the results as a continuous signal.

Dehumidifier

The device comprises a self-regenerative silica gel dehumidifier which dehumidifies the air required for generating ozone. The dehumidifier comprises two cylinders. While one cylinder is active the other is regenerated. The silica gel is heated to approx. 160 °C for about 135 minutes for this purpose in order to remove humidity. This process is followed by a cooling phase of about 45 minutes. Both cylinders are switched every 180 minutes in order to ensure constant drying.

9.4 TYPE APPROVAL OF ANALYZER

Type approvals for various analyzers:

HORIBA APNA 370 (NO/NO_x) – TUV Rheinland, Certificate No. 0000028755-03

HORIBA APMA 370 (CO) – TUV Rheinland, Certificate No. 0000028754-03

HORIBA APOA 370 (O₃) – TUV Rheinland, Certificate No. 0000028756-03

HORIBA APSA 370 (SO₂) – TUV Rheinland, Certificate No. 0000028757-03



EKONERG

Energy Research and Environmental Protection Institute



THANK YOU FOR YOUR ATTENTION

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This project is funded by the European Union