



# SLOVENSKI STANDARD

## SIST EN 17346:2020

01-julij-2020

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### Kakovost zunanjega zraka - Standardna metoda za določevanje koncentracije amoniaka z difuzijskim vzorčenjem

Ambient Air Quality - Standard method for the determination of the concentration of ammonia by diffusive sampling

Außenluftqualität - Messverfahren zur Bestimmung der Konzentration von Ammoniak mit Passivsammlern

Air ambient - Méthode normalisée pour la détermination de la concentration d'ammoniac au moyen d'échantillonneurs par diffusion

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Ta slovenski standard je istoveten z: EN 17346:2020

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#### ICS:

13.040.20      Kakovost okoljskega zraka      Ambient atmospheres

**SIST EN 17346:2020**

**en,fr,de**

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EUROPEAN STANDARD

EN 17346

NORME EUROPÉENNE

EUROPÄISCHE NORM

May 2020

ICS 13.040.20

English Version

## Ambient air - Standard method for the determination of the concentration of ammonia using diffusive samplers

Air ambiant - Méthode normalisée pour la détermination de la concentration en ammoniac au moyen d'échantillonneurs par diffusion

Außenluft - Messverfahren zur Bestimmung der Konzentration von Ammoniak mit Passivsammlern

This European Standard was approved by CEN on 13 April 2020.

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**EN 17346:2020 (E)****European foreword**

This document (EN 17346:2020) has been prepared by Technical Committee CEN/TC 264 “Air quality”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2020, and conflicting national standards shall be withdrawn at the latest by November 2020.

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

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## Introduction

Atmospheric ammonia ( $\text{NH}_3$ ) is a pollutant of major environmental concern with adverse effects on forests, species composition of semi-natural ecosystems and soils [1-4]. Emission and deposition of  $\text{NH}_3$  can contribute significantly to total nitrogen deposition to the environment, contributing to eutrophication (nutrient enrichment) and acidification (oxidation of  $\text{NH}_3$  to nitrate resulting in release of  $\text{H}^+$  ions) of land and freshwaters, leading to a reduction in both soil and water quality, loss of biodiversity and ecosystem change [5-10].

In addition to these effects,  $\text{NH}_3$  is the major precursor for neutralization of atmospheric acids, affecting the long-range transport distance of both  $\text{SO}_2$  and  $\text{NO}_x$  and leading to the formation of secondary particles (primarily ammonium sulphate and ammonium nitrate) [11-13]. These particles have multiple impacts including effects on atmospheric visibility, radiative scattering (and the greenhouse effect) and on human health.

The recognition of  $\text{NH}_3$  as an important air pollutant led to its inclusion in international agreements to reduce air pollutant emissions, first under the 1999 UNECE Gothenburg Protocol and then the National Emissions Ceilings Directive (NECD) (2001/81/EC) of the EU. The target of both these agreements is that  $\text{NH}_3$  emissions should not exceed emission ceilings set for EU member states, with a particular focus on reducing the extent of critical loads exceedance for acidification and eutrophication effects. Revision of the Gothenburg Protocol (2012) and the NEC Directive (2016) include new, more stringent emission ceilings for 2020 that seek more environmental protection and improvement in air quality than has so far been committed, including the introduction of an emissions ceiling for particulate matter (PM). Under the 2012 UNECE Gothenburg Protocol, EU member states have to jointly cut their emissions of  $\text{NH}_3$  by 6 % and particles by 22 % between 2005 and 2020. As a precursor of PM, controlling  $\text{NH}_3$  is important to reducing particle emissions of  $\text{PM}_{2.5}$  and  $\text{PM}_{10}$ . A recent study employing three chemical transport models found that the models underestimated the formation of ammonium particles and concluded that the role of  $\text{NH}_3$  on PM is larger than originally thought [14]. Thus the implementation of 2020 targets detailed above may not be enough to deliver compliance with proposed particle limit values, and further local measures may be required to be compliant. 63e/sist-en-17346-2020

Other legislations to abate  $\text{NH}_3$  emissions include the Industrial Emissions Directive (IED) (2010/75/EU) which requires pig and poultry farms (above stated size thresholds) to reduce emissions using Best Available Techniques. For the protection of vegetation and ecosystems, new revised "Critical Levels" (CL) of  $\text{NH}_3$  concentrations were adopted in 2007 (see Table 1), of  $1 \mu\text{g}/\text{m}^3$  and  $3 \mu\text{g}/\text{m}^3$  annual mean for the protection of lichens/bryophytes and higher plants under field conditions, respectively, which replaced the previous CL annual mean value of  $8 \mu\text{g}/\text{m}^3$ . A monthly critical level of  $23 \mu\text{g}/\text{m}^3$  was retained as a provisional value in order to deal with the possibility of high peak emissions during periods of manure application (e.g. in spring) ([15]). In Germany, the recommended exposure limit for the protection of ecosystems is  $10 \mu\text{g}/\text{m}^3$  (TA Luft, Annex 1, [16]).

**Table 1 — Summary of upper limits of NH<sub>3</sub> concentrations for protection of ecosystems under field conditions**

Concentration (µg/m <sup>3</sup> )	Specification	Types of locality
1	UNECE Critical Level (annual mean) for lower plants (lichens, bryophytes)	Sensitive ecosystems in which the lichens and bryophytes are important components, e.g. designated sites for nature conservation and protection of sensitive species, e.g. Natura 2000 sites
3	UNECE Critical Level (annual mean) for higher plants	Sensitive ecosystems in which the higher plants are important components, e.g. designated sites for nature conservation and protection of sensitive species, e.g. Natura 2000 sites
10	German First General Administrative Regulation Pertaining to the Federal Immission Control Act Maximum near installations where ecological monitoring undertaken.	Near installations
23	UNECE critical level (monthly mean) – for peak emission periods such as in months where slurry spreading takes place.	In close proximity to emission sources

Improving knowledge on levels of NH<sub>3</sub> in the ambient air and near sources is therefore important for the assessment of:

- environmental effects on ecosystems (Contribution to eutrophication and acidification processes);
- contributions to the formation of PM<sub>10</sub> and PM<sub>2,5</sub>;
- effectiveness of current and future abatement measures to reduce NH<sub>3</sub> emissions.

The simplest to the latest state-of-the-art techniques for measurement of atmospheric NH<sub>3</sub> are presented in Table 2.



**Table 2 — Measurement methods suitable for determination of atmospheric NH<sub>3</sub> gas and ammonium particle concentrations**

Monitoring Methods	Time resolution	References
<b>Integrative methods: passive</b>		
Passive diffusion samplers	daily to monthly	[17] [18] [19] [20]
<b>Integrative methods: active</b>		
Simple denuder systems with offline chemical analysis	daily to monthly	[17] [19] [21]
Annular denuder systems (ADS) with offline chemical analysis	hourly to daily	[22]
Conditional sampling with denuders at different heights (COTAG)	weekly to monthly	[23]
<b>Continuous: wet chemistry methods</b>		
Annular Denuder Systems with online analysis Membrane stripping with online analysis	hourly or better depending on set-up	[24]
Steam Jet Aerosol Collector Systems for gas and aerosol	hourly or better depending on set-up	[25] [26]
<b>Continuous: optical methods</b>		
Differential Optical Absorption Spectrometry (DOAS)	hourly or better depending on set-up	[27]
Tunable Diode Laser Absorption Spectrometry and Quantum Cascade Laser (TDL and QCL AS, respectively)	hourly or better depending on set-up	[28]
Photoacoustic spectrometry	hourly or better depending on set-up	[29]
Chemiluminescence with catalytic conversion	hourly or better depending on set-up	[30]

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Integrative atmospheric sampling methods such as passive diffusion samplers and active samplers provide measurement of concentrations of NH<sub>3</sub> averaged over the chosen sampling time. The diffusive samplers used include those that are available commercially and those that have been developed in-house by organisations to meet specific research requirements. A full validation of diffusive sampling methods for NH<sub>3</sub> in accordance with the European Standard (EN 13528-2 [31]) would be costly and would also require specialist facilities only available at well-equipped large metrological institutes. Validation of the quantitative measurement of NH<sub>3</sub> through comparison with “reference” methods is problematic for NH<sub>3</sub> as there is no currently accepted and defined reference method. Automatic continuous analysers for NH<sub>3</sub>, employing spectroscopic or other techniques (Table 2) are available commercially, but there is a lack of robust published calibration data and procedures for reliable field measurements under ambient concentrations and conditions [32].

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## 1 Scope

This document specifies a method for the sampling and analysis of NH<sub>3</sub> in ambient air using diffusive sampling.

It can be used for NH<sub>3</sub> measurements at ambient levels, but the concentration range and exposure time are sampler dependent, and the end user is therefore advised to match the sampler type to the measurement requirement and to follow the operating instructions provided by the manufacturer.

## 2 Normative references

There are no normative references in this document.

## 3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp/ui>

### 3.1

**combined standard measurement uncertainty**  
**combined standard uncertainty**

standard measurement uncertainty that is obtained using the individual standard measurement uncertainties associated with the input quantities in a measurement model

[SOURCE: JCGM 200:2012, 2.31] [33]  
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### 3.2

**extraction efficiency**

ratio of the mass of analyte extracted from a sampling device to that applied

### 3.3

**diffusive sampler**

device which is capable of taking samples of gases or vapours from the atmosphere at a rate controlled by a physical process such as gaseous diffusion through a static air layer or a porous material and/or permeation through a membrane, but which does not involve the active movement of air through the device

Note 1 to entry: Active normally refers to the pumped movement of air.

[SOURCE: EN 13528-2:2002, 3.6] [31]

### 3.4

**diffusive sampling rate**

**diffusive uptake rate**

rate at which the diffusive sampler collects a particular gas or vapour from the atmosphere

Note 1 to entry: The sampling rate is usually expressed in units of (m<sup>3</sup>/h), (ml/min) or (cm<sup>3</sup>/min).

Note 2 to entry: cm<sup>3</sup>/min may be converted to SI units of m<sup>3</sup>/s by factor 1,67 × 10<sup>-8</sup>.

**EN 17346:2020 (E)****3.5  
expanded measurement uncertainty**

product of a combined standard measurement uncertainty and a factor larger than the number one

Note 1 to entry: The factor depends upon the type of probability distribution of the output quantity in a measurement model and on the selected coverage probability.

Note 2 to entry: The term “factor” in this definition refers to a coverage factor.

[SOURCE: JCGM 200:2012, 2.35]

**3.6  
field blank**

unused sampler, taken from the same batch used for NH<sub>3</sub> monitoring, handled in the same way as a sampler that is used for NH<sub>3</sub> monitoring, except it is not used for collecting a sample

Note 1 to entry: Adapted from EN 14902:2005, 3.1.6.

Note 2 to entry: The results from the analysis of field blanks are used to identify contamination of the sample arising from handling in the field and during transport.

[SOURCE: EN 1540:2011, 3.3.8] [34]

Note 3 to entry: A transport blank is considered to be a special case of a field blank. A transport blank is taken to the exposure site, left unopened and returned to the laboratory immediately after placement or collection of the samplers. Transport blanks may be used when regular field blanks reveal an unacceptable level of ammonium to investigate the possibility of contamination of samplers during transport. This blank is only used for quality control purposes.

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**3.7  
laboratory blank**

sealed sampler drawn from the same batch as the samplers being used for NH<sub>3</sub> monitoring which is stored for the duration of the sampling period and is analysed at the same time as the exposed samplers

**3.8  
measurement uncertainty  
uncertainty of measurement**

non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

Note 1 to entry: For notes to the definition the reader is referred to the parent document JCGM 200:2012.

[SOURCE: JCGM 200:2012, 2.26]

**3.9  
standard measurement uncertainty  
standard uncertainty**

measurement uncertainty expressed as a standard deviation

[SOURCE: JCGM 200:2012, 2.30]

## 4 Description of samplers

### 4.1 Principle

The diffusive sampler is exposed in air for a measured time period.  $\text{NH}_3$  migrates through the sampler along a diffusion path of defined dimensions and is collected by reaction onto an acid sorbent.

Determining the sampling rate is essential when deploying diffusive  $\text{NH}_3$  samplers in the field, either by:

- calculation based on Fick's first law of diffusion (see EN 13528-3 [35] and Annex E),
- calibration by exposure to standard atmospheres, or
- co-located calibration studies against another well characterized  $\text{NH}_3$  measurement method in the field.

NOTE Denuders can be used as a cost effective surrogate reference method until there are improvements in the continuous optical methods.

Details of these approaches shall be documented.

Samplers can be provided with manufacturer measured sampling rates. Samplers in networks often have on-going measurements of sampling rates. Users can calculate a locally derived sampling rate. Sampling rates are also documented in literature [see [36], and Annex D].

The sampling rate in the field is a function of local meteorology. Samplers can be deployed with protective shelters to minimize meteorological influences. When doing so, the user shall apply a suitable protocol to ensure a consistent approach for all samplers. Ideally, the effect of the shelter on the sampler performance should be characterized.

### 4.2 Implementation

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Samplers shall be sealed and stored under cool conditions, for example at temperatures between 0 °C and 4 °C, in the dark, in order to minimize any undesired reactions before and after deployment. After deployment, samplers shall be analysed as soon as possible, according to manufacturer's specifications.

Disposable gloves shall be worn at all times, including during deployment in the field. This serves to protect the samples from contamination by contact with the skin. It is also advised to avoid breathing directly on the samples, as exhaled breath contains  $\text{NH}_3$ .

Since there are different sampler designs, each common sampler type is briefly described below.

### 4.3 Tube-type samplers

The tube-type samplers are hollow cylindrical tubes oriented vertically. A cap at the top end holds in place either a cellulose filter paper, glass fibre filter or stainless steel grid, which is coated with a sorbent that collects the gas of interest. This type of sampler is characterized by a high length to cross sectional area ratio [15, 37]. To collect  $\text{NH}_3$ , sorbents used include citric, phosphoric, phosphorous, sulphuric and tartaric acid [38]. The analysis is carried out using various methods including ion chromatography, flow injection analysis with detection of conductivity and spectrophotometry.

There is one commonly used design of tube type samplers, the 3,5 cm short membrane diffusion tube.

For more information, see Annex A.