



# SLOVENSKI STANDARD

SIST EN 14671:2006

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Characterization of sludges - Pre-treatment for the determination of extractable ammonia using 2 mol/l potassium chloride

Charakterisierung von Schlämmen - Vorbehandlung zur Bestimmung des extrahierbaren Ammoniums unter Verwendung von 2 mol/l Kaliumchlorid

Caractérisation des boues - Prétraitement pour le dosage de l'azote ammoniacal extractible par une solution de chlorure de potassium a 2 mol/l

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Ta slovenski standard je istoveten z: EN 14671:2006

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ICS 13.030.20

English Version

## Characterization of sludges - Pre-treatment for the determination of extractable ammonia using 2 mol/l potassium chloride

Caractérisation des boues - Prétraitement pour la détermination de l'Azote ammoniacal extractible utilisant 2mol/l de chlorure de potassium

Charakterisierung von Schlämmen - Vorbehandlung zur Bestimmung des extrahierbaren Ammoniums unter Verwendung von 2 mol/l Kaliumchlorid

This European Standard was approved by CEN on 24 May 2006.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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## Foreword

This document (EN 14671:2006) has been prepared by Technical Committee CEN/TC 308 “Characterization of sludges”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by December 2006, and conflicting national standards shall be withdrawn at the latest by December 2006.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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## Introduction

Because sludge may be applied to land as manure or as a disposal, there is a need to monitor extractable ammonia.

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## 1 Scope

This European standard specifies a procedure for the determination of extractable ammonia using a 2 mol/l potassium chloride solution in raw and digested sewage sludges. The range of the method is up to 100 g/kg N assuming appropriate dilution of the potassium chloride extract in the final measurement step.

NOTE The above wording will be modified once the round robin results are available. It may be suitable for other types of sludges, but the user should validate the method using these sludges.

## 2 Normative references

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

EN 12880:2000, *Characterization of sludges — Determination of dry residue and water content*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

## 3 Terms and definitions

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

### 3.1

#### **extractable ammonia**

ammonia that is released when the homogenised sludge is shaken with 2 mol/l potassium chloride for 1 h at room temperature

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### 3.2

#### **dry residue**

dry mass portion of the sludge obtained after the specified drying process, expressed as a percentage

[EN 12880:2000, 3.1]

## 4 Principle

An aliquot of the homogenised sludge is shaken for 1 h with 2 mol/l potassium chloride at room temperature. The sample is then filtered and the ammonia determined by a suitable method. Potassium chloride (2 mol/l) is used rather than water to ensure that both soluble and ion exchangeable forms of the determinand are determined. It also minimises biological activity in the extracted sample.

All results are expressed as nitrogen.

Although undried samples are taken for analysis, it is recognised practice to report results on a dry weight basis. Consequently the dry residue content of the homogenised sample used for analysis should also be determined using a separate test portion (see EN 12880).

## 5 Limitations and interferences

Substances present at their normal concentration in these types of samples should not cause significant interference in the pre-extraction step. For the vast majority of sewage sludges, the relatively high concentration of ammonia compared with potential interferences should not result in any significant

interferences in the final measurement stage. The user should carry out suitable recovery checks to confirm this.

## 6 Hazards

### 6.1 General

Waste and sludge samples may contain hazardous and inflammable substances. They may contain pathogens and be liable for biological action. Consequently it is recommended that these samples should be handled with special care. The gases, which may be produced by microbiological activity are potentially inflammable and will pressurise sealed bottles. Exploding bottles are likely to result in infectious shrapnel and/or pathogenic aerosols. Glass bottles should be avoided whenever possible. National regulations should be followed with respect to microbiological hazards associated with this method.

NOTE All equipment used in the vicinity should be flame proof to avoid any risk of ignition.

### 6.2 Storage

It is not advisable to store samples in an open laboratory. If samples are to be stored, store them between 0 °C and 4 °C. Sample containers that enable the escape of any generated gas should be used.

## 7 Reagents

7.1 **Water** complying with purity grade 2 of EN ISO 3696:1995

7.2 **Potassium chloride c(KCl)** = 2,5 mol/l

Dissolve (931,9 ± 1) g of potassium chloride (dried at (105 ± 2) °C for 1 h) in approximately 3 l of water and dilute to 5 l with water in a volumetric flask.

## 8 Apparatus

Ordinary laboratory apparatus and the following:

### 8.1 Extraction bottles

Wide-necked glass or plastics bottles with secure stoppers or caps, for example 250 ml nominal capacity.

### 8.2 Mechanical shaking apparatus

Any suitable horizontal or inversion shaker shall be used (e. g. horizontal shaker (20 rpm to 40 rpm with ~20 mm throw); roller table rotating at about 10 rpm; end-over-end tumbler (5 rpm to 10 rpm)).

NOTE Other shaking devices may be used provided that they can be shown to provide equivalent results. The material being leached should not settle or be agitated too vigorously so that particle abrasion occurs.

8.3 **Balance** with an accuracy ± 0,05 g.

8.4 **Drying oven**, capable of maintained at (105 ± 5) °C

8.5 **Suitable apparatus for the determination of ammonia in diluted sludge filtrates**

(See 10.4).



## 8.6 Filter papers

Cellulose filter papers with a pore size of 20  $\mu$  to 25  $\mu$ . (For example Whatman 541<sup>1)</sup>).

## 9 Sample pre-treatment

### 9.1 Sample preservation

Sludge samples can change composition through biological and/or chemical activity. They shall be kept in cool boxes at less than 4 °C in transit to the laboratory. They shall be analysed as soon as possible (within 24 h) after sampling.

If prolonged storage is unavoidable, then store the samples at or below a temperature of –18 °C in a freezer for a maximum period of 4 weeks and note this on the test report.

### 9.2 Preparation of sample

The purpose of pre-treatment is to obtain a representative and homogeneous sample. Samples shall not be subject to any drying prior to homogenisation because this will result in a loss of ammonia. For frozen samples see NOTE below.

NOTE For frozen sludges, it is recommended that a suitable aliquot (equivalent to 0,25 g to 1 g of dry matter) of the frozen homogenised sample is directly transferred to the potassium chloride solution, as transformation of nitrogen compounds can occur during the thawing process.

## 10 Procedure

**10.1** Transfer a known weight ( $m$  gram) of suitably homogenised (as sampled) sludge (equivalent to (0,25 to 1) g  $\pm$  2 mg of dry matter, see NOTES 1 and 2) to an extraction bottle (8.1). Add (80  $\pm$  1) ml 2,5 mol/l potassium chloride (7.2) and (20 –  $m$ )  $\pm$  1 ml of water (7.1) (See also 9.2, NOTE).

NOTE 1 If a dry solids result is not available and an immediate analysis is required, then visually estimate the dry solids content.

NOTE 2 The maximum “as received” weight of sludge taken should not exceed 20 g.

**10.2** Replace the bottle cap and place on a shaker for (60  $\pm$  5) min at room temperature (see 8.2).

**10.3** Owing to the difficulty in filtering many types of sludge samples, it is recommended that a suitable aliquot of the leached sample is centrifuged. Then filter a suitable volume aliquot through a fluted filter paper (8.6) discarding at least the first 10 ml. In some cases paper filtration is too slow or even impossible. In such cases a validated alternative procedures for obtaining a clear supernatant are acceptable and the technique used shall be reported. The filtrate is stable for some hours in a hermetically closed polyethylene bottle if stored in a refrigerator at (0 to 4) °C. The filtrate can be stored for longer periods in a deep freezer at less than –18 °C.

NOTE Vacuum filtration should not be used as it can result in loss of ammonia.

In most instances the ammonia concentration will be sufficiently high to dilute the leached sample extract 10 times prior to this filtration step. This will greatly aid the speed of the filtration step. This dilution must be

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1) This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.