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EUROPEAN STANDARD

EN 13342

NORME EUROPÉENNE

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English version

## Characterisation of sludges - Determination of Kjeldahl nitrogen

Caractérisation des boues - Détermination de l'azote  
KjeldahlCharakterisierung von Schlämmen - Bestimmung des  
Stickstoffs nach Kjeldahl

This European Standard was approved by CEN on 5 August 2000.

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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## Foreword

This European Standard 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of an identical text or by endorsement, at the latest by February 2001, and conflicting national standards shall be withdrawn at the latest by February 2001.

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

The annex A is informative.

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## Introduction

Because sludge may be applied to land as a nutrient or as a disposal route, there is a need to monitor nitrogen content and application rates.

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## 1 Scope

This method describes a procedure for the determination of "Kjeldahl Nitrogen" in sludge and sludge products. The digestion is catalysed by selenium or copper, the temperature being raised by a high concentration of sodium sulphate.

Although wet samples are normally taken for analysis, it is recognised practice to report results on a dry mass basis (g/kg). Consequently, it is also necessary to determine the dry residue of the homogenised sample used for analysis (see EN 12880).

## 2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 12880, *Characterization of sludges – Determination of dry residue and water content*.

EN 25663, *Water quality – Determination of Kjeldahl nitrogen – Method after mineralization with selenium* (ISO 5663 : 1984).

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

EN ISO 5667-13, *Water quality – Sampling – Part 13: Guidance on sampling of sludges from sewage and water-treatment works* (ISO 5667-13 : 1997).

ISO 5664, *Water quality – Determination of ammonium – Distillation and titration method*.

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## 3 Terms and definitions

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

**3.1**  
**Kjeldahl nitrogen**  
nitrogen that is contributed by free ammonia, inorganic ammonia compounds and those types of organic nitrogen compounds that are converted to ammonium sulphate by the digestion process described (catalysed sulphuric acid digestion)

**3.2**  
**dry residue**  
the dry mass portion of the sludge obtained after the specified drying process. It is expressed in percent or in grams per kilogram (see EN 12880)

## 4 Principle

Rigorous acid digestion of the sample in the presence of selenium or copper converts most nitrogen compounds present to ammonium sulphate. Sodium sulphate is added to raise the digest to the appropriate temperature.

Distillation of the digest under alkaline conditions into excess dilute sulphuric acid (or excess boric acid) liberates the ammonia giving a solution of ammonium sulphate (or ammonium borate). This solution can then be analyzed for ammonia using the appropriate method (see ISO 5664 and EN 25663).

## 5 Limitations and interferences

### 5.1 Limitations

The digestion fails to determine nitrogen in the form of azide, azine, azo-compounds, hydrazine, nitrate, nitrite, nitroso-compounds, oxime, semi-carbazone and nitrile.

### 5.2 Nitrate/Nitrite

The presence of nitrate or nitrite may cause erratic results, but not at the levels normally found in sludge.

## 6 Reagents

During the analysis, use water of purity grade 2 as specified in EN ISO 3696 and reagents of recognised analytical grade.

### 6.1 Sulphuric acid, $\text{H}_2\text{SO}_4$ , $\rho = 1,84$ g/ml

### 6.2 Digestion catalyst

#### 6.2.1 General

Previous experience has shown that equivalent results can be obtained using a less toxic copper catalyst (6.2.3) in place of the selenium catalyst (6.2.2). However, before employing this catalyst, the user should demonstrate that equivalent results can be obtained for the typical range of sludges analysed. If the results are to be used for legal purposes or to resolve trade disputes then the selenium catalyst should be used. The selenium catalyst method should be regarded as the absolute reference method. For the vast majority of sludges the copper catalyst should give fit for purpose results.

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#### 6.2.2 Selenium catalyst

Thoroughly mix (1 000 ± 20) g of anhydrous sodium sulphate with (50 ± 1) g of selenium powder or previously ground selenium pellets.

**WARNING** This mixture is toxic. Inhalation of any dust resulting from its preparation or use shall be avoided. All residues containing selenium shall be collected for selenium recovery or controlled disposal.

NOTE This catalyst is commercially available as tablets.

#### 6.2.3 Copper catalyst (See 6.2.1)

Thoroughly mix (1000 ± 20) g of anhydrous sodium sulphate with (100 ± 2) g of copper sulphate pentahydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ).

NOTE This catalyst is commercially available as tablets.

### 6.3 Sodium hydroxide solution

Cautiously add approximately 800 ml of water to (500 ± 20) g of sodium hydroxide pellets contained in a plastics beaker, with stirring and dissolve. It is strongly recommended that the outside of the beaker is cooled with running cold water whilst adding the 800 ml of water. When cooled, dilute with water to 1000 ml in a stoppered measurement cylinder. Store in a polyethylene bottle.

### 6.4 Ammonia receiving reagents

#### 6.4.1 General

A reagent for a colorimetric and a reagent for a titrimetric endpoint are given.



#### 6.4.2 Sulphuric acid, solution $c(\text{H}_2\text{SO}_4) = 0,05 \text{ mol/l}$ (colorimetric endpoint)

Add  $(2,8 \pm 0,1)$  ml of sulphuric acid (6,1) to 800 ml water (6.5) and dilute with water to  $(1\ 000 \pm 5)$  ml.

#### 6.4.3 Boric acid solution, $\text{H}_3\text{BO}_3$ 40g/l (titrimetric endpoint)

Add  $(40 \pm 1)$  g of boric acid to 800 ml water and dissolve. Add 6 ml pH 4,5 indicator solution and dilute to  $(1\ 000 \pm 5)$  ml with water. One drop of 0,1 mol/l sodium hydroxide should be sufficient to change the colour of 20 ml of this reagent from purple to green. If not discard and prepare freshly.

NOTE This solution is commercially available.

## 7 Apparatus

### 7.1 Digestion vessels

The digestion vessel shall be suitable for the mass or volume of the test portion used. For example: -

Kjeldahl distillation flasks, 300 ml nominal capacity

or

Glass digestion tubes, 250 ml capacity for use with a temperature controlled heating block.

### 7.2 Heating device

Kjeldahl flasks can be heated singly or more usually, in a rack carrying several vessels, by use of either a Bunsen burner or electric heating mantle. It is important, however, that the glass surface above the digestion mixture receives no direct heating.

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Heating blocks capable of maintaining a temperature sufficient to reflux the digestion mixture are commercially available. These hold typically up to 24 digestion tubes allowing for high laboratory throughput.

### 7.3 Distillation apparatus

The digestion vessel should be suitable for direct attachment to glass apparatus incorporating an anti-splash distillation head, a vertical water condenser and a delivery tube, the outlet of which is submerged in the absorbent solution.

Commercial automatic Kjeldahl steam distillation apparatus is suitable for this determination.

### 7.4 Boiling aids

Anti-bumping granules or glass beads.

## 8 Sample pretreatment

Drying of sludge will tend to deplete free ammonia and, depending on the pH, ammonium salts. For this reason, a test portion of a wet homogenised sample shall be used (see EN ISO 5667-13).

## 9 Procedure

9.1 Transfer a known mass of homogenised, wet sludge (equivalent to 0,25 g to 0,5 g of dry mass) to a digestion vessel (7.1).

A blank shall be run by substituting 5 ml of water instead of the wet sludge with each batch of samples.