



SLOVENSKI STANDARD
SIST EN 12176:1999
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Ugotavljanje lastnosti blata - Določevanje pH vrednosti

Characterization of sludge - Determination of pH-value

Charakterisierung von Schlamm - Bestimmung des pH-Wertes

Caractérisation des boues - Détermination de la valeur du pH

Ta slovenski standard je istoveten z: EN 12176:1998

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Characterization of sludge - Determination of pH-value

Caractérisation des boues - Détermination de la valeur du
pHCharakterisierung von Schlamm - Bestimmung des pH-
Wertes

This European Standard was approved by CEN on 16 January 1998.

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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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of an identical text or by endorsement, at the latest by August 1998, and conflicting national standards shall be withdrawn at the latest by August 1998.

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.

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0 Introduction

The pH value is a necessary parameter for monitoring the treatment of urban and comparable industrial sludges as well as of sludges from water supply treatment plants and to assess the suitability of the use of sludge in agriculture or for its disposal to land fill. The pH value of liquid sewage sludge may be used to monitor digester performance.

1 Scope

The scope of this standard is the sludges, i.e. and sludges products from :

- storm water handling ;
- night soil ;
- urban wastewater collecting systems ;
- urban wastewater treatment plants ;
- treating industrial wastewater similar to urban wastewater (as defined in Directive 91/271 EEC) ;
- water supply treatment plants ;
- water distribution systems ;
- but excluding hazardous sludges from industry.

This method is applicable to the determination of pH values of sludges which include liquid, paste-like or solid matter. If the sludge sample is sufficiently liquid, direct measurement should be made. If the sludge sample contains relatively high percentages of solids the measurement should be carried out after dilution with water.

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 amendment 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.

prEN 12880	Characterization of sludges – Determination of dry residue and water content
ISO 3696	Water for analytical laboratory use - Specification and test methods
ISO 10523:1994	Water quality - Determination of pH

3 Definitions

For the purpose of this standard the following definitions apply :

3.1 pH-value

The pH-value of an aqueous solution is theoretically a function of the electromotive force of a measuring cell (e.g. a glass electrode and a calomel electrode) which is defined by the equation given in Annex A of ISO 10523:1994. The practical pH-scale refers to exactly specified buffer solutions.

3.2 Standard buffer solutions

Solutions of certain concentrations of pure substances or mixtures thereof with defined pH values.

4 Principle

A suitable aqueous suspension of the solid or paste-like sludge is prepared and the pH value of the suspension is determined potentiometrically. In liquid samples the measurement is made directly.

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5 Interferences

Substances present at their normal concentration in these types of samples do not cause significant interference. The pH value of suspensions is dependent, to a considerable extent, on the carbon dioxide equilibrium. Therefore manipulation after the sampling of liquid sludge and after the preparation of suspensions should be kept to a minimum. If oil or grease is visible in the sample, it should be removed by filtration through a pad of cotton wool (8.9) prior to carrying out the measurement. The presence of sulphides or arsenic may poison the glass interface of the electrode.

Electrodes with a low "alkali error" should be used for the measurement of sludges expected to be of a high pH.

NOTE 1 : Glass electrodes normally respond to sodium ions at pH values above 9. This may cause errors in the measurement of the pH values of samples such as lime conditioned sludges.

NOTE 2 : A film may form on the surface of the pH-electrodes due to oil and grease. This may lead to errors in the measuring results. Inorganic film-forming substances may also interfere if not removed from the pH-electrodes between determinations.

NOTE 3 : If sludge samples are taken from a digester, digester gas may escape causing changes in the carbon dioxide content, which may lead to errors in the measured results.

6 Hazards

Samples of sewage sludge are liable to ferment. Do not store them in the open laboratory. If samples must be stored, then store them at (0 to 4) °C. When handling a stored sample wear gloves, face and eye protection and sufficient body protection to guard against bottles bursting. Use bottle carriers. The gas evolved is usually flammable so all equipment in the vicinity should be flame-proof and sources of ignition absent. Bursting glass bottles can produce micro-organism contaminated shrapnel. Plastic bottles can also burst and produce a hazardous spray and aerosol. Ensure that all spillages are cleared up. Do not handle broken glass directly.

There is also a danger of hydrogen sulphide formation during storage. Cap loosely to avoid pressure build-up.

Sludge usually contains harmful organisms. Cleanliness is essential. Keep sludge away from food and eating. Protect all cuts.

7 Reagents

Analytical grade-reagents shall be used.

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7.1 Water

For the preparation of standard buffer solutions (see 3.2) freshly distilled or deionised water freshly taken from a column according to ISO 3696, Grade 3 shall be used. It shall be free from carbon dioxide and have a conductivity of $\leq 2 \mu\text{S/cm}$.

7.2 Standard buffer solutions

These shall be in accordance with ISO 10523 and Annex A (normative). Such solutions are available commercially.

7.3 Ethanol, (CH₃CH₂OH), 70 % V/V.

7.4 Acetone, (CH₃COCH₃).

8 Apparatus

8.1 pH-meter

Temperature compensated input resistance $\geq 10^{12} \Omega$ readable to at least pH = $\pm 0,05$, slope correction in mV/pH.

8.2 Glass electrode and reference electrode

The glass electrode measuring system shall have a zero voltage at pH = 7. This may consist of a glass electrode and a reference electrode, or a combination electrode system, see [2] and [3].

NOTE : Some problems are associated with the reference electrodes and it is recommended that these should be tested before use. The use of a symmetrical pH-measuring system is suggested, i.e. glass and reference electrode of the same type, e.g. Ag/AgCl electrode system. Reference electrodes with gelled filling solutions should not be used.

8.3 Thermometer, capable of measuring to the nearest 0,5 °C.

8.4 Mechanical shaking apparatus

8.5 Sample bottles, plastics or borosilicate glass, wide mouthed.

8.6 Stoppered conical flask, Erlenmeyer type, 150 ml

8.7 Balance, accuracy $\pm 0,01$ g

8.8 Drying oven, (105 ± 5) °C

8.9 Cotton pads

8.10 Soft tissue

and other usual laboratory devices.

9 Preparation of samples

9.1 Sample preservation

Sludge samples can change composition through biological or chemical activity. Therefore, they should be analyzed as soon as possible after sampling. Liquid sludge samples will lose carbon dioxide on exposure to air. They shall therefore be kept in a closed partially filled container until the pH value is determined. Handling should be minimized to avoid loss of carbon dioxide. If prolonged storage of sludge samples is unavoidable they should be stored at (0 to 4) °C in a refrigerator.