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SWEDISH STANDARDS INSTITUTE

Markundersökningar – Bestämning av total cyanid (ISO 11262:2011, IDT)

Soil quality – Determination of total cyanide (ISO 11262:2011, IDT)

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Den internationella standarden ISO 11262:2011 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av ISO 11262:2011.

Denna standard ersätter SS-ISO 11262:2004, utgåva 1.

The International Standard ISO 11262:2011 has the status of a Swedish Standard. This document contains the official version of ISO 11262:2011.

This standard supersedes the Swedish Standard SS-ISO 11262:2004, edition 1.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

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Denna standard är framtagen av kommittén för Karaktärisering av avfall, mark och slam, SIS/TK 535.

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 11262 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

This second edition cancels and replaces the first edition (ISO 11262:2003), which has been technically revised. This edition specifies two methods for the determination of the total cyanide content. It is only validated for direct liberation using orthophosphoric acid, Annex A provides new validation data. A method with an alkaline extraction before liberation is described in Annex B. In addition, the text has been editorially revised.

Introduction

Cyanides form simple salts with alkali earth cations and ionic complexes of varying strengths with numerous metal cations; the stability of these compounds is dependent on the cation and on the pH. Cyanide forms complexes with gold, mercury, cobalt and iron that are very stable even under mildly acidic conditions. Metal cyanide complexes also form salt-type compounds with alkali or heavy-metal cations, such as potassium ferrocyanide ($K_4[Fe(CN)_6]$) or copper ferrocyanide ($Cu_2[Fe(CN)_6]$). Cyanides can be present in soil both as cyanide ions and as complex cyanides.

Determination of cyanides can be carried out under different conditions. When using mild acidic conditions (e.g. pH = 4), only so-called “easily liberatable cyanides” (also known as “weak-acid dissiciable cyanides”) are measured. Under strong acidic conditions (e.g. pH = 1), all cyanides (both easily liberatable and complex cyanides) can be determined, these are called “total cyanides”.

A number of studies in soil samples have demonstrated that it is impossible to obtain reliable results for easily liberatable cyanide (ELC) using a manual ELC cyanide extraction/reflux method. Consequently, this revised International Standard does not include an ELC method.

NOTE ISO 17380 gives details of both an automated ELC method and a total cyanide method.

This International Standard specifies manual methods for the determination of total cyanide only. An alternative method for alkaline extraction prior to liberation using orthophosphoric acid is described in Annex B.

Soil quality — Determination of total cyanide

WARNING — Hydrogen cyanide and its salts are toxic. Therefore, care shall be exercised when manipulating cyanide-contaminated samples. Volatile hydrogen cyanide (with a smell of bitter almonds) is released from acidified solutions containing cyanide salts. As a minimum, all work shall be carried out in a fume hood and suitable plastic gloves shall be worn when handling contaminated samples.

Analytical wastes containing cyanides shall be placed in a special container with a lid, in the laboratory, for temporary storage. This container shall be clearly marked with labels such as "toxic waste" or "cyanides". Periodically, the container shall be emptied and the wastes containing cyanides disposed of as "special waste" by an appropriate waste-management contractor.

1 Scope

This International Standard is applicable to as-received (field-moist) samples and specifies two different procedures for the liberation of cyanide from the soil:

— direct liberation of hydrogen cyanide using orthophosphoric acid (normative);

— extraction with sodium hydroxide solution and subsequent liberation using orthophosphoric acid (informative, see Annex B).

The liberated cyanide is determined either by a photometric method or a titrimetric method using an indicator.

The method is applicable to all types of soil.

Under the conditions specified in this International Standard, the lower limit of application is 0,5 mg/kg of total cyanide (expressed on the as-received basis) for photometric determination and 10 mg/kg for titrimetric determination.

NOTE Using the alkaline extraction followed by liberation using phosphoric acid, the lower limit of application is 1 mg/kg of total cyanide (expressed on the as-received basis) for photometric determination and 30 mg/kg for titrimetric determination.

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.

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

ISO 9297, *Water quality — Determination of chloride — Silver nitrate titration with chromate indicator (Mohr's method)*

ISO 11464, *Soil quality — Pretreatment of samples for physico-chemical analysis*

ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

ISO 14507, *Soil quality — Pretreatment of samples for determination of organic contaminants*

3 Terms and definitions

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

3.1
total cyanide
all compounds which form hydrogen cyanide under the conditions of this method

3.2
recovery factor
F_{rc}
recovery factor (F_{rc}) of the liberation apparatus for total cyanide is the fractional recovery of a mid-range standard containing potassium hexacyanoferrate(III) carried through the whole procedure against an equivalent calibration standard of potassium cyanide not carried through the liberation stage, but only through the final detection stage of the method (m_{found}/m_{known})

4 Principle

4.1 Direct liberation of hydrogen cyanide using orthophosphoric acid

The field-moist sample is homogenized and pretreated in accordance with ISO 14507, removing visible coarse constituents. It is then treated with orthophosphoric acid and the liberated hydrogen cyanide is transported by an airflow and absorbed into 1 mol/l sodium hydroxide. Tin(II) and copper(II) salts are added to suppress the interference from sulfur compounds and catalyse the decomposition of complex cyanides during the liberation process.

4.2 Determination of total cyanide content

Cyanide ion in the sodium hydroxide absorber solutions is determined either

— photometrically (see Clause 9) by a procedure based on the reaction of cyanide with chloramine-T with the formation of cyanogen chloride; this reacts with pyridine-4-carboxylic acid and 1,3-dimethylbarbituric acid to form a coloured complex, the absorbance of which is measured at 606 nm, or

— titrimetrically (see Clause 10) by a titrimetric procedure involving titration with silver nitrate. When in excess relative to the $Ag(CN)_2^-$ ion, silver ions form a red-coloured complex with the end-point indicator, 5-(4-dimethylaminobenzylidene)rhodanine.

5 Reagents

All reagents shall be of recognized analytical grade and the water used shall conform to grade 2 of ISO 3696:1987. All reagents are stable for at least 3 months unless stated otherwise.

5.1 Reagents for liberation and absorption of cyanide

5.1.1 Orthophosphoric acid, $w(H_3PO_4) = 85$ % (mass fraction), $\rho = 1{,}69$ g/ml.

5.1.2 Sodium hydroxide solution, $c(NaOH) = 1$ mol/l.

Dissolve 40 g of NaOH in water and dilute with water to 1 000 ml, or use commercially available solutions. Store in a polyethylene bottle.

5.1.3 Hydrochloric acid solution, $c(HCl) = 1$ mol/l.

Dilute 98,6 g of concentrated hydrochloric acid (37 %, $\rho = 1{,}18$ g/ml) with water to 1 000 ml or use commercially available solutions.

5.1.4 Tin(II) chloride solution.

Dissolve 50 g of tin(II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$) in 40 ml of the hydrochloric acid solution (5.1.3) and dilute with water to 100 ml. Prepare a fresh solution daily.

5.1.5 Copper(II) sulfate solution.

Dissolve 200 g of copper(II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) in water and dilute with water to 1 000 ml.

5.2 Reagents for the photometric determination of cyanide

5.2.1 Sodium hydroxide solution, $c(NaOH) = 0{,}8$ mol/l.

Dissolve 32 g of NaOH in water and dilute with water to 1 000 ml. Store in a polyethylene bottle.

5.2.2 Glacial acetic acid, 20 % (volume fraction).

Dilute 100 ml of glacial acetic acid ($\rho = 1{,}049$ g/ml) to 500 ml in a measuring cylinder with water.

NOTE 100 % glacial acetic acid ($\rho = 1{,}049$ g/ml), as well as 96 % glacial acetic acid ($\rho = 1{,}06$ g/ml), are commercially available.

5.2.3 N-Chloro-4-methylbenzenesulfonamide sodium salt (chloramine-T) solution.

Dissolve 0,5 g of chloramine-T trihydrate [$C_7H_7ClNO_2S \cdot Na(3H_2O)$] in water in a 50 ml volumetric flask and dilute to the mark. Prepare a fresh solution daily.

5.2.4 Colour reagent.

Dilute 7,0 g of sodium hydroxide (NaOH) in 500 ml of water. Add 16,8 g of 1,3-dimethylbarbituric acid ($C_6H_8O_3N_2$), and 13,6 g of pyridine-4-carboxylic acid (isonicotinic acid) ($C_6H_5NO_2$), and dilute to 1 000 ml with water. Mix well for 1 h at 30 °C and then filter (pore size approximately 8 µm) through a pleated filter. This solution can be kept for at least 1 week, provided it is stored below 10 °C in the dark, and filtered through another pleated filter (pore size approximately 8 µm) before use.

5.2.5 Potassium cyanide stock solution, corresponding to 100 mg/l of cyanide ion.

Dissolve 250 mg of potassium cyanide (KCN) in the 0,8 mol/l sodium hydroxide solution (5.2.1) and dilute with the same sodium hydroxide solution to 1 000 ml in a volumetric flask. Standardize this solution by titration with the 0,01 mol/l silver nitrate solution (5.3.1), once each day if determinations are carried out (see Clause 9). Commercially available stock solutions may also be used. Store in the dark and at a temperature below 10 °C.

5.2.6 Potassium cyanide standard solution, corresponding to 10 mg/l of cyanide ion.

Dilute 10 ml of stock solution (5.2.5) to 100 ml in a volumetric flask using the 0,8 mol/l sodium hydroxide solution (5.2.1). Prepare daily.

5.2.7 Paranitrophenol (0,1 % m/V) in ethanol.

Dissolve 0,1 g of paranitrophenol in 100 ml of ethanol.

5.3 Reagents for the titrimetric determination of cyanide

5.3.1 Silver nitrate solution, $c(AgNO_3) = 0{,}01$ mol/l.

Dissolve 1,699 g of silver nitrate in approximately 400 ml of water and dilute to 1 000 ml in a volumetric flask with water. Check the actual concentration of the 0,01 mol/l silver nitrate by titration with sodium chloride in accordance with ISO 9297 on a two-weekly basis. Store this solution in the dark.

5.3.2 Silver nitrate solution, $c(AgNO_3) = 0{,}001$ mol/l.

Prepare daily from the 0,01 mol/l silver nitrate solution (5.3.1). Add 25,00 ml of 0,01 mol/l silver nitrate solution to a 250 ml volumetric flask and dilute to 250 ml with water. Cover the flask with aluminium foil to exclude light.