



# SLOVENSKI STANDARD

## SIST EN 14345:2005

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Characterization of waste - Determination of hydrocarbon content by gravimetry

Charakterisierung von Abfällen - Bestimmung des Kohlenwasserstoffgehalts mittels Gravimetrie

Caractérisation des déchets - Détermination de la teneur en hydrocarbures par gravimétrie

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Ta slovenski standard je istoveten z: <sup>SIST EN 14345:2005</sup> EN 14345:2004

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EUROPEAN STANDARD

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## Characterization of waste - Determination of hydrocarbon content by gravimetry

Caractérisation des déchets - Détermination de la teneur en hydrocarbures par gravimétrie

Charakterisierung von Abfällen - Bestimmung des Kohlenwasserstoffgehalts mittels Gravimetrie

This European Standard was approved by CEN on 9 July 2004.

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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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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## Foreword

This document (EN 14345:2004) has been prepared by Technical Committee CEN/TC 292 "Characterization of waste", the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by March 2005, and conflicting national standards shall be withdrawn at the latest by March 2005.

Anyone dealing with waste and sludge analysis should be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples may contain hazardous (e.g. toxic, reactive, flammable, infectious) substances, which can be liable to biological and/or chemical reaction. Consequently these samples should be handled with special care. Gases which may be produced by microbiological or chemical activity are potentially flammable and will pressurise sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations should be followed with respect to all hazards associated with this method.

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, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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EN 14345:2004 (E)

## Introduction

Hydrocarbons have been determined up to now mainly by infrared spectroscopy after extraction with halogenated solvents such as 1,1,2-trichloro-1,2,2-trifluoroethane or tetrachloromethane. The objective of the new standard is to provide an analytical method for the determination of hydrocarbon content by gravimetry avoiding the use of such solvents.

The user of this standard should be aware that the results obtained with the new method might not be comparable with those obtained when using infrared spectroscopy.

This document is restricted to waste samples containing large amounts of relatively high boiling hydrocarbons (boiling point above 250 °C) and may be used as a test that is easy to perform.

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## 1 Scope

This document specifies a gravimetric method for the determination of the hydrocarbon content in solid waste. It is applicable to hydrocarbon content greater than 0,5 % (m/m) on dry matter basis. This method does not permit to provide qualitative information on the nature and the source of the hydrocarbons.

NOTE Aqueous liquid waste samples can be analysed in accordance with prEN ISO 9377-1 or the procedure specified in Annex B.

## 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.

prEN 14346, *Characterization of waste — Calculation of dry matter by determination of dry residue and water content*

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

## 3 Terms and definitions

For the purposes of this document, the following term and definition applies.

### 3.1

#### hydrocarbon content by gravimetry

sum of compounds extractable with acetone/petroleum ether (2+1) which do not adsorb on Florisil and do not evaporate during the drying process.

For practical reasons, the term "hydrocarbon content by gravimetry" is abbreviated "hydrocarbon content" in the following text

NOTE 1 Substances that comply with this definition are mainly non-polar long-chain or branched aliphatic, alicyclic, polycyclic or alkyl substituted aromatic hydrocarbons.

NOTE 2 This definition differs from that given in EN 14039.

## 4 Principle

Depending on the type of waste, a known amount of the homogenised waste sample is extracted by mechanical shaking twice with acetone/petroleum ether (2+1). The combined extracts are washed twice with water. Polar compounds are removed by chromatography on Florisil. The final extract is evaporated to dryness after which the mass of the residue is determined by weighing.

## 5 Hazards

Acetone and petroleum ether are highly flammable solvents and shall therefore be handled with caution. Special care is required during centrifugation. During extraction substantial pressure can built-up in the extraction vessel. This pressure shall be released by occasional venting of the vessel in a fume hood. Due to its toxicity, the contact with the skin and eyes and the inhalation of petroleum ether vapours shall be avoided.

## EN 14345:2004 (E)

**6 Interferences**

High concentrations of more polar compounds, e.g. animal and vegetable fats and oils, may exceed the adsorption capacity of the Florisil used. A mass fraction of up to 1,0 % of such compounds will not interfere with the determination of hydrocarbon content.

**7 Reagents**

**7.1 In general**, all reagents shall be at least of reagent grade and suitable for their specific purposes Use water complying with at least EN ISO 3696, grade 3..

**7.2 Acetone**,  $(\text{CH}_3)_2\text{CO}$ ;

**7.3 Petroleum ether**, (boiling range 40 °C to 60 °C);

NOTE Other hydrocarbon solvents, boiling range 36 °C to 69 °C can be used providing extraction efficiencies are proven to be comparable.

**7.4 Florisil<sup>1</sup>**, particle size 150 µm to 250 µm (mesh 100 to 60), heated for at least 16 h at 140 °C and stored in a desiccator over a molecular sieve. Alternatively, commercially available Florisil cartridges containing 2 g of Florisil and 2 g of sodium sulfate are also applicable.

**7.5 Anhydrous sodium sulfate**,  $\text{Na}_2\text{SO}_4$ , heated for at least 2 h at 550 °C;

**7.6 Test solution** of n-octadecanoic acid octadecyl ester,  $\text{C}_{36}\text{H}_{72}\text{O}_2$ . Dissolve about 2 g of n-octadecanoic acid octadecyl ester in 100 ml petroleum ether [7.3].

**7.7 Hydrocarbon standard solution for recovery check**

Dissolve about 2 g of an heavy mineral oil such as a lubricating oil, without any additives, in 100 ml of petroleum ether [7.3].

**7.8** A plug of pre-washed glass wool or a PTFE frit is pushed down into the column [8.10]. Then, successively 2 g of Florisil [7.4] and 2 g of sodium sulfate [7.5] are added. The column shall be prepared immediately before use.

**8 Equipment**

**8.1 Standard laboratory glassware**, which shall be heated or rinsed with acetone [7.2] and dried before use;

**8.2 Mechanical shaker**, e.g. horizontal or rotary shaker;

**8.3 Evaporation device**, e.g. a rotary evaporator;

**8.4 Analytical balance** (Accuracy equal or better than 0,1 mg);

**8.5 Glass extraction vessel** of at least 100 ml, with ground glass stopper or screw caps incorporating a septum coated with polytetrafluoroethylene (PTFE);

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<sup>1</sup> Florisil is the trade name of a prepared diatomaceous substance, mainly consisting of anhydrous magnesium silicate. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product. Equivalent products may be used if it can be shown to lead to comparable results.



- 8.6 Conical flask**, 100ml or 250 ml, with ground glass stopper;
- 8.7 Separating funnel** of at least 500 ml, with ground glass stopper;
- 8.8 Glass evaporation vessel**, e.g. 100 ml or 250 ml round bottom flask;
- 8.9 Glass beaker**, 25 ml;
- 8.10 Chromatography column for clean-up**, glass columns of about 10 mm internal diameter. shall be used. The upper part of the column should be widened to use as solvent reservoir and the lower part to be narrowed to form a tip.

## 9 Sample conservation and pre-treatment

The samples shall be kept sealed in darkness at a temperature of about 4 °C and extracted within a period of one week. If this is not possible, the samples shall be stored at -18 °C or lower.

Before analysis, the samples shall be homogenised.

## 10 Procedure

### 10.1 Blank

With each series of samples, a blank determination has to be carried out in accordance with [10.3.], using all reagents in identical amounts but without a sample. If blank values exceed 0,1 % (20 mg for a typical mass of the test portion of 20 g), every step in the procedure shall be checked to find the reason for these high blanks.

### 10.2 Dry matter

Determine the dry matter on a separate sub-sample (or each of the phases when phase separation has taken place) in accordance with prEN 14346. . When stable emulsions exist, the sample shall be analysed without phase separation.

### 10.3 Extraction

Weigh to the nearest 0,1 g about 20 g of the homogenised sample into a glass extraction vessel [8.5], add 40 ml  $\pm$  1 ml of acetone [7.2]. After short shaking by hand add 20 ml  $\pm$  0,1 ml of petroleum ether [7.3]. Extract the sample for 10 min to 15 min on a mechanical shaker [8.2]. After settling of the solid material transfer as much as possible of the supernatant into a separating funnel [8.7]. Repeat extraction with the same volumes of fresh solvent. Combine the extracts and remove the acetone by washing with 100 ml of water twice. Collect the organic layer in an conical flask [8.6]. Add sufficient amount of sodium sulfate [7.5] so that no lumps are formed anymore.

NOTE 1 Alternative extraction procedures, e.g. ultrasonic or accelerated solvent extraction (ASE) can be used provided they give comparable extraction performances.

NOTE 2 To improve and accelerate phase separation centrifugation can be applied provided that the necessary safety precautions, especially with regard to inflammable solvents are taken into account.

### 10.4 Clean-up

Fill the clean-up column with Florisil in accordance with [7.8.]. Transfer the extract quantitatively to the top of the column. Rinse the conical flask containing the sodium sulfate twice with about 5 ml of petroleum ether and add this to the column. Collect the entire eluate in the evaporation vessel [8.8]. Rinse the column twice with about 5 ml of petroleum ether and add that eluate to the evaporation vessel [8.8]