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**Karakterizacija odpadkov – Vsebnost halogena in žvepla – Zgorevanje s kisikom v zaprtem sistemu in metode za določevanje**

Characterization of waste - Halogen and sulfur content - Oxygen combustion in closed systems and determination methods

ICS 13.030.40

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# Characterization of waste - Halogen and sulfur content - Oxygen combustion in closed systems and determination methods

Caractérisation des déchets - Teneur en halogènes et en soufre - Combustion sous oxygène en système fermé et méthodes de dosage

Charakterisierung von Abfällen - Halogen- und Schwefelgehalt - Sauerstoffverbrennung in geschlossenen Systemen und Bestimmungsmethoden

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# Foreword

This document (prEN 14582:2005) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

This document is currently submitted to the second CEN Enquiry.

# Introduction

Sulphur and halogens (fluorine, chlorine, bromine and iodine) may be found in various forms in materials. During the combustion of these materials, corrosive and harmful compounds may be released. The determination of sulphur and halogens may be used for assessing the suitability of waste for incineration.

Oxygen combustion in a closed system is the preferred method used to waste samples for halogen and sulphur determination. Other preparatory techniques may be used, e.g. high furnace combustion, acid digestion, alkaline fusion, Wickbold oxy-hydrogen flame combustion, if equivalence with the method described can be proven.

The determination of the resultant halides and sulphate can be achieved by many different techniques, e.g. using atomic emission spectrometry, titrimetry, ion chromatography.

These methods do not lead to comparable results in all cases.

# 1 Scope

This document describes two combustion methods for the determination of halogen and sulphur contents in materials by combustion in a closed system containing oxygen, and the subsequent analysis of the combustion product using different analytical techniques.

These preparation procedures are:

— Method A — oxygen bomb combustion (calorimetric bomb);
— Method B —- oxygen flask combustion (Schöniger flask).

Method A is applicable to solid, pasty and liquid samples containing more than 0,025 g/kg of halogen and/or sulphur content.

Method B is about 10 times less sensitive.

For both methods, the limit of detection depends on the element and on the determination technique used.

Insoluble halides and sulphate present in the original sample or produced during the combustion step are not completely determined by these methods.

# 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.*

prEN 14899, *Characterization of waste — Sampling of waste materials — Framework for the preparation and application of a sampling plan.*

prEN 15002, *Characterization of waste — Preparation of test portions from the laboratory sample.*

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

EN ISO 10304-1, *Water quality — Determination of dissolved fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulphate ions, using liquid chromatography of ions — Part 1: Method for water with low contamination (ISO 10304-1:1992).*

EN ISO 10304-2, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 2: Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulphate in waste water (ISO 10304-2:1995).*

EN ISO 10304-3, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 3: Determination of chromate, iodide, sulphite, thiocyanate and thiosulphate (ISO 10304-3:1997).*

EN ISO 11885, *Water quality — Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885:1996).*

ISO 9280, *Water quality — Determination of sulphate — Gravimetric method using barium chloride*.

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

ISO 10359-1, *Water quality — Determination of fluoride — Part 1: Electrochemical probe method for potable and lightly polluted water.*

# 3 Terms and definitions

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

**3.1**
**sulphur content**
the sum of sulphur contained as organic and inorganic compounds which can be converted to sulphate by combustion and then absorbed or dissolved in aqueous solution

**3.2**
**halogen content**
the sum of halogens contained as organic and inorganic compounds which can be converted to halides (fluoride, chloride, bromide, iodide) by combustion and then absorbed or dissolved in aqueous solution

NOTE Be aware that the above definitions are valid for this empirical EN only and do not comply with scientific definitions of sulphur and halogen content.

# 4 Principle

## 4.1 General

The sample is oxidized by combustion in a closed system (a bomb containing oxygen under pressure for the method A and a heavy walled glass flask - Schöniger apparatus - filled with oxygen for the method B). Halogenated and sulphur containing compounds are converted respectively to fluoride, chloride, bromide, iodide and sulphate which are absorbed and/or dissolved in an absorption solution.

Several methods may be used for the determination of the halides and sulphate concentrations in the absorption solution.

## 4.2 Applicability

In general, method A is applicable for concentrations over 0,025 g/kg, depending on the element and on the determination technique. It may be used for aqueous samples or samples which burn with difficulty. This involves the use of a combustion enhancer.

The method B is faster and easier to perform It uses a smaller amount of sample therefore needs more attention on the homogenization. In general, it is applicable for concentrations over 0,25 g/kg, depending on the element and on the determination technique.

# 5 Interferences

There are no interferences in the combustion step described in the present standard but interferences may occur during the subsequent determination of sulphate and halides (see corresponding standards).

Insoluble halides and sulphate present in the original sample or produced during the combustion step are not completely determined by these methods.

# 6 Hazards

Hydrogen peroxide is very caustic: the operator shall wear goggles and gloves and shall work under a fumehood when handling this reagent. These two methods use a gas (oxygen) at high temperature and high pressure, precautions shall be taken by the operator.