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English Version

**Characterization of waste - Digestion of waste samples using
alkali-fusion techniques**

Caractérisation des déchets - Digestion d'échantillon de
déchets par Mise en solution par fusion alcaline - Guide de
bonnes pratiques pour la mise en solution par fusion - Les
différentes méthodes et protocoles existants

Aufschluss von Abfallproben mittels Alkalifusion

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Contents

Introduction.....	4
1 Scope.....	5
2 General information.....	5
2.1 Digestion of samples.....	5
2.2 Digestion by fusion.....	5
3 Fluxes.....	6
3.1 Alkaline fluxes.....	7
3.2 Acid fluxes.....	10
3.3 Oxidising fluxes.....	11
3.4 Reducing fluxes (alkaline fluxes + reducing agents, sulphides).....	12
3.5 Digestion by sintering.....	12
4 The crucibles.....	12
4.1 Platinum.....	12
4.2 Silver.....	13
4.3 Nickel.....	13
4.4 Vitreous carbon.....	13
4.5 Iron.....	13
4.6 Porcelain.....	13
5 Protocols currently used within industry.....	13
5.1 Analysis of 16 metallic elements in crushing residues [11].....	13
5.2 Determination of Si, Al, Fe, Mn, Mg, Cr, Ti, and F in slags [12].....	15
5.3 Fusion with carbonate or borate mixture [15].....	16
5.4 Fusion with lithium borate [14].....	17
5.5 Standards (non exhaustive list).....	17
5.6 Fluxes and their applications.....	18
6 Comparison of different digestion techniques.....	21
7 Conclusion.....	23
Bibliography.....	24

Foreword

This Technical Report (CEN/TR 15018:2005) has been prepared by Technical Committee CEN/TC 292 "Characterization of waste", the secretariat of which is held by NEN.

This Technical Report is the translation of the French guideline BP X 30-428 "Digestion by fusion – Good practice guide for digestion by fusion: the different existing methods and protocols" and adoption as a CEN/TR. It gives information about the digestion of the waste samples using alkali-fusion techniques.

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Introduction

EU regulations (e.g. hazardous waste, waste incineration, European waste catalogue) ask in many cases for the total content of certain elements. In the European landfill directive, knowledge of total composition is given as an example of waste property-based criteria and is part of the basic characterization of waste.

In these special cases the total content of certain elements has to be determined. The standard based on acid digestion of waste samples (EN 13656) is in almost all cases applicable. However for some elements or waste composed of very refractory matrix (e.g. silicates, carbides, oxides), or when some residue is left after acid digestion, alkali-fusion may be used to bring the waste sample completely into solution.

1 Scope

This Technical Report describes digestion methods for the determination of element contents of waste samples by using different alkali-fusion techniques.

2 General information

2.1 Digestion of samples

The determination of the elemental chemical composition of waste material includes a pretreatment of the sample comprising several stages:

- sampling for analysis (drying, crushing, homogenisation, sample reduction);
- digestion.

This last stage of digestion is essential because it allows to obtain a homogenous medium compatible with current analytical methods (atomic absorption spectrometry AAS, inductively coupled plasma and atomic emission spectrometry ICP/AES, inductively coupled plasma mass spectrometry ICP/MS, molecular absorption spectrometry MAS, X-ray fluorescence spectrometry XRF).

The diversity of the materials is such that this stage remains very complex and can give rise to major errors due mainly to:

- contamination of the sample by digestion reagents;
- incomplete digestion;
- loss of elements by adsorption onto the mineralization residue, onto the filter, or onto the walls of the mineralization reaction vessel;
- loss of elements by volatilisation (over and above those connected with drying and crushing);
- loss by reprecipitation in the form of hardly soluble salts.

Digestion is generally conducted in two stages. The attack, which consists in destroying the sample's organic matter and in dissolving the mineral residue by possibly modifying the specification by a very aggressive medium, followed by a dilution of the residue by a liquid allowing to obtain a homogeneous solution compatible with the subsequently implemented analytical techniques. Specific methods have been developed for volatile elements.

While numerous digestion methods exist, none is universal. The choice depends, on the one hand, on the nature of the sample (matrix type) and, on the other hand, on the sought after element(s) or on the targeted objective : determination of the total content or search for exogenous contaminants. Digestion can be performed by a wet (acid attack) or dry (fusion, calcination, combustion) technique.

The purpose of this code of good practices is to inventory those fusion methods which allow the mineralisation and digestion of waste for which acid attacks do not give satisfactory results.

2.2 Digestion by fusion

Fusion is often employed for the digestion of mineral materials (silicates, alumino-silicates, ...) and more particularly of certain refractory oxides (zircon, chromite, ...), but it is unsuitable for the digestion of volatile elements.