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Waste - State-of-the-art document - Halogens and sulfur by oxidative pyrohydrolytic combustion followed by ion chromatography detection

National foreword

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TECHNICAL REPORT

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English Version

**Waste - State-of-the-art document - Halogens and sulfur by
oxidative pyrohydrolytic combustion followed by ion
chromatography detection**

Caractérisation des déchets - État de l'art - Halogènes
et soufre par combustion pyrohydrolytique oxydative
suivie d'une détection par chromatographie ionique

Abfall - Dokument zum Stand der Technik -
Bestimmung von Halogenen und Schwefel mittels
oxidativer pyro-hydrolytischer Verbrennung mit
Ionenchromatographie Detektion

This Technical Report was approved by CEN on 4 February 2019. It has been drawn up by the Technical Committee CEN/TC 444.

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European foreword

This document (CEN/TR 17345:2019) has been prepared by Technical Committee CEN/TC 444 “Test methods for environmental characterization of solid matrices”, the secretariat of which is held by NEN.

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

Introduction

The content of sulfur, chlorine, fluorine and/or bromine has to be determined in various waste streams such as refuse derived fuel, rubber granulates, post-shredder residue and plastics from wastes of electrical and electronic equipment (WEEE).

At the moment the determination of these elements is performed according to EN 14582. This European standard specifies a combustion method for the determination of halogen and sulfur contents in materials by combustion in a closed system containing oxygen (calorimetric bomb), and the subsequent analysis of the combustion product using different analytical techniques. Because the combustion has to be conducted for each sample separately and no automation is possible, this method is time-consuming and labour-intensive compared to combustion ion chromatography (C-IC).

The use of the combustion ion chromatography (C-IC) instrument would allow in one single run the combustion of the material and the simultaneous determination of fluorine, chlorine, bromine, and sulfur by ion chromatography. Moreover, the combustion module enables the sample digestion of different type of samples under pyrolysis and oxidation conditions. The instrument may also be equipped with automatic sample introduction modules for solids and liquids, which will benefit the automation and reduce significantly the labour-intensive process. The system is already offered commercially by different manufacturers.

Many laboratories are using none coupled customized hydropyrolysis systems for different kind of applications. Offline systems can be used as sample preparation systems for IC measurement, too. Coupling is no requirement for using the C-IC technique.

This document provides a technical description of the C-IC technique, an overview of available commercial instruments, the strengths and limitations of this technique, and analytical results for halogens and sulfur obtained on waste samples.

1 Scope

In the framework of EU Directive 99/31/EC [1] and EU Directive 2000/76/EC [2] halogens and sulfur need to be determined on waste samples. The implementation of the combustion-IC technique would allow in one single run the combustion of the sample followed by the determination of the halogens and sulfur with ion chromatography. Moreover, this instrument may be provided with a sample carousel for both solids and liquids, allowing an automation of these type of analyses.

Recent developments of the C-IC technology have made this technique interesting for the determination of halogens and sulfur in waste samples. Therefore, a document on the current progress of the C-IC technology was prepared, including the evaluation of the performance of different commercially available systems and the presentation of analytical results obtained on certified reference materials and waste samples.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Description of the combustion-IC technique

4.1 Principle

Samples are introduced in the combustion tube using an automatic boat control device. First samples are thermally combusted under argon atmosphere, followed by a combustion at 800 °C to 1 100 °C with oxygen under pyrohydrolytic conditions. Sulfur in the samples converts to SO_x and halogens to hydrogen halide. These volatile compounds are trapped in an aqueous absorbing solution and subsequently injected for ion chromatographic analysis. The basic equipment configuration is shown in Figure 1.

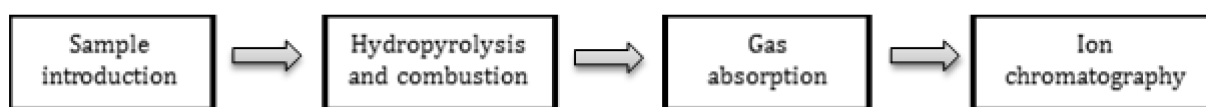


Figure 1 — Basic configuration of a C-IC system