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Standards

Standard Recommendation  
S.R. CEN/TR 16184:2011

# Characterization of Waste - State-of-the-art document - Analysis of eluates

## S.R. CEN/TR 16184:2011

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## Characterization of Waste - State-of-the-art document - Analysis of eluates

Caractérisation des déchets - Document de l'état de l'art -  
Analyse des éluats

Charakterisierung von Abfällen - Stand der Technik -  
Analyse der Eluate

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## **Foreword**

This document (CEN/TR 16184:2011) has been prepared by Technical Committee CEN/TC 292 "Characterization of waste", 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 [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

## Introduction

For determining the acceptability of waste at landfills, test methods are described in two umbrella European Standards:

- EN 12506, *Characterization of waste – Analysis of eluates – Determination of pH, As, Ba, Cd, Cl<sup>-</sup>, Co, Cr, Cr VI, Cu, Mo, Ni, NO<sub>2</sub><sup>-</sup>, Pb, total S, SO<sub>4</sub><sup>2-</sup>, V and Zn* (analysis of inorganic constituents of solid waste and/or its eluate; major, minor and trace elements);
- EN 13370, *Characterization of waste – Analysis of eluates – Determination of Ammonium, AOX, conductivity, Hg, phenol index, TOC, easily liberatable CN<sup>-</sup>, F* (analysis of inorganic constituents of solid waste and/or its eluate (anions)).

At the moment these standards are under revision and therefore this state-of-the-art document is prepared in order to verify the following items:

- Are all parameters mentioned in the Council Decision of 19 December 2002 included in these two European Standards?
- Should new relevant standards (e.g. EN ISO 17294 series) be included?
- Are the current analytical techniques capable of verifying the prescribed limit values with an acceptable confidence level?

In Clause 5, a proposal is given for the revision of the EN 12506 and EN 13370 umbrella standards.

The major changes between the umbrella standard EN 12506:2003 and the proposal for revision are:

- Addition of the parameters Sb and Se together with the related analytical methods;
- Revision of the standards ISO 11885 (ICP-AES) and ISO 10304-1 (IC) (the versions of ISO 10304-1:1992 and ISO 10304-2:1995 have been combined in one standard ISO 10304-1:2007);
- Addition of the ICP-MS method (EN ISO 17294-1:2006 and EN ISO 17294-2:2004);
- Addition of the AAS with graphite furnace technique (EN ISO 15586:2003);
- Addition of the flow analysis techniques for Cl<sup>-</sup> (ISO 15682:2000), Cr(VI) (ISO 23913:2006) and SO<sub>4</sub><sup>2-</sup> (ISO 22743:2006).

The major changes between the umbrella standard EN 13370:2003 and the proposal for revision are:

- Addition of the parameter TDS (total dissolved solids) together with the related analytical method;
- Replacement of the parameter TOC (total organic carbon) by DOC (dissolved organic carbon);
- Revision of the standards EN ISO 11732 (ammonium by flow analyser) and ISO 10304-1 (IC);
- Replacement of EN 1485 (AOX) by EN ISO 9562:2004;
- Revision and addition of new standards for Hg determination – EN 1483, EN ISO 17852 (and EN 12338);

- For the determination of  $F^-$  using the ion selective electrode it is recommended to use the buffer as described in DIN 38405-4 instead of the buffer as described in ISO 10359-1, considering the higher efficiency as decomplexing agent for Al, Fe, Mg and Ca-fluoride compounds.

With regard to the revised standards, there is no need for an additional validation round robin test when implementing the revised standards into the umbrella standards EN 12506 and EN 13370.

Regarding the additional standards, all of these have waste water and/or leachates included in their scope, meaning there are strong indications that the method is suitable for the analysis of eluates. Nevertheless, an additional validation of these standards for eluate matrices is required when implementing them into the umbrella standard, with the exception of:

- EN 12338:1998 (Hg – enrichment methods by amalgamation): There is no need to incorporate this standard into the umbrella standard EN 13370 because a measurement in this low concentration range is not required;
- EN 15126:2007 (TDS): As this parameter was validated for eluates in the EN 15126:2007, no additional validation is required when implementing this standard into the umbrella standard EN 13370.

For the determination of the elements Mo, Ba, Cr, Cd, Sb and Se in eluates validation data are available from a round robin test between 19 recognized laboratories (from Flanders – Belgium and the Netherlands) organized by VITO (see Clause 6). Based on these results it can be concluded that for the various elements using the ICP-AES technique according to ISO 11885 and the ICP-MS technique according to ISO 17294, there are enough validation data available to implement in the umbrella standard. No additional validation of the standard according to EN ISO/IEC 17025 is required. On the other hand, for the determination of Sb and Se using the hydride generation procedure or the graphite furnace technique, additional validation data according to the prescribed standards are required.

For all other parameters determined according to the new standards, additional validation data need to be gathered. Once a year (in March) VITO organizes a round robin test for the analysis of eluates in the framework of the acceptability of waste at landfills (see results of 2005, 2007 and 2008 in Clause 8). For this purpose, round robin samples are yearly prepared. Having these samples available, it may be interesting to distribute the same samples also to other European laboratories in order to validate the various parameters according to the new European Standards.

Additionally, from the laboratories there is an increasing interest to implement the discrete analyser technique in their laboratory to measure parameters such as ammonium, nitrite, chloride and sulfate in water samples. At the moment, no International (ISO) or European (EN) Standard method is available. Moreover, the Netherlands Standardization Institute (NEN) has developed and validated a standard method NEN 6604:2007 (Water – Determination of ammonium, nitrate, nitrite, chloride, ortho-phosphate, sulfate and silicate using a discrete analyzer and spectrophotometric detection). This method should also be taken into consideration when revising the standard.

The elements Hg and S are not included in the scope of the ICP-MS standard, however it is advisable to investigate the determination of Hg and S with ICP-MS. Therefore, additional validation is required.

## **1 Scope**

In the Council Decision of 19 December 2002 establishing criteria and procedures for the acceptance of waste at landfills pursuant to Article 16 of and Annex II to Directive 1999/31/EC, the test methods are described for determining the acceptability of waste at landfills. In section 3 of the annex of this decision the following umbrella European Standards are included for the analysis of eluates:

- ENV 12506 Analysis of eluates – Determination of pH, As, Ba, Cd, Cl, Co, Cr, Cr(VI), Cu, Mo, Ni, NO<sub>2</sub>, Pb, total S, SO<sub>4</sub>, V and Zn (analysis of inorganic constituents of solid waste and/or its eluate; major, minor and trace elements);
- ENV 13370 Analysis of eluates – Determination of ammonium, AOX, conductivity, Hg, phenol index, TOC, easily liberatable CN, F (analysis of inorganic constituents of solid waste and/or its eluate (anions)).

In 2003 both European Standards were approved and became final standards i.e. EN 12506 and EN 13370. At the moment these standards are under revision and therefore a state-of-the-art document is prepared in order to verify the following items:

- Are all parameters mentioned in the decision included in these two European Standards?
- Should new relevant standards (e.g. EN ISO 17294 series) be included?
- Are the current analytical techniques capable of verifying the prescribed limit values with an acceptable confidence level?

## **2 Normative references**

Not applicable.

## **3 Legislation**

In the Council Decision of 19 December 2002 limit values for waste acceptable at the landfills are defined. The limit values differ depending on the type of waste i.e.

- inert waste;
- non-hazardous waste;
- non-hazardous waste (stable, non-reactive);
- hazardous waste.

The limit values taken over from the Council Decision of 19 December 2002 are presented in Table 1.



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