

ENVIRONMENT AGENCY

**GUIDANCE ON SAMPLING AND TESTING OF
WASTES TO MEET LANDFILL WASTE
ACCEPTANCE PROCEDURES**

Version 1

April 2005

ACKNOWLEDGEMENTS

We are grateful for the considerable technical advice received from David Hall and his colleagues at Golder Associates (UK) Ltd. This document benefits from the responses from the previous consultation exercise and includes at least one new diagram from that source.

EXECUTIVE SUMMARY

This guidance has been produced prior to the finalisation of the Landfill (England and Wales) Regulations 2005 and as such will need to be reviewed to take into account any amendments to the Regulations once issued.

Whilst reviewing the document we shall also hope to improve its usefulness to all concerned and so would welcome feedback to assist us with the review.

The guidance has been produced for Environment Agency officers, waste producers and the waste management industry. It will assist in the development of sampling plans, in undertaking testing programmes for waste characterisation and in compliance testing and the use of the data to determine acceptance of granular and monolithic wastes to landfill. It has been prepared to:

- outline responsibilities in relation to waste testing for acceptance to landfill;
- provide background on the technical aspects of waste characterisation; and
- provide guidance on compliance measures for waste acceptance to landfill.

This document presents our guidance on sampling and testing of wastes to determine acceptance at landfill of both granular and monolithic wastes. It also details the proposed approach to determining compliance with the waste acceptance procedures.

The full landfill waste acceptance criteria for granular wastes are being implemented in the UK via the Landfill (England & Wales) Regulations 2002 and their subsequent amendments. The criteria for granular wastes are based on maximum limit values for eluate concentrations derived from a European standard batch leaching test and total or sum parameters for certain other determinands. The proposed landfill acceptance criteria for monolithic wastes have been published in the draft Landfill (England and Wales) Regulations 2005. These are based on limit values for cumulative leached concentrations from a diffusion leaching test as well as organic content of input wastes to the process that produces the waste form. The waste acceptance criteria will be introduced in July 2005 at landfills for hazardous waste and as part of the Pollution Prevention and Control (PPC) permitting process for other sites.

All waste producers must obtain characterisation data that, among other uses, will determine the destiny of each of their waste streams. First, there may be a need to determine whether the waste is hazardous or non-hazardous. Then, if it is to be landfilled, the class of landfill at which it can be accepted must be identified. With the exception of certain listed and one-off wastes, each waste will be tested periodically by the landfill operator to ensure that the waste complies with the waste acceptance criteria for that class of landfill.

This guidance is primarily aimed at the sampling and testing of wastes that are either generated on a regular basis and are reasonably consistent in quality, or, where separation at source can generate waste streams that are reasonably consistent.

The characterisation of wastes is required under The Environmental Protection (Duty of Care) Regulations 1991 (as amended) (for example for declaration of hazard properties) as well as for assessing waste acceptance to landfill. Sampling and testing can be time-consuming and expensive. The Agency recommends that every opportunity is taken to obtain additional value from the sampling exercise by including samples collected under average and worst-case operational conditions. The information on variation of waste characteristics might assist decisions about alternative treatment or disposal options. It can also be cost-effective to commit a high proportion of the required sampling and testing budget to an initial testing programme. This should ensure that reliable information is collected about the sources of variability in any waste stream before the sampling plan for a longer term testing programme is devised.

Testing is not always required if there is considerable relevant knowledge about the production process, and the technical expertise of the waste producer can be harnessed effectively to determine the characteristics of the waste. For many industries there is an opportunity for the trade association to co-ordinate a well-designed, stratified sampling programme so that information on plant-to-plant and within-plant variability in the composition and leaching behaviour of wastes can be obtained. A comprehensive dataset is likely to be very useful when negotiating with the landfill operator and the Agency.

An effective dialogue between the waste producer and landfill operator will be of great assistance in the operation of the waste acceptance procedures. Co-ordinated testing can reduce the analytical burden of both the waste producer (characterisation data) and landfill operator (compliance data). However, this may represent a significant change in culture as currently there may be a reluctance for the waste producer and landfill operator to exchange potentially commercially sensitive information.

The ultimate aims of the Landfill Directive - to reduce the volume and the hazardous nature of waste going to landfill - can be assisted by the characterisation exercise. Guidance is provided on leaching tests that can be used to demonstrate that leaching behaviour of wastes may be modified through waste treatment.

Since this guidance was produced in draft form, other guidance documents and regulations have been published relating to requirements for landfill pre-treatment and the waste producers' obligations to produce characterisation information. The reader is referred to these for additional information throughout the text.

This guidance provides clarification on a range of issues, the most significant of which are:

- **Sampling to confirm waste classification – hazardous or non-hazardous waste:** For wastes destined for landfill, the waste producer's obligations include undertaking a basic (fundamental) characterisation of the waste (in its treated state, if appropriate) prior to transport and disposal. Under Duty of Care, the European Waste Catalogue code, status as hazardous or non-hazardous waste and hazard properties of hazardous wastes must be declared. Representative samples should be collected for the hazard assessment and analysis of fundamental characteristics of the waste.
- **Sampling for testing against the landfill waste acceptance criteria:** As part of the sampling and testing programme, samples that represent worst-case as well as average conditions should be collected for testing against the appropriate landfill waste acceptance criteria. The dataset is needed to demonstrate that as a hazardous waste, or a stable, non-

reactive hazardous waste, or an inert waste, any load of waste meets the appropriate landfill waste acceptance criteria (WAC). The landfill WAC are maximum limits which must not be exceeded and should be viewed as treatment specifications for landfill. If the waste producer knows that a post-production treatment process generates off-specification residues from time to time, these episodes should be targeted as part of his own private compliance testing programme and the treatment operations improved. The waste producer should be confident that his wastes will always meet the landfill operator's waste acceptance testing at the agreed scale (see below).

- **Scale:** The scale defines the minimum quantity (mass or volume) of material below which variations in quality are judged to be unimportant. The scale for assessing waste against the landfill waste acceptance criteria has been set at the scale of the load at which it is accepted to landfill, be it a skip, roll-on-off or tipper truck. Representative sampling techniques will be needed to obtain a sample that represents the load.

We have also produced an overview of sampling and testing requirements in the document **“Requirements for the sampling and testing of wastes destined for landfill. A guide for waste producers and waste managers”** which is available on our web-site.

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GLOSSARY

Aqua regia	Hydrochloric/nitric acid, used hot to digest solid samples for determination of mineral acid soluble ‘total’ concentration.
Acid/base neutralisation capacity	The volume of acid or alkaline used to bring a waste to a specific pH value measured in mol acid per dry kg waste (mol/kg).
Availability for leaching	Potential leachability under worst case environmental conditions assessed using the maximum availability leaching test (EA NEN 7371) expressed in mg kg ⁻¹ dry residue leached.
BS EN 12457:2002	European standard leaching tests – compliance leaching test for granular wastes (Parts 1-3 are a series of batch tests for material at <4mm particle size, at L/S2, L/S10 or L/S2 and L/S2-10 l/kg).
CEN	Comité Européen de Normalisation (European Standards Organisation).
CEN TC292	CEN Technical Committee 292 (Characterisation of Waste)
Diffusion test ‘tank’ test	Leaching test conducted on monolithic samples in ‘tanks’ of deionised water, with or without pH control (e.g. EA NEN 7375 and CEN TC292/WG2 draft WI 292010).
Eluate	The solution obtained after leaching a solid material with a leachant during a standardised laboratory leaching test. (The term ‘leachate’ is reserved for a liquid collected in the field).
Granular	Granular waste is neither monolithic, liquid, gas or sludge (BS EN 12457). For the purposes of landfill acceptance, granular wastes include all wastes that are not monolithic ¹
L/S	Abbreviation for liquid-to-solid ratio prevailing during the leaching test, as quantity of liquid (‘L’) in litres and solid (‘S’) in kg dry matter. L/S is expressed in l kg ⁻¹ .
Leachant	Solute used in the leaching procedure.
Monolithic	The Agency considers that monolithic wastes will normally mean wastes that have been deliberately treated to solidify them. These requirements would apply to any monolithic material, be it cementaceous or otherwise, but provided that the waste is disposed of so that it forms large blocks or slabs and not as a simple mixed stabilised waste (e.g. as cement

¹ The Landfill (England and Wales) (Amendment) Regulations 2004.

	stabilised granular waste).
pH dependence tests	E.g. prCEN/TS 14429:2003 and prEN 14997:2004 leaching tests where separate samples of granular waste are subjected to pH controlled leaching between pH4 and 14 for 24 hours at L/S10.
Scale	The scale defines the minimum quantity (mass or volume) of material below which variations in quality are judged to be unimportant. The scale for assessing waste against the landfill waste acceptance criteria has been set at the scale of the load at which it is accepted to landfill, be it a skip, roll-on-off or tipper truck. Representative sampling techniques will be needed to obtain a sample that represents the load.
Upflow percolation test	E.g. prCEN/TC 14405:2003 a leaching test conducted in a column packed with granular waste, through which deionised water is pumped continuously and eluate removed at cumulative liquid to solid ratios of 0.1 to 10 l/kg.
Wastes in liquid form	For the purposes of landfill acceptance: <ul style="list-style-type: none"> - any waste that near instantaneously flows into an indentation void made in the surface of the waste, or - any waste load containing free draining liquid substance in excess of 250 litres or 10% of the load volume, whichever represents the lesser amount. ‘Free draining’ means a liquid as defined in (1) irrespective of whether that liquid is in a container.²

² DEFRA, 2004a. Government Interpretation of the Landfill (England and Wales) Regulations 2002 (As Amended), September 2004.

1. INTRODUCTION

1.1 Purpose

This guidance document has been prepared to:

- outline responsibilities in relation to waste testing for acceptance to landfill;
- provide background on the technical aspects of waste characterisation;
- provide guidance on compliance measures for waste acceptance to landfill as required by the European Council Decision 2003/33/EC³.

This guidance has been produced prior to the finalisation of the Landfill (England and Wales) Regulations 2005 and as such may need to be amended to take into account any amendments to the draft Regulations that are currently available.

We have also provided an overview of the sampling and testing requirements in our guidance document 'Requirements for the sampling and testing of wastes destined for landfill. A guide for waste producers and waste managers.'

1.2 Intended use

The guidance is for Environment Agency officers, waste producers and the waste management industry. It will assist the end user in developing sampling plans, in undertaking testing programmes for characterisation and in compliance testing and the use of the data to determine acceptance of granular and monolithic wastes to landfill.

1.3 Structure

Section 2 presents the levels of testing, as defined by the Landfill Directive and information requirements for waste characterisation, particularly for landfill acceptance.

Section 3 provides advice relevant to waste producers.

Section 4 provides advice relevant to landfill operators.

Most of the technical detail relating to sampling, testing and the specified analytical methods is presented in a series of Annexes.

³ EC Council Decision 2003/33/EC of 19th December 2002 establishing criteria and procedures for the acceptance of waste at landfills pursuant to Article 16 and Appendix II of Directive 1999/31/EEC on the landfill of waste. Official Journal of the European Communities L11 16.1.2003.

1.4 Definitions

The following broad definitions of waste producer are used throughout the report.

A primary waste producer refers to the producer of a waste that is sent directly to landfill.

A secondary waste producer⁴ treats the waste or changes its chemical characteristics in some way, before sending it to landfill. The secondary waste producer may be:

- the same company as the primary waste producer; or
- a third party treatment plant operator; or
- a treatment plant operator within the same waste management company as the landfill operator.

The distinction is made because it is the waste producer's responsibility to characterise a waste and the landfill operator's responsibility to only accept waste for which his landfill is permitted. Depending on where the landfill pre-treatment occurs, the primary or secondary waste producer must be able to demonstrate that the appropriate class of landfill has been selected for the treated waste.

Waste acceptance criteria have been developed for both granular and monolithic wastes. We consider that monolithic wastes will normally mean wastes that have been deliberately treated to solidify them. These requirements would apply to any monolithic material, be it cementaceous or otherwise, but provided that the waste is disposed of so that it forms large blocks or slabs and not as a simple mixed stabilised waste (e.g. as cement stabilised granular waste)⁵.

Granular wastes are considered to be all wastes that are not monolithic wastes.

⁴ The term is not to be confused with secondary material, a processed waste destined for reuse rather than disposal.

⁵ Environment Agency 2005a. Requirements for the acceptance of waste in landfill.

2. INFORMATION REQUIREMENTS AND APPROACHES

2.1 Hierarchy of testing for waste acceptance to landfill

Appendix II of the Landfill Directive states that:

“The general characterisation and testing of waste must be based upon the following three level hierarchy:

Level 1: Basic⁶ characterisation. This constitutes a thorough determination, according to standardised analysis and behaviour-testing methods, of the short and long-term leaching behaviour and/or characteristic properties of the waste.

Level 2: Compliance testing. This constitutes periodical testing by simpler standardised analysis and behaviour-testing methods to determine whether a waste complies with permit conditions and/or specific reference criteria. The tests focus on key variables and behaviour identified by basic characterisation.

Level 3: On-site verification. This constitutes rapid check methods to confirm that a waste is the same as that which has been subjected to compliance testing and that which is described in the accompanying documents. It may merely consist of a visual inspection of a load of waste before and after unloading at the landfill site.

In summary before a waste can be landfilled, all of the properties of the waste which determine whether it meets the general criteria at Schedule 1(5) of the Landfill Amendment Regulations⁷ must be known (Level 1). The waste should then be periodically checked (Level 2) to ensure that those properties have not changed, and it should be checked at the landfill (Level 3) to verify that it is the expected waste and has not been contaminated in storage or transport.

We expect that most wastes will require some testing to determine the relevant properties. The exceptions will be:

- wastes where a comprehensive dataset is already available from previous testing,
- wastes which are listed in Regulations or Agency guidance as not requiring testing, or
- wastes whose composition can be predicted to the satisfaction of the Agency from a knowledge of the process producing the waste.

In general, the responsibilities of the waste producer and landfill operator can be summarised as follows:

⁶ The term ‘basic’ as used in the Directive means ‘comprehensive’ or ‘full’. Hence, “this constitutes a thorough determination.....of leaching behaviour and/or characteristic properties...”

⁷ SI 1375:2004 The Landfill (England and Wales) (Amendment) Regulations..

- Level 1 testing is the responsibility of the waste producer; it should however be noted that the landfill operator should only accept waste that has been appropriately tested and characterised.
- Level 2 testing may be undertaken by both the producer and the landfill operator, but it is the responsibility of the landfill operator to ensure that he only accepts wastes that fulfil the waste acceptance criteria for his site; and
- Level 3 is the responsibility of the landfill operator.

Details of these relationships and responsibilities are provided in Sections 3 and 4.

The information obtained from a level 1 characterisation provides a sound basis for the provision of information to comply with Duty of Care requirements given in regulation 3 of the draft Landfill (England and Wales) Regulations, 2005 and to assess the opportunities for reuse, treatment or disposal for any waste (or secondary material) including testing for waste acceptance to landfill.

2.2 Information required for waste acceptance to landfill

Basic characterisation (level 1) is the first step in the acceptance procedure. Basic characterisation constitutes a **full** characterisation of the waste by gathering all the necessary information for the safe disposal of the waste in the long term. Basic characterisation is required for each type of waste.

The functions of basic characterisation are defined in SI 1375:2004, the Landfill (England & Wales) (Amendment) Regulations (that implement the European Council Decision 2003/33/EC³) as the following.

- a) Basic information on the waste (type⁸ and origin, composition, consistency, leachability and – where necessary and available - other characteristic properties).
- b) Basic information for understanding the behaviour of waste in landfills and options for treatment as laid out in Regulation 10(1) of the Landfill Regulations 2002.
- c) Assessing waste against limit values.
- d) Detection of key variables (critical parameters) for compliance testing and options for simplification of compliance testing (leading to a significant decrease of constituents to be measured, but only after demonstration of relevant information). Characterisation may deliver ratios between basic characterisation and results of simplified test procedures as well as frequency for compliance testing.

If the basic characterisation of a waste shows that the waste fulfils the criteria for a landfill class, the waste is deemed to be acceptable at this class of landfill. If this is not the case, the waste is not acceptable at this class of landfill.

⁸ For example, whether the waste is regularly or not regularly generated (see Section 3.2).

The producer of the waste, or in default, the person responsible for its management, is responsible for ensuring that the characterisation information is correct.

The information required for basic (full) characterisation is:

- a) *The source and origin of the waste;*
- b) *the process producing the waste (including a description of the process, its SIC Code and characteristics of its raw materials and products);*
- c) the waste treatment applied in compliance with Regulation 10(1) of the Landfill Regulations, 2002, or a statement of reasons why such treatment is not considered necessary;
- d) the composition of the waste including where relevant, an assessment of it against the relevant limit values in Part 3 of Schedule 1 of the Landfill Amendment Regulations 2004, and, where necessary and available, its other characteristic properties;
- e) *the appearance of the waste (including its smell, colour, consistency and physical form);*
- f) *the code applicable to the waste under the European Waste Catalogue⁹.*
- g) in the case of hazardous waste, the relevant hazard properties according to Appendix III of the Hazardous Waste Directive (91/689/EEC);
- h) *a demonstration that the waste is not prohibited by regulation 9;*
- i) the landfill class at which the waste may be accepted;
- j) the likely behaviour (including, where relevant, leaching behaviour) of the waste in a landfill and any additional precautions to be taken at the landfill;
- k) *whether the waste can be recycled or recovered;*
- l) identification of the key variables for compliance testing and options for simplification of compliance testing.

Where the waste is not being consigned to landfill the minimum information requirements which are likely to be provided¹⁰ by the (primary) producer are italicised ((a), (b), (e), (f), (h) and (k)). The remaining information ((c), (d), (g), (i), (j) and (l)) must be obtained by the final holder of the waste prior to consigning the waste to landfill, so that all information listed in (a) to (l) is supplied to the landfill operator before it is accepted at the landfill.

The guidance provided in this document is primarily aimed at the information required in (b), (d) and (i).

⁹ Council Decision 2000/532/EC as amended by Council Decisions 2001/118 EC, 2001/119 EC and 2001/573/EC.

¹⁰ Consultation draft of the Landfill Regulations (England and Wales) 2005.

2.3 Testing requirements

Most wastes will require sampling and testing to obtain information needed to determine the fate of a specific waste stream from a specific plant or location. The exceptions are those wastes specified in Section 2.1.

2.3.1 As a minimum, information will be needed to classify the waste as a hazardous or non-hazardous waste and to identify the class of landfill at which it may be disposed. As most hazardous wastes will require treatment prior to landfilling, this classification and assessment of acceptability will usually be on treated wastes. Waste classification

The Landfill Regulations (as amended) require the waste producer to classify his waste as hazardous or non-hazardous waste as part of the basic characterisation. This is provided for by:

- f) the code applicable to the waste under the European Waste Catalogue¹¹;
- g) in the case of hazardous waste, the relevant hazard properties according to Appendix III of the Hazardous Waste Directive (91/689/EEC).

NOTES: WASTE CLASSIFICATION

Wastes are classified as hazardous or non-hazardous wastes on the basis of:

(a) *European Waste Catalogue (EWC) coding.* The EWC (2002) lists wastes by industry sector. It defines wastes according to their known hazard characteristics, as:

- hazardous (absolute);
- mirror-entry (hazardous or non-hazardous depending on the presence of hazardous properties/dangerous substances);
- non-hazardous wastes.

Note: the EWC does not define inert wastes.

(b) *Hazard assessment.(remove bold)* The presence or absence of the 14 hazard properties listed in the Hazardous Waste Directive (91/689/EC) (e.g. H4 irritant, H6 toxic, H14 ecotoxic) defines a mirror-entry waste as hazardous or non-hazardous respectively. The hazard properties of an absolute hazardous waste and mirror-entry absolute waste must be declared prior to transport and disposal under Duty of Care.

The hazard assessment requires knowledge of the average quality of the waste with respect to the hazard properties.

Detailed guidance on the hazard assessment of wastes is provided by the Environment Agency (2003a)¹²

¹¹ Council Decision 2000/532/EC as amended by Council Decisions 2001/118 EC, 2001/119 EC and 2001/573/EC.

Classifying the waste as a hazardous or non-hazardous waste (see text box) may require testing to demonstrate the presence or absence of hazard properties¹² H1-H14 for Duty of Care purposes, or to determine reuse, treatment or disposal options.

2.3.2 Appropriate class of landfill

For those wastes requiring landfill disposal, the waste producer will need to show that the waste is destined for disposal at the correct class of landfill (see text box) as the basic characterisation obligations of the Landfill Regulations require identification of:

- h) the landfill class at which the waste may be accepted.

NOTES: LANDFILL CLASSIFICATION

Landfills are classified as follows:

- **Hazardous waste landfills** may only accept wastes that are classified as hazardous waste (according to EWC code and/or hazard assessment) and (from 16 July 2005) that meet the waste acceptance criteria for hazardous wastes.

- **Non-hazardous waste landfills** may accept wastes that are classified as non-hazardous wastes, either on the basis of their EWC code, or, in the case of mirror-entry wastes, demonstrated absence of hazard properties. A date of October 2007 has been proposed for the introduction of landfill pre-treatment requirements for non-hazardous wastes accepted at landfill permitted only for non-hazardous wastes.

- Cells for **stable, non-reactive hazardous waste** can be established within non-hazardous waste landfills. Any non-hazardous waste or stable, non-reactive hazardous waste accepted within that cell must comply with the landfill waste acceptance criteria for stable, non-reactive hazardous wastes.

Wastes meeting the inert landfill waste acceptance criteria can also be accepted at landfills for non-hazardous wastes.

- **Inert waste landfill** can accept inert wastes that are on a list of inert wastes and/or that meet the landfill waste acceptance criteria for inert wastes.

¹² Hazardous waste: interpretation and classification of hazardous waste. Technical Guidance WM2 (Environment Agency, 2003a)

2.4 Landfill waste acceptance criteria

In most cases, wastes due to be landfilled will require testing to demonstrate compliance with the appropriate waste acceptance criteria (WAC). The WAC have been set as maximum limit values which must not be exceeded and should be viewed as minimum treatment specifications for landfill. The sampling and testing programme should include samples that represent worst-case as well as average operational conditions for testing against the appropriate landfill waste acceptance criteria. The dataset is needed to demonstrate that the average concentration of any load of waste meets the appropriate limit values. If the waste producer knows that a post-production treatment process generates off-specification residues from time to time, these episodes should be targeted as part of his own compliance testing programme and the treatment operations improved, or those loads consigned to a different class of landfill. The waste producer should be confident that his wastes will always meet the landfill operator's waste acceptance testing at the agreed scale (see text box on 'scale').

NOTES: SCALE

The 'scale' is crucially important to defining a sampling programme. It defines the minimum quantity (mass or volume) of material below which variations in quality are judged to be unimportant. For example, if the scale is defined to be 'a 6m³ skip of waste', then variations in any characteristic of the waste within the volume of a skip are declared to be of no concern. This means that during characterisation of the waste, concentration and leaching data must be representative of average concentrations within a skip. Samples collected at a smaller scale would be expected to exhibit greater variability. They would need to be combined to produce a composite sample and thoroughly mixed before analysis.

It is important, therefore, that all involved parties agree the appropriate scale at the outset of any sampling and testing programme. For landfill acceptance compliance testing, it is the waste producer (or treatment plant operator) and the landfill operator that must agree the appropriate scale for the sampling programme. Any enforcement samples taken by the regulator should also be assessed at the same scale. In the example of a 6m³ skip, this would require collection of incremental samples from the full depth of the waste at a number of points in the skip. Alternatively the skip should be emptied and incremental samples taken throughout the waste.

Note: composites should not be created that are representative of a volume of waste that is greater than the agreed scale.

2.4.1 Landfill waste acceptance criteria for granular wastes

The Regulations state that granular waste can be considered to be any waste that is not monolithic waste.

Quantitative limit values have been set for regulating the chemical characteristics of granular wastes accepted into landfills for hazardous waste, cells within non-hazardous sites for stable non-reactive hazardous waste and inert waste landfills. WAC have not currently been set for non-hazardous waste landfills.

The parameters used as the basis for determining the class of landfill at which granular waste may be accepted are listed in Table 2.1. The background to the test methods and their use is given in Section 3.3 and Appendix C. The range of eluate determinands may also need to be expanded beyond the restricted suite listed.

Where landfill acceptance criteria cannot be met, information from leaching behaviour tests undertaken as part of the Level 1 characterisation, can help to make an informed decision about waste treatment that will allow landfill or other alternative management options. Leaching behaviour tests include the maximum availability leaching test, pH dependence test and upflow percolation test (Table 3.3 and Appendix C).

It is therefore recommended that, before effort is committed to the preparation of a sampling plan and the collection of a potentially large number of samples, additional sampling objectives should be developed.

Table 2.1 Landfill waste acceptance criteria for granular wastes

Parameter	Inert waste landfill	Stable non-reactive hazardous waste in non-hazardous landfill [¶]	Hazardous waste landfill
Parameters determined on the waste			
Total organic carbon (w/w %)	3%	5%	6%*
Loss on ignition			10%*
BTEX (mg kg ⁻¹)	6		
PCBs (7 congeners) (mg kg ⁻¹)	1		
Mineral oil C ₁₀ -C ₄₀ (mg kg ⁻¹)	500		
PAHs (x congeners) [^]	100 mg kg ⁻¹		
pH		>6	
Acid neutralisation capacity		To be evaluated	To be evaluated
Limit values (mg kg⁻¹) for compliance leaching test using BS EN 12457- 3 at L/S 10 l kg⁻¹			
As (arsenic)	0.5	2	25
Ba (barium)	20	100	300
Cd (cadmium)	0.04	1	5
Cr (chromium (total))	0.5	10	70
Cu (copper)	2	50	100
Hg (mercury)	0.01	0.2	2
Mo (molybdenum)	0.5	10	30
Ni (nickel)	0.4	10	40
Pb (lead)	0.5	10	50
Sb (antimony)	0.06	0.7	5
Se (selenium)	0.1	0.5	7
Zn (zinc)	4	50	200
Cl (chloride)	800	15,000	25,000
F (fluoride)	10	150	500
SO ₄ (sulphate)	1,000 [#]	20,000	50,000
Total dissolved solids (TDS) ⁺	4,000	60,000	100,000
Phenol index	1		
Dissolved organic carbon at own pH or pH7.5-8.0 [@]	500 [⊕]	800	1,000

¶ And non-hazardous wastes deposited in the same cell

* Either TOC or LOI must be used for hazardous wastes

** UK PAH limit values are being consulted upon (Draft Landfill Amendment Regulations 2005)

If an inert waste does not meet the SO₄ L/S10 limit, alternative limit values of 1500 mg l⁻¹ SO₄ at C₀ (initial eluate from the percolation test (prCEN/TS 14405:2003)) AND 6000 mg kg⁻¹ SO₄ at L/S10 (either from the percolation test or batch test BS EN 12457-3), can be used to demonstrate compliance with the acceptance criteria for inert wastes.

+ The values for TDS can be used instead of the values for Cl and SO₄.

@ DOC at pH 7.5-8.0 and L/S10 can be determined on eluate derived from a modified version of the pH dependence test, prCEN/TS 14429:2003, if the limit value at own pH (BS EN 12457 eluate) is not met.

⊕ In the case of soils, a higher TOC limit value may be permitted by the Environment Agency at an inert waste landfill, provided the DOC value of 500mg/kg is achieved at L/S 10 l/kg, either at the soil's own pH or at a pH value between 7.5 and 8.0.

From: Landfill Regulations 2002 as amended.

Total dissolved solids (TDS) is not a primary parameter. There is no requirement to meet the limit value for TDS unless the waste holder has opted to do so in preference to meeting the individual chloride and sulphate limits. It may be used as an optional replacement for chloride and sulphate leaching values, particularly if a waste is unable to comply with the chloride and sulphate limits individually but is able to meet the TDS values.

The Council Decision allows Member States to introduce more stringent protective measures than those established in the decision document and states that this could be of particular relevance to the limit values for cadmium and mercury. The UK did not agree to the leaching test criteria for cadmium and mercury for non-hazardous and hazardous waste sites and proposed lower limit values of 0.1 and 1 mg kg⁻¹ Cd and 0.02 and 0.4 mg kg⁻¹ Hg respectively. Following consultation of the Landfill (England and Wales) Amendment Regulations 2004, the EU limits for Cd and Hg have been applied, however, this may be reviewed in 2006.

2.4.2 Proposed Landfill waste acceptance criteria for monolithic waste

We consider that monolithic wastes will normally mean wastes that have been deliberately treated to solidify them. These requirements would apply to any monolithic material, be it cementaceous or otherwise, but provided that the waste is disposed of to form large blocks or slabs and not as a simple mixed stabilised waste (e.g. as cement stabilised granular waste).

Landfill WAC for monolithic wastes only apply to landfills accepting hazardous wastes and those accepting stable, non-reactive hazardous wastes.

More detailed guidance for monolithic waste treatment plant operators is provided in section 3.3.4. However, all producers and receivers of monolithic wastes need to be aware of the following proposed limit values for monolithic wastes accepted at landfills for stable, non-reactive hazardous and hazardous wastes.

- a) Limit values on input wastes to treatment process generating monolithic wastes.
- b) Limit values on cumulative release at 64 days from a diffusion test for the purposes of characterising the output of the treatment plant.
- c) Limit values on cumulative release at 4 days from a diffusion test for compliance purposes.

Proposed Limit values for input wastes to monolithic treatment process

Current legislative proposals will require that all wastes entering the plant producing the monolithic waste forms must meet the TOC or LOI limit values reproduced in Table 2.2.

Table 2.2 Limit values for TOC and LOI on for wastes entering the monolithic treatment plant

Parameter	Stable non-reactive hazardous waste in non-hazardous landfills	Hazardous waste in hazardous landfills
LOI ¹³ (Loss on ignition)	10%	10%
TOC (Total organic carbon)	6%	6%

Proposed Limit values on outputs from monolithic treatment process

Quantitative limit values have been proposed for regulating the chemical characteristics of monolithic wastes accepted into landfills for hazardous waste and stable non-reactive hazardous waste.

The parameters used as the basis for determining the class of landfill at which the monolithic waste may be accepted are listed in Table 2.3. The background to the test methods and their use is given in Table 3.3 and Appendix C.

The same diffusion (tank) test is used for both characterisation and compliance. For characterisation, the full 64 day tank test is followed and cumulative leaching at 4 days and 64 days used to assess short and long term leaching behaviour. As a routine compliance test, cumulative leaching over the first 4 days is required to be below the 4-day cumulative limit values. Further information is provided in Sections 3.3.4, 4.3.4 and Appendix C.

¹³ Either Loss on Ignition or Total Organic Carbon

Table 2.3 Proposed UK landfill waste acceptance criteria for the leachability of monolithic wastes⁽¹⁾

	Stable non-reactive hazardous waste in non-hazardous landfill and non-hazardous waste in same cell		Hazardous waste landfill	
Parameter	Cumulative limit values (mg m⁻²)			
	For compliance (cumulative 4 day leaching)	For characterisation (cumulative 64 day leaching)	For compliance (cumulative 4 day leaching)	For characterisation (64 day leaching)
As (arsenic)	0.325	1.3	5	20
Ba (barium)	11.25	45	37.5	150
Cd (cadmium)	0.05	0.2	0.25	1.0
Cr (chromium total)	1.25	5	6.25	25
Cu (copper)	11.25	45	15	60
Hg (mercury)	0.025	0.1	0.1	0.4
Mo (molybdenum)	1.75	7	5	20
Ni (nickel)	1.5	6	3.75	15
Pb (lead)	1.5	6	5	20
Sb (antimony)	0.075	0.3	0.625	2.5
Se (selenium)	0.1	0.4	1.25	5
Zn (zinc)	7.5	30	25	100
Cl ⁻ (chloride)	2500	10,000	5000	20,000
F ⁻ (fluoride)	15	60	50	200
SO ₄ ²⁻ (sulphate)	2500	10,000	5000	20,000
DOC (Dissolved Organic Carbon)	must be determined	must be determined	must be determined	must be determined
pH	must be determined	must be determined	must be determined	must be determined
Electrical Conductivity (μS.cm ⁻¹ .m ⁻²)	must be determined	must be determined	must be determined	must be determined

(1) ESART 2004 and draft Landfill (England and Wales) Regulations 2005

2.5 Approach to information gathering

2.5.1 The role of waste producers and landfill operators

Waste producer

The role of the waste producer in the characterisation of waste has specified in the draft Landfill (England and Wales) Regulations 2005.

Waste producers will be seeking a balance between (a) their outlay in effort and costs associated with sampling and testing of their wastes, and (b) negotiations and costs associated with securing their favoured landfill disposal route. The two will be directly linked in cases where waste management charges are increased to incorporate a waste sampling and testing service. In all cases the waste producer will be seeking minimum disruption to his operations.

For producers who are undertaking in-house landfill pre-treatment of their residues, there is a clear benefit from investing in the collection of a reliable dataset well ahead of the introduction of full EU landfill waste acceptance criteria for hazardous wastes in July 2005, particularly if this increases understanding of the extent and causes of variability in waste quality. For example, where wastes are border-line cases for waste classification, or hazardous WAC are exceeded, there is still time for modifications to be made to production or residue treatment processes and for further testing to be conducted. In some cases alternative treatment or disposal routes may be required. Testing will not be required in all cases, particularly where the technical knowledge of the process and its impact on waste quality is well known or an historical dataset can be demonstrated to be relevant and reliable.

However, a waste producer with little knowledge of the characteristics of his waste, who has invested in only the bare minimum of testing, may be faced with limited or no landfill disposal options, should routine compliance monitoring conducted by the landfill operator indicate even occasional failure. For example, suppose the producer of a granular waste undertakes the batch leaching test, pH and TOC analysis for just one or two samples. Comparison of the leached metal levels with the waste acceptance criteria limit values may suggest that the waste is acceptable at a landfill for hazardous waste. But with no knowledge of the variability of the waste, he may be caught unawares when a routine compliance test undertaken by the landfill operator demonstrates failure, showing that the waste is not acceptable at any landfill for hazardous waste without further treatment to reduce leachability and/or organic carbon content. Alternatively the landfill operator might take the view that the Level 1 data are inadequate before this stage has been reached and refuse to accept the waste at his landfill.

Landfill operator

The landfill operator will seek to minimise the disruptions associated with non-compliance with waste acceptance procedures, such as enforcement action. He will also want to avoid renegotiating the disposal contract to utilise a higher class of landfill site, or losing a customer where he has no suitable site. The landfill operator will be attracted to demonstrably consistent or well-characterised wastes that are not borderline classification cases. He may adjust disposal charges accordingly,

particularly where the waste management service includes a sampling and testing element.

2.5.2 Partnership

Both the waste producer and landfill operator will be looking for a sound but practicable approach to reducing the risks of non-compliance associated with inadequate knowledge of waste variability. They will also want to limit the burden of sampling and testing. A partnership approach between the two parties that encourages free exchange of data on the characteristics of the waste (whilst recognising commercial sensitivities) will assist both to meet their goals.

For some parts of the waste management industry this may require some modification of current practices. However, for some waste producers, the very concept of regular sampling and testing of waste materials will itself require a significant change.

For both parties the exercise is made easier in the context of reliable, publicly available data on generic waste streams. Ultimately there is a great opportunity for the trade associations of producers of generic industrial wastes such as foundry sands, incineration residues, coal-fired power generation residues or water utility waste streams. Although the waste from each plant may have a specific chemical characteristic 'fingerprint', related to feedstock and operational conditions, the overall characteristics may be broadly similar and hence the waste streams could be described as 'generic'. The Agency recommends that the trade body co-ordinates a structured programme of testing according to the principles of stratified sampling (see Appendix B1) to establish the full range of waste characteristics. If the waste from a particular process can be shown to be broadly the same, regardless of the plant, then it may be possible to rely on level 2 monitoring data rather than needing level 1 characterisation data for each plant. Information on process-related factors controlling waste variability should be pooled. As a participating member sets up a new plant, initial data from the waste can be set against the larger dataset. The costs of assessing the leaching behaviour and other waste characteristics beyond those required for waste acceptance to landfill can also be shared, generating information which can be used as source-term data for risk assessments or to assess treatment or alternative disposal options.

With an authoritative body of relevant information available, the trade body may be able to negotiate inclusion of the waste on a list of wastes requiring minimum testing, set up on either a national or a site-specific basis (such as the inert wastes listed in the Landfill Regulations 2002, as amended) or a site-based list of wastes, specific to a particular landfill.

3. WASTE PRODUCER

3.1 Preamble

This section lists the information that is required to determine the correct class of landfill for a waste, and provides guidance on the sampling and testing needed to produce that information.

The primary waste producer is defined as the producer of a waste that is being sent directly to landfill without further treatment. In general, the volume and types of wastes that can be landfilled without pre-treatment will significantly decline. The exceptions are those wastes for which treatment is not feasible or where treatment would not reduce its quantity or hazardousness (Defra 2004a).

Where the waste producer is unable to meet the treatment/acceptance criteria for landfilling, further treatment will be required. For the purposes of this guidance, this treatment stage is conducted by secondary waste producers, who are either:

- the same company as the primary waste producer (in-house treatment plant on- or off-site), or
- an external waste management company, which may or may not also operate the landfill at which the waste is finally accepted.

In some cases characterisation data collected by the primary waste producer for Duty of Care purposes may also have been suitable to determine acceptance for landfill. Where treatment has changed the wastes characteristics, and the secondary waste producer wishes to consign the waste for disposal at a landfill for inert or hazardous waste, he will have to test the treated waste against the landfill WAC to determine suitability for landfill.

The waste producer is responsible for ensuring the accuracy of any characterisation data. He may commission independent sampling and testing of his wastes or pay his waste disposal contractor to undertake it as part of his waste management service. The testing of the waste may be carried out by the waste producer or landfill operator if they have set up an appropriate quality assurance system including periodic independent checking. Further information is provided in Appendix C. The waste producer should have unrivalled technical knowledge about the impact of external and operational factors on the quality of his waste streams, and this must be harnessed in the design of any sampling programmes and the review of any compositional or leaching data.

The sequence of events that should be undertaken by the waste producer is summarised as a flow-chart (Figure 3.1).

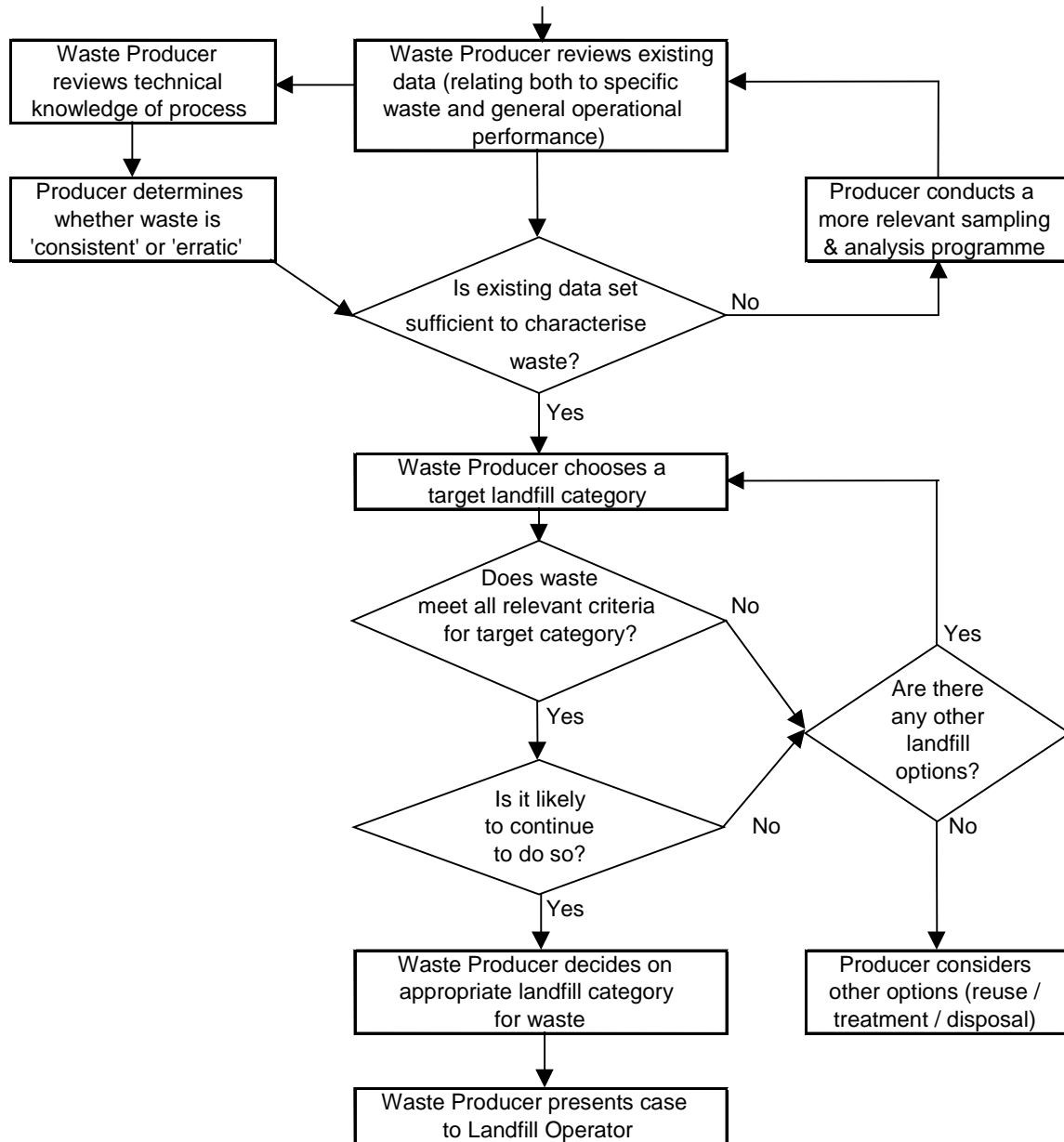


Figure 3.1 Generic flow-chart for a waste producer

Note: In general, the decision process will relate to treated wastes.

3.2 Requirements for basic characterisation and compliance testing

As discussed in Section 2.1 above, the characterisation and testing of waste must be based on a three-level hierarchy of characterisation, compliance testing and on-site verification. The Landfill Decision document 2003/33/EC⁽³⁾ explains that a general exception to the requirement to conduct compliance testing is when the waste is one that is not regularly generated.

3.2.1 Regularly Generated Waste

It is unlikely that the whole population of a regularly generated waste will be available at one time for characterisation, as the process will probably be producing waste over a considerable period. Such wastes can be divided into two categories for the purposes of testing as follows.

- The waste generation process(es) **and** input materials are both consistent and well defined. The waste(s) may be from a single installation or different installations that produce a stream with common characteristics and known boundaries that is also consistent, (e.g. bottom ash from the incineration of municipal waste). Characterisation must include the range and variability of the characteristic properties of the waste. The waste can then be considered to be characterised and shall subsequently be subject to compliance testing only, unless significant changes in the generation process occur.
- The waste is regularly generated by the same process and the generic input streams are the same but, either the inputs are more variable and/or a variable process recipe both yield an end-product of less consistent quality. In this case, characterisation must specifically address the expected variability of the waste stream over time. This scenario is relevant to facilities where a number of waste streams are bulked/ mixed or treated by a third party (e.g. merchant treatment plant, aggregate recovery plant or waste transfer plant).

The waste producer's requirements for testing regularly generated wastes are described in Sections 3.3 - 3.5 of this guidance. For these wastes, characterisation will comprise compositional analysis, determination of characteristic properties and leachability tests. The dataset should be sufficient to enable the variability of those characteristics to be assessed and in particular to demonstrate that the waste is consistently below the appropriate landfill waste acceptance criteria. Compliance, commonly limited to a restricted list of key variables, should then be undertaken at regular intervals by the waste producer to ensure the process remains within the identified boundaries.

3.2.2 Non-regular Wastes

Where it is not possible to characterise the waste stream over time, such waste should be regarded as 'non-regular'.

The test for the decision about the need for compliance testing is whether the whole population of a waste is available for sampling and testing for basic characterisation. If the whole of the waste has been subjected to basic characterisation directly before transfer to the landfill, there is probably no need to conduct compliance testing, even if the waste arrives at the landfill in several loads over several days. In all other cases, compliance testing is required.

The waste should then be divided into sub-populations (batches) which can be characterised and the guidance in Section 3.3.2 should be applied. Examples include building materials from site demolition, or soils recovered from a site excavation.

a) Entire population available for characterisation

For example, during a site redevelopment, the waste materials have been stockpiled as separate streams rather than being consigned immediately for landfill. Of the separate C&D streams, the 'clean' soils and contaminated soils are due to be landfilled as non-hazardous wastes and hazardous wastes respectively. The whole population of contaminated soil is available for sampling and can be

characterised (including hazard assessment and suitability for acceptance at a landfill for hazardous waste). A probabilistic approach in terms of numbers, sizes and sample locations could be taken to sampling the stockpiles. To ensure characterisation is undertaken at the same scale as the compliance sampling by the landfill operator the entire population of waste should be considered as being a series of loads (of a size that is consistent with the intended disposal method to landfill) which would then be subjected to a stratified or systematic sampling programme. Once the waste was consigned to landfill (as a series of loads) the landfill operator will undertake compliance sampling as for a regularly generated waste.

Alternatively, where the wastes are still *in situ* it is possible to use a well-designed site investigation as the characterisation exercise, assuming an effective 3D sampling grid can be undertaken on a probabilistic basis. The SI report will be the characterisation report, assuming all the required information (section 2.2) is provided. As in the example sited above the site could be divided into a sampling grid, whose units each represent a potential load to the landfill, to ensure comparability between the characterisation programme and landfill operator compliance scheme.

b) Entire population not available for characterisation

Sometimes only a sub-population of the total population that is due to be consigned to landfill is available for sampling and testing. For example, only part of a site has been demolished/excavated or a waste collector/treatment plant operator is handling/ treating differing generic input wastes in any given week or month.

There are two potential testing routes, either:

- Undertake a comprehensive characterisation exercise and repeat this as new material is generated, or (in the case of a process) when it is known or suspected that the waste characteristics are likely to have changed. This approach effectively divides the waste into a number of one-off wastes (each requiring characterisation but not compliance testing), OR;
- Undertake characterisation when knowledge of the activity indicates that sampling will encompass the expected variability in the waste stream, then undertake regular compliance checks over the period of the process or activity to check that the waste stream is within the ranges previously determined. Characterisation would be for a comprehensive determinand list (e.g. WAC determinands) to identify the key variables and concentration ranges that might be expected. Even where prior use of the site indicated that a sole contaminant might be expected, initial compositional and leaching tests would be needed to confirm this to be the case.

There will therefore be a need to undertake compliance testing on non-regularly generated wastes, where it is not possible to ‘characterise’ the entire population or consignment of waste requiring disposal.

3.3 Level 1 – basic characterisation

3.3.1 Review of existing information

In practice, waste producers will have varying levels of technical knowledge about their production processes and the factors that cause variation in their wastes. They will also have varying amounts of data on the composition or leaching behaviour of the wastes. The four extreme combinations of these two key inputs to the Level 1 characterisation process are summarised in Figure 3.2. It can be seen from the discussion boxes that, although both inputs are important, the most critical element is *process knowledge*. Given a sound understanding of the process, it is relatively straightforward to decide what sampling needs to be done. But without that knowledge, even a substantial amount of data is not sufficient in itself to give full assurance that the waste has been assigned to the correct class of landfill.

Historical data may have been obtained on the waste material for many reasons. These may include quality control, reprocessing of off-specification products, potential for reuse as an (engineered) product, waste disposal, environmental or human health risk assessments, or site investigations.

Some of the data may be relevant to acceptance at landfill and some may provide general information on the variability of the waste. Some historic data will be of limited use if key waste characteristics have been fundamentally changed through landfill pre-treatment processes. Data evaluation will need to focus on post-treatment samples.

3.3.2 Information requirements

Section 2.2 lists the types of information required to be collected as part of a basic characterisation for a range of purposes, including requirements under Duty of Care, as well as landfill acceptance. It is possible that different aspects of waste characterisation may be undertaken by different parties, particularly where the primary waste producer does not undertake the pre-landfill treatment.

A review should be conducted to seek any information or data indicating the degree of consistency of the waste, and in particular:

b) the process producing the waste (including description of the process, its SIC code and characteristics of its raw materials and products).

d) the composition of the waste, including where relevant, an assessment of it against the relevant limit values in Part 3 of the Landfill (England and Wales) (Amendment) Regulations, 2004.

Key to the latter is knowledge about the extent and causes of variability in the composition and leaching behaviour of the waste that could compromise predictions regarding acceptance to a particular class of landfill, particularly for borderline cases.

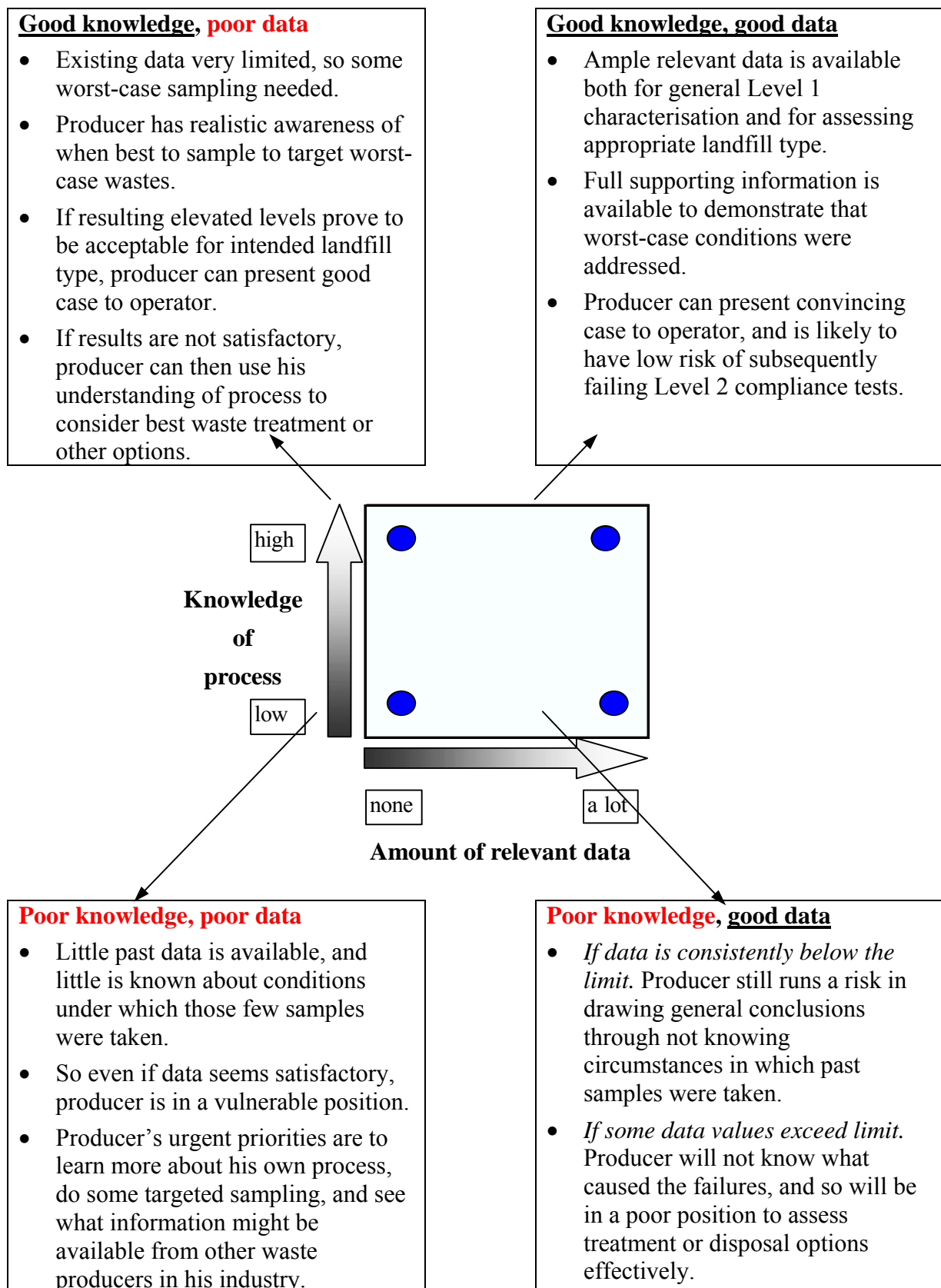


Figure 3.2 Possible level 1 starting points for the waste producer

Variability may be introduced by many factors, including:

- types, or variations in the quality of, feedstock, or other process inputs;
- cyclical factors;
- operational conditions (e.g. plant start up /shut down, changes between shifts); and
- scale (see Section 2.4.1). For example, spatula sized samples of incinerator bottom ash could show extreme variability whereas the average concentration of skip loads of waste would be more consistent.

Information of this sort is a valuable input to the process of designing a suitable sampling plan. The more that is reliably known about the effect of such factors on waste variability, the smaller is the amount of new sampling likely to be needed.

Existing information can be used to assess other options higher up the waste hierarchy such as energy recovery and reuse. Hence the review of existing information should not be limited to parameters for which there are limit values for waste acceptance to landfill. In some cases supplementary testing may be required to fill the gaps. In most cases dialogue with the landfill operator will indicate where the pertinent gaps are.

The treatment process owner's approach to information gathering is the same as that of the primary waste producer. Firstly, he needs to assess the quality and quantity of existing data (see Figure 3.2). Where the gross characteristics and leaching behaviour have been modified, a Level 1 characterisation should be carried out on the product of the treatment process. New test data will need to be compared with limit values for landfill acceptance. In addition, the inherent variability of the process will need to be established with respect to process inputs and operational conditions in order to determine whether the waste is of consistent or erratic quality (See Figure 4.3).

3.3.3 Sampling

The principal aim of Level 1 characterisation is to provide (for all relevant parameters) a reliable overall description of the waste, including for example the mean, the standard deviation, and any major changes or trends through time (with as much understanding as possible of the factors causing these). For example, such information would be needed to carry out a risk assessment.

As noted in Section 3.3.2, the fullest possible use should be made of existing information so as to minimise the amount of additional sampling required. Indeed, at one extreme so much may be known about the homogeneity of the waste that an assessment of the leaching behaviour of a few samples may be sufficient to characterise it adequately. At the other extreme - and especially for critical wastes - it may be necessary to carry out a comprehensive representative sampling programme. This would cover each waste type and all major sources of variation over time, and tests would be undertaken for all parameters likely to be important. In many cases, a phased characterisation programme will be needed that could extend over several months.

In general, therefore, the type of sampling carried out depends on knowledge of the waste, the objective(s) of the exercise, and the desired precision and confidence to be achieved. Ensuring that all these aspects are explicitly addressed before sampling is one of the key benefits of having a sampling plan. The preparation of a sampling plan will become mandatory when the full waste

acceptance procedures take effect (from 16th July 2005). Background to the development of a sampling plan is presented in Appendix A, drawn from the draft European sampling standard prEN 14899:2004 (CEN 2004a). Essentially it provides a shop-shelf approach to conducting a sampling exercise, with supporting technical reports providing examples of best-practice rather than prescriptive techniques for all aspects of sampling. Due to site-specific and waste-specific complexities, it is not possible to prepare a single sampling plan for landfill waste acceptance for all wastes. However, ESART (2004) published best practice guidance on the application of sampling plans to waste testing and provided examples of sampling plans for several waste sampling scenarios which illustrate the approach that can be taken.

Importantly, the sampling plan requirements should be related to scale (see textbox in Section 2.4). The waste producer, landfill operator (and regulator in the case of any enforcement samples) should agree the appropriate scale before undertaking a sampling programme.

For waste acceptance to landfill, the scale of sampling has been set at the scale of the load (whether a skip or single rail freight container). Representative sampling¹⁴ should be undertaken of that load to avoid over-reliance on analyses of contamination hot-spots picked up at a finer scale that do not represent the average concentration. As the WAC are maximum limits the producer should also include loads of wastes which are expected to represent worst-case operational conditions at the plant. If the average concentration of incremental samples from a worst-case load is compliant with the WAC, then it is likely that the average concentration of incremental samples of any waste load will generally also be WAC compliant under normal conditions. This may not always be the case and should be tested for all WAC parameters.

By comparison, for a waste hazard assessment, information on the average waste characteristics is required to confirm whether it exhibits any hazard properties. Results from worst-case loads would be included in the average results for that waste for comparison with relevant hazard thresholds.

In both scenarios the average concentration relating to any one load is compared with the appropriate WAC or hazard threshold. The maximum value from any incremental sample within the load would not be considered in isolation.

3.3.4 Testing

Overview

This section addresses the testing required to satisfy one of the information requirements of the waste acceptance procedures:

(d) the composition of the waste, including where relevant, an assessment of it against the relevant limit values in Part 3 of the Landfill (England and Wales) Regulations, 2002, as amended.

Interpretation of compositional information may be required to determine whether or not a waste is

¹⁴. Examples might include combining incremental samples obtained either by coring through the entire waste depth, or grab-samples obtained over the entire period of discharge/unloading of the container. Guidance on sampling techniques is provided in TR2 a supporting technical report to prEN 14899 (see Appendix A).

hazardous (Section 2.3.1.) After the waste classification has been established and the appropriate management option has been identified as landfilling as an inert, non-hazardous or hazardous waste, a producer's characterisation programme will be driven by the need to demonstrate that the appropriate class of landfill has been identified for that waste. Although the landfill operator must demonstrate that the waste meets the appropriate landfill waste acceptance criteria, the producer must also be satisfied that his wastes meet them before they are consigned to landfill.

Granular wastes

The minimum testing requirements to determine the acceptance of granular wastes to landfill are presented in Table 3.1. These cover a range of inorganic determinands in eluates derived from the European standard leaching test BS EN 12457:2002, and several specific parameters determined on the waste itself such as pH, acid neutralisation capacity and total organic carbon. Specified test methods of waste analysis are outlined below and in Appendix C. Sufficient samples should be tested to provide information on variability including the worst-case operational position and in sufficient quantity for all the tests that may undertaken.

In addition to ensuring that the waste will be acceptable for the chosen class of landfill, other characterisation tests, for example, leaching behaviour tests may also be required (see later subsection).

Monolithic wastes

The minimum testing requirements proposed to determine the acceptance of monolithic wastes to landfill are presented in Table 3.2. Details of the leaching behaviour tests cited are provided in Table 3.3.

Where the monolithic wastes are being produced at a treatment plant, the plant operator must determine the leaching behaviour of key variables in the output wastes and monitor the quality of input wastes to the plant.

- *Input wastes:* the treatment plant operator would be advised to obtain sufficient characterisation data (preferably from the waste producer) for each of the wastes entering his plant, particularly with respect to variability in composition and leachability. He should aim to obtain characterisation data for waste streams produced under normal and worst-case operational conditions in order to ensure that his product recipe is robust enough to generate a monolithic waste that is a consistent WAC-compliant product for landfilling.

The wastes entering the plant producing the monolith must comply with the either the TOC or LOI values given in Table 2.2.

Table 3.1 Minimum data requirements for determining granular waste acceptance to landfill⁽¹⁾

Parameters ⁽²⁾	Class of landfill			
	Inert waste	Non-hazardous waste	Cells for 'stable, non-reactive hazardous wastes' within landfills for non-hazardous waste ⁽³⁾	Hazardous waste
BS EN 12457-3 eluate determinations in mg/kg at L/S10 for As, Ba, Cd, Cr (total), Cu, Hg, Mo, Ni, Pb, Sb, Se, Zn, Cl, F, SO ₄ , total dissolved solids (TDS), phenol index, dissolved organic carbon (DOC) (at own pH or pH 7), pH ⁽⁴⁾	√		√	√
pH			√	√
Acid neutralisation capacity (ANC)			√	√
Total organic carbon (TOC)	√		√	√ (or LOI)
Loss on ignition (LOI)				√ (or TOC)
Benzene, toluene, ethylene, xylene (BTEX) compounds	√			
Polychlorinated biphenyls (PCBs 7 congeners)	√			
Polyaromatic hydrocarbons (PAHs)	√			
Mineral oil/petroleum hydrocarbons (C10 to C40)	√			

Notes

- (1) Determining waste acceptance to landfill is only a sub-set of testing that may be required for basic characterisation. For example the waste may have required prior testing to determine its status as a hazardous or non-hazardous waste and leaching behaviour tests may also have been undertaken.
- (2) Other leaching behaviour tests may be required – for example availability, pH dependence, upflow percolation. In many cases the information can assist the producer in identifying potential pre-landfill treatment options.
- (3) And non-hazardous waste deposited in the same cell
- (4) Certain parameters can be used interchangeably for compliance purposes (see Section 2.4.1)

Table 3.2 Minimum data requirements for determining monolithic waste acceptance to landfill⁽¹⁾

Parameters	Cells for 'stable, non-reactive hazardous wastes' within non-hazardous waste landfills ⁽²⁾	Landfill for Hazardous waste
<p>EA NEN 7375:2004 (Agency translation of the Dutch 'tank' test) cumulative eluate concentrations in mg/m² over the 8 stages of the test (64 days) and from the first 4 stages of the test (4 days). Cumulative limit values for: As, Ba, Cd, Cr, Cu, Hg, Mo, Ni, Pb, Sb, Se, Zn, Cl, F and SO₄ in mg m⁻².</p> <p>Eluate DOC concentration, pH and EC should also be determined.</p>	√	√
<p>On the ground monolith:</p> <ul style="list-style-type: none"> - maximum availability for leaching (EA NEN 7371:2004 – Agency translation of the Dutch availability test) for WAC determinands and other key variables - pH dependence leaching test (prCEN/TS 14429:2003 or prEN 14997:2004) and with analysis of WAC determinands or other key variables in the eluate plus - calculation of ANC/BNC. 	√	√
<p>Total organic carbon (TOC) and/or loss on ignition (LOI) limits for input wastes to the treatment plant producing the monolith</p>	√	√

- (1) Determining waste acceptance to landfill is only a sub-set of testing that may be required for basic characterisation. For example the waste may have required prior testing to determine its status as a hazardous or non-hazardous waste
- (2) And non-hazardous waste deposited in the same cell

- *Output wastes*: as part of the characterisation of the outputs from the treatment plant should be subjected to the following tests:
 - 64-day EA NEN 7375:2004 tank test for monolithic waste. The 64-day tank test quantifies long term diffusive leaching from the stabilised waste product. Cumulative data from the full test should meet full 64 day leaching limit values for monolithic wastes given in Table 2.3. If a cementaceous binder is used the test should be conducted on waste forms that have cured for 28 days, as this test also serves to indicate the longevity of the waste form. The full 8 stage 64-day test enables the demonstration that the emission follows a solely diffusive form and not an advective/ fully solubility controlled form similar to granular wastes. Cumulative leaching at 4 days should also be compared with the 4 day limit values presented in Table 2.3.
 - Maximum availability for leaching (EA NEN 7371:2004) and pH dependent leaching (prCEN/TS 14429:2003 or prEN 14997:2004) (see Table 3.3) and calculation of ANC/BNC (see ‘composition/general characteristics’ sub-section below).

Quality assurance

Before the sampling and testing programme is started, quality assurance issues should be agreed and committed to the sampling plan. The QA programme should address all factors that could affect the quality of the samples both before they arrive at the laboratory and during the testing itself (Appendix A). It is important that the correct storage conditions (e.g. absence of light, temperature <4°C, appropriate preservative, use of sample containers appropriate to the analytes being determined) are agreed during the preparation of the sampling plan. In addition these should be confirmed on the sampling record or chain of custody form. Details of sample preparation for analysis or leaching tests and any deviations from the test methods should be reported with the data.

A technical report (CEN 2004b) is being prepared to support the European sampling standard, which outlines best practice for sub-sampling in the field and storage, transport and delivery. Further detail is provided in Appendix A and in ESART 2004.

Sample preparation

Sample preparation methods have a large impact on the quality of the sample and inappropriate handling can affect the results of compositional analyses and the leaching behaviour of the sample. PrEN 15002:2004 details procedures for the preparation of test portions from laboratory samples.

All test methods require specific sample preparation methods and these are summarised in Appendix C3. For example, samples taken for ‘total composition’ analysis must be finely ground due to the small quantity of sample digested (< 1g). However, for most granular leaching tests, crushing of the sample is kept to the minimum and restricted to the oversize particles. The whole sample may require air-drying¹⁵ to facilitate sieving and removal and quantification of non-crushable items. BS EN 12457:2002 requires a minimum 2 kg weight of test sample for preparation.

¹⁵ Or oven-drying to constant weight, but the temperature must be below 40°C to prevent loss of mercury.

Of this up to 200g will be required on a dry weight basis for an individual leaching test (i.e. without replication). In some cases the 2 kg may yield a sufficient quantity of prepared material for the majority of the testing requirements although this will depend on moisture content, non-crushable content and the number and type of determinations required.

Composition/general characteristics

Examples of the types of compositional testing required for characterisation and to assess acceptance to landfill are listed below.

- *Hazard criteria H1-H14 under the Hazardous Waste Directive ((91/689/EEC) of 12 December 1991.* (See Technical Guidance Note WM2¹⁶). This may involve compositional analysis for comparison with thresholds for H5 harmful, H6 toxic, H14 ecotoxic, H7 carcinogenic etc. Additional testing (for example for flash point or ecotoxicological testing of the waste) may also be required under Duty of Care for hazardous wastes, or to demonstrate the absence of hazardous properties for disposal of mirror-entry wastes to non-hazardous waste landfill (see Section 2.3.1).
- *'Total' concentrations of inorganic components.* These may include mineral acid soluble metals determined by ICP-OES/MS or AAS (for example) on aqua regia digests, microwave assisted acid digests; true 'totals' derived from hydrofluoric/perchloric acid digests or alkali fusions. Fused-bead X-Ray fluorescence may be more appropriate than acid digestion for some wastes (Appendix C).
- *Total concentrations of organic components.* These may include total organic carbon, BTEX (benzene, toluene, ethylene, xylene compounds), PAHs (polycyclic aromatic hydrocarbons), PCBs (polychlorinated biphenyls) and mineral oils (or total petroleum hydrocarbons). Information on these parameters is mandatory for wastes to be accepted at inert waste landfill.
- *pH of a water extract.* The pH of the waste will have a strong influence on the leachability of many metals and is therefore a powerful characterisation tool. It is mandatory for acceptance to landfills for hazardous and stable, non-reactive hazardous wastes.
- *Acid or base neutralisation capacity.* This is the quantity of acid or base required to achieve a specific pH reported in mol kg⁻¹. ANC/BNC is an indicator of buffering capacity or resistance to external influence on the pH of the material. The difference between the ANC/BNC of the waste at natural pH and at the predicted final pH of the landfill leachate should be established. It must be evaluated by the landfill operator as part of the WAC compliance programme. However, where the waste producer is undertaking his own pre-treatment it can be a valuable characterisation parameter (see Appendix C4.3.4).
- *Other parameters.* These include, for example, dry residue content (moisture content at 105°C) required for leaching tests and loss on ignition at 550°C.

The principles of the specified test methods are presented in Appendix C5, Tables D1 to D6. In some cases, the methods specified in the Council decision document are different from those

¹⁶ Hazardous Waste – Interpretation of the definition and classification of hazardous waste. Environment Agency, 2003 (ISBN 1 84432 130 4)

commonly used within the UK. In these cases comparable 'blue book' methods (as prepared by the UK Standing Committee of Analysts) are listed, if available. In some instances XRF and XRD can also be useful analytical tools. For example XRD can verify the presence of specific compounds in waste prior to undertaking a desk-based hazard assessment of existing composition data and XRF can be useful for wastes that are not compatible with acid digestion methods.

Leaching behaviour

Leaching tests can be divided into leaching behaviour tests and compliance tests. The principal uses of the tests are presented in Table 3.3.

The methods for a number of leaching behaviour (characterisation) tests and the compliance batch test are summarised in Appendix C4 and as a series of flow charts in Figures C1 to C4.

Leaching behaviour tests enable predictions of contaminant release to be made for different conditions, for example under different pH conditions or at very low liquid to solid ratios. Examples of how to use the data in combination with information from the compliance leaching test, BS EN 12457:2002, are provided in Appendix C4.3.

Leaching behaviour information would be required to support any application from a waste producer or landfill operator for acceptance of a waste onto a national or site-specific list of wastes that have limited or no further Level 1 testing requirements.

Table 3.3 Examples of leaching tests for characterisation and compliance testing

Test method	Purpose
<p>BS EN 12457 Parts 1-3:2002. Compliance test for granular waste materials. Pt 1: L/S2, <4mm Pt 2, L/S10, <4mm Pt 3: L/S2 and L/S8 (cumulative L/S10), <4mm</p>	<p>Characterisation and compliance batch test for granular wastes. If used as a compliance test, it must also be undertaken as part of basic characterisation.</p> <p>To assess leachability under mild extraction conditions for waste disposal or material reuse options. The pH of the eluate is not externally controlled during this test, i.e. the eluate pH is determined by the pH of the waste material itself ('own pH' or 'natural pH').</p> <p><i>As a compliance test:</i></p> <p>L/S10 values from the two step test (Part 3) are required for assessing compliance with waste acceptance criteria leaching limit values at L/S10 (Table 2.1) for: As, Ba, Cd, Cr, Cu, Hg, Ni, Mo, Pb, Sb, Se, Zn, Cl, F and SO₄, phenol index, TDS and DOC in mg kg⁻¹ dry residue. Eluate pH should always be determined.</p> <p>There may be technical reasons for using the one-step tests (parts 1 and 2) instead of the two-step test, e.g. where the samples are not suitable for serial leaching (see Appendix C4.1).</p> <p>This test is mandatory for assessing compliance with the waste acceptance criteria for inert, hazardous and stable, non-reactive hazardous landfill.</p> <p><i>As a characterisation test:</i></p> <p>Increasing L/S ratio represents increased flushing of the waste with water, correlating with leaching timescales. The two-step test provides basic information about relative timescales for release particularly when compared with maximum availability for leaching and when placed in the context of data from the upflow percolation test.</p> <p>Testing samples targeted for worst-case as well as average operational conditions, or over different time frames will highlight whether the waste quality is vulnerable to WAC non-compliance at certain times. Such screening programmes may lead to the need to identify alternate management options for the waste or for on-site procedures to be modified.</p>
<p>Characterisation (leaching behaviour) tests for granular wastes</p>	
<p>Maximum availability leaching test (EA NEN 7371: 2004)</p>	<p>To determine the potential (maximum) availability of components by leaching under worst-case environmental conditions rather than natural infiltration conditions. Samples are finely ground, tested at high L/S ratios and with pH control (pH 7 and 4). The test is for the assessment of maximum potential for leaching of metals rather than 'total' concentrations determined on hot aqua regia digests. Can be used for granular and monolithic wastes.</p> <p>Limited acid neutralisation capacity (ANC) values at pH7 and pH4 only can be determined from the test data.</p>

Test method	Purpose
Upflow percolation test (prCEN/TS 14405: 2004)	<p>To determine the rate of leaching of various contaminants from granular wastes as a function of liquid to solid ratio (i.e. relative time). The test conditions approximate to the leaching process occurring when rainwater or other liquids infiltrate and percolate through a granular waste material. Cumulative L/S ratios are 0.1, 0.2, 0.5, 1, 2, 5 and 10. Initial leaching data are particularly relevant to the very low L/S ratios prevailing in landfill and provide context for L/S2 and L/S10 data from BS EN 12457:2002 batch tests. Data can be used as source term data for risk assessments for reuse scenarios or (monofill) disposal and for consideration of the acceptable waste sulphate concentrations at landfills for inert waste.</p>
pH dependence tests (prCEN/TS 14429:2004 and draft prEN 14997: 2004)	<p>To determine the effect of falling or increasing pH conditions on leachability of granular wastes. Two test methods either with continuous pH control or in batch mode. Two main applications are leachability predictions for waste:</p> <ul style="list-style-type: none"> • after chemical treatment (e.g. admixing with acid or alkaline wastes) prior to landfilling; • after landfilling, should local porewater/leachate pH conditions change. <p>A full range of acid/base neutralisation capacity (ANC/BNC) values can be determined from both test methods. Acid/base consumption at extreme pH values and neutral pH is also determined initially.</p> <p>Dissolved organic carbon can be determined on the pH7.5-8 eluate for comparison with the appropriate limit value. This second test may be needed where DOC at natural pH (as controlled only by the waste material itself from BS EN 12457) exceeds the limit value. (Section 3.3.2)</p>
Characterisation (leaching behaviour) and compliance test for monolithic wastes	
Diffusion test (tank test for monolithic wastes) (EA NEN 7375: 2004)	<p>To assess the leachability of wastes which have been solidified¹ for reuse or disposal.</p> <p><i>As a characterisation (leaching behaviour) test for monolithic wastes.</i></p> <p>The test is conducted on samples > 40mm in any direction using a volume of leachant approx. 5 times greater than that of the solid. 8 leaching steps are carried out over 64 days. The test is static (no agitation) and is conducted at natural pH (unbuffered deionised water). Results are generally interpreted on a surface area basis (mg m⁻²) rather than a liquid to solid ratio basis (mg kg⁻¹)</p> <p><i>As a compliance test:</i></p> <p>The first four days of the test are completed, eluates analysed and cumulative leaching in mg m⁻² compared with the compliance limit values for monolithic wastes (Table 2.3).</p>

Test method	Purpose
Draft WI 292010 (Compliance test for leaching of monolithic waste, 2004)	<p>A draft CEN/TC2 compliance test has been prepared based on 3 leaching steps conducted within a 48 hour period. The leaching container is sealed and a vacuum applied for 15 minutes to ensure that the pores of the test sample are saturated. Gentle stirring is then applied from the remainder of the test with eluate removal and leachant renewal after 6, 24 and 48 hours from the start of the test. The eluates are filtered and analysed and data reported on a mg cm⁻² basis. (Data interpretation on a mg kg⁻¹ basis could be compromised by variation in the leachant volume to surface area ratio and therefore sample dimensions must be controlled within tight tolerances).</p> <p>A longer dynamic monolithic leaching test is also in preparation for the characterisation of monolithic wastes.</p>

3.3.5 Interpretation

Overview

Interpretation of the results of the characterisation exercise will allow:

- initial identification of landfill class at which the waste could be accepted based on a comparison of the waste analyses and leaching test data with the waste acceptance criteria;
- an assessment of the variability of the data both in relation to previous data and with respect to demonstrating confidence in the initial landfill class identified;
- where leaching behaviour tests have been performed, an indication of whether treatment might improve waste characteristics of borderline cases, so that acceptance to a particular class of landfill can be achieved.

The first two points can be assessed simultaneously, but the issue of variability is discussed below.

Assessing variability of results

When the new targeted sampling programme has been completed and the results are to hand (for any particular parameter of interest) four main cases can be distinguished. These are discussed in Table 3.4.

Classification of waste and identification of suitable landfill class

A comparison of the waste analyses and leaching test data from BS EN 12457-3:2002 L/S10 (for granular waste) and EA NEN 7375:2004 (for monolithic waste) with appropriate hazard thresholds and the waste acceptance criteria (Table 2.1 and 2.3) will provide an initial classification of the waste stream and an indication of the class of landfill at which it may be accepted.

Figure 3.3 presents a generic procedure for classifying the waste and identifying the appropriate class of landfill for its disposal.

Table 3.4 Possible findings from targeted sampling programme

Variability of new data	Status of data relative to limit for particular type of landfill	
	<i>All values below limit</i>	<i>Some values above limit</i>
<i>Relatively low</i>	<ul style="list-style-type: none"> • If data values are generally higher than those of past routine samples, this gives reassurance about the effectiveness of the system used for targeting worst-case wastes. • If they are not higher, some investigation may be warranted to check the assumptions behind the targeting system. • In either case, the producer has good evidence to place before the landfill operator that the waste is suitable for this class of landfill. 	<ul style="list-style-type: none"> • Because the data are quite consistent, the presence of values above the limit indicates that the choice of landfill type is inappropriate. • The data should be re-evaluated for a different class of landfill. • If that is not possible, some form of treatment will be needed - perhaps only for the identifiable worst-case fraction of the waste.
<i>Relatively high</i>	<ul style="list-style-type: none"> • As noted above, if data values are generally higher than those of past routine samples, this gives reassurance about the effectiveness of the targeting. • If operational factors can be identified that explain some of the higher values, then (provided these are potentially controllable) this will improve the producer's case for the waste being suitable for the nominated type of landfill. 	<ul style="list-style-type: none"> • The choice of landfill may still be appropriate <u>provided</u> the reason for values exceeding the limit is clearly identified <u>and</u> there is a convincing remedy. • Otherwise the choice of landfill type is inappropriate, and the options in the cell above should be pursued.

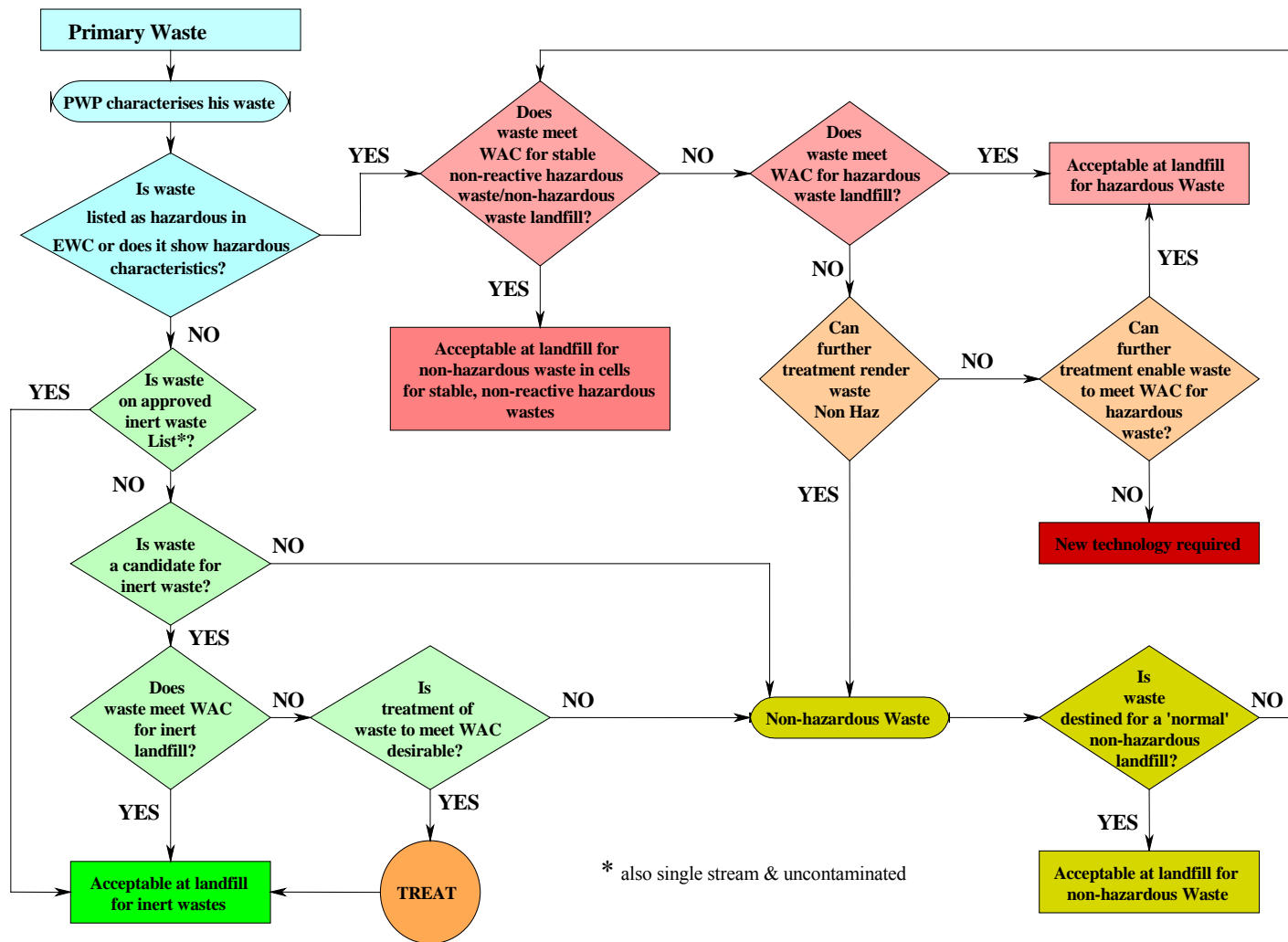


Figure 3.3 Generic flow chart for identifying appropriate waste and landfill class

The main steps are as follows:

- Classify the waste as hazardous or non-hazardous according to the Hazardous Waste Directive¹⁷ and the European Waste Catalogue and proceed with (a) or (b) below for hazardous or non-hazardous waste respectively.

a) *Producers of hazardous wastes*

- In addition to any hazard characteristics specific to that waste (H1-H14 under the Hazardous Waste Directive, 91/689/EEC), check data against the waste acceptance criteria for sites for hazardous wastes (Table 2.1).

Either:

- all limit values are met, in which case the waste is acceptable at a site for hazardous wastes, or
- one or more of the limit values are exceeded, in which case the waste is not acceptable at any class of landfill in its present state, and either:
 - i. further treatment must be undertaken to reduce the organic content and/or the leachability of the waste. The treated waste must then be retested and reassessed against the hazardous waste acceptance criteria; or
 - ii. an alternative, non-landfill disposal outlet must be sought.
- Where a hazardous waste has been treated¹⁸ to render it ‘stable, non-reactive’ hazardous waste, check data against the waste acceptance criteria for ‘stable, non-reactive’ hazardous waste sites (Tables 2.1 and 2.3).

Either

- all limit values are met, in which case the waste is acceptable at a site for non-hazardous wastes in a cell for stable, non-reactive hazardous wastes, or
- one or more of the limit values are exceeded in which case the waste is not acceptable at this type of cell without further treatment, and either:
 - i. further treatment must be undertaken to reduce the organic content and/or the leachability of the waste. The treated waste must then be retested and reassessed against the stable, non-reactive hazardous waste acceptance criteria; or
 - ii. an alternative, disposal outlet must be sought.

¹⁷ See also Hazardous Waste - Interpretation of the definition and classification of hazardous waste. Technical Guidance WM2. Environment Agency, Bristol (2003).

¹⁸ Even if a waste inherently meets the stable, non-reactive hazardous waste criteria, treatment must be undertaken to reduce its volume or hazardousness unless a suitable method is not available (Defra 2004).

- If it does not meet the ‘stable, non-reactive’ limit values but does meet all the limit values for sites for hazardous waste it can be accepted at a landfill for hazardous waste.

b) *Producers of non-hazardous wastes*

- The principle action required by producers is to ensure that their waste is not hazardous (see above)
- No action is required by the producers of treated wastes on a national or site-specific list of non-hazardous wastes exempted from testing such as municipal solid waste (RGN11¹⁹). (Organic and inorganic treated wastes listed on the EWC as non-hazardous currently require no further testing to secure acceptance at landfills for non-hazardous waste (but see Appendix C for other testing options). However, it may be necessary to demonstrate that a waste has not been contaminated in such a way as to render it hazardous. It may also be necessary to demonstrate compatibility with other wastes taken at the site.
- Wastes that are destined for disposal in specific cells with stable, non-reactive hazardous wastes must meet the same criteria as those stable, non-reactive hazardous wastes. The limit on total and leachable organic carbon is particularly important to minimise mobilisation of some of the heavy metals associated with hazardous wastes. This is principally a matter for the landfill operator. However, the waste producer may wish to undertake the testing to determine which options are available particularly when the waste producer also operates a landfill. The producer of a non-hazardous or hazardous waste will classify his waste in accordance with the Hazardous Waste Directive and European Waste Catalogue. The producer of an inert waste will be able to propose an initial classification of waste to determine the appropriate class of landfill for disposal. However, if sufficient information on waste variability was not obtained or reviewed during the characterisation exercise, it is possible that subsequent compliance monitoring by the landfill operator may require a change of classification (e.g. to non-hazardous or hazardous waste) if limit values for acceptance at a landfill for inert waste are not met.

c) *Producers of inert waste*

- No action is required by the producers of wastes that are on the list of inert wastes²⁰ other than to demonstrate that it is uncontaminated, single-stream waste from a single source.
- Other ‘inert’ waste producers will need to demonstrate compliance with the inert landfill WAC. They will need to check that their treated wastes are essentially inorganic (<3w/w% TOC), with very low levels of organic contaminants (BTEX, PCBs, PAHs and mineral oil), very low leachabilities with respect to the listed metals, anions and

¹⁹ Environment Agency 2005b. Landfill Directive Regulatory Guidance Note 11. The disposal in landfills for non-hazardous waste of: stable, non-reactive hazardous wastes, asbestos wastes, wastes with high sulphate or gypsum contents.

²⁰ The full list is given in the October 2003 consultation draft of the Landfill (England & Wales) (Amendment) Regulations, 2004.

phenol index and containing low eluate levels of dissolved solids and dissolved organic carbon. The main steps are summarised below.

Check data against the waste acceptance criteria for inert waste sites (Table 2.1).

Either:

- all limit values are met, in which case the waste is acceptable at an inert waste landfill, or
- one or more of the limit values are exceeded, in which case:
 - i. further treatment must be undertaken to reduce the organic content and/or the leachability of the waste. The treated waste must then be retested and reassessed, or
 - ii. the waste must be handled as a non-inert waste (see below), or
 - iii. an alternative disposal outlet/management option must be sought, e.g. if fails the TOC limit, the waste may be appropriate for use as a landfill restoration material.

Leaching behaviour

One or more leaching behaviour tests may be used as part of the characterisation. In combination with the two-step batch test these can significantly increase the level of information available for predicting the leachability of granular and crushed monolithic material under different conditions, thereby allowing a number of different questions about the leaching behaviour to be posed.

For example:

- if the waste is stockpiled in the open, what level of contaminant release might be expected with continued infiltration? (percolation test);
- how much of the available contaminant will remain after the waste has been flushed by varying amounts of water? (percolation test and maximum availability leaching test);
- will prolonged ageing/washing reduce leachability to below waste acceptance limit values? (percolation test and batch leaching test);
- if ageing also brings about a decrease (or increase) in porewater pH within the stockpile, would this reduce the leachability of pH-dependent metals below limit values? (pH dependence test, batch leaching test).

Further information on the relationships between data from the leaching tests is provided in Appendix C4.3.

Using the characterisation data

For granular wastes, data from leaching behaviour tests can be used in conjunction with the compliance test data to highlight opportunities for simple treatment measures that could be

undertaken on-site. This might be needed to ensure that waste acceptance criteria are met, or could be employed where a lower classification of site is sought (e.g. hazardous waste to stable, non-reactive hazardous waste).

For example the following enquiries could be made where failure of limit values has been reported.

- Is it a ‘blip’, unlikely to happen again, or can the process be ‘tweaked’ to prevent a repeat occurrence?
- Is it an artifact of the process and therefore likely to be repeated? If so, can the process be modified to deliver treated wastes that will consistently meet the waste acceptance criteria? (For example, improve the organic carbon content to meet 3% TOC limit for inert wastes, reduce the leachability of metals to meet eluate limit values.) This may include scrutinising feedstock or other inputs to the plant to exclude contaminated inputs to the process.
- Is it easier or more cost-effective to separate contaminated ‘hot-spots’ of waste from the main waste stream? The more contaminated stream could be further treated and/or sent to a higher class of landfill, leaving the rest of the waste less contaminated and able to take the usual disposal route.
- If contaminated ‘hot-spots’ cannot be identified and separated, is it feasible to modify the characteristics of the waste to reduce leachability? (For example use information from the pH dependence test and percolation test to conduct ageing/washing/pH adjustment trials on site).
- If neither the process nor waste characteristics can be modified to ensure that the limit values can be met consistently, the waste must go to a higher class of landfill - or, if an alternative landfill cannot be used and a further treatment process cannot be found to make it acceptable for landfilling, an alternative non-landfill disposal route must be sought (e.g. high temperature incineration).

3.3.6 Reporting

Characterisation

As a minimum the characterisation report should provide the required information for basic characterisation listed as (a) to (k) in Section 2.2.

The following should be included:

- the presentation of the test results for assessing compliance against the waste acceptance criteria (for example, TOC concentrations and for granular wastes, L/S10 results for batch leaching test BS EN 12457-3 for a number of samples), and
- the scale selected for the sampling (e.g. 20m³ container, 10m³ stockpile), and
- an initial classification of landfill at which the waste may be accepted; and
- a demonstration that failure of limit values is not expected in the next compliance

assessment period (e.g. even under worst-case operational conditions the plant delivered loads of waste with average concentrations that were WAC-compliant).

This must be supported by a dataset derived from a sufficient number of samples²¹ to demonstrate that a satisfactory attempt has been made to quantify the variability within the waste for all parameters. Evidence should also be provided that some samples were collected to coincide with periods when the worst-case situation with respect to waste quality would be predicted. This might be after a period of down-time at the plant, or when a non-routine feedstock or process input was being used. The report can be supplemented by data from other similar plants.

The report should also provide characterisation data on more typically representative samples of the waste. For example, the batch test L/S 2 and L/S10 data for granular wastes should be compared with maximum availability of metals within the waste and either the impact of increased flushing with water or a change of pH conditions on the leachability of the waste.

We require use of the two-part test, BS EN 12457/3, because it provides more information about the leaching behaviour of the waste over time.

The full report should be retained for auditing purposes but does not need to be submitted to the Environment Agency.

Waste producer's compliance monitoring programme

Where a compliance monitoring programme is being conducted by the waste producer for his own purposes (e.g. to monitor the impact of process modifications or waste treatment trials) any cyclical factors should be highlighted to ensure that testing is periodically undertaken at the worst point in the cycle. In addition, the dataset can be used to highlight where a reduction in testing can be incorporated into the waste producer's compliance monitoring programme. The report should distinguish between testing that has been carried out on fresh waste as it is generated and wastes that may have been stockpiled on site in the open or treated in some way to modify their composition or leaching characteristics.

In a partnership approach with the landfill operator involving free exchange of information, the waste producer may not need to undertake his own compliance monitoring programme.

Application for inclusion on national lists

Where the waste producer wishes to make an application for inclusion of the waste on a national list of wastes requiring a reduced level of testing, all the information outlined above will be required. Evidence must be provided that the levels of key variables and, for granular wastes, the L/S10 values of the treated wastes are well below the limit values and that the leaching behaviour at L/S2 and L/S10 is consistent. This must be the case for samples collected at all points in the operational cycle, including under worst case conditions. Where waste from multiple installations is to be considered for inclusion on a national list, the waste

²¹ This will depend on the waste characteristics. Key statistical aspects to be considered in designing a sampling programme are discussed in Section 4.3.1.

from all plants must be of demonstrably consistent quality and predictably compliant with all relevant acceptance criteria.

Information on leaching characteristics under increasing liquid to solid ratio (upflow percolation test for granular wastes) and/or different pH conditions (pH dependence test) should be presented to show that the wastes will behave in a predictable manner under a range of landfill conditions. Where the upflow percolation test is not undertaken, the relationship between the potential leachability (derived from the maximum availability leaching test) and contaminant release at L/S2 and L/S10 must be demonstrated for granular wastes.

3.4 Level 2 – compliance

3.4.1 Overview

This section refers to the ‘private’ compliance monitoring scheme conducted by the waste producer or the product QA monitoring scheme operated by the treatment plant operator rather than that conducted by the landfill operator. The waste producer would be advised to conduct such a compliance scheme, particularly where the waste may be close to the limit values for acceptance to a particular class of landfill. This should provide early warning of any possible compliance problems and hence reduce the risk of the waste producer inadvertently sending waste to the wrong landfill. This is particularly true for an inert or hazardous waste which may be close to the limit values. For example, if the waste were to prove to be more heterogeneous than had been thought, the resulting information could be used to identify opportunities for treatment or process modification, and this might result in the waste going to a different class of landfill (e.g. non-hazardous to inert).

However, additional targeted, worst-case samples would be needed to assess compliance with the waste acceptance criteria. Where waste acceptance criteria were failed, the reason for failure should be established immediately (e.g. was it due to an operational problem, or to the input of poorly characterised waste?) and rectified. The ‘off-spec’ waste would require further treatment until it met the relevant waste acceptance criteria or be consigned to a different class of landfill. For example the leachability of inorganic elements and/or the total organic content of the waste may need to be reduced, or the pH or acid neutralisation capacity of the treatment residue may need further modification. Where the waste has already been accepted to landfill without further testing, the landfill operator should be informed immediately. Procedures to prevent a reoccurrence should be implemented and the landfill operator and Environment Agency should be notified of the management or process changes undertaken.

There is no legal requirement for the waste producer to undertake his own routine WAC-compliance monitoring programme, and it may not be needed in a partnership arrangement with the landfill operator. Nor will it be needed if there is no doubt in the integrity of the historic dataset or in the ability of the plant to deliver consistently WAC-compliant wastes. If this is not the case, then the waste producer may wish to balance the costs savings from not doing any compliance monitoring himself against the risk of the landfill operator rejecting his waste if repeated non-compliance data are returned from the landfill operator’s own monitoring programme.

The landfill operator must inform the waste producer immediately if a waste has failed a compliance test. The operator should refuse to accept all further consignments of the waste

until the producer has provided a satisfactory explanation of the failure and evidence that the waste will meet the waste acceptance criteria in the future.

The landfill operator should supply the waste producer and the Agency with a compliance report on a regular basis and at the end of the compliance period (normally a year) as detailed in Section 4. This will not be a detailed report, but a tabulation of reference information for loads that have been sampled and a list of non-compliant parameters.

Even if only a small number of samples are taken as part of the waste producer's own compliance monitoring programme, the information is still a useful addition to the historic dataset. This would be available for review as part of the initial assessment of data when negotiating a new waste disposal contract. A more extensive, reliable dataset, particularly where targeted worst-case monitoring can be demonstrated, may reduce the need for rigorous checking by the landfill operator. This reduction in risk for the landfill operator may yield commercial benefit for both parties.

Where analytical results are not of acute commercial sensitivity, it is recommended that the waste producer provides all relevant characterisation data to the landfill operator. In particular this should include background information relating to variability of those parameters for which waste acceptance limits have been set. The landfill operator and waste producer would then work together to draw up a compliance monitoring programme that would generally be more comprehensive than the absolute minimum scheme described in Section 4 - and so be more informative to both parties.

3.4.2 Sampling

An important secondary aim of Level 1 characterisation will be to provide information regarding the likely performance of the waste against the compliance criteria governing waste acceptance to landfill (Section 4). As these limits are to be viewed as maximum concentrations of leachable metals (and other determinands), the most effective approach is for the sampling to target the occasions when the quality of the waste might be expected to be closest to the appropriate limit values. This is referred to subsequently as 'targeted worst-case' sampling.

The need for targeted sampling poses a problem, as it is incompatible with the representative sampling typically required in meeting the Level 1 characterisation objective. This highlights the general principle that different objectives can lead to quite different sampling requirements. Here, however, the conflict can in principle be resolved by the adoption of a stratified sampling programme. Assuming that there is sufficient information about the nature of the waste for it to be classified into several categories - 'Best', 'Typical' and 'Worst', say - Level 1 sampling would be conducted for each of these identifiable groups. For an overall assessment of the waste, the data from the three groups would be combined statistically, as outlined in Appendix B1. Then to assess the likely performance of the waste under the operator's Level 2 compliance programme, the data for just the 'Worst' category would be used.

3.4.3 Testing

Of the leaching tests available, only the two-step batch test (BS EN 12457-3) can be used for

granular waste to assess compliance with the leaching test limit values. This test should also have been carried out as part of the characterisation exercise to determine initial waste classification. However, technical issues may preclude the use of the two-step batch leaching test (BS EN 12457-3) and the single-step L/S10 test (BS EN 12457-2) may be used instead in certain circumstances (see Appendix C4.1.1). Its use must be justified.

Testing requirements for most wastes include an assessment of the total organic carbon (TOC) of the waste and the leachability of dissolved organic carbon (DOC) from the waste. Wastes destined for landfills for inert wastes, stable non-reactive hazardous wastes or hazardous wastes must be essentially inorganic in nature and therefore must be shown to have a total organic carbon content (TOC) of less than 3, 5 or 6% w/w C respectively (or have a loss on ignition content below the appropriate limit value). For granular wastes, where failure of the DOC limit at natural pH (from the BS EN 12457-3) test has occurred and chemical contamination rather than biodegradable organic carbon is believed to be the reason, a second DOC extraction at neutral pH can be undertaken (the test method is based on the pH dependence test, prCEN/TS 14429:2004 see Appendix C).

For wastes destined for an inert or hazardous waste landfill, any other parameters for which there are limit values should also be determined until periodic testing demonstrates that they are not present at borderline levels.

3.4.4 Interpretation

If any one of the targeted samples produces values that exceed the limit (Tables 2.1 and 2.3), the waste is deemed to have failed the acceptance criteria for that particular class of site. The options mentioned in Section 3.3.5 would then be relevant.

3.4.5 Reporting

The results of the waste producer's compliance monitoring programme - together with all relevant detail about the times and locations of samples, operational conditions, etc. - should be held in a clearly defined location (e.g. a spreadsheet kept on a secure PC with controlled access). This will allow the data to be reviewed as and when necessary - for example, in support of a breach of compliance reported by the landfill operator. In many cases, simple scanning of the data will be sufficient to confirm that there are no spurious results (when compared with the historical data set and/or the characterisation report), and that there are no obvious time trends to indicate that a repeat of the characterisation exercise might be required.

3.5 Recommendations for the waste producer

Wastes on a national list (e.g. the list of inert wastes) may not require a sophisticated sampling programme. Waste producers who are passing their wastes to a third-party for treatment prior to landfilling may also not need to undertake the leaching tests required as part of level 1 characterisation of the waste, as it is the treated wastes that will be landfilled. However the remaining information should be provided in order to comply with Duty of Care requirements and to provide the treatment plant operator with sufficient information to be able to predict the outputs of his process.

The waste producer, who is meeting the treatment requirements of the Landfill Directive in-house, would be advised to undertake a carefully planned stratified sampling programme at the earliest opportunity. This should include samples collected when the waste quality is expected to be at its poorest, i.e. when the highest levels of organic matter and/or leachable metals would be predicted to occur.

A sampling and testing programme spanning a group of waste producers can be co-ordinated by a national trade body or institution. Some industrial processes generate wastes that are relatively consistent in gross characteristics and leachability from plant to plant. In these cases, sampling should include older or poorly performing plant as well as flag-ship examples. The programme may show that all plant produce waste with similar characteristics or that plant of a certain type or a certain age may produce what is essentially a different waste stream. A specific waste producer may then be able to avoid a full level 1 characterisation if he can show that his waste is similar, in all relevant respects, to the waste produced by the generic group.

A satisfactory characterisation report, backed up with compliance monitoring data indicating that the generic waste is essentially consistent in quality, or that processes can be modified to produce consistent wastes, would demonstrate that the waste was a suitable candidate for inclusion within a national list of wastes with a lower testing requirement.

4. LANDFILL OPERATOR

4.1 Preamble

For any given waste stream, the primary responsibility of the landfill operator is to test compliance with the waste acceptance criteria for his landfill. He must ensure and demonstrate that he is accepting only wastes that meet the conditions of his permit. In order to do this he will need evidence that the waste producer's characterisation exercise has resulted in the waste being classified correctly. He will be looking particularly for assurance that there will be no appreciable 'hot-spots' - abnormal or unusual runs/batches/episodes in the production process - which might generate wastes that would fail the test.

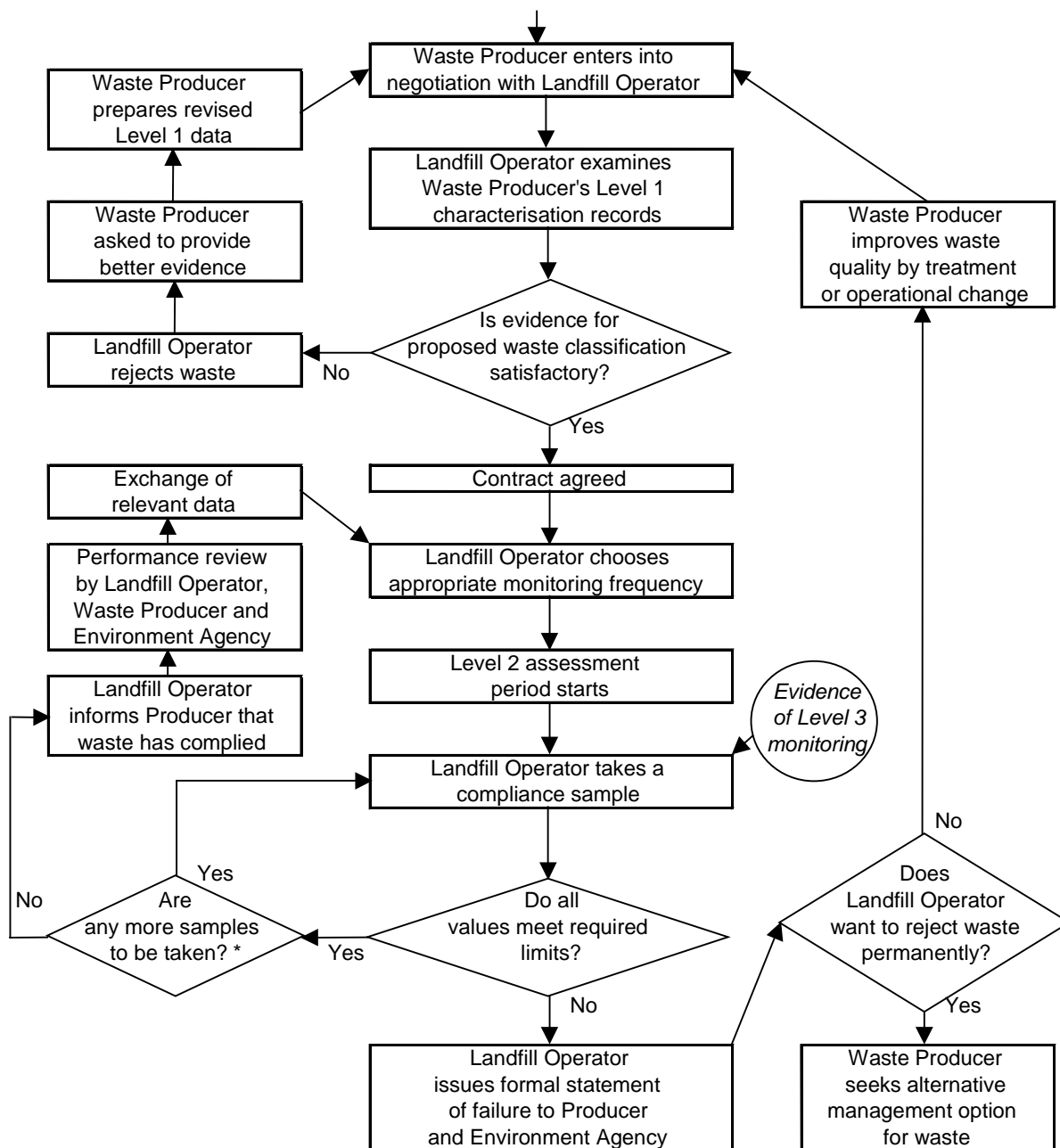
The surest way of gaining such assurance is by seeing all relevant findings from the producer's characterisation programme. The Environment Agency will require the landfill operator to provide at least an annual compliance report that should be copied to the waste producer. However, there is, at present, no similar obligation, other than Duty of Care, for the waste producer to share his data and process knowledge with the landfill operator. Thus, as noted in Section 2.5, there may need to be a change in attitude by waste producers towards sharing the results of the monitoring programme: it would be most cost-effective for both the waste producer and landfill operator to view the data collection as a partnership and share the information, as far as is commercially possible. A detailed description of the required compliance assessment scheme is set out in Section 4.3. There are a number of parallels with the procedures that have been successfully applied since the late 1980s for the regulatory monitoring of sewage treatment works effluents. The main elements of effluent compliance assessment, and their points of similarity with the present waste assessment scheme, are outlined briefly in Appendix E.

The testing requirements for the landfill operator are relatively straightforward and therefore the reader is referred to the background in Sections 2.4 and 2.5, relevant sections in the chapter related to the waste producer (Section 3) and to Appendix C.

The sequence of events that should be undertaken by the landfill operator is summarised as a flow-chart (Figure 4.1).

4.2 Use made of data from Level 1 characterisation

The waste producer will in some cases, provide information from Level 1 characterisation. In other instances it may be appropriate for the operator to commission or undertake the testing on behalf of the waste producer and include the costs in the waste disposal charges.



* For example, have a minimum of 6 samples been tested?

Figure 4.1 Generic flow-chart for an operator of a specific class of landfill

Given the limited time likely to be available for evaluating a producer's waste, the landfill operator may want to target effort towards managing the compliance scheme according to his prior knowledge of the waste producer and the producer's process. He will not have time to review all of the characterisation report; much of the information may not be relevant to him,

and some waste producers may in any case be reluctant to pass on all the data.

Two extremes, and a potentially beneficial ‘middle way’, can be envisaged, in the way that the landfill operator handles information from three different types of waste producer.

- Producer of a low-variability waste: He is an existing client, the landfill operator has accepted similar waste for some time and it seems to be consistent. The producer states that he has L/S10 or other data to justify his assertion that the waste is consistent, and nowhere near the limit values. This is a low risk customer, and the landfill operator deems it unnecessary to review the characterisation reports in any detail.
- New client: There is no information on consistency so the landfill operator may ask for evidence to back up waste producer’s claim that the waste is consistent and that the dataset does not just relate to best case and/or representative samples. The landfill operator will concentrate on the maximum values provided - and will look for any evidence to indicate whether these relate to normal conditions or to potentially unfavourable wastes.
- Partnership: The waste producer and landfill operator have negotiated the contract on the basis of free transfer of data between the parties. The Level 1 dataset is ‘on the table’ and the waste producer and landfill operator are combining effort in sampling and testing for the compliance programme (see Section 3.3.1). In this scenario, as long as the landfill operator is confident that the load has not been tampered with en route, then it may be simpler to collect a representative sample of the load during loading at the waste producer’s premises than have to take a representative sample at the landfill gate.

Whichever approach is taken it is essential that the scale selected for the sampling is the same for the waste producer and landfill operator (and the regulator, if enforcement samples are required).

4.3 Level 2 – compliance

4.3.1 Basis of the scheme

The main elements of the Level 2 compliance approach are as follows.

Assessment period

In principle, compliance may be assessed over any number of months. Thus, the more critical the waste (whether on account of the expected concentrations of critical parameters or the quantities arriving at the landfill), the shorter the landfill operator might wish to make the assessment period. However, because of the organisational complexities of maintaining a variety of different assessment periods, a standard period of one calendar year is recommended as a reasonable default.

Statistical interpretation of leaching test limit

The L/S10 limits for granular wastes and the tank test limit values for monolithic wastes are to be interpreted as strict maximum limits. For compliance to be achieved, all parameters must meet their respective leaching test limits for all samples at the appropriate scale

Sampling plan requirements should be related to scale (see textbox in Section 2.4). The waste producer, landfill operator (and regulator in the case of any enforcement samples) should agree the appropriate scale before undertaking a sampling programme.

For waste acceptance to landfill, the scale of sampling has been set at the scale of the load (whether a skip or single rail freight container). Representative sampling²² of that load should be undertaken to avoid over-reliance on analyses of contamination hot-spots picked up at a finer scale.

Type of sampling

The recommended approach is targeted worst-case sampling, whereby the landfill operator deliberately focuses sampling effort on occasions when he believes the waste has the greatest risk of failing the limit. If the average concentration of incremental samples from a worst-case load is compliant with the WAC, then it is likely that the average concentration of incremental samples of any waste load will generally also be WAC compliant under normal conditions. This may not always be the case and should be tested for all WAC parameters.

(Note that this is in marked contrast to the approach needed when assessing compliance against *mean* or *percentile* limits, when targeted sampling would be entirely invalid and sampling that is representative of the total waste population would be adopted).

Targeting relies on the landfill operator having useful information from the Level 1 characterisation about factors affecting waste quality, perhaps reinforced by past experience of that waste. It may also be possible to use the results of the routine Level 3 checks to flag up occasions where a targeted compliance sample could be advantageous. However, there will be many circumstances where the landfill operator has insufficient knowledge about the waste to take a targeted approach - or has good reason to believe that the waste is sufficiently homogeneous over time for leaching test results to be similar whenever the samples are taken. In that case some form of random or representative sampling may be used instead. A convenient option then would be systematic sampling, whereby samples are taken at regular intervals (e.g. one sample every 2 months, or one representative sample from every fortieth skip).

²² e.g. combining incremental samples obtained either by coring through the entire waste depth, or grab-samples obtained over the entire period of discharge/unloading of the container.

Number of samples

In situations where non-targeted sampling is adopted, the degree of protection achieved by any given number of samples can be calculated. The 'statistical power' for various sample numbers is shown in Figure 4.2. The quantity measured along the 'horizontal' axis is the unknown proportion of the total amount of waste arriving at the landfill over the year's assessment period that is truly above the leaching limit. (For many wastes, this may of course be 0%.) The chance of a compliance failure is measured on the 'vertical' axis. The curves then show, for various numbers of samples, how the chance of failure increases as the waste truly gets worse.

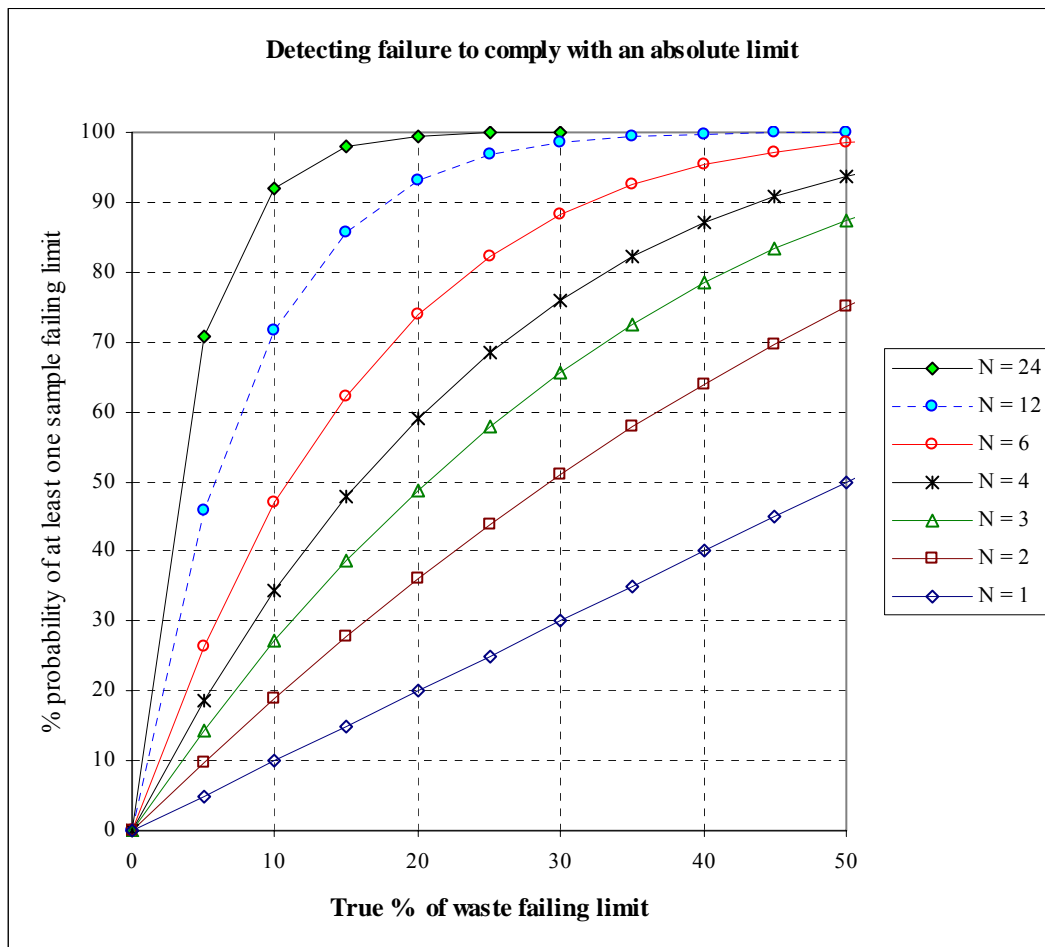


Figure 4.2 Protection provided by a random sampling compliance programme

Take, for example, the case of $N = 6$ samples (shown by the curve with hollow circles). If 10% of the waste over the assessment period is truly non-compliant, a programme based on 6 random samples has a chance of just under 50% of detecting a failure. If, however, as much as 30% of the waste is truly non-compliant, the chance that the landfill operator will detect a failure on the basis of 6 samples is nearly 90%.

The figure provides the landfill operator with a rational basis for deciding how much or little compliance sampling to devote to a particular waste. For example, if it were deemed

important to fail the waste when (for a particular parameter) as much as 10% by volume was truly above the limit, this could be achieved with about 24 samples. Conversely, a single annual sample might be sufficient for a waste that was believed to pose little or no problem. Of course, in any one year the detection rate would then be very poor, as Figure 4.1 shows. But if a non-compliant situation was thought very unlikely to arise, the operator might be willing to work to a much longer time horizon than one year. Over a six-year assessment period, for example, he would have accumulated 6 samples, and hence have a good chance of detecting a waste that was truly over the limit for 25-30% of the time. In many cases this will be an ample level of protection if the waste is of relatively low volume and/or of less critical composition in comparison with the totality of landfill inputs.

Where targeted sampling is used, the statistical power is likely to be substantially greater than the 'long-stop' guarantee provided by the Figure 4.2 power curve. However, there is no exact way of calculating in advance how much better it will be, as this will depend entirely on the quality of the information possessed by the landfill operator. The more adroit the targeting, the more likely the monitoring will be to pick up cases of non-compliance. There is, nevertheless, one way of estimating the improvement in certain circumstances. This is outlined in Appendix B; and the example given there shows that the information provided by just 5 targeted samples can be superior to that provided by 24 'conventional' representative samples.

The Environment Agency considers that a minimum of six targeted samples per year will generally be required for each waste stream.

4.3.2 Implications of a successful Level 2 compliance assessment

Suppose that, over a year's compliance monitoring, all the leaching test results for a particular parameter are well below the limit. Such an outcome might be thought to justify dropping that parameter from the compliance suite. However, this would be appropriate only if there was *strong supporting evidence* that the waste was broadly consistent through time. The evidence of the samples alone is not sufficiently compelling. As Figure 4.3 illustrates, 100% compliant data could quite conceivably arise from either a 'consistent' waste (Case A) or an 'erratic' waste (Case B). This is supported by the power curves of Figure 4.2. For example, even if 6 out of 6 samples all comply, it is quite possible that as much as 10% of the waste is failing the limit, as this degree of non-compliance would have less than a 50% chance of being picked up.

4.3.3 Actions following a Level 2 compliance failure

Suppose the landfill operator had decided that 6 samples are to be taken over a 12-month period, and that one of these samples fails. It is important to appreciate that this does not simply mean that that *particular* consignment of waste has failed. It means that *the whole year's waste* from that producer has been deemed to fail. This must be so, because the annual compliance rate is demonstrably less than 100%. In fact, the true failure rate could easily lie anywhere between 1% and 58%. (The statistical details are in Appendix B3.) In other words, there is an appreciable chance that *over half of the year's waste has failed the limit*. This demonstrates that even a 'marginal' compliance failure needs to be taken seriously.

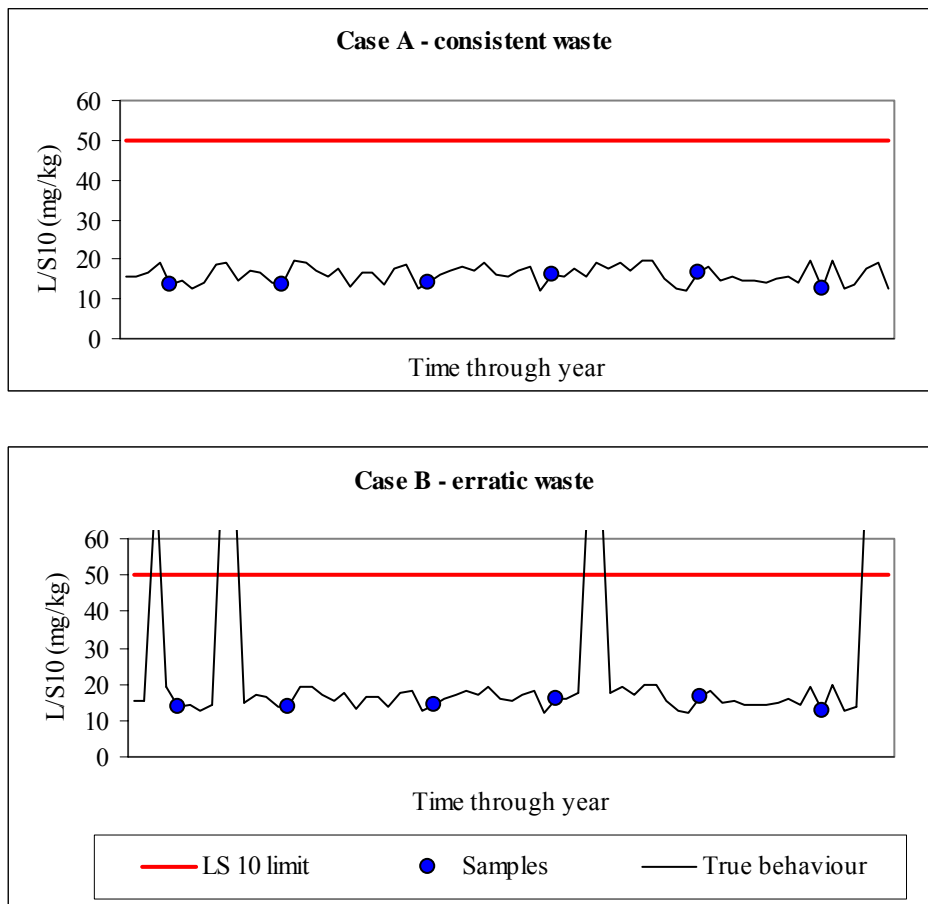


Figure 4.3 Examples of consistent and erratic wastes

The response to a compliance failure will, nevertheless, vary according to factors such as the severity of the failure, the potential impact of the waste, and the operator's previous experience of the waste producer. The most extreme option would be to ban the waste permanently from the site. Alternatively the waste producer would be required to do a full Level 1 re-characterisation exercise, with greater emphasis placed on identifying factors leading to waste 'hot spots'. The producer would then need to demonstrate that these problem wastes could be identified and separated out (and then treated and/or sent to a different class of site), and that the remaining bulk of the waste would then comply. Another option would be to see whether the characterisation information indicated that the leachability of the waste could be reduced (e.g. pH-dependent metal leaching – see Appendix C).

The primary consequence of failure is an obligation for the landfill operator to inform the waste producer. The producer will be required to explain the reason for the failure and how he is going to prevent compliance failure in the future - whether through tighter operational control, or treatment either on-site or at a merchant treatment plant to meet the limit values.

In all cases, compliance failures must be recorded by the landfill operator together with the operational or management changes effected by the producer to prevent recurrence. If the waste producer is unable to provide this information, any further consignments of the waste should be refused. The Environment Agency will require regular returns on compliance monitoring from the landfill operator as a requirement of permit conditions. Failure to provide the records will be a breach of permit conditions and may lead to enforcement action.

The guidance provided in Section 3.3.4 with respect to quality assurance and sample preparation apply to samples collected under the landfill operator's compliance scheme. Information on the specified test methods of waste analysis and the compliance leaching test are outlined in Section 3.2 and in Appendix C.

Granular wastes

The waste acceptance criteria for granular wastes are reproduced in Table 2.1. The WAC parameters constitute the minimum testing requirements to determine the acceptance of granular wastes at landfill. These cover a range of inorganic determinands in eluates derived from the European standard leaching test BS EN 12457-3:2002 and several specific parameters determined on the waste itself, such as pH, acid neutralisation capacity, total organic carbon and/or loss on ignition.

The operator of an inert waste landfill²³ will have the largest number of parameters to monitor against the full waste acceptance criteria, as organic contaminants such as BTEX, PCBs, PAHs and mineral oil (or total petroleum hydrocarbons) must also be determined. There may also be site specific acceptance criteria for prohibited parameters (e.g. cyanide). In addition, the waste must be rejected or tested if there is doubt regarding its source (single stream/single source) or contamination status, since despatch by the waste producer. The Level 3 verification checking may indicate when compliance sampling is required (see Section 4.4).

In most cases the operator of a non-hazardous waste landfill will need to undertake less sampling and testing than for other classes of site as limit values do not apply at present for non-hazardous wastes received at non-hazardous waste landfills. However, the operator of a non-hazardous waste site will need to demonstrate that none of the wastes exhibit hazard properties H1-H14 under the hazardous waste directive and therefore testing will focus on these characteristics.

It will also be necessary to consider that:

- the wastes to be accepted in a cell are compatible;
- the leachate source term used in the risk assessment used to gain the PPC permit for the site was appropriate for the waste that it is planned to accept (see RGN18); and
- there may be a need to apply waste acceptance ratios to control the waste mix in a cell.

²³ The operator of an inert waste landfill can also avoid sampling and testing wastes by only accepting single-stream listed exempt wastes that are known to be from a single-source.

The acid or base neutralisation capacity (ANC/BNC) must be evaluated by the operator of landfills for hazardous and stable, non-reactive hazardous wastes in the context of other wastes in the landfill. It is the quantity of acid or base required to achieve a specific pH reported in mol kg⁻¹. ANC/BNC is an indicator of buffering capacity or resistance to external influence on the pH of the material. Wastes with a high neutralisation capacity can have a significant impact on the pH of surrounding, less well-buffered materials compromising leachability predictions derived from tests conducted at 'natural' pH (e.g. BS EN 12457 and upflow percolation test for granular waste). In this instance such wastes should not be mixed unless the leachability of the less buffered waste has been assessed under different pH regimes, via the pH dependence test. This test provides the most detailed information on the leachability of pH-dependent metals and the neutralisation capacity of wastes that are potentially incompatible under the predicted final pH range for the landfill leachate (Appendix C4.3.4).

Monolithic wastes

In some circumstances the landfill operator will have a direct role in producing monolithic wastes, in which case the testing outlined for the waste producer in Section 3.3.4 apply. Where he receives the waste in monolithic form from the waste producer he will need to undertake compliance testing, but will also require characterisation information in order to undertake the site risk assessment. In either situation, as the testing is the same, a partnership approach would provide best value to both parties.

Characterisation for the site risk assessment.

Characterisation for a site risk assessment for the receiving landfill should comprise the following tests.

- ***64-day EA NEN 7345:2004 tank test for monolithic waste.*** The 64-day tank test quantifies long term diffusive leaching from the solidified waste product. Cumulative data from the full test should meet full 64 day leaching limit values for monolithic wastes given in Table 2.3. If a cementaceous binder is used the test should be conducted on waste forms that have cured for 28 days, as this test also serves to indicate the longevity of the waste form. The full 8 stage 64-day test enables the demonstration that the emission follows a solely diffusive form and not an advective/ fully solubility controlled form similar to granular wastes.
- ***Maximum availability for leaching (EA NEN 7371:2004)*** on the ground monolith, pH dependent leaching (prCEN/TS 14429:2004) and calculation of ANC/BNC, both on the ground monolith. The tests on the ground monolith can be used for landfill site risk assessments to quantify the source term and to predict changes in leachability should the monolith be overlain by waste of different pH and buffering capacity.

Compliance testing of the monolithic waste at the landfill

The compliance test for monolithic waste entering a landfill is a shortened version of the standard 64 day diffusion test (EA NEN 7375:2004). The 4 day compliance limits are

provided in Table 2.3 and are $\frac{1}{4}$ of the 64 day limit values which are used for characterising²⁴ the output of the treatment plant. Cumulative leaching from the first four steps of the test will be the level 1 characterisation benchmark against which periodic compliance testing will be checked. Electrical conductivity does not have to be determined on the diffusion test eluates for compliance.

In addition, the monolith must be formed from waste that contained no more than 6% total organic carbon or 10% loss on ignition (Table 2.2).

The sample used for compliance testing prior to acceptance at the landfill must be at least 40mm in any direction, but there will be no requirement to cure the sample before compliance testing.

Key variables

The key variables to be identified should primarily be dictated by what parameters are known to be present in non-trivial quantities in the feedstock. Where such knowledge is not available, the full suite of parameters should generally be included, and the list reviewed when sufficient samples have accumulated for a considered assessment of the data to be possible.

A reduction in the list of determinands to be tested can only be agreed when it is known or it can be shown that:

- the element in question cannot be introduced during the process and is not present in any feedstock;
- the characterisation demonstrates that it is not a significant parameter in the results of the waste testing; and
- no contamination from that element could be expected to occur between testing and acceptance at landfill.

4.4 Level 3 – verification

The Landfill operator is required to undertake a visual inspection of each load of waste delivered to the site before unloading (if the waste is accessible) and after unloading, and to check all required documentation.

For wastes deposited by the waste producer at a landfill in his control, this verification may be made at the point of dispatch.

The waste may be accepted at the landfill if it is the same as that which has been subjected to basic characterisation and compliance testing and which is described in the accompanying documents. If this is not the case, the waste must not be accepted.

The appearance, odour²⁵ and any other relevant, readily determined properties must be

²⁴ The landfill operator will also require characterisation data for the monolithic waste for his site risk assessment

²⁵ A risk assessment should be undertaken prior to actively assessing an odour.

checked. In addition, tests may be required to ensure compatibility with the engineering requirements of the landfill (for example, for stable, non-reactive hazardous wastes).

The landfill operator may use the verification checking procedure to identify samples which are suitable for compliance testing. These samples must be maintained under appropriate storage conditions until the analytical results have been reported, and for at least one month.

4.5 Reporting

4.5.1 Routine compliance

Compliance monitoring reports will be sent to the waste producer and the Agency on a regular basis. These reports will describe testing undertaken in the preceding period and list all parameters that failed the compliance limits with appropriate reference information. Where a partnership approach is being taken it would be appropriate to copy the full analytical report to the other party. However, in general an electronic report tabulating the following details, would suffice for submission to the Environment Agency and waste producer:

- reference information to uniquely identify the load sampled (including EWC code),
- a list of non-compliant parameters (if any) for each sample
- information on the selected scale,

At the end of each assessment period (in most cases 12 months) the compliance monitoring data will be reviewed by the landfill operator. This will be in addition to the immediate reporting of any non-compliance. The review report will indicate what measures have been taken to prevent any further failure(s) in the following assessment period. The landfill operator should have obtained this information from the waste producer at the time of failure. The review and reporting of data will be a requirement of the site permit. Within the waste management companies, compliance will normally also be reported internally, as for example through compliance assessment reports prepared as part of the company's Environmental Management System.

4.5.2 Application for inclusion on a site-based list

Where the landfill operator believes that a waste accepted at a specific landfill is of consistent quality and wishes to make an application for inclusion of a waste stream on a site-specific list provided in the permit, this must be supported by a detailed report. The results of the compliance monitoring conducted at least yearly intervals, must show that the leaching behaviour and levels of other key variables is consistent and that all relevant values are well below the limit values. Characterisation data from the waste producer or commissioned by the landfill operator must show that the leachability of the treated waste can be predicted at least for the range of liquid to solid ratios and/or pH conditions likely to be prevalent at that particular landfill. For granular wastes, where results from the upflow percolation test are not available the relationship between potential availability for leaching (derived from the maximum availability leaching test) and contaminant release at L/S2 and L/S10 must be demonstrated.

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APPENDIX A EUROPEAN STANDARD ON THE SAMPLING OF WASTES

Working Group 1 (Waste Sampling) of the Comité Européen de Normalisation (CEN) Technical Committee 292 (TC 292 Waste Characterisation) has prepared a draft European sampling standard (prEN 14899²⁶) and supporting advisory documents for the sampling of wastes.

The principal component of the sampling standard is the requirement to prepare a sampling plan, which will become mandatory when the full waste acceptance procedures take effect (on or after July 2005).

A1 FRAMEWORK FOR THE PREPARATION OF A SAMPLING PLAN

The key components to be recorded in a sampling plan are presented in Figure A1, which acts as a route map through the rest of the standard. The sampling plan details everything from the objectives for sampling through to the toolbox of appropriate sampling methods for the type of waste and specific situation. The plan translates and documents the primary objectives of the testing programme (e.g. as set by the Landfill Directive or national legislation) into practical technical goals that take account of the physical state, accessibility and size of the material to be sampled. Secondary objectives can be linked to specific data analysis requirements and a select number of statistical criteria that provide a consistent means of assessing and interpreting testing data. Such tools ultimately provide the means to verify that the testing objective(s) have been met or not.

This diversity of aims affects the location, number, volume and minimum testing requirements for the sampling exercise and it is therefore important that the ultimate aim of the testing programme and any specific objectives are clearly identified to ensure that the samples collected meet these aims. The sampling plan should identify any restrictions or limitations on the reliability of the testing data.

A2 TECHNICAL REPORTS ON SAMPLING

The remaining parts of the standard will not be mandatory but provide guidance on the type, number and locations for sampling, sampling techniques, sub-sampling in the field and sample packaging/preservation/storage/transport/delivery. These reports are as follows:

TR 1 xxxx (WI 292002) Waste – Technical Report on Sampling – Part 1: Information on the selection and application of a basic statistical approach to sampling under various conditions.

TR 2 xxxx (WI 292017) Waste – Technical Report on Sampling – Part 2: Information on sampling techniques.

²⁶ PrEN 14899. Characterisation of Waste - Sampling of waste materials: Framework for the preparation and application of a Sampling Plan.

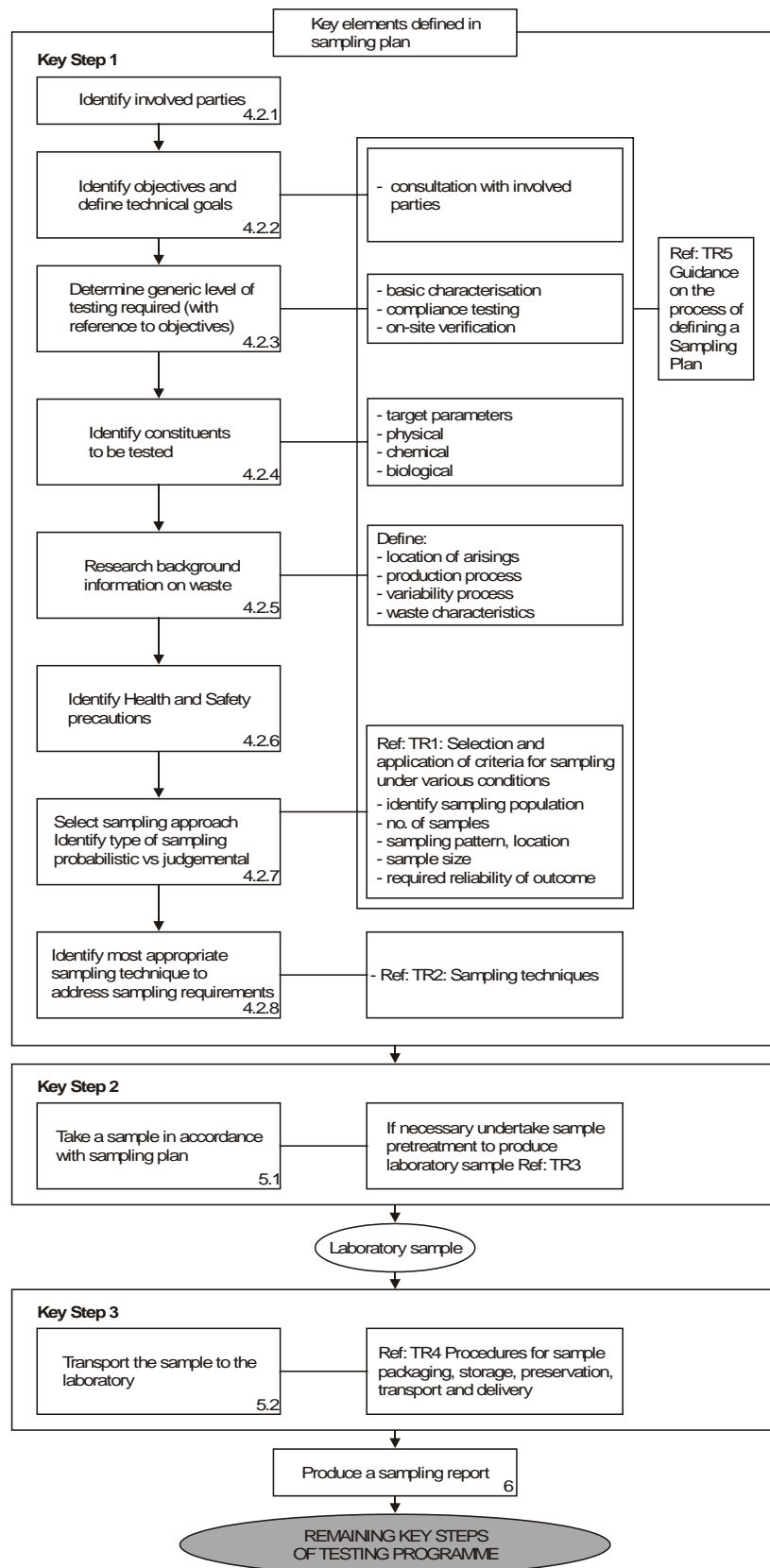


Figure A1 Key components in a sampling plan (after prEN 14899)

Note: The numbers in the boxes refer to the appropriate section within the sampling standard: 'Framework for the Preparation of a Sampling Plan', CEN2004a

TR 3 xxxx (WI 292018) Waste – Technical Report on Sampling – Part 3: Information on procedures for sub-sampling in the field.

TR 4 xxxx (WI 292019) Waste – Technical Report on Sampling – Part 4: Information on procedures for sample packaging, storage, preservation, transport and delivery.

TR 5 xxxx (WI 292041) Waste – Technical Report on Sampling – Part 5: Information on the process of defining the Sampling Plan.

TR1 provides outline guidance on the statistical elements of sampling. It is recognised that in some situations it may be necessary to consult a statistician to elaborate on this advice. TR2 provides a so-called ‘shop shelf approach’ to the collection and handling of different waste matrices (e.g. granular or liquid) in a range of sampling situations (e.g. falling stream, stockpile or drum). Process maps/flow charts are provided at the beginning to guide the reader through the selection of appropriate sampling techniques listed in the document. It is not possible to provide detailed technical guidance for every conceivable sampling situation, so TR5 will provide example sampling plans for commonly occurring sampling scenarios (e.g. specific waste, specific sampling situation).

TR3 and TR4 detail best practice for reducing the size of the sample in the field and provide instructions on returning the sample to the laboratory such that the risk of changing the physical, chemical or biological characteristics of the sample is minimised.

Representative sampling of selected loads has been advocated for the collection of samples for testing against the waste acceptance criteria. The loads should include those expected to contain waste generated under worst-case operational conditions (see Appendix B2). TR2 provides examples of representative sampling methods for a range of waste matrices.

APPENDIX B BACKGROUND TO SAMPLING PROGRAMME DESIGN

B1 A STRATIFIED SAMPLING APPROACH TO LEVEL 1 CHARACTERISATION

It was noted in Section 3 that the waste producer will need random or representative sampling for the main Level 1 characterisation exercise, but should take a targeted worst-case approach when determining which class of landfill the waste can be accepted into.

These incompatible sampling requirements can largely be resolved by the adoption of a stratified sampling scheme. The general principle can be illustrated here with a simple example (though in specific cases it may be necessary to consult a statistician on matters of detail). The approach relies on the waste producer being able to categorise his waste over a suitable time period (3 months, say) into several groups *within* each of which the waste is thought to be relatively homogenous, but *between which* there are substantial differences. The identifiable groups might, for example, be labelled Best, Typical and Worst, and respectively represent about 30%, 60% and 10% of the total waste production. Clearly the degree of success with which the waste can be stratified in this way relies on a good understanding of the processes that give rise to the waste - hence the importance of the initial review of information discussed in Section 3.

The sampling would then consist of taking agreed numbers of samples at random from each of the three groups. Suppose the results (for some parameter of interest) were as follows:

Statistic	Waste groups			Population estimate
	Best (30%)	Typical (60%)	Worst (10%)	
No of samples	5	10	15	30
Mean	10.0	20.0	40.0	19.0
Standard deviation	6.0	14.0	32.0	17.3
Maximum	23.0	52.0	122.0	122.0

For basic Level 1 characterisation, the overall mean estimate for the whole waste population is required. This can be estimated by calculating a weighted average of the means in each group, or 'stratum'. Thus $19.0 = 0.3 \times 10 + 0.6 \times 20 + 0.1 \times 40$. The population standard deviation can similarly be estimated knowing the means and standard deviations in the individual groups (though the calculation is slightly more complicated).

In contrast, for considering the waste in relation to the various Level 2 leaching limits the appropriate measures of performance are then those in the shaded column of the table, as these have been obtained by purposefully targeting the sampling on worst-case wastes. For example, it might be thought useful to estimate the 99-percentile assuming logNormality. Using the worst-case mean and standard deviation, the answer in this example is 161 - a value that is much higher (and so more relevant to the question of appropriate landfill class) than if data representative of the whole waste population had been used.

B2 ESTIMATING THE IMPROVEMENT IN POWER ACHIEVED USING TARGETED WORST-CASE SAMPLING

Figure 4.2 in the main text has shown that a programme of 6 random samples would have a better than 90% chance of detecting a waste that was truly failing for 30% of the time.

Suppose it is known that any problems with a particular waste will be confined to three critical months of the year. A programme of 6 random samples *targeted on those three months* would, as before, have a >90% chance of detecting a waste that was truly failing for 30% of the time. But as the waste can be assumed to have a 0% failure rate for the other nine months, the detectable failure rate *over the year as a whole* is therefore just 7.5%. To detect a waste as marginally poor as this using an annual random sampling programme would be an ambitious goal requiring substantially more than 24 samples (see Figure 4.2 again).

Thus 6 targeted samples (coupled with reliable prior knowledge about the waste) produces better information than will 24 untargeted samples. This is a good example of the cost-effectiveness of targeted sampling.

B3 IMPLICATIONS OF A MARGINAL LEVEL 2 COMPLIANCE FAILURE

The example below provides more detail to the scenario developed in Section 4.3.3.

Suppose that a Level 2 compliance assessment based on 5 random samples produces a single failed sample. The estimated proportion of the waste that is failing the limit is clearly 1/5, or 20%. However, this is only an estimate, and it is important to know how much better or worse than this the true figure might be. These limits can be determined, for any desired confidence level, using the binomial distribution. The table below shows, for various different numbers of samples, 90% confidence limits for the true failure rate.

Number of random samples	Observed number of failures	90% confidence limits for true failure rate	
		Lower limit	Upper limit
2	1	2.5%	97.5%
3	1	1.7%	86.5%
4	1	1.3%	75.1%
5	1	1.0%	65.7%
6	1	0.9%	58.2%
12	1	0.4%	33.9%

Thus, in the case of 1 failure out of 5 samples, the true failure rate may lie anywhere between 1% and 66%.

APPENDIX C TESTING/ANALYTICAL ISSUES

C1 PREAMBLE

In this Appendix the details are provided of the test methods used to determine parameters for which acceptance limit values have been set, both parameters determined in the waste and on eluates derived from the BS EN 12457:2002 batch leaching test. Where European standards have been cited the appropriate 'blue book' (SCA)²⁷ method more commonly used in the UK has been referenced.

While there is considerable experience in the use of batch leaching tests in the UK, many laboratories are less familiar with the leaching behaviour tests that have been developed. Technical background to the tests in terms of potential problems are highlighted here and flow charts to summarise the methods for the end-user of the data are included. In addition, examples of how the data can be presented and used are provided.

C2 FRAMEWORK FOR CHARACTERISATION

The requirements for testing will have been detailed in the sampling plan. CEN/TC292²⁸ have promoted a tool-box approach and different working groups have been charged with different remits (e.g. long and short term leaching etc.). Working Group 6 has prepared guidance (Draft prEN 12920)²⁹ on test selection depending on the waste to be tested and the disposal or reuse scenario to be evaluated. The step-wise approach covers the following:

- question to be answered;
- description of the scenario;
- description of the waste;
- determination of the influence of parameters on release;
- modelling of the leaching behaviour;
- model validation; and
- conclusions and study report.

Additional testing may be needed to determine:

- suitability for energy recovery (e.g. moisture content and calorific value); or
- reuse as a secondary material (e.g. 'total' aluminium or particle size distribution to assess

²⁷ Standing Committee of Analysts

²⁸ Comité Européen de Normalisation, Technical Committee TC292 (Waste Characterisation)

²⁹ Draft prEN 12920:2004 Characterisation of waste – Methodology for the determination of the leaching behaviour of waste under specified conditions (to supersede ENV 12920:1998)

compliance with specifications for engineered products); or for

- environmental and/or human health risk assessments during those operations (e.g. ‘total’ metals for a dust inhalation assessment).

During the Level 1 characterisation, the waste producer will also be required to test for hazard properties listed under the Hazardous Waste Directive. The landfill operator will need to check these properties as part of the Level 2 testing.

It is therefore recommended that, while effort is committed in the preparation of a sampling plan and the collection of a potentially large number of samples, additional sampling objectives are developed.

CEN TC292 have developed a three-tiered framework for waste characterisation:

- total composition, in particular for TOC and other organic components (so called ‘sum parameters’ e.g. PAHs, PCBs or BTEX) for which leaching tests are not yet validated;
- potentially leachable amount (the maximum leachability determined using aggressive agents or conditions in the testing or cumulative quantity leached over a range of liquid to solid ratios);
- actual leachability or mobility under likely exposure conditions (mild extraction). Research has demonstrated that the leaching behaviour of particular constituents in granular waste is governed by a limited number of main solubility controlling influences:
 - *the pH of the solution* - this is dictated by the matrix and is one of the most crucial parameters in determining solubility of contaminants;
 - *redox status and presence of complexing agents* (chloride, organic or dissolved organic compounds etc.) - these influence the release of heavy metals in particular;
 - *liquid to solid ratio (L/S)* - this is important because it can relate results to a time scale through the rate of infiltration;
 - *major element chemistry* - because the major elements dictate the pore water composition, which in turn controls the trace element leachability.

The assessment of leachability is largely limited to inorganic parameters as assessments of the leachability of many organic components are susceptible to many sources of analytical error and a suitable method has yet to be validated. However, the leachability of dissolved organic carbon (DOC) at neutral pH is believed to be a robust indicator of the stability of organic matter in the waste (see Appendix C4.1.2)).

C3 SAMPLE PREPARATION FOR TESTING

Traditional sample preparation for ‘totals’ analysis has involved drying and grinding a relatively small sub-sample to a fine powder. However, a larger quantity of prepared sample is required for most leaching tests and, in most instances drying above 40°C and grinding must be avoided. Most leaching tests have specific sample preparation requirements and the correct preparation of the samples is therefore critical to the correct interpretation of the

leaching test data.

For the batch tests, 95% of the particles in the sample must be < 4mm. If the material is greater than 4mm and damp it may require air-drying (or oven-drying at below 40°C) to facilitate sieving and crushing of the >4mm portion prior to recombination with the <4mm fraction. The moisture content and portion not passing through a 4mm screen (e.g. metal) must be quantified and reported. The sample must be large enough to allow for both these stages, for example the mass of an incineration bottom ash sample may be reduced by 80% after moisture content and non-crushable elements have been removed. In addition, the residual moisture content at 105° C must be determined so that an appropriate quantity of material is leached to give the correct dry residue content.

The pH dependence test requires size reduction to <1mm rather than <4mm and the maximum availability leaching test does require grinding (to <350 µm) as a worst case leaching scenario is being simulated.

Further guidance on the preparation of test portions from laboratory samples has been prepared by CEN³⁰.

The remainder of this Appendix presents information on leaching test methods, how leaching test data can be presented (Appendix C4.3) and information on methods for the analysis of wastes and eluates (Appendix C4).

C4 LEACHING TESTS

C4.1 Method summaries

Table 3.3 presented the aims of the batch compliance test and a selection of leaching behaviour tests. The test methods are summarised below.

C4.1.1 Batch leaching tests (BS EN 12457:2002)

The minimum leaching requirement for testing for waste acceptance is the European compliance leaching test BS EN 12457. Three parts are of relevance to the UK and all are conducted on waste which has been prepared so that 95% of the sample passes through a 4 mm sieve. The three parts differ only in the liquid-to-solid ratios (L/S) at which the test is conducted and number of leaching steps.

- BS EN 12457 Part 1: single stage batch tests at liquid to solid ratio 2 l/kg with particle size below 4mm. (Single-step leaching at L/S 2 l kg⁻¹ dry residue for 24 hours);
- BS EN 12457 Part 2: single stage batch tests at liquid to solid ratio 10 l/kg with particle

³⁰ PrEN 15002:2004 Characterisation of waste - preparation of test portions from laboratory samples. Consultation draft July 2004.

size below 4mm. (Single-step leaching at L/S10 1 kg⁻¹ dry residue for 24 hours);

- BS EN 12457 Part 3: two stage batch tests at liquid to solid ratio of 2 l/kg and 8 l/kg with a high solid content and with a particle size below 4mm. (Two-step leaching at L/S2 1 kg⁻¹ dry residue for 6 hours and 8 l kg⁻¹ for 18 hours; the results are aggregated to provide cumulative L/S10 leaching information).

With the exception of L/S ratio and number of leaching steps, the general test conditions for all three parts are the same. Deionised water is used as the leachant (leaching medium) and the tests take a total of 24 hours each. As 95% of the sample must be <4 mm, preliminary drying, sieving and/or crushing may be required. Additional material is added to compensate for any residual moisture content determined on a separate sub-sample at 105°C (The test portion itself is not dried at temperatures above 40°C). The test requires continuous agitation of the eluates using an end-over-end shaker with no pH control. The final pH of the eluates is at, or close to, the actual pH of the test material, similar to the pH of the waste's porewater if stockpiled or monofilled.

The eluates are separated from the sample by filtration or centrifugation, sub-sampled and preserved as appropriate. The two step test yields two eluates that are submitted for separate analysis. As for most of the tests above, the eluates are subjected to the determination of at least pH, electrical conductivity, dissolved organic carbon and a range of inorganic determinands. The results of the determinations are recalculated as mg leached per kg dry residue at L/S2 and L/S10. The latter makes appropriate allowances for the material leached from the first leaching step.

For basic characterisation, eluate determinations should not simply be limited to pH and the parameters for which limit values have been published. Major element chemistry can be a major controlling influence on the solubility of minor and trace metals and therefore inclusion of major cations and other parameters would be advised. (In addition, quality assurance checks may necessitate the occasional inclusion of an ion balance suite) Assessment of the characterisation data may allow a reduction in the list of determinands for compliance monitoring. (Information on test methods for eluate analyses is presented in Appendix C5).

In most instances the two-step leaching test (BS EN 12457-3:2002) will be undertaken. The BS EN 12457-3 batch test is mandatory, as the L/S 10 leached concentrations in mg kg⁻¹ are directly compared with the waste acceptance criteria limit values to assess the class of landfill at which the waste may be accepted. The test data will therefore have a role in assessing waste classification at Level 1 and determining compliance with waste acceptance criteria at Level 2.

However, there are a number of situations where the use of the single-step leaching tests (BS EN 12457-1 and 2) will be used; these are when the use of a serial batch leaching test is precluded on technical grounds and when single-step L/S10 data are collected as part of screening exercise during characterisation, in addition to undertaking the two-step test:

- **Screening exercise as part of characterisation:** An important part of the Level 1 characterisation will be an assessment of waste variation during which the factors controlling waste quality will be determined. This may involve large numbers of samples over a prolonged time-frame depending on the operational/seasonal cycles which may need to be taken into account (see Section 3.3). While 'total' concentrations will provide a

relatively inexpensive indication of waste quality, these often bear no relation to the leaching behaviour of the waste material and are not a viable alternative to carrying out leaching tests. The single-step leaching test BS EN 12457-2, which generates L/S10 data only on samples of particle size below 4mm, is a useful test for such screening exercises on a potentially large number of samples. A smaller sub-set of these samples, composites or fresh samples collected according to a sampling plan informed by the data from the screening exercise, can be subjected to other testing, including the two-step L/S2 and L/S2-10 test (BS EN 12457-3).

- Technical reasons precluding use of BS EN 12457-3:2002:
 - *serial batch tests not feasible*: some sludges and organic or clay-rich samples generate eluates which are unsuitable for multiple-step tests due to blinding of the filters during separation of the sample during the first leaching step. If centrifugation is also unsuccessful, and L/S2 leaching is technically possible (see below) single-step leaching at both L/S2 (BS EN 12457-1) and L/S10 (BS EN 12457-2) may be undertaken, although the limitations of the single-step data must be recognised. Alternatively, testing may be restricted to single-step testing at L/S10 (BS EN 12457-2).
 - *sludges with a dry solids content below 50%*: in the case of very low dry solid sludges where L/S2 cannot be achieved, the single-step L/S10 batch test (BS EN 12457-2) may be undertaken. In some cases it may still be possible to undertake serial leaching by analysing the porewater (already in equilibrium with the waste) and calculating this natural L/S ratio. The amount of leachant required to establish L/S10 can then be calculated. Such deviations to the standard method are permitted but must be reported with the data.

Where the single-step L/S10 test (BS EN 12457-2) is used instead of the two-step test (BS EN 12457-3) full justification must be provided with the data and should be agreed with the Environment Agency before commencing the testing.

It should be noted that non-leaching test methods are also required to determine waste acceptance to landfill, notably for the determination of acid neutralisation capacity (see Appendix C4.3.4), total organic carbon and/or loss on ignition and other organic contaminants (Appendix C5).

C4.1.2 Other leaching tests recommended for basic characterisation

Although only the batch tests are mandatory for granular wastes, interpretation of waste characterisation from the data is limited. When placed in the context of data from other characterisation tests information on the leaching behaviour, and the potential for predicting or modifying the leaching potential of the waste is increased.

A number of characterisation leaching tests can assist predictions of leaching behaviour of granular wastes under changing conditions which might prevail during waste treatment or within the landfill, for example with increasing liquid to solid ratio or different porewater pH domains, as summarised below.

Flow charts are provided in figures D1 to D4 to indicate to the end-user of the data the

complexity of the procedures. These are not intended to be used as method sheets by the laboratory – the analyst must refer to the full standard.

- *Maximum availability leaching test*: (EA NEN 7371:2004)³¹ A two-step agitated leaching test conducted on ground materials, at high liquid to solid ratios (L/S50 at pH 7 and L/S100 (cumulative) during the second step at pH 4) representing aggressive conditions. The two eluates are analysed separately but the results are reported as mg kg⁻¹ dry residue for the whole test. The test conditions present a worst case leaching scenario rather than the conditions that might be expected to prevail in a landfill, for example. While ‘total’ metals determined on a hot acid digest represent potential release under extreme geological scenarios and timescales, the ‘availability for leaching’ represents potential release under aggressive environmental conditions. Availability for leaching is therefore a useful source term characteristic when assessing the potential for release of a contaminant to an aqueous environment, particularly when assessing the quantity of potentially leachable contaminant remaining after a waste has been leached at L/S10.

The quantity of acid used to maintain pH at 7 and 4 during the test can be used to calculate acid neutralisation capacity (Appendix C4.3).

A translation of this standard is available on the Environment Agency web-site.

- *Upflow percolation test* (prCEN/TS 14405:2004)³². Sequential flushing of a column of granular material with acidified water is carried out at increasing liquid to solid ratios (0.1-10). The leachant (which can be site specific) is passed through a vertical column of the waste material in upflow mode, so that the column of waste is water saturated. The eluate is collected in fractions at increasing liquid to solid ratios (from as low as 0.1 l kg⁻¹), filtered and analysed. A flow rate which fulfils localised equilibrium conditions within the waste matrix must be used. Column tests are well suited to describe the progression of leaching in the liquid to solid ratio range of 0-2 l kg⁻¹ and complement batch leaching tests of the type described below. The results of the test are reported as a function of L/S ratio as mg released per litre of eluate and mg released per kg of waste material (dry matter). The method is summarised as a flow chart in Figure C1.

³¹ EA NEN 7371:2004 Determination of the availability of inorganic compounds for leaching – solid earthy and stony materials. Environment Agency translation of the Netherlands Normalisation Institute Standard NEN 7371. Available on Environment Agency web-site.

³² PrCEN/TS 14005 : 2004. Characterisation of waste – leaching behaviour tests – Upflow percolation test (under specified conditions).

prCEN/TS 14405: Upflow percolation test

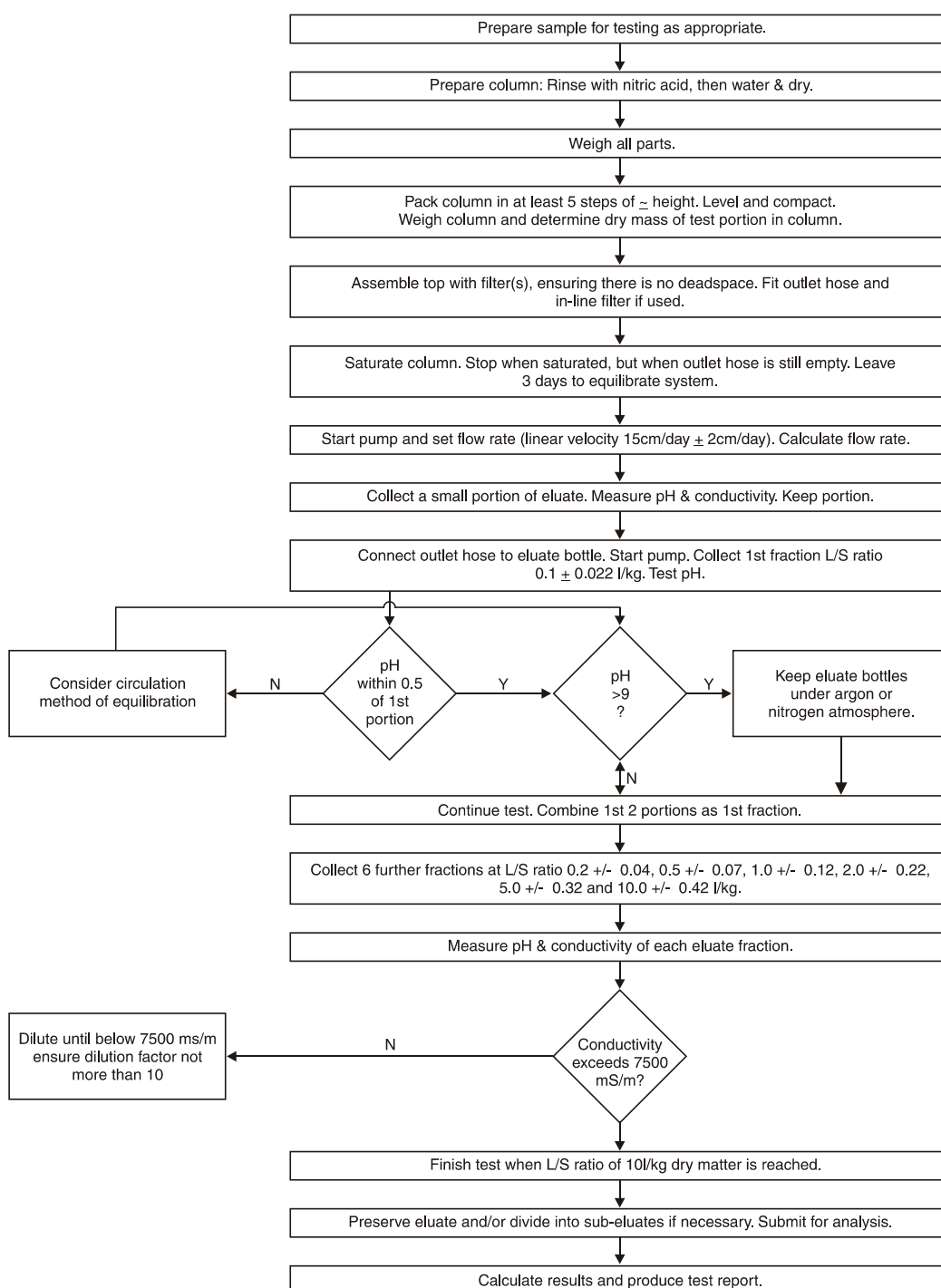


Figure C1 Outline of prCEN/TS 14405:2004 Upflow percolation test

- *pH dependence tests*: Replicate samples of granular materials are leached for 48 hours. Each sample is controlled at a specific pH value between pH 4 and 12. Two methods are available:
 - prCEN/TS 14429:2004³³ Influence of leaching with initial acid/base addition, (where pre-determined amounts of acid or base are added to reach the target pH value) (summarised in Figure C2), and
 - prEN 14997:2004³⁴ Influence of leaching with continuous pH control (to reach equilibrium by the end of the test) (summarised in Figure C3).

The samples are size-reduced to <1mm to accelerate leaching. The tests provide information on:

- pH dependent leaching: The resulting leaching curves demonstrate the impact of adjusting the pH of the material outside its normal pH domain. Of particular interest is the prediction of leachability under the likely final leachate pH conditions of the landfill. For most landfills (other than industrial monofills) this would be expected to be between pH7 and 8.
- Acid/base neutralisation capacity: The quantity of acid/base used to achieve each leaching step is used to calculate ANC/BNC which are presented for each pH value both as mg leached per litre of eluate and as the quantity of acid or base added per dry kg⁻¹ of material (expressed as mol H⁺kg⁻¹ or negative mol H⁺kg⁻¹ for base addition).

³³ PrCEN/TS 14429 : 2004. Leaching behaviour tests – Influence of pH on leaching with initial acid/base addition.

³⁴ PrEN 14997 : 2004. Leaching behaviour tests – Influence of pH on leaching with continuous pH control

prCEN/TS 14429: Influence of pH on leaching with initial acid/base addition.

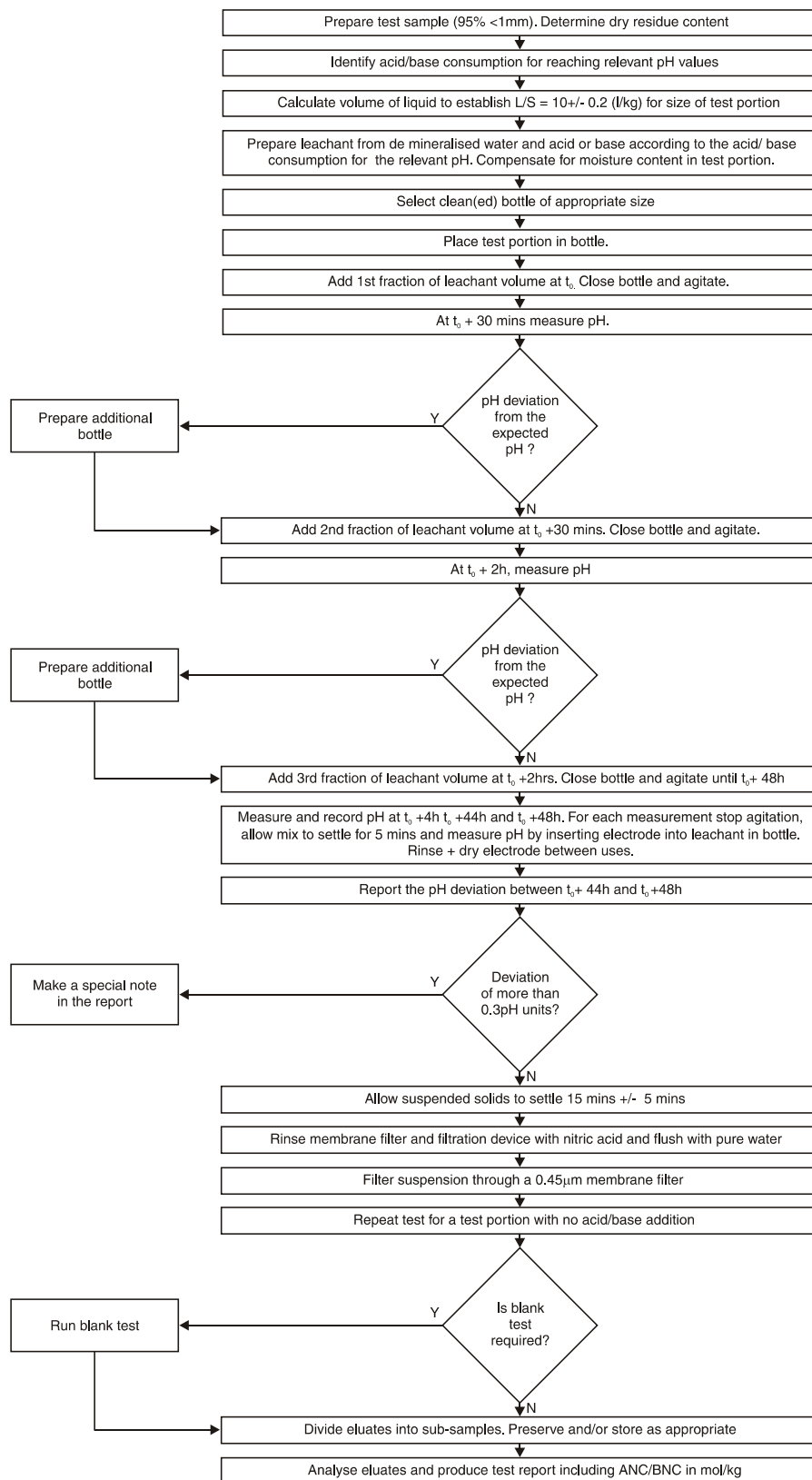


Figure C2 Outline of PrCEN/TS 14429 : 2004 Influence of pH on leaching with initial acid/base addition

prEN 14997: Influence of pH on leaching with continuous pH control

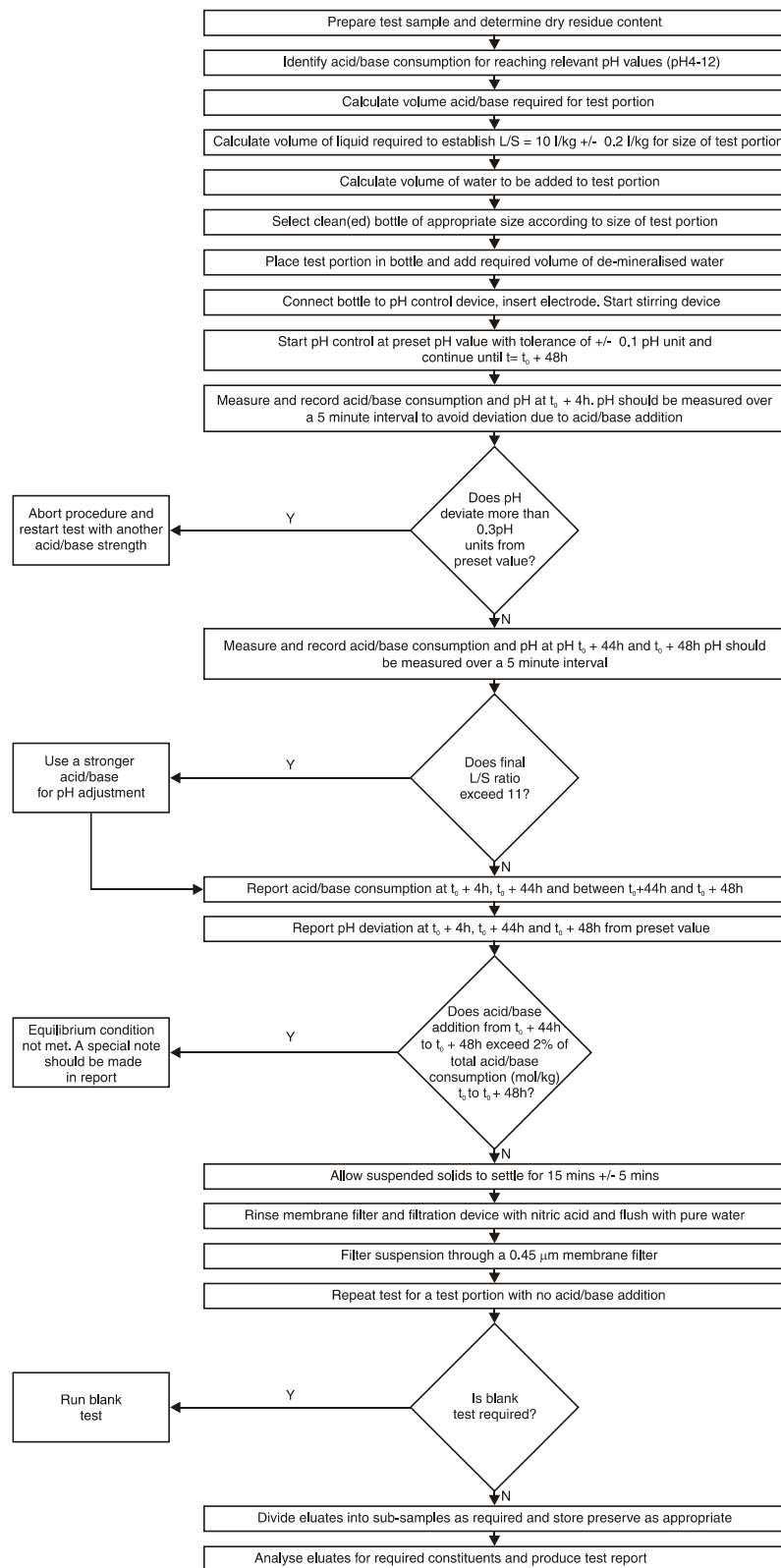


Figure C3 Outline of prEN 14997:2004 Influence of pH on leaching with continuous control

- *Tank test* (monolithic wastes) (EA NEN 7345:2004). This test is used to assess leaching of solidified materials. Cubes or cores etc. of solidified material are subjected to sequential extraction at in pH neutral deionised water over a maximum of 64 days. Leachant is renewed at 7 time intervals. Results are usually reported with reference to the surface area of the sample as mg cm² rather than mg kg⁻¹ leached.

The reader is referred to the English translation of the standard, which is available on the Environment Agency web-site. The test methodology is summarised in Figure C4, with some deviations from the test method based on operational experience (e.g. measurement of pH and electrical conductivity on unfiltered eluate, automatic weighing of sample at each leachant renewal stage).

As a landfill waste acceptance test for monolithic wastes, the 4-day and 64-day cumulative mg m⁻² results are compared with the compliance and characterisation limit values presented in Table 2.3. The standard also provides a series of calculations for determining whether leaching has been dominated by diffusion or other leaching mechanisms. If diffusive leaching has not occurred, the status of the waste as a monolith is questionable and the waste should be crushed and tested as for granular wastes.

- *Compliance test for monolithic wastes*. A draft CEN/TC2 compliance test (WI 292010) has been prepared based on 3 leaching steps conducted within a 48 hour period. The leaching container is sealed and a vacuum applied for 15 minutes to ensure that the pores of the test sample are saturated. Gentle stirring is then applied from the remainder of the test with eluate removal and leachant renewal after 6, 24 and 48 hours from the start of the test. The eluates are filtered and analysed and data reported on a mg/cm² basis. Data interpretation on a mg kg⁻¹ basis could be compromised by variation in the leachant volume to surface area ratio and therefore sample dimensions must be controlled within tight tolerances.
- *DOC at pH 7.5-8*. Under final storage conditions, landfill leachate would be expected to be in the range pH 7.5-8. Leachability assessed at natural pH (e.g. using BS EN 12457) may be very different from the leaching behaviour that might be predicted to occur at pH 7.5-8. Where DOC release at natural pH exceeds the waste acceptance criteria for landfill, and the pH is above or below the predicted final leachate pH, a second leaching test can be undertaken.

The test is still in draft form within CEN TC292/Working Group 6 but is based on the pH dependence test (prCEN/TS 14429:2003). In essence it is a 48 hour deionised water shake test with pH control at 7.5-8, under a liquid to solid ratio of 10 l kg⁻¹ dry matter.

The DOC mobilised at neutral pH should reflect the level of low molecular weight organic substances resulting from the degradation of organic matter. DOC release outside a neutral pH range could include organic micropollutants which indicate chemical contamination rather than biological activity.

Background work in this area has been carried out by Hans van der Sloot under the EU funded project, Harmonisation of Leaching/Extraction tests (SMT4-CT96-2066) and by Oonk and Welders, 1999).

NEN 7375: Diffusion test

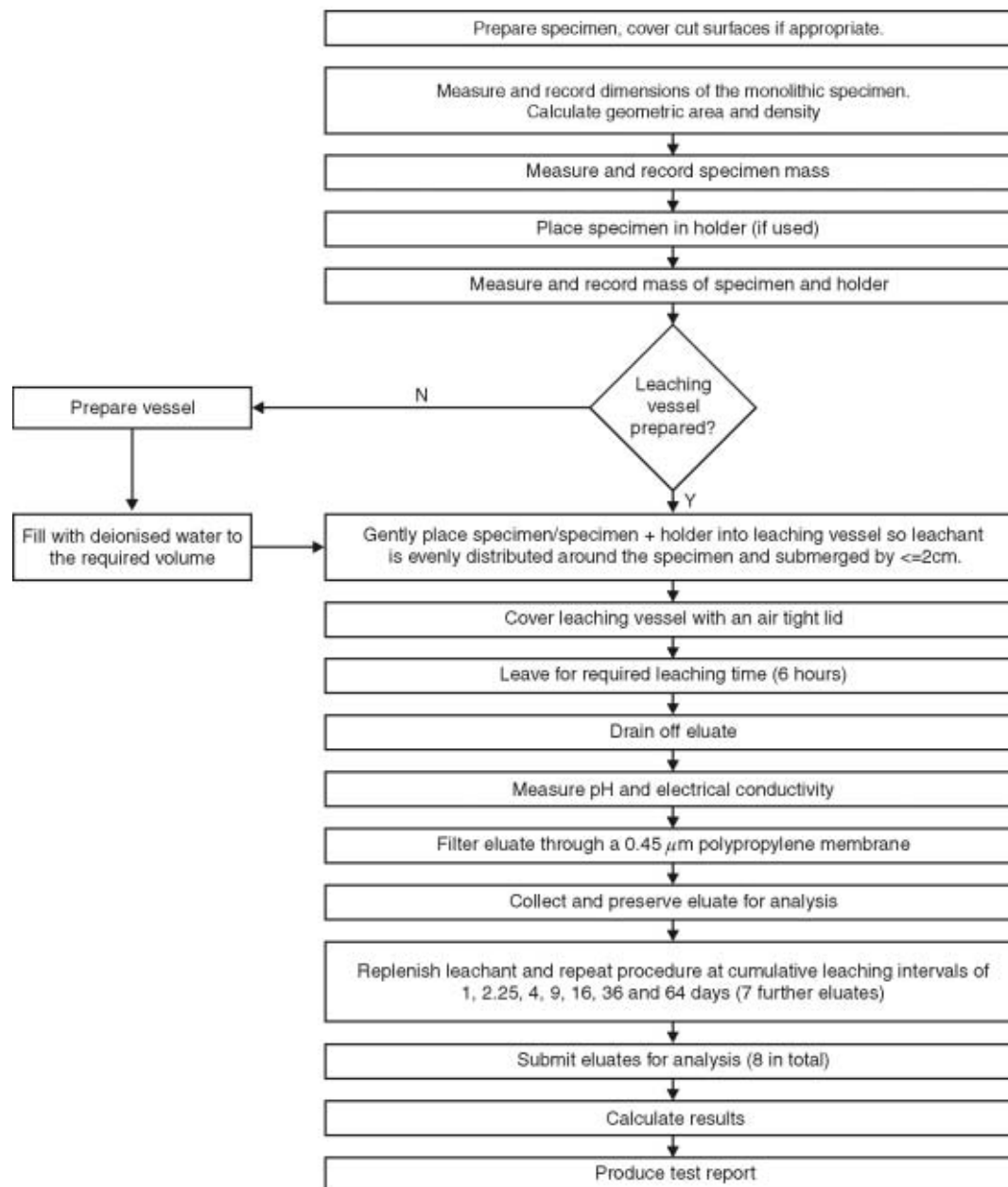


Figure C4 Outline of EA NEN 7375:2004 Diffusion test for monolithic materials

C4.2 Practical issues

During the preparation of the sampling plan, the project manager should have discussed laboratory procedures with the analyst. It is recommended that the following issues are raised and, if required, details are reported with the analytical data.

- *Leachant flow rate for upflow percolation test:* Preliminary testing of the waste material is required to ascertain the optimal flow rate of leachant through the packed column to deliver the correct liquid to solid ratios at practical times. The pre-test also allows suitability of the waste for the tests to be assessed as it is not compatible with all sample matrices. An equilibration period (at least 24 hours) is required between introducing the leachant and pumping out the first eluate. During this time materials with pozzolanic properties can set hard, destroying the column. Samples with a high clay content may blind the filters at the top of the column preventing flow out of the column. Some guidance on suitability is provided in the standard.
- *Volume of leachant:* The pH dependence test also requires preliminary testing to be undertaken with the test material. The acid or base neutralisation capacity of the test material and the quantity and strength of acid required to maintain the appropriate pH during each step of the pH dependence test must be determined. This ensures that the liquid to solid ratio is not compromised, for example, by the addition of a large volume of acid that is too weak.
- *Filtration:* The eluates are usually separated from the sample by filtration. The correct filter medium (appropriate to the required determinands), poresize and cleaning methods are required.
- *QA/QC:* As with any laboratory procedure, QA/QC samples are essential and the analyst should arrange routine AQC replicates and blanks. A batch blank (leaching vessel containing leachant only) should be included with the test samples at appropriate intervals to demonstrate lack of contamination from apparatus, reagents or the laboratory air.
- *Reporting units:* The analyst should report data from the batch tests as mg kg⁻¹ leached (on a dry residue basis) except where inappropriate (e.g. pH), to allow rapid comparison with regulatory limits. Suspect results should be dealt with as soon as possible, and, if necessary, repeat determinations should be performed before the eluates are discarded.

C4.3 Interpretation of Leaching Test Data for Granular Wastes

C4.3.1 Characterisation tests

A characterisation report should present the salient details from the sampling plan, in particular any sample preparation techniques, the analytical methods employed as well as previous data or literature values if relevant. The full approach to data evaluation cannot be presented here and a detailed description of how pH-dependent leaching can be interpreted is provided in CEN 2002d. However, two main aspects affecting release from granular materials have been combined in a unified approach to leaching that has been developed by CEN/TC292 and its working groups as indicated in Figure C5.

Release as a function of liquid to solid ratio from the upflow simulation test is shown in the left hand plot. Release as a function of pH as evaluated using the pH dependence test is shown in the right hand plot. In this example the two step batch test (BS EN 12457-3) has also been undertaken. From the combination of the graphs, conclusions can be drawn for conditions other than those tested in a single batch test (such as BS EN 12457).

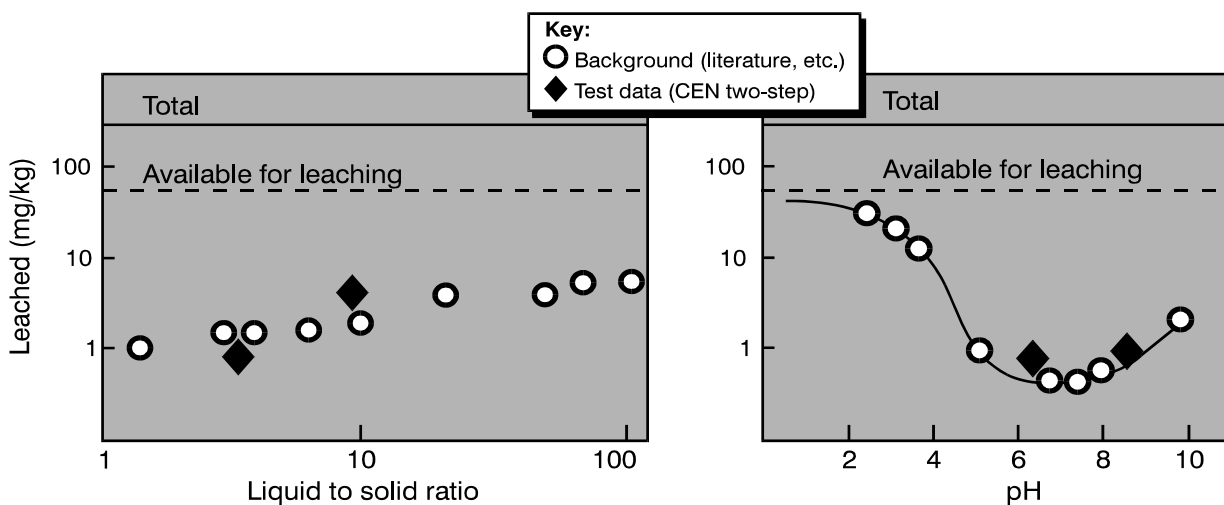


Figure C 5 The CEN TC292 model for comparing contaminant release as a function of pH and L/S ratio to determine controlling influences over leaching behaviour

The two-step serial batch test (BS EN 12457-3 at L/S2 and L/S2-10) allows the recognition of the following leaching behaviour:

- *Where constituents are so readily soluble that they are almost completely leached out after the first leaching cycle* - In this case the amount released is the total available for leaching. Peak concentrations in porewater can be estimated for these constituents through calculation. This will be indicated by a ratio of L/S2-10 to L/S2 data of approximately 1.
- *Where the leaching of constituents is solubility controlled* - In this case the concentration in the eluate is about equal in both extractions and the amount released increases proportionally to the liquid to solid ratio until the leachable fraction is depleted.
- *Where constituents show a sharp increase in concentration in the second leaching cycle* - This delayed release, caused for example by the wash-out of a solubility controlling phase, indicates the potential for an uncontrolled condition. Identification of the factors which cause this behaviour is necessary through more rigorous 'basic' characterisation tests.

C.4.3.2 Compliance tests

Interpretation of isolated test results from a single batch extraction without a framework of reference is very limited and will only allow a direct comparison with regulatory limits on a pass/fail basis. The conditions which develop in a laboratory leaching test may not be at all representative of the conditions that may prevail in a reuse or disposal scenario. But an approach to testing in which key aspects of leaching behaviour are integrated and full use is made of previous knowledge of the material will lead to a situation where the number of false positive and false negative decisions can be reduced.

In Figure C5, background data from full characterisation tests can be compared with the less rigorous batch test approach. When the data fall within the trends shown by the background data, more confidence can be placed on the batch test approach and solubility controls can be confirmed as the main influence over leaching. In other words, the batch leaching test results only reveal a small part of the leaching story. But, the combination of the tests provide more insights into how factors influence release, which in turn can be used for controlling/managing the release.

C4.3.3 Example

Figure C6 highlights the benefits of undertaking leaching behaviour tests in addition to the compliance test during the level 1 characterisation. The results of the compliance leaching test data at L/S2 and L/S10 from BS EN 12457-3 and L/S10 from BS EN 12457-2 are plotted against eluate pH. The sample fails the L/S10 limit value for hazardous wastes at below pH 6.5 (dotted line). However, the leachability of Zn is clearly pH dependent and behaviour of the waste under the ultimate pH conditions of a landfill can now be predicted.

C4.3.4 Acid neutralisation capacity (ANC)

ANC/BNC (acid/base neutralisation capacity) is an indicator of the buffering capacity of the waste. This value is determined from the acid/base consumption needed to attain a fixed pH and is therefore a by-product of pH-controlled leaching tests such as the pH dependence test or the maximum availability test. It is important to identify the pH value at which the ANC/BNC has been determined.

The ANC/BNC is calculated from the volume of acid (or base) used to attain a specific pH value, allowing for the moisture content of the material as follows:

$$ANC_{\text{pHx}} = V_{\text{A/B}} * \text{Molarity}_{\text{A/B}} * M_{\text{dr}}$$

where ANC_{pHx} = ANC (or BNC) at pH value = x (mol dry kg^{-1})

$V_{\text{A/B}}$ = volume of acid added to attain pHx (litres)

$\text{molarity}_{\text{A/B}}$ = molarity of acid/base

M_{dr} = mass of sample (dry weight) (kilogrammes)

Two of the leaching tests described in C4.1.2 are pH controlled tests. ANC at pH 7 and 4 can be established from acid consumption in the maximum availability test and ANC/BNC over a full range of pH values (4-12) can be determined from the pH dependence test. A draft test is

also in preparation solely for the determination of ANC/BNC.³⁵ Some examples of ANCs for a range of inorganic wastes are provided in Table C1 and C2.

³⁵ WI 292046, CEN 2004. Characterisation of waste – acid and base neutralisation capacity test.

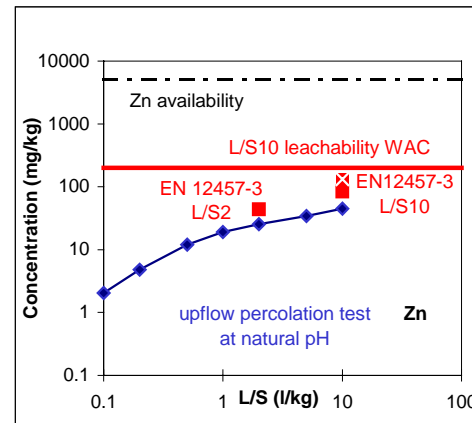
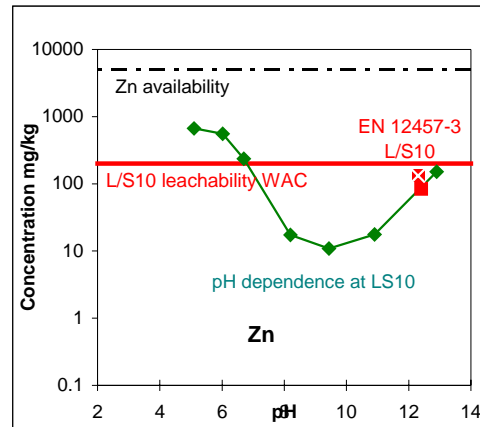
Maximum availability for leaching NEN 7371

Maximum quantity of contaminant that can be leached under worst case environmental conditions (as opposed to 'total' concentrations).
2-step leaching at L/S50 and cumulative L/S100 at pH7 and 4, on ground material is very aggressive. Calculated value in mg/kg release.
Acid addition is used to calculate ANC at pH7 and pH4. See note on ANC.

BS EN 12457-2 compliance test. EN 12457-3 compliance test

Single-step L/S10 2-step L/S2 and cumulative L/S10 (or L/S2-10)
(red square = L/S 2 value, red square/white cross = L/S10 values)
Particle size <4mm, 24 hour, unbuffered DIW. Eluate pH controlled by test sample i.e. at 'natural' or 'own' pH. Eluate pH must be measured to place data in context vs pH (as in left-hand plot). Not meant to represent field conditions. Extrapolate L/S2 and L/S10 slope to availability line to predict (on order of magnitude basis), the L/S at which supply of available contaminant would be exhausted and leaching cease.

ANC/BNC - acid base neutralisation capacity. Quantity of acid/base used to maintain a set pH in mol/kg. E.g. low ANC means weak resistance to external influence on porewater pH. Leachability predictions at 'own' pH could be compromised by changes in landfill leachate pH.



L/S - liquid to solid ratio in l/kg (litres of leachant/dry kg sample). E.g. at L/S2 mass of liquid is twice mass of dry sample. Low L/S is representative of landfill scenario, higher L/S represents uncontrolled infiltration e.g. in stockpile, or during washing process.

pH dependence test prEN 14429.

<1mm particles, 48 hour, pH controlled between pH4-14. Each step at L/S10
Not cumulative leaching. Enables leachability to be predicted if porewater pH rises/falls through treatment or in landfill.
Acid/base addition used to calculate **ANC/BNC**.
For very alkaline/acid wastes, DOC value for eluate from pH7.5 step can be compared with DOC limit if EN 12457 eluate (natural pH) failed the limit value.

Upflow percolation test prCEN/TS 14405.

<4mm particle size material packed in a column. Leachant (unbuffered DIW) equilibrates for 48 hours before pumping in UPFLOW mode (to allow uniform saturation). Eluate pH controlled by test sample itself. Eluate collected for analysis at cumulative L/S of 0.1, 0.2, 0.5, 1, 2, 5 & 10 l/kg, l/kg, initial eluate = C0. Time to attain specified L/S is dependent on permeability of material/pumping rate. No pH control, leaching reflects continued flushing/infiltration of water at own pH, not influenced by other wastes, i.e. represents a monofill/single waste stockpile situation.

Figure C6 Example of compliance leaching test data placed in context with characterisation data and waste acceptance criteria

Table C 1 Acid neutralisation capacities determined from the maximum availability leaching test (NEN 7341:1995³⁶)

Waste	ANC at pH 7 mol kg ⁻¹ (dry weight)	ANC at pH4 mol kg ⁻¹ (dry weight) ¹
Foundry sands	<0.03	0.02-0.3
Steel slags	0.2-4	2-5
MSW incineration bottom ash	0.9-1.5	2-4
PFA	0.1-0.5	0.2-0.6
MSW incineration air pollution control residue	8-10	9-12
Cement kiln dust	2-10	9-12

Source: Environment Agency 2000³⁷

Table C 2 Examples of ANC values obtained from the pH dependence test (prCEN/TS 14429: 2004)

MSW IBA		Nickel sludge		Contaminated soil		Metallurgical slag	
pH	mol kg ⁻¹	pH	mol kg ⁻¹	pH	mol kg ⁻¹	pH	mol kg ⁻¹
3.5	2.65	3.6	2.3	4.0	0.191	4.0	0.068
4.0	1.80	4.5	1.3	5.0	0.093	5.6	0.033
5.7	1.50	5.6	0.2	6.0	0.042	5.6	0.013
7.3	1.0	7.2	0.0	7.0	0.003	7.6	0.004
8.8	0.63	8.1	-0.2	8.1	-0.027	9.0	0.002
9.6	0.50	9.0	-0.6	9.0	-0.053	10.0	0.000
10.7	0.25	10.9	-1.5	10.5	-0.119	11.0	-0.021
11.9	0.13	12.0	-2.0	12.0	-0.361	12.0	-0.974

Source prEN 14429:2004.

Notes:

1. A value of zero indicates the step at which the material was tested at its own pH, i.e. without acid or base addition
2. BNC is indicated as a negative ANC

However, an initial ANC/BNC may be calculated before commencing the pH dependence test to ensure that the molarity and volume of acid are correct for maintaining L/S10 l kg throughout each step of the test. This initial calculation allows the extremes of neutralisation capacity to be established (e.g. pH 4 and pH 12 as well as at pH 7).

³⁶ This test has now been superseded by NEN 7371:2004.

³⁷ Evaluating the environmental performance of wastes with respect to their classification under the UK classification scheme. Environment Agency R&D Technical Report P1-297/TR.

The ANC/BNC that is expected to prevail under the predicted final pH leachate conditions are of most interest. The ultimate quality of landfill leachate would be expected to be approximately pH7-8 and therefore the difference in ANC/BNC at natural pH (0 mol/kg) and pH 7 should be calculated (or at predicted final leachate pH for the landfill if not neutral pH). For most wastes the difference will be <2 mol/kg (see Table C1 and C2). Mixing a high ANC/BNC waste of extreme pH with a less well-buffered waste of different pH should be avoided unless the pH dependent leaching of both wastes has been fully assessed. This is because the former waste will swamp the local porewater pH conditions, compromising any leachability predictions based on natural pH leaching tests (e.g. BS EN 12457 batch tests or prCEN/TS 14405 upflow percolation test). Where a waste exhibits a difference of more than 3 mol/kg between the ANC recorded at natural pH and the ANC recorded at pH 7 and it is to be accepted in cells for hazardous (or stable, non-reactive hazardous) wastes with other waste streams of less extreme pH and ANC, pH dependent leaching of all wastes must be fully assessed, and potential impact upon the leachate quality evaluated as part of the site risk assessment.

C4.3.5 Total and Dissolved Organic carbon

A limit value has been set for total and dissolved organic carbon in wastes going to hazardous and inert waste landfills. This is determined by combustion in oxygen after removal of the inorganic carbon as in Appendix C5. Eluates are filtered through a 0.45 µm filter either at the time of separation from the solid material or prior to analysis, therefore it is dissolved organic carbon (DOC) rather than total organic carbon (TOC) which is determined in filtered eluates.

During the BS EN 12457 test, pH is not controlled. The final pH of the eluate is therefore very close to the pH of the material itself ('own pH' or 'natural pH'). A limit value has been set for DOC in eluates. However, EU-funded research undertaken by van der Sloot for the Network for the harmonization of leaching extraction tests, (SMT4-CT96-2066) has shown that DOC at neutral pH tends to reflect the degradability of the material, i.e. the more DOC that is released at neutral pH, the more 'bio-reactive' is the waste. As it is the organic carbon related to the biodegradability of the waste rather than from organic micropollutants which are of greatest concern for waste acceptance to landfill, a draft method is being considered by CEN TC292/WG6 to establish the DOC on eluate generated at neutral pH. The draft method is based on the pH dependence test. As described above, a requirement of the full test is a preliminary assessment of acid/base consumption at extreme and neutral pH. This therefore generates an eluate at pH 7 that is subsequently available for analysing for DOC regardless of whether a pH 7 eluate is generated from the full pH dependence test.

C5 ANALYSES ON WASTE AND ELUATES

A large number of analytical test methods exist for determining the concentrations of the various determinands of interest in extracts from leach tests. Most of these were originally developed for the analysis of water samples, but can often be used for waste eluate matrices.

The European Standards prEN 13370:2002 and ENV 12506 are intended to define the analytical methods to be used for eluates obtained from waste characterisation tests.

Tables C2-C4 give a summary of the principles of the determinations and the equivalent recognised UK 'Blue Book' methods (Methods for the Examination of Waters and Associated Materials, HMSO) which would be expected to give similar analysis results.

If the methods listed below are inappropriate because of interferences or inadequate limits of detection, other validated methods such as GF-AAS or ICP-MS can be used (and in some cases are actually preferable). For ICP-MS techniques see *ISO 17924 parts 1&2* and 'Blue Book' method

Inductively Coupled Plasma Spectrometry, 1996

It is the responsibility of laboratory performing the analyses to ensure that the methods are suitable for the waste eluate being analysed, as there are a large number of potential interferences, which are outlined in the individual standards.

The standard based on acid digestion (EN 13656) is a suitable method for many but not all waste matrices. For refractory matrices (e.g. silicates, carbides, oxides) or where residues remain after acid digestion, alkali fusion can be effective in bringing the whole sample into solution. Draft PrCEN/TR 15018³⁸ provides methods for the use of a range of such techniques. In addition, alternative waste composition methods such as X-Ray fluorescence and X-Ray diffraction can be more appropriate in certain circumstances. A CEN standard for the former technique is in preparation³⁹.

C5 QUALITY ASSURANCE

Where waste producers or landfill operators conduct their own landfill waste acceptance testing, the laboratory must operate to a written quality assurance system, with a minimum:

- standard operating procedures, approved for use by management, for all of the test methods in use;
- validation data generated in the waste producer or landfill operators' own laboratory to show the performance of the method;
- testing of ongoing quality control samples, to demonstrate that the method remains under control whilst in use;
- evidence of the initial training and approval, and the ongoing competence of the staff performing the test; and
- independent third party audit of the in-house quality system at least annually.

The Agency prefers the in-house quality system to be externally accredited or certified to an international standard e.g. ISO 9001 or ISO 7025 and would expect the laboratory to be working towards one of these. It is good practice for a selection of samples (10% minimum) to be tested in an independent third party laboratory, which should also operate to the minimum standards outlined above.

³⁸ Draft PrCEN/TR 15018 : 2004. Characterisation of wastes – digestion of waste samples using alkali fusion techniques. CEN TC292, August 2004

³⁹ CEN TC292 WI 292038 : 2004. Characterisation of waste and soil – determination of elemental composition by X-Ray fluorescence. CEN TC 292, December 2004.

Table C3 Principles of prescribed test methods

Determinand	Prescribed Method	Principle	UK Equivalent 'Blue Book' method Standing Committee of Analysts, HMSO.
<i>General Waste Properties</i>			
Total Organic Carbon	EN 13137	Inorganic carbon is removed using sulphurous acid, and TOC is determined by combustion in a stream of oxygen. Ideally suited for elemental analysers (CHN)	None. Methods provided by the manufacturers of suitable instruments should be suitable
Loss on Ignition	EN 12879	Determined gravimetrically by ashing a portion of the sample at 550°C.	The Conditionability, Filterability, Settleability and Solids Content of Sludges. 1984 ISBN 0117517879
Dry Matter Content	prEN 14346	Determined gravimetrically by drying a portion of the sample to constant weight at 105°C.	The Conditionability, Filterability, Settleability and Solids Content of Sludges. 1984 ISBN 0117517879
<i>Digestion of Raw Waste</i>			
Aqua Regia Extractable Metals	EN 13657	A portion of the dried sample is digested with aqua regia and diluted to volume with water.	Methods for the Determination of Metals in Soils, Sediments, Sewage Sludge and Plants by Hydrochloric-Nitric Acid Digestion. 1986 ISBN 0117519081
Total Metals Content	EN 13656	A portion of the dried sample is brought into solution using microwave-assisted digestion with HF, HNO ₃ and HCl acids.	See reference above for Aqua Regia, Method B for determination of total metals. HF/Perchloric acid digestion is also common, & likely to be more aggressive than EN 13656
Dry Matter Content	prEN 14346	Determined gravimetrically by drying a portion of the sample to constant weight at 105°C.	The Conditionability, Filterability, Settleability and Solids Content of Sludges. 1984

Table C4 ENV 12506 Analysis of eluates – Determination of pH, As, Cd, Cl, Cr, CrVI, Cu, Ni, NO₂, Pb, SO₄, and Zn

Determinand	Prescribed Method	Principle	UK Equivalent ‘Blue Book’ method Standing Committee of Analysts, HMSO.
pH	ISO 10523	pH electrode and suitable meter	The Measurement of Electrical Conductivity and the Laboratory Determination of the pH Value of Natural, Treated and Waste Waters. 1978.
Arsenic	EN 26595 EN ISO 11885 EN ISO 11969	<p>Arsenic is converted to As(III), which is then reduced to arsenic trihydride (AsH₃) and this is then absorbed into a solution of silver diethyldithiocarbamate (in a solvent) to form a red complex which is measured spectrophotometrically at 510 nm.</p> <p>The sample is introduced in aerosol form into a high temperature plasma, which is produced and sustained by electromagnetic coupling through a coil in a RF circuit. Determinands in the sample are excited in the plasma and emit radiation at characteristic wavelengths.</p> <p>Arsenic is converted to As (III), and the As(III) is reduced to gaseous arsenic(III) hydride by reaction with sodium tetrahydroborate in HCl. The AsH₃ is stripped and passed through a flowcell where its absorbance is measured at 197.3 nm</p>	<p>None.</p> <p>This method uses unpleasant organic solvents.</p> <p>Inductively Coupled Plasma Spectrometry. 1996 ISBN 0117532444</p> <p>Selenium in Waters. 1984. ISBN 0117519332 Selenium and Arsenic in Sludges, Soils and Related Materials 1985. A note on the Use of Hydride Generator Kits 1987.</p>

Determinand	Prescribed Method	Principle	UK Equivalent 'Blue Book' method Standing Committee of Analysts, HMSO.
Cadmium Copper Nickel Lead Zinc Cobalt	ISO 8288	The liquid sample is aspirated into an air-acetylene flame where the determinands of interest absorb light of a characteristic wavelength.	Cadmium in Potable Waters by AAS. 1976 ISBN 0117511366 Copper in Potable Waters by AAS. 1980 ISBN 0117515426 Nickel in Potable Waters. 1981 ISBN 011751604X Lead in Potable Waters by AAS. 1976 ISBN 0117511374 Zinc in Potable Waters by AAS. 1980 ISBN 0117515418 Cobalt in Potable Waters by AAS. 1981 ISBN 0117516031
Cadmium Copper Nickel Lead Zinc Cobalt Molybdenum Vanadium	EN ISO 11885	The sample is introduced in aerosol form into a high temperature plasma, which is produced and sustained by electromagnetic coupling through a coil in a RF circuit. Determinands in the sample are excited in the plasma and emit radiation at characteristic wavelengths.	Inductively Coupled Plasma Spectrometry. 1996 ISBN 0117532444

Determinand	Prescribed Method	Principle	UK Equivalent 'Blue Book' method Standing Committee of Analysts, HMSO.
Chloride	ISO 9297 EN ISO 10304-1 & 2	Chloride reacts quantitatively with silver nitrate to form insoluble silver chloride. Potassium chromate is added to indicate the titration end point, when excess silver ions form red silver chromate. Ion Chromatography. Chloride is separated from other anions using a suitable combination of column and eluant, and is then determined using a conductivity detector.	Chloride in Waters, Sewages and Effluents. 1981 ISBN 0117516260 The Determination of Anions and Cations, Transition Metals, Other Complex Ions and Organic Acids and Bases in Water by Ion Chromatography. 1990 ISBN 0117523313
Chromium (VI)	ISO 11083	Cr(VI) reacts with 1,5-diphenyl carbazide to form a red complex, the absorbance of which is measured at 545nm	Chromium in Raw and Potable Waters and Sewage Effluents. 1980. ISBN 0117515280 Method B can be adapted to determine only Cr(VI)
Nitrite	EN 26777 EN ISO 10304-1&2	Under acidic conditions, nitrite reacts with sulphanilamide and N-1-Naphylethylenediamine dihydrochloride to form a pink azo-dye which is measured at 520 nm. Manual technique. Ion Chromatography. Nitrite is separated from other anions using a suitable combination of column and eluant, and is then determined using a conductivity detector.	Usually used as the automated test (see EN ISO 13395 – below) The Determination of Anions and Cations, Transition Metals, Other Complex Ions and Organic Acids and Bases in Water by Ion

Determinand	Prescribed Method	Principle	UK Equivalent 'Blue Book' method Standing Committee of Analysts, HMSO.
Nitrite contd.	EN ISO 13395	Under acidic conditions, nitrite reacts with sulphanilamide and N-1-Naphylethylenediamine dihydrochloride to form a pink azo-dye which is measured at 520 nm. Automated technique.	Chromatography. 1990 ISBN 0117523313 Oxidised Nitrogen in Waters. 1981 ISBN 011715930
Sulphate	ISO 9280 EN ISO 10304-1&2	The sample is acidified with HCl and boiled with barium chloride solution to precipitate barium sulphate which is collected by filtration, dried and weighed. Ion Chromatography. Sulphate is separated from other anions using a suitable combination of column and eluant, and is then determined using a conductivity detector.	Sulphate in Waters, Effluents and Solids. 1988. ISBN 0117522406 The Determination of Anions and Cations, Transition Metals, Other Complex Ions and Organic Acids and Bases in Water by Ion Chromatography. 1990 ISBN 0117523313

Table C5 ENV 13370 Characterisation of waste – Analysis of eluates – Determination of Ammonium, AOX, conductivity, Hg, phenol index, TOC and easily liberatable CN⁻ and F⁻

Determinand	Prescribed Method	Principle	UK Equivalent 'Blue Book' methods
Ammonium	EN ISO 11732	i) FIA, gas diffusion into indicator reagent. ii) CFA photometric determination, salicylate chemistry	Ammonia in Waters 1981
AOX	EN 1485	Adsorption onto activated carbon, combustion in oxygen, and determination of halide ions.	None.
Conductivity	EN 27888	Direct reading using conductivity meter.	The measurement of Electrical Conductivity and the Laboratory Determination of the pH value of Natural, Treated and Waste waters. 1978
Mercury	EN 1483	CV-AAS. Mercury is converted to the elemental form and determined at 253.7 nm	Mercury in Waters, Effluents and Sludges by Flameless Atomic Absorption Spectrophotometry. 1978
Phenol Index	ISO 6439	In the presence of potassium hexacyanoferrate(III), phenolic compounds react with 4-Aminoantipyrine at pH 10.0 to form a coloured antipyrine dye which is measured at 510 nm	Phenols in Waters and Effluents by Gas Chromatography, 4-Aminoantipyrine and 3-Methyl-2-Benzothiazolinehydrazone. 1981.
Total Organic Carbon	EN1484	Oxidation of organic carbon to carbon dioxide by combustion, by the addition of an appropriate oxidant, by UV radiation or any other high-energy radiation. Combustion methods are preferred for samples with a high organic content.	The Instrumental Determination of Total Organic Carbon, Total Oxygen Demand and Related Determinands. 1979

Determinand	Prescribed Method	Principle	UK Equivalent 'Blue Book' methods
Easily Liberated CN	ISO 6703-2	Cyanide is distilled at 125°C and determined spectrophotometrically by the reaction of cyanide with chloramine-T to form cyanogen chloride, which then reacts with pyridine-4-carbonic acid and 1,3-dimethylbarbituric acid to form a red dye.	Cyanide in Waters etc. 1988
Fluoride	EN ISO 10304-1	Ion Chromatography. Fluoride is separated from other anions using a suitable combination of column and eluant, and is then determined using a conductivity detector.	The Determination of Anions and Cations, Transition Metals, Other Complex Ions and Organic Acids and Bases in Water by Ion Chromatography. 1990
Fluoride	ISO 10359-1	Ion Selective Electrode For highly contaminated eluates: Evaporation to dryness, distillation, ISE For lightly contaminated eluates direct ISE	Fluoride in Waters, Effluents, Sludges, Plants and Soil. 1982

APPENDIX D PARALLELS WITH THE REGULATORY MONITORING OF SEWAGE EFFLUENTS

D1 INTRODUCTION

The compliance assessment system set out in this guidance document is likely to represent a substantial change to current methods of working for many waste producers and landfill operators, and so it is natural that there will be some concern as to how well the system will work in practice. For this reason, it may be reassuring to know that there are a number of parallels with the procedures that have been successfully applied since the late 1980s for the regulatory monitoring of sewage treatment works (STW) final effluents. The main elements of effluent compliance assessment are outlined briefly below, with the various parallels with waste assessment indicated parenthetically in italics.

D2 COMPLIANCE ASSESSMENT PERIOD

The assessment period for effluent compliance assessment is one calendar year. (*This is the default period recommended in the present guidance.*)

D3 TYPES OF EFFLUENT CONSENT LIMIT

For each relevant determinand (typically BOD, suspended solids and ammonia) the effluent is required to meet two numerical consents:

1. a 95-percentile ('95%ile') limit; and
2. an absolute maximum, or 'upper-tier' limit.

(The limits in the present guidance are to be interpreted as absolute maxima.)

D4 TYPES OF SAMPLING PERMITTED

D4.1 Sampling for assessment of performance against 95%ile limit

For testing compliance with the 95%ile limit, the monitoring programme carried out by the Environment Agency is required to be statistically representative of the 12-month period, and there must be a sampling schedule stating in advance the dates on which samples are to be taken. Sewage effluent quality typically shows a seasonal variation, and so the scheme nearly always adopted is to take a fixed number of samples per month (commonly 1 or 2), giving annual frequencies of typically 12 or 24. No amendments to the programme during the year are permitted. (Thus, if the Agency does choose to take additional samples, these cannot be included in the compliance assessment.)

D4.2 Sampling for assessment of performance against upper-tier limit

In contrast, for testing compliance with the upper-tier limit, Agency staff may take additional samples over and above those in the routine monitoring programme whenever they have reason to suspect that effluent quality may have deteriorated. (*This is the worst-case targeted sampling approach described in the present guidance.*) Because the limit must be met for 100% of the time, there is no statistical requirement for the sampling to be representative - and by the same token the discharger knows that he has no grounds for complaining that worst-

case targeted sampling is 'unfair'.

The targeted sampling approach, although a potentially powerful tool in the hands of the regulator, is seldom seen as a problem in practice. STW operators can tolerate the imposition of a strict upper-tier limit because they are confident from their knowledge of the treatment process that these limits can comfortably be met in normal day-to-day operation. *(This reinforces the emphasis placed in this guidance document on understanding the process that generates the waste.)*

D5 TIMING OF A COMPLIANCE FAILURE

Failure of the 95%ile limit generally becomes apparent only near the end of the year's effluent monitoring programme. It also tends to be indicative of a gradual decline in STW performance rather than something more serious. It is usual, therefore, for the Agency to wait until the end of the assessment period before reporting any such failures to STW management.

In contrast, a single failure of the upper-tier limit (however early in the year) gives an unequivocal demonstration that the effluent has failed, and so in this case there is no question of delaying any response until the end of the assessment period. An upper-tier failure triggers immediate action from the Agency. *(Similarly, in the event of a single compliance failure at the landfill the operator will immediately deem the waste to have failed for the whole assessment period.)*

D6 CONSEQUENCES OF A COMPLIANCE FAILURE

By the time a failure of the 95%ile limit has been demonstrated, almost a year's effluent load may have entered the aquatic environment - and clearly there is no question of 'recalling' the sub-standard effluent for further treatment. The same is true even for one-off episodes of poor performance when the effluent exceeds the upper-tier limit. By the time the alarm has been raised for a gross pollution incident, and a tripartite sample taken to provide formal evidence of failure, the bulk of the offending effluent will already have entered the receiving water and passed downstream. *(Similarly, deliveries of a waste may have been accepted by the landfill for a number of months before the operator happens to obtain a sample which fails the limit. It is clearly not then a feasible option to identify all that year's waste and return it to the producer.)*

The primary consequence of failure, accordingly, is an obligation for STW management to tell the Agency how they are going to improve future effluent performance - whether through tighter operational control, or through capital investment to increase the treatment capacity of the works. *(These options are broadly analogous to those available to the waste producer following a compliance failure at the landfill.)*