

## III.1

# Assessment of pollution potential from solid waste

Irena Twardowska

### III.1.1. Introduction

The majority of disposed wastes, including recyclable waste, is not environmentally safe. Waste as a freshly generated anthropogenic material usually is not geochemically stable. Hence, contaminant release at different stages of waste exposure to environmental conditions, either at the disposal site, or in case of its bulk use for construction purposes, e.g. as a common fill, can be anticipated. The leaching of soluble constituents upon contact with water is regarded as a main mechanism of release, which results in a potential risk to the environment.

The need for reliable assessment and prediction of waste material behavior under specific conditions of exposure resulted in the development of a multitude of leaching/extraction tests and testing schemes by different groups of analysts and national regulatory bodies. In the USA, the basic set of guidelines for solid waste analysis for environmental risk assessment includes a multi-volume continuously updated assemblage, USEPA SW-846 (1996–2003). There, different techniques for waste sampling, preservation, storage and analysis dependent upon the physical state of a material, test aim, frequency of sampling and type of contaminants are presented. The testing program comprises waste material, disposal site and all the compartments of the environment in the vicinity of the site in the area of waste processing and utilization (i.e. waste, landfill air, groundwater, soil, run-off water, soils and pore solution of the vadose zone and plants). These guidelines are widely available and are in common use in the USA and several other countries.

In Europe, the variety of data and schemes developed in many countries evoked a need of integration and unification of approaches towards evaluating the leaching behavior of waste materials that are either disposed or used in construction. The main goal behind these efforts is the development and harmonization of reliable testing procedures for short- and long-term risk assessments, which would consider specificity, but also similarities in the leaching behavior of different waste and other materials. The stronger links between different research groups involved in this issue in Europe were developed through the Measurements and Testing Program of the EC, Working Group B of ISCOWA – The International Society for the Environmental and Technical Implications of Construction with Alternative Materials (Laboratory Testing and Environmental Impact Assessment), WASCON conferences on environmental implications of construction with waste

materials and technology developments held in 1991, 1994, 1997, 2000 and 2003, the Workshop on the Harmonization of Leaching/Extraction Tests for Environmental Risk Assessment held in 1996 and follow-up development of the European Network for Harmonization of Leaching/Extraction Tests (2000).

One of the most important results from these studies is the leaching similarity of different waste materials having corresponding geochemical properties. These similarities allow for a common approach in the characterization of leaching by focusing on the relevant key contaminants and a limited number of controlling parameters in relation to quality control and regulatory testing (van der Sloot et al., 1993, 1994a,b, 1996, 1997; Eighmy and van der Sloot, 1994; van der Sloot, 1996, 2000a) affords unification of data reporting and comparison in different fields, and therefore facilitates evaluation and regulatory control of waste and non-waste materials with respect to long-term environmental impact and its mitigation, in particular when usable materials are transformed into waste and vice versa.

The above works and studies set a foundation for the development of a protocol for assessing environmental risk from solid wastes and generically relevant materials based on their leaching behavior. This protocol has already started to materialize in the outcome of standardization activity of CEN/TC 292 – European Committee for Standardization/ Technical Committee Characterization of Waste, and in a linkage with eight other core Environmental and related Technical Committees (TCs), e.g. TC 308 on sludges, and five other liaisons outside CEN: International Organization for Standardization ISO/TC 190 on Soil Quality, UNEP, EFTA, EC/DGX I and EC/DGX II. These European Standards are being developed primarily to support the needs of the EU and EFTA countries, in particular to provide methods for testing of waste in a standardized form for the EU member states to fulfill the requirements of the EU Directives related to waste management.

The EC Landfill Directive (EC, 1999) imposed a large number of requirements with respect to the quality of the waste that may be accepted for landfill; a list of waste characterization methods to be standardized had to be prepared by July 2001. To fulfill this requirement, TAC Subcommittee on the EC Landfill Directive developed a so-called “toolbox” of methods for testing of waste, which the EC member states should have available as CEN or ISO Standards (TAC Landfill, 2001). This toolbox comprises a list of methods and procedures for sampling, pre-treatment, evaluation of general waste properties, methods of digestion of raw waste, waste analysis, and leaching tests, as well as specifies the needs for research for development of further methods and procedures.

Recently, the initiative has also been undertaken with the aim of development within the CEN Environmental and related TCs, and external liaison bodies of horizontal standards for upcoming EU directives on sludge, soil and biowaste and thus to produce, where possible, one standard as opposed to several elaborated in a vertical manner (CEN BT, 2001; CEN/TC 292, 2002c, 2003; Project HORIZONTAL, 2003).

The three basic fields of waste characterization are being covered by the European standardization activity: (1) waste sampling; (2) leaching behavior; and (3) analysis of waste and eluates. A complementary activity for all these fields is (4) terminology – related terms and definitions that assure univocal data comparison and interpretation. This activity is aimed to developing reliable testing procedure for short- and long-term environmental risk assessment from waste disposed of or utilized for different purposes.

Below, the current stage of studies and European standards development, along with problems and pitfalls of prognoses based on standard tests will be discussed.

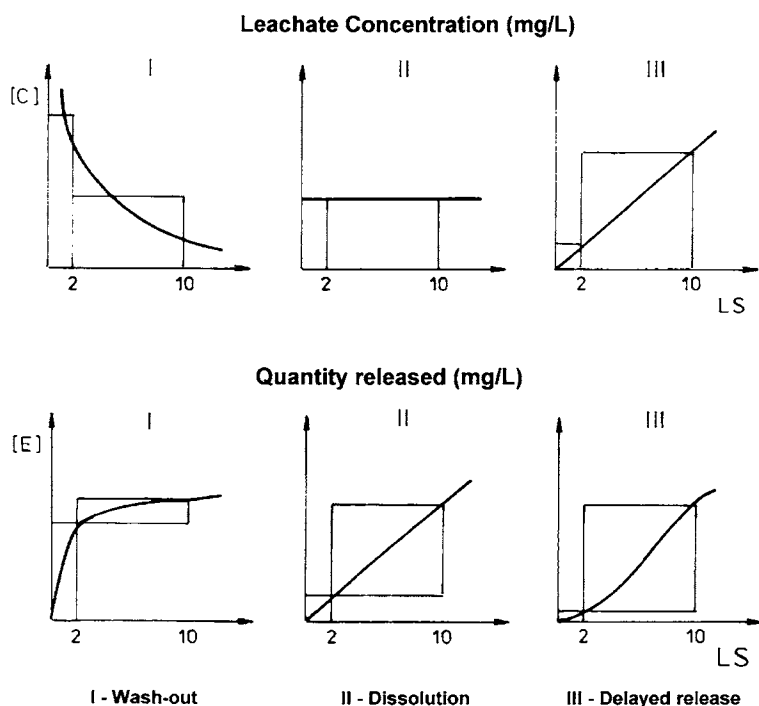
### **III.1.2. Testing procedures for risk assessment**

#### ***III.1.2.1. General approach to characterization and testing of waste***

Simplified arbitrary test procedures related to regulatory limits, and thus having direct economic consequences, very often bear no relation to the actual situation to be controlled and hence should not be considered as a sound decision-making tool. The comparison of different regulatory test methods designed for different purposes, mainly to assess the potential for long-term environmental impact and used in the USA (EP-tox, TCLP, availability test), Germany (leach test DIN 38414 S4), France (leach test X 31-210), Switzerland (leach test TVA) and the Netherlands (availability test NVN 2508, column test NVN 2508, serial batch extraction NVN 2508, and monolith tank leaching NVN 5432) showed a low efficiency in achieving that goal (van der Sloot et al., 1991b). The reason for the failure was explained by the attempt of evaluating all materials for all disposal/use options by one decision test. It was further concluded that the best approach toward the testing procedure to be applied as a basis for decision making on different aspects of waste handling (e.g. treatment, recycling, utilization, disposal) requires application of scenarios based on actual environmental data, matrix composition and origin of the materials, specific for the purpose and exposure conditions. Due to a number of research projects undertaken in the last decades in various research centers, the understanding of leaching behavior of waste has grown substantially and resulted in a better identification of release mechanisms and controlling factors. One of the fundamental conclusions derived from these studies has been the need of a strong consideration of the time-dependent release for prediction of the long-term leaching behavior of waste on a time scale far beyond that of any realistic laboratory-leaching test. The major premise for the short- and long-term assessment is the systematic behavior of inorganic constituents controlled by several basic variables – of these liquid to solid ratio ( $L/S$ ) (reflecting the time factor), pH, redox Eh and complexation have been considered the most important ones.

#### ***III.1.2.2. Generic leach pattern of waste***

A crucial observation for the long-term prediction of leaching behavior of constituents is the III-stage generic leach pattern that comprise wash-out (I), solubility-controlled dissolution (II) and delayed release (III) stages shown in Figure III.1.1 (van der Sloot et al., 1993). This pattern reflects a situation when after decline of a solubility controlling phase, e.g. availability of adequate buffering agents, the massive release of constituents at a high rate may occur at some point delayed in time. In general, the correct prediction of the occurrence and intensity of the delayed release (III) stage appears to be a particularly problematic task. In the most frequent case, the development of this phase is determined by two kinetically defined processes of acid generation and buffering of constituents release, either due to the direct attack of generated acid loads (instant neutralization), or independent dissolution of buffering constituents, when dynamics of acid generation may



*Figure III.1.1.* Patterns of concentrations in leachate and cumulative loads in the three-stage mechanisms of constituent release from waste matrix (after van der Sloot et al., 1993).

prevail over the buffering agents availability in pore solution in spite of their still abundant total content in the system (e.g. carbonate dissolution in microenvironments of heterogeneous waste material). The complexity of real systems makes the correct prediction of the delayed release (III) stage development extremely difficult and requires complete and detailed information not just on the chemical composition, but also on the phase composition of a waste material, including the forms, dispersion and specific surface of the phases in the matrix, which influence their reactivity and availability (Twardowska et al., 1988; Twardowska and Szczepańska, 2002).

### **III.1.2.3. Long-term leaching behavior issues**

The kinetically defined processes of constituent release makes simulation of long-term leaching behavior in a laboratory particularly complicated. It is generally agreed that a single batch test will never allow for long-term prediction. The compression of a time scale during accelerated testing may cause a deep distortion of the actual pattern, as adequate acceleration of process kinetics is generally not possible. Therefore, a reasonable level of compromise should ensure proper information and a good confidence in the tests for long-term risk assessment from waste relating to mass transfer, environmental physico-chemical parameters and the time scale (Quevauviller, 1996).

The comparison of agreement of different regulatory leaching test procedures for waste materials and construction materials with field data showed numerous examples of discrepancy, e.g. poor agreement of USEPA tests such as EP-tox, TCLP extracts, synthetic acid rainwater (SAR) extracts, CO<sub>2</sub> saturation test and deionized (DI) water extracts with the field leachate due to the difference between low Eh potential in the field leachate compared to high Eh in the laboratory tests (van der Sloot et al., 1991b). Also studies on leaching characteristics of coal combustion fly ash (FA) deposits in the natural conditions showed significant differences in comparison with the data obtained in laboratory leaching tests (Fällman and Hartlén, 1994; Janssen-Jurkovičová et al., 1994; Meij and Schaftenaar, 1994; Meij and te Winkel, 2000; Twardowska and Szczepanska, 2002). Due to the much more complicated nature of the environmental interactions, the distortion of the time scale may cause serious qualitative and quantitative errors in prediction of the leaching behavior of a material. In particular, kinetically determined processes and reactions such as weathering, dissolution of amorphous phases and formation of secondary minerals, as well as the effect of flow conditions upon the actual composition and ionic strength of pore solutions are not adequately considered in these tests. Correct prediction of the leaching behavior of trace elements from the material requires the precise modeling of processes occurring within the macro-components of a material, which are responsible for the formation of factors controlling trace metal release (pH, Eh, exposure). The complexity of real systems makes this task extremely difficult. Nevertheless, significant progress in the development of reliable testing procedures for prediction of the leaching behavior of waste within the wash-out (I) and dissolution (II) stages has already been achieved.

#### ***III.1.2.4. Waste environmental evaluation scheme***

As a result of complex studies and analysis on constituent release from granular material carried out in numerous European laboratories, a substantial part of them being conducted since the late eighties within the research program of the Netherlands Energy Research Foundation ECN, a more flexible material- and site-specific approach to waste environmental evaluation than one unified decision test has been proposed in order to improve the basis for decisions concerning waste management options (van der Sloot et al., 1991a,b, 1994b, 1996, 1997; Eighmy and van der Sloot, 1994; van der Sloot, 2000a). Material characteristics, site-specific information and long-term aspects of major element chemistry as input data, as well as the modeling of constituent release and the sensitivity of the system to environmental factors are essential elements in this approach (Fig. III.1.2). Besides material specificity, the interactions between waste and soil were found to be of great importance in controlling the net release of contaminants from waste disposal and reuse activities (van der Sloot et al., 1991a; Hockley et al., 1992; Hjelmar et al., 2000; Odegard et al., 2000). The current regulatory waste testing methods neglect the waste–soil interface effects and are focused on evaluating entirely the waste properties. This results in inconsistency with field data, most often giving false-positive evaluation of the environmental hazard from waste. The novel approach suggests site-specific evaluation of the environmental hazard, which considers inclusion of waste–soil interaction into the waste testing procedure. A classification system for waste–soil interactions, with diffusion-dominated interfaces and equilibrium reactions has been used as a basis for an approach to the subject that aims to incorporating this model into the macroscopic soil

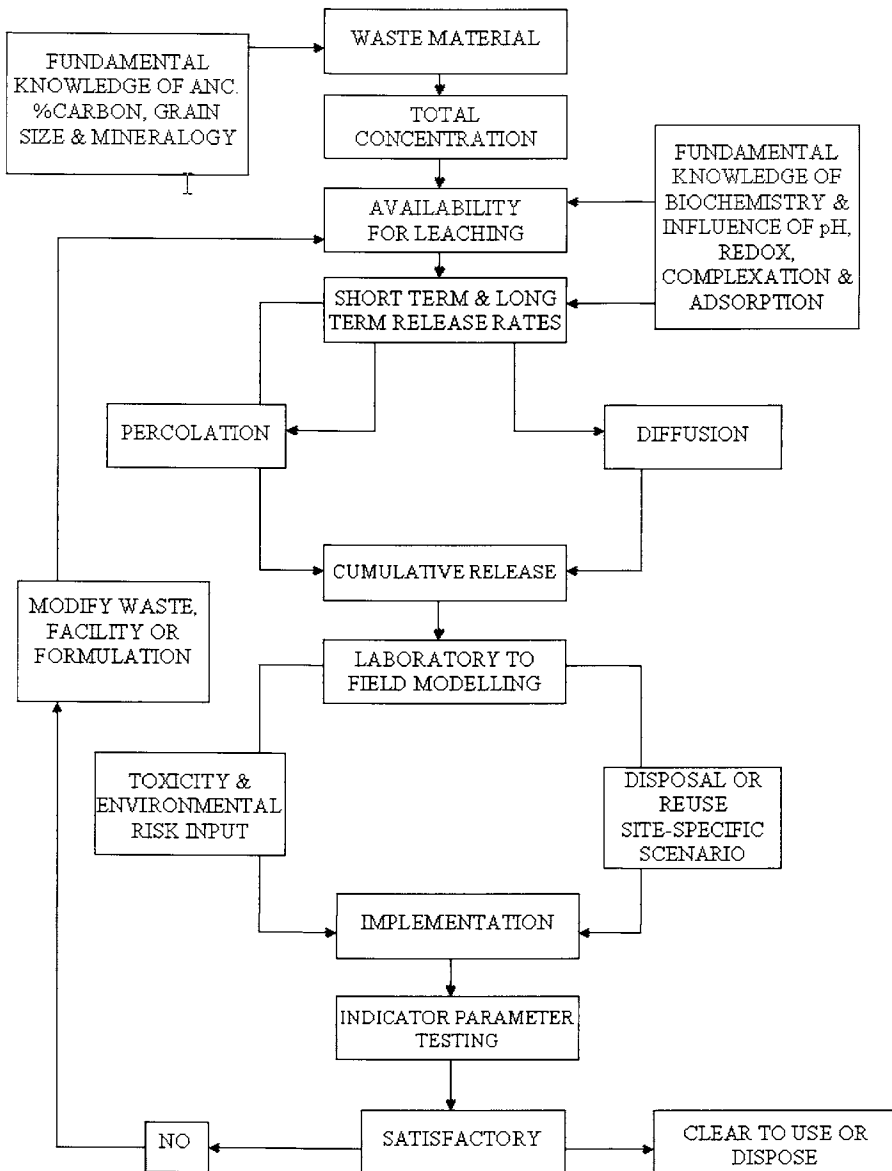


Figure III.1.2. Solid waste testing and evaluation flow chart (after van der Sloot et al., 1991b).

models and creating a link between macroscopic soil and groundwater contamination models for long-term environmental impact assessment and public policy (van der Sloot et al., 1991a; Hockley et al., 1992; Odegard et al., 2000). This approach, though rational, still presents considerable practical difficulties in developing reliable predictive models and thus has not yet been implemented in standardization and regulatory test procedures.

A flow chart summarizing the above approach to evaluation of waste materials based on leaching data and environmental factors, which distinguish inorganic, organic and volatile organic compounds, is presented in Figure III.1.3 (van der Sloot et al., 1991b). Each of these kinds of compounds is to be treated differently in the subsequent steps. The specific features of this scheme are as follows:

- The leaching is addressed in terms of constituent release as a function of time.
- The evaluation procedure includes different tests for waste material and stabilized waste material.
- The procedure comprises sampling, waste analysis and leaching.
- The properties required for long-term environmental impact assessment are indicated and addressed in relevant levels of testing.
- The aspects to be considered at the different levels of testing are also indicated.
- The waste–soil interfaces and a field validation is the last step preceding potential environmental impact assessment.

It has been assumed that the integration of leaching data, controlling factors and environmental conditions ultimately leads to an assessment of potential environmental impact and to the decision concerning the environmental sustainability of a waste site with or without the controlling measures.

The scheme gives the outline of the testing procedure required for an environmental impact assessment, but not yet necessarily developed in detail for diverse kinds of contaminants, waste materials and tests. To date, the particular tests and levels of testing in this scheme display different extent of development. In the research and standardization activity at the European level, the advances in these areas reflect the place of a standard in the business plan and target dates.

The comprehensive framework of the unified systematic approach to evaluation of leaching behavior of granular inorganic waste based on the general geochemical principles (pH and redox-dependent precipitation/dissolution, liquid phase complexation and sorption), applied to a wide range of waste materials by a number of authors was outlined by Eighmy and van der Sloot (1994). The subsequent stages of integrating information on leaching behavior of waste comprise:

- I “Basic Characterization”: physical properties (structure of matrix, particle size, specific surface), solid phases at the particle surface, mineralogical and chemical composition.
- II Evaluation of “Systematic Leaching Behavior” with use of the fundamental information derived from step I. The framework of the evaluation procedure includes: (i) serial batch, column, or lysimeter tests to assess cumulative release rate (mg/kg) vs. cumulative  $L/S$  ratio/or time, or pH, in relation to the total content (mg/kg) or environmentally available fraction of element (mg/kg) over geologic time (i.e. 1000–10,000 years. Here though, entirely soluble compounds available at the moment of testing can be assessed. No generation of new loads of soluble compounds is considered in this test); (ii) determination whether the leaching process is kinetic- or equilibrium-based to estimate the duration of required observation of a leaching process; (iii) additional leaching tests for elucidating the controlling effect of Eh, complexation and sorption processes; (iv) geochemical thermodynamic modeling to

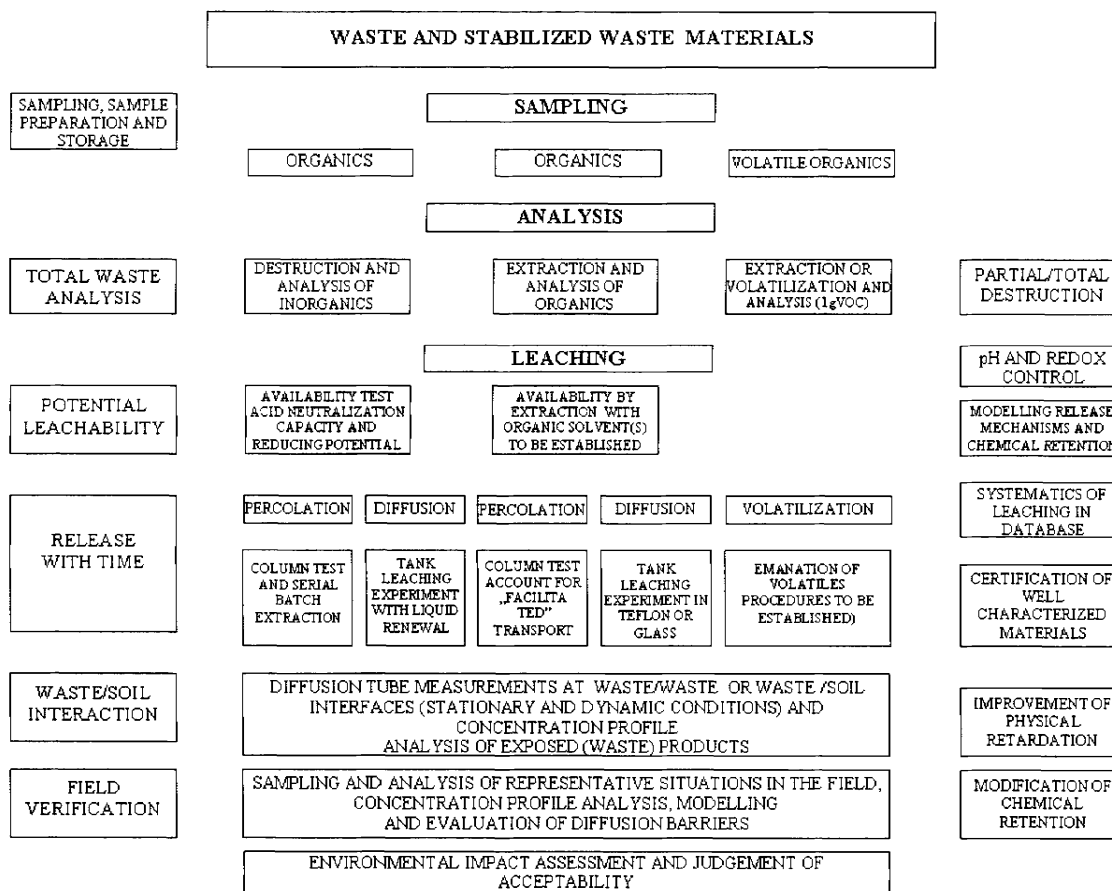


Figure III.1.3. Scheme for the evaluation of long-term release from waste and stabilized waste material (after van der Sloot et al., 1991a).

verify equilibrium-based leaching behavior; and (v) kinetic modeling to verify kinetic-based leaching behavior (to be developed).

- III “Field Verification” (comparative evaluation) of laboratory test- and predictive data identified in step II by means of: (i) full-scale data from applied projects; (ii) large-scale pilot or demo data; and (iii) lysimetric studies.
- IV “Accelerated Testing”: simplified leaching procedures, and long-term prediction of leaching behavior, in particular for assisting industry with QC, upstream and downstream modifications in waste stream, and cost reduction. The framework for these procedures includes: (i) rapid, concise, reliable tests for characterization, compliance and verification of data derived from the step II; (ii) accelerated aging, weathering, destruction tests; and (iii) simulated long-term leaching or extraction tests.

### III.1.3. European standardization activity

#### III.1.3.1. Testing levels and categories

The above frameworks set a foundation for the development of a protocol and a set of European Standards for assessing environmental risk from solid wastes and generically relevant materials based on their leaching behavior.

Standardization activity comprising basic fields of waste characterization, i.e. waste sampling, evaluation of leaching behavior and analysis of waste and eluates in different stages of development (CEN/TC 292, 2002b). The status of standardization of terminology on waste (material and management related terms and definitions) that assures univocal data comparison and interpretation has been addressed in Chapter I.

The test standards in force, which are intended to identify the leaching properties of granular waste and sludges, are generally divided into three categories (EN 12457–1/2/3/4, 2002):

1. Basic characterization tests are used to obtain information on the short- and long-term leaching behavior and characteristics properties of waste materials. *L/S* ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity and physical parameters are addressed in these tests.
2. “Compliance” tests are used to determine whether the waste complies with specific reference values. The tests focus on key variables and leaching behavior identified by basic characterization tests.
3. “On-site verification” tests are used as a rapid check to confirm that the waste is the same as that, which has been subjected to the compliance test(s).

These categories are adequate to the three-tier procedure of characterization and testing of waste provided in Annex II of the EC Landfill Directive (1999) referring to Levels 1, 2 and 3 of testing (Fig. III.1.4).

The Level 1 of testing is considered to be the key to the waste acceptance system. Its purpose is to determine the intrinsic properties of the waste in order to decide on the appropriate methods and site for the treatment, disposal or reuse of the waste. According to the Landfill Directive, the waste producer, before removal from the producer’s premises,

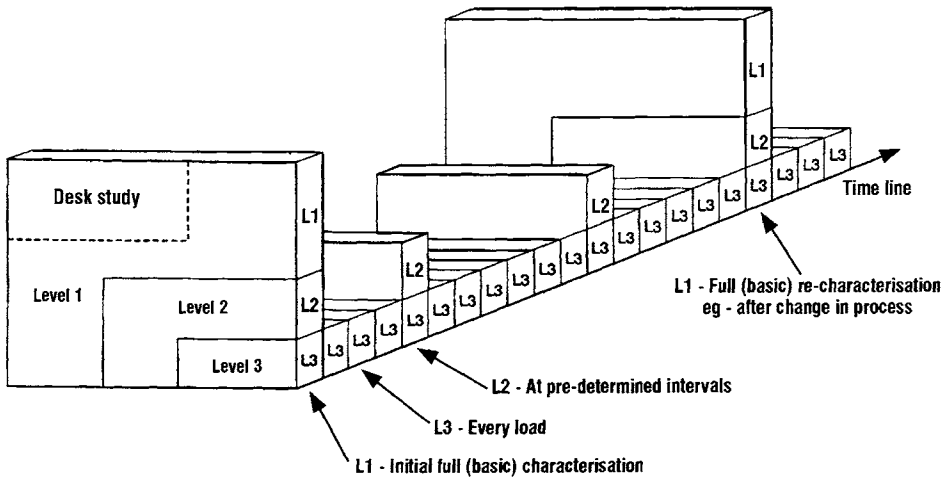


Figure III.1.4. Scheme of a three-tier hierarchy for characterization, sampling and testing of waste (according to Annex II of the Landfill Directive, 1999).

should use it for assessment of waste characteristics. Once the comprehensive characterization of the waste material is documented, provided the waste is of a consistent nature, only infrequent confirmation of this characterization by the waste producer is necessary. Therefore, the periodic monitoring in Levels 2 and 3 is based on the bank of characterization data provided by Level 1.

### III.1.3.2. Waste sampling

Sampling is the first step and an essential part of the reliable environmental risk assessment from waste material related to its treatment/disposal/reuse options. It should follow the three-tier procedure of waste characterization and give representative material for testing. The hydrogeochemical monitoring practice shows that this step is critical for quality requirements: about 30% of errors are being committed during collecting and transport of samples, 60% of errors falls to sample treatment and preparation for analysis and just 10% are the analytical errors (Ramsey et al., 1992; Ramsey, 1993). In waste characterization testing the errors are probably more evenly distributed between sampling and the testing scenario and its interpretation, while analytical errors also play a marginal role. Due to the variety of waste material and other related issues such as different sampling goals, strategies, techniques, and the risk posed by this waste to the environment, the sampling scenario should be designed accordingly on an individual basis.

As a result, for European Standards for Waste Sampling the concept of the “shop shelf” approach was developed, which allows the appropriate parts of the standard to be selected according to a sampling program. This idea is being materialized in the development of the series of coordinated basic Draft European Normative Standards dealing with sampling techniques and procedures (CEN/TC 292.WG1, 2000). The standards in this series, which already underwent the CEN-enquiry are: characterization of waste – sampling of liquid and granular waste materials including paste-like materials and sludges – a framework

for sampling plan preparation (WI 29001, 2003); Part 1: selection and application of criteria for sampling under various conditions (WI 292002, 2001); Part 2: sampling techniques (WI 292017, 2001); Part 3: sample pre-treatment in the field (WI 292018, 2001); Part 4: procedures for sample packaging, storage, preservation, transport and delivery (WI 292019, 2001). Terms and definitions related to sampling constitute an integral part of these standards.

This approach tends to acknowledge waste material, process and objective variability, allowing the standard to be adaptable to technical environment and objectives for sampling. The sampling objectives, along with the sequence of operations required to fulfill them are detailed in an overall *sampling program* that is defined as “total sampling operation, from the first step in which the objectives of sampling are defined to the last step in which data is analyzed against these objectives.” The details of the program must be discussed and agreed with all involved parties.

The links between the essential elements of a sampling program, *sampling plan* being one of these elements, are illustrated in a process map (Fig. III.1.5). The Draft European Standard (WI 292001) sets out the general principles to be applied in the preparation of a sampling plan for the characterization of waste materials to previously set objectives. Key elements of a sampling plan are defined in Figure III.1.6. Waste sampling plan with reference to the program objectives should be in conformity with a relevant level of testing according to the three-tier general procedure for waste characterization and testing (Fig. III.1.4) and ensures a representative nature of the sampling. In this pre-standard, probabilistic sampling is seen as the preferred option. The appropriate sampling techniques are considered to be selected from PrEN, WI 292017 (Part 2) using statistical guidance from prEN, WI 292002 (Part 1).

The Draft European Standard PrEN, WI 292002 (Part 1): “selection and application of criteria for sampling under various conditions” presents statistical principles and purpose of sampling, types and pattern of sampling (probabilistic and methodology-agreed), as well as methodology of determining the size and number of samples, defining the sampling scheme and statistical principles (objectives, types of variability, population parameters and sample statistics, the scale of sampling and reliability).

The Draft European Standard PrEN, WI 2920017 (Part 2): “sampling techniques” describes techniques used in the recovery of the sample and defined as “the physical procedure employed by the sampler to collect part or parts of a discarded or secondary material for subsequent investigations.” The standard details two types of sampling procedures:

- Primary sampling that is representative of the whole mass being sampled (e.g. a core taken through a well-mixed stream).
- Spot sampling that removes a portion of a total mass. It is generally used when sampling large masses, where access across or coring through the material is impractical or dangerous.

The sampling technique adopted depends on a combination of different characteristics of the material and circumstances encountered in the sampling location. This part of the (draft) European Standard describes techniques for sampling liquid and granular waste material, including paste-like materials and sludges, found in a variety of locations. The standard also gives guidance on the selection and preparation of equipment, apparatus

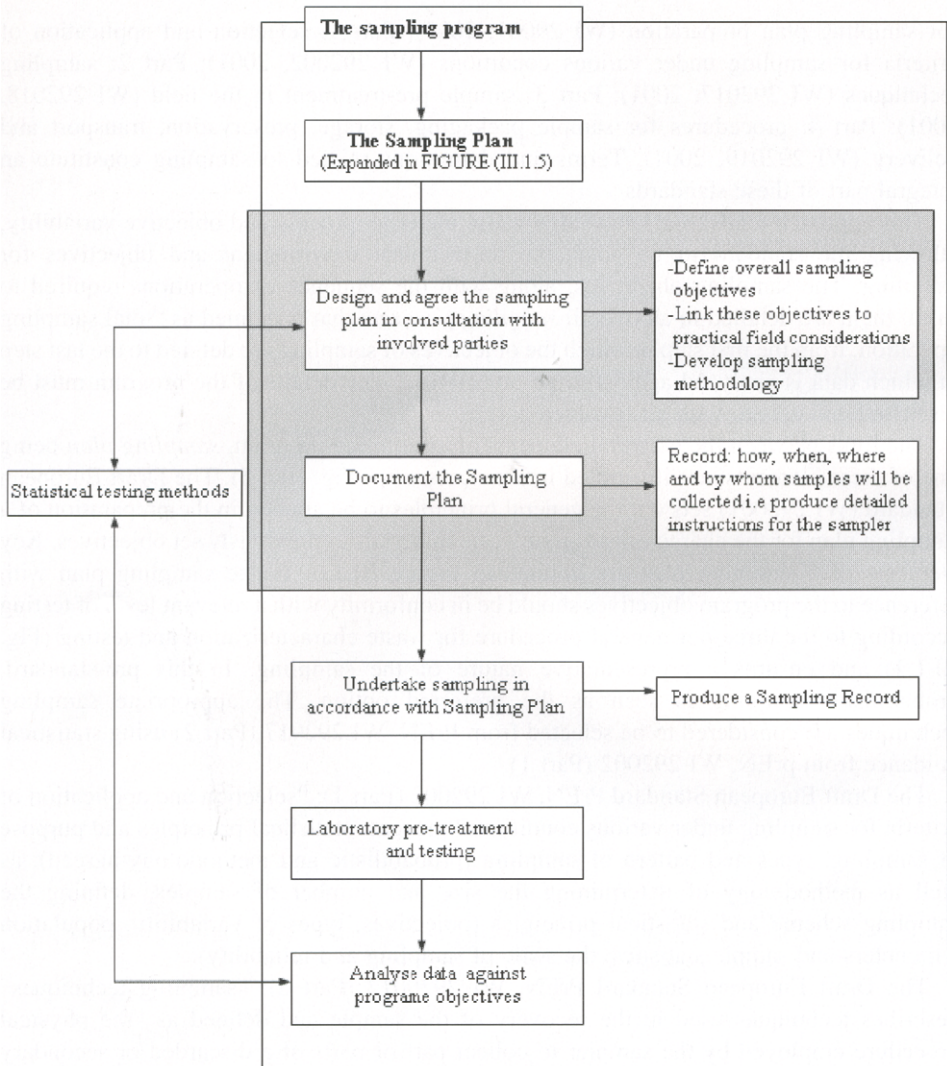


Figure III.1.5. Links between the essential elements of a sampling program (after WI 292001, CEN/TC 292/WG 1, 2001).

used in the waste sampling program and recommendations on sample handling, along with the relevant terms and definitions.

The Draft European Standard PrEN, WI 292018 (Part 3): “sample pre-treatment in the field” specifies procedures for field sample size reduction for the above kinds of waste, among them for generic sub-sampling of solid waste to provide sub-sample in the field that is representative of the overall sample and suitable for submission to the laboratory. It does not deal with sub-sampling in the laboratory or the preparation of samples for analysis.

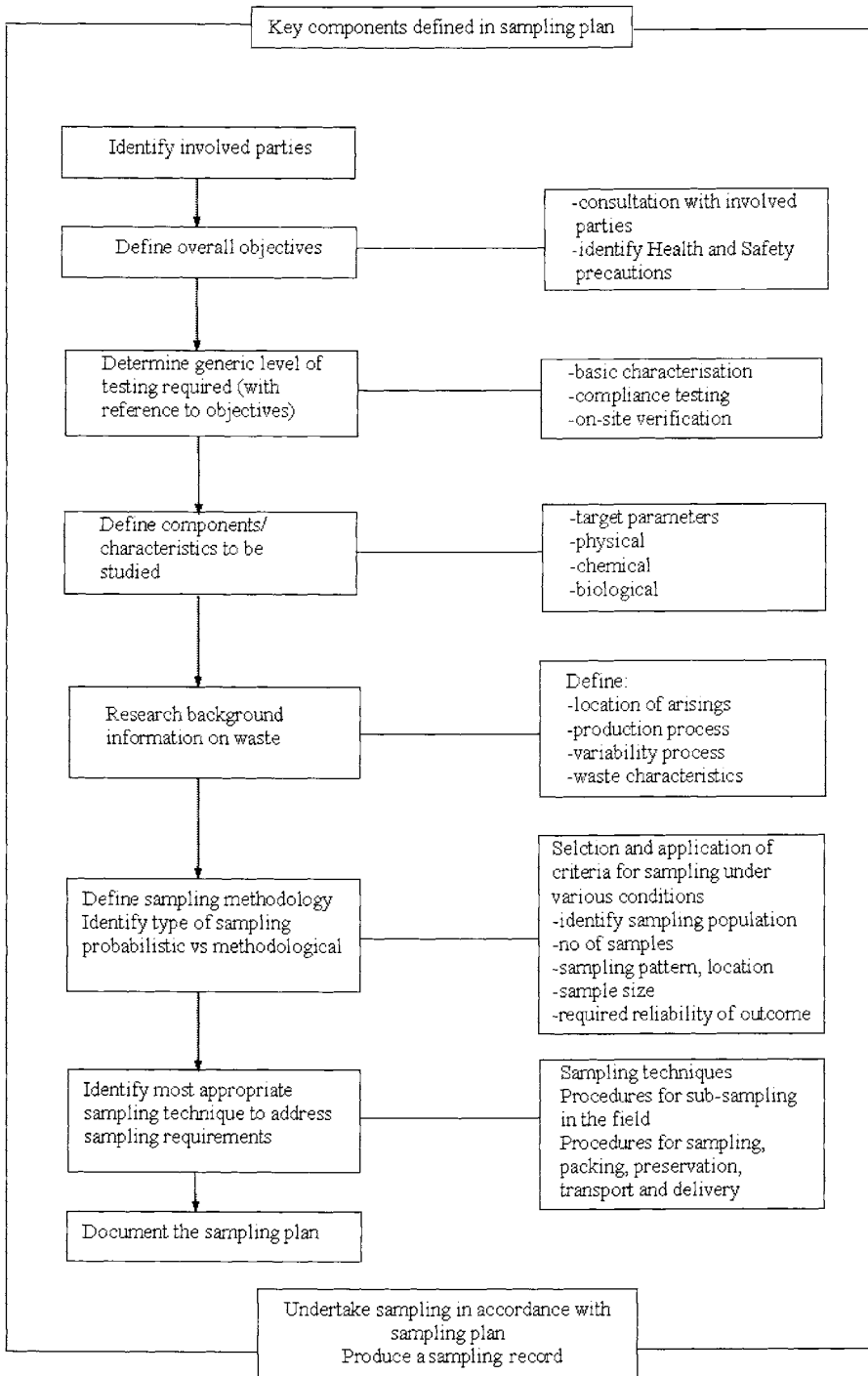


Figure III.1.6. Key elements of a sampling plan (after WI 292001, CEN/TC 292/WG 1, 2001).

The Draft European Standard PrEN. WI 292019 (Part 4): “procedures for sample packaging, storage, preservation transport and delivery” describes recommendations or methods for the packaging, preservation, short-term storage and transport of samples of both solid and liquid waste, including paste-like substances and sludges, or for samples of similar materials. It is applicable for all wastes or secondary materials, excluding domestic wastes.

In order to facilitate the enforcement of the Landfill Directive (EC, 1999), EC requested CEN/TC 292 (2002a) to prepare the Draft European Standard on Sampling – Part 5 (WI 292041, 2003) that should incorporate the examples of several sampling scenarios of the typical sampling situations relevant for the Landfill Directive: piles, moving belts, falling streams, truckloads and tanks. They may be generic sampling plans for typical situations or sampling plans developed especially for specific situations and include sampling of granular, monolithic, paste-like waste and sludge, as well as sampling of inert, non-hazardous and hazardous waste (the target date for Formal Vote for this Standard is 2005).

The role of sampling, sub-sampling, storage and pre-treatment at different levels in the characterization of waste is presented in Figure III.1.7.

### ***III.1.3.3. Determination of the leaching behavior of waste***

#### ***III.1.3.3.1. Basic characterization tests***

The release of soluble constituents upon contact with water is regarded as a main mechanism of release, which results in a potential risk to the environment during the reuse or disposal of waste materials. The basic assumption for testing is that leaching behavior of waste is influenced by several parameters and external factors, of which the chemical nature of waste in terms of pH, reducing properties and degradable organic matter content, the nature of the leachant, the contact time of the leachant with the waste and release mechanism (solubility or diffusion), as well as the chemical, physical and geotechnical natures of the environment, to which the waste is exposed, are considered of particular importance. For examination of the influence and importance of these factors, the basic characterization tests have been developed.

For basic characterization, a methodology for the determination of the leaching behavior of waste under specified conditions has been formulated in the European Standard EN 12920: (2003), where the steps required to achieve such a characterization are specified. This generally requires several tests to be performed, to use or establish a behavioral model and the validation of the model.

The standardization procedures that will allow supplying reliable data for the significant part of waste stream and for its site-specific long-term leaching behavior are currently in progress. Up-flow percolation test (under specified conditions) (prCEN/TS 14405, 2002) belongs to the category basic characterization tests and specifies a test to determine the leaching behavior of inorganic constituents from granular waste without or with size reduction. The waste body is subjected to percolation with water as a function of  $L/S$  ratio under hydraulically dynamic conditions. The method is a once-through column leaching test and the test results establish the distinction between different release patterns, for instance washout and

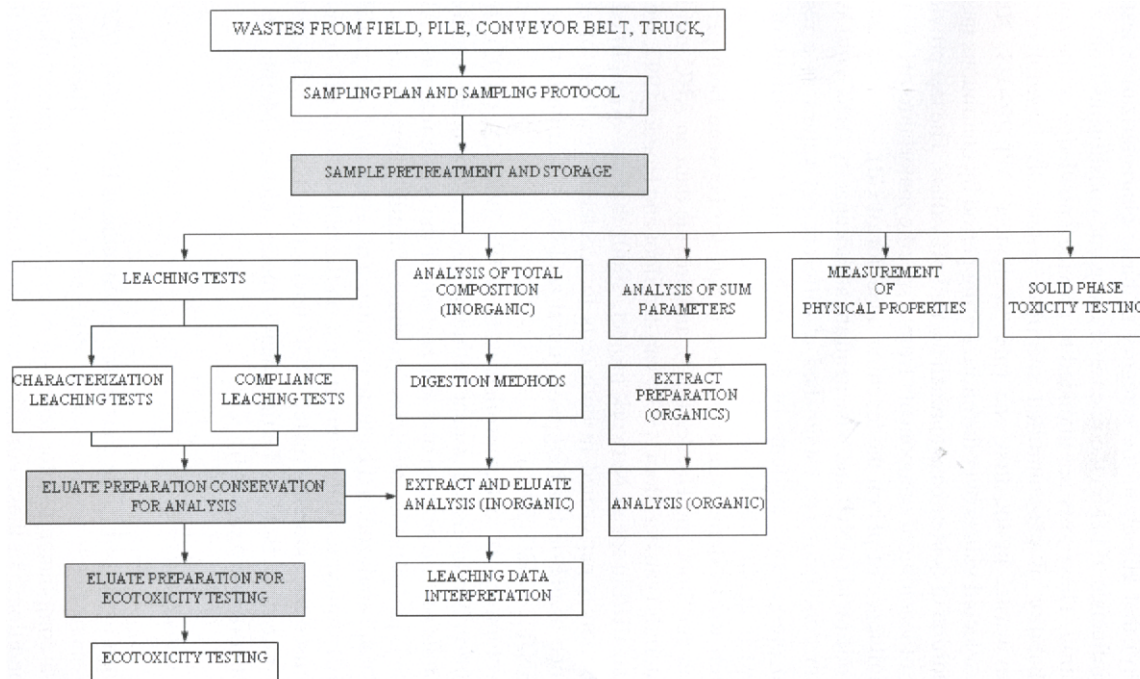


Figure III.1.7. Role of sampling, sub-sampling, storage and pretreatment of waste at different levels in the characterization of waste (after van der Sloot, 2002).

release under the influence of interaction with the matrix, when approaching local equilibrium between waste and leachant. The release of soluble constituents upon contact with water is regarded as a main mechanism of release, which results in a potential risk to the environment during the reuse or disposal of waste materials.

Other leaching behavior tests under development (expected Formal Vote in 2004 and 2005, respectively) that belong to the category of basic characterization tests consider determining the influence of pH on the release of inorganic constituents from a waste into the aqueous solution. These tests are intended to provide knowledge of the potential and anticipated leachability of pH-controlled specified, potentially harmful or hazardous components from waste. In the first one (pH “static test” WI 292033, 2003) pH is controlled at pre-selected values over the entire testing period by continuous measurement and automatic addition of acid or base to reach desired pH values. The test provides insight in the sensitivity of leaching of components from a specific material to pH (Figure III.1.8 exemplifies the influence of pH on the leaching behavior of Cd).

In the second one (acid (ANC) and base neutralization capacity (BNC) test WI 292046, 2003) equilibrium conditions are established at different pH values as a result of the reaction between pre-selected amounts of acid or base and test portions of the waste material. This test is applicable to determine the ANC and BNC of a waste material. Preceding research works demonstrate the data difference between both pH-controlled leaching tests (van der Sloot and Hoede, 1997), and provide data on ANC and BNC for a wide range of materials (van der Sloot, 1996; van der Sloot and Hoede, 1997; van der Sloot et al., 1997,2000a; EU/European Network project, 2000) that are exemplified in Figure III.1.9. Size reduction is performed in both tests to accelerate reaching of equilibrium condition. Influence of pH on leaching with initial acid/base addition is to be evaluated with use of prCEN/TS 14429 (2003).

A further development of pH-controlled tests is WI 292XXX (2002). In the test, equilibrium condition is established at near neutral pH as a result of the reaction between pre-selected amounts of acid or base and test portion of the waste material. Also, in this test, size reduction is performed to accelerate reaching of equilibrium condition. Dissolved organic carbon (DOC) analyzed in the eluate provides a measure for biodegradability (e.g. see Figure III.1.10). Analytical data from the test on the influence of pH on the leaching behavior may be used for modeling metal–DOC interaction (Fig. III.1.11) that has been found to be an important factor affecting heavy metal release (Meima et al., 1999; EU/European Network project, 2000).

#### *III.1.3.3.2. Compliance tests*

The four procedures described in the four European Standards EN 12457–1/2/3/4 (2002) are one- or two-stage batch tests based on different *L/S* ratios. They deal with the specifications of a compliance test for leaching of granular waste materials and sludges under specific conditions. In these compliance tests, the final conditions of the test are imposed by the waste itself. The key factors influencing leaching, which are considered in these tests are contact time, *L/S* ratio, pH, reducing properties and the leaching of organic contaminants.

The compliance tests comprised by EN 12457–1/2/3/4:2002 are based on the assumption that equilibrium or pseudo-equilibrium is reached under the test conditions;

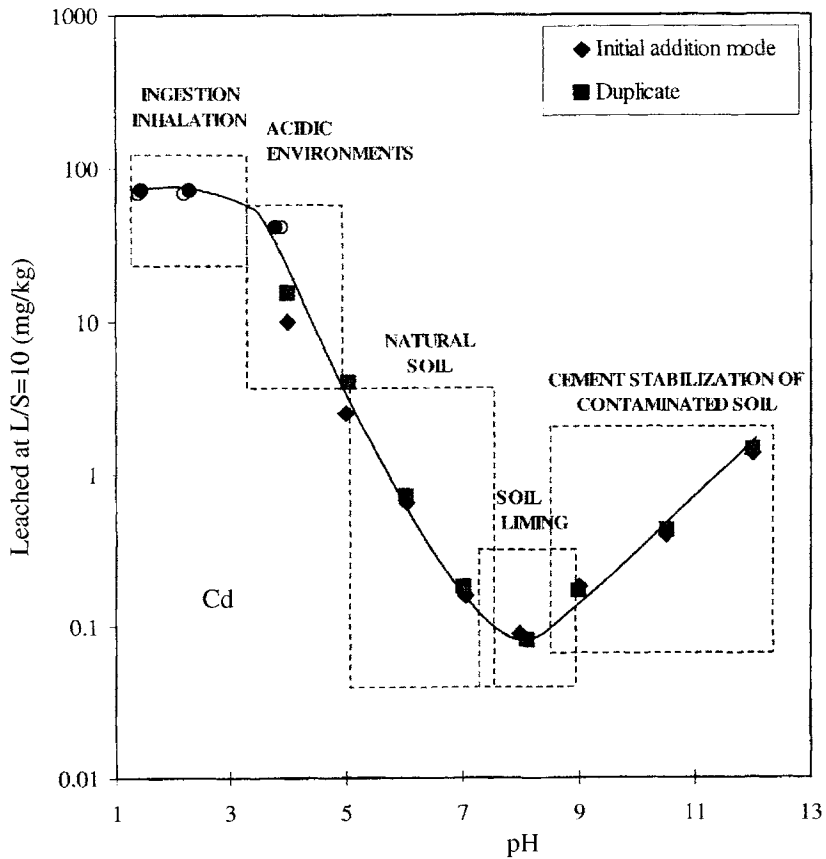


Figure III.1.8. Illustration of the influence of pH on the leaching behavior of Cd in a heavily sewage sludge amended soil in relation to different scenarios (test performed with initial acid/base addition) (after EU project SMT4-CT96-2066).

24 h are considered to be sufficient to reach this condition. Influence of  $L/S$  ratio is the major factor addressed in this standard. In its four parts, different  $L/S$  are specified (10, 8 and 2), leading generally to different test results. As in this standard the waste itself imposes the final conditions of the test, sample handling and storage, as well as laboratory preparation such as size reduction, performance of the leaching test and analysis tend to limit the changes of these factors induced by the external exposure or fine grinding. Considering that the leaching of organic contaminants is governed by processes, which differ substantially from that of inorganic contaminants and still are not well addressed, the standard specifies a scope that excludes organics.

The informative part of the standard (Annex A) underlines, that “the test results obtained with the compliance test specified in this standard only allow a direct comparison with regulatory limits on a pass/fail basis”. A comprehensive evaluation of the leaching behavior requires a basis or framework of reference such as that

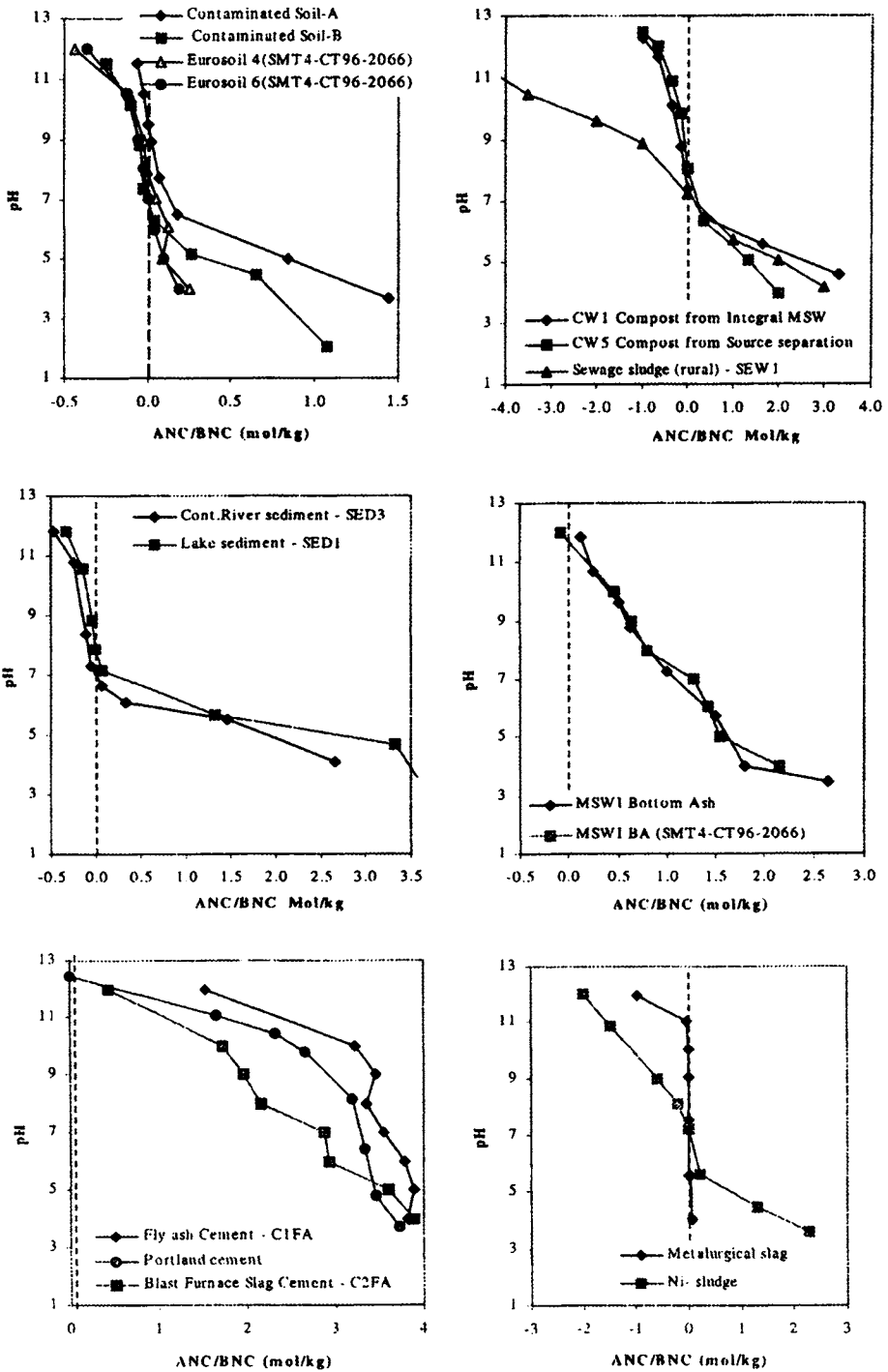


Figure III.1.9. Acid/base neutralization curves for different materials (after Meima et al., 1999; EU project SMT4-CT96-2066; van der Sloot et al., 2000). The base addition is given as negative values.

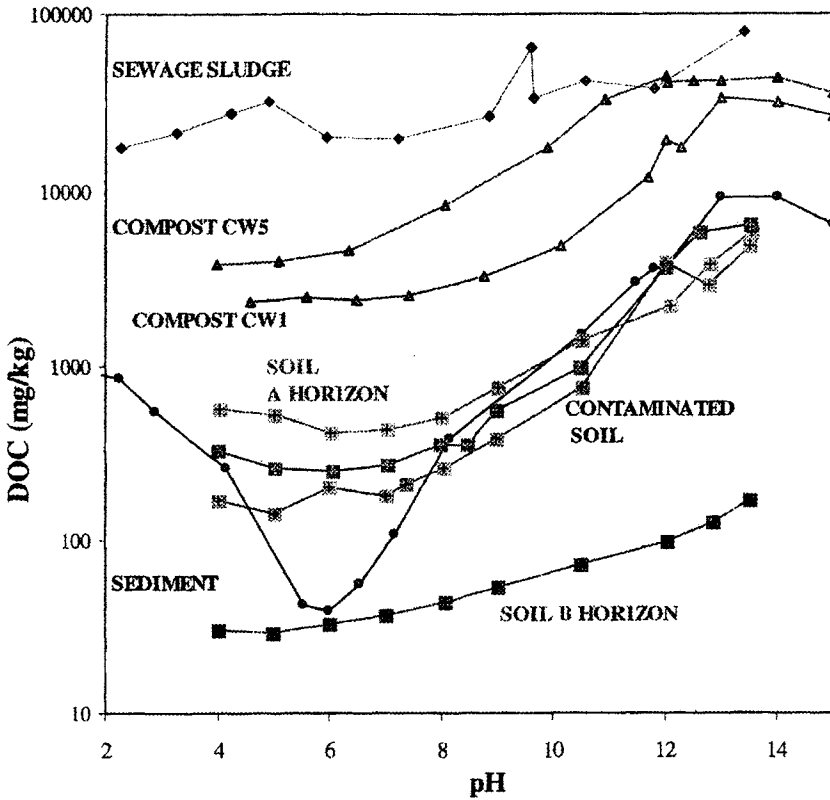


Figure III.1.10. Comparison of DOC as a function of pH for different bioactive materials illustrating the differences in response between fresh bioreactive materials and aged fully reacted materials (natural soil) (after EU project SMT4-CT96-2066).

provided by ENV 12920 (2003) "Methodology for the determination of the leaching behavior of waste".

It can be easily seen that the application and informative area of this test has considerable limitations, also for regulatory purposes, and is not relevant either to the full scope of waste materials (e.g. monolithic, organic, mixed organic-inorganic) or to the conditions of the environmental exposure. These, in general, display a much lower  $L/S$  ratio of infiltration water under the vadose zone conditions, and significant transformations of waste properties in time due to simultaneously occurring intrinsic processes of different kinetics induced by external factors. A leaching procedure for  $L/S = 2$  has been developed in view of assessing waste for landfill. In case some form of infiltration reduction is applied, an  $L/S$  ratio of about 1 may only be reached in  $>1000$  years (van der Sloot, 2000a). The transformations of waste properties within a much shorter time scale may cause dramatic changes in the leaching behavior of waste.

Nevertheless, this test is a valuable source of information on the contamination potential of waste at the moment of testing, and generally allows prediction of short-term

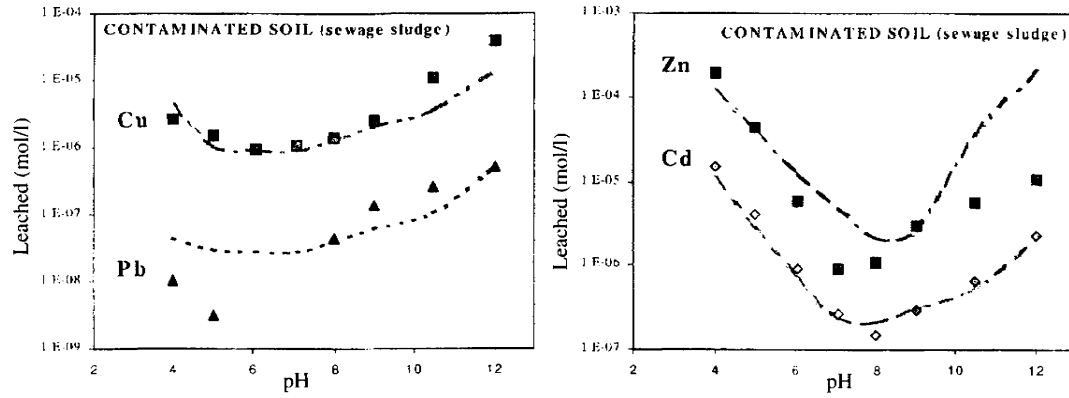


Figure III.1.11. Leaching behavior of Cd, Cu, Pb and Zn in a heavily sewage sludge amended soil. The main factors controlling metal mobility are the interaction of metals with particulate and dissolved organic matter (DOC). Geochemical speciation is modeled by ECOSAT computer program (after EU project SMT4-CT96-2066).

environmental behavior of waste in the wash-out (I) and dissolution (II) stages provided that no fast kinetically defined transformations occur within the time scale of these stages.

Leaching Tests EN 12457, 1–4 underwent in 2001 validation procedure of CEN/TC 292 (2001) in view of their possible use in a regulatory context, such as the EU Landfill Directive (EU, 1999); they were adopted as EU standards in 2002.

Construction with waste materials needs correct prediction of leaching behavior of cement-based solidified waste that has been studied in several research works (e.g. Kosson and van der Sloot, 1997; Tiruta-Barna et al., 2000, 2001). Compliance leaching tests for monolithic material (WI 292010, 2002; WI 292040) are now at the initial stage of development and anticipated to be ready for a Formal Vote in 2006. The proposed scope of this European standard is to determine the flow-through leaching behavior of these materials as a function of time. The test can be used to determine the dominant release mechanisms of inorganic constituents from regularly shaped specimens of monolithic wastes, including relatively water impermeable monoliths. It consists of a series of subsequent leachant renewal cycles, of which the contact time increases to reflect the predominant leaching mechanisms. At the development of the procedure, an extensive review of existing testing methods was utilized (CEN/TC 292/WG6, 2001). The assessment of the monolithic character of wastes is addressed in the draft EN standard WI 292031 (2002).

Dynamic leaching occurring in the anthropogenic (waste dump) and the natural vadose zone under the actual conditions of contaminant release and transport within the waste and in the underlying soil layer is addressed in the up-flow (prCEN/TS 14405 = WI 292034) and down-flow percolation simulation tests WI 292035 (Formal Vote and publication in 2003 for prCEN/TS 14405 and 2006 for WI 29035).

Recognition of the specificity of mining waste resulted in 2002 in the taking into consideration a preparation of a separate standard jointly with the CEN/TC 345 Soil Quality (CEN/TC 292, 2002d).

Another important direction for standardization is ecotoxicological testing of waste and different aspects of this issue (CEN/TC, 1999). The rationale behind this set of tests of different scope is that effects on living organisms goes through the liquid phase even in case of inhalation or ingestion, and that pH as a controlling factor of toxic constituents release in the environmentally accepted range 5–9 at low *L/S* (1–2) should be a basis for ecological testing (van der Sloot, 2000a). For this purpose, a modification of EN 12457 1 with manual pH control in the relevant pH window has been suggested as the most suitable one. In the waste, in which the role of dissolved organic matter is of importance, an upper boundary in the pH range reflecting the highest DOC and oxyanions (As, Mo, Sb, Se) and Cr (VI) mobility is considered to be relevant for ecotoxicity testing, while for predominantly inorganic waste a lower boundary in the pH range will reflect the highest mobility of metal cations (Pb, Cd, Zn, Ni, etc.).

The test on ecotoxicity (prEN 14735, 2003) aims to provide standardized test methods for ecotoxicological properties of raw waste and water extracts from waste that will describe the necessary steps to be performed before the ecotoxicological tests themselves, such as: taking the sample, transport, storage, preparation of raw waste sample and preparation of water extract to be tested. In a recognition of the applicability of other biological tests than those considered in the WI 292027 (2002), the extension to other applications and other biological tests by CEN/TC 262 is planned.

### III.1.3.3.3. Further directions of test development and validation

These and other standards under development constitute further steps towards the harmonization of tests for the environmental risk assessment from waste. Though the systematic leaching behavior of different waste materials has been already well documented (van der Sloot et al., 1991b, 1994a,b, 1996, 1997, 2000; Eighmy and van der Sloot, 1994; van der Sloot, 1996, 2000a) and leaching tests are in wide use for regulatory purposes as a tool for the environmental risk assessment from waste, there is also awareness that a single test is not a reliable method for long-term risk assessment, considering possible transformations of physico-chemical parameters of a waste in time. To ensure good confidence in the tests for this purpose, more sophisticated dynamic and sequential testing schemes (or combinations of weak and strong extractions), and a need of the validation of leaching/extraction tests in relation to the actual field conditions have been suggested (Quevauviller et al., 1996; van der Sloot et al., 1997).

An evidence of discrepancies of different nature between singular regulatory tests, long-term risk assessment based on accelerated simulation tests or predictive models and actual field conditions reported by different authors (Fällman and Hartlén, 1994; Jansen-Jurkovičová et al., 1994; Meij and Schaftenaar, 1994; Meij and te Winkel, 2000) has been supported by the case study on powerplant FA surface pond (Upper Silesia, Poland) reported elsewhere (Twardowska and Szczepańska, 2002) and discussed in detail in the Chapter III.7. This typical high-volume waste disposal site was subjected to field validation of the results of laboratory leaching/extraction tests and long-term column experiments on FA leaching behavior under controlled conditions for environmental risk assessment. The study proved inconsistency of the laboratory leaching tests and the actual leaching behavior of trace metals, particularly when equilibrium conditions are dictated by kinetically determined reactions; the test results reflected entirely wash-out (I) and dissolution (II) stages, but did not comprise the delayed release (III) stage. Life-cycle monitoring or singular screening of waste profiles at well-defined dumping sites (by waste age and hydrogeochemical characteristics) for contaminant release to the infiltration water as a function of the primary (pH–Eh, ionic strength, ionic composition of solute) and secondary controlling factors (actual *L/S* ratio, water percolation conditions) along the vertical profile of an anthropogenic or natural vadose zone can be utilized in the development of the long-term predictive hydrogeochemical models based on the input data from standard testing and their field validation. The pH (and Eh) as a function of time-dependent (kinetically defined) processes appeared to be a key issue for a correct prediction of the leaching behavior of waste. In the European Standardization, the influence of pH is considered to be covered by the Leaching Behavior Tests prCEN/TS 14429 (2003); WI 292033 under the development by CEN/TC 292/WG 6 with a final target date 2003. The influence of the specific conditions of the *L/S* phase contact is to be tested by the Leaching Behavior Tests ENV 12920 (methodology for the determination of the leaching behavior of waste under specified conditions), prEN14405 = WI 292034 and WI 292035 (up-flow and down-flow percolation simulation tests), and WI 292010 (compliance leaching test for monolithic material). Nevertheless, due to a high degree of simplification, these tests do not characterize well kinetically defined processes of contaminant generation and leaching.

Another area that is still not well addressed in standardization activity and needs more attention is the leaching of organic and inorganic contaminants from mixed and pure organic waste (EN 12457-1, Annex A, 2002). The difference in release and immobilization mechanisms (e.g. sorption) makes the leaching pattern different for organic and inorganic contaminants. The same source points out also a significant difference in properties and mechanisms of release between more polar, relatively water-soluble compounds and non-polar, hydrophobic organic contaminants.

Partially, these issues have been considered in WI 292033 (2003) “influence of pH on leaching with continuous pH control” and WI 292XXX (2002) “measure for biodegradability of waste”, where the difference of DOC release from fresh bioreactive material and aged fully reacted material as a function of pH (Fig. III.1.10), as well as interaction of metals with DOC as the main factor controlling metal mobility are illustrated (Fig. III.1.11). Nevertheless, much more attention should be paid to the leaching behavior of organic contaminants and to the interaction of organic–inorganic phases under different conditions, due to the abundance of mixed-phase waste.

#### **III.1.3.4. Waste analysis**

The discussion of methods of waste analysis are beyond the major scope of this chapter, though the European Standardization activity on characterization of waste comprises waste analysis and validation of analytical procedures as an integral part of standardization process. A number of standards on analytical procedures have currently received the status of an European standard: EN 13137 (2001) on determination of total organic carbon (TOC) in waste, sludge and sediments; EN 13656 (2002) on microwave-assisted digestion with hydrofluoric (HF), nitric (HNO<sub>3</sub>) and hydrochloric (HCl) acid mixture for subsequent determination of elements in waste; EN 13657 (2002) on digestion for subsequent determination of *aqua regia* soluble portion of elements in waste. Prior to the Formal Vote, the standards on waste digestion underwent a detailed validation procedure (Environment Institute JRC, 1999). Several standards concerning analysis of constituents in waste and eluates are in the last stage of voting before getting the status of National Standards of CEN member states, e.g. standards on determination of hydrocarbons in waste by gas chromatography (prEN 14039) and by gravimetry (prEN 14345), both were subject to validation procedure; also standards on calculation of dry matter (prEN 14346) and on determination of halogen and sulfur content, as well as oxygen combustion in closed systems (WI 292007 = prEN 14582). Analysis of eluates comprise European Standards 12506 (2003) on determination of pH, heavy metal ions, and anions Cl<sup>-</sup>, NO<sub>2</sub><sup>-</sup> and SO<sub>4</sub><sup>-</sup>; EN 13370 (2003) on analysis of ammonium-(N), AOX (sum of adsorbable organic halogen compounds), conductivity, Hg, phenol index, TOC, CN-easy liberable, F. Several standards on waste samples preparation (WI 292042) and pre-treatment (WI 292030), and determination of polychlorinated biphenyls (PCB) in waste (WI 292028) are in the initial stage of development.

Due to high mobility and high health and ecological hazards, much attention is paid to Cr(VI) determination in the environmental matter, though this task is considered difficult to handle because of instability of the oxidation states of Cr and the complex character of environmental samples (van der Sloot, 2000b; Jitaru et al., 2001). Analysis method for

determination of Cr(VI) in waste was published in 2003 (CEN/TR 14589, 2003). Target date for acceptance of a draft standard WI 292037 (2002) as European Standard is 2006. A non-destructive method of determination of waste elemental composition by X-ray fluorescence (XRF) was also accepted by CEN as a work item (WI 292038, 2002).

To date, a large number of different methods are available for the determination of the content of trace elements in different matrices. Most of them are designed for soil and sediment samples, and not for inorganic solid waste. These methods are often used at random and without proper justification, guidance and documentation, and considerable confusion exists in this area. An overview of the different methods, a discussion of the advantages and drawbacks associated with each method, along with the documentation on the comparative efficiency and the areas of application of various methods, in order to initiate a set of guidelines for the selection of methods for various purposes of waste analysis has been presented recently by Hjelm and Holm (1999). The scope of an overview comprises brief discussion of principles and descriptions of standard methods and novel methods or methods under development based both on destruction of the solid matrix, among them of digestion with strong acids and oxidizing mixtures, decomposition of samples by fusion and analysis of digested and decomposed samples, as well as non-destructive methods such as XRF spectroscopy, neutron activation analysis (NAA) and other techniques. On the basis of comparison of the different methods, the application of various types of methods has been summarized and discussed in relation to the scope of the analysis and the different types of matrices and elements to be considered. This project thus provides a simple guidance for the selection of suitable methods for determining the contents of various trace elements in various solid matrices for various purposes.

In particular, if the purpose of the analysis is to perform mass balance calculations of specific elements in different matrices and media, the complete destruction of matrix, e.g. with use of HF in conjunction with other acids or non-destructive methods for the total content analysis have been suggested. Digestion with e.g. HNO<sub>3</sub> or aqua regia could be used for samples not containing silicates.

It has been stated that since in several regulatory systems the results of analysis of waste and soil are based on partial digestion, these results can be used for comparative purposes with a clear specification of digestion method and a labeling the results as "partial content" to avoid confusion and wrong conclusions.

The final conclusion of the review points out the disadvantages of the methods based on total digestion of the matrices, which consisted in using aggressive and potentially hazardous acids and small-size samples. Non-destructive methods for the analysis of the total content are not yet effective enough to be used as a sole method and thus "should be further developed to yield sensitive and accurate analytical results based on fast, simple, non-expensive and non-destructive methodology" (Hjelm and Holm, 1999).

The question arises, whether a total content evaluation of an inorganic element is indeed of a crucial importance for environmental analysis and risk assessment from waste. The total or "nominal" metal concentration in a matrix does not give enough information on environmental risk, while contents of soluble metal species more closely reflects the bioavailable fraction (Gupta et al., 1996; Allen and Batley, 1997).

### **III.1.4. Evaluation of metal mobility in a matrix as a tool for risk assessment**

The European Standardization activity in the area of Environmental impact assessment of waste is focused mainly on inorganic contaminants, in particular heavy metals and their leachability. An approach that is not yet addressed in the European Standardization activity is the evaluation of metal fractions of different susceptibility to release in the waste matrix with use of a sequential extraction scheme. A number of studies on application of chemical extraction as a decision-making tool clearly confirm the reliability of this method for evaluation of risk assessment from waste (Prudent et al., 1996), sediments (Kersten and Förstner, 1986; Förstner and Kersten, 1988; Tack and Verloo, 1996) and soil (Gupta et al., 1996; Houba et al., 1996; McGrath, 1996; Quevauviller et al., 1996; Ure, 1996; Twardowska et al., 1999). For site- and use-specific actual and potential risk assessment from waste, as well as for estimating long-term effects of the changing controlling factors on metal release and leachability, the identification of metal-binding strength in matrix is of fundamental importance for evaluation of their susceptibility to mobilization under different exposure conditions with respect to different risk receptors, of which humans (adults and children), farm animals and wildlife, soil organisms and groundwater should be specified.

Recently, many authors involved in the project on harmonization of leaching/extraction tests for environmental risk assessment emphasize a necessity for determination of metal fractions of different mobility as a requirement for risk assessment (Gupta et al., 1996; McGrath, 1996; Ure, 1996). For this purpose, sequential extraction schemes for distinguishing metal-binding fractions appear to be an extremely useful tool. The concept of these schemes is that elements occur in the soil or waste matrix in various pools of different binding strength, which can be assessed by different reagents.

Since 1973, more than a dozen sequential extraction procedures using different extractants and defining from one to nine extraction schemes, mainly to identify chemical "forms" of metal binding have been developed, among them those by Tessier et al. (1979) modified by Kersten and Förstner (1986), Zeien and Brümmer (1989), Kaszycki and Hall (1996) and Han and Banin (2001) are currently the most widely used for general or specific purposes. The chemical extraction sequences by many authors are still subject to arguments concerning the selectivity of extractants and the redistribution of metals among phases during fractionation (e.g. Tessier and Campbell, 1991a,b; Xiao-Quan and Bin, 1993; Tack and Verloo, 1996; Hall and Pelchat, 1999). The attempts of many authors are focused on using a sequential extraction procedure mainly for the identification of chemical associations of pollutants in different organic–inorganic and mixed matrices. The greatest advantage of the chemical extraction sequences, though, is a possibility to differentiate between the fractions of different binding strength onto particular matrix and to compare partitioning in different organic, inorganic and complex matrices (Twardowska and Kyzioł, 2003). An extreme variety of waste materials with differing mechanisms of metal-binding needs to be tested for bioavailability, e.g. metal bonding onto material that is predominantly organic like fresh sewage sludge (e.g. Förstner et al., 1981), soil and solid waste particles that are predominantly inorganic (Förstner et al., 1981; Harrison et al., 1981; Lum et al., 1982; Twardowska et al., 1999), and complex material like municipal waste (Prudent et al., 1996). These materials vary with respect to

the fractions that are mobile thus reflecting differences in key metal transfer pathways from waste to risk receptors.

Of these pathways, groundwater is endangered by metal leaching from waste by percolating water; the food chain is considered the most important pathway leading from waste/contaminated soil to humans, farm animals and wildlife. Direct uptake is of importance for small children, grazing farm and wild animals, and soil organisms. The relevant fractions in waste or contaminated soil that reflect the health risk for the anthroposphere from these transfer paths are generally termed the mobile fraction (active = bioavailable and easily leachable), mobilizable (potentially bioavailable or leachable) and "pseudo total". The mobile fraction is a deciding factor for risk caused by leaching, mobile and mobilizable fractions reflect metal intake through the food chain, while pseudo total fraction is crucial for the direct ingestion of waste-soil particles under intestinal conditions. As extracting media for the mobile fraction, neutral unbuffered salt solutions are commonly used; mobilizable fractions are extracted with buffered and unbuffered complexing and chelating reagents, while for simulation of intestinal conditions, strong acid solutions are used (Gupta et al., 1996).

For the purposes of testing for binding strength of waste for metals, the optimum sequential extraction procedure should be simple both analytically and conceptually and display an order of a consecutive increase of binding strength. These are the properties shown by the most widely applied six-step sequential extraction procedure developed by Tessier et al. (1979) and modified by Kersten and Förstner (1986) for partitioning sediment samples, but which has also been used for different matrices, e.g. for soils (e.g. McGrath, 1996; Twardowska et al., 1999) or municipal waste (Prudent et al., 1996). Due to the variety of waste, in many cases it is rather difficult to identify the chemical forms of binding associated with each step. Nevertheless, the fractionation according to binding strength as a decisive parameter with use of this scheme ensures a very good confidence and repeatability. With respect to a certain group of materials (sediments and soils), the identification of major binding phases with use of this scheme is also possible. A growing number of extraction schemes and the advantages of this procedure as a useful tool for risk assessment from waste, clearly stresses the need for harmonization, identification of the areas of their applicability and standardization.

The need for standardization of methodology resulted in The Community Bureau of Reference (BCR) coordinating the development and validation of soil extraction schemes and in producing in 1995 two reference soils with certified extractable contents of a group of heavy metals (Ure, 1996). Also two Polish reference soils (PL-1 and BPGL-1) with certified extractable contents were prepared in parallel.

The metal aquatic toxicity testing for regulatory purposes requires consideration of metal species and a careful selection of the appropriate conditions for testing sparingly soluble substances that determine their bioavailability (Allen and Batley, 1997). In 1999, within the CEN/TC 292 *ad hoc* group on ecotoxicology of wastes has been established in order to provide standardized *test* methods as tools for the application of Annex III, Hazardous Waste Directive 91/689/EEC (EEC, 1991), which defines "ecotoxic" substances and preparations as the ones, which present or may present immediate or delayed risks for one or more sectors of the environment (CEN/TC 292, 1999). Ecotoxicity tests for raw wastes and water extracts from waste are currently under development; standard prEN 14735 (2003) describes preparation of waste samples for ecotoxicity tests.

### **III.1.5. Horizontal standardization**

Recently, the initiative has also been undertaken with the aim of development within the CEN Environmental and related TCs, and external liaison bodies of horizontal standards for relevant EU Directives and thus to produce, where possible, one standard as opposed to several elaborated in a vertical manner (CEN BT, 2001).

Besides standards applicable specifically for waste, there is a need to develop horizontal and harmonized European standards that are suitable for a wide range of materials, including waste, soil, sludge, and treated biowaste, lead to equivalent results and permit to avoid unnecessary differences in standards and duplication of work. The development of horizontal standards is aimed to facilitating implementation of upcoming EU Directives on sludge, biological treatment of biodegradable waste, and on the soil monitoring, as well as of the Council Directive 1999/31/EC on the landfill of waste. For this purpose, the collaborative European project HORIZONTAL started at the end of 2002 (CEN/TC 292, 2002c). Part of the work within the project will focus on co-normative horizontal standardization of existing ISO and CEN standards developed by the relevant TCs for the same parameters. Another part will comprise pre-normative research required to develop new needed horizontal standards for these materials. The workplan includes horizontal standards on sampling, on hygienic, biological, organic and inorganic parameters, on mechanical properties and leaching behavior of the most frequent contaminants in waste, sludge, treated biowaste and soils in Europe, in view of the potential impact on human and animal health, plant uptake, soil function and groundwater quality. In particular, the project considers an evaluation of: (i) inorganic compounds such as heavy metal cations, oxyanions and nutrients (N, P); (ii) volatile to semi-volatile compounds (chlorinated compounds etc.); (iii) strongly sorbed, non-volatile, relatively low water-soluble compounds (polycyclic aromatic hydrocarbons (PAHs), PCBs and phthalates); and (iv) soluble non-volatile organic compounds such as oxygenated and heterocyclic compounds.

In the process of horizontal standardization, a number of tests developed for the characterization of waste are considered to be evaluated in relation to their implementation in the Landfill Directive, and suitability for sewage sludge, soil and biowaste.

### **III.1.6. Conclusions**

In general, the current approach to the testing procedures for a short- and long-term environmental risk assessment from waste shows growing understanding of release mechanisms and factors controlling leaching behavior. This has resulted in developing testing schemes and scenarios based on the consideration of both intrinsic properties of waste material and external factors specific for the exposure conditions and interactions instead of a single regulatory test. The European standardization activity, which is directed to unification and harmonization of the numerous testing procedures, tends to simplification of the testing procedure based on the use of the observed geochemical similarity of the leaching behavior of waste and a limited number of parameters controlling the contaminants' release. Significant progress has been achieved in the development of a reliable testing procedure for prediction of short-term leaching behavior

within the wash-out (I) and dissolution (II) stages. Nevertheless, there are still considerable difficulties in the simulation of kinetically defined processes that in many cases determine long-term leaching behavior in the delayed release (III) stage, and the leaching behavior in the specific conditions of a liquid/solid phase contact (in particular, under vadose zone conditions). The current waste testing methods do not include site-specific evaluation of the environmental hazard, which should comprise interaction of waste with soil of the vadose zone into the waste testing procedures.

Another area that is not adequately addressed in standardization activity is the leaching of organic and inorganic contaminants from pure organic and mixed organic–inorganic waste due to the difference in the release and immobilization mechanisms. There is still limited progress in the harmonization and optimization of procedures for prediction of metal mobility and bioavailability in the waste matrix that is of a particular importance for site- and use-specific risk assessment from waste. The validation of laboratory data by field leaching studies for different solid wastes, their interpretation based on the understanding of the processes of contaminant generation and release, controlling factors and interactions under the actual conditions of exposure should ultimately lead to development of an optimized environmental evaluation scheme in order to make a correct decision concerning the life-cycle environmental sustainability of a waste site, which excludes both false-positive and false-negative errors.

The development of harmonized horizontal European standards suitable for waste, and for a wide range of other materials such as sludge, soil, and treated biowaste is anticipated to facilitate the European standardization and its implementation in the relevant regulatory fields governed by EU Directive on waste landfill and upcoming Directives on sludge, biowaste and soil.

## References

- Allen, H.E., Batley, G.E., 1997. Kinetics and equilibria of metal-containing materials: ramifications for aquatic toxicity testing for classification of sparingly soluble metals, inorganic metal compounds and minerals. *Hum. Ecol. Risk Assess.*, 3 (3), 397–413.
- CEN BT, 2001. Development of horizontal standards for EU directives on sludge, soil and biowaste. Draft Resolution BT C82/2001, N 6472, August.
- CEN/TC 292, 1999. Decisions of the meeting of the CEN/TC 292 *ad hoc* group “Ecotoxicology of wastes” – Paris – 990211, N 339.
- CEN/TC 292/WG1: TC292/WG 1, 2000. European standards for waste sampling – the story so far, N 435, May.
- CEN/TC 292/WG6, 2001. State of the art review from a standardization point of view on a dynamic leaching test for monolithic waste materials, N 239 (revised), May 2001, p. 41.
- CEN/TC 292, 2001. Validation of CEN/TC 292 leaching tests and eluate analysis methods PfEN 12457 part 1–4, ENV 13370 and ENV 12506 in co-operation with CEN/TC308, CEN.
- CEN/TC 292, 2002a. Examples of sampling scenarios, N 596.
- CEN/TC 292, 2002b. Overview of the scopes of (draft) standards of CEN/TC 292, N 602, May.
- CEN/TC 292, 2002c. Project HORIZONTAL: horizontal standards for implementation of EU directives on sludge, soil and treated biowaste, N 622, December, p. 37.
- CEN/TC 292, 2002d. Resolutions of the 17th meeting CEN/TC 292, N 639.
- CEN/TC 292, 2003. Time frame project HORIZONTAL, N 667, July, p. 4.
- EEC: Council Directive 91/689/EEC of 12 December 1991 on hazardous waste. OJ L 377, 31.12.1991, pp. 20–27.
- EC: Council Directive 1999/31/EC of 26 April 1999 on the landfill of waste. OJ L 182, 16.07.1999, pp. 1–19.

- Eighmy, T.T., van der Sloot, H.A., 1994. A unified approach to leaching behavior of waste materials, pp. 979–987. In: Goumans, J.J.J.M., van der Sloot, H.A., Aalbers, Th.G. (Eds), WASCON'94 Int. Conf. Environmental Aspects of Construction with Waste Materials, 1–3 June 1994, Maastricht, the Netherlands, Elsevier, Amsterdam, p. 988.
- EN 12457-1, 2002. Characterization of waste – leaching – compliance test for leaching of granular waste materials and sludges – part 1: one stage batch test at a liquid to solid ratio of 2 l/kg with high solid content and with particle size below 4 mm (without or with size reduction), CEN, Brussels.
- EN 12457-2, 2002. Characterization of waste – leaching – compliance test for leaching of granular waste materials and sludges – part 2: one stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 4 mm (without or with size reduction), CEN, Brussels.
- EN 12457-3, 2002. Characterization of waste – leaching – compliance test for leaching of granular waste materials and sludges: part 3: two stage batch test at a liquid to solid ratio of 2 l/kg and 8 l/kg for materials with high solid content and with particle size below 4 mm (without or with size reduction), CEN, Brussels.
- EN 12457-4, 2002. Characterization of waste – leaching – compliance test for leaching of granular waste materials and sludges: part 4: one stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 10 mm (without or with size reduction), CEN, Brussels.
- EN 13137, 2001. Characterization of waste – determination of total organic carbon (TOC) in waste, sludges and sediments, CEN, Brussels.
- EN 13656, 2002. Characterization of waste – microwave assisted digestion with hydrofluoric (HF), nitric (HNO<sub>3</sub>) and hydrochloric (HCl) acid mixture for subsequent determination of elements in waste, CEN, Brussels.
- EN 13657, 2002. Characterization of waste – digestion for subsequent determination of aqua regia soluble portion of elements in waste, CEN, Brussels.
- EN 12920, 2003. Characterization of waste – methodology guideline for the determination of leaching behavior of waste under specified conditions, CEN, Brussels.
- EN 12506, 2003. Characterization of waste – analysis of eluates – determination of pH, As, Ba, Cd, Cl<sup>-</sup>, Co, Cr, Cr(VI), Cu, Mo, Ni, NO<sub>2</sub><sup>-</sup>, Pb, total S, SO<sub>4</sub><sup>2-</sup>, V and Zn. CEN, Brussels.
- EN 13370, 2001. Characterization of waste – analysis of eluates – determination of ammonium-(N), AOX, conductivity, Hg, phenol index, TOC, CN<sup>-</sup> easy liberable, F. CEN, Brussels.
- Environment Institute JRC, 1999. Inter-laboratory test for validation of CEN/TC 292/WG 3 Draft Standards, Contract Number TR 14410-98, Final Report, Vol. 1–4, EC-JRC (EC Joint Research Centre), Ispra.
- EU/European Network, 2000. Technical work in support of the network on harmonization of leaching/extraction tests. EU project SMT4-CT96-2066, unpublished; website: <http://www.leaching.net/>.
- Fällman, A.M., Hartlén, J., 1994. Leaching slags and ashes – controlling factors in field experiments versus laboratory tests, pp. 39–54. In: Goumans, J.J.J.M., van der Sloot, H.A., Aalbers, Th.G. (Eds), WASCON'94 Int. Conf. Environmental Aspects of Construction with Waste Materials, 1–3 June 1994, Maastricht, the Netherlands, Elsevier, Amsterdam, p. 988.
- Förstner, U., Kersten, M., 1988. Assessment of metal mobility in dredged material and mine waste by pore water chemistry and solid speciation, pp. 214–237. In: Salomons, W., Förstner, U. (Eds), Chemistry and Biology of Solid Waste, Springer, Berlin, p. 305.
- Förstner, U., Calmano, W., Conradt, K., Jaksch, H., Schimkus, C., Schoer, J., 1981. Chemical speciation of heavy metals in solid waste materials (sewage sludge, mining wastes, dredged materials, polluted sediments) by sequential extraction, pp. 698–704. Proc. Int. Conf. Heavy Metals in the Environment, Amsterdam, 1981, CEP Consultants, Edinburgh.
- Gupta, S.K., Vollmer, M.K., Krebs, R., 1996. The importance of mobile, mobilizable and pseudo total heavy metal fractions in soil for three-level risk assessment and risk management. *Sci. Total Environ.*, 178, 11–20.
- Hall, G.E.M., Pelchat, P., 1999. Comparability of results obtained by the use of different selective extraction schemes for the determination of element forms in soils. *Water Air Soil Pollut.*, 112, 41–53.
- Han, F.X., Banin, A., 2001. Selective sequential dissolution techniques for trace metals in arid-zone soils: the carbonate dissolution step. *Commun. Soil Sci. Plant Anal.*, 32, 2691–2708.
- Harrison, R.M., Laxen, D.P.H., Wilson, S.J., 1981. Chemical associations of lead, cadmium, copper, and zinc in street dusts and roadside soils. *Environ. Sci. Technol.*, 15, 1378–1383.
- Hjelmar, O., Holm, P.E., 1999. Determination of total or partial trace element content in soil and inorganic waste material. Nordtest Report, NT Techn. Report 446, Espoo (Finland), p. 44.
- Hjelmar, O., Holm, P.E., Lehmann, N.K.J., 2000. Testing of soil and inorganic residues prior to utilization: development of rational limit values and adaptation of test methods. WASCON'2000 Abstracts, Int. Conf. on

- the Environmental and Technical Implications of Construction with Alternative Materials, 31 May–2 June 2000, Leeds/Harrogate, UK, Web site: <http://www.efm.leeds.ac.uk/wascon2000/>.
- Hockley, D.E., van der Sloot, H.A., Wijkstra, J., 1992. Waste–soil interactions, ECN-R-92-003, Netherlands Energy Research Foundation ECN, Petten (The Netherlands), p. 62.
- Houba, V.J.G., Lexmond, Th. M., Novozamsky, I., van der Lee, J.J., 1996. State of the art and future developments in soil analysis for bioavailability assessment. In: Ph Quevauviller (Ed.), Special Issue: Harmonization of Leaching/Extraction Tests for Environmental Risk Assessment, *Sci. Total Environ.*, 178, 21–28.
- Janssen-Jurkovičová, M., Hollman, G.G., Nass, M.M., Schuiling, R.D., 1994. Quality assessment of granular combustion residues by a standard column test: prediction versus reality, pp. 161–178. In: Goumans, J.J.J.M., van der Sloot, H.A., Aalbers, Th.G. (Eds), WASCON'94 Int. Conf. Environmental Aspects of Construction with Waste Materials, 1–3 June 1994, Maastricht, the Netherlands, Elsevier, Amsterdam, p. 988.
- Jitaru, P., Tirez, K., De Brucker, N., 2001. State of the art: chromium VI speciation in solid matrices, CEN/Draft Technical Report, ÖN – Austrian Standards Institute and CEN/TC 292/WG3, p. 36.
- Kaszycki, C.A., Hall, G.E.M., 1996. Application of phase selective and sequential extraction methodologies in surficial geochemistry, pp. 155–168. In: Bonham-Carter, G.F., Galley, A.G., Hall, G.E.M. (Eds), EXTECH I: A Multidisciplinary Approach to Massive Sulphide Research in the Rusty Lake – Snow Lake Greenstone Belts, Manitoba. Geological Survey of Canada, Bull. 426.
- Kersten, M., Förstner, U., 1986. Chemical fractionation of heavy metals in anoxic estuarine and coastal sediments. *Water Sci. Technol.*, 18, 121–130.
- Kosson, D.S., van der Sloot, H.A., 1997. Integration of testing protocols for evaluation of contaminant release from monolithic and granular wastes. In: Goumans, J.J.J.M., Senden, G.J., van der Sloot, H.A. (Eds), *Waste Materials in Construction – Putting Theory into Practice. Studies in Environmental Science 71*, Elsevier, Amsterdam, The Netherlands, pp. 201–216.
- Lum, K.R., Betteridge, J.S., MacDonald, R.R., 1982. The potential availability of P, Al, Ca, Co, Cr, Cu, Fe, Mn, Ni, Pb, and Zn in urban particulate matter. *Environ. Technol. Lett.*, 3, 57–62.
- McGrath, D., 1996. Application of single and sequential extraction procedures to polluted and unpolluted soils. *Sci. Total Environ.*, 178, 37–44.
- Meij, R., te Winkel, B.H., 2000. Seven years of experiments with lysimeter leaching of pulverized fly ash. WASCON'2000, International Conf. on the Environmental and Technical Implications of Construction with Alternative Materials, 31 May–2 June 2000, Leeds/Harrogate, UK, website: <http://www.efm.leeds.ac.uk/wascon2000/>.
- Meij, R., Schaftenaar, H.P.C., 1994. Hydrology and chemistry of pulverized fuel ash in a lysimeter or the translation of the results of the Dutch column leaching test into field conditions, pp. 491–506. In: Goumans, J.J.J.M., van der Sloot, H.A., Aalbers, Th.G. (Eds), WASCON'94 Int. Conf. Environmental Aspects of Construction with Waste Materials, 1–3 June 1994, Maastricht, the Netherlands, Elsevier, Amsterdam, p. 988.
- Meima, J.A., Van Zomeren, A., Comans, R.N.J., 1999. The complexation of Cu with dissolved organic carbon in municipal solid waste incinerator bottom ash leachates. *Environ. Sci. Technol.*, 33, 1424–1429.
- Odegard, K.E., Kartensen, K.H., Lund, W., 2000. Speciation of metals in soil solutions – the concept of forced-shift equilibration: quantification of a complexing ability of soil solutions. WASCON'2000, International Conf. on the Environmental and Technical Implications of Construction with Alternative Materials, 31 May–2 June 2000, Leeds/Harrogate, UK, website: <http://www.efm.leeds.ac.uk/wascon2000/>.
- prCEN/TR 14589, 2003. Characterization of waste – determination of chromium Cr(VI) in waste – state-of-the-art document. CEN, Brussels.
- prCEN/TS 14429, 2003. Characterization of waste — leaching behaviour tests — influence of pH on leaching with initial acid/base addition. CEN/TC 292/WG6 (Formal Vote 2003).
- prEN 14039, 2002. Characterization of waste – analysis of hydrocarbons (C<sub>10</sub> to C<sub>40</sub>) by gas chromatography. CEN/TC 292/WG 5 (target date for the Formal Vote 2003).
- prCEN/TS 14345. Characterization of waste – determination of hydrocarbons by gravimetry. CEN/TC 292/WG 5, status 2002 (target date for the Formal Vote 2003).
- prEN 14346. Characterization of waste – calculation of dry matter by determination of dry residue or water content. CEN/TC 292/WG 5, status 2002 (target date for the Formal Vote 2003).
- prCEN/TS 14405, 2003. Characterization of waste – leaching behaviour of a waste material under standardized percolation conditions – up-flow percolation test. CEN/TC 292/WG6 (Formal Vote 2003).

- prEN 14429. Characterization of waste – leaching behaviour tests – influence of pH on leaching with initial acid/base addition. CEN/TC 292/WG 6, status 2002 (target date for the Formal Vote 2003).
- prEN14582, 2003. Determination of halogen and sulfur content; oxygen combustion in closed systems and determination methods. CEN/TC 292/WG5 (target date for the Formal Vote 2005).
- prEN14735, 2003. Characterization of waste – preparation of waste samples for ecotoxicity tests. CEN/TC 292/WG7, (target date for the Formal Vote 2003).
- Project HORIZONTAL, 2003 (pending). Horizontal Standards for Implementation of EU Directives on Sludge, Soil and Treated Biowaste. ECN website: <http://www.ecn.nl/library/horizontal/>.
- Prudent, P., Domezel, M., Massiani, C., 1996. Chemical sequential extraction as decision-making tool: application to municipal solid waste and its individual constituents. *Sci. Total Environ.*, 178, 55–62.
- Quevauviller, Ph., van der Sloot, H.A., Ure, A., Muntau, H., Gomez, A., Rauret, G., 1996. Conclusions of the workshop: harmonization of leaching/extraction tests for environmental risk assessment. *Sci. Total Environ.*, 178, 133–139.
- Ramsey, M.H., 1993. Sampling and analytical quality control (SAX) for improved error estimation in the measurement of Pb in the environment using robust analysis of variance. *Appl. Geochem.*, Suppl. Issue No. 2, 149–153.
- Ramsey, M.H., Thompson, M., Hale, M., 1992. Objective evaluation of precision requirements for geochemical analysis using robust analysis of variance. *J. Geochem. Explor.*, 44, 33–36.
- TAC Landfill. Toolbox of testing methods and procedures for testing waste for landfilling, TAC Subcommittee on the Landfill Directive, version 30.05.2001.
- Tack, F.M., Verloo, M.G., 1996. Impact of single reagent extraction using  $\text{NH}_4\text{OAc-EDTA}$  on the solid phase distribution of metals in a contaminated dredged sediment. In: Ph Quevauviller (Ed.), Special Issue: Harmonization of Leaching/Extraction Tests for Environmental Risk Assessment, *Sci. Total Environ.*, 178, 29–36.
- Tessier, A., Campbell, P.G.C., 1990. Comment on pitfalls of sequential extractions by P.M.V. Nirel and F.M.M. Morel. *Water Res.*, 24, 1055–1056.
- Tessier, A., Campbell, P.G.C., 1991. *Water Res.*, 25, 115–117.
- Tessier, A., Campbell, P.G.C., Bison, M., 1979. Sequential extraction procedure for the speciation of particulate trace metals. *Anal. Chem.*, 51, 844–851.
- Tiruta-Barna, L., Imyim, A., Barna, R., Méhu, J., 2000. Prediction of inorganic pollutant release from various cement based materials in disposal/utilization scenario based on the application of a multi-parameter leaching tool box, pp. 318–324. In: Wooley, G.R., Goumans, J.J.J.M., Wainwright, P.J. (Eds), *Waste Materials in Construction: Science and Engineering of Recycling for Environmental Protection*, Pergamon, Amsterdam, The Netherlands, pp. 318–324.
- Tiruta-Barna, L., Barna, R., Moszkowicz, P., 2001. Modelling of solid/liquid/gas mass transfer for environmental evaluation of cement-based solidified waste. *Environ. Sci. Technol.*, 35, 149–156.
- Twardowska, I., Kyziol, J., 2003. Sorption of metals onto natural organic matter as a function of complexation and adsorbent–adsorbate contact mode. *Environ. Int.*, 28 (8), 783–791.
- Twardowska, I., Szczepanska, J., 2002. Solid waste: terminological and long-term environmental risk assessment problems exemplified in power plant fly ash study. *Sci. Total Environ.*, 285 (1–3), 29–51.
- Twardowska, I., Szczepanska, J., Witczak, S., 1988. *Impact of Coal Mining Waste on the Aquatic Environment: Risk Assessment, Prognosis, Prevention. Works and Studies 35*, Ossolinski National Publishers, Polish Academy of Sciences, Warsaw–Warszawa–Krakow–Gdansk, p. 251, in Polish.
- Twardowska, I., Schulte-Hostede, S., Ketrup, A.A.F., 1999. Heavy metal contamination in industrial areas and old deserted sites: investigation, monitoring, evaluation, and remedial concepts, pp. 273–319. In: Selim, H.M., Iskandar, I.K. (Eds), *Fate and Transport of Heavy Metals in the Vadose Zone*, Lewis Publishers, CRC Press, Boca Raton, p. 328.
- Ure, A.M., 1996. Single extraction schemes for soil analysis and related applications. In: Quevauviller, Ph. (Ed.), Special Issue: Harmonization of Leaching/Extraction Tests for Environmental Risk Assessment. *Sci. Total Environ.*, 178, pp. 3–10.
- US EPA SW-846, Test Methods for Evaluating Solid Waste. Physical and Chemical Methods, 3rd edn, T 1 ABC + T 2 novel 1.2. US EPA, Washington DC, 1989–2003 (continuously updated). Web sites: <http://www.epa.gov/epaoswer/hazwaste/test/main.htm>; <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>; <http://www.epa.gov/epaoswer/hazwaste/test/new-meth.htm>.

- van der Sloot, H.A., 1996. Developments in evaluating environmental impact from utilization of bulk inert wastes using laboratory leaching tests and field verification. *Waste Manag.*, 16 (1–3), 65–81.
- van der Sloot, H.A., 2000a. Ecological testing of waste: considerations on the Work of WG7, ECN, Petten (materials for the 13th meeting of CEN/TC 292 in Thessaloniki, Greece, unpublished).
- van der Sloot, H.A., 2000b. Topic – Cr VI in solid phase as discussed in WG 3, ECN, Petten (materials for the 13th meeting of CEN/TC 292 in Thessaloniki, Greece, unpublished).
- van der Sloot, H.A., 2002. Diagram with an overview of the role of sampling, subsampling, storage and pretreatment at different levels in the characterization of waste. CEN/TC 292, N 600, p. 1.
- van der Sloot, H.A., Hoede, D., 1997. Comparison of pH static leaching test data with ANC test data. ECN R-97-002, Petten, The Netherlands.
- van der Sloot, H.A., de Groot, G.J., Hoede, D., Wijkstra, J., 1991a. Mobility of trace elements derived from combustion residues and products containing these residues in soil and groundwater, ECN-R-91-008, Netherlands Energy Research Foundation ECN, Petten (The Netherlands), p. 33.
- van der Sloot, H.A., Hoede, D., Bonouvrie, P., 1991b. Comparison of different regulatory leaching test procedures for waste materials and construction materials, ECN-C-91-082, Netherlands Energy Research Foundation ECN, Petten (The Netherlands), p. 90.
- van der Sloot, H.A., Hjelmar, O., Aalbers, Th.G., Wahlstrom, M., Fällman, A.-M., 1993. Proposed leaching test for granular solid wastes, ECN-C-93-012, Netherlands Energy Research Foundation ECN, Petten (The Netherlands), p. 75.
- van der Sloot, H.A., Hoede, D., Comans, R.N.J., 1994a. The influence of reducing properties on leaching of elements from waste materials and construction materials, pp. 483–490. In: Goumans, J.J.J.M., van der Sloot, H.A., Aalbers, Th.G. (Eds), WASCON'94 Int. Conf. Environmental Aspects of Construction with Waste Materials, 1–3 June 1994, Maastricht, the Netherlands, Elsevier, Amsterdam, p. 988.
- van der Sloot, H.A., Kosson, D.S., Eighmy, T.T., Comans, R.N.J., Hjelmar, O., 1994b. Approach towards international standardization: a concise scheme for testing of granular waste leachability, pp. 453–466. In: Goumans, J.J.J.M., van der Sloot, H.A., Aalbers, Th.G. (Eds), WASCON'94 Int. Conf. Environmental Aspects of Construction with Waste Materials, 1–3 June 1994, Maastricht, the Netherlands, Elsevier, Amsterdam, p. 988.
- van der Sloot, H.A., Comans, R.N.J., Hjelmar, O., 1996. Similarities in the leaching behaviour of trace contaminants from waste, stabilized waste, construction materials and soils. *Sci. Total Environ.*, 178, 111–126.
- van der Sloot, H.A., Heasman, L., Quevauviller, Ph. (Eds), 1997. Harmonization of Leaching/Extraction tests. *Studies in Environmental Science*, Vol. 70, Elsevier Science, Amsterdam, 292 pp.
- van der Sloot, H.A., Rietra, R.P.J.J., Hoede, D., 2000. Evaluation of leaching behaviour of selected wastes designated as hazardous by means of basic characterization tests, ECN-C-00-050, Petten (The Netherlands).
- WI 292001, 2003. Characterization of waste — sampling of waste materials — framework for preparation of a sampling plan. CEN/TC 292/WG1 (target date for the Formal Vote 2004).
- WI 292002, 2001. Characterization of waste — sampling of waste materials — part 1: Information on selection and application of criteria for sampling under various conditions. CEN/TC 292/WG1 (target date for the Formal Vote 2004).
- WI 292010, 2002. Characterization of waste – compliance leaching test for monolithic material, CEN/TC 292/WG2 (target date for the Formal Vote 2006).
- WI 292017, 2001. Characterization of waste — sampling of waste materials — part 2: Information on sampling techniques. CEN/TC 292/WG1 (target date for the Formal Vote 2004).
- WI 292018, 2001. Characterization of waste — sampling of waste materials — part 3: Information on procedures for sub-sampling in the field. CEN/TC 292/WG1 (target date for the Formal Vote 2004).
- WI 292019, 2001. Characterization of waste — sampling of waste materials — part 4: Information on procedures for sample packaging, storage, preservation, transport and delivery. CEN/TC 292/WG1 (target date for the Formal Vote 2004).
- WI 292028, 2003. Characterization of waste – determination of polychlorinated biphenyls (PCB) in waste. CEN/TC 292/WG5, (target date for the Formal Vote 2006).
- WI 292030, 2003. Characterization of waste – preparation of a test portion from the laboratory sample. 292/WG3 (target date for the Formal Vote 2006).
- WI 292031. Characterization of waste – assessment of the monolithic character. CEN/TC 292/WG 2, status 2002 (target date for the Formal Vote 2005).

- WI 292033, 2003. Characterization of waste – leaching behaviour tests – influence of pH on leaching with continuous pH control. CEN/TC 292/WG6 (target date for the Formal Vote 2004).
- WI 292035. Characterization of waste – simulation of the leaching behaviour of a waste material under specific conditions – down-flow percolation test. CEN/TC 292/WG6, status 2003 (target date for the Formal Vote 2006).
- WI 292037. Characterization of waste – determination of chromium Cr(VI) in waste – analysis method. CEN/TC 292/WG3, status 2003 (target date for the Formal Vote 2006).
- WI 292038. Characterization of waste – determination of elemental composition by X-ray fluorescence. CEN/TC 292/WG3, status 2003 (target date for the Formal Vote 2007).
- WI 292040. Characterization of waste – dynamic leaching test for monolithic waste. CEN/TC 292/WG6, status 2003 (target data for a Formal Vote 2006).
- WI 292041, 2003. Characterization of waste — sampling of waste materials — part 5: guidance on the process of defining the sampling plan. 292/WG1 (target date for the Formal Vote 2005).
- WI 292042, 2003. Characterization of waste — digestion of waste samples using alkali-fuzion techniques. 292/WG3, (target date for the Formal Vote 2006).
- WI 292046, 2003. Characterization of waste – leaching behaviour tests – acid and base neutralization capacity test. CEN/TC 292/WG 6 (target data for a Formal Vote 2005).
- WI 292XXX, 2002. Characterization of waste – leaching behaviour tests – measure for biodegradability of waste. CEN/TC 292/WG 6 (no target data for a Formal Vote).
- Xiao-Quan, S., Bin, C., 1993. Evaluation of sequential extraction for speciation of trace metals in model plenary soil containing natural minerals and humic acid. *Anal. Chem.*, 65, 802–807.
- Zeien, H., Brümmer, G.W., 1989. Chemische extraktionen zur bestimmung von schwermetallbindungsformen. *Böden. Mitt. Dtsch Bodenkund. Gsch.*, 59/I, 505–510, in German.