Primary Raw Materials

Alternative raw materials

Stage 1: Raw material supplies

Stage 2: Manufacture of construction materials and elements

Stage 3: Construction Process

Stage 4: Service Life

Stage 5: Demolition

Release into the environment

Energy

Dust, noise emissions

End of Life

Supply of information on technical and environmental quality

Characterisation of monolith/granular leaching behaviour

Monolith/granular QC and compliance leaching test

Environmental impact (leaching)

Release into the environment

Contaminated soil

Infiltration

Field pH

Roadbase

Infiltration

Groundwater

Energy

Characterisation of granular leaching behaviour

Granular compliance test

Field pH

Defining characteristics

Dust, noise emissions

‘End of Life’
The mission of the IES is to provide scientific and technical support to EU policies for the protection of the environment contributing to sustainable development in Europe.
WORKSHOP PROCEEDINGS

PROBLEMS AROUND SOIL AND WASTE I
- HORIZONTAL ASPECTS OF LEACHING

DG JRC WORKSHOP
ISPRA, 14-15 FEBRUARY 2005

B. M. GAWLIK AND G. BIDOGLIO (EDS.)
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Introduction

**Background information**

With its new workshop series “Problems around Soil and Waste”, the European Commission DG JRC’s Institute for Environment and Sustainability is highlighting those technical regarding the characterization of soil and waste, which are relevant for the respective EU legislation. In this context, the question was raised whether the development of a commonly underlying approach towards the use of leaching test for soil, soil-related matrices, waste and also other solid materials would facilitate the implementation of EU policies. This initiative goes along with a strong support of the JRC for the activities related to horizontal standardization, in particular as performed in the so-called Project HORIZONTAL, dealing among other with the development of an across-matrix standardization framework for soil, (treated) biowaste and sludges.

The judgment of impact on soil and groundwater from a wide range of anthropogenic activities is hampered by (1) the wide variety of assessment tools and approaches and (2) an unclear situation with respect to objectives aimed for.

Impacts on soil and groundwater may result from, e.g., utilisation of alternative materials in road construction, application of construction materials containing increasing amounts of secondary materials, disposal of waste, treated wood application, release from contaminated sites, agricultural activities, etc. If all of these impacts need to be judged, there is a gain to be made in harmonising the approaches to assess impact from the wide range of different materials. In fact, the mechanisms leading to release and factors controlling release are not unique properties for specific material classes, but rather generic (chemical) mechanisms that are commonly applicable to all materials. There are obvious differences in specific aspects (e.g. reducing properties, type of organic matter, pH), which may vary between materials. Also, release behaviour of specific contaminants is more systematic than generally recognised. Often too simple tests are used to try and assess impacts. This is counter productive as a hierarchy in testing allows detail, when needed, and simplicity, when sufficient knowledge is available. Already, a toolbox of suitable test methods is available and standardised or almost finalised as harmonised standards in CEN. Also, the approach to link this information describing a source term for a given material to impact evaluation on soil, surface- and groundwater has advanced significantly in recent years. In addition, basic characterisation data are now available for a large number of materials (construction materials, such as concrete, asphalt, brick, lime silicate bricks, aggregates, roofing materials, drinking water pipes, preserved wood, soil, contaminated soil, sediments, sludges, compost, a wide variety of wastes and stabilised wastes), which show that specific materials and material classes have very similar leaching characteristics, which in turn implies that unnecessary duplication of work can be avoided.

**Aim of the Workshop**

The above evaluation of the current situation with respect to impact evaluation to soil and groundwater is relevant for several EU Directives in preparation. It is important to evaluate the degree to which these observations are recognised by others and can be taken as starting point for subsequent work in validating the basic characterisation leaching tools developed in CEN TC 292 for a wider range of materials than strictly
Introduction

belonging to CEN TC 292 work field (wastes, stabilised waste, mining waste), such as soil (covered in ISO/ TC190/SC7/WG6) and in the horizontal test development in connection with the Construction Products Directive (CPD). The ultimate goal is to develop a scenario based evaluation similar to the one used in the development of criteria for the EU landfill Directive (Annex II) for different material classes for which regulation needs to be developed now or in the near future. Further there is a need to develop a European database/expert system containing basic characterisation data for the wide range of materials for which decisions need to be made. Development of a framework of testing, which makes use of a hierarchy in testing: more detailed characterisation to understand the problem and short compliance tests to verify compliance with earlier characterisation data.

Benefits

The major benefit in a common approach to assess impact from widely different materials through leaching in different applications is the transparency in regulation. Impact on soil and groundwater caused by, for instance, landfill leachate or percolate from a roadbase is not fundamentally different. The utilization of a limited selection of assessment tools ensures comparability between fields as well as between different applications of the same material. Maximum exchange between fields can be obtained in this manner. Significant gain in efficiency in standardisation work and in testing can be realized and duplication of work can be avoided. A Europe-wide accessible database/expert system will provide users at different levels with the most relevant and up to date information on material characteristics. The conclusion may well be that steel slag from Sweden is not significantly different in leaching behaviour from steel slag produced in Spain and in Germany. This is particularly relevant for the new Member States, where the availability of resources is limited and significant steps in understanding the problem to be dealt with can be achieved.
Harmonisation of leaching approaches  
– a challenge for legislation

Presented by
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Abstract

Surface water, drinking water, soil and groundwater can be polluted by different types of discharges, such as contaminants released from polluted soil, landfills, primary and recycled construction materials, preserved wood, drinking water pipes, etc. Those sources fall under different national legislations and policies and under different EU-direc-
tives and EU policies.

For some types of discharges criteria and limit values are set on a national or even on EU-level. For other sources criteria and limit values are set on a local or regional level by competent authorities, often on a case by case basis.

The same materials and products may fall under different regulations and policy fields. For example, concrete can be used as construction product, but also as material for drinking water pipes. Waste materials can be landfilled, but often also (in part) recycled as construction material. Preserved wood can be used for tables and chairs in gardens, but also as construction material. In each of these product areas different test methods are available or under development at Member State level or at EU-/CEN level.

The awareness of the need for harmonization within the sector of environmental legislation is growing. However at European level, there is still not a strong incentive formulated on harmonization of leaching tests from the environmental sector. The project ‘Horizontal’ included some starting points for harmonization of leaching tests, but this has not received a high priority, within this project.

The Construction sector feels the need for urgent harmonization of all kinds of test methods for evaluating the quality of construction products. The ‘New Approach’ Construction Products Directive (CPD) aims at development of harmonised CEN-product standards for all kinds of construction products. More than a hundred of such standards are available now, concerning technical parameters. Now a program has been started to select and harmonize test methods for evaluation of ‘dangerous substances’, released from or available in construction products. Mandated by the EU (DG-Enterprise) CEN will elaborate a working plan in 2005 and select and harmonize such test methods in the near future.

This process will cover most legislative environmental sectors, since construction products are produced with many types of primary and secondary materials in many
different situations. So this pressure on development of a harmonised market will influence the whole environmental sector.

It is important to join forces and to merge the needs emerging from the CPD and the knowledge, experience and risk assessment demands from the environmental sector to realise harmonization; methods which fit both the needs of the producers of construction products and other products to comply as well as possible for the setting of limit values in the environmental sector. It is a real challenge to make this combination work. Not in the least because of the strongly vertical (by product type) organized CEN structure.

Recently in several occasions experiences on leaching in different sectors were indicatively compared and discussed. It strongly looks like that in many cases the same mechanisms dictate the leaching process. This knowledge provides a good basis for further harmonization and for making progress in development of adequate test methods and harmonised evaluation procedures that can cover a range of product types.

It is evident that the three step approach of the Landfill Directive (1.Characterisation/Evaluation, 2.Compliance and 3.On site verification) can structure development and use of test methods significantly. It proved to be impossible to develop a simple test that provides quick and cheap answers which also give sufficient insight in the risks associated release of substances. Splitting up may help. Characterization and evaluation can be done with some test procedures, that provide good insight in the risks of a specific product. Availability of a database with data from similar products may reduce costs and time for evaluation of individual cases. Based on this information, routine testing for compliance purposes can be reduced to critical parameters. Under the CPD, terms as ‘Initial Type Testing’ (ITT) and ‘Factory Production Control’ (FPC) are rather similar to ‘Characterization’ and ‘Compliance’.

For common understanding it is important that different approaches converge. Producers of materials and products stress the selection of tests that can serve to provide answers for different environmental sectors in a simple and not to expensive way. In many cases evaluation approaches started with just the selection of a certain leaching test.

The ‘environmental sector’ often starts with defining what kind of protection is aimed at and what information is needed for adequate risk assessment or other environmental assessments, e.g. source oriented assessments. So starting from different starting points it is a challenge to find ways to meet each other in an adequate way. A lot of knowledge and experience is already available. Now it is time to bring this information together.
Harmonisation of leaching approaches – a challenge for legislation

Rein Eikelboom

Direct / Indirect discharges into ground water

Pollution by leaching

Sources
- Constructions
- Waste-store/landfill
- Garden furniture, Boats, Cars, etc
- Drinking water materials
- Food production facilities
- Food containment

Targets
- Surface water
- Ground water
- Soil
- Indoor and outdoor air
- Human health
- Animal care
- Plants

Interactions with aquatic and terrestrial ecosystems? Links with SW status and EQS

Risks of pollution from diffuse/point sources (incl. landfills, wastes, contaminated soils, agriculture)

Prevent / Limit

Run-off

Quality standards / thresholds?

Trend identification and reversal

Time
## Policy and legislation

### EU-Legislation

<table>
<thead>
<tr>
<th>Directive/Strategy</th>
<th>Level of technical instructions</th>
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<tbody>
<tr>
<td>Water Framework Directive 2000/60/EC,</td>
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<td>Soil Strategy (... under development)</td>
<td>national, local, permits</td>
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### Policy and legislation

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### Policy and legislation – CPD and Test instructions

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Harmonisation of leaching approaches

**Release; products oriented approach**

- Sources (products, waste, soil, etc)
- Leaching test methods
- Product Scenario approach
- Assessment effects to the environment
- Evaluation

**Evaluation; environment oriented approach**

- Product Scenario's
- Limit Values on product quality
- Limit Values on products related to burdening of the environment
- General Limit Values on environmental quality
- Environmental system approach
- Environmental risks
  - soil, water, drinking water, air, food, etc.
- General environmental Criteria
- Environmental / health protection
  - soil, water, drinking water, air, food, etc.

**Combined; release evaluation approach**

- Sources (products, waste, soil, etc)
- Leaching test methods
- Product Scenario approach
- Limit Values on product quality
- Limit Values on products related to burdening of the environment
- General Limit Values on environmental quality
- Environmental system approach
- Environmental risks
  - soil, water, drinking water, air, food, etc.
- General environmental Criteria
- Environmental / health protection
  - soil, water, drinking water, air, food, etc.
Testing levels

1. Characterisation + general evaluation
   (Use of all available data (+data base)
   and all relevant test methods.)

2. Compliance testing
   (simplified testing on critical parameters only,
   based on characterisation tests.)

3. Acceptation procedures
   (Mainly visual and administrative procedures; only short tests or
   ‘compliance like tests’ if strictly needed.)

Environmental evaluation schemes

EU-legislation/policy
- Water Framework Directive
- Groundwater directive
- Soil Strategy
- Drinking water
- Waste
- Substances
- Air
- Biocides
- Construction products directive

EU-Environmental criteria development, including leaching
- Guidelines WG2C - WG3-Discharges
- EAS (European Acceptation Schemes)
- Landfill Directive; giving selected test procedures
- WHO/EU statements demanding adequate approaches. (e.g. WHO air guidelines; EHAP / NEHAP)
- Evaluation procedures (e.g. wood preservation)
- Mandate; demanding uniform test methods

Time tables (examples)
- Landfill Directive
  - Introduction in national legislation: July 2005
  (as far as available in CEN)
- CPD
  - Mandate
  Acceptation: beginning 2006
  Harmonisation: 2006 - 2008-2010?
- Drinking water
  - Going on procedure (EAS); some parts available,
  some parts under development
- WFD/GWD
Questions

- Is a transparent uniform systematic approach of leaching possible for different materials and sectors?
- Is it possible to limit the total number of test methods, without loss of relevant information?
- Is it clear what the real needs of information are from the different environmental sectors?
- How can we give adequate guidance to further developments? (resulting in an efficient, transparent and useful system of test procedures and approaches for legislation and public parties)

Conclusions / actions

- Different types of legislation overlap,
- Same Products may fall under several environmental legislations,
  → So, further harmonisation is necessary for legislation, the environment, industry, local authorities and others.
  → The environmental sector should make clear what their demands are on evaluation of materials that may leach.
  → Actual demands from e.g. WFD/GWD, CPD, Drinking water, Biocides directive, etc, should be the basis for an integrated program of further developments.
Presentations

Leaching and standardisation developments – a status report

Presented by
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Abstract

Standardisation of leaching tests for waste and soil takes place in within CEN (European level) and ISO (globally). CEN/TC 292: “Characterization of waste” is responsible for standardisation of leaching tests for waste, and ISO/TC 190/SC7/WG6: “Soil and site assessment: Leaching” is responsible for standardisation of leaching tests for soil. In addition, a new European technical committee, CEN/TC 345: “Characterization of soil”, has been formed with the purpose of reviewing the ISO standards on soil analysis and testing and to propose those that fulfil the requirements in the EU policy on soils as European (CEN) standards.

From a European point of view, CEN standards are more important than ISO standards since they are incorporated into EU legislation and often developed under mandate from the EU Commission. ISO standards may coexist with similar national standards in Europe, whereas parallel national standards automatically are cancelled by CEN standards in the CEN member states. ISO standards that are likely to be adapted as CEN standards are, of course, equally important.

The test hierarchy (basic characterization, compliance testing and on-site verification), which was developed by CEN/TC 292, has proven a very useful administrative tool as far as waste is concerned and has been incorporated into EU legislation (e.g. the Landfill Directive) as well as national legislation. For waste, basic characterization generally applies to a major waste stream or waste production unit, whereas for soil, basic characterization rather describes the general knowledge about the leaching behaviour of soils as a function of soil properties and external influences. The tiered approach will, however, be useful in both cases.

Another useful tool developed by CEN/TC 292 is prEN 12920: Characterization of waste – Methodology for the determination of the leaching behaviour of waste under specified conditions. This pre-standard, which is expected to gain status as a proper standard (EN) by the end of 2005, is a guideline for the use of leaching tests for various purposes such as e.g. impact assessment. One of the key points of prEN 12920, which is outlined schematically in figure 1, is to start by clearly defining the problem at hand and asking the right questions in a precise manner. Relevant questions could for instance be: What is the flux of leached (specified) contaminants from a (specified) waste material in a (specified) utilisation or landfill scenario under (specified) climatic conditions as a function of time? and: How can the relationship between the resulting concentration of a contaminant in the groundwater at a certain distance downstream of the waste application and the result of a laboratory leaching test on that material be established? Once the
O. Hjelmar

problem and the solutions sought are clearly defined, the methodology proceeds with technical descriptions of the waste construction scenario and the surrounding environmental and climatic scenario, description of the geotechnical and chemical properties of the waste, selection of the correct leaching methods to investigate the leaching properties as a function of L/S and pH as well as the influence of various internal (waste) properties and external factors on the release of contaminants. When the appropriate results of the leaching tests have been procured, a suitable model describing the problem under investigation must be selected/developed, set up and run. If possible, the model should be validated, e.g. by lysimeter test results or field observations, before the final conclusions are drawn. The conclusions may be that the problem is solved, that it cannot be solved or that it may be solved if more information is gathered at one or more stages of the procedure.

Figure 1 - Principle of the methodology described in prEN 12920.
CEN/TC 292 has two working groups (WGs) dedicated to the standardisation of leaching tests. WG2 works with compliance leaching tests, and WG6 with characterization leaching tests. A list of waste leaching standards developed or currently being developed by CEN/TC 292 is shown in Appendix 1. The list includes five basic characterization leaching tests (two percolation tests, two pH-dependence leaching tests – all to be carried out on granular or crushed material under conditions approaching equilibrium - and one dynamic leaching test for monolithic waste) and two compliance leaching tests (one batch leaching test with four options related to L/S ratio and particle size to be carried out on granular waste, in most cases under equilibrium-like conditions, and one leaching test for monolithic material).

The tests developed so far by CEN/TC 292 are applicable mainly to the determination of the leaching behaviour of inorganic constituents (although the leaching of DOC and some highly soluble organic species such as phenols are often included). A proposal for a new work item dealing with the leaching of organic constituents has been put forward by WG6, but no work has commenced yet. There are a number of similarities between leaching mechanisms of organics and inorganics (in terms of flow regimes – percolation/diffusion) but the chemistry of organics is different from that of inorganics, and leaching and test requirements for specific organic components depends strongly on solubility, DOC and pH (which also affects DOC). Biodegradation of organics must be taken into account. In general, it is technically much more difficult to get meaningful results from leaching of organics than from leaching of inorganics, particularly for organics with low solubility.

The usefulness of a standard leaching test for instance in a regulatory framework is obviously dependent on its reliability, i.e. its ruggedness, repeatability and reproducibility. These properties are generally determined by performing a so-called validation with participation of many laboratories. It has proven very hard to raise the funding necessary for proper validation of the leaching standards developed by CEN/TC 292. So far, only the compliance leaching standard EN 12457 – part 1-4 has been validated for a number of largely inorganic constituents. CEN/TC 292 has decided only to issue standards that have been properly validated. Method descriptions that have not been validated will be published as Technical Specifications (CEN/TS).

ISO/TC 190: Soil Quality, SC7: Soil and site assessment, WG6: Leaching, is currently developing four leaching standards for soils (two batch leaching tests at L/S = 2 l/kg and L/S = 10 l/kg, one percolation test and one pH dependence test). These tests are all based on the corresponding leaching tests from CEN/TC 292 but with minor adjustments to account for the differences between waste and soil (e.g. by using a $10^{-3}$ M CaCl$_2$ solution instead of demineralised water to facilitate the separation of fine clay particles from the eluent). However, the leaching standards developed by ISO/TC 190/SC7/WG6 have a much broader scope than those developed by CEN/TC 292: In their current form they are meant to apply both to inorganic and (non-volatile) organic constituents and to provide eluate for chemical analysis and ecotoxicological testing. If a very stringent procedure is followed, particularly with respect to choice of materials and separation of eluate and soil, it may be possible to determine the release of some organics and inorganics in the same procedure. If only inorganic contaminants are of interest, a much less stringent procedure may often be followed. The current versions of the standards do not provide sufficient guidance on these issues, but they are being amended and will hopefully improve considerably before they are issued as finalised standards. This is important.
because ISO/TC 190 does not require standards to be validated before they are issued. A list of the leaching tests for soil being developed by ISO/TC 190: Soil Quality, SC7: Soil and site assessment, WG6: Leaching, is also shown in Appendix 1.

Appendix 1 - Leaching standards developed or under development by CEN/TC 292 and ISO/TC 190

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<td>CEN/TS 14405</td>
<td>Characterization of waste: Leaching behaviour tests – Up-flow percolation test (under specified conditions)</td>
<td>Published 06-2004</td>
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<tr>
<td>WI 292035</td>
<td>Characterization of waste: Leaching behaviour tests – Simulation of the leaching behaviour of a waste under specified conditions - Down-flow percolation test</td>
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<td>prEN 14977</td>
<td>Characterization of waste: Leaching behaviour tests – Influence of pH on leaching with continuous pH control</td>
<td>End of 2005</td>
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<tr>
<td>prCEN/TS 14429</td>
<td>Characterization of waste: Leaching behaviour tests – Influence of pH on leaching with initial acid/base addition</td>
<td>2005</td>
</tr>
<tr>
<td>WI 292040</td>
<td>Characterization of waste: Leaching behaviour tests – Dynamic leaching test for monolithic waste</td>
<td>After 2005</td>
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</table>

ENV 12920: Characterization of waste – Methodology for the determination of the leaching behaviour of waste under specified conditions. Published: 06-1998. Under conversion to EN.
A new work item, Leaching tests for organic constituents, is under consideration.

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<td>Published 11-2002</td>
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<td></td>
<td>Part 1: One stage batch test at a liquid to solid ratio of 2 l/kg for materials with high solid content and with particle size below 4 mm (without or with size reduction).</td>
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<tr>
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<tr>
<td></td>
<td>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).</td>
<td></td>
</tr>
<tr>
<td></td>
<td>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).</td>
<td></td>
</tr>
<tr>
<td>WI 292010</td>
<td>Characterization of waste – Leaching – Compliance test for monolithic material</td>
<td>After 2005</td>
</tr>
</tbody>
</table>
### Leaching tests developed/under development by ISO/TC 190/SC7/WG6

<table>
<thead>
<tr>
<th>ID</th>
<th>Title</th>
<th>Finalised (expected)</th>
</tr>
</thead>
</table>

Problems around Soil and Waste I
Horizontal aspects of leaching

Leaching and standardisation developments - a status report

Ole Hjelmar
DHI – Water & Environment

JRC Workshop
European Commission DG JRC
Ispra Site, Italy
14 – 15 February 2005

Standardisation of leaching tests for waste and soil

CEN/TC 292: Characterisation of waste
WG 2 (Compliance leaching tests)
WG 6 (Characterisation leaching tests)
Work on leaching tests started in 1992

ISO/TC 190: Soil Quality
SC7/WG6: Soil and site assessment: Leaching
Work started approx. 1998

CEN/TC 292: Hierarchy of testing

Basic characterisation
Information on short and long term leaching behaviour and characteristic properties of materials. L/S ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity and physical parameters are addressed in these tests.

Compliance testing
Compliance tests are used to determine whether the waste complies with specific reference values. The tests focus on key values and leaching behaviour identified by basic characterisation tests.

On site verification
On-site verification tests are used as a rapid check to confirm that the material is the same as that which has been subjected to the compliance test(s).
CEN/TC 292: Characterisation of waste

prEN 12920:
Methodology guideline
for the determination of
the leaching behaviour of
waste under specified
conditions

ENV published 1998

CEN enquiry until 11
April 2005

CEN/TC 292: Characterisation tests

CEN/TS 14405: Up-flow
percolation test (under
specified conditions)

Granular materials, largely
inorganic components,
based on local equilibrium
assumption (LEA),
saturated conditions,
leaching as a function of
L/S

Published June 2004
Mandated
Not validated

CEN/TC 292: Compliance leaching tests

EN 12457-1,2,3, and 4:
Compliance leaching test for
granular waste materials and
sludges

Part 1: L/S = 2 l/kg, < 4 mm
Part 2: L/S = 10 l/kg, < 4 mm
Part 3: L/S = 2 and 8 l/kg, < 4 mm
Part 4: L/S = 10 l/kg, < 10 mm

Granular materials, largely
inorganic components, batch
test, equilibrium-like conditions

Published in 2002
Mandated
Validated
Advantages/uses of percolation tests

Identification of solubility control versus wash out
Indication of pore water concentrations relevant to field leachate from low L/S data
Local equilibrium established quite rapidly
Basis for geochemical speciation modelling
Allows comparison with lysimeter and field data provided L/S value can be obtained from such measurements
Projection towards long term behaviour possible
- Solubility controlled release
- Wash-out of non-interacting species

Applicable to many materials. Limited or not applicable to e.g. clayey soils and sediments due to low permeability

CEN/TC 292: Characterisation tests

Influence of pH on leaching
prEN 14977: With continuous pH control
prCEN/TS 14429: With initial acid/base addition

Performed on finely ground material under equilibrium conditions at L/S = 10 l/kg

Both to be published in 2005
Mandated
Not validated

Advantages/uses of pH dependence tests

Identification of sensitivity of leaching to small pH changes
Provides information on pH conditions imposed by external influences
Basis for comparison of international leaching tests
Basis for geochemical speciation modelling
Provides acid neutralisation capacity information
Mutual comparison of widely different materials to assess similarities in leaching behaviour
Recognition of factors controlling release
Applicable to almost any material
CEN/TC 292: Basic characterisation tests

WI 292040: Dynamic leaching test for monolithic materials (DMLT)

Tank leaching (batch) test similar to NEN 7345 (flow through system also suggested) – useful for diffusion controlled release from monolithic materials, results expressed in mg/m² (cumulative) vs. time

Method still being discussed, study carried out by WG6 in 2004 (complicated by need to describe equilibrium controlled scenarios)

Mandated

CEN/TC 292: Compliance leaching tests

WI 292010: Compliance leaching test for monolithic material

Method under discussion in WG2 (and WG6), a need to address both diffusion controlled components and equilibrium situations have been identified

Definition of “monolithic” often insufficient

Mandated

CEN/TC 292: Leaching of organics

A proposal for a new work item dealing with the leaching of organics has been circulated by WG 6 – the outcome is not yet known

There are a number of similarities between leaching mechanisms of organics and inorganics (percolation/diffusion) but the chemistry is different and leaching and test requirements for specific organic compounds depends strongly on solubility, DOC and pH (which also affects DOC). Biodegradation must be taken into account.

In general, it is technically more difficult to get meaningful results from leaching of organics, particularly organics with low solubility.
4 procedures are under preparation based on methods developed for waste by CEN/TC 292 (but with some adjustment to the soil media – e.g. 0.001 M CaCl₂ as leachant instead of DMW).

Each standard covers both leaching of inorganics and organics and the use of the eluate for ecotoxicological testing. There are several good reasons to separate the leaching of inorganics (generally easy) and organics (often very difficult). However, there is disagreement within WG6 on this issue. At the very least the current draft standards must have much more precise descriptions on the procedures to be used for the leaching of organics.

CD: Committee draft    DIS: Draft international standard

ISO/DIS 21268-1: Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials – Part 1: Batch test at L/S = 2 l/kg (based on EN 12457-1)
ISO/DIS 21268-2: Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials – Part 2: Batch test at L/S = 10 l/kg (based on EN 12457-2)
ISO/DIS 21268-3: Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials – Part 3: Up-flow percolation test (based on CEN/TS 14405)
ISO/CD 19492: Soil quality: pH dependent leaching test for soil and soil-like materials (based on prCEN/TS 14429)

NWI (new work item): Soil quality: Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials – Guidance standard (under preparation)
Conclusions and points to be made

There is plenty of room for horizontal use of the leaching standards and methods developed by CEN/TC 292 for waste.

In some cases, minor changes are needed to account for specific properties of a new medium.

It is generally technically more demanding to perform leaching tests on organics than on inorganics.

Standardisation is often a slow process....

Funding is needed for validation.
Leaching data – interpretation, modelling and scenarios

Presented by
Hans van der Sloot

ECN
Westerduinweg 3, NL - 1755 ZG Petten, The Netherlands
mailto: vandersloot@ecn.nl

Abstract
Leaching is the process by which constituents in any solid material are released to the environment through contact with water. Understanding the rate and extent to which constituents of interest may be released is central to defining
(i) potential environmental impacts through water-borne mechanisms including soil, groundwater and surface water contamination, (ii) human health and ecological risks from beneficial use and disposal of commercial materials and wastes, (iii) effectiveness of certain treatment processes for wastes and products, (iv) designs and acceptance criteria for waste management facilities, and (v) degradation of structural performance of certain materials in the environment, especially cement-based materials. The specific rates and extents of constituent release are a function of (i) the chemical and physical properties of the material under consideration, (ii) the chemistry of the constituent(s) of interest, (iii) characteristics of the local environment in which the material is placed, including chemical properties (e.g., pH, oxidation-reduction potential, presence of reacting constituents such as carbon dioxide, oxygen) and the nature of water interaction (e.g., frequency, amount, interfacial contact area).

Fundamental understanding of leaching processes is achieved through study and research on material testing, geochemistry, constituent mass transfer, and development and verification of mathematical models to estimate long-term behavior and characterize risks under varied environmental conditions. Extensive research and evolution in understanding fundamental aspects of leaching processes and impact evaluation has been carried out over the past two decades. This research provides a foundation for practical applications in leaching characterization and impact assessment.

Recognizing the risks and environmental damage caused by uncontrolled materials use and waste disposal, national and regional regulatory organizations have developed widely variable, and often disparate, test methods and regulatory control frameworks to characterize leaching and make decisions about acceptable and unacceptable use of materials, waste management practices, and contaminated site restoration needs. These regulations, which began evolving in the early 1980’s, were based on the best understanding at the time, but are largely inadequate in the context of current understanding and needs. Many circumstances that have resulted in misapplication of procedures, erroneous decisions based on inadequate science, and resulting misuse of economic resources, damage to the environment or human health impacts have been documented based on these shortcomings.
Efforts have been undertaken in the European Union and the United States to develop a more robust and scientifically sound, while practical, framework for characterization of materials subject to environmental leaching and decision-making based on assessment of potential impacts. Consensus is evolving on an overarching framework and methodological details for implementation [1,2]. The framework is a tiered approach, allowing the user to select the level of testing and evaluation required based on the degree of conservativeness required, prior information available, and balancing costs of testing against benefits from more detailed information (e.g., reduced management costs or alternative management options). Use of this approach is beginning in both the European Union and the United States. Demand for such a system is also great in other countries.

A central challenge to the project will be to convert the depth and breadth of knowledge currently available to decisions on selection of characterization methods, assessment models and decisions on acceptability, and implementation of these choices based on specific needs of a diverse set of users. Thus, we believe there is a important need for an expert system which guides choices based on user needs, carries out data management and assessment based on testing results, and provides a comparison with results from others with similar materials and needs. This is coupled with a need for extensive training and technology transfer, as well a consultancy and further research for problems that challenge the current state-of-the-art.

Information needs

There are a range of questions to be answered in relation to possible contamination of soil and sediments, use of alternative materials in construction, application of concrete for drinking water pipes, use of biocides in wood preservation, disposal of municipal, industrial and mining waste. These questions cover aspects such as to whether that contamination is critical to plants (agricultural perspective), or critical to organisms (ecosystem function), or through release likely to adversely affect groundwater and surface water quality. The prevention of contamination and preservation of the groundwater quality are key element of the European Water Framework Directive [3]. Several regulations such as the Sewage Sludge Directive [4], the Working Document on Biowaste [5], the Construction Products Directive and the Council Directive on Landfill of Waste [6] are linked with the European Water Framework Directive as they are aimed to control the transport and distribution of contaminants from the respective fields.

The EC communication COM (2002) 179 “Towards an EU Thematic strategy for Soil protection”[7] identifies the lack of reliable and updated information on the status of the major threats to soils as the major problem, including threats such as decline in organic matter, salinization, contamination, etc, resulting from a wide range of human activities (e.g. agriculture, construction, disposal, diffuse atmospheric inputs). Regarding soil protection issues, existing soil policies are largely based on total content of contaminants. However, leaching/extraction tests may provide a better insight in the mechanisms controlling release of contaminants to the environment. Understanding and modelling of the processes governing availability and transport of contaminants in soils and sediments is needed for long-term environmental risk assessment.
Total composition versus leaching behaviour

In many existing regulations, particularly in the field of soil and sediments, total composition is used as the basis to set criteria rather than leaching behaviour. It is argued that degradation of organic matter leads to mineralization and subsequently a different uptake by plants and organisms. Using total composition would therefore provide a better safety measure than leaching. The underlying misconception is that adsorbed metals, when released by degradation of associated organic matter are 100% available for uptake, whereas in reality these metals are largely re-distributed over other available active surfaces (aluminium-oxide, iron-oxide and particulate non-degradable organic matter).

Furthermore, it is not possible to describe leaching behaviour properly from total composition in combination with a single Kd – value (solid/solution distribution coefficient). This is because the Kd value depends strongly on the system conditions, and testing conditions generally do not match the conditions for which judgement is required. The Kd approach is therefore a poor surrogate for impact assessment.

Testing methods

The types of characterisation tests that provide essential information to assess leaching behaviour under a variety of exposure conditions are:

- the pH dependence leaching test, which provides a relationship of element mobilisation as a function of pH (PrEn 14429,[8]),
- a percolation leaching test, which mimics percolation behaviour (PrEn 14405, [9]) and
- a tank leach tests or a compacted granular leach test (CGLT, NVN7347, [10]) for materials which behave as monolithic entity in their application. This method addresses the surface related release phenomena relevant for such materials.

To properly reflect release behaviour in practise, measures may be necessary to maintain reducing conditions during the test. The liquid to solid ratio (L/S) can be related to a time scale through the infiltration rate, the height and density of the material. For monolithic materials (many construction products) and in some cases release from a soil (clay) or sediment, is not percolation driven, but rather governed by surface related release phenomena, when the material under consideration has a very low permeability (< 10⁻⁹ m/s). Therefore the three characterisation leaching tests cover more than 80% of the questions to be addressed.

Development of an integrated approach

The development of a more integrated framework started with the recognition of similarities in leaching behaviour and the need for a more unified approach in testing of leaching [11, 12, 13, 14]. The characterisation test development started in CEN TC 292 Characterisation of waste in 1994 and has led to the formulation of a percolation test (PrEN 14405), a pH dependence leaching test (PrEN 14429) and a dynamic monolith leach test in development. To address the relevant questions in proper context a methodology guideline was issued (ENV 12920, [15]). In the framework of the EU project on Harmonization of Leaching/extraction tests [16] a comparison was made between leaching/extraction methods applied or applied to a broad range of materials (soil, sludge, compost, wood, waste, stabilized waste and construction materials).
has resulted in the conclusion that a limited number of test is suitable to address leaching from a wide range of materials and secondly that the pH dependence leaching test can form the basis of comparison for a wide range of existing leaching tests. A robust and scientifically sound, while practical, framework for characterisation of soils, sludge, wastes and constructions materials subject to environmental leaching and decision-making based on assessment of potential impacts is in development [17]. The framework is a tiered approach, allowing the user to select the level of testing and evaluation required based on the degree of conservatism required, prior information available, and balancing costs of testing against benefits from more detailed information (e.g., reduced management costs or alternative management options). A database of characterisation leaching data (LeachXS) is a means to facilitate cross-field comparison of data, thus avoiding unnecessary duplication of work [18].

Parallel, significant improvements in modelling geochemical speciation and chemical reaction and transport have been achieved [17, 18] using ORCHESTRA. Particularly important in this respect are the capabilities to distinguish speciation in solid and liquid phase, as well as the partitioning between iron-oxide and aluminium-oxide surfaces, clay surfaces and sub-fractions of particulate and dissolved organic matter. The latter is important for bioavailability in relation to plant uptake and sensitivity of organisms. The experimental and modelling approach has been demonstrated to be successful for a range of contaminated soils [18] and is now being extended in other fields.

A further important aspect is the identification of parameters besides leaching that need to be covered to be able to make a full evaluation of material behaviour in a given scenario [20,21]. This is best done in an expert system (currently coupled to LeachXS), that allows tying the relevant aspects together. The output from the expert system is a source term description for release of a set of contaminants to soil and groundwater. This source term can be the input to an impact evaluation tool similar to the one developed in connection with the setting of criteria at EU level for acceptance of waste at the various classes of landfills described in the EU Landfill Directive [19]. Since the release controlling factors are largely the same the same method may be applied to scenarios describing release from a wide range of materials to surface water or to sub-soil and groundwater. Now integration of all of these aspects into an environmental impact evaluation approach which is capable of addressing a large number of different questions related to use, treatment, judging of quality of materials in different exposure scenarios is in progress.

Geochemical and transport modelling
Leaching tests can not be used directly to assess relevant concentrations under field conditions or release in field scenarios. Therefore, answering the questions posed in the introduction requires modelling to provide a better understanding of the chemical speciation aspects that control release in the long term, or to obtain a source term for release under influence of changes in materials properties with time by external factors (carbonation, oxidation, infiltration, ageing). Using the recently developed JAVA-based modelling tool ORCHESTRA [17], predictive modelling can be carried out to predict leaching of elements, and to identify the solubility controlling processes. In addition, partitioning between free ion and complexed metal species in solution can be assessed. In the modelling, element interaction with aluminium-oxide, iron-oxide, clay surfaces, particulate and dissolved organic matter can be quantified provided that the relevant information is available (e.g., clay content, organic matter content and the amount of
reactive Fe and Al surfaces obtained by selective extractions). The model approach is based on a combination of a number of recently developed, mechanistic adsorption models such as the NICA-Donnan approach for adsorption to organic matter [22], the Diffuse Layer model of Dzombak and Morel for adsorption to hydrous ferric oxides [23]. Recently, the approach has been used also with success for a range of contaminated soils in the study of Dijkstra et al [18]. In table 1 a list of management scenarios and beneficial material use scenarios are given that can be covered by the proposed methodology.

Table 1 - List of Potential Management and Beneficial Materials Use Scenarios

<table>
<thead>
<tr>
<th>Management Scenarios</th>
<th>Beneficial Materials Use Scenarios</th>
</tr>
</thead>
<tbody>
<tr>
<td>Landfill (with/without leachate control)</td>
<td>Fertilizer application</td>
</tr>
<tr>
<td>Contaminated site evaluation</td>
<td>Soil use in embankment (construction)</td>
</tr>
<tr>
<td>Mining waste disposal</td>
<td>Soil improvement</td>
</tr>
<tr>
<td>Dredge spoil disposal</td>
<td>Plant uptake</td>
</tr>
<tr>
<td>Evaluation Treatment of soil (stabilisation)</td>
<td></td>
</tr>
</tbody>
</table>

The conclusions that can be drawn from the geochemical modelling on impact of contaminant release on plants and organisms is far reaching, as all relevant species (such as free element concentrations) can be quantified. These latest development in modelling surpass the claimed speciation possibilities with sequential extraction schemes [24, 25] in many respects. For evaluation of release in a given scenario, modelling the dynamic release from a material under specific exposure conditions will be relevant. In this case, dedicated models focused on a combination of chemical reaction and transport are relevant. Both types of modelling are important to reach the understanding of processes controlling release in a given scenario.

References


10. **Compacted granular leach test (CGLT) NVN 7347. NEN. 2002.**


MAIN CONCERNS IN RELATION TO LEACHING TEST USE AND DEVELOPMENT

- Too many leaching tests addressing the same question
- Too many ways of data representation
- Too limited relation of test conditions with the actual problem (for example, TCLP is used far outside its scope of development)
- Too limited use and relevance of the vast amount of leaching test data generated annually in the industry and research (missing parameters)
- Key information relevant to the outcome and possible interpretation of a leaching test often not measured/reported (pH, EC, Eh, DOC)

STRONG NEED FOR HARMONISATION OF LEACHING TEST METHODS AND DATA EVALUATION!

ROLE OF CHARACTERISATION LEACHING TESTS IN ENVIRONMENTAL JUDGEMENT

Judgement of the application of materials

Limit values

Relation lab-practice (Scenarios)

Modelling

Regulation

Assessibility of data: data base/expert system

Characterisation leaching tests (identification of mechanisms and processes)

Development of criteria for regulation

Quality control of products

Efficient measurements

Precision measurement data

Product improvement

Product modification

Measurement for verification
Leaching data – interpretation, modelling and scenarios

**CURRENT APPLICATION OF LEACHING TESTS**

- Waste classification
  - "Hazardous" versus
  - "Non hazardous"
- Treatment process effectiveness
  - Best Demonstrated Available Technology (BDAT)
  - Process development
- Waste management options
  - Alternative disposal scenarios
- Site assessment and remediation endpoints
  - Contaminated soils
  - Brownfields sites
- Beneficial Use Scenarios
- Soil quality issues
- Source term evaluations
  - Release flux
  - Risk assessment
  - Long-term monitoring requirements

**KEY QUESTIONS**

Is the distinction between percolation and surface area related mass transfer sufficient to distinguish release from most materials that need to be judged?

Are the chemical factors controlling leaching from soil, incinerator bottom ash or concrete fundamentally different?

Which aspects are crucial for the relationship or lack thereof between lab and field?

**DIFFERENT LEACHING ISSUES**

- Total versus leaching
- Release mechanisms
- Chemical speciation issues
- Examples of systematic leaching behaviour
- Lab field relationships
- Scenario approaches
- Criteria development
- Compliance test selection
- .............
### Comparison of a Judgement Based on Composition Versus Leaching

**MSWI Bottom ash (repeatability, n=15)**

<table>
<thead>
<tr>
<th>Element</th>
<th>Total content (mg/kg)</th>
<th>Leaching (mg/kg, L/S=10)</th>
<th>Ratio Total/Leaching</th>
<th>Absolute SD's</th>
</tr>
</thead>
<tbody>
<tr>
<td>As</td>
<td>13.9</td>
<td>1.7</td>
<td>12.4</td>
<td>0.0086</td>
</tr>
<tr>
<td>Ba</td>
<td>713</td>
<td>287</td>
<td>29.0</td>
<td>0.11</td>
</tr>
<tr>
<td>Cd</td>
<td>4.4</td>
<td>0.87</td>
<td>19.9</td>
<td>0.0007</td>
</tr>
<tr>
<td>Cr</td>
<td>193</td>
<td>31</td>
<td>15.9</td>
<td>0.018</td>
</tr>
<tr>
<td>Cu</td>
<td>3652</td>
<td>2815</td>
<td>77.1</td>
<td>0.55</td>
</tr>
<tr>
<td>Mo</td>
<td>4.4</td>
<td>1.9</td>
<td>42.9</td>
<td>0.051</td>
</tr>
<tr>
<td>Ni</td>
<td>158</td>
<td>63</td>
<td>29.9</td>
<td>0.0030</td>
</tr>
<tr>
<td>Pb</td>
<td>1218</td>
<td>524</td>
<td>43.0</td>
<td>0.010</td>
</tr>
<tr>
<td>SO₄ as S</td>
<td>2885</td>
<td>240</td>
<td>8.3</td>
<td>104</td>
</tr>
<tr>
<td>Sb</td>
<td>28</td>
<td>4.3</td>
<td>15.0</td>
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<tr>
<td>Sr</td>
<td>199</td>
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<td>19.0</td>
<td>0.95</td>
</tr>
<tr>
<td>V</td>
<td>24</td>
<td>5.4</td>
<td>22.2</td>
<td>0.014</td>
</tr>
<tr>
<td>Zn</td>
<td>2275</td>
<td>1045</td>
<td>45.9</td>
<td>0.017</td>
</tr>
</tbody>
</table>

**pH**

11.0 0.059

Deviation expressed in percent can be very misleading. Absolute deviations crucial!

### Misconceptions in Judgement of Total Content Versus Leaching

The uncertainty (in mg/kg) about a possible environmental effect hidden in the analysis of content is a multitude of the uncertainty obtained by a leaching test.

Carrying out a leaching test is not more complex than carrying out a content analysis. Both require a form of extraction.

From a sampling point of view leaching may be preferable as larger quantities of material can be used in the test (less stringent sample pretreatment).
Leaching data – interpretation, modelling and scenarios

FUNDAMENTAL APPROACH

- **Measure intrinsic characteristics**
  - Solubility and Release as function of pH (redox, DOC)
  - Solubility and Release as function of LS or time
  - Mass transfer rate (monolith and compacted granular)

- **Evaluate release in context of field scenario**
  - External influencing factors such as carbonation, oxidation
  - Hydrology
  - Mineralogical changes

Mimicking or simulating complex field scenarios will fail or have very limited applicability of the data

BASIC CHARACTERISATION TESTS

**GRANULAR MATERIALS**

- TANK LEACH TEST (MONOLITH)
- COMPACTED GRANULAR LEACH TEST.
- pH DEPENDENCE TEST : BATCH MODE AND COMPUTER CONTROLLED

**MONOLITHIC MATERIALS**

- pH DEPENDENCE TEST : BATCH MODE AND COMPUTER CONTROLLED
- PERCOLATION LEACHING TEST (prEN 14405)

**CEN/TC 292**

ENV 12920

Chemical speciation aspects

Time dependent aspects of release

RELEASE PROCESSES FROM MATERIALS

- Graphs showing release processes from materials

- **F** for solubility control
- **Cl** for wash out
- **C1** for concentration vs. L/S (kg)
Example where lab data will lead to an over-prediction of release. There are also situations (oxy-anions) where a significant under-prediction occurs based on a single step lab test.

LAB - FIELD VERIFICATION OF A SUSTAINABLE LANDFILL

Pilot Nauerna (12,000 m³), NL

Lysimeters (1.5 m³), NL

PERCOLATION TEST EQUIPMENT (0.0005 m³)

EU LFD

Non-

hazardous

COMPARISON OF TEST RESULTS AT DIFFERENT SCALES OF TESTING – LAB, LYSIMETER AND FIELD

Lead leaching behaviour of mixed waste very systematic and consistent with known Pb behaviour. Testing at different scales consistent and indicative of solubility control.
Organic contaminants can be evaluated in the same manner as inorganic contaminants. Increased organic matter level leads to increased PAH release. Batch test leads to higher release than a percolation test. PAH behaviour very consistent at different scales of testing (highly correlated with DOC release)!
**FULL SPECIATION OF MSWI BOTTOM ASH**

- Black line: reducing conditions at L/S=10
- Dotted red line: prediction at L/S=0.5

**Concentration profiles modelled during leaching of concrete in water.**

- Interface
- Pore water diffusion profile
- Pore sealing by mineral formation at the interface
- Pb precipitation at the interface
- Depth (cm)

**SCENARIO APPROACH IN JUDGING IMPACT**

**APPLICABLE TO:**
- CONSTRUCT, MATERIALS, SOIL, SLUDGE, SEDIMENT, WASTE, LANDFILL, PRESERVED WOOD, etc

**Expert system/database**
- Problem definition and test selection
- Lab, lysimeter, field data collection, data management, data formatting, storage and retrieval
- Management Scenario Description – configuration, design specifications, infiltration, climate
- Physical, chemical, biological properties
- pH, L/S & time dependence – redox, DOC, EC, ANC
- Granular, Percolation related, Monolithic, Surface area
- Source term description
- Release with time
- Impact evaluation subsoil and groundwater
- Judgement and decision making
- Treatment, Disposal, Utilization, Remediation end-points, long-term stewardship requirements

**Data integration between fields and tests, modelling and verification against field data**

**ENV 12920**
DIFFERENT IMPACT SCENARIOS........

Drinking water well
Landfill
Contaminated soil
Road base
Mining
Coastal protection
Agriculture
Plant
Industrially contaminated soil

LINKING LABORATORY TESTING THROUGH LAB-FIELD RELATIONSHIPS WITH REGULATORY ASPECTS

BASIS: Scenario approach for specific applications using a limited number of impact scenarios

The step-wise procedure is summarised below:
1. Choice of primary target(s), critical parameters and quality criteria
2. Description of the material application scenario
3. Description of the environment scenario
4. Description of the source term of the potential contamination
5. Description and modelling of the migration of the contaminants from the application to the POC(s)
6. Performance of “forward” modelling to determine attenuation factors
7. Application of the results to criteria setting (“backwards” calculation)
8. Transformation of the source term criteria to limit values at different L/S values

LEVELS OF TESTING

- Characterization forms the basis
  - Detailed evaluation to define material or material type characteristics and performance (more similarities than often anticipated)
  - Understanding of the key controlling factors for a material type or class

- Compliance or simplified testing
  - Simplified testing in close relationship with previous characterisation to ensure that material is within bounds of previously characterized material or class (short duration and limited parameters)

- Verification or internal QC
  - Rapid quality control tests based on key characteristics derived from characterisation
  - Completed within minutes to a few hours
### DIFFERENT OVERLAYS OF ACTIVITIES IN RELATION TO LEACHING

Hierarchy in testing: Characterisation – compliance  
Laboratory – Lysimeter – Field relationships  
Parameters: Inorganic -, organic constituents and radionuclides  
Sampling – pretreatment – testing – analysis - evaluation  
Modelling - pH stat, time dependent tests and field data  
Different scenarios for the same material  
Different exposure environments – oxidised, carbonated, reducing  
Simple modelling approach versus detailed chemical reaction transport models

### CONCLUSIONS

Leaching from materials is far more systematic than commonly believed based on single step leaching tests.  
A limited number of leaching tests can provide the crucial answers needed to assess long-term impact for a wide range of materials.  
Systematic comparison of lab, lysimeter and field data has shown great potential to make predictions of long term behaviour.  
The approach presented for inorganic contaminants is equally relevant for organic contaminants and radionuclides.

### CONCLUSIONS

The leaching framework, using characterization as a comparative basis, provides for multiple questions to be answered instead of new testing for every individual situation.  
The proposed hierarchy in testing provides the necessary detail required by regulators and developers of treatment techniques. It also provides for cost effective testing.  
Widely different utilization and disposal scenarios can be addressed with a similar impact evaluation methodology using basic characterisation test results to derive a source term.  
Making leaching data currently inaccessible in pdf's and (worse) in paper files available for comparison provides new insights from earlier data.
CONCLUSIONS

Chemical speciation using mineral solubility, sorption and organic matter interactions provides identification of release controlling minerals and similarities amongst widely different materials.

Understanding chemical speciation provides insights for system improvement and enhances long-term release prediction for many constituents of concern.

FUTURE

Development of an international database/expert system incorporating leaching and related data (with consideration to confidentiality issues).

Validation of the widely applicable characterisation leaching tests for a wide scope of materials.

Develop simple compliance methods targeted at key parameters in reference to the characterisation data.

Organization of training for the use of tests, interpretation of data, modelling and guidance in judgement.

INFORMATION ON LEACHING AVAILABLE AT:

LEACHING BACKGROUND
www.leaching.net (Wascon 2003 workshop)

CONSTRUCTION PRODUCTS DIRECTIVE

LEACHING IN PROJECT HORIZONTAL
www.ecn.nl/library/horizontal

GRACOS
www.uni-tuebingen.de/gracos
Abstract
A framework for the evaluation of inorganic constituent leaching from wastes and secondary materials is presented. The framework is based on the measurement of intrinsic leaching properties of the material in conjunction with mathematical modeling to estimate release under field management scenarios. Site-specific and default scenarios are considered, which may be selected based on the evaluation context. A tiered approach is provided to allow the end user to balance between the specificity of the release estimate, the amount of testing knowledge required, a priori knowledge, and resources required to complete an evaluation. Detailed test methodologies are provided for a suite of laboratory leaching tests.
A View From Across The Ocean – Leaching Issues in the United States

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“Problems around Soil and Waste I – Horizontal aspects of leaching”
European Commission DG JRC, Ispra Site
14-15 February 2005

e-mail: David.Kosson@Vanderbilt.edu

Key Messages
- TCLP defined under RCRA for use in waste classification but currently widely used for leaching assessment in applications where not mandated.
  - Measure intrinsic leaching characteristics, use geochemical speciation and mass transfer models in conjunction with management scenarios to estimate constituent release.
  - Use results to assess impacts, develop acceptance criteria and monitoring strategies.
- Several Integrated Leaching Framework uses in-progress and regulatory applications under consideration.
- Most important research and development needs are
  - Field validation of laboratory-based release estimates
  - Guidance documents for broad-based implementation
  - Inter-laboratory methods validation
  - Integrated database and decision tools

RCRA Background
- EPA regulates waste management under the Resource Conservation and Recovery Act (RCRA).
- Groundwater contamination is a key waste management concern.
- Leach testing has been used in regulatory programs to help determine:
  - What waste is hazardous: listings, delistings, Toxicity Characteristic (TC) regulation.
  - What treatment is adequate: Land Disposal Restriction (LDR) treatment requirements.
- TCLP is the most used leaching test.

*courtesy of Mr. Gregory Helms, USEPA, Office of Solid Waste, Washington, DC.
RCRA Background

- TCLP was designed as a screening test to consider conditions that may be present in a MSW landfill:
  - Acetic acid buffered to pH 5 (initial); 20:1 liquid/solid ratio; particle size reduction to 9.5 mm; equilibrium.
  - Co-disposal of industrial solid waste with MSW is designated as plausible “worst case” management of unregulated waste.

*courtesy of Mr. Gregory Helms, USEPA, Office of Solid Waste, Washington, DC

RCRA Background

- Because of its regulatory program use, TCLP is also used when not required:
  - Site remediation where LDRs are not triggered (usually in-place or on-site treatment and disposal)
  - Corrective Action Management Units
  - Non-hazardous waste reuse (state Beneficial Use programs)
  - Industrial non-hazardous waste landfills

*courtesy of Mr. Gregory Helms, USEPA, Office of Solid Waste, Washington, DC

Technical Issues with TCLP

- TCLP is a screening test that evaluates leaching potential under a single set of environmental conditions:
  - Initially acidic conditions; final conditions were not considered critical, and usually are not known
  - Generally oxidizing environment
  - For most metals, leaching is pH dependent; many landfills achieve reducing conditions.
  - For many applications, landfill co-disposal with MSW is not relevant.

*courtesy of Mr. Gregory Helms, USEPA, Office of Solid Waste, Washington, DC
Science Advisory Board Concerns

  - The SAB expressed concern about over-broad use of the TCLP test.
  - The SAB urged the Agency to undertake new leaching research on both occasions.
  - SAB urged development of tests that consider actual disposal conditions affecting leaching.
  - SAB urged field validation of new tests.

Agency Response to Concerns

- Seeking approaches that can be developed into reliable tests for routine use:
  - Most non-TCLP testing has been research or ad hoc modifications of TCLP (Hg waste treatability studies, regional delisting programs)
  - Need defined protocols that are validated for particular uses; validation includes interlab and field evaluations
  - Most existing alternatives have not been validated (particularly field validation)

Agency Response to Concerns

- In considering new approaches to leach testing, the Agency is seeking:
  - Broad applicability (regarding both waste types and management conditions)
  - Consideration of factors affecting leaching
  - Validation in both the lab and field
  - Practical applicability of tests
EPA Future Direction: Waste Programs

- No plans to revise the TCLP or its use in supporting the TC or LDR regulations.
- Regional delisting guidance and Framework demonstration.
- Hope to develop new approaches for use in:
  - Evaluating leaching potential in reuse
  - Site remediation especially for effectiveness of in situ/on-site solidification/stabilization treatment of soil
- Development of Decision Support Tool (with ORD)

*courtesy of Mr. Gregory Helms, USEPA, Office of Solid Waste, Washington, DC

Integrated Leaching Framework Uses In-progress and Regulatory Applications Under Consideration

- Evaluation of impacts from new regulations on emissions and residues from coal fired power plants
- Mine filling with coal combustion residues
- Delisting of hazardous waste
- Beneficial use of secondary materials
- Management of nuclear waste
- Site remediation, especially contaminated soils and solidification/stabilization treatment
- Treatment process evaluations (Determination of Equivalent Treatment)

Overarching Framework
(Kosson, van der Sloot et al., 2002, Environ. Engr. Sci., 19, 159-203)

- Broad-based impact and release evaluation for environmental management (regulators, producers, re-use contractors, treatment vendors)
- Framework flexibility
  - Choice of testing levels or tiers
  - Considers "conservatism" of estimate
  - Definition of characteristic leaching parameters and scenario conditions
  - Selection of various deterministic source term models
    - Degree of complexity commensurate with release phenomena
  - Probabilistic impact estimation
    - Variability of environmental conditions
    - Uncertainty in parameter measurement
Conceptual Approach to Leaching Evaluation

- Measure intrinsic leaching characteristics of material
- Evaluate release in the context of field scenario
  - External influencing factors such as carbonation, oxidation
  - Hydrology
  - Mineralogical changes
- Use geochemical speciation and mass transfer models to estimate release for alternative scenarios
  - Model complexity to match information needs
  - Many scenarios can be evaluated from single data set

*Do NOT mimic field scenarios with specific tests! Too many tests with limited data comparability!*

Measuring Intrinsic Leaching Characteristics

- Aqueous-solid partitioning as a function of pH and Liquid to Solid ratio
  - Batch extractions
  - Constituent fraction readily leached
  - Controlling mechanism for release (mineral dissolution and solubility, solid phase adsorption, aqueous phase complexation)
- Release kinetics
  - Percolation (column tests)
  - Diffusion (monolithic or compacted granular tank leaching tests)
  - Use results in conjunction with understanding of pore water chemistry to determine mass transfer rate constants (e.g., effective diffusivities)

Main Types of Leaching Tests

- Equilibrium-based leaching tests
  - Carried out on size reduced material
  - Aim to measure contaminant release related to specific chemical conditions (pH, LS ratio)
- Mass transfer-based leaching tests
  - Carried out either on monolithic material or compacted granular material
  - Aim to determine contaminant release rates by accounting for both chemical and physical properties of the material
- Percolation (column) leaching tests
  - May be either equilibrium or mass transfer rate
Leaching issues in the United States

**Equilibrium Characterization**

**Solubility and Release as a Function of pH (SR002.1)**

- 11 parallel solubility extractions
- DI with HNO3 or KOH addition
- Size reduced material
- Contact time based on size
- LS ratio: 10 mL/g dry
- Endpoint pH
  - Distributed 3 ≤ pH ≤ 12

<table>
<thead>
<tr>
<th>Particle size</th>
<th>Contact time</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 0.3 mm</td>
<td>18 hr</td>
</tr>
<tr>
<td>&lt; 2 mm</td>
<td>48 hr</td>
</tr>
<tr>
<td>&lt; 5 mm</td>
<td>168 hr</td>
</tr>
</tbody>
</table>

**Equilibrium Characterization**

**Solubility and Release as a Function of LS (SR003.1)**

- 5 parallel extractions
- DI water
- Size reduced material
- Contact time based on particle size
- LS ratios: 0.5, 1, 2, 5, and 10 mL/g dry

<table>
<thead>
<tr>
<th>Particle size</th>
<th>Contact time</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 0.3 mm</td>
<td>18 hr</td>
</tr>
<tr>
<td>&lt; 2 mm</td>
<td>48 hr</td>
</tr>
<tr>
<td>&lt; 5 mm</td>
<td>168 hr</td>
</tr>
</tbody>
</table>

**Mass Transfer Rate Characterization**

**Tank Leaching Tests (MT00x.1)**

- Two protocols
  - Monolithic (MT001.1)
  - Compact granular (MT002.1)
- DI water (own pH)
- Liquid to surface area ratio: 10 cm²/cm²
- Refresh intervals: 2, 3, 16 hr, 1, 2, 4, days, ...

Cumulative release as a function of time - Results in mg/m²
Release Modes

- Percolation through granular materials
  - Granular or highly permeable material
  - Local equilibrium controls release
  - Preferential flow may be important

- Flow around low permeability (monolithic) materials
  - Coupled diffusion and pore-water chemistry controls release
  - Boundary conditions are important

Long-term Assessment Models

- Simple release models
  - Percolation/equilibrium model
  - Diffusion model

- More sophisticated release models to account for
  - Chemistry between solid-liquid phases (empirical or geochemical speciation)
  - Effect of intermittent wetting
  - Effect of external stresses (e.g., carbonation)

- Use of probabilistic approach to allow for
  - Consideration of a range of management sites and conditions
  - Consideration of a range of expected climate conditions and waste characteristics
  - Bounded levels of confidence and distribution frequencies for release estimates

100-year Release Estimates

- Comparison of release estimates using the simple diffusion and IMT model precipitation data to saturated release models (monolithic materials)
A Comparison of Treatment Processes for Mercury Contaminated Soils for Determination of Equivalent Treatment

- 2 Hg-contaminated soils (ca. 4500 mg Hg/kg) as mixed waste
- Test cases
  - Untreated soils
  - Thermal desorption (1 vendor)
  - Solidified/stabilized soil (2 vendors)
  - Sulfur polymer cement (1 vendor)
- Test Conditions
  - Total content, Availability
  - Solubility & Release as function of pH & LS
  - Mass transfer release rate (monolithic or compacted granular)

Solubility & Release from Untreated and Treated Hg-contaminated Soils

Vendor 3 treatment resulted in a significant increase in Hg solubility for pH between 4 and 8.

Mercury Release Rates (Untreated & Treated Hg-contaminated Soil)

- $D_{obs}$ untreated Am soil = $9.8 \times 10^{-16}$ m$^2$/s
- $D_{obs}$ SPC treated Am soil = $8.9 \times 10^{-18}$ m$^2$/s
Research is in support of EPA’s regulatory program to control mercury from coal-fired power plants. Interest is in changes that may occur to coal combustion residues as a result of implementing mercury or multi-pollutant control technologies. Changes to CCRs that may increase environmental impacts from their management are to be addressed in the RCRA Subtitle D regulations under development for coal combustion waste.

USEPA Evaluation of Coal Combustion Residues from Facilities with Enhanced Mercury Control

- USEPA-ORD Research Program (S. Thorneloe)
  - Programmatic input from EPA-OSW (G. Helms)
  - Consultation with EPA Science Advisory Board (Environmental Engineering Committee)
  - Leaching tests carried out by ARCADIS (R. Keeney)
  - Technical support by Vanderbilt Univ. (F. Sanchez and D. Kosson)

- Extensive QAQC program development
  - Methods validation with mass balance on reference CCR
  - QAQC within leaching methods, chemical analysis, data evaluation

- Leaching characterization
  - Release as function of pH (SR002.1)
  - Release as function of LS (SR003.1)
  - Mass transfer release (when considered necessary)

- Comparison with field data
  - Leachate concentrations reported in USEPA database
  - Field sampling from CCR management facilities

- Release Scenario Assessment
  - Land disposal
  - Probabilistic release estimates based on range of conditions (pH, LS) reported in USEPA database (improved estimates to be based on EPRI data)
  - Release estimates for default scenarios at 3 pHs (acid, alkali, own)
Leaching issues in the United States

CCR Evaluation Results (USEPA Program)

- Completed
  - CCRs from 5 facilities
- In process
  - CCRs from 5 additional facilities (representing 8 different units)
- Just received
  - Fly ash from facility with brominated sorbent for Hg control [w/ cold-side electric static precipitator (CS-ESP) and low Sulfur coal] — evaluation includes organic Brominated compounds in fly ash and leachate

Probabilistic Assessment of Release for Land Disposal (based on USEPA database)

Example
Arsenic Leaching as a Function of pH

Baseline
As total content*: 80.5 ± 1.9 µg/g

w/ activated carbon injection
As total content*: 27.8 ± 2.1 µg/g
**Example**

Probability Distribution of 100 yr Release Estimates for Land Disposal

<table>
<thead>
<tr>
<th>Percentile</th>
<th>MT min</th>
<th>MT 25%</th>
<th>MT 50%</th>
<th>MT 75%</th>
<th>MT 95%</th>
<th>MT max</th>
</tr>
</thead>
<tbody>
<tr>
<td>B (µg/kg)</td>
<td>0.1</td>
<td>0.12</td>
<td>0.17</td>
<td>0.22</td>
<td>0.27</td>
<td>0.50</td>
</tr>
<tr>
<td>P (µg/kg)</td>
<td>0.001</td>
<td>0.0301</td>
<td>0.10</td>
<td>0.20</td>
<td>0.77</td>
<td>0.10</td>
</tr>
</tbody>
</table>

**Leachability and Degradation of Solidified Secondary Waste from High-Temperature Treatment of Hanford Tank Waste**

**Hanford Tank Wastes**

- 144 Underground Tanks
- Concrete reinforced carbon steel
- 10^6 gallon capacity
- Single and double shelled
- Several tanks have leaked
- Tank Waste
  - Supernatant, sludge, filter cake
  - 2x10^8 m³ of radioactive waste
- DOE Remediation by 2028
  - Separate low activity waste (LAW) from high level waste (HLW)
  - HLW → Vitrification with off-site disposal
  - LAW → Vitrification with off-site disposal plus supplemental technology
  - Secondary wastes from high temperature treatments ???
Leaching issues in the United States

Secondary Waste Uncertainties
- Composition of Secondary Waste
  - $^{129}$I (assumed 100% of tank inventory may be incorrect)
  - $^{99}$Tc (assumed 10% of tank inventory)
- Formulation of Solidified Secondary Waste (SSW)
- Retention of I and Tc in SSW
  - Constituent speciation (AgI, TcO$_4^-$)
  - Retention of Iodine not strong in cement
- Long-Term Performance of SSW
  - Leaching rates
  - Equilibrium chemistry
  - Long-term alteration in leaching behavior

Current assessment approaches do not consider the interaction between leaching and durability with aging and degradation mechanisms

European Commission DG JRC
14-15 February 2005

Durability and Leaching
- Durability Metrics
  - Compressive strength
  - Porosity
  - Permeability
  - Acid resistance
- Monolithic Matrix
  - Flow-around
  - Low interfacial area
  - Diffusive release
- Stressed Matrix
  - Flow-through
  - Higher interfacial area
  - Diffusive/convective release
- Spalled Matrix
  - Very high permeability
  - Very high interfacial area
  - Equilibrium-based release

European Commission DG JRC
14-15 February 2005
Project Objectives

- Characterize leaching from SSW
  - Equilibrium – solid/liquid chemistry with pH, LS ratio
  - Mass Transport – physical parameters, leaching rates
  - Intermittent Wetting – approach toward release conditions
- Evaluate effects degradation and aging on durability and leaching performance
  - Carbonated SSW
  - Aged SSW
  - Cracked SSW
- Assess structural durability of SSW as function of
  - Leaching
  - Progressive cracking
- Develop constitutive relationships for durability and leaching as functions of external and internal stresses

Understanding PAH Leaching and Bioavailability from Estuary Sediments

Equilibrium Partitioning Piles Creek Sediment

- Measured sediment-water partitioning for each PAH in each sediment fraction.
- Plotted Koc of each compound in each fraction vs. corresponding Koc in whole sediment.
- Results show fundamentally different carbon-normalized partitioning behavior in low-density sediment.

Desorption Assay

- Shake sediment slurry with Tenax beads
- Transfer slurry, add new Tenax beads & resume shaking
- Extract PAHs from used beads and quantify via HPLC-PDA/Fluorescence

Desorption Assay

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- Transfer slurry, add new Tenax beads & resume shaking
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Desorption Controls PAH Bioavailability

<table>
<thead>
<tr>
<th></th>
<th>Newtown Creek</th>
<th>Piles Creek</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>% biodegraded</td>
<td>% biodegraded</td>
</tr>
<tr>
<td>Phenanthrene</td>
<td>19 ± 4</td>
<td>17</td>
</tr>
<tr>
<td>Anthracene</td>
<td>31 ± 2</td>
<td>42</td>
</tr>
<tr>
<td>Pyrene</td>
<td>72 ± 0</td>
<td>80</td>
</tr>
</tbody>
</table>

The percent of total sediment PAHs that can be biodegraded equals the fraction in the fast-diffusion regime, f.

PAH Desorption Kinetics

PAH bioavailability by sediment fraction: High-ρ >> whole > Low-ρ


The fraction of PAHs that desorb in various time intervals varies dramatically by sediment and compound.


Key Messages

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Acknowledgements

- USEPA
  - Office of Solid Waste
  - National Risk Management Research Laboratory (RTF)
  - Northeast Hazardous Research Center
- Consortium for Risk Evaluation with Stakeholder Participation (CRESP) with support from DOE-EM
- Recycled Materials Resource Center (UNH/FHWA)
Environmental aspects of construction materials in relation to the Construction Products Directive (ER3) – Impact on soil and groundwater

Presented by
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mailto: vandersloot@ecn.nl

Abstract
In the beginning of 2005 the European Commission, DG Enterprise, will mandate the European Standardization Organisation CEN to prepare test methods with which construction products in the EU can be tested with respect to the potential release of dangerous substances to soil and groundwater and to indoor air. This action is intended to satisfy the needs resulting from Essential requirement number 3 as specified in the Construction Products Directive (89/106/EEC). These test methods will be coupled to regulatory limits that a broad range of construction products has to fulfil to be accepted on the EU market (Construction Products Directive, CPD). An expert working group under the European Commission, DG Enterprise, Construction Unit has supported the drafting of the mandate. CEN is also involved already, as the Environmental Project of the Construction Sector Network (CSNPE) has taken the initiative to set up a horizontal Technical Committee.

It is generally recognized that one of the most important environmental risks associated with the use of materials for construction purposes is the potential release and subsequent migration of contaminants from the material into the environment. This may occur during initial material use, use after recycling, and after final disposal. The contaminants, which are released upon contact with water and transported by water, may pose a risk to the quality of e.g., groundwater, surface water and soil.

However, existing tests based on release appear to be very different from each other in many cases, while they largely address the same question: what is the release of a constituent from a product in general or in a specific situation? The wide range of tests often results from an approach of attempting to simulate individual field conditions for a specific material, rather than focusing on quantifying common underlying mechanisms that control release. Such test methods are typically "conditional", which means that results from the test only apply to a specified scenario (e.g. migration tests that are only suitable for drinking water pipes and which only answer some rather specific questions.) However, the consequence of "conditional" testing is that neither test results can be compared to other test results or changed conditions, nor can they be interpreted in a mechanistic way. For the above named reasons, the development of as many different test methods as there are materials and application scenarios, seems therefore very inefficient.

A preferred approach to assessing contaminant release is to use a common set of leaching tests that define and quantify the underlying mechanisms that control contaminant release
under a wide range of environmental conditions. Through this approach, a common set of testing results can be used to assess material performance under a range of use, recycling and disposal scenarios and thus facilitating life-cycle evaluations. In addition, this information facilitates materials improvement and uniform comparison of materials within and between categories of materials and under varying use and management scenarios.

Through a hierarchy in testing a well-balanced approach is laid out, which uses sufficiently detailed characterization to understand the main question and provides information for adequate evaluation. In this hierarchy sufficiently quick methods for quality control purposes are available, once the work has moved in to a stage of monitoring quality and ensuring compliance with regulation.

Such an approach could in principle lead to a "level playing field" of requirements that all types of (construction) materials have to fulfil. Firstly, because legislation based on "release" can be derived from testing information in one consistent way. Secondly, apart from efficiency (in terms of time and finance), an important advantage would be a high flexibility, because many (emerging) impact/exposition scenarios could be treated in a similar way based on the same tests. Finally, the approach leads to a rational relationship between perceived risks and the criteria set to provide protection against those risks.

Following acceptance of the CPD mandate by CEN a work plan needs to be developed in 2005 for the proposed horizontal TC.
ENVIRONMENTAL ASPECTS OF CONSTRUCTION MATERIALS IN RELATION TO THE CONSTRUCTION PRODUCTS DIRECTIVE (ER3)

Impact on soil & groundwater

H.A. van der Sloot
ECN

Based on work in ECRICEM and ECO-Serve with VDZ, NORCEM, HOLCIM

“Problems around Soil and Waste I – Horizontal aspects of leaching”
European Commission DG JRC, Ispra Site
14-15 February 2005

CPD - Dangerous substances

Essential requirements nr. 3: Hygiene, health and environment:
- the giving-off of toxic gas,
- the presence of dangerous particles or gases in the air,
- the emission of dangerous radiation (e.g. Radon)
- pollution or poisoning of the water or soil,
- faulty elimination of waste water, smoke, solid or liquid wastes,
- the presence of damp in parts of the works or on surfaces within the works.

Note: CPD covers service life only

CPD - ER3 Dangerous substances

Mandate accepted by Standing Committee Construction on October 26 2004

Beginning 2005 mandate issued to CEN

Work plan to be drafted in 8 month

Start of the horizontal standardisation work on:
  Sampling
  Indoor air
  Impact to soil and groundwater
**LIFE CYCLE ISSUES IN SOIL & GROUND AND SURFACE WATER IMPACT**

- Service life: different exposure scenarios
  - Constructions in water
  - Constructions on land
  - Drinking water pipes/basins
- Recycling stage (bound): same as service life
- Reuse stage (unbound): different exposure scenarios
  - Road base/embankment
  - Structural fill (dikes, soundbarriers)
- "End of life": Landfill scenarios
  - Inert landfill
  - Non-hazardous waste landfill

Characterisation testing provides information on the above mentioned life-cycle stages.

**ROLE OF LEACHING TESTS IN THE BUILDING CYCLE**

<table>
<thead>
<tr>
<th>Primary Raw Materials</th>
<th>Alternative raw materials</th>
<th>Granular compliance test</th>
<th>Supply of information on environmental quality</th>
<th>Characterisation (ITT) of monolith/granular-leaching behaviour</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stage 1: Site material supply</td>
<td>Stage 2: Manufacture of construction materials and elements</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stage 3: Construction Process</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Stage 4: Service Life</td>
<td></td>
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<tr>
<td>Stage 5: Demolition</td>
<td></td>
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<tr>
<td>Energy</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Environmental impact (sketch)</td>
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<tr>
<td>Release into the environment</td>
<td></td>
<td></td>
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</tbody>
</table>

**CONSTRUCTION PRODUCTS DIRECTIVE (CPD)**

**SAMPLING RELEASE SCENARIOS**

<table>
<thead>
<tr>
<th>Main aspect</th>
<th>Sample strategy</th>
<th>Pretreatment</th>
<th>Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target Sampling</td>
<td>Storage</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sub topics</td>
<td>Inorganic</td>
<td>Organic</td>
<td></td>
</tr>
</tbody>
</table>

**PRODUCT FAMILIES IN CONSTRUCTION**

- Aggregates
  - Aggregate in concrete (bound)
  - Aggregate in road base (unbound)
  - Aggregate as structural fill (unbound)
- Cements, Building limes and other hydraulic binders
- Concrete, mortar, Grout and related products
- Construction adhesives
- Curtain walling
- Doors, windows, shutters, gates and related building hardware
- External thermal insulation
- Fire stopping, fire sealing and fire protection products
- Fixed fire fighting systems
- Floorings
- Geotextiles
- Glass products
- Gypsum products
- Internal & external wall and ceiling finishes
- Internal partition kits
- Light composite wood based beams and columns
- Liquid applied waterproofing kits
- Masonry and related products
- Membranes
- Metal anchors for concrete
- Metal injection anchors for use in concrete to fix lightweight systems
- Non-load bearing permanent shuttering kits
- Pipes, tanks and ancillaries not in contact with water
- Plastic anchors for use in concrete and masonry
- Post-tensioning kits for pre-stressing of structure
- Prefabricated stair kits
- Reinforcing and pre-stressing steel for concrete
- Road construction products
- Asphaltic products
- Roof coverings, rooflights, roof windows and ancillary products

**Release to Soil, Surface & Groundwater**

- Granular materials
- Monolithic materials

**Environmental quality**

- Soil protection, surface water quality, groundwater quality
- Drinking water quality
- Landfill/similar use

**Availibility of Test methods for Construction products**

<table>
<thead>
<tr>
<th>Test methods for Construction products</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material Flow</td>
<td>Characterisation of granular leaching</td>
</tr>
<tr>
<td>Primary Raw Materials</td>
<td>Energy</td>
</tr>
<tr>
<td>Alternative Raw Materials</td>
<td>Environmental impact (sketch)</td>
</tr>
<tr>
<td>Granular Compliance Test</td>
<td>Release into the environment</td>
</tr>
<tr>
<td>Supply of Information on Environmental Quality</td>
<td>Characterisation (ITT) of Monolith/Granular Leaching Behaviour</td>
</tr>
</tbody>
</table>

**Characterisation of Granular Leaching**

- Soil Protection, Surface Water Quality, Groundwater Quality
- Drinking Water Quality
- Landfill/Similar Use

**Availability of Test methods for Construction products**
Environmental aspects of construction materials

SCHEME TO DECIDE ON WT, WFT AND FT 
(in discussion)

WT = Without Testing
WFT = Without Further Testing and
FT = Further Testing

CONCLUSIONS

- Scenario approach most suitable to meet the requirements of CPD and the regulatory needs associated with it
- For the wide range of construction products the focus should be on the common release mechanisms and similar release controlling factors rather than on the individual material properties
- Hierarchy in testing advised – start with proper characterisation of material classes and then select the relevant parameters and compliance method for QC and WFT (without further testing)
- For more than 90% of the material – scenario combinations with existing tests or tests in development can be adopted
- The proposed methods can be used to assess behaviour in service life, recycling and for end-of-life evaluation.

CONCLUSIONS

- Measurement of intrinsic leaching parameters permits estimation of constituent release for default or site-specific scenarios and comparison of treatment or management options.
- Although a very significant amount of work has been done on cement mortars, similar observations will apply to a range of other construction materials relevant to the CPD.
- After appropriate characterisation simple compliance tests can be selected for quality control (frequency still to be decided) on crucial parameters identified in the characterisation or initial type testing.
- Modelling capabilities of release are advancing rapidly as shown by the results for field exposed concrete using ORCHESTRA
- Establishment of standardised protocols and an (web-based) international database would extensively leverage resources.
Evaluation of the application of standardized methods for mining waste characterisation

Presented by
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Co-authors
J. Dijkstra, N. Erikson, L. Lindahl

Abstract
The European Commission has presented a proposal for a Directive to regulate the management of waste from the extractive industries (COM (2003) 319). Together with the revision of the Seveso II Directive on the control of major industrial accidents, and a Best Available Techniques document on the management of tailings and waste rock (IPTS, 2004), the proposed Directive will ensure sound management of wastes from the extractive industry throughout the EU. Waste from extractive industries is subject to the general provisions of the Waste Framework Directive (75/442/EEC). Furthermore, facilities for the disposal of waste from the extractive industries are considered to be covered by the Landfill Directive (1999/31/EC). However, the latter contains provisions, which are not always adapted to mining waste. It has therefore been necessary to create an appropriate legal framework that would exempt waste from the extractive industries from the provisions of the Landfill Directive and establish tailor-made rules. The proposal (COM (2003)319) focuses on the waste management in all stages of the life-cycle of a mine (exploration, planning, design, construction, operation, closure and after-care) and on issues such as accident prevention, emergency planning and minimization of day-to-day pollution. Environmental problems that are addressed in the proposal are primarily the pollution of surface water, groundwater and soil, due to e.g. spills and the creation of contaminated leachates from waste facilities. The proposed Directive covers wastes from all sectors of the extractive industry. Its provisions are expected to affect mainly wastes that are potentially dangerous to the environment. Lower requirements are foreseen for wastes with a low environmental risk. Excluded are wastes inappropriate to be managed under the proposal, such as food waste and waste from offshore industry.

In CEN TC 292 (committee on waste characterization in the European standardization organisation) methods have been developed for sampling, analysis and the leaching characteristics of wastes, as well as a framework for an approach to address specific waste utilization and management scenarios (ENV 12920). Even though these developments were strongly focused on waste in general, the methods proved to be suitable for characterisation and evaluation of more types of mineral and other materials in other scenario's. In an ad hoc group established by CEN TC 292, the suitability of existing methods developed in CEN and additional needs for standardised protocols is evaluated, which may lead to establishing a new working group in CEN TC 292. So far a need has been established to be able to the acid rock drainage (ARD) generation potential and the acid neutralisation capacity of sulphidic mining wastes.
Evaluation of the application of standardized methods for mining waste characterisation

Hans van der Sloot (ECN, NL), Joris Dijkstra (ECN, NL), Nils Eriksson (EUROMINES) and Lars-Ake Lindahl (Metallgruppen SE) with contributions from Ann-Marie Fällman (EPA, SE), Rein Eikelboom (VROM, NL) and Claus Gerhard Bannick (UBA, D).

EUROMINES, Brussels, June 7, 2004

Wastes from the extractive industries

Origin of mining wastes
- Topsoil
- Overburden and waste rock
- Tailings

Nature of mining wastes
- Sulphidic
  - Pb, Cu, Zn, Au mining
- Non-sulphidic
  - Potash (high salt load)
  - Red mud (highly alkaline)

Disposal and utilisation scenarios of wastes from the extractive industries

- Backfill
- Construction of embankment
- Tailings pond
- Tailings heap
- Beneficial uses of some waste streams
  - Sand fraction from aluminium production.
  - Fertilizer and soil improvement capabilities of red mud

……………
……………
AVAILABLE TESTS FOR MINING WASTE

No European standards specifically for mining waste
At international level no standards available either. In Canada the humidity cell test has been standardised.

Methods mainly used in the mining industry are:
- Acid Base Accounting (ABA) test (similar to ANC)
- Net acid generation test (NAG)
- Humidity cell test

Incidentally used are among others:
- Column and lysimeter tests
- Sequential chemical extraction

Suitability of existing leaching test methods for mining waste

The methodology developed in CEN TC 292 (ENV 12920) matches well with the approaches followed in the mining waste sector

The test methods developed in CEN TC 292 for characterisation (PrEn 14405 and PrEN 14429) are suitable to address a range of questions related to mining waste.

The main missing aspect is the acid producing capacity of sulphidic wastes. The method developed in the mining waste area has similarities with the ANC method. Methods to assess the kinetics of ARD formation for which there is no formalised standard

TESTING APPROACH FOR MINING WASTES

1. Samples collection and preparation
2. Physical/geotechnical characterisation
3. Geochemical characterisation
4. Chemical/mineralogical analysis
   - Sulphide leach
   - Non-sulphide leach
   - Acid Base Accounting tests
5. Evaluation of ARD potential
   - Yes
   - No
       - Short term extraction test
       - pH dependence test
       - Column test
       - Acid leachability
       - No further ARD testing
6. Kinetic testing

ECN
CONCLUSION

- Similarities between the chemical characteristics of mining waste, soil material and other waste types
- Tests as developed in CEN TC 292 cover many aspects that also apply to a large variety of mining waste
- Standards on sampling, sample pre-treatment, extraction and chemical analysis can possibly be used directly
- A major distinction can be made between reactive (sulphidic) and non-reactive (non-sulphidic) mining waste
- Reactive mining wastes require the characterisation of the ARD generation potential and possibly also the reaction kinetics in order to evaluate possible disposal scenarios
- For non-sulphidic mining wastes the available methods in TC 292 seem adequate
- To reach a high degree of acceptance it is important to bear the global aspect of the mining industry in mind
Presentations

Leaching processes of soil, sludges and compost - Recent developments in geochemical modeling

Presented by
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Abstract

Geochemical modelling is increasingly being used in the environmental assessment of waste utilisation, particularly to predict contaminant leaching and mobility beyond the conditions of laboratory measurements (i.e. translation to field situations and long-term predictions). However, the predictive power and the reliability of modelling predictions are strongly determined by the modelling approach and the estimation of the necessary environmental modelling parameters. It is particularly challenging to apply thermodynamical constants that have been extracted from model systems to the strongly heterogeneous matrices of waste materials and soils, without the need for parameter fitting.

A successful combined experimental and geochemical modelling approach has been developed to assess contaminant speciation in waste materials and contaminated soils, and its implications for contaminant fixation, leaching and mobility. The experimental approach is largely based on the application of pH-dependent leaching experiments to obtain geochemical fingerprints of the controlling processes. Additional specific chemical extractions are performed to quantify reactive organic matter constituents such as humic and fulvic acids, as well as mineral surfaces that may control contaminant adsorption to the soil matrix. The modelling approach is based entirely on generic thermodynamic constants and involves no fitting of parameters. The new modelling platform ORCHESTRA is used for the calculations.

A step-by-step approach is followed to define the appropriate geochemical model for the waste or soil system to be evaluated. In addition to mineral precipitation/dissolution and aqueous complexation reactions, the approach includes models and generic thermodynamic parameters for the sorption of contaminants to metal(hydr)oxides (Diffuse Double Layer surface complexation model), clay minerals (Donnan model) and both dissolved and particulate organic (humic) substances (Non-Ideal Competitive Adsorption, NICA, model). Given the major role of organic matter in the speciation, leaching and mobility of metals in waste materials and soils, specific attention is paid to its nature and binding properties, in direct support of the development of a generic modelling approach.

It is demonstrated that the pH-dependence leaching test captures the information from a multitude of (batch) leaching tests and that it provides the necessary geochemical fingerprints to identify the underlying leaching processes. As such, the pH-dependence leaching test is shown to form the basis of the outlined and powerful “horizontal” modelling approach that does not require any parameter fitting and is widely applicable to materials such as soils, sludges and compost. Specific examples of the modelling approach include novel speciation diagrams (including both mineral and adsorbed species) and pH-dependent leaching curves of major and minor elements in soil, sludge and compost. The speciation diagrams demonstrate the dominant role of (dissolved) organic carbon (DOC) in contaminant leaching from soils and many soil-like (waste) materials. Near-future developments include forward prediction of pH and DOC solubility, as well as inclusion of the full modelling approach in the ECN database/expert system LeachXS.
Leaching processes of soil, sludges and compost

Recent developments in geochemical modelling

Rob N.J. Comans, Joris J. Dijkstra, Hans A. van der Sloot, Andre van Zomeren & Johannes C.L. Meeussen

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Outline

- Comparison of different leaching/extraction tests
- Approach to identification of processes controlling contaminant leaching
- Estimation of modelling parameters
- Examples for soil, sludge and compost
- Conclusions

Comparison of different leaching/extraction tests:

\( pH \) – the major controlling variable

From: EU Network on harmonisation of leaching/extraction tests
Problem definition
How to identify the major processes that control the leaching properties of complex heterogeneous systems?

Source: Surface and Aqueous Geochemistry Group, Stanford, USA

Approach to identification of geochemical processes
“Geochemical fingerprints”

Modelling contaminant speciation

Modified after M. Gfeller & R. Schulin, ETH, Zürich
How to estimate key parameters?

Concentration of exchangeable contaminant ("availability")

Examples of availability vs. total concentration

How to estimate key parameters?

pH/E_H and major elements

Solution (leachate) composition:
→ Fixed pH and redox potential (E_H)
→ Fixed total conc. major elements (incl. DOC)
→ Calculation of mineral saturation (SI)
Examples of solubility-control

How to estimate key parameters?
Complexation by DOC

Fulvic Acid Separation Technology ("FAST")
Rapid batch procedure for determination of humic and fulvic acids

1. Acidify → pH = 1
2. HA → centrifugation
3. HA → TOC analysis = HA
4. FA → HY → centrifugation
5. FA → HY → TOC analysis = FA + HY
6. 1 hour equilibration
7. HA redissolution
8. TOC analysis = HA

van Zomeren & Comans, 2004 (submitted)

Example of DOC speciation and its effect on Cu-leaching

DOC speciation in CW5 compost

Cu - MSWI BA/CW5 compost

Enhanced Cu leaching by dissolved HA

How to estimate key parameters?

Organic (humic) matter:
- [DOC] in alkaline extraction (IHSS procedure)
- Fractions of humic, fulvic & hydrophilic acids
- NICA model & generic parameters

Hydrous Ferric Oxide (HFO):
- [Fe] in ascorbic acid extraction
- Diffuse Layer Model & parameters

Amorphous Al(OH)₃:
- [Al] in oxalate extraction
- Diffuse Layer HFO Model & parameters

Clay:
- Mineral fraction < 2 µm
- Illite as assumed clay mineral (CEC = 0.25 mol/kg)
- Donnan Model
Modelling metal leaching from MSWI bottom ash mixed with different amounts of compost

Modelling metal leaching from three different soils

Metal speciation in solid phase and leachate of a contaminated soil

HFO-soluble Fe-hydroxides, DFA-soluble fulvic acid, SHA-soluble humic acid, Free=dissolved free metal ion, INORG=dissolved inorganic complexes, DFA=dissolved fulvic acid, DHA=dissolved humic acid
Multiple major and minor element speciation modelling of EUROSOIL 4

ORCHESTRA:
- Selected finite minerals;
- Sorption on Fe(hydr)oxide & organic matter;
- Complexation with DOC and inorganic ligands

→ Consideration of multi-element interactions is crucial

Geochemical speciation modelling of soil and soil-like materials: fields of application

- Identification of mobile chemical "species", that may be transported towards and in groundwater (e.g. free, inorganically and organically complexed ions)
- Assessment of potential bioavailability in relation to uptake by plants and soil organisms, and ecotoxicity (predominantly free ions)
- Risk assessment of contaminated sites (from both industrial and agricultural soil use)
- Evaluation and improvement of soil remediation techniques (binding potential vs. contaminant "availability")
- Monitoring of soil quality ("available" species in addition to total composition)
- Input to more sophisticated reactive transport models (more widely applicable than $K_d$-based models)

Conclusions

- pH-dependence leaching test captures information from a multitude of (batch) leaching tests and provides the necessary geochemical fingerprints to identify the underlying leaching processes
- Powerful "horizontal" approach towards process identification: pH-stat, specific chemical extractions and geochemical modelling
- Great perspective for geochemical modelling of leaching processes, based on fundamental published thermodynamic parameters (i.e. without fitting parameters)
- Dominant role of (dissolved) organic carbon in contaminant leaching from soils and many soil-like (waste) materials: new rapid techniques to quantify reactive organic matter fractions (humic and fulvic acids)
- The way forward:
  → prediction of pH and DOC solubility
  → inclusion of full modelling approach in LeachXS
Leaching of biocides from treated wood in service

Abstract
Wood preservatives are regulated under the European Biocides Directive 98/8/EC. This directive requires authorisation procedures for active substances and biocidal products that include an environmental risk assessment. The risk assessment is based on the comparison of predicted environmental concentrations (PEC-values) and predicted no effect levels (PNEC-values, derived from ecotoxicological data). The release into the environment can occur via leaching during either storage or service. This process is described by time dependent flux rates that can be converted into cumulative emissions during the assessment periods for different emission scenarios.

The environmental conditions that treated wood is exposed to can be quite different. This is considered by the differentiation of several use classes. The CEN TC38 WG27 developed two laboratory methods that are related to different use conditions. These procedures are under discussion as OECD guideline proposals. The methods have been investigated and optimised during research projects that have been supported by National and EC funding.

An important question is the transfer of laboratory data to variable service conditions. First comparisons of laboratory data that were obtained from the OECD guideline tests with data from treated specimens that were exposed to natural weathering demonstrated that this relation did not only depend on the weathering conditions, but also on the substances that were investigated. A simple transfer of laboratory data to the service conditions is impossible. This limits the use of laboratory data for the prediction of environmental concentrations.

An alternative approach might be a description of the leaching potential of certain substances on the basis of laboratory tests. These intrinsic data can be used in models that simulate service conditions. Van der Sloot and his colleagues (ECN Petten) applied the ORCHESTRA programme to wood that was treated with a preservative containing copper and tebuconazole and has been exposed to use class 3 conditions. The first simulations of tebuconazole concentrations in rainwater as well as cumulative emissions were quite promising when the data were compared to actual measurements.

There are certain aspects that should be considered if modelling is applied to treated wood in service. Leaching of substances is controlled both by the availability of water and the distribution of the active ingredients in the wood. Accurate predictions of emissions via leaching require knowledge on the depth of penetration of water into the wood as well as the duration of wetting periods (especially for use class 3). The distribution of the active ingredients depends on e.g. the treatment procedures and wood species. Leaching data on treated wood probably must be related to the wood species and treatment procedure. The leachability of a certain active ingredient may be dependent on
other substances that are contained in the formulated wood preservatives. Leaching from wood can depend on the direction of the transport processes, i.e. the permeability of wood is lower across the grain than along the grain.
Leaching of biocides from treated wood in service

Ute Schoknecht
Federal Institute for Materials Research and Testing (BAM)
Berlin, Germany

European Biocides Directive 98/8/EC

- environmental risk assessment for the authorisation of active substances and biocidal products
- PEC/PNEC calculation

PEC/PNEC concept

- release into the environment: time dependent leaching process during either storage or service
- flux rates ➔ cumulative emissions during the assessment period ➔ OECD ESD models for calculation of PEC
Use classes for preserved wood
Without water contact (use classes 1 and 2)
Use class 3 weathering during storage and under service conditions
Use class 4 soil/permanent water contact
Use class 5 contact with sea water

Different environmental conditions!

PNEC
Calculation based on ecotoxicological experiments with model organisms

Estimation of emissions
OECD guideline proposals by CEN TC38 WG27
- Use Class 3
  - dipping events on certain ‘immersion days’
  - 60 days

- Use Classes 4 and 5
  - permanent water contact
  - 60 days
Leaching of biocides from treated wood in service

Project supported by the EC

- March 2004 until March 2005
- Test of OECD guideline proposals
  ➢ repeatable results for different types of preservatives
- Recommendations
  ➢ methods can be optimised

What about the relation between emissions in laboratory tests and emissions under service conditions?

Emission of tebuconazole in laboratory and field tests
Factors that influence leaching of biocides from treated wood

- Storage and service conditions
  - weather conditions
  - orientation of exposed surfaces

- Formulation of the preservative
- Wood species
- Treatment procedure

Limitations of the PEC/PNEC concept

- laboratory data do not represent variable storage and service conditions
- lack of experiences
- literature data that allow to balance PEC/PNEC calculations are available only for certain types of preservatives (formulations based on Cu and Cr)

Potential benefits of modelling

- leaching potential of substances in certain formulations
  - diffusion coefficient, retention factor
  - pH dependence
- characteristic data for wood

  model variable environmental conditions
Specific aspects of treated wood

- availability of preservatives depends on
  - uptake of water
  - distribution of the preservative in the wood
  - leaching direction (different permeability along/across the grain)
- differences between wood species
- formulation effects leachability
Exposure to natural weathering (UC 3)

Leaching of copper from Cu/tebuconazole by natural weathering

Leaching of tebuconazole from Cu/tebuconazole by natural weathering
Leaching of biocides from treated wood in service

Horizontal aspects of Leaching, Workshop Ispra, 14-15 February 2005

Leaching of Cu and tebuconazole vs. rain

Emission of copper in laboratory and field tests

Emission of copper from different preservatives in laboratory and field tests
Emission of Cu from different preservatives in guideline 2 experiments

![Graph showing emission of Cu from different preservatives over time.](image)
Leaching tests for products in contact with drinking water

Abstract

The objective of the Drinking Water Directive “shall be to protect human health from the adverse effects of any contamination of drinking water by ensuring that it is wholesome and clean” (Article 1). The parametric values have been set using the WHO “Guidelines for drinking water quality” and the advice from the Scientific Committee on Toxicology, Ecotoxicology and Environment (SCTEE). The parametric value is selected to ensure that the drinking water can be consumed safely on a life-long basis. “The Member States shall take all measures necessary to ensure that regular monitoring of the quality of drinking water is carried out in order to check that the water available to consumers meets the requirements.... Sample should be taken so that they are representative of the quality of the water consumed throughout the year” (Article 7 §1).

Further the DWD regulates that appropriate monitoring programmes as established by the competent authorities “shall meet the minimum requirements set out in Annex II” (Article 7 §2). All parameters mentioned in Annex I are subject to audit monitoring. However the minimum annual amount of samples to be taken is very low compared to the amount of inhabitants in a supply zone; 1 sample for >500-5000 inhabitants, 1-4 samples for >5000-50.000 inhabitants, 4-13 samples for >50.000-500.000 inhabitants and 14 samples for >500.000 inhabitants the exact amount of samples depending of the real amount of inhabitants. In the Member States (MSs), the use of Construction Products in contact with Drinking Water (CPDW) is regulated by National Acceptance Schemes (NASs) to protect the consumer against bad drinking water quality. The various compounds used to produce CPDW and reaction products emerging from the manufacturing may migrate into the drinking water. The various NASs differ significantly in philosophy, assessments and tests, and acceptance criteria. Therefore NASs are a barrier for the internal European Market. The MSs requested Enterprise Directorate General to solve this problem. It was decided to set up a Regulators Group on CPDW (RG-CPDW) which should prepare a European Acceptance Scheme (EAS) for CPDW. The RG-CPDW concluded that it was not possible simply to merge all NASs in the EAS: in some cases the MSs use different types of assessments for one parameter. For most assessments the RG-CPDW agreed on the test to be used. For these tests the European Organisation for Standardisation (CEN) developed prototype European Norms which are in the procedure to become EN standards. However, for some assessments the tests were not available or differed very much in the various Member States. The RG-CPDW proposed to carry out co-normative research for these assessments, which resulted in this project. This presentation gives an overview on role of the JRC IES IMW Unit in carrying out and co-ordinating this co-normative research.
Leaching tests for products in contact with drinking water

Releasing of chemicals to drinking water

Problems of odour, taste, colour, turbidity, microbial growth, toxicity

Eddo J. Hoekstra

Legal Framework

Drinking Water Directive (98/83/EC)
- compliance of water quality at the consumers' tap (art. 6.1)
- acceptable effect of distribution system on drinking water quality (art. 10)

- CE-marking of construction products fit for their intended use
  - compliance with harmonised technical specifications
  - Interpretative document No. 3: Hygiene, health and the environment

European Acceptance Scheme for products in contact with drinking water
- mechanical requirements (Mandate 131)
  - AoC 4: testing by manufacturer and review by certification body
- health-related aspects (Mandate 136)
  - AoC 1+: certification body certifies production control system, does testing and surveillance

EAS certification

- Provision of full formulation and composition
- Acceptance of substances and materials
  - Positive List, Composition List, Approved Constituent List
- Testing to ensure product conforms specification
  - Preparation of migration water
    - Organoleptic assessments
    - odour, flavour, colour, turbidity
  - General hygiene assessments
    - TOC, chlorine demand
  - Migration/dissolution of toxic substances
    - DWD, Positive List, Composition List, unsuspected compounds by GC-MS
  - Toxicological assessments (in future???)
    - cytotoxicity, genotoxicity
  - Enhancement of microbial growth
  - Surface residues on metals
Leaching tests for products in contact with drinking water

Test parameters

<table>
<thead>
<tr>
<th>Material</th>
<th>Organic</th>
<th>Metallic</th>
<th>Cementitious</th>
<th>Glassy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Odour</td>
<td>+</td>
<td>–</td>
<td>+</td>
<td>–</td>
</tr>
<tr>
<td>Flavour</td>
<td>+</td>
<td>–</td>
<td>+</td>
<td>–</td>
</tr>
<tr>
<td>Colour</td>
<td>+</td>
<td>–</td>
<td>+</td>
<td>–</td>
</tr>
<tr>
<td>Turbidity</td>
<td>+</td>
<td>–</td>
<td>+</td>
<td>–</td>
</tr>
<tr>
<td>Chlorine demand</td>
<td>+</td>
<td>–</td>
<td>?</td>
<td>–</td>
</tr>
<tr>
<td>TOC</td>
<td>+</td>
<td>–</td>
<td>+</td>
<td>–</td>
</tr>
<tr>
<td>Relevant DWD parameters</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>PL substances</td>
<td>+</td>
<td>–</td>
<td>+/–</td>
<td>–</td>
</tr>
<tr>
<td>Unsuspected substances</td>
<td>+</td>
<td>–</td>
<td>+/–</td>
<td>–</td>
</tr>
<tr>
<td>Composition</td>
<td>–</td>
<td>+</td>
<td>–</td>
<td>+</td>
</tr>
<tr>
<td>Microbial growth</td>
<td>+</td>
<td>–</td>
<td>+/–</td>
<td>–</td>
</tr>
<tr>
<td>Surface residues</td>
<td>–</td>
<td>+</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

Migration water – organic

- Tap water with <0.2 mg/l as Cl₂
  - Flushing
  - Stagnation
  - Test water (0, 1, 50 mg/l free Cl₂)
  - Migration

Migration water – cementitious – prEN 14944

- Preconditioning water
  - Test water (0 or 1 mg/l free Cl₂)
  - Migration

Realistic S/V = 5-40 /dm
Migration water – metallic

Three purposes:
• Qualification of material as a reference material
  – for a category of materials
  – covering a broad range of water compositions.
• Comparative testing of materials
• Interaction of material with a local water

Terme di Diocleziano, Museo Nazionale Romano, Roma

Dynamic Test Facility

Control pipe
• Single length stainless steel
• EN 10088-1:1995, material no. 1.4401
Test pipe
• Single length test pipe
• ID = 13±1 mm or the next largest commercially available size

Dynamic Test Facility at JRC
Leaching tests for products in contact with drinking water

**Operation conditions**

- The outlet ≥ 300 mm than the inlet
- The test line shall not at any point be lower than the previous section
- Operation temperature of 20±5°C
- Pressure of test water at entrance of test rig ≥ 3 bar
- Flushing test rig using local water (1 h; 5±0.5 L/min) before operation
- Regular daily operation controlled automatically
- Turbulent flow (5±0.5 L/min)
- Equipotentially bonding of test pieces
- Measurement stagnation curves (month 1-6 then week 39, 52,65, 78, 91, 104)
- 4 HS sample every week

**Test water – general**

- Local drinking water not influenced by the domestic system
- Quality check of test water on metallic contaminants

<table>
<thead>
<tr>
<th>Element</th>
<th>Max. conc. (µg/L)</th>
<th>Max. % DWD limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>100</td>
<td>10</td>
</tr>
<tr>
<td>Cu</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Zn</td>
<td>50</td>
<td>10</td>
</tr>
<tr>
<td>Mn</td>
<td>5</td>
<td>10</td>
</tr>
<tr>
<td>As</td>
<td>1</td>
<td>10</td>
</tr>
</tbody>
</table>

- Determination of water composition
  - T, pH, conductivity, dissolved O₂, total alkalinity, TOC
  - Ca, K, Na, Mg, Al, Bi, P, Si
  - Cl, NO₃, SO₄

**Test water – specific**

Materials approved compared to reference materials

<table>
<thead>
<tr>
<th>Modification of local water, if necessary, to meet the requirements:</th>
</tr>
</thead>
<tbody>
<tr>
<td>[Cl] = 220 ± 20 mg/l adjusted by adding NaCl</td>
</tr>
<tr>
<td>pH = 6.7 ± 0.2 adjusted by adding gaseous CO₂ or dilute HCl</td>
</tr>
<tr>
<td>O₂ saturation at testing temperature min. 70%</td>
</tr>
<tr>
<td>alkalinity min. 0.5 mmol/l</td>
</tr>
<tr>
<td>[PO₄] &lt; 0.2 mg/l</td>
</tr>
<tr>
<td>DOC &lt; 1.5 mg/l</td>
</tr>
<tr>
<td>Local water treated by addition of corrosion inhibitors shall not be used for testing</td>
</tr>
</tbody>
</table>

Materials approved as reference materials in four different natural water types:

<table>
<thead>
<tr>
<th>pH</th>
<th>HCO₃</th>
<th>Ca+Mg</th>
<th>Cl+(2×SO₄)</th>
<th>Conductivity</th>
<th>Org. carbon</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>mmol/l</td>
<td>mmol/l</td>
<td>mmol/l</td>
<td>µs/cm</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>&lt;7.3</td>
<td>&gt;5</td>
<td>&gt;2.5</td>
<td>&gt;4</td>
<td>&gt;800</td>
</tr>
<tr>
<td>2</td>
<td>7.5±0.2</td>
<td>2±0.2</td>
<td>1±0.1</td>
<td>&gt;4</td>
<td>?</td>
</tr>
<tr>
<td>3</td>
<td>8±0.2</td>
<td>1±0.2</td>
<td>0.5±0.1</td>
<td>&lt;2</td>
<td>&lt;600</td>
</tr>
<tr>
<td>4</td>
<td>6±0.2</td>
<td>1±0.2</td>
<td>0.5±0.1</td>
<td>&lt;1</td>
<td>&lt;600</td>
</tr>
</tbody>
</table>
Leaching tests for products in contact with drinking water

Modelling – polymers

Boundary conditions:
- Homogeneous distribution of the migrant in the polymeric material
- Finite volume of the polymeric material and the drinking water (DW) is well mixed
- No boundary resistance for the transfer between P and DW during the migration process
- Constant total amount of the migrant in P and DW during the migration process

\[ \frac{m_{t}}{A} = \frac{c_{P,0}}{\rho_{P}} \frac{d_{P}}{\alpha} \int_{0}^{\infty} 2a \left( 1 + \alpha \right) \exp \left( \frac{D_{r} \alpha^{2}}{\alpha} \right) \alpha \cdot \frac{V_{P}}{Q_{L}} \cdot c_{L,\infty} \frac{c_{L,\infty}}{c_{L,\infty}} \]  

\[ K_{P,DW} = \frac{c_{P,0}}{c_{L,\infty}} \]

- Surface in steady corrosion state
- Initial water concentration uniform throughout the tube (\( C = C_{0}, 0 < r < r_{int}, t = 0 \))
- The average concentration (\( C_{a} \)) in the cylinder as function of time:

\[ C_{a} = \frac{C_{\text{ave}}}{C_{\text{ave}}} = \frac{1}{\sum_{n=1}^{\infty} 4 \cdot \exp \left( - D \cdot \alpha_{n}^{2} \cdot t \right)} \]

\[ C_{a} = \frac{1}{\sum_{n=1}^{\infty} 4 \cdot \exp \left( - \frac{2.4048}{r_{\text{ave}}} \cdot D \cdot r_{\text{ave}} \right) \frac{4 \cdot 5.52012}{5.52012} \exp \left( - \frac{5.52012}{r_{\text{ave}}} \cdot D \cdot r_{\text{ave}} \right) \]  

where \( \alpha_{n} \) are the roots of \( J_{0} (r_{\text{ave}} \cdot \alpha_{n}) = 0 \) (first kind Bessel function of the zero order)

New versus aged materials

What about aging effects?

EAS = new products
Waste = aged products
**Point in distribution – nickel**

- **Service pipe of polyvinyl chloride (±34.8 m)**
- **Copper tubing before and after watermeter**
- **Kitchen: Damixa (50-100 ml); Cu tube (±26 ml); flexible hose (±30 ml)**
- **Bathroom: Tap? (? ml); SS tube (±16 ml)**
- **500 ml ~ 3.8 m ? length Cu tube**
- **Median saturation [Cu] = 990 µg/l**

**Stagnation time – copper**

- 
- **Stagnation time (h)**
- **Conc. Cu (µg/l)**
- **• K 1st**
- **• K 2nd**
- **• B 1st**
- **• B 2nd**
- **• FF**
- 

**Stagnation time – lead + zinc**

- **[Pb] determined by diffusion**
- **Median saturation [Pb] = 2.9 µg/l**
- **[Pb] strong correlation with [Cu]**
- **Source is different**
- **[Zn] not determined by diffusion, but by dissolution process**
Review of a 12 years experience in leaching standardisation

Abstract
This review concerns the standardisation work undertaken in AFNOR in the early nineties. At that time the French Government has established a new waste law with the concept of “ultimate” waste. AFNOR established a standardisation committee on long term behaviour of waste promoted by users, industries and regulators addressing both recycling and landfilling.

The first step was the development of a methodology standard published in 1995 XP X30 407 “Wastes – Methodology for determining long term behaviour” benefiting of in depth practical works. It contributed later on to the development of EN 12920.

The second step was a specific programme started in 1995 for a selection of materials. This was done in the framework of theses methodology standards : standardise by requirements expressed as much as possible in terms of performance criteria, standardisation of test selected further to a first review of utilisation leaching scenario and focus on prediction of behaviour (material related). The main aspects addressed are the continuous renewal of leachant at high or zero or scenario rate, the scenario driven composition and temperature. This resulted in six Afnor standards on top of the methodology standard.

The third step was launched more recently with experimental validation of the two main standards. The validation results including improvements of the test standards requirements were incorporated in a standard under publication that is applicable to a much wider range of materials and combines different tests of the second step as well as experience gathered with other materials than those selected in the second step.
ISPRA leaching workshop – Feb. 2005

Review of a 12 years experience in leaching standardisation

by

Jean-François VICARD
STRATENE - Lyon
Chairman of the AFNOR committee for coordination of environmental measurement methods

• Enforcement of a new waste law in the early nineties
• Concept of “ultimate waste”
• Establishment in AFNOR of a standardisation committee on long term behaviour of waste promoted by users, industries and regulators

• Development of a methodology standard XP X30 407 “Wastes – Methodology for determining long term behaviour” benefiting of in depth practical works. Publication in 1995
• Contribution to CEN TC 292 activities. In particular to WG 6
Review of a 12 years experience in leaching standardisation

Specific programme started in 1995
• How to standardise: requirement expressed as much as possible in terms of performance criteria
• Which tests to standardise: first review of utilisation leaching scenario
• Focus on prediction of behaviour (material related)

Review of a 12 years experience in leaching standardisation

Specific programme started in 1995
• Renewal rate of leachant
  - high
  - Zero
  - scenario
• Composition
• Temperature

Review of a 12 years experience in leaching standardisation

• From the beginning of this century on-going work on horizontal approaches for standards
• Importance of validation (robustness – variability)
• First step of validation
Application of ENV12920 - French experience

Abstract
In order to assess the application of the ENV12920 standard during the last five years, a study on the main data of studies and R&D programs has been realized. The whole data has been structured on a database form (ACCESS®) which allows doing requests on funding organisms, wastes, materials and/or utilizations and essays. This database is aiming at future potential extensions.

The critical analysis of each of the 10 studied programs put into evidence four main difficulties (field of improvement) linked to the performing of this methodology:

- description of the considered scenario and links with the choice of the studied influence factors are not often explained or described,
- links between identified influence factors at previous steps and the performed tests are not always explicit,
- the modeling step doesn’t always end in a relevant or exploitable behavioral model towards data and influence factors,
- validation tests are often performed, but their interpretation doesn’t always allow to reach objectives, that is to say to validate the behavioral model.

According to these different points, recommendations can be made, as for instance:

- in the scenario description step, the distinction between the description of the considered application and the description of a conceptual model that would constitute a simplification and a schematization of this application,
- the performing of two validation levels, the first validates the model at the conceptual model scale, and the second validates the model at the considered application scale.

A diagram has been proposed in order to facilitate the application of the methodology.

The usefulness of this standard is an evidence according to the several situations where wastes (or others pollutants origins) are exposed to leaching in contact with the natural medium. Nevertheless, its application absolutely needs to be supervised at two levels:

- by a guide of the best practices as far as coherence between the different steps and justification of the methods choices are concerned,
- by the competence and the experience of the staff in charge of the implementation of these different steps and methods.
Application of ENV12920

French experience

January 2005

10 Programmes taken into account

(1) **BRITE EURAM METALEUROP**: Valorization of Pb-Zn primary smelters slag, METALEUROP (for the European Commission) 1997 (ref. POLDEN: 94A)

(2) **CAEN**: Underground quarry backfilling with MSWI bottom ash-containing mortars: INERTEC 2003

(3) **LANDFILL DIRECTIVE**: "Acceptation of inert wastes, annex 2 of Directive 1999/31/CE concerning landfill of wastes", TAC working group: France (BRGM, INSA de Lyon, Ministère de l'Environnement), The Netherlands (ECN, VROM), UK (WRC, Ministry of Environment), Germany (Ministry of Environment - BMU), Denmark (DHI, Ministry of Environment), Austria (Ministry of Environment), Suède (SGI)

(4) **ECOCOMP**: Research programme on ecocompatibility of waste, Document réalisé par POLDEN - ADEME 1999

(5) **GHOST**: Research programme on ecocompatibility of a Heap of salted wastes from the soda industry. POLDEN SOLVAY ADEME

(6) **REVASOL**: Utilisation of treated fly ash from MSWI issued from NEUTREC process, POLDEN - BERTIN – SOLVAY - ADEME en 2003

(7) **SNET-EDF**: Assessment of Environmental Impact of utilisation of silico-aluminoeous coal fly ash in road application, POLDEN - EDF-SNET in 2000

(8) **SVDU**: Environmental impact assessment of the use of municipal solid waste incineration bottom ash in roadwork, CREED-LCPC-BRGM-SVDU-ITASCA – ADEME

(9) **VALOMAT**: New civil engineering materials based on bottom ash valorisation, SOLETANCHE-BACHY – POLDEN (for the European Commission) in 2001

(10) **VIVALDI**: Programme "VIVALDI" on vitrified fly ash from MSWI Bordeaux Métropole : Annex 1: Assessment of long term leaching behaviour EUROPLASMA - ADEME - CEA in 2000
**Considered Combinations**

<table>
<thead>
<tr>
<th>Scenario Evaluation Criteria</th>
<th>Cost by unit</th>
<th>Model by unit</th>
<th>Data by unit</th>
<th>Model API module</th>
<th>Data API module</th>
<th>Validation API module</th>
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<tbody>
<tr>
<td>Building</td>
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<td>BOTE EUGAR</td>
</tr>
<tr>
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<td>Gutter drainage</td>
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<td>BOTE EUGAR</td>
<td>BOTE EUGAR</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Main problems encountered**

1. **Description of the scenario**
   - Confusion between data from the industrial reality (extreme complexity) and the significative data to be considered (conceptual scenario)
   - Exposures conditions not always specified

2. **Selection of the tests**
   - Not always links between identified influence factors at previous step and the performed tests
   - Non justified test or without any exploitation
   - Identified influence factors without any associated tests

3. **Modelling**
   - Mentioned factors and/or measured data non integrated in the predictive mathematical modelling
   - Decision or diagnosis based on a direct exploitation of the experimental data (pilot scale) without any calculation
   - Selection of mathematical tools not always rational (i.e. based on the true nature of the phenomena to be considered) but often linked to the scientific background of the operators (mineralogy, chemistry, hydrodynamics, mass transfer,...):
     - "My colleagues always did like this..."
     - "I have downloaded a new funny tool...let's try it!"
Main problems encountered

4. Validation

- Not always a validation step
- Pilot scale in most of the cases
- Pilots with a non expected behaviour (not in the conceptual scenario (accidental spill))
- Results different from the prediction without resolution of the problem (like an adaptation of the model with integration of neglected factors in the conceptual scenario (carbonation, bio leaching, ...)
- Problem of different scales of validation (pilots and instrumented full scale works) leading to different results
Conclusions of the Workshop

This workshop was launched to address the complex issue of leaching from a scientific and standardisation point of view. Accordingly, the below statements shall not be considered and construed in a regulatory framework. They shall be understood in a scientific and standardisation way.

The following conclusions were achieved.

1. The needs from current regulatory actions in relationship to leaching were illustrated by some speakers and it was shown that the different sectors, e.g. groundwater protection, construction products for drinking water, soil characterization, treated biowaste, sludge, etc. are not necessarily interacting very well whereas the questions that need to be answered seem to be similar. A top-down (material characterisation and assessing release) and bottom-up approach (environmental quality objectives and what that means for acceptance of releases) need to be linked.

2. A presentation of the characterisation methods and their potential to address different questions was given covering the waste and soil field. The fact that the methods developed by working groups in close contact through common experts have a close resemblance hold promise to come to horizontal standards in the future, provided that the remaining differences could be overcome. There was a general consensus on the need in leaching standards to document the testing conditions (pH, REDOX, etc.) in the report, including the relevant information on the overall measurement process, in accordance with the general requirement for test methods (EN ISO 17025).

3. The leaching of organic contaminants was identified as an issue of further discussion and will require further research. Some of the critical issues discussed were the role of particulate and dissolved organic matter (POM and DOC), degradation of the target compounds during leaching experiments, the interaction with materials (sorption), the nature of DOC sub-fractions as well as repeatability/reproducibility issues. When testing the leaching of organics of low solubility, the separation of eluate and solids becomes critical to the outcome of the test, and very stringent procedures become necessary. Other major influence factors may also need further research, e.g. role of micro-biology in organic compounds degradability process.

4. In all application of leaching, the need for support to validation work was emphasized by the participants. In the environmental field, such validation is generally considered as a prerequisite for adoption of the standards as EN and for being fit for purpose.

5. Several contributions highlighted a framework of leaching tests for asking the proper questions to get the right answers. The European framework specified in the EN 12920 methodology was addressed by some European speakers. The U.S. framework that was addressed by an American speaker may evolve from the strict TCLP basis. Possible interactions between US and EU approaches in this regard were highlighted and are welcome.

6. Some speakers presented as a way forward the development of scenario descriptions for specific applications using the characterisation tests as means to
identify and determine relevant intrinsic material properties for that scenario in the framework of EN 12920. The prediction as to what will be the environmental impact of a certain management option seems to be the main interest from a regulatory perspective. Some speakers underlined the need for the development of specific scenario descriptions as guidance to practitioners for facilitating the application of the EN 12920 methodology. Given a certain question, how must one select an appropriate test and what steps are necessary to come to a conclusion?

Some speakers also addressed the question of extrapolation of lab data to field conditions that needs to be discussed further. This could significantly enhance the usefulness and applicability of leaching tests.

7. The limitations of simulation tests applicable to one situation were shown when used alone. Measuring intrinsic properties and modelling with parallel verification against field observations, as e.g. provided in the EN 12920 methodology, appears to have a greater potential.

8. The horizontal approach in assessing soil and groundwater impact in relation to the CPD needs is promising, too. The fact that testing of wood preservatives is headed in the same direction is a step in the right direction. This applies also for the approach taken in dealing with mining waste. Here a special property namely the reducing properties and its potential to create acid rock drainage is not yet addressed in standardisation. The issue of reducing properties is not limited to mining waste. So it is definitely relevant for waste as well.

9. In the drinking water sector test methods are already standardised in CEN. The testing facilities developed in JRC-Ispra apply these CEN test methods for material in contact with drinking water and were shown to be quite promising.

10. A modelling work on soil, sludge and biowaste has been shown to be quite promising in dealing with relevant factors controlling leachability.

11. Hierarchy in testing was addressed and its potential to do detailed characterisation when needed and apply more simple testing when sufficient knowledge has been gained already. In order to be able to make most efficient use of the more costly characterisation, it makes a lot of sense to develop a database/expert system to facilitate comparison, avoid unnecessary duplication, identify gaps in the knowledge and use it as basis of reference for compliance testing, quality control testing or treatment efficiency verification. The participants were generally interested in the development of such a system and many of the participants would be ready to support such an initiative.
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