

Leaching tests in Europe



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Leaching tests in Europe

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- ” Overview types of analytical tests
- ” Real Total analyses
- ” So-called %Real Total+analyses
- ” Leaching tests
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- ” Legislation related to leaching and bioavailability
- ” Conclusions

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Who am I?

- “ Business Development Manager ALcontrol Laboratories
- “ Responsible for:
 - “ Special projects, e.g. for ministry Environmental Ministry NL (e.g. development of laboratory guidelines related to legislation)
 - “ National and international Committees (NEN, SIKB, CEN, ISO)
 - “ Assessment of and implementation of new analytical developments
 - “ FeNeLab (Dutch Federation of Laboratories, chair Environmental Council of directors)

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Statement

“The unnecessary full analysis of soil to learn if it is fertile or not, cannot be argued enough. The long and short of it is availability, which cannot be derived beforehand. The analysis shows what there is, agriculture must draw its own conclusions from that+”

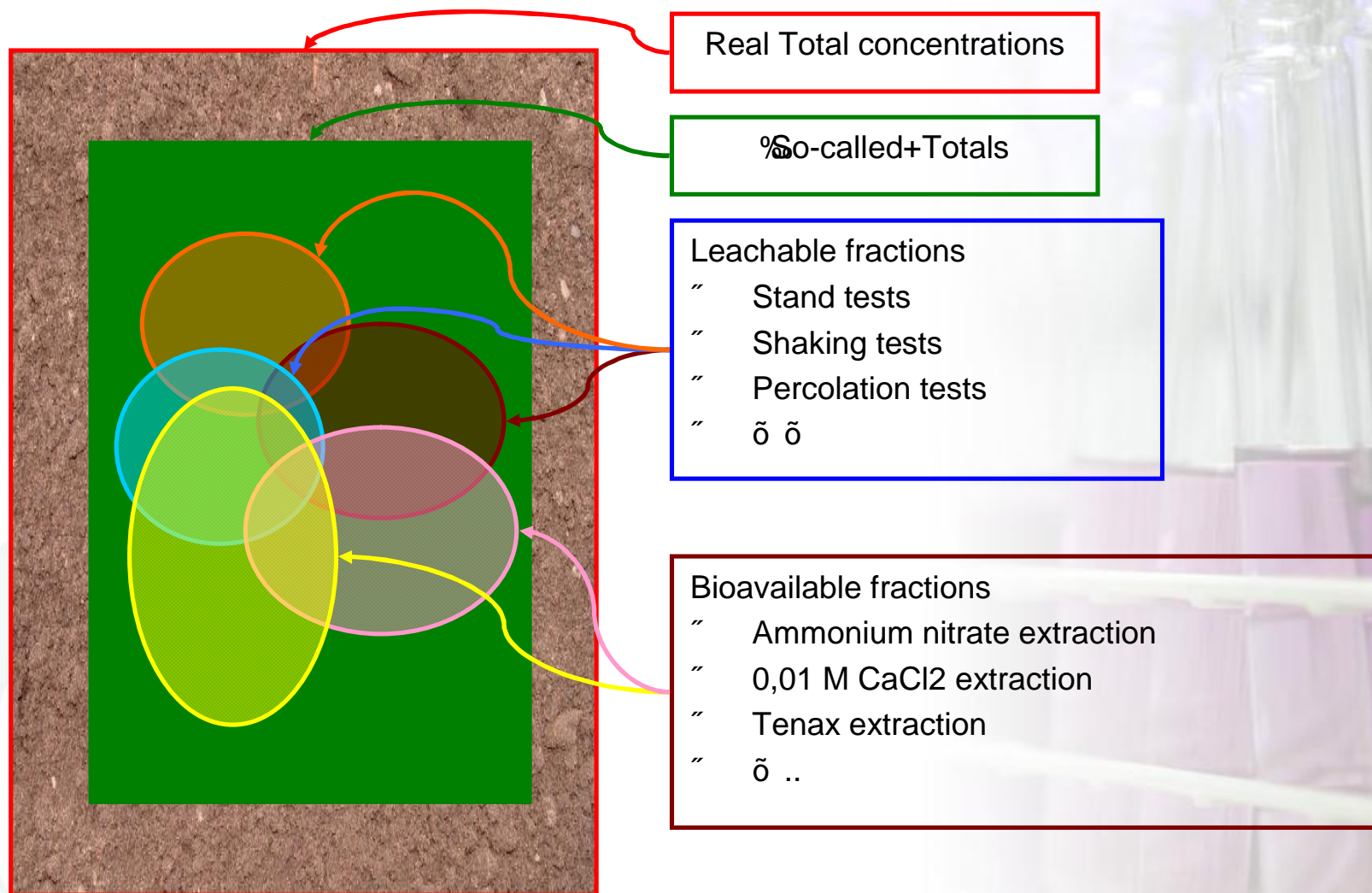
Mulder, 1860

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Overview types of analytical tests



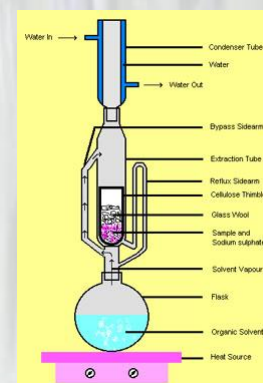
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Real Total extraction

“ Aim: extract everything up to the last molecule of a compound

- Total extraction of elements from soil, e.g. by cooking with a mixture of $\text{HCl} + \text{HNO}_3 + \text{HF}$
- Total extraction of organic compounds
 - “ Soxhlett extraction (few hours cooking)
 - “ Cooking under high pressure



“ Examples legislation

- OVAM Compendium (Flanders, Belgium) for heavy metals and for semi-volatile organic compounds
- Legislation related to asphalt testing (content of PAH)

“ Labor intensive, strong chemicals used → safety issues on the lab

“ NOT MUCH USED in LEGISLATION!



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So-called “Total extraction”

” Aim: get a good indication of %Real+total concentration

- . Aqua Regia ($\text{HCl} + \text{HNO}_3$) extraction for elements (by 2 hr cooking or microwave extraction)
- . HNO_3 extraction for elements (less strong than Aqua Regia)
- . Shaking soil with a organic solvent, e.g. acetone + petroleum ether, methanol

” Legislation examples

- . Aqua Regia: most European countries (France, Spain, Italy, Germany, Netherlands, etc.) → done by most labs in Europe
- . HNO_3 extraction: Nordic countries (Norway, Denmark, Sweden)
- . Shaking for semi-volatile organic compounds: all European countries except Belgium



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Leaching tests (1)

” Aims

- . Impact assessment
- . Compliance test

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Leaching tests (2)

” Impact assessment

- . Methodology described in EN12920
- . Method dependent on information needed:
 - ” Forecast of a property after a specified time, e.g. release
 - ” Comparison of treatment options
 - ” Data related to regulatory requirements
- . Depending on the scenario or the soil characteristics, different leaching test procedures may be needed

Leaching tests (3)

“ Compliance tests (1)

- . Methodology described in ISO 18772
- . For comparing with background values, e.g. targets set by legislation, treatment efficiency
- . Once a suitable choice is made, the test procedure remains unchanged for the next time
- . 2 types of tests
 1. Static tests
 2. Dynamic tests
- . Test conditions which influence results
 - “ pH of the leachant
 - “ L/S (liquid . solid) ratio
 - “ Contact time
 - “ Temperature of the (lab) environment

Leaching tests (4)

“ Compliance tests (2)

- . 3 different aims, classified in ascending order of representativity and complexity
 1. Compliance and quality control (QC) leaching tests
 2. Basic characterization
 3. Simulation test

Leaching tests (5)

” Compliance tests (3)

- . Compliance and quality control (QC) leaching tests
 - . Initial screening of release to water
 - . Generally one or two days
 - . Comparison with legislation target values
 - . Gives no information on the behavior of soil in a given scenario
 - . Example: ISO/TS 21268-1 and -2:
 1. Batch (shaking) test using L/S ratio of 2 l/kg dry matter
 2. Batch (shaking) test using L/S ratio of 10 l/kg dry matter

Leaching tests (6)

“ Compliance tests (4)

- Basic characterization

- Aim: measuring intrinsic properties of soil, used for release prediction, like:
 - » Diffusion coefficients, solubility, physical properties
- Test takes generally longer period, up to a few weeks
- Examples:
 - » ISO/TS 21268-4: Influence of pH on leaching with initial acid/base addition → %parametric test± information for specific parameters
 - » ISO/TS 21268-3: Up-flow percolation test (= %column test±) → %multiparametric test± information of combined effect of different parameters
 - » NEN 7347: Diffusion test (%stand test±): e.g. used for leaching behavior of materials used under water

Leaching tests (7)

“ Compliance tests (5)

- Simulation tests
 - Aim: reproduce, as far as possible, field conditions for checking behavior towards leaching predicted by parametric and multiparametric leaching test (see foregoing slide)
 - Examples:
 - » Lysimeter
 - » Large-scale column test

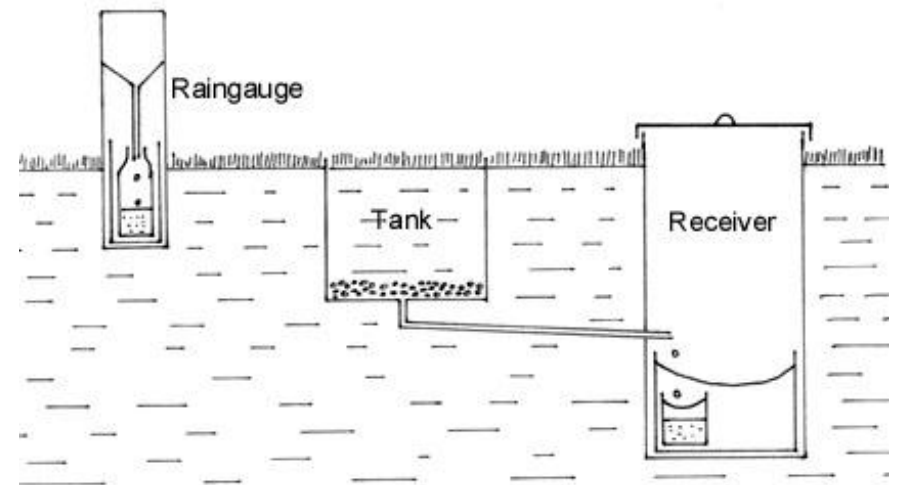


Fig 2. Lysimeter apparatus for measuring evapotranspiration

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Examples of Leaching tests

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Shaking tests (= batch tests)

- “ Dynamic tests for worst case leaching behavior
- “ In France often referred to as 24 hr shaking test+

- “ Conditions

- Temperature: 20 ± 2 °C
- Shaking (overhead)
- Water, initial pH = neutral or 0,001 M CaCl_2

- “ Options:

- EN 12457-1: L/S=2 (water, 1 time shaking 24hr), particles < 4mm
- EN 12457-2: L/S=10 (water, 1 time shaking 24hr), particles < 4mm
- EN 12457-3: L/S=2 and 8 (water, 2 times shaking, 6 and 18 hr), particles < 4mm
- EN 12457-4: L/S=10 (water, 1 time shaking 24hr), particles < 10mm
- ISO/TS 21268 . 1: L/S=2 (0,001 M CaCl_2 , 1 time shaking 24 hr), < 4 mm
- ISO/TS 21268 . 2: L/S=10 (0,001 M CaCl_2 , 1 time shaking 24 hr), < 4 mm



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Column test (1)

- “ Dynamic test to determine the leaching (release) from L/S (Liquid/Solid ratio) 0,1 to 10.
- “ The total Emission at L/S 10 is the maximum leachable quantity over a period from 60 to 100 years in mg/kgDM
- “ Conditions
 - Temperature: 20 ± 2 °C
 - Constant flow of water upwards through column
 - Initial pH = 4.0 or neutral



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Column test (2)

“ Options:

- . Gather 7 fractions (test takes then about 23 days)
 - “ L/S (cumulative): 0,1, 0,2, 0,5, 1,0, 2,0, 5,0 and 10 l/kgDM
 - “ Gives information about leaching over time
- . Gather only one or more fractions

“ Standards (international)

- . ISO 21268 . 3, percolation test
- . EN/TS 14405, percolation test

Diffusion test

- “ Static test to determine the leaching (release) and stability of monolithic and moulded (building) materials.
- “ The Emission is the maximum leachable quantity over a period from 60 to 100 years in mg/m^2 .
- “ Conditions
 - . Temperature: 20 ± 2 °C
 - . Flow zero!
 - . Initial pH = 4.0
- “ Procedure:
 - . Let stand sample in water
 - . Gather 8 fractions (during 64 days)
 - “ Fractions taken: 0,25, 1,0, 2,25, 4, 9, 16, 36 and 64 days
 - “ Gives information about leaching over time



Bioavailability tests

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Bioavailability tests (1)

- ” ISO 17402: methodology description
- ” Aims at risk assessment of contaminants
- ” 2 types of tests
 - . Chemical tests
 - ” Chemical analysis in leachates depending on the specific risk assessed
 - . Biological tests
 - ” exposure of organisms to the soil, or to soil eluates
 - ” Assessment of mortality, growth inhibition etc.
 - ” No leaching → not further dealt with in this presentation

Bioavailability tests (2)

" %Bioavailability+

- . Does not exist as such!
- . It is always: %Bioavailability for± depends on variables like the contaminant, the target, actual soil properties
- . Levels of %protection goals+
 - " Human and higher animals
 - " Soil habitat function
 - " Soil retention function
- . Levels of availability
 - " Actual available fraction
 - " Potentially available fraction
 - " Non-available fraction

" Humans
" Grazing animals, e.g. cattle
" Wildlife

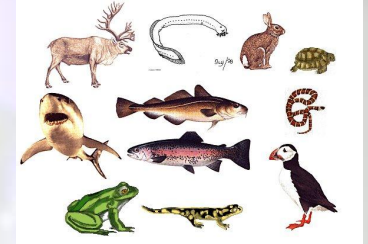
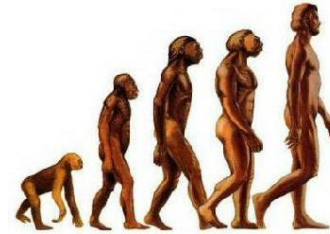
" Invertebrates
" Soil micro-organisms
" Vegetation
" Food

" Water organisms
" Groundwater and surface water

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Bioavailability tests (3)



” Pathways

- . Human, higher animals
 - ” Soil ingestion
 - ” Dermal contact
 - ” Inhalative intake
 - ” Groundwater (→ drinking water)
- . Soil organisms
 - ” See under human (depends on type of organism)
- . Plants
 - ” Uptake by roots
 - ” Systematic translocation
 - ” Uptake by the leaves
 - ” Adsorption on leaves through dust (soil the plant is growing in)
 - ” Deposition on leaves (diffuse sources)



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Bioavailability tests (4)

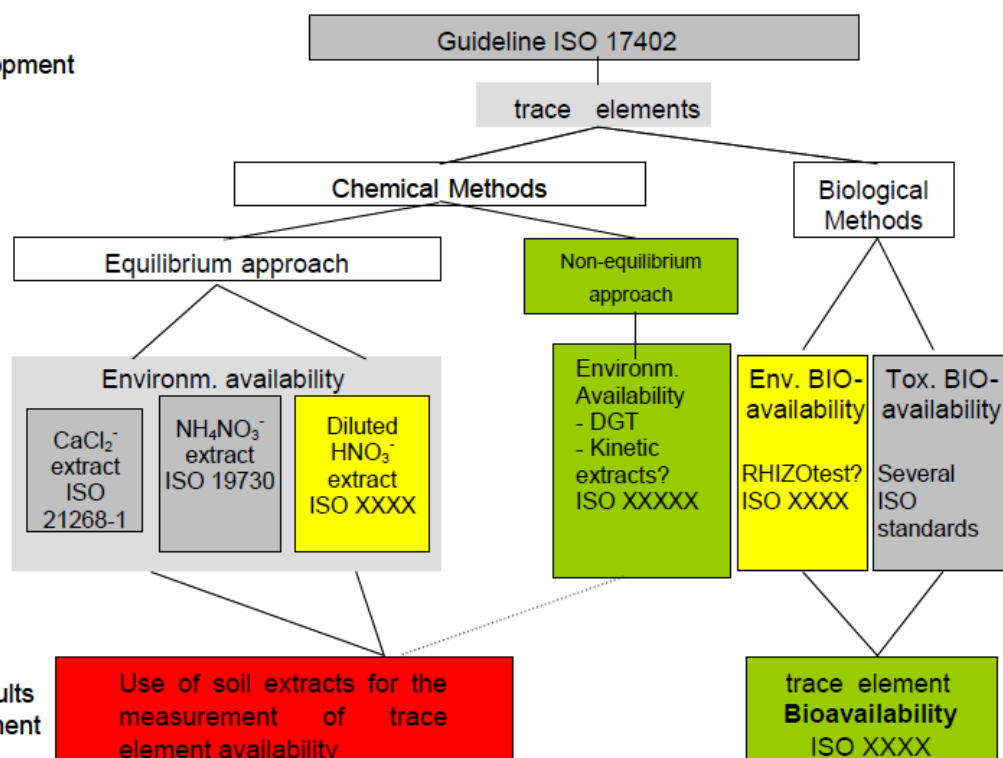
Examples of tests

- ISO/TS 17924: soil ingestion
- ISO 19730: ammonium nitrate extraction for plant uptake
- ISO 21268-1: CaCl_2 extraction, plant uptake
- Biodegradation test for organic contaminants: Tenax-, XAD-extraction, cyclodextrine method (no International standards yet; ISO is going to start with standards)

Selection
and development

Specific
Methods

Use of results
in assessment



Grey: ISO ready
Red/yellow: in development
Green: future development



Legislation

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Legislation (1)

“ EU (examples)

- . Landfill Directive
 - “ EN 12457-2 or -4 (shaking test), or EN 14405 (1st step of percolation test)
 - “ → in most countries EN 12457 is used (either part 2 or part 4)
- . Soil Directive
 - “ Drafts has been made. Very doubtful if it will ever come.
 - “ In draft: risk orientation related to contamination is mentioned
- . Water Framework Directive
 - “ Sediment
 - . Tenax extraction is mentioned as possibility
- . CPD, Construction Products Directive
 - “ Many tests are developed for assessing leaching behavior to the environment (water, air). This is done by CEN/TC 351.

Legislation (2)

“ France (examples)

- Landfill Directive implementation
 - “ Inert waste, non dangerous and dangerous granular waste: **EN 12457-2**.
 - “ Monolithic dangerous waste: **NF X31-211** (24 hr batch test)
- Reuse of materials for road building (under development), which should include different types of leaching tests (percolation, shaking test, surface test ò)

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Conclusions

- “ Leaching is more than just a shaking test+with water
- “ Leaching behavior depends on a lot of parameters, e.g.
 - . pH
 - . Temperature
 - . Type of leaching (shaking, percolation, stand+test)
 - . Time
- “ Leaching tests are used for assessing
 - . Risks of leaching of contaminants to the environment
 - . Risks for humans or ecosystem: bioavailability tests
- “ Future: leaching and bioavailability tests will be used more and more in order to assess risks related to contamination

Questions?



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