ALcontrol Laboratories

Leaching and EU Landfill Directive

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Who am I?

- Business Development Manager ALcontrol Laboratories

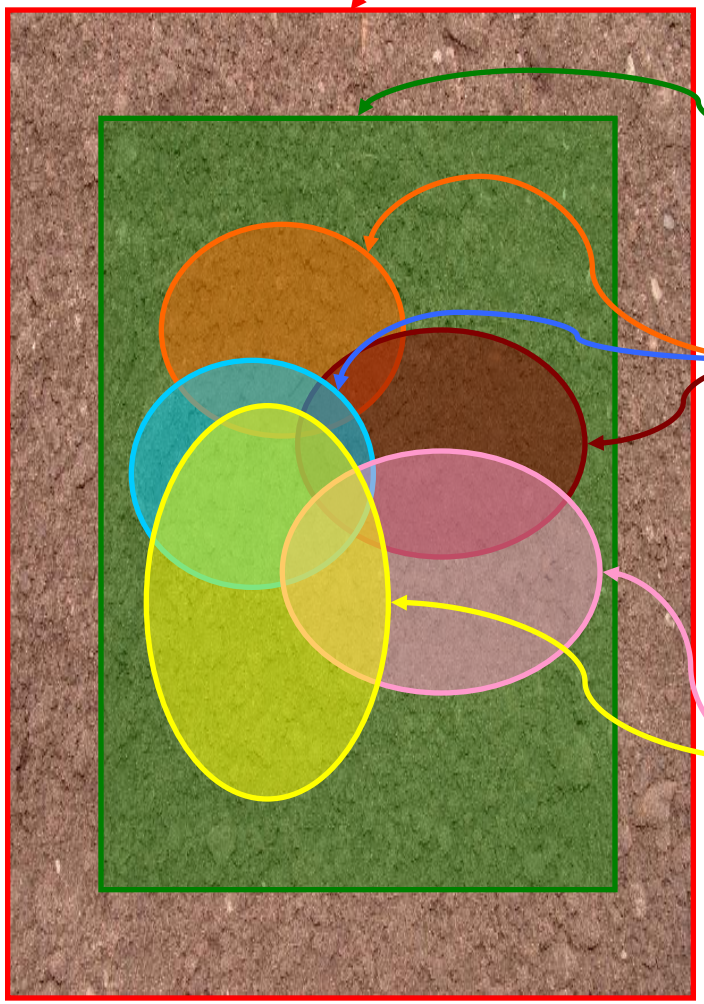
- Responsible for:
  - Special projects, e.g. for ministry Environmental Ministry NL (e.g. development of laboratory guidelines related to legislation)
  - National and international Committees (NEN, SIKB, CEN, ISO)
  - Assessment of and implementation of new analytical developments
  - FeNeLab (Dutch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

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

Overview types of analytical tests



Real Total concentrations

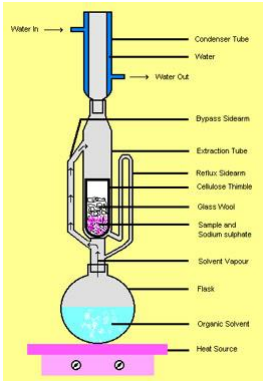
"So-called" Totals

Leachable fractions
- Stand tests
- Shaking tests
- Percolation tests
- ……

Bioavailable fractions
- Ammonium nitrate extraction
- 0,01 M CaCl2 extraction
- Tenax extraction
- …..

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 $HCl+HNO_3+HF$
  - 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!

So-called "Total extraction"

- Aim: get a good indication of "Real" total concentration
  - Aqua Regia ($HCl+HNO_{3)}$ 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 lab's in Europe
  - HNO_3 extraction: Nordic countries (Norway, Denmark, Sweden)
  - Shaking for semi-volatile organic compounds: all European countries except Belgium

Leaching tests

Leaching tests (1)

- Aims
  - Impact assessment
  - Compliance test

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

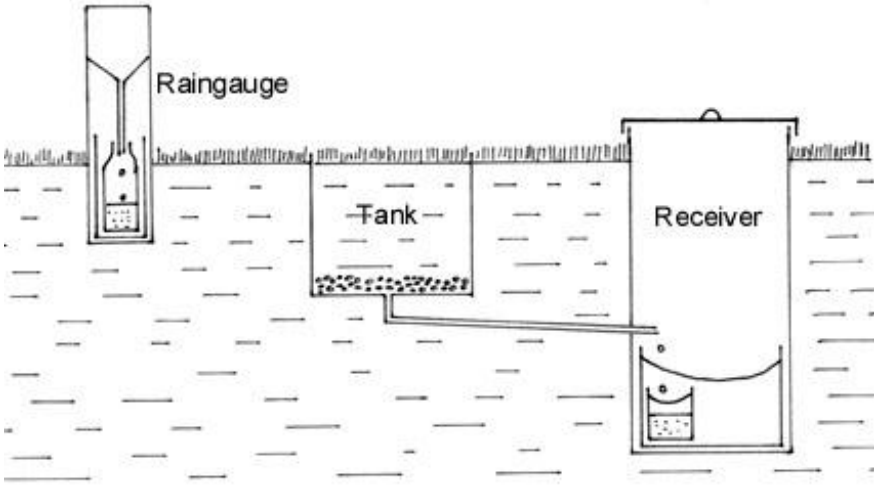


Fig 2. Lysimeter apparatus for measuring evapotranspiration

Examples of Leaching tests

Shaking tests, lixiviation (= 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

Column test, percolation (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

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 monolitic 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 $\pm$ 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

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

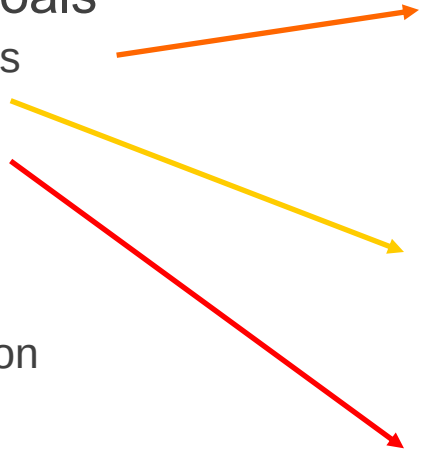
    exposure of organisms to the soil, or to soil eluates

    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



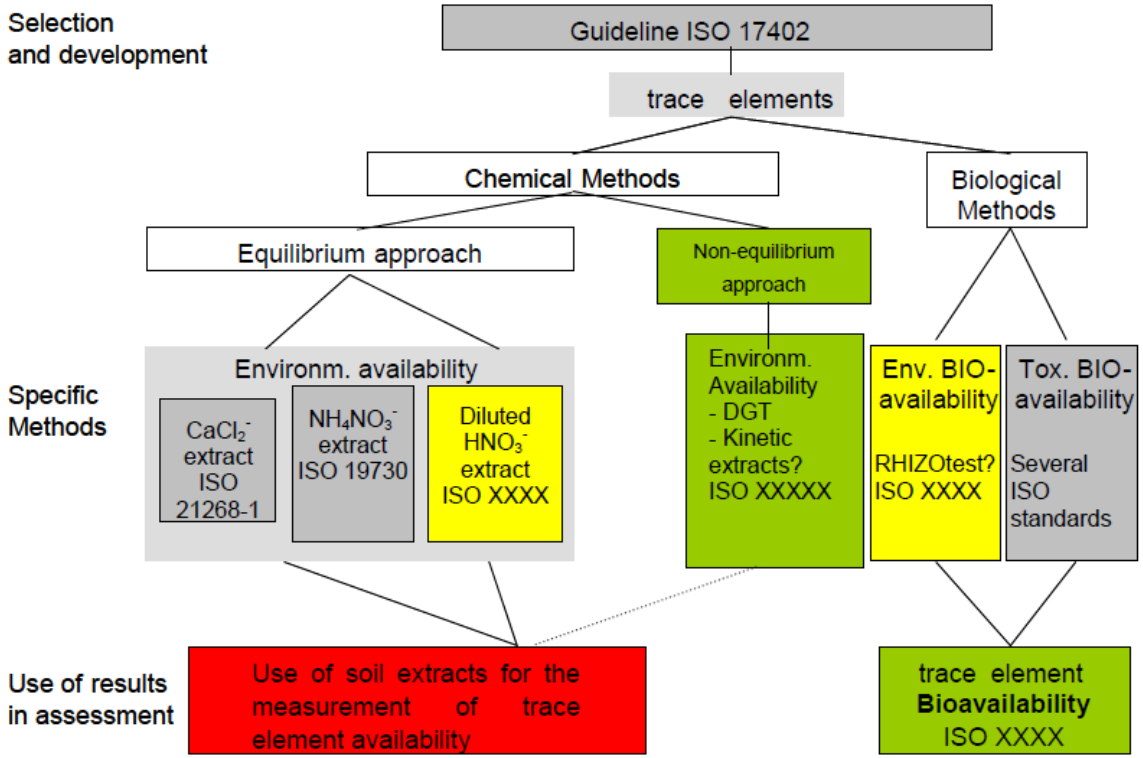
- Humans
- Grazing animals, e.g. cattle
- Wildlife

- Invertebrates
- Soil micro-organisms
- Vegetation
- Food

- Water organisms
- Groundwater and surface water

Bioavailability tests (3)

- Examples of tests
  - ISO/TS 17924: soil ingestion
  - ISO 19730: ammonium nitrate extraction for plant uptake
  - ISO 21268-1: CaCl2 extraction, plant uptake
  - Biodegradation test for organic contaminants: Tenax-, XAD-extraction, cyclodextrine method (no International standards yet; ISO has started with development of standards)



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

Legislation

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 (28/10/2010), non dangerous and dangerous granular waste: **EN 12457-2** and if necessary **EN 14405.**

    Monolithic dangerous waste: **NF X31-211** (24 hr batch test)
  - Reuse of materials for road building

    (SETRA guideline - published in 2011), which include different types of leaching tests (leaching test **EN12457-2**, percolation **EN 14405**)

Leaching and Landfill Directive

Regulation (1)

EU Directive

- 5 sorts of Landfills:
  - Inert waste,
    ➔ not in all EU countries (e.g. Netherlands)
  - Non-hazardous waste
  - Non-hazardous waste pursuant to Article 6(c)(iii)
  - Hazardous waste
  - Underground storage

Regulation (2)

- France
  - Each landfill must comply to local legislation/decision
  - Requirements are defined by the local authorities
  - Requirements are different per landfill according to its activities and geographic location
  - Requirements may differ from national legislation, mostly stricter
  - Local legislation prevails above national legislation
  - Enforcement by local environmental police (DREAL)

Compliance testing

Acceptance criteria, general remarks

- Up to **3 times higher** limit values are accepted in certain circumstances, but not for:
  - DOC (inert, non-hazardous, hazardous waste)
  - BTEX, PCB and mineral oil (inert waste)
  - TOC and pH (non-hazardous art. 6c (iii))
  - LOI and or TOC (hazardous waste, leaching)
  - TOC: **up to 2 times higher** acceptable for inert waste, leaching
- Acceptable if:
  - Permit is given by competent authority
  - Emissions for the landfill (including leaching) will present no additional risk, based on a risk assessment
  - This is reported to EU at intervals of 3 years
- Each EU member country may choose which leaching test to use
  - most countries use shaking test EN 12457 (faster than perculation test CEN/TS 14405)
  - France uses EN 12457-2 (< 4 mm), Netherlands 12457-4 (<10 mm)

Leaching Criteria in Landfill Directive

Criteria, Landfills for inert waste (1)

- Leaching

Compound	Inert material		
	LS=2	LS=10	LS=0.1
	Batch test	Batch test	(perculation test)
	mg/kgds	mg/kgds	mg/l
As	0.1	0.5	0.06
Ba	7	20	4
Cd	0.03	0.04	0.02
Cr	0.2	0.5	0.1
Cu	0.9	2	0.6
Hg	0.003	0.01	0.002
Mo	0.3	0.5	0.2
Ni	0.2	0.4	0.12
Pb	0.2	0.5	0.15
Sb	0.02	0.06	0.1
Se	0.06	0.1	0.04
Zn	2	4	1.2
Cl	550	800	460
F	4	10	2.5
SO4	560	1000	1500
Phenolindex	0.5	1	0.3
DOC	240	500	160
TDS	2500	4000	

Criteria, Landfills for inert waste (2)

Leaching (continued, remarks)

(*) **SO4:** If the waste does not meet these values for sulphate, it may still be considered as complying with the acceptance criteria if the leaching does not exceed either of the following values: 1 500 mg/l as C0 at L/S = 0,1 l/kg and 6 000 mg/kg at L/S = 10 l/kg. It will be necessary to use a percolation test to determine the limit value at L/S = 0,1 l/kg under initial equilibrium conditions, whereas the value at L/S = 10 l/kg maybe determined either by a batch leaching test or by a percolation test under conditions approaching local equilibrium.

(**) **DOC:** If the waste does not meet these values for DOC at its own pH value, it may alternatively be tested at L/S = 10 l/kg and a pH between 7,5 and 8,0. The waste maybe considered as complying with the acceptance criteria for DOC, if the result of this determination does not exceed 500 mg/kg. (A draft method based on prEN 14429 is available).

(***) **TDS:** The values for total dissolved solids (TDS) can be used alternatively to the values for sulphate and chloride.

Criteria, Landfills for non-hazardous waste (1)

- Leaching

Compound	Non-hazardous waste		
	LS=2	LS=10	LS=0.1
	Batch test	Batch test	(perculation test)
	mg/kgds	mg/kgds	mg/l
As	0.4	2	0.3
Ba	30	100	20
Cd	0.6	1	0.3
Cr	4	10	2.5
Cu	25	50	30
Hg	0.05	0.2	0.03
Mo	5	10	3.5
Ni	5	10	3
Pb	5	10	3
Sb	0.2	0.7	0.15
Se	0.3	0.5	0.2
Zn	25	50	15
Cl	10000	15000	85000
F	60	150	40
SO4	10000	20000	7000
Phenolindex			
DOC	380	800	250
TDS	40000	60000	

Criteria, Landfills for non-hazardous waste (2)

- Leaching, remarks

(*) **DOC:** If the waste does not meet these values for DOC at its own pH, it may alternatively be tested at L/S = 10 l/kg and a pH of 7,5-8,0. The waste maybe considered as complying with the acceptance criteria for DOC, if the result of this determination does not exceed 800 mg/kg (A draft method based on prEN 14429 is available).

(**) **TDS:** The values for TDS can be used alternatively to the values for sulphate and chloride

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Criteria, Landfills for non-hazardous waste , pursuant to art. 6c (iii) (1)

- Leaching and content

Compound	Non-hazardous waste, art. 6c (iii)			
	Content	LS=2	LS=10	LS=0.1
		Batch test	Batch test	(perculation test)
		mg/kgds	mg/kgds	mg/l
As		0.4	2	0.3
Ba		30	100	20
Cd		0.6	1	0.3
Cr		4	10	2.5
Cu		25	50	30
Hg		0.05	0.2	0.03
Mo		5	10	3.5
Ni		5	10	3
Pb		5	10	3
Sb		0.2	0.7	0.15
Se		0.3	0.5	0.2
Zn		25	50	15
Cl		10000	15000	85000
F		60	150	40
SO4		10000	20000	7000
Phenolindex				
DOC		380	800	250
TDS		40000	60000	
LOI				
TOC	50 g/kgds			
pH	> 6			
ANC	***)			

Criteria, Landfills for non-hazardous waste , pursuant to art. 6c (iii) (2)

Leaching (continued, remarks)

(*) **DOC:** If the waste does not meet these values for DOC at its own pH, it may alternatively be tested at L/S = 10 l/kg and a pH of 7,5-8,0. The waste maybe considered as complying with the acceptance criteria for DOC, if the result of this determination does not exceed 800 mg/kg (A draft method based on prEN 14429 is available).

(**) **TDS:** The values for TDS can be used alternatively to the values for sulphate and chloride.

(***) **ANC:** has to be measured, no target value

Criteria, Landfills for hazardous waste (1)

– Leaching and content

Compound	Dangerous waste			
	Content	LS=2	LS=10	LS=0.1
		Batch test	Batch test	(perculation test)
		mg/kgds	mg/kgds	mg/l
As		6	25	3
Ba		100	300	60
Cd		3	5	1.7
Cr		25	70	15
Cu		50	100	60
Hg		0.5	2	0.3
Mo		20	30	10
Ni		20	40	12
Pb		25	50	15
Sb		2	5	1
Se		4	7	3
Zn		90	200	60
Cl		17000	25000	15000
F		200	500	120
SO4		25000	50000	17000
Phenolindex				
DOC		480	1000	320
TDS		70000	100000	
LOI	10% opds			
TOC	60 g/kgds			
pH				
ANC	***)			

Criteria, Landfills for hazardous waste (2)

Leaching (continued, remarks)

(*) **DOC:** If the waste does not meet these values for DOC at its own pH, it may alternatively be tested at L/S = 10 l/kg and a pH of 7,5-8,0. The waste maybe considered as complying with the acceptance criteria for DOC, if the result of this determination does not exceed 1 000 mg/kg. (A draft method based on prEN 14429 is available.)

(**) **TDS:** The values for TDS can be used alternatively to the values for sulphate and chloride.

(***) **LOI / TOC:** either LOI (Loss On Ignition) or TOC must be used.

(****) **TOC:** If this value is not achieved, a higher limit value may be admitted by the competent authority, provided that the DOC value of 1 000 mg/kg is achieved at L/S = 10 l/kg, either at the material's own pH or at a pH value between 7,5 and 8,0.

Conclusions

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?