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Background
Two ECETOC reports:
• TR 117: Understanding the Relationship between Extraction Technique and Bioavailability;
• TR 118: Development of interim guidance for the inclusion of non-extractable residues (NER) in the risk assessment of chemicals.
At the moment NER is considered either as a chemical time bomb or as a sustainable detoxification.

Objective
• Validate the extraction regime of ECETOC TR 117.
• Develop a “soup test” to assess the toxicity of the different fractions of chemicals.
• Provide a clear, mechanistically driven definition to NER and residual toxicity caused by NER.

Conceptual approach
In our approach a contaminant is adsorbed by the soil. At time zero all of the contaminant is bioavailable and may cause risks. All of the contaminant is in equilibrium with the water phase. In time, the contaminant is distributed over different sorption fractions. Biodegradation may be responsible for a decrease of the contaminant is distributed over different sorption fractions. Some fractions are measurable, some are not.

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Desorption rates are much larger in a slurry test system, compared to an unsaturated soil.

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Slurry system (test method)</th>
<th>Unsaturated system (soil)</th>
<th>Method to measure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dissolved/activity</td>
<td>0</td>
<td>DMPE or SPE, 0.01 M CaCl₂</td>
<td></td>
</tr>
<tr>
<td>Rapidly desorbing</td>
<td>1 day</td>
<td>2600</td>
<td>Microtox EC50: 40 mg/kg</td>
</tr>
<tr>
<td>Slowly desorbing</td>
<td>weeks</td>
<td>Months to years</td>
<td>No effect</td>
</tr>
<tr>
<td>Very slowly desorbing</td>
<td>months to years</td>
<td>0.1-0.3 decades</td>
<td>No effect</td>
</tr>
<tr>
<td>Irreversibly sorbed</td>
<td>years to decades</td>
<td>&lt;0.01</td>
<td>No effect</td>
</tr>
</tbody>
</table>

We define the desorption by a sum of three first order decreases.

\[
\frac{dx}{dt} = F_{\text{rapid}} e^{-kt} + F_{\text{slow}} e^{-kt} + F_{\text{irr}} e^{-kt}
\]

(Cornelissen et al., 1997, Harmsen, 2004. ISO 17402 and Rhodes et al., 2010).

In the mathematical model no non-desorbing fraction is defined.

We translate the research results into practical and explainable recommendations for regulators.

Contamination left can be measured using radiolabelled compounds; Bioassays react on the bioavailable fraction.

Selection of test chemicals
Criteria: (1) occurrence in soil and/or sediment, (2) formation of NER, (3) relevant effect on benthic and/or soil organisms, (4) sufficient analytical accuracy for different extraction methods, (5) known biodegradation and metabolism in soils, (6) variety of chemistry, (7) availability of 14C radiolabelled compound. The candidate test chemicals, Trinitrotoluene TNT, Benzo(a)pyrene, Cypermethrin and Carbendazim can be measured sensitive, using LC/MS.

A proper bioassay is still under investigation.

The way to go
• C-14 studies to assess partitioning over different fractions of soil;
• Comparison of C-14 study results to standard chemical analysis of the measurable fractions;
• Measurement of formation rate of NER;
• Use of suitable bioassays to follow toxicity in extracts AND extracted soils.
• Translate the research results into practical and explainable recommendations for regulators.

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• Tenax/cyclodextrine can be used to measure \( F_{\text{rapid}} \);
• An exhaustive extraction (standard method), to measure total, measures the sum of the rapidly, slowly and very slowly desorbing fractions.