The Chemical Monitoring Activity Under the Water Framework Directive (WFD) – Legal Framework and Analytical Challenges

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What’s new in the WFD?

- Protection of aquatic ecosystems
- Objective by 2015:
  - Good status (ecological and chemical)
  - No deterioration principle
- Water management based on river basin districts
- Environmental quality standards and emission controls
- „Phasing out“ of priority hazardous substances
- Use of economic instruments
- Public participation
- Integration of other directives related to water issues
Implementation Schedule for WFD

Scope of the Chemical Monitoring

- All surface waters
  - Rivers, lakes, and artificial waters
  - Transitional waters
  - Coastal waters up to one sea mile
  - For chemical status, also territorial waters, which may extend to 12 sea miles from the territorial baseline of a Member State

- Groundwater
Types of Chemical Monitoring

- Surveillance
- Operational
- Investigative
- Protected areas

What to Monitor?

- Priority Substances – Compliance with European Environmental Quality Standards (EQS)
- Other pollutants (relevant at river basin level) – Compliance with national EQS
- Physico-chemical parameters supporting interpretation of biological data
- Parameters required for interpretation of the results of chemical measurements (*e.g.*, DOC, Ca, SPM)
Chemical Monitoring – Status Assessment

- Good Status Surface Water
- Good Chemical Status
- Good Ecological Status/Potential
- Priority Substances
- “Other Pollutants”
- Biological Quality Elements
- Hydromorphological Quality Elements
- Physico-chemical Parameters

Frequency of Monitoring

- Minimum given for all quality elements in WFD
- More frequent may be necessary (e.g., to detect long term changes or to estimate pollution loads)
- Less frequent if justifiable
- Seasonally targeted
- Achievement of acceptable levels of confidence and precision in assessing status of water bodies
  - Balance between costs of monitoring and costs of measures
**Frequency of Monitoring**

<table>
<thead>
<tr>
<th>Physico-Chemical Quality Elements</th>
<th>Rivers</th>
<th>Lakes</th>
<th>Transitional</th>
<th>Coastal</th>
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</thead>
<tbody>
<tr>
<td>Thermal Conditions</td>
<td>3 months</td>
<td>3 months</td>
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<td>Oxygenation</td>
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<td>Salinity</td>
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<td>Nutrient status</td>
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<td>Acidification status</td>
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<tr>
<td>Other pollutants</td>
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<td>3 months</td>
<td>3 months</td>
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<tr>
<td>Priority substances</td>
<td>1 month</td>
<td>1 month</td>
<td>1 month</td>
<td>1 month</td>
</tr>
</tbody>
</table>

Less frequent if justifiable (e.g., biota, sediment)

- Biota: once a year
- Sediment: once a year up to once every six years

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**Annex X – Priority Substances**

**DECISION 2455/2001/EC**

**List of Priority Substances (33)**

- **Priority Substances**
  - Present a significant risk to or via the aquatic environment

- **Priority Hazardous Substances**
  - Subset of priority substances, which are toxic, persistent, liable to bioaccumulate or give rise to equivalent level of concern
Annex X – Priority Substances

- **Priority substances**
  - Progressive reduction of discharges, emissions and losses

- **Priority hazardous substances (PHS)**
  - Cessation of discharges, emissions and losses or phasing-out by 2020

### Priority Substances/Other Pollutants

<table>
<thead>
<tr>
<th>Priority Hazardous Substances</th>
<th>Priority Substances</th>
<th>Other Specific Pollutants</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anthracene</td>
<td>Alachlor</td>
<td>DDT / p,p'-DDT</td>
</tr>
<tr>
<td>Pentabromodiphenylether</td>
<td>Atrazine</td>
<td>Aldrin</td>
</tr>
<tr>
<td>Cadmium and its compounds</td>
<td>Benzene</td>
<td>Dieldrin</td>
</tr>
<tr>
<td>C10-C13-Chloroalkanes</td>
<td>Chlorpyrifos</td>
<td>Endrin</td>
</tr>
<tr>
<td>Endosulfan</td>
<td>Chlorfenvinphos</td>
<td>Isodrin</td>
</tr>
<tr>
<td>Hexachlorobenzene</td>
<td>1,2-Dichloroethane</td>
<td>Carbontetrachloride</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>Dichloromethane</td>
<td>Tetrachloroethylene</td>
</tr>
<tr>
<td>Hexachlorocyclohexane</td>
<td>Di(2-ethylhexyl)phthalate (DEHP)</td>
<td>Trichloroethylene</td>
</tr>
<tr>
<td>Mercury and its compounds</td>
<td>Duran</td>
<td></td>
</tr>
<tr>
<td>Nonylphenol</td>
<td>Fluoranthene</td>
<td></td>
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<tr>
<td>Pentachlorobenzene</td>
<td>Isoproton</td>
<td></td>
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<tr>
<td>Polyaromatic Hydrocarbons (PAH)</td>
<td>Lead and its compounds</td>
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<tr>
<td>Tributyltin compounds</td>
<td>Naphthalene</td>
<td></td>
</tr>
<tr>
<td>Nickel and its compounds</td>
<td>Octylphenol</td>
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<tr>
<td>Pentachlorophenol</td>
<td>Simazine</td>
<td></td>
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<tr>
<td>Trichlorobenzenes</td>
<td>Trichloromethane</td>
<td></td>
</tr>
<tr>
<td>Trifluralin</td>
<td></td>
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</tr>
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</table>
Environmental Quality Standards (EQS)

- At this stage, only for surface water except for HCB, HCBD and Hg
- Eco-toxicological assessment according to the TGD on Risk Assessment of Chemicals
- Rule of thumb: Lowest NOEC*0,1
- AA-EQS
  - Environmental quality standard expressed as annual average concentration
- MAC-EQS
  - Environmental quality standard expressed as maximum allowable concentrations
- EQS may be different for inland and other surface waters

Compliance with EQS

- AA-EQS
  - For each representative point within the water body, the arithmetic mean of the concentrations measured at different times during the year is below the EQS
- MAC-EQS
  - The measured concentration at any representative monitoring point within the water body measured at any time must not exceed the EQS

- With the exception of metals, EQS refer to total concentration in the whole water samples
- In the case of metals, the EQS refer to the dissolved concentration
- The Directive does not specify decision rules on how to perform compliance checking
### Environmental Quality Standards

#### Inland Surface Waters

<table>
<thead>
<tr>
<th>No.</th>
<th>Priority Substance</th>
<th>AA-EGS (µg/L)</th>
<th>MAO-EGS (µg/L)</th>
<th>AA-EGS (µg/L)</th>
<th>MAO-EGS (µg/L)</th>
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<tr>
<td>(1)</td>
<td>Alachlor</td>
<td>0.3</td>
<td>0.7</td>
<td>0.3</td>
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<tr>
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<td>Atrazin</td>
<td>0.6</td>
<td>2</td>
<td>0.6</td>
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<tr>
<td>(4)</td>
<td>Benzene</td>
<td>10</td>
<td>50</td>
<td>8</td>
<td>50</td>
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<tr>
<td>(5)</td>
<td>Pentabromodiphenylether</td>
<td>0.0005</td>
<td>not applicable</td>
<td>0.0002</td>
<td>not applicable</td>
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<tr>
<td>(6)</td>
<td>Cadmium and its compounds</td>
<td>0.08-0.2</td>
<td>0.45-1.5</td>
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<tr>
<td>(7)</td>
<td>C10-C13 Chloroalkanes</td>
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<td>1.4</td>
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<td>0.1</td>
<td>0.3</td>
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<td>Chlorpyrifos</td>
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<td>0.03</td>
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<td>1,2-Dichloromethane</td>
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<td>10</td>
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<td>(11)</td>
<td>Dichloromethane</td>
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<td>(12)</td>
<td>Di(2-ethylhexyl)phthalate (DEHP)</td>
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<td>1.3</td>
<td>not applicable</td>
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<td>(13)</td>
<td>Dieldrin</td>
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<td>0.04</td>
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<td>Isoproturon</td>
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<td>1.0</td>
<td>0.3</td>
<td>1.0</td>
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<td>(20)</td>
<td>Lead and its compounds</td>
<td>7.2</td>
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<td>7.2</td>
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#### Other Surface Waters

<table>
<thead>
<tr>
<th>No.</th>
<th>Priority Substance</th>
<th>AA-EGS (µg/L)</th>
<th>MAO-EGS (µg/L)</th>
<th>AA-EGS (µg/L)</th>
<th>MAO-EGS (µg/L)</th>
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<tbody>
<tr>
<td>(21)</td>
<td>Mercury and its compounds</td>
<td>0.05</td>
<td>0.07</td>
<td>0.05</td>
<td>0.07</td>
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<td>(22)</td>
<td>Naphthalene</td>
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<td>(23)</td>
<td>Nickel and its compounds</td>
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<td>20</td>
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<td>Nonylphenol</td>
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<td>0.3</td>
<td>2</td>
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<tr>
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<td>Octylphenol</td>
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<td>0.01</td>
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<td>(26)</td>
<td>Pentachlorobenzene</td>
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<td>0.0007</td>
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<tr>
<td>(27)</td>
<td>Pentachlorophenol</td>
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<td>1</td>
<td>0.4</td>
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<td>Polycyclic Hydrocarbons (PAH)</td>
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<td>0.05</td>
<td>0.1</td>
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<td>Simazine</td>
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<td>(30)</td>
<td>Tributyltin compounds</td>
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<td>0.0001</td>
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<td>0.0001</td>
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<td>(31)</td>
<td>Trichlorobenzene</td>
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<td>Trichloromethane</td>
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<td>(33)</td>
<td>Trifluralin</td>
<td>0.03</td>
<td>not applicable</td>
<td>0.03</td>
<td>not applicable</td>
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Environmental Quality Standards

<table>
<thead>
<tr>
<th>No</th>
<th>Other Pollutants</th>
<th>Inland Surface Waters</th>
<th>Other Surface Waters</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>AA-EQS (µg/L)</td>
<td>MAQ-EQS (µg/L)</td>
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<tr>
<td>(1)</td>
<td>DDT total</td>
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<td>p,p’ DDT</td>
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<tr>
<td>(2)</td>
<td>Aldrin</td>
<td>Σ&lt;0.01</td>
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<tr>
<td>(3)</td>
<td>Dieldrin</td>
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<td>Σ&lt;0.005</td>
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<tr>
<td>(4)</td>
<td>Endrin</td>
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<td>not applicable</td>
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<tr>
<td>(5)</td>
<td>Isodrin</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(6)</td>
<td>Carbon tetrachloride</td>
<td>12</td>
<td>not applicable</td>
</tr>
<tr>
<td>(7)</td>
<td>Tetrachloroethylene</td>
<td>10</td>
<td>not applicable</td>
</tr>
<tr>
<td>(8)</td>
<td>Trichloroethylene</td>
<td>10</td>
<td>not applicable</td>
</tr>
</tbody>
</table>

What Matrix is Best Suited for WFD Monitoring?

Selection of matrix depends on

- Physico-chemical properties of the substance to be analysed
  - Solubility in water, log K<sub>sw</sub>, K<sub>obs</sub>, BCF
- Properties of the water body to be assessed
  - pH, SPM content, f<sub>oct</sub>, K<sub>o</sub>
- Objective of the monitoring
  - Compliance with EQS
  - Assessment of long term effects of anthropogenic activity
  - Temporal trends
  - No deterioration principle
  - Estimation of pollution loads
  - .........................
Monitoring in Water

- **Metals**
  - Filtered water sample (dissolved fraction)
    - Natural background
    - Bioavailability (DOC, pH, hardness etc.)

- **Organic compounds**
  - Results have to be referred to whole water
    - Analysis of whole water samples
    - Separate analysis of solid particulate matter (SPM) and the liquid phase
    - SPM or the filtered water samples may be used as substitute for the whole water sample if justifiable

Monitoring in Biota and Sediment

- Monitoring in biota compulsory for mercury, HCB, and hexachlorobutadiene

- The following concentrations in prey tissue must not be exceeded
  - Mercury 20 µg/kg w.w.
  - HCB 10 µg/kg w.w.
  - Hexachlorobutadiene 55 µg/kg w.w.

**Instead of checking compliance with biota EQS**
- Member States may set up a more stringent EQS for water (replacing the one suggested by the Commission)
- Providing the same level of protection as the biota standard
Monitoring in Biota and Sediment

- **EQS for sediment and/or biota possible**
  - Apply them instead of EQS for water
  - Derivation at national level
  - Notifications to the Commission and other Member States
  - Level of protection equivalent to the EQS for water

- **Long-term trends to be monitored in biota and sediment**
  - Substances listed in Directive 2008/105/EC with significant accumulation potential
  - Ensuring that concentrations do not significantly increase in sediment and/or relevant biota

Group Parameters

- **Chlorpyrifos (CAS number 2921-88-2)**
  - Chlorpyrifos-ethyl

- **Pentabromodiphenyl Ether**
  - BDE28, BDE47, BDE99, BDE100, BDE153, BDE154

- **Total DDT**
  - p,p'-DDT, o,p'-DDT, p,p'-DDE, p,p'-DDD

- **Endosulfan (CAS number 115-29-7)**
  - α- and β-endosulfan

- **Hexachlorocyclohexane (CAS number 608-73-1)**
  - α-, β-, γ- and δ-hexachlorocyclohexane
**Group Parameters**

- **Nonylphenol** (CAS number 104-40-5)
  - 4-nonylphenol isomers
- **Octylphenol** (CAS number 140-66-9)
  - *para-tert*-Octylphenol
- **Trichlorobenzenes**
  - 1,2,4-, 1,3,5-, and 1,2,3- trichlorobenzene

**Priority Substances Difficult to Analyse**

- **Organochlorine pesticides**
  - Sensitivity insufficient for cyclodiene pesticides, endosulfane, and pentachlorobenzene
  - Difficulties in meeting required LoQ for DDT, hexachlorocyclohexane and hexachlorobenzene

- **Polycyclic Aromatic Hydrocarbons**
  - Sensitivity insufficient for Indeno[1,2,3-cd]pyrene and benzo[ghi]perylene compounds as the LoQ
  - Difficulties in meeting required LoQ for benzo[k]fluoranthene and benzo[b]fluoranthene

- **Tributyl tin compounds**
  - Requirements on LOQ impossible to achieve since proposed EQS is 0.2 ng/L
  - Lowest reported LoQ was 1 ng/L
Priority Substances Difficult to Analyse

- Pentabromodiphenylether
  - No standardised method for water available
  - Requirements on LoQ hard to meet
    - the sum concentration of 6 congeners has to be below 0.5 ng/L and 0.2 ng/L, respectively
  - ISO 22032 for Analysis of PBDE in Sediments

- Short-chain chlorinated paraffins
  - No standard method for water available
  - Analysis not under control also in research laboratories
  - Most frequently applied method is GC-ECNI-MS
    - Unsolved Problems:
      - Calibration, dependency of response on degree of chlorination
      - Isomers with less than five chlorine cannot be detected


Substances subject to review for possible identification as priority or priority hazardous substances

- AMPA
- Bentazon
- Bisphenol-A
- Dicofol
- EDTA
- Free Cyanide
- Glyphosate
- Mecoprop (MCPP)
- Musk xylene
- Perfluoroctane sulphonic acid (PFOS)
- Quinoxyfen (5,7-dichloro-4-(p-fluorophenoxy)quinoline)
- Dioxins
- PCB

The Commission shall report outcome of the review to the EP and to the Council by 13 January 2011
Commission Directive – Objectives

- **Provision of data of proper scientific quality**
  - Basis for programme of measures

- **Comparability of monitoring results across Europe**
  - Implementation of common principles and harmonised procedures for chemical monitoring

What to be Specified?

- Subject matter
- Analytical methods
- Minimum performance criteria on analytical methods
- Requirements on laboratories involved in chemical monitoring including sampling
- Requirements on quality assurance and quality control
- Responsibilities

- **Standardised and other validated methods**
  - Any method provided it is properly validated and meets certain performance criteria may be applied

- **Requirements on analytical methods**
  - Validation according to EN ISO/IEC 17025
    - **Limit of Quantification**
      - ≤ 30% of the relevant EQS
    - **Relative target uncertainty at EQS level**
      - ≤ 60%
  - If there is no EQS or no method that meets the performance criteria
    - ‘Best available technique’


- **Quality assurance and quality control**
  - Quality management system according to EN ISO/IEC 17025
  - Demonstration of competence by
    - Participation in laboratory proficiency testing schemes
    - Analysis of appropriate reference materials
  - **Proficiency tests**
    - Accredited or internationally or nationally recognised organisations
    - Meeting the requirements of ISO/IEC 43-1
    - Evaluation according to ISO13588 or ISO/IEC 43-1 (z-scores)

- Data below the limit of quantification (LoQ)
  - Results below LoQ shall be set half of the value of LoQ when calculating annual average concentrations
  - If annual average below LoQ
    - to be reported as “less than LoQ”
  - Does not apply to sum concentrations, e.g. PBDE
    - For individual substances/isomers, results below LoQ are set zero before calculating sum concentrations

Chemical Monitoring Activity

- Chemical Monitoring Activity
  - One representative from each Member State, stakeholders

- Objective
  - Support MS in implementing WFD

- Three Working Groups
  - Exchange of best practices
  - QA/QC
  - Standardisation/Research
Chemical Monitoring Activity

STRATEGIC CO-ORDINATION GROUP

WG E

CMA Plenary

CMA-1
Exchange of best practices

CMA-2
Quality Assurance/Quality Control

CMA-3
Standardisation/Research Needs

- Chemical Monitoring Guidance
- On-site workshops (Field trial)
- Sediment and Biota Guidance
- Follow-up of COM Directive
- QA/QC strategy (EAQC-WISE)
- New Methods
- Mandate M424
- Links to DG Res

CMA On-Site 2

2nd Field Trial, Budapest, September 17/18, 2008

- Objective
  - Comparison of monitoring methods (sampling and analysis) among Member State laboratories

- Target compounds
  - PAH
  - PBDE
  - Alkylphenols

- Participants
  - 18 laboratories
  - 12 countries

- Organisation
  - JRC-IES
CMA On-Site 2

- **Comparison at three levels**
  - Riverwater samples (whole procedure including sampling)
  - River water extract (measurement including natural sample matrix)
  - Standard solution of undisclosed concentration (instrumental performance, calibration)

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**Sampling Scheme CMA On-Site 2**

<table>
<thead>
<tr>
<th>Time</th>
<th>In-situ Probe, temperature, turbidity</th>
<th>SPM gravimetric</th>
<th>DOC/POC</th>
<th>PAH/PBDE homogeneity sampling</th>
<th>Alkylphenol homogeneity sampling</th>
<th>PAH/PBDE sampling (filtration/adsorption)</th>
<th>Large volume sampling (centrifuge)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9:00</td>
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Chemical Monitoring of Surface Water

Guidance Document No. 19
Guidance on Surface Water Chemical Monitoring
Final Draft


Mandate 424

- Mandate 424 addressed to CEN to develop or improve methods to support WFD monitoring

Requirements/problems to solve
- Analysis of whole water samples
  - The method should enable the analysis of samples containing up to 0.5 g/L of suspended solids
- LOQ equal or less than 30% EQS
- Measurement uncertainty equal or less than 50%
- Method should be fully in-house validated and tested for ruggedness prior to inter-lab validation
- Validation by international inter-comparison according to ISO 5725
## Mandate 424

### Substance groups included in the mandate

- **Organochlorine pesticides**
  - Alachlor, endosulfan, HCB, HCH isomers, pentachlorobenzene, DDT and metabolites, aldrin, dieldrin, endrin, and isodrin
- **PBDE**
  - Indicator congeners BDE28, BDE47, BDE99, BDE100, BDE153, BDE154
- **PAHs**
  - Benzo(k)fluoranthene, benzo(b)fluoran-thene,
  - indeno(1,2,3-cd)pyrene and benzo(ghi)perylene
- **TBT**
- **SCCP**

## Mandate 424

### Status of the mandate

- The mandate M/424 was adopted by CEN/TC/230 after a formal enquiry in June 2009
- Call for tender was launched at the CEN webpage (July 2008)
- Some 20 proposal have been submitted
- Evaluation by an independent expert group on September 22, 2009
- Consortium meeting was held on November 14, 2009
- WP leader meeting was held on January 16, 2009
- Final DoW is expected to be submitted via CEN CS to DG ENTR in February, 2009
Information on the European Water Framework Directive

http://circa.europa.eu/Public/irc/env/wfd/library


Needs for reliable analytical methods for monitoring chemical pollutants in surface water under the European Water Framework Directive