The water is not too deep:

Our approach to implementing new demands for analysing environmental contaminants in the laboratory.

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National Laboratory Service
Product Manager
Identifying, buying & developing…

BEST AVAILABLE TECHNOLOGY

(Not Entailing Excessive Cost)
Background

Directives

Analytical gap analysis

Funding, Procurement Process including Tendering

Method Development

What next
3 Laboratories:

Solids

Waste Water

Clean Waters

Water Quality

Instrumentation Facility - Reading

Environmental Quality Standards (Directive 2008/105/EC) (EQSD) and 2013 revision


UKTAG CTT guidance on QAQC Directive
• The Limit of Quantification (LoQ) must $\leq \frac{1}{3}$ of the Environmental Quality Standard (EQS).

• As the LoQ is a function of the Limit of Detection (LoD), i.e. $10 \times \text{xsd}$ or $2.15 \times \text{LoD}$, the LoD will need to be $\leq \frac{1}{7}$ EQS.
EQS for B(α)P in water is 0.00017µg/l so LoD = ~0.00002µg/l

EQS for Cypermethrin is 0.00008µg/l so LoD = ~0.00001µg/l

Precision (2x%RSD) at EQS must be ≤ 50%

Method must be demonstrated to be in control at the LoQ
Alconifen
Alachlor
Bifenox
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(ghi)perylene
Benzo(k)Fluoranthene
Fluoranthene
Oestrone
17-alpha-ethinylstradiol
17-beta-estradiol

Surface (fresh) waters

Triclosan
Heptachlor
Heptachlor Epoxide
Hexachlorocyclohexane
Methiocarb
Pentachlorobenzene
PFOS & PFOA
Terbutryn
Tributyl tin chloride
Tri-phenyl tin
Cr VI
Toxic 6 metals

Biota (Mytilus, crayfish & fish)

Saline (TRAC) waters
Cypermethrin

1 ng\textsuperscript{-L} \rightarrow 0.01 ng\textsuperscript{-L}

Benzo(α)pyrene

1 ng\textsuperscript{-L} \rightarrow 0.02 ng\textsuperscript{-L}
PFOS
5ng\textsuperscript{-L} \rightarrow 0.09ng\textsuperscript{-L}

HBCDD
n/a \rightarrow 0.2ng\textsuperscript{-L}
HBCDD in biota

n/a \rightarrow 24 \mu g^{-kg}

Dicofol in biota

n/a \rightarrow 5 \mu g^{-kg}
Heptachlor in water

1 ng\textsuperscript{-L} \rightarrow 0.00008 ng\textsuperscript{-L}

Heptachlor in biota

n/a \rightarrow 0.001 \mu g\textsuperscript{-kg}
### Workloads:

- **PAH’s**: 800 spm
- **Organo tins**: 600 spm
- **Cypermethrin**: 300 spm
- **Nonylphenols & Triclosan**: 600 spm
- **Misc Pesticides**: 600 spm
- **PFOS & PFOA**: 100 spm
- **Biota (various test)**: 250 pa

Sampling workloads renegotiated every year based on govt. funding.
and a sceptical customer!

Are you sure?

We’ve heard it can’t be done!

How are Europe doing it?

How much?

Will it be BAT?
| Substance | CAS Number | Unit | Threshold Limit Value | Permissive Ceiling Value | Recommended Limit Value | Biological Monitoring Guideline Value | Standard | Comment |
|-----------|------------|------|-----------------------|--------------------------|-------------------------|--------------------------------------|---------|
| Arsenic   | 7440-38-2  | mg/m³ | 0.00034                 | 0.0017                   | 1.000                  | 1.600                  | Yes     | 400 ppm |
| Benzene   | 71-43-2    | mg/m³ | 0.0031                   | 0.0089                   | 0.060                   | 0.060                   | No      | 200 ppm |
| Chloroform | 67-66-3  | mg/m³ | 0.00034                 | 0.0017                   | 1.000                  | 1.600                  | No      | 400 ppm |
| Ethylbenzene | 108-88-3 | mg/m³ | 0.0031                   | 0.0089                   | 0.060                   | 0.060                   | No      | 200 ppm |
| Toluene   | 108-88-3   | mg/m³ | 0.0031                   | 0.0089                   | 0.060                   | 0.060                   | No      | 200 ppm |
| Xylenes   | 106-42-3   | mg/m³ | 0.0031                   | 0.0089                   | 0.060                   | 0.060                   | No      | 200 ppm |

*Note: Table continues with more substances and values.*
A sceptical supplier(s)

We don’t know

Real world detection limits

Not used to comparing instrument to Directives

Whole method solutions

Not been done before

Be aware of false promises
Evaluating new instrument

• Price 50%
• Quality 50%
• BAT?
• LoD equivalent solutions trialled before awarding contract
• Install and “sign off” purchase
Monitoring and Budgetary cycles

- 2019 all water bodies and TRaC waters – “Good Chemical Status”
- Monitoring to start April 2014 – conceptual gap in capability
- No money assigned to development including equipment
- April 2014 - Start development in existing equipment (Nonyl / Octyl Phenol and Biota)
- April 2014 - start procurement / tender process
- June 2014 - reassign limited money and prioritise PAH development
- November 2014 award contract to instrument companies and start buying equipment. 2xGCQQQQ
Monitoring and Budgetary cycles – cont.

- November 2014 start bidding process for DEFRA GiA for more equipment and development costs

- April 2015 - Agency reports first Chemical Status data to DEFRA/Europe

- April 2015 – due to funding delays earlier in 2014 miss opportunity to develop sufficient methods in time to make a full bid for the water industry CIP programme.

- Monitoring of the majority of contaminants for the Environment Agency can now not start until April 2016 (e.g. PFOS, Cpermethrin, Misc pesticides)

- Remaining funding is awarded and remaining instrumentation can be purchased. Mixture of GC and LC QQQ

- Methods developed

- April 2016 remaining methods go live
New methods

- Regulation Oct 2013
- Budget Estimation & Funding Feb 2014
- Identify BAT Jun-Nov 2014
- Procurement Nov 2014 - April 2015
- Monitoring & Reporting cycles 2015 - 2019
- Funding April 2015 for bulk
- Develop methods Up to Mar 2016
Validation Requirements

11x2 in UHP, River Water, Saline Water, Biota & some sediments

✓ Detection Limit – preferred approach to spike at the target.

✓ Limit of Quantification – UKTAG requires government labs to demonstrate control. Method to validate a QC at the LoQ followed by embed the level in the curve.

Precision & BIAS measured at:-

✓ LoQ (2x target LoD)

✓ EQS or below

✓ 15-20% range

✓ 75-80% range
Limit of Detection
Validation Data
Benzo (α) pyrene
ng l⁻¹
Benzo(α)pyrene (3ng⁻¹)
Benzo(α)pyrene (0.02ng⁻¹)
Limit of Detection
Validation Data
Benzo (α) pyrene
ngl⁻¹
<table>
<thead>
<tr>
<th>Water</th>
<th>Biota</th>
<th>BAT</th>
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<tbody>
<tr>
<td>Nonylphenol</td>
<td>PFOS</td>
<td>Dichlorovos</td>
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<tr>
<td>Octylphenol</td>
<td>HBCDD</td>
<td>Endosulfan</td>
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<tr>
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<td>Heptachlor</td>
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<td>Methiocarb</td>
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<td>HBCDD</td>
<td>April 1st</td>
<td>Pentachlorobenzene</td>
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<td>Tributyl tin chloride</td>
<td>Cypermethrin</td>
<td>Drins</td>
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<tr>
<td></td>
<td>cis and trans Permethrin</td>
<td>Methiocarb</td>
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<td></td>
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New methods

Regulation

Develop methods

Budget Estimation & Funding

Identify BAT

Funding

Monitoring & Reporting cycles

Procurement
- Chloroalkanes (SCCP) in water and biota

- Bis(2-ethylhexyl) phthalate (DEHP) in biota

- Investigation in effect of particulates on PAH, PFOS & HBCDD

- 2008 EQS – 2013 lowered the LoD’s

- 2018?

- Mercury 10ng to 0.07ng/l
Questions?