National Laboratory Service
Innovation

Science
Service
Solutions

# GC-MS (Multi-Residue) Screening for Pesticides; Emerging Organic Pollutants (EOP's) and Persistent Organic Pollutants (POP's).

National Laboratory Service
Innovation

Science
Service
Solutions

The GC-MS Target Based Screening methods allow for virtually all GC-amenable pesticides as well as hundreds of other organic pollutants to be identified from a single sample, incorporating over 950 target Chemicals and including both VOCs and SVOCs into a single unique target database.

National Laboratory Service
Innovation

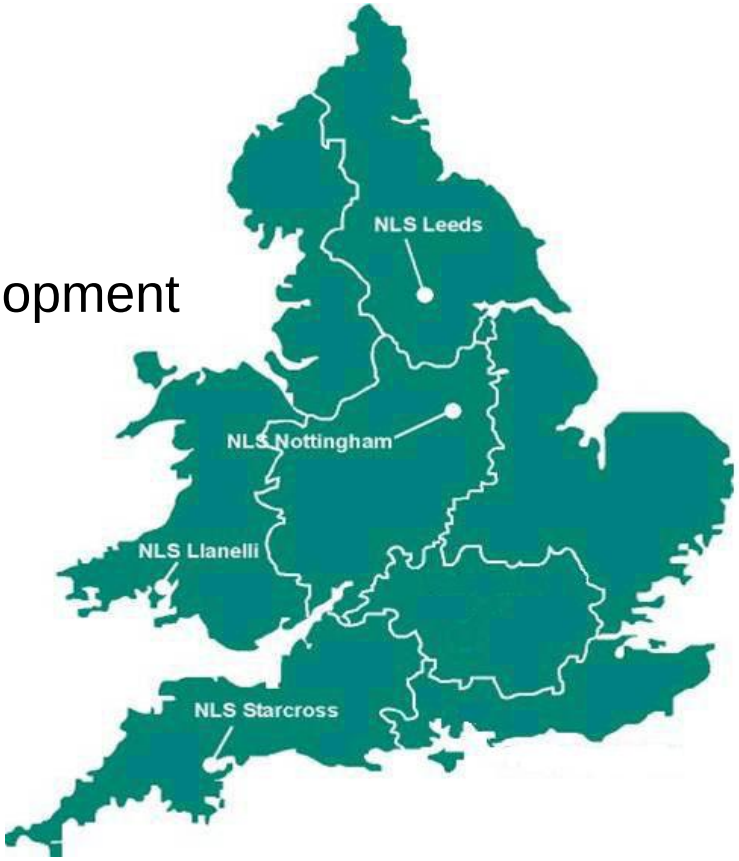
# Our Laboratories

Science
Service
Solutions

- Four laboratories, each one specialising in specific areas of environmental testing & analysis

- Soils and Contaminated Land
- Wastewater Analysis / Forensic
- Centre for Analytical Research & Development
- Clean Water Analysis
  & Microbiological Analysis

Full range of services visit
www.natlabs.co.uk



National Laboratory Service
Innovation

Science
Service
Solutions

# Water Framework Directive (WFD)

A strategic framework for managing the water environment

National Laboratory Service
Innovation

# Water Bodies

Science
Service
Solutions

**The Water Framework Directive applies to all:**

- Surface freshwater bodies (including lakes, streams, canals and rivers);
- Groundwaters;
- Transitional water bodies (estuaries);
- Coastal waters out to 1 mile from low-water (low-tide).

This contrasts with most existing EU Directives which only applied to designated or identified waters.

National Laboratory Service
Innovation

# Initial Specification

Science
Service
Solutions

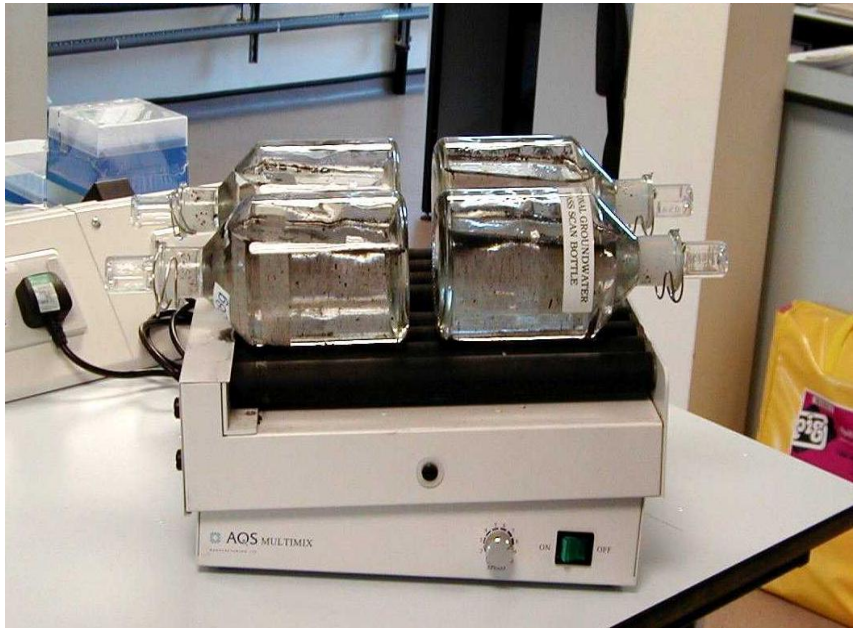
- Screening method to detect the Presence / Absence of Organic substances in a given water body
- Identification of both VOCs & SVOCs from a single sample
- Provision to add new substances
- Typical Limit of Detection (LoD) 0.1µg/l
- Economical option for validating the pressures and risks to water bodies.

National Laboratory Service
Innovation

# Sample Extraction

Science Service Solutions

- Dichloromethane solvent
- Extraction 15min
- Bottles are rocked from side to side whilst being rolled
- Maximise solvent-matrix interaction
- Reduced formation of emulsions



National Laboratory Service
Innovation

# Instrumentation

Science
Service
Solutions

## ❑ Hardware

- 5975C inert XL MSD with Triple-Axis detector (TAD)
- 7890A GC System
- 7683B Series Injector
- Programmed Temperature Vaporization Inlet (PTV)

## ❑ Software

- GC/MSD Chemstation
- Deconvolution Reporting Software (DRS)
  Automated Mass Spectral Deconvolution and Identification Software (AMDIS)
  NIST08 Mass Spectral compound library
- Hazardous Industrial Chemicals (HIC) database 567 compounds, Retention Time Locked (RTL)

National Laboratory Service Innovation

# Experimental

Science Service Solutions

## ❑ GC Instrument Conditions

- Inlet – Cold splitless 20 C (0.2) - 720 - 300 C (key parameter)
- Oven: 40 C (0.2) - 10 - 300 C (15)
- Constant pressure
- RTL to Fluorene at 15.577mins
- Run time: 36mins
- 30m x 0.25mm ID x 0.25um HP5-MS UI

## ❑ MS Instrument Conditions

- Acquisition mode – Full Scan
- Scan range 35 – 565u
- Source and Quad temps. 250 / 150 C

National Laboratory Service
Innovation

# VOCs Inclusion

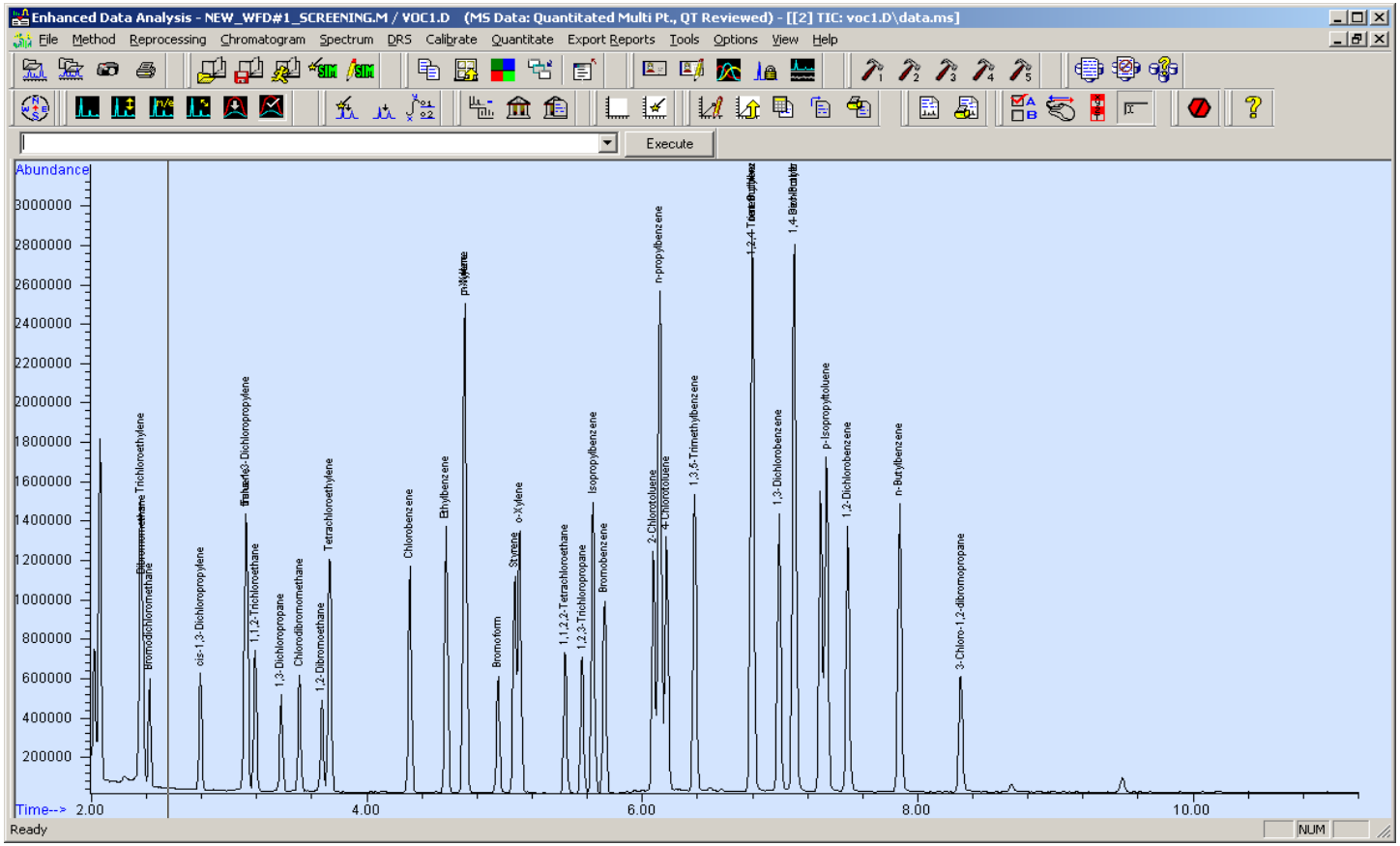
Science Service Solutions

- The 5975C inert XL MSD with Triple-Axis detector (TAD) has enabled us to include many of the volatile solvents
- This delivered the necessary increase in sensitivity
- Our previous GCMS screen method used a 10µl solvent vent injection (2 x 5µl)
- Not a suitable technique for the analysis of volatile solvents
- Use a 1µl splitless, cold inlet,  injection

National Laboratory Service
Innovation

# VOC Chromatogram

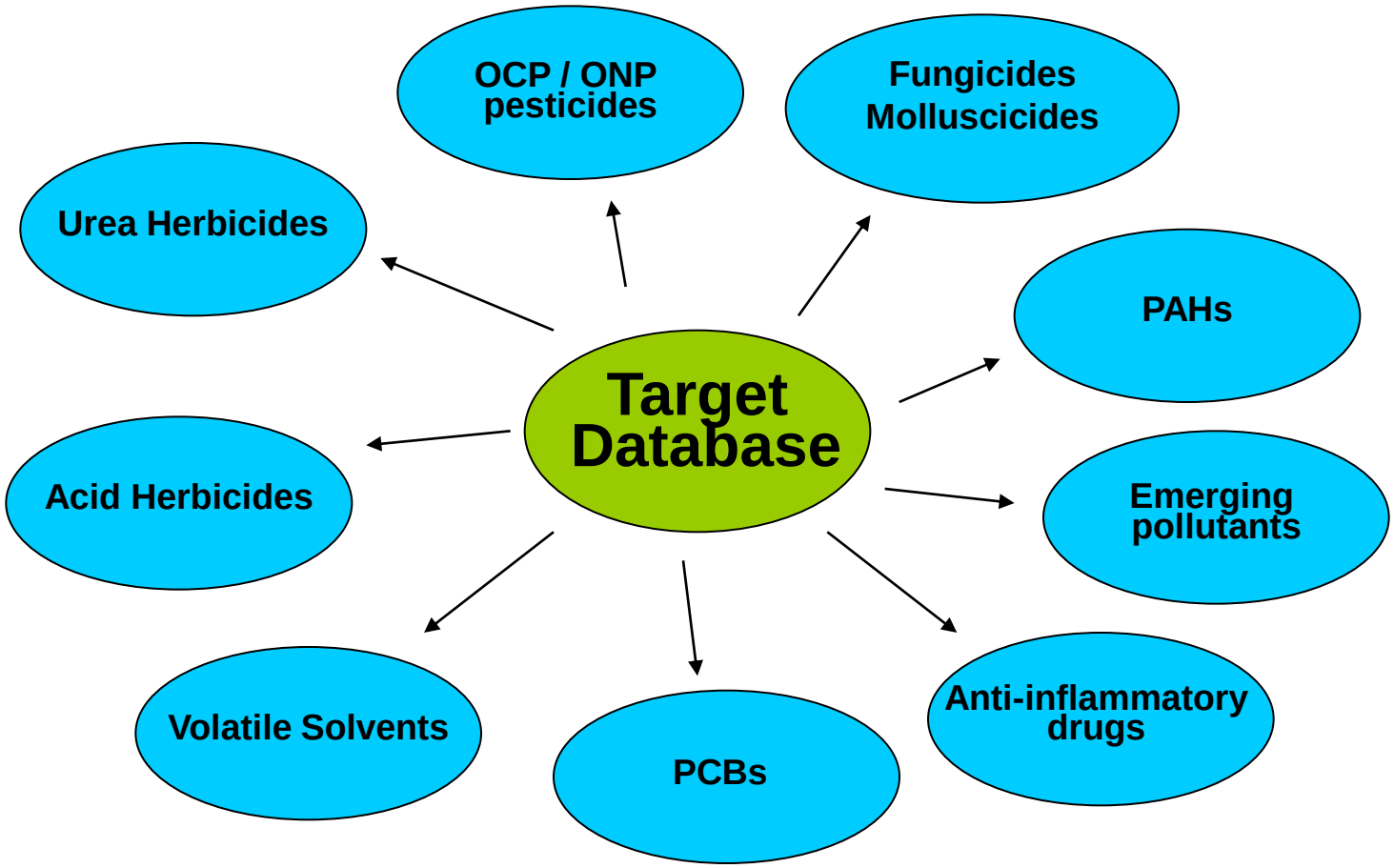
Science
Service
Solutions



National Laboratory Service
Innovation

# Make it Relevant

Science
Service
Solutions



National Laboratory Service
Innovation

# Limit of Detection

Science
Service
Solutions

## River water fortified with targets at 0.1µg/l

| Target Compound | River |
|---|---|
| 2-Chlorophenol | √ |
| Trichloroethylene | √ |
| 2,4-D methyl ester | √ |
| 2,4,5-Trichlorobiphenyl | √ |
| p,p'-DDE | √ |
| Pyrene | √ |
| Aldrin | √ |
| Atrazine | √ |
| Benzo (a) pyrene | √ |
| Caffeine | √ |
| Metaldehyde | √ |
| Carbetamide | √ |
| Chlordane | √ |

| Target Compound | River |
|---|---|
| Dichlorvos | √ |
| Ethyl Benzene | √ |
| Fenchlorophos | √ |
| Hexachlorobenzene | √ |
| Methoxychlor | √ |
| Pentachlorobenzene | √ |
| Pirimicarb | √ |
| Simazine | √ |
| Trietazine | √ |
| Vinclozolin | √ |
| Propachlor | √ |
| Tetrachloroethylene | √ |
| Methoxychlor | √ |

National Laboratory Service Innovation

# Reporting Limit

Science Service Solutions

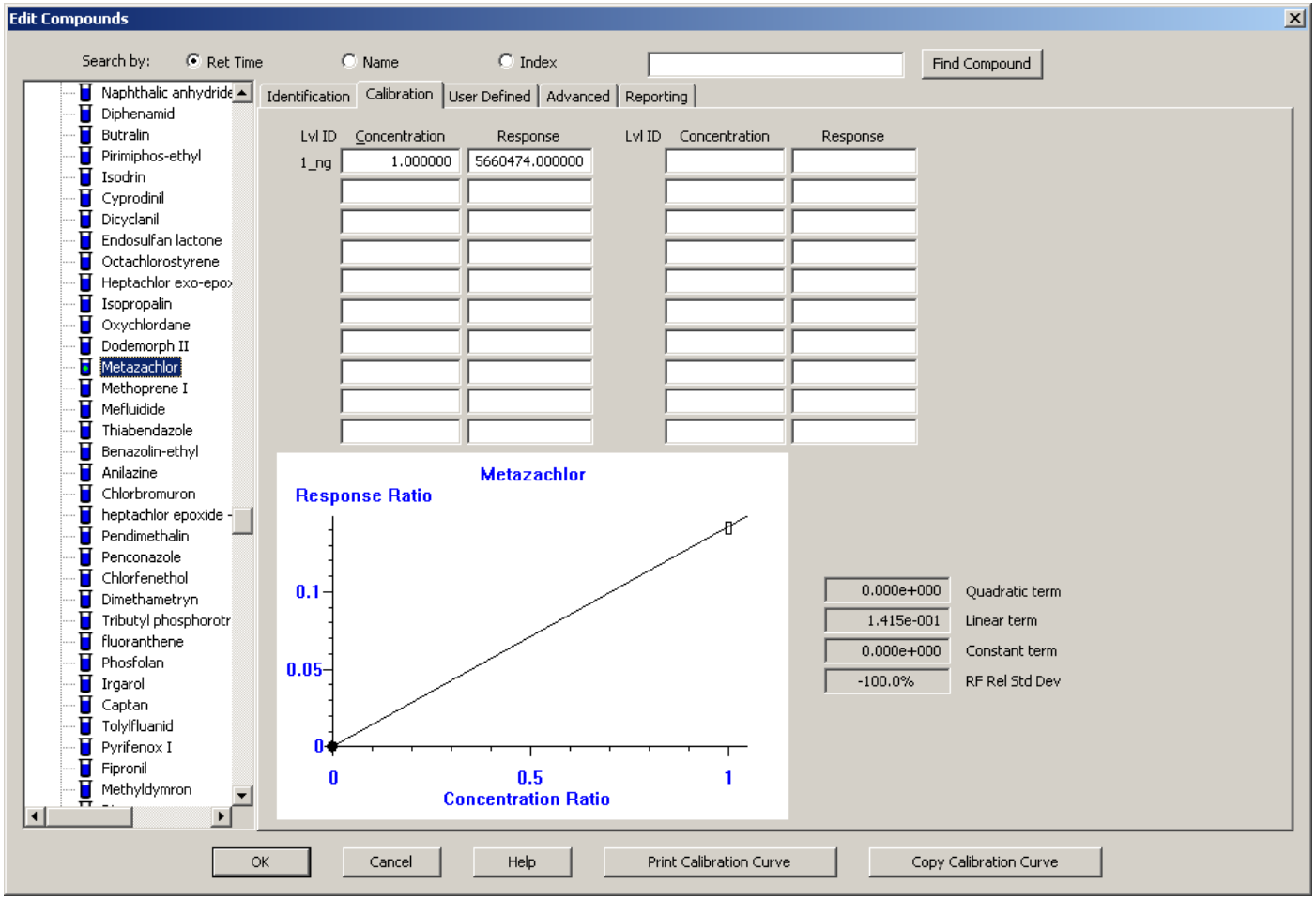
## In fact, 75% of the targets tested were detected at «0.02µg/l

| | | | Amount (µg/l) approx. | | AMDIS | | NIST | |
|---|---|---|---|---|---|---|---|---|
| R.T. | Cas # | Compound Name | Chem Station | | Match | R.T. Diff Sec. | Match | Hit Num. |
| **2.4158** | **79016** | **Trichloroethylene** | **0.02** | **0.01** | **48** | **4.8** | **90** | **1** |
| **7.7070** | **108623** | **Metaldehyde** | **0.02** | **0.02** | **45** | **-0.8** | **82** | **1** |
| **17.8784** | **1912261** | **Trietazine** | **0.02** | **0.01** | **40** | **-3.2** | **60** | **1** |
| **21.4323** | **129000** | **Pyrene** | **0.02** | **0.01** | **93** | **-4.4** | **93** | **2** |
| **24.6484** | **72435** | **Methoxychlor** | **0.01** | **0.01** | **90** | **-7.8** | **80** | **1** |

National Laboratory Service
Innovation

# Estimate of Concentration

Science
Service
Solutions



National Laboratory Service
Innovation

# Hours to Minutes

Science
Service
Solutions

- DRS has enabled us to dramatically increase the number of samples processed
- Now processing over 24 samples per day compared with just 8 using the traditional manual GCMS screen method approach
- Realised a real cost saving to the business
- Year on year increase since introducing the method in 2007/08 predicted workload of 650, actual received 1600 samples
- Demand continues to grow, predicted workload for 2009/10 of 3500 samples

National Laboratory Service
Innovation

# Summary

Science
Service
Solutions

- Large number of target compounds, over 800
- Combined VOC / SVOC detection
- Increased accuracy of target identification
- Fast data interpretation, from hours to minutes
- Low detection limits, even in complex matrices
- Strong link with (and support for) WFD
- Tailored to suit individual requirements
- Economical option for the identification of organic substances

National Laboratory Service
Innovation

# Other Applications

Science
Service
Solutions

- Sewage & Trade effluent discharge
- Potable raw water monitoring
- Sea life centres & aquaria
- Supporting University research projects
- Fish farms (freshwater & saline)
- Pollution investigations