Spotlight
Sample Preparation & Processing

Sample Management and Laboratory Testing

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The National Laboratory Service (NLS) recently announced its largest set of commercial contracts to date after having been appointed by UK Water operators to assist in the preparation for the adoption of the latest EU water legislation. Ian Rippin, Commercial Director at the NLS, describes how the programme will work in the new regulatory landscape.

The Chemical Investigations Programme

The UK Water Chemical Investigations Programme (CIP) is a massive scale environmental testing programme facilitated by the UK Water Industry Research organisation in collaboration with the Environment Agency. The CIP is an investigation into the management and control of 'priority substances' and other pollutants. In this context, 'priority substances' are defined in the Priority Substances Daughter Directive (PSDD), which supports the EU Water Framework Directive (WFD).

At the heart of the WFD and its daughter directives exists the establishment of Environmental Quality Standards (EQSs); values that have been determined for all EU water bodies, which define if concentrations of chemical contaminants are considered of 'good chemical status'.

Although water companies are not solely responsible for taking measures to meet these EQSs, they are required to assess the contribution their actions have on contaminant loads present in receiving waters. The CIP was launched to aid this by gathering data that will provide for a much clearer understanding of the frequency, concentration and behaviour of 'priority substances' throughout the wastewater treatment cycle. With the data captured, the measures to be implemented into the next cycle of the WFD, from 2014 forwards, can be more tailored to the cause.

The collaboration between water companies for the delivery of the CIP was essential to the collection of test data in a cost effective way. As part of the partnership the individual water companies will not have to assess the treatment options separately and they are able to share the data delivered by the whole programme.

The CIP for each individual water company will vary dependent on the extent and complexity of their regional networks, however it is expected that over 20,000 samples will be taken over the duration of the programme, breaking down into 6,000 sewage effluents, 8,000 catchment samples, 5,000 Wastewater Treatment Work process samples and 1,000 sludge samples.

Having started in 2010, the CIP is now in delivery with the majority of data expected before the middle of 2012. Given the size and complexity of the programme, the majority of water companies have decided to outsource the entirety of their CIP sample collection and associated laboratory analysis.

Determining substance stability

Given the substances involved, obtaining the necessary information to a proven and adequate level of reliability is a considerable challenge. To help meet this challenge prior to commencing with the main programme, the NLS conducted substance stability trials in final effluent samples. Completed in May 2010, the aim was to determine how stable the compounds of interest were in wastewater treatment work discharges. The results from the trials helped to determine the best testing regime to conclude the effectiveness of the control of these substances throughout the treatment process.

Stability trials were designed to establish any changes in substance concentrations after one, three and five day intervals, following a prescribed 'trend analysis' approach that was performed in triplicate. In some cases, the NLS continued testing after the prescribed five days to further confirm analytical results. For example, for Glyphosate – the world's biggest selling herbicide – stability testing proceeded for a total of nine days due to an expectation at the outset that this compound would prove to be unstable. Glyphosate is widely understood to be harmless to the environment, as current data suggests it breaks down quickly following its use.

It was assumed from the beginning that the substances of interest to the trials were relatively unstable or those for which no feasible or proven methods of chemical preservation are known. It was therefore surprising to discover that the majority of the substances included in the trials were found to be relatively stable under specific conditions. These conditions were designed to maintain the integrity of each sample taken, from its source to eventual analysis.

The net result is a definition for the CIP's sample preservation, storage and maximum holding times - between sample collection and analysis in the receiving laboratory. The findings suggest that to maintain sample integrity, chilling samples after collection to approximately 4°C and subsequently transporting and storing them at the same temperature is required. Samples must also be analysed or otherwise stabilised within three to five days of their collection.

The main areas of stability testing to date have focused on the following substances:

DEHP – a general purpose plasticiser used to make PVC plastic soft and flexible. DEHP was thought to be relatively unstable and is on the Priority Substance list.

TBT – an aggressive biocide that is used as a fungicide in agriculture and as an anti-fouling agent in ship paints. TBT is classed as a Priority Hazardous Substance (PHS) and of key concern because of its prescribed low EQS.

Pharmaceuticals and PSDD (Annex 3) substances – both of these groups were included in the trials on the basis that their determination on effluent and sewage process samples is not yet well established.

Reactive aluminium – as the name suggests, a substance that is likely to be subject to change.

Total mercury – included on the basis that mercury stability and contamination has long been shown to be an issue.

Bentazone and Mecoprop - both man-made chemicals that are used as herbicides. Bentazone is known to be harmful to fish and Mecoprop - widely used as an ingredient in household weed killers - to wildlife in general. Both substances were found to be more stable than previous data suggests.

Sample management considerations

Important to the success of the CIP and any large sampling programme is the sample management protocols deployed. Perhaps of most significance to any design is to ensure that samples taken are representative of the prevailing conditions in the environment being tested, therefore the identification of the appropriate sampling points is critical.

In the first element of the programme known as C1, screening of wastewater treatment works final effluents helps to confirm the concentrations and loads of substances being released to receiving waters. The majority of this screening work is now currently complete.