

**DAIMON Toolbox Fact Sheets:**

*Methods for measurement of warfare agents' pollution in sediments*

**Assessment category 2: Hazardous substances**

**Toolbox component: Water chemistry**

**Fact Sheet 2.4: Chemical analysis of CWA-related compounds in pore water with LC-MS/MS**

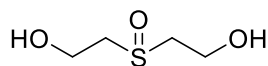
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**What is it?**

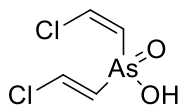
Chemical analysis of degradation products of chemical warfare agents in sediment pore water samples is based on liquid chromatography-tandem mass spectrometric technique (LC-MS/MS) utilizing multiple reaction monitoring (MRM). Identification criteria of detected chemicals are based on European Union guidelines of ion ratios.<sup>1</sup>

Target chemicals are degradation products of sulfur mustard, lewisite and phenylarsenic chemicals. Degradation products of sulfur mustard were screened for their presence. All analysed compounds are listed in the DAIMON Report on Chemical Analysis of Sea-Dumped Chemical Warfare Agents in pore water samples (WP2).<sup>2</sup>

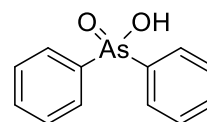
Example compound structures:



Thiodiglycol sulfoxide



Bis(2-chlorovinyl)arsinic acid



Diphenylarsinic acid

**What does it tell you?**

Chemical analysis of CWAs in sediment indicates if dumped munitions are present and leaking. A positive analysis result is a specific indicator for leakage of specific CWA.

**Type of Indicator** (tick box)

- non-specific stress indicator
- specific for groups of contaminants incl. CWA or explosives
- CWA-specific indicator
- specific for substances related to explosives (e.g. TNT)

### How to measure it?

Detailed sample preparation and analytical techniques are available in the DAIMON Report on Chemical Analysis of Sea-Dumped Chemical Warfare Agents in pore water samples (WP2).<sup>2</sup>

**Species:** Degradation products of CWAs in sediment pore water samples.

**Matrix:** Sediment pore water

**Equipment:** For measurement of CWAs in pore water samples, special laboratory equipment for sample preparation and analysis is required.

**Measurements and units:** Frozen sediment samples are thawed, and specific sample preparation techniques are applied prior to quantitative analysis utilizing LC-MS/MS technique in MRM-mode. Calibration standards (~ 1-150 pg/μl) are run prior and post the sample batch. Dimethyl methylphosphonate (DMMP) is used as an internal standard. The results of the analyses are presented as ng/ml from pore water removed from original sediment sample. Only values above the lower limit of quantitation (LLOQ) are reported.

### How to analyze and assess the data?

The identification of the detected chemicals are based on EU guidelines.<sup>1</sup> In case of selected scan techniques (like MRM), reliable compound identification requires relative intensities of the detected ions, expressed as a percentage of the intensity of the transition, to correspond to those of the reference standard, at comparable concentrations, measured under the same conditions. For each target chemical detected from the samples, ion ratio between the qualifier ion (q) and the quantifier ion (Q) is calculated (q/Q –ratio) and compared to that of the reference standard. The calculated ion ratios must fall in a certain tolerance window. If the q/Q–ratio does not fulfill the tolerance criteria, the identification is not considered reliable and the data will be rejected.

Concentrations of the target compounds are calculated based on the calibration standard curves. The LLOQ value for target chemicals is 8.7 pg/μl.

### Contact information of the analyzing laboratory

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### References

<sup>1</sup> COMMISSION DECISION of 12 August 2002 implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results, Official Journal of the European Communities, 2002, 2002/657/E, pp. 36

<sup>2</sup> DAIMON report *Chemical Analysis of Sea-Dumped Chemical Warfare Agents in Pore water samples*