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Application of Headspace and Direct Immersion Solid-Phase Microextraction in the Analysis of Organothiophosphates related to the *Chemical* Weapons Convention from Water and Complex Matrices

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## **ACCEPTED MANUSCRIPT**

Application of Headspace and Direct Immersion Solid-Phase Microextraction in the Analysis of Organothiophosphates related to the Chemical Weapons Convention from Water and Complex Matrices Marc André ALTHOFF<sup>a,b</sup>, Andreas BERTSCH<sup>a</sup>, Manfred METZULAT<sup>a,b</sup>, Thomas M. Klapötke<sup>b</sup> and Konstantin L. KARAGHIOSOFF<sup>b,\*</sup>

#### Abstract

The successful application of headspace (HS) and direct immersion (DI) solid phase microextraction (SPME) for the unambiguous identification and characterization of a series of toxic thiophosphate esters, such as Amiton (I), from aqueous phases and complex matrices (e.g. grass and foliage) has been demonstrated. A Thermo Scientific gas chromatograph (GC) – tandem mass spectrometer (MS/MS) system with a TriPlus RSH® autosampler and a SPME tool was used to investigate the effect of different parameters that influence the extraction efficiency: e.g. pH of the sample matrix and extraction temperature.

The developed methods were employed for the detection of several Amiton derivatives (Schedule II of the CWC) that are structurally closely related to each other; some of which are new and have not been reported in literature previously. In addition, a novel DI SPME method from complex matrices for the analysis of organophosphates related to the CWC was developed. The studies clearly show that DI SPME for complex matrices is superior to HS extraction and can potentially be applied to other related compounds controlled under the CWC.

#### **Abbreviations**

CBRN, Chemical, Biological, Radiological, Nuclear; CWA, Chemical Warfare Agent; CWC, Chemical Weapons Convention; DI, direct immersion; DVB, Divinylbenzene; GB, Sarin, (RS)-Propan-2-yl methylphosphonofluoridate; GC, gas chromatograph; HS, head space; HPLC, high performance liquid chromatography; ICI, Imperial Chemistry Industries; LLE, liquid-liquid extraction; LOD, limit of detection; LOQ, limit of quantification; MS, mass spectrometry or mass spectrometer; PDMS, Polydimethylsiloxane; SPE, solid phase extraction; SPME, solid phase microextraction; VX, Oethyl S-[2-(diisopropylamino)ethyl] methylphosphonothioate

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