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Detection of arsenic-containing hydrocarbons in canned cod liver tissue

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ABSTRACT

Arsenic is a metalloid well known to be potentially toxic depending of its species. Lipid-soluble arsenicals (arsenolipids) are present in a wide range of biological samples in which they could play a role in the biosynthesis of organoarsenic compounds from inorganic arsenic compounds. Arsenolipids have recently attracted considerable interest. In order to gain deeper insights into the impact of arsenolipids new analytical approaches for reliable determination of this class of arsenic-containing hydrocarbons in various matrices are needed.

High concentrations of arsenolipids were found in seafood which served as sample material in this study. We report the investigation of three arsenolipids found in canned cod liver from which they were extracted and purified by solid phase extraction (SPE) using a silica gel column and ethyl acetate/methanol as eluent. Analytical studies were conducted by means of gas chromatography coupled with ICP-MS, MIP-AES and EI-qMS and by TOF-MS. The results obtained by GC-ICP-MS and GC-MIP-AES showed the existence of numerous arsenic compounds in the SPE fractions collected. Three major peaks were found within a retention time window between 10 and 25 min. The presence of arsenic compounds in the fish tissue could be confirmed using GC-EI-qMS analysis. Corresponding information of the molecular weights of the major arsenic species were provided by TOF-MS which allows highly accurate mass determinations. The results showed the presence of the arsenic-containing hydrocarbons with the following molecular formulas: $C_{17}H_{37}AsO$ (calculated for $[M+H]^+$ 333.2133; found 333.2136; $\Delta m = 0.90$ ppm); $C_{19}H_{41}AsO$ (calculated for $[M+H]^+$ 361.2446; found 361.2446; $\Delta m = 0.00$ ppm); $C_{23}H_{37}AsO$ (calculated for $[M+H]^+$ 405.2133; found 405.2145; $\Delta m = 2.96$ ppm). Suggestions for the corresponding structures are discussed.

1. Introduction

Expect a multiplicity of water-soluble arsenic species lipid-soluble forms of arsenic compounds are present in the environment in a wide range, particularly in marine organisms [1–4]. In order to investigate the relevance of long-chain arsenic-containing hydrocarbons (in the following called 'arsenolipids') in organisms, new analytical approaches for reliable determination of this class of arsenic compounds in various matrices are necessary.

More than 40 years ago Lunde [5–7] reported that lipids extracted from various marine organisms contain elevated concentrations of arsenic as organic compound. Total concentrations of arsenic in oil and fatty acid fraction extracted from marine fishes and invertebrates were found to be between 4.7 and 84 mg As kg^{-1}, and from seaweed between 5.7 and 221 mg As kg^{-1}, respectively [7]. A fractionation of fish lipids on silica gel column was also performed using different polar eluents. Investigations on lower and higher freshwater plants based on isotope labelled experiments with ^{74}As indicated the presence of a high percentage (52–80%) of arsenolipids beside arsenite and water-soluble, lipid-related compounds like anionic glycosidic derivatives of trimethylarsoniumlactate [8].

The distinction in water-soluble and lipid-soluble arsenic-containing compounds which plays an important role in marine organisms was also made by Maher [9,10] and by Kaise et al. [11]. An unidentified, lipid-soluble dimethylated arsenic species was the major component analysed in marine organisms [11]. The fractionation in lipid-soluble and water-soluble forms was carried out with water, methanol, chloroform, or mixtures of these solvents. Recently, such fractionations were also applied for the determination of organoarsenicals in seafood products [12] and in Japanese flying squid tissues [13].

Benson et al. [14] suggested that o-phosphatidyltrimethylarsoniumlactate is a major arsenic-containing product in marine

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organisms, similar in structure to lecithin. In 1988 a phosphatidylarsenosugar was detected and characterized in brown algae (Wakame) by Morita and Shibata [15] by ^{1}H NMR. Edmonds et al. [16] determined phosphatidylarsenocholine and phosphatidyldimethylarsinylribose in digestive gland of lobster using HPLC-ICP-MS.

Several investigations were focused on the extraction of arsenolipids by alkaline solutions to separate them in alkaline-stable and alkaline-labile fractions [17,18] analysed by using HPLC-ICP-MS. Neutral and polar organoarsenicals were also found and determined in fish oil after acid digestion [19] using IC-ICP-MS.

Recently, intact natural occurring arsenic-containing long-chain compounds were detected in fish oil [20] using liquid chromatography. Long-chain hydrocarbons containing arsenic could be analysed in oil of capelin [21] and Sashimi tuna [22]. Arsenic-containing long-chain fatty acids were found in cod liver oil by using HPLC-ICP-MS and HPLC-ESI-MS and identified by high-resolution MS [23].

Also in organometalloid analysis the gas chromatographic methods offer an excellent option combining effective separation and a variety of detection features. Table 1 depicts some examples of the application of gas chromatography to the analysis of organoarsenicals in environmental and food samples. Furthermore, the optimization of the preceding extraction and/or derivatization can lead to a significant improvement on the resulting complete analytical methodology.

The aim of the present work was to investigate the occurrence of recently discovered arsenolipids in fish tissues and to identify as much as possible arsenolipids by combination of comprehensive methods. Therefore, two sensitive and element-specific detectors (ICP-MS and MIP-AES) combined with gas chromatography to identify As (m/z 75, λ = 189 nm) were employed. The capabilities of both techniques for sensitive determination of the arsenolipids should be investigated and compared with results obtained by GC-EI-qMS as a molecule selective method. Complementarily, flow injection electrospray ionization high-resolution mass spectrometry in combination with interpretation of isotopic pattern should be applied for identification of arsenolipids without any chromatographic separation.

2. Experimental

2.1. Instrumentation

2.1.1. GC-ICP-MS:

The GC-ICP-MS setup consists of a gas chromatograph (HP 6890) coupled online with an ICP-MS 7500ce via a heated transfer line (both Agilent Technologies, Wilmington, DE, USA). The conditions for separation and detection are listed in Table 2.

2.1.2. GC-MIP-AES

A gas chromatograph (HP 6890, Agilent) was coupled with a microwave induced plasma atomic emission detector (G 2350 AED, jas, Moers, Germany). The conditions for separation and detection are presented in Table 2.

2.1.3. GC-MS

GC-EI-qMS consisting of Network GC System 6890N, mass selective detector 5973 and injector 7683 Series served as a method for the identification of the metalloid species based on their molecular mass. Also, chromatographic as well as detection conditions are summarized in Table 2.

2.1.4. TOF-MS:

For high-resolution mass spectrometry, a micrOTOF (Bruker Daltonics, Bremen, Germany), equipped with an Agilent CE-ESI-MS sprayer kit (G1607A), was used. A mixture of iso-propanol and water (50:50, v/v) containing 0.2% formic acid served as sheath-liquid and was pumped with a syringe pump model KDS 601553 (KDScientific, Holliston, MA, USA) selecting a flow rate of 3 μL min^{-1}. Sample introduction was performed applying pressure to the sample vial which was connected with the coaxial sheath-liquid sprayer via a short piece of fused silica capillary (50 μm I.D., 360 μm O.D.).

2.2. Sample preparation

2.2.1. Extraction of arsenolipids from canned cod liver (CCL)

Cod (*Gadus morhua*) liver (60 g of canned fish liver) was cleaned from the surrounding oil, cut in small pieces, and mixed with methanol (250 mL). Afterward, the slurry consisting of the hackled cod liver in methanol was sonicated for 24 h at room temperature. The supernatant (MeOH + arsenolipids) was transferred to plastic tubes (15 mL), and centrifuged at 5000 rpm for 10 min. The yellow liquid was collected from the Eppendorf tubes and the excess of solvent was removed under reduced pressure to yield a cream-yellow liquid (10 mL). This liquid was transferred to tubes and centrifuged again at 5000 rpm for 10 min. The solvent was evaporated under reduced pressure resulting in about 1 mL of yellowish oil. This oil was fractionated by preparative column chromatography on silica gel. The column was packed with 24 g of silica gel (0.063–0.200 mm). The eluent was a mixture of ethyl acetate/MeOH with changing compositions added in the order described in Table 3.

Between 12 and 13 fractions of 20 mL each were collected. Afterwards, the excess of solvent in each fraction was evaporated under reduced pressure to dryness. Then, each dry fraction was dissolved with MeOH (1 mL) and finally analysed. Canned cod liver from different brands were analysed with the result that the main volatile arsenic components under investigation were identically. However, the concentrations of these As-compounds were quite different depending on many factors not specified on the can.

3. Results and discussion

Derived from the aim of the present work the occurrence of the known long-chain arsenolipids in cod liver tissue as an up to now not investigated sample will be investigated by the combination of comprehensive gas chromatographic methods. With only GC-EI-qMS a detection of the organoarsenicals is impossible even if their molecular masses are known. This method lacks on selectivity for the detection of arsenic that m/z interferences can occur always. Therefore, two sensitive and element-specific detectors (ICP-MS and MIP-AES) were combined with gas chromatography to identify the element arsenic in the compounds under investigation. Both detectors are able to be employed element-specifically using independent principles of determination. The molecular masses of the organoarsenicals should be also monitored with high-resolution MS and isotopic pattern.

3.1. Optimization of GC-ICP-MS conditions

The ICP-MS conditions were optimized with respect to maximum sensitivity using Xe m/z 124 added to the Ar-carrier gas flow. The carrier gas flow rate of the ICP-MS and the Ar/O_2 pressure as well had the main influence on peak height and shape. Optimal conditions were achieved using 0.6–0.7 L min^{-1} carrier gas and an Ar/O_2 gas pressure of 138 kPa. To avoid dead volumes in the system the separation capillary column was conducted from the injector up to the tip in the torch.

Table 1
Examples of the hyphenated techniques used for the analysis of organometal(loid)s in natural samples.

Method	Target analysed compounds	Extraction	Derivatization	Samples	Ref.
GC-MS	Organoarsenic warfare agents	Dichloromethane	+	Sediments	[24]
GC-ICP-MS	Methylated Ge-, As-, Sn-, Sb-, Te-, Hg-species	Hydride generation for liquid samples	+	Soil, sediments, waste, compost	[25]
GC-ICP-MS	As-, Sb-, Sn-species	Hydride generation (purge and trap)	–	Urban soils	[26]
GC-MIP-AES	Dimethylarsinic acid, monomethylarsonic acid	Liquid extraction	+	Seawater, wine, beer, infant food	[27]

Table 2
Parameters for GC-ICP-MS, GC-MIP-AES, GC-MS and TOF-MS.

GC-ICP-MS	
GC	
Column	HP-5MS (30 m × 0.25 mm × 0.25 μm) up to torch (Agilent)
Carrier gas	He 2 mL min^{-1}
GC-Programs	A: 40 °C, 10 °C min^{-1} to 60 °C, 30 °C min^{-1} to 250 °C, 40 °C min^{-1} to 280 °C B: 50 °C, 1 min, 50 °C min^{-1} to 180 °C, 3 °C min^{-1} to 220 °C 1 min, 15 °C min^{-1} to 270 °C 8 min [30]
Injector mode	Splitless
Transfer temperature	280 °C
Injector temperature	280 °C
Make-up gas flow rate	Ar/O_2 (20%) 20 psi
ICP-MS	
RF power	1500 W
Plasma gas flow rate	Ar 15 L min^{-1}
Carrier gas flow	0.5–0.7 L min^{-1}
Sample depth	6–7 mm
Element monitored	*m/z* 75 (As)
GC-MIP-AES	
GC	
Column	HP-5MS (30 m × 0.25 mm × 0.25 μm)
Carrier gas	He
GC-Program	See GC–ICP-MS
Injector mode	Splitless
Transfer temperature	280 °C
Injector temperature	280 °C
AED	
Reagent gas	H_2, 20 psi
Cavity temperature	300 °C
Make-up gas flow	90 mL
Wavelength	As 189 nm
GC-MS	
GC	
Column	HP-5MS (30 m × 0.32 mm × 0.25 μm)
Carrier gas	He 2 mL min^{-1}
GC-Program	See GC-ICP-MS
Injector mode	Splitless
Injector temperature	250 °C
MS	
Compound monitored	*m/z* 315, 333, 388 [M–O]
TOF-MS	
Capillary voltage	–4500 V
End plate offset	–500 V
Capillary exit	150.0 V
Nebulizer gas	(N_2): 0.4 bar
Drying gas	(N_2): 4.0 L min^{-1}
Drying temperature	200 °C
Skimmer	1: 50.0 V, 2: 23.0 V
Hexapole voltage	1: 23.0 V, RF: 300 V
Transfer time	49.0 μs
Pre-pulse storage	10.0 μs
Detector voltage	–0 V

3.2. Comparison of GC-ICP-MS and GC-MIP-AES

To compare and verify the performance of both analytical systems used GC-ICP-MS and GC-MIP-AES, two commercially available standard compounds were analysed. Triethylarsine (Et_3As, STREM, 99%, Royston, UK) with a boiling temperature (T_B) of 140 °C [28] and triphenylarsine (Ph_3As, Merck, Darmstadt, Germany) with T_B of 360 °C were used for characterization of sensitivity in different retention time windows. The Et_3As (RT = 2.75 min) appears at the beginning of the gas chromatogram and Ph_3As (RT = 15.8 min) at the end. Related to the arsenic content of the compounds the sensitivity was similar for both standard compounds using peak areas for quantification. With GC-ICP-MS a calibration was performed in the (As) concentration range of 1.1–11.0 ng μL^{-1} for Et_3As and of 0.74–7.4 ng μL^{-1} for Ph_3As, respectively. The limit of detection (LOD) calculated using the 3δ-criterion with peak height was 0.05–0.13 ng μL^{-1} for Et_3As and Ph_3As, respectively.

Table 3
Composition and elution strength of the eluent used for cleaning and fractionation of a methanolic cod liver tissue extract by different mobile phase mixtures (E° (SiO_2) = 0.77 E° (Al_2O_3) [29]).

Fraction	Ethyl acetate (%)	MeOH (%)	E° (SiO_2)
1	100	0	0.45
2	90	10	0.48
3	80	20	0.51
4	70	30	0.53
5	60	40	0.56
6	50	50	0.59
7	40	60	0.62
8	30	70	0.65
9	20	80	0.67
10	10	90	0.70
11	0	100	0.73
12[a]	0	100	0.73

[a] Repeated extraction with methanol.

3.3. Analysis of cod liver tissue samples

Preliminary extraction and dilution experiments with cod liver tissue and cod liver oil naturally occurring by conservation in the can, respectively, have shown that the kind and degree of extractable arsenic species depends strongly on the polarity of the extracting agents. In a first trial cod liver tissue and oil were extracted by n-hexane as well as methanol. Using n-hexane as extractant only small quantities of arsenic-containing compounds could be extracted and determined. Much higher extraction efficiencies were achieved by using methanol as extracting agent. In this extract three major arsenolipids could be detected in the retention time window of 10–20 min by means of GC-MIP-AES, GC-ICP-MS and GC-EI-qMS under nearly identical separation conditions (injection mode, column, temperature program) (Fig. 1). For the selective extraction of the cod liver tissue a scheme was developed and applied to clean and fractionate the primary cod liver extract with the aim to isolate the free major arsenic-containing compounds detected by MIP-AES, ICP-MS and EI-qMS after GC separation. Therefore, a preparative glass column was filled with silica gel and conditioned by ethyl acetate. After addition of the primary extract the composition of the mobile phase was changed stepwise from 100% ethyl acetate to 100% methanol according to the description in the experimental part and Table 3. During this procedure the polarity was varied from E° (SiO_2) = 0.45–0.73. Each of the fractions were collected separately, the excess of solvent was evaporated and the residue subsequently analysed

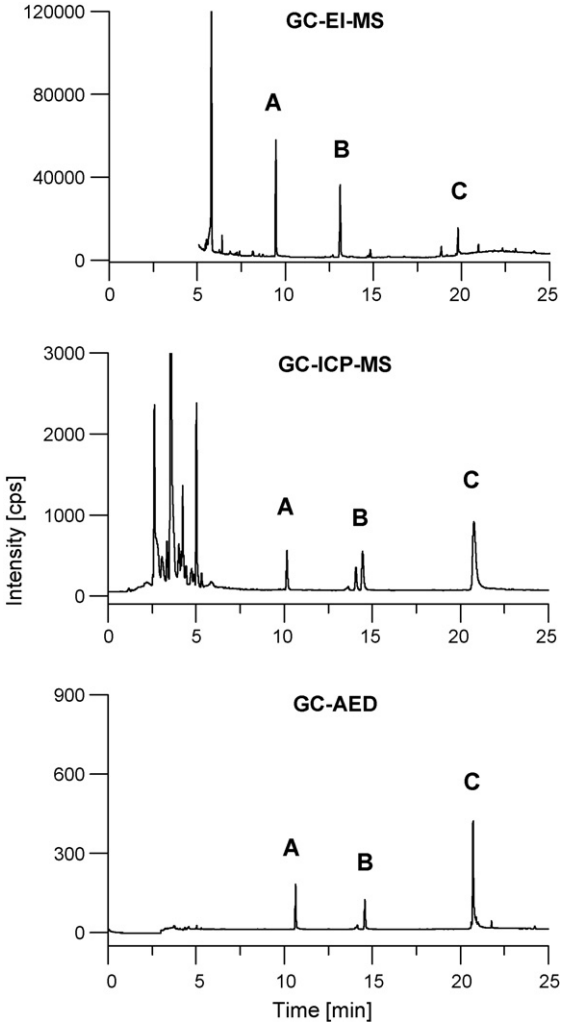

Fig. 1. Comparison of the separation and detection using GC-MIP-AES, GC-ICP-MS and GC-EI-qMS. (A) Arsenolipid m/z 316; (B) arsenolipid m/z 344; (C) arsenolipid m/z 388 (all for GC-MS) (for arsenolipid identification see Fig. 5); GC-Program: B.

by gas chromatography (method A) with MIP-AES and ICP-MS detection (Figs. 2 and 3) under optimized separation conditions. The fractionation of the extract did not lead to a separation of the major arsenic components. However, surprisingly remarkable amounts of high volatile organoarsenicals (HVAs) could be detected in the more non-polar fractions 7 and 8 in the retention time window of 2–9 min with the ICP-MS detection (Fig. 3) as well as within 3.5–9 min with the MIP-AES detection (Fig. 2). With increasing polarity of the eluting solvent mixture, the concentrations of the lower volatile arsenic species (LVAs) increased in the fractions 10, 11 and 12 occurring in the retention time window of 9–15 min. Additionally, to these major aliphatic long-chain arsenolipids, decreasing amounts of higher volatile arsenic species were detected with increasing polarity of the extractant. For example, in the fraction 11 around 90% of the chromatographed arsenic is bound in form of LVAs and approximately 10% as HVAs.

Unfavourable is the soaring peak broadening with increasing retention time in GC-ICP-MS (Fig. 3) compared to GC-MIP-AES (Fig. 2). However, a multiplicity of arsenic-containing compounds (e.g. Fig. 3) appeared in the retention time window of HVAs between 2 and 9 min. Ten to twenty times higher peaks were detected in the fractions 7 and 8 compared to the fractions 10, 11, and 12. Due to the high number of detected arsenic-containing compounds,

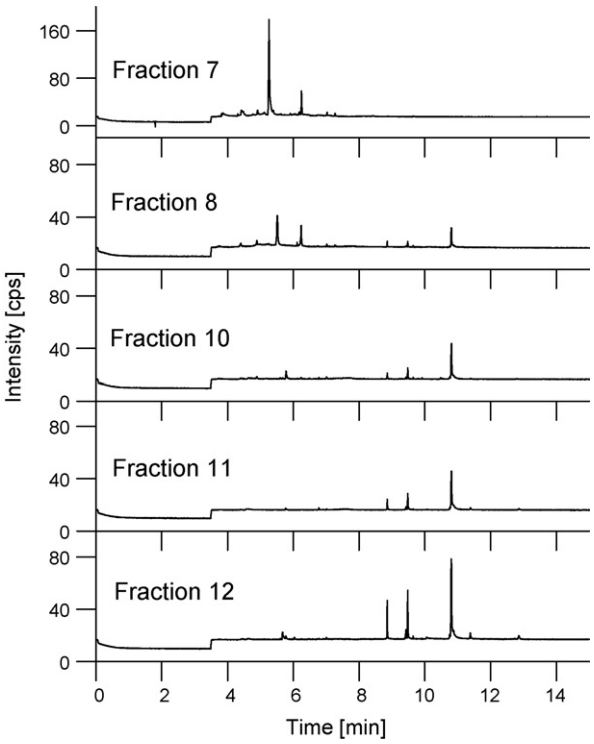

Fig. 2. GC-MIP-AES, CCL fractions 7, 8, 10, 11, 12; GC-Program: A.

the assumption arose that interferences by $ArCl^+$ can occur caused by chlorine-containing organic compounds. Parallel detection of masses 35 and 37 showed no appropriate peaks in the whole retention time range of the chromatogram.

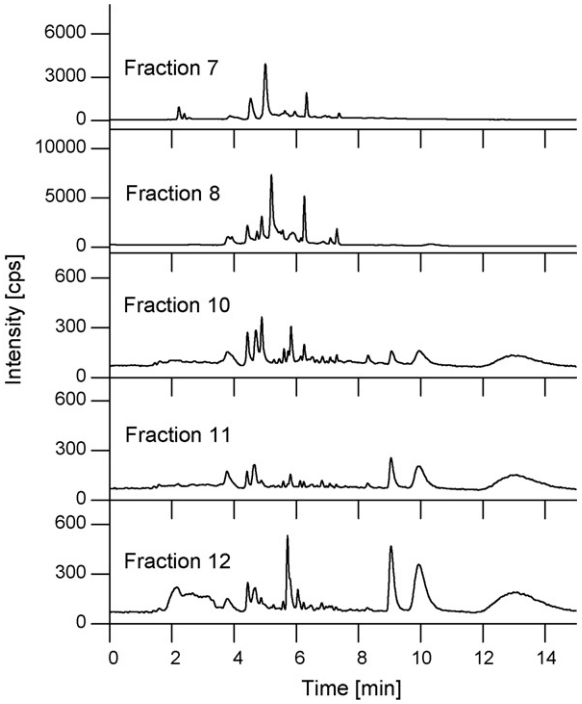

Fig. 3. GC-ICP-MS, CCL fractions 7, 8, 10, 11, 12, GC-Program: A.

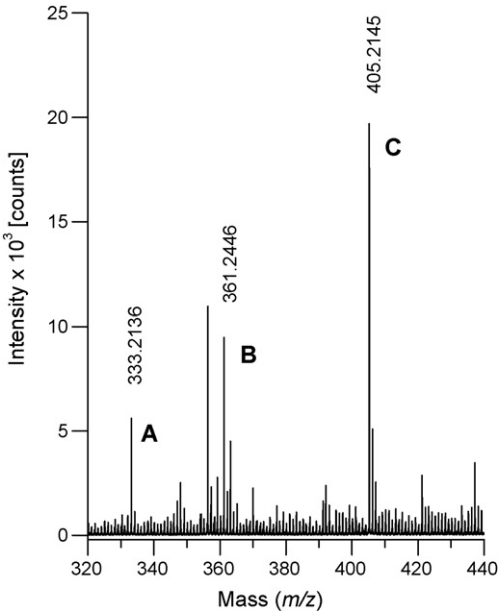

Fig. 4. ESI-TOF-MS spectrum of three arsenolipids identified in cod liver with the following molecular formulas: (A) $C_{17}H_{37}AsO$; (B) $C_{19}H_{41}AsO$; (C) $C_{23}H_{37}AsO$; fraction 12.

3.4. Identification of arsenolipids

3.4.1. GC-ICP-MS and GC-MIP-AES

As seen in Fig. 1, using arsenic-specific detectors (ICP-MS m/z 75 and MIP-AES 189 nm) combined with GC quite similar results were obtained regarding the retention time and number of major peaks. All three peaks could be detected element-specifically in all samples under investigation. Compared with GC-EI-qMS small differences in the retention times of the corresponding peaks could be observed attributed to the vacuum system of the MS-detector.

3.4.2. GC-EI-qMS

According to the findings of Raber et al. [30] the mass to charge ratios of three arsenolipids [M−O] were detected in the single ion-monitoring mode as seen in the upper chromatogram in Fig. 1. The m/z 316, 344, and 388 correlate clearly with peak A (RT 9.47 min), peak B (RT 13.11 min), and peak C (RT 19.80 min), respectively. Structural information for these three arsenolipids is given later. However, also other isomeric structures may be detectable by EI-qMS, judging by the additional peaks detecting in the single ion-monitoring mode. In the case of m/z 388 a structural derivative of docosahexaenoic acid ($C_{22}H_{32}O_2$, CAS: 6217-54-5) can also be detected. However, the pattern of the major peaks in the GC-EI-qMS chromatogram corresponded very well with the arsenic-selective signals of MIP-AES (189 nm) and ICP-MS (m/z 75). Therefore, it is most likely that they concern arsenolipids.

Unfortunately, the sensitivity of GC-MS in the scan mode did not allow the identification of additional arsenic-containing compounds.

Summarizing the results obtained by GC-MS analysis of cod liver tissue extracts, identical arsenolipids could be analysed and detected as in capelin fish (*Mallotus villosus*) oil [21]. It is known that capelin fish is the major food source for cods; however, the direct uptake of arsenate and its metabolism by the fish could be possible.

3.4.3. TOF-MS

TOF-MS which allows highly accurate mass determinations was used to gain complementary information. The extracts were injected hydrodynamically into the ESI-source of the TOF-MS via a fused silica capillary. The results in the positive ionization mode presented in the MS spectrum (Fig. 4) show the presence of these arsenic-containing hydrocarbons with the following molecular formulas: $C_{17}H_{37}AsO$ (calculated for $[M+H]^+$ 333.2133; found 333.2136; $\Delta m = 0.90$ ppm); $C_{19}H_{41}AsO$ (calculated for $[M+H]^+$ 361.2446; found 361.2446; $\Delta m = 0.00$ ppm); $C_{23}H_{37}AsO$ (calculated for $[M+H]^+$ 405.2133; found 405.2145; $\Delta m = 2.96$ ppm). The agreement between the calculated and experimentally found data is excellent and supports the identification by GC-MS. Fig. 5(A–C) shows in detail the simulated isotopic pattern (SIP) of the high-resolution mass spectra of the 3 compounds. In addition to the high mass accuracy of TOF-MS the isotopic pattern calculated from the isotope abundance of C, H, O and the monoisotopic mass of As fits very well with the experimental data.

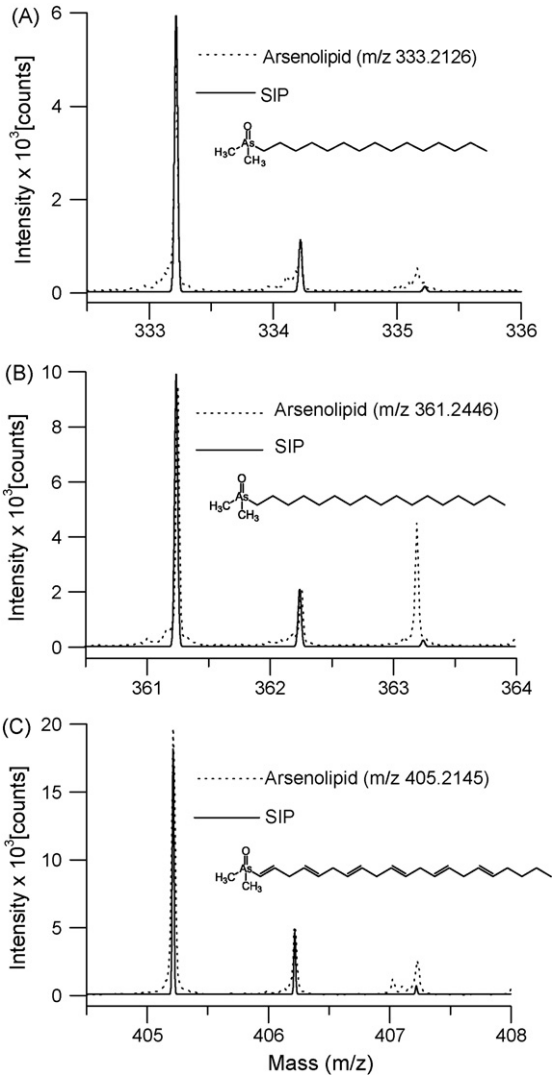

Fig. 5. Overlay of the ESI-TOF-MS spectra with simulated isotopic pattern (SIP). (A) $m/z = 333.2136$; (B) $m/z = 361.2446$; (C) $m/z = 405.2145$.

4. Conclusion

Arsenic species are not only widely distributed in the aqueous environment, but also in non-aqueous (hydrophobic) compartments of aquatic organisms. In analogy to free fatty acids characterized by a polar carboxylic group and a long-chain aliphatic

tail, the corresponding arsenic compounds, in which the carboxyl group is replaced by a dimethylarsinoyl group as polar unit, are present in aquatic animal tissue. Those volatilizable compounds can be determined by gas chromatographic methods. It could be shown that after a successful separation of numerous arsenic-containing compounds both element-specific detectors (ICP-MS or MIP-AES) can be applied for sensitive determination of the arsenolipids investigated. Applying only GC-EI-qMS a detection of the organoarsenicals is only possible if their molecular masses are known. Interferences caused by similar m/z ratios but without arsenic are very probable. Therefore, GC-EI-qMS identifications must be assured by GC-ICP-MS or GC-MIP-AES measurements. On the other hand the element-specific detection has some advantages in the determination of low concentrations of the arsenolipids.

Additionally, the ICP-MS detection has the benefit that the chromatogram can monitor also higher volatile arsenic compounds by the absence of the solvent peak.

Fractionation with a polarity gradient can be used to differentiate between low volatile and high volatile arsenic compounds. The resulting fractions contain a variety of arsenic species in the boiling point range of 40–300 °C, which still need further work for identification. High-resolution mass spectrometry with the capability of isotopic pattern analysis can effectively support the GC-MS data. Coupling of TOF-MS with liquid chromatographic or electrophoretic separation technique may further improve the identification capabilities. Additional research efforts are necessary to isolate and preconcentrate such arsenolipids for toxicological tests.

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