

Speciation of Arsenic species in marine products by HPLC-ICP-MS

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In general, speciation of arsenic species is known for several years and wide a scope of analytical methods have been developed since then. A number of publications have been written on the speciation of arsenic compounds in drinking waters, marine products and plant materials (e.g. 1-2). However the number of publications on arsenic speciation in animal feed products is limited, while e.g. seaweed and algae are often used as feed ingredient. This contribution describes the method development based on HPLC separation of the species and the ICP-MS detection of organic and inorganic arsenic species in fishery products and seaweed based animal feed products.

European legislation on arsenic species in fishery products is lacking. Maximal residue level (MRL) for fruit and vegetables is based on total amount of arsenic and is 0.1 mg/kg product. In animal feed the legislation on total and inorganic arsenic are given by EU2003/100 for compound feed based on fish and fishery products, or feed products based on seaweed. The maximal residue level for inorganic arsenic (some of As³⁺ and As⁵⁺) in these feed products is 2 mg/kg.

Speciation of organic and inorganic arsenic is usually done by using an unselective extraction method combined with a LC-ICP-MS. Concentrations of the inorganic species, As³⁺ and As⁵⁺, can be summed mathematically afterwards, however our preference is the chemical conversion of As³⁺ to As⁵⁺ by redox reaction with hydrogen peroxide during the extraction before HPLC separation. The chromatography was therefore optimized for the As⁵⁺ separation and the ICP-MS was used for arsenic detection. The total amount of inorganic arsenic was expressed as As⁵⁺. The total As concentration was determined by a second measurement using the ICP-MS.

200 mg seaweed (or compound feed) is weighted and 10 ml 0.07 Molar HCl prepared in 10% H₂O₂ is added. The sample was placed in the microwave (600 W) at 90°C for 25 minutes. The extract was centrifuged and 100 µl was analysed on an HPLC equipped with an anion PRP-X100 column. Eluting compounds were detected on the online coupled ICP-MS. Performance of the method and quality control was done using seaweed and fish tissue reference materials, respectively BCR-279 and TORT-2.

Organic and inorganic arsenic species in marine products were successfully extracted with 0.07M HCl prepared in 10% H₂O₂ and analysed with LC-ICP-MS. The concentrations inorganic arsenic in seaweed and compound feed were between 0.1 and 1 mg/kg, i.e. always below the MRL. Total As contents were in seaweed in the range from 20 to 40 mg/kg (n=12) and for compound feed 1 to 10 mg/kg (n=13).

References

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