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Comparison of INAA and LC-ICP-MS for the determination of As species in marine tissues

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The aim of this work was to acquire traceable quantitative results for total As in whole samples as well as in extracts containing As species. Instrumental neutron activation analysis (INAA) is well suited for such measurements and is used in this work to validate the As mass fraction determined by liquid chromatography combined with inductively coupled plasma mass spectrometry (LC-ICP-MS) in tuna fish and kelp candidate RMs and other marine samples, including certified RMs.

INAA was used to determine total As and trace elements in original solids, extracted solids, and in extracts as well as LC fractions; LC-ICP-MS was limited to the determination of As species in extracts. Extraction yields were determined by INAA for a number of common solvents and extraction techniques; the best results were acquired after methanol/acetone/water extraction with sonication. This procedure was used for quantitative As species evaluation with LC-ICP-MS incorporating internal standards and single point standard addition, while the sum of all As fractions was monitored by INAA. In the case of tuna tissue, AB was the predominant species determined by LC-ICP-MS and its As mass fraction was 4.41 ± 0.09 mg/kg. The total extracted As by INAA was 4.88 ± 0.27 mg/kg. In case of the BCR 627 certified RM the sum of AB + DMA was 4.15 ± 0.10 mg/kg, measured as AB = 3.99 ± 0.08 mg/kg and DMA = 0.148 ± 0.010 mg/kg in good agreement with the certified values, and the total As extracted was 4.28 ± 0.18 mg/kg by INAA summing up with As in the residue to 4.75 ± 0.17 mg/kg in excel-lent agreement with 4.81 ± 0.11 mg/kg determined by INAA in the original material. These differences between the techniques may be explained by relatively too high dissolution required by LC-ICP-MS not detecting very low mass fractions of other species in the extracts, and/or retention of As on the column.

To completely evaluate the LC-ICP-MS process INAA detection limits were lowered utilizing the Compton suppression technique in the gamma spectrometry to reach below 0.2 ng sensitivity, which is sufficient to determine As in LC effluent fractions. LC effluent fractions were collected according to the time intervals recorded by the ICP-MS, reduced in volume, and transferred and dried on Whatman 542 filter substrate. These were subsequently submitted to INAA. The initial results showed As in the background (blank) fractions of the chromatogram, however most of it due to solvent blank. Arsenic in these fractions is not captured by ICP-MS and thus would explain the difference. Further, the quantitative determination of As by INAA in the chromatogram peaks provided a direct measurement of each separated species and allowed for calibrated determination of each species. Species, including those not available as standards, e.g., the arsenosugars found in kelp, can be accurately determined by INAA and used as calibrants. INAA and LC-ICP-MS thus were successfully used as complementary techniques for characterization and traceability studies related to the development of RMs for As species.

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