

Application of electrochemical sensors able to discriminate against arsenic species relevant for food safety assessment

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Introduction

Toxicity of arsenic depends on its molecular nature



Conventional technique for As speciation Anion exchange Cation exchange Column **PRP-X100** Zorbax 300 SCX T (°C) 30 40 Mobile phase $20 \text{ mM NH}_4\text{H}_2\text{PO}_4$ 10 mM pyridine (pH 2.3) (pH 6) Q_1 (mL min⁻¹) 1.5 1.5 AB, Gly, TMAP Analytes DMA, MMA, PO_4 , SO₃, As^{III}, As^V **ICP-MS** HPLC

Electrochemical sensors for As speciation



RE: Ag/AgCl AE: Platinum filament WE: NEE Supporting electrolyte: HCl 0.2 M + Hydrazine 62 mM

Deposition: As(III) + $3e^- \rightarrow As^\circ$

Stripping: $As^{\circ} \rightarrow As(III) + 3e^{-1}$

Electrochemical sensors for As speciation

NEEs: Nanoelectrode ensembles



- (a) Track-etched golden membrane
- (b) Cooper adhesive tape with conductive glue to connect to instrumentation
- (c) Aluminium adhesive foil with non-conductive glue
- (d) Insulating tape

Real samples:

- <u>Sea products</u>: Canned tuna, fresh tuna, mussels, kombu algae, shrimps. Certified krill, dogfish muscle and lobster hepatopancreas.
- <u>**Rice products</u>**: spaghetti, flour, cracker, gallette, Jasmine rice, arborio rice and certified flour rice.</u>

Sample preparation:

- A portion (~100 mg for sea products and 500 mg for rice products) of the dried sample was weighed directly into a 15 mL polypropylene tube.
- 5 mL of 20% (v/v) methanol were added and the mixture was shaken top over bottom at ambient temperature overnight.
- Centrifugation (4500 rpm) × 20 min at room temperature.
- Filtration through an 0.45-µm PVDF filter (Millipore).

Analytes of interest



Results and discussion HPLC/ICPMS As speciation



Results and discussion

HPLC/ICPMS As speciation

<u>Sea products</u>: Canned tuna, fresh tuna, mussels, kombu algae, shrimps, certified krill, dogfish muscle and lobster hepatopancreas.





Results and discussion (HPLC/ICPMS As speciation)

<u>Rice products</u>: spaghetti, flour, cracker, gallette, Jasmine rice, arborio rice and certified flour rice.









Method validation







Method application: Rice flour



Method application: Kombu algae



62 ppm As

Conclusions

Features of HPLC/ICPMS

- The HPLC/ICPMS is the most widely technique for speciation analysis because the connection between them is easy and the ICPMS detector provides excellent detection limits, wide dynamic range and multi-element capabilities.
- But it is an expensive technique and requires a infrastructure (clean room environment...) and qualified technical personnel.

Features of voltammetry with NEE

- Easy to use
- Cheap
- Very sensitive

Work in progress...

- More accurate interference studies
- Matrix effects
- Reproducibility

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Method application

