Invitation for participation in collaborative studies:
Determination of inorganic arsenic (iAs) by SPE-HG-AAS after microwave assisted extraction in marine feeding stuffs

Copenhagen, 10th September 2010

Dear colleague,

You are hereby invited to participate in a collaborative study of a method for the determination of inorganic arsenic in animal feed. The method principles are based on microwave assisted extraction, selective separation by solid phase extraction (SPE) and element-specific determination by hydride generation atomic absorption spectrometry (HG-AAS).

There has recently been drawn much attention on the content of inorganic arsenic in food and feed. Emphasis has from official side been pointing at the need for selective methods for the determination of inorganic arsenic [1, 2]. In the EU directive on undesirable substances in animal feedingstuffs maximum levels for total arsenic are laid down for a range of feed products [3]. However, it is also stressed that the responsible operator should be able to document that the inorganic arsenic concentration is below 2 mg Kg$^{-1}$ [3]. The participation in this collaborative study will provide knowledge to your laboratory on the measurement of inorganic arsenic and furthermore provide a unique set of samples with elevated concentrations of inorganic arsenic to be used for quality assurance purposes.

I hope you will find it attractive to participate in the development of a future European CEN standard method for feed control. Further information can be found in the following pages and if interested please fill in and send the registration form. Your efforts are very much appreciated, thanks in advance.

Best regards,

Dr. Jens J. Sloth (senior scientist)

References:
Introduction:
An international collaborative study will be conducted under the CEN leadership to evaluate a method for the determination of inorganic arsenic (iAs) marine animal feeds. The proposed method was discussed within CEN/TC 327/WG 4. The National Food Institute at the Technical University in Denmark will organize this collaborative trial. CEN members and other interested laboratories are invited to participate.

Principle of the method:
Extraction of inorganic As is done by microwave assisted acidic extraction with a mixture of dilute hydrochloric acid and hydrogen peroxide. Inorganic arsenic is selectively separated from organic arsenic compounds using solid phase extraction (SPE) and thereafter determined by hydride generation atomic absorption spectrometry (HG-AAS).

Samples:
The set-up and execution of the collaborative study will be done according to the IUPAC protocol for the design, conduct and interpretation of method-performance studies [ref ?]. According to this guideline in minimum 5 different samples should be analysed in more than 8 (valid) laboratories.

5-6 marine samples with unknown concentrations (concentration range LOQ - 4 mg Kg\(^{-1}\)) will be sent out for the statistical validation of the CEN methods. Furthermore for lab training one blank and one control sample (with known concentration) and a standard solution will be sent to the participating laboratories. SPE cartridges will be provided together with the samples.

Requirements to the participating laboratories:
The following equipment and reagents should be available at the participating labs.

- Microwave oven - preferably capable of controlling the temperature
- Vacuum chamber for solid phase extraction (Vac-Elut or similar)
  (SPE cartridges will be provided together with the samples)
- HG-AAS apparatus with an arsenic specific lamp
- Reagents: NaBH\(_4\), KI, HCl, H\(_2\)O\(_2\), acetic acid, (NH\(_4\))\(_2\)CO\(_2\), methanol, ascorbic acid
- NOTE: SPE cartridges will be provided together with the samples
Time schedule:
- Dispatch of samples – 8. October 2010
- Discussion of results in CEN TC327/WG 4
- Reports to participants will be sent out after WG discussions

Organisation of the study:
The studies are organised by CEN/TC 327/WG 4. The National Food Institute at the Technical University of Denmark (DTU Food) will be in charge to organize.

The contact address is:
Jens J. Sloth (project leader)
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Mørkhøj Bygade 19
DK-2860 Søborg
Denmark

Participation in the study:
If you want to participate, please send your reply to the email-address indicated (use the attached registration form), before September 30, 2010.

If you have any questions please send a mail to: jjsl@food.dtu.dk or call +45 35887625
Registration form **Collaborative study:**

**Animal feeding stuffs - Determination of inorganic arsenic by SPE-HG-AAS after microwave assisted extraction in marine feedingstuffs**

Yes, I will participate in the CEN/TC 327 collaborative study in feeds for: 

□ Inorganic arsenic by SPE-HG-AAS

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Please send this registration form by mail to: [jjsl@food.dtu.dk](mailto:jjsl@food.dtu.dk)