

Selezione pubblica per titoli ed esami, con eventuale preselezione, per il reclutamento di n.1 posto di personale di categoria D – posizione economica 1 – area tecnica, tecnico-scientifica ed elaborazione dati, da assumere con rapporto di lavoro subordinato a tempo determinato, della durata di dodici mesi, per le attività previste presso il Dipartimento di scienze agroalimentari, ambientali e animali (DI4A) dell'Università degli Studi di Udine (2022_PTA_TD_003)

Ai sensi, per gli effetti e per gli adempimenti previsti dall'art. 19 del D.lgs. 33/2013, n. 33 (*"Riordino della disciplina riguardarne il diritto di accesso civico e gli obblighi di pubblicità, trasparenza e diffusione di informazioni da parte delle pubbliche amministrazioni"*) e successive modificazioni e integrazioni, la Commissione, nominata con Provvedimento Dirigenziale n. 138 del 25/03/2022, riunitasi in seduta preliminare in data 04/04/2022, e così composta:

Presidente	prof.ssa MORET Sabrina	prof.ssa Associata – Dipartimento di scienze agroalimentari, ambientali e animali – Università degli Studi di Udine
Componente	prof. BRAIDOT Enrico	prof. Associato – Dipartimento di scienze agroalimentari, ambientali e animali – Università degli Studi di Udine
Componente	dott. CUDINI Andrea	cat. D – Area tecnica, tecnico-scientifica ed elaborazione dati – Dipartimento di scienze agroalimentari, ambientali e animali – Università degli Studi di Udine
Segretaria	dott.ssa BOSCO Michela	cat. C – Area amministrativa – Dipartimento di scienze agroalimentari, ambientali e animali – Università degli Studi di Udine

COMUNICA

di aver formulato i seguenti quesiti per la prova orale:

QUESITI DELLA BUSTA N. 1

Quesito n. 1: Il candidato illustri quali sono le potenzialità della stampa 3D nell'ambito della chimica analitica.

Quesito n. 2: Il candidato illustri, attraverso qualche esempio pratico, quali sono i vantaggi e svantaggi degli strumenti portatili a basso costo utilizzabili in ambito analitico, rispetto a quelli da banco.

Prova di conoscenza informatica:

Il candidato apra il file Excel presente sul pc denominato "Tabella dati" e proceda alla preparazione di un grafico a dispersione sulla base della serie di dati forniti nel file. Effettui il calcolo della regressione lineare e riporti la formula della retta di interpolazione con il relativo coefficiente di regressione.

Prova di conoscenza della lingua inglese:

Il candidato legga il testo tratto da un articolo scientifico e lo traduca dimostrando di averne compreso il contenuto (si allega testo n. 1).



Comparison of extraction procedures for methylmercury determination by a SPME-GC-AFS system

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Abstract

In this study the comparison of three different alkaline extraction techniques carried out with methanolic NaOH followed by phenylation derivatization for the determination of methylmercury in marine fish was performed. The investigated three methods differed in the technique how the extraction assisting energy was introduced to the sample. Namely, closed vessel ultrasonic bath, an open vessel technique using an ultrasonic probe and the microwave assisted extraction procedures were characterized and optimized. Optimum values of 3 h at 75 °C for the ultrasonic bath, 25 min for the ultrasonic probe and 6 min at 60 W for the microwave method were obtained. All three methods were validated using the BCR-464 tuna fish certified reference material.

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Keywords: Methylmercury; Fish; Extraction; Ultrasonic bath; Ultrasonic probe; Microwave

1. Introduction

As it is well known, when speciation analysis is aimed the preservation of the original forms of the species present in the sample during the entire measurement is a key issue [1]. Among the steps that are generally performed from the sample collection stage until the analysis presumably, extraction of the analyte from the sample matrix is the most critical one. On one hand, extraction efficiencies very close to 100% are a basic requirement in order to be able to identify the entire species pattern of a given sample. On the other hand the more drastic extraction technique is applied for obtaining better extraction efficiencies the higher the risk of destroying the original species pattern [2]. That is the reason why several extraction techniques have been developed during the years among which ultrasonic energy assisted procedures have become the most widespreadly used ones [3–7]. In this study two different ultrasound assisted extraction procedures were characterized, namely the ultrasonic bath method and a relatively new technique using an ultrasonic probe. The ultrasonic probe (or commonly termed as ultrasonic processor) is basically consists of a metal rod (sonotrode)

vibrating at in the range of tens of kHz with an amplitude of a few hundreds of micrometers. Depending on the instrument type the amplitude and the pulse range can be adjustable. During operation the sonotrode is immersed into the sample, directly transmitting the ultrasonic waves into the solution.

Apart from the ultrasonic assisted extraction technique a microwave energy based procedure was also applied. In this case the energy indirectly irradiates the sample via the wall of the sample container vial such in the case when ultrasonic bath is used. The extraction efficiencies and especially the minimal required time for obtaining satisfactory (i.e. 100%) efficiencies are strongly dependent of the techniques and of course of the doses applied. In this work the comparison of the mentioned three techniques for methylmercury extraction capability of fish samples at different doses was carried out.

2. Materials and methods

2.1. Instrumentation

A manual solid phase microextraction (SPME) device (Supelco, Bellefonte, PA, USA), equipped with a fused silica fiber coated with a 100- μ m film of poly(dimethylsiloxane) (PDMS) was used for extractions in all experiments. GC

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QUESITI DELLA BUSTA N. 2

Quesito n. 1: Il candidato discuta criticamente le potenzialità delle tecnologie “open source” (hardware e software) in ambito analitico.

Quesito n. 2: Il candidato illustri l'impiego della tecnologia della stampa 3D in ambito analitico, con particolare riferimento ai diversi materiali che possono essere impiegati in fase di stampa.

Prova di conoscenza informatica:

Il candidato apra il file Word presente sul pc denominato "Testo" ed effettui la formattazione del testo utilizzando il font Arial 14 punti, un valore di interlinea doppia e un allineamento giustificato. Inserisca il numero di riga e nell'intestazione indichi la denominazione del Dipartimento DI4A.

Prova di conoscenza della lingua inglese:

Il candidato legga il testo tratto da un articolo scientifico e lo traduca dimostrando di averne compreso il contenuto (si allega testo n. 2).

Improvement of the BCR three step sequential extraction procedure prior to the certification of new sediment and soil reference materials



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The Standards, Measurements and Testing Programme (formerly BCR) of the European Commission proposed a three-step sequential extraction procedure for sediment analysis, following extensive expert consultations and two interlaboratory studies. This scheme was recently used to certify the extractable trace element contents of a sediment reference material (CRM 601). Although this procedure offers a means to ensure the comparability of data in this field, some difficulties concerning the interlaboratory reproducibility still remain, and a new project is currently being conducted to determine the causes of poor reproducibility in the extraction scheme. The final objective of the project is the certification of new sediment and soil reference materials for their extractable contents of Cd, Cr, Cu, Ni, Pb and Zn. This paper presents the results of a small-scale interlaboratory study, which aimed to test a revised version of the extraction schemes by comparing the original and the modified protocols using the CRM 601 sample. This work offers an improvement to the BCR sequential extraction procedure through intercomparison exercises. This improved procedure will allow the obtaining of CRMs to validate analytical data in the analysis of soils and sediments, and it will also facilitate comparability of data in the European Union.

Introduction

Environmental studies on soil and sediment analysis are often based on the use of leaching or extraction procedures (e.g. single or sequential extraction procedures) which enable broader forms or phases to be measured (e.g. 'bioavailable' forms of elements) and which are, in most cases, sufficient for the purpose of environmental policy.¹ The development and use of these types of extraction schemes started in the early 1980s and aimed to evaluate the metal fractions available to plants (for estimating the related phytotoxic effects and/or nutritional properties of elements) and the environmentally accessible trace metals (evaluation of the mobility of metals).²⁻⁴ The lack of uniformity in the procedures used did not allow the results to be compared world-wide or the methods to be validated since the results obtained are 'operationally defined', i.e. the 'forms' of metals are defined by the determination of extractable elements and, therefore, the significance of the analytical results is highly dependent on the extraction procedures used. In order to improve this situation, the European Commission through the BCR Programme and its successor (Standards, Measurements and Testing Programme) launched a collaborative project which aimed to (1) design a three-step sequential extraction scheme, (2) test the selected scheme in interlaboratory studies involving expert European laboratories and (3) certify the extractable trace element contents of a sediment reference material. This project has been extensively described in the literature.⁵⁻⁸

Recent findings have shown that the three-step sequential extraction scheme is not without difficulties and that research is necessary to identify the causes of poor reproducibility. A

follow-up of the above-mentioned project was selected for funding by the European Commission in 1996⁹ in order to investigate the causes of error and test an optimised version of the protocol in an interlaboratory study prior to certification of new sediment and soil reference materials. This paper presents the results of the collaborative work to improve the so-called BCR sequential extraction scheme.

Design of the study

Initially, a group of European experts (see Acknowledgements) met to discuss the necessary amendments to the so-called BCR sequential extraction scheme. Following these discussions, a modified protocol was proposed for interlaboratory testing, using CRM 601 as a reference material. This material was certified for extractable contents of various elements (see Table 1) and was shown to be stable over a long-term period.¹⁰ Each laboratory participating in the study received four bottles of CRM 601 and was requested to apply both the original sequential extraction scheme and the modified protocol (five replicate analyses to be performed on two different days, for each protocol, for each of the trace metals Cd, Cr, Cu, Ni, Pb and Zn; in addition, laboratories were supplied with six bottles containing about 200 mg l⁻¹ calibrant solutions of the metals to be determined). This procedure allowed the testing of the repeatability of the extraction methods, taking into account the homogeneity variance in the material (five replicate analyses in two different bottles) and their reproducibility (analyses carried out on two separate days); in addition, the analysis of calibrant solutions allowed the identification of possible problems occurring in the measurement step (hence avoiding

di aver formulato i seguenti quesiti per la prova pratica:

QUESITI DELLA BUSTA N. 1

Quesito n. 1: Il candidato illustri lo sviluppo in un dispositivo ottico per misure in riflettanza.

Quesito n. 2: Il candidato faccia una dimostrazione pratica di stampa di un dispositivo paper-based per applicazioni analitiche.

QUESITI DELLA BUSTA N. 2

Quesito n. 1: Il candidato illustri lo sviluppo in un dispositivo ottico per misure in assorbanza.

Quesito n. 2: Il candidato faccia una dimostrazione pratica di una stampa di un componente per un dispositivo analitico.

Si comunica altresì che il candidato, ha estratto per la prova orale la busta n. 2 e per la prova pratica la busta n. 1

Dei quesiti non estratti è stata data lettura.

Udine, 13/04/2022

La Presidente della Commissione esaminatrice
Prof.ssa MORET Sabrina

