Oceanografia Química - Práticas

Aula 04

Profa. Juliana Leonel

Ténicas Instrumentais

- FAAs
- GFAAs
- ICP OES
- ICP MS
- Cromatografia

Em todas essas técnicas as amostras são usualmente introduzidas no estado líquido.

1. Definição do problema analítico
2. Escolha do método de análise
3. Amostragem
4. Processamento (tratamento) da amostra
5. Medida analítica
6. Avaliação dos resultados
7. Ação

Preparo da Amostra

Obtenção de soluções a partir da amostra ambiental.

→ (ainda) é a parte mais cara: tempo, dinheiro e esforço.

→ determina (junto com a amostragem) o tempo máximo para execução de um análise.

Por que a etapa de preparo de amostras é um "problema"?

Qual a melhor forma de preparar uma amostra?

Qual a melhor forma de preparar uma amostra?

→ Preparo nenhum (análise direta)

→Apenas diluição (“Dilua-Determine”)

→ Mínimo de preparo (possível)

Sociedade Brasileira de Química (SBQ)

Determinação direta de Cu, Cd, Ni e Pb em substâncias húmicas aquáticas por espectrometria de absorção atômica em forno de grafite

Danielle Goveia[1,2] (PG), Adriana Paiva de Oliveira [3*] (PQ), Fabiana Aparecida Lobo[1,2] (PG), André Henrique Rosa [1] (PQ), Ricardo Dalla Villa[3] (PQ) *dri_poliv@yahoo.com.br

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Palavras Chave: *metais, GFAAS, substâncias húmicas aquáticas.*

ELSEVIER

Aquatic Toxicology 67 (2004) 127–142

AQUATIC TOXICOLOGY

www.elsevier.com/locate/aquatox

Rapid assessment of polycyclic aromatic hydrocarbon (PAH) exposure in decapod crustaceans by fluorimetric analysis of urine and haemolymph

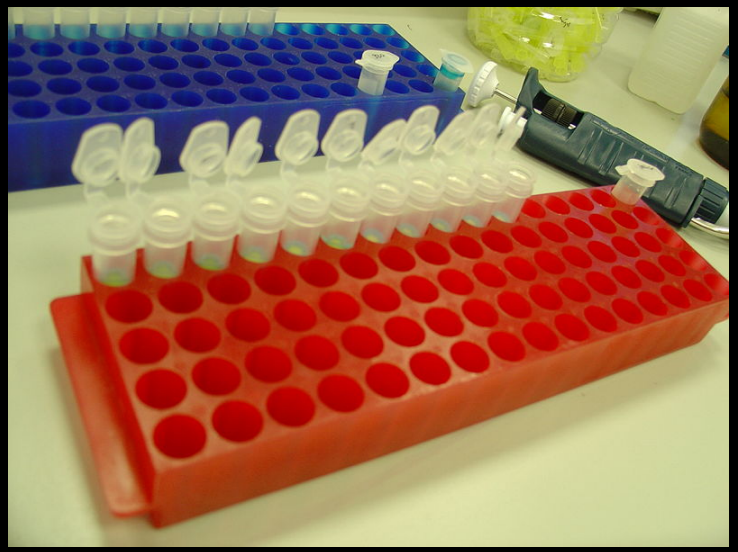
Giles M. Watson [a,*], Odd-Ketil Andersen [b], Tamara S. Galloway [a], Michael H. Depledge [a,c]

2.5. *Urine and haemolymph sampling*

In the exposure–response experiment, urine and haemolymph samples were taken from each crab after 48 h exposure using the technique described by Bamber and Naylor (1997). Briefly, crabs were re-

2.6.5. *Urine and haemolymph samples*

Urine (diluted 1:100) and haemolymph (diluted 1:20) samples from exposed and control crabs were analysed for pyrenes as described above. Fluorescence intensity was measured at the assigned wavelength pair of Ex345/Em382 nm (FF) and Em381.4 nm (SFS) and expressed as $\mu g\, l^{-1}$ of 1-OH pyrene equivalents following comparison to standards. Before analysis of either standards or samples, a blank of 50% ethanol was scanned by FF and SFS. This provided a value for the background fluorescence contribution of the solvent, for comparison with samples and standards.



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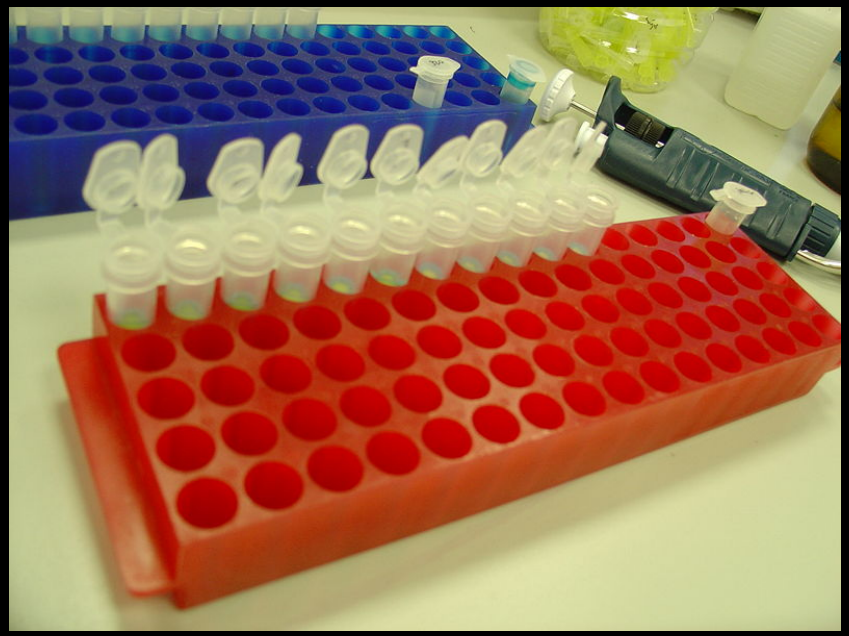
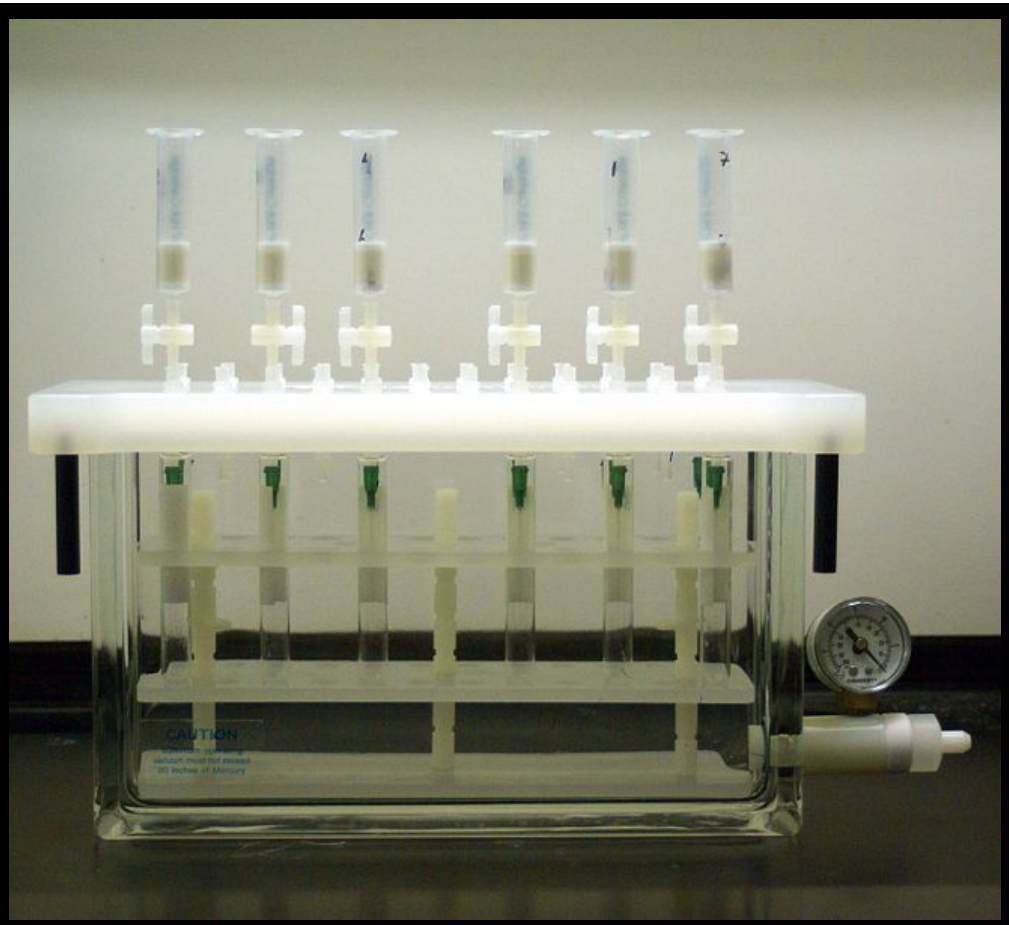


http://commons.wikimedia.org/wiki/File:Gradillas.JPG

2.6.7. GC/MS analysis of water samples

To investigate the uptake of waterborne pyrene by exposed crabs, a series of tanks were set up in duplicate (one containing crabs, the other without). Pyrene was added to the water at the same nominal concentrations as for the exposure experiment and water samples taken at 0, 2, 6, 12, 24, 48 and 96 h. Samples were analysed for parent PAH using GC/MS analyses (Hewlett-Packard Model 5890 II Plus GC and a 5972 mass selective detector (MSD) (Palo Alto, CA)). Internal standard spiked (pyrene-d_{10}) water samples (100–500 ml) were concentrated using C_{18} cartridges (IST, Hengoed, UK), which were subsequently eluted (three times) using 3 ml of ethyl acetate. The eluent was then concentrated down to 1 ml before analyses by GC/MS.



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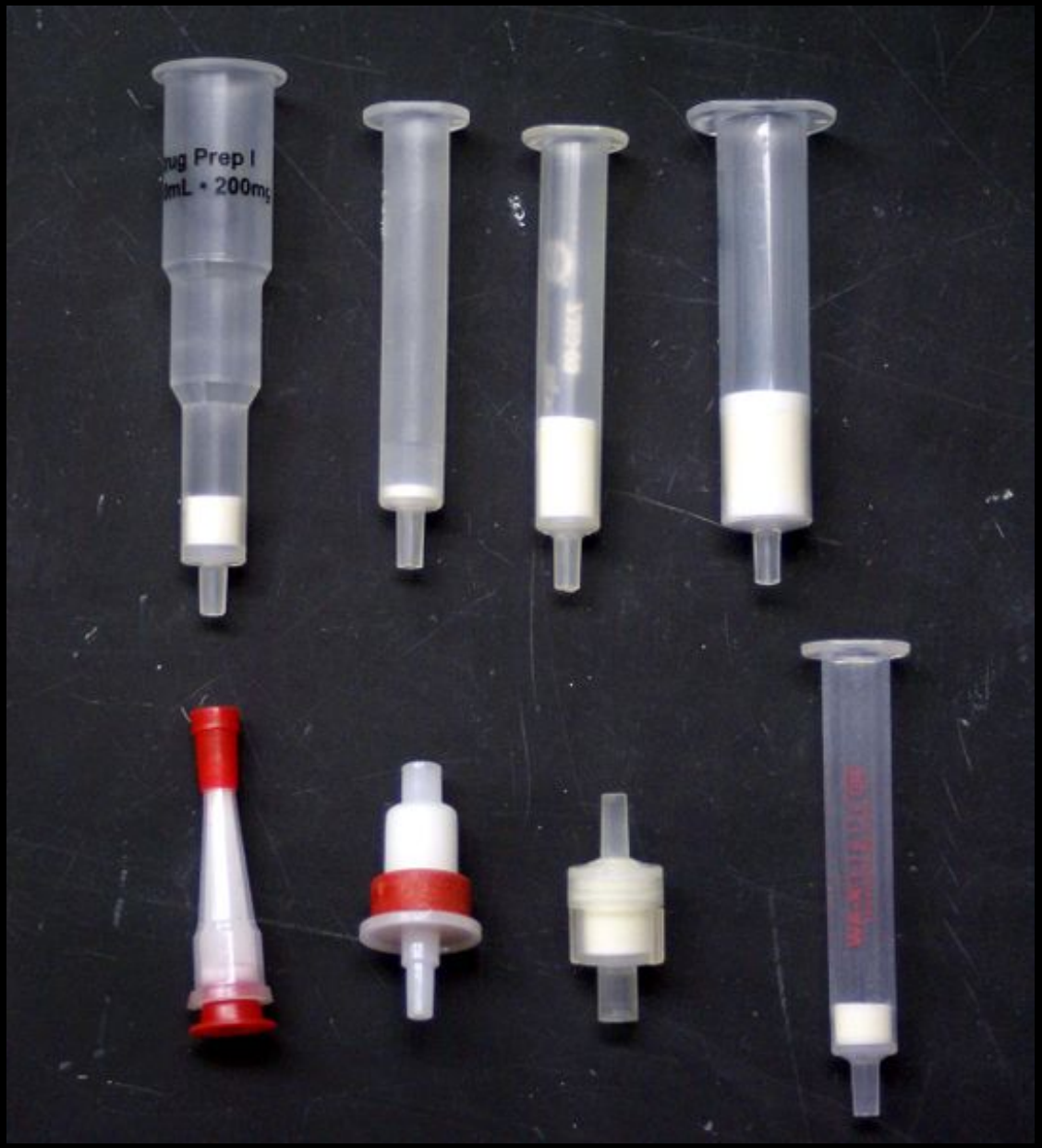

http://en.wikipedia.org/wiki/File:SPE_Cartridges.jpg


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Chemosphere 86 (2012) 741–746

Contents lists available at SciVerse ScienceDirect

Chemosphere

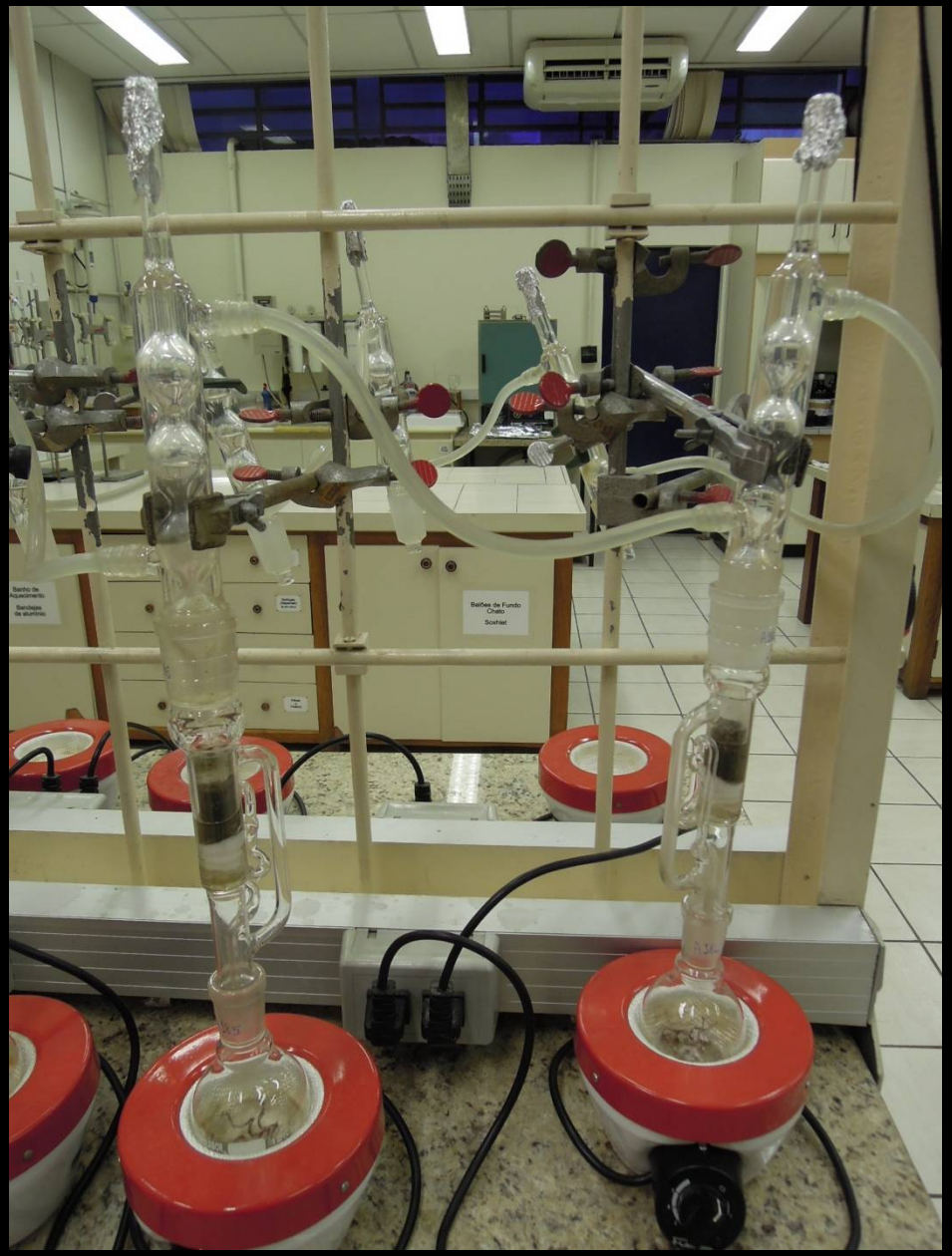
journal homepage: www.elsevier.com/locate/chemosphere

Contamination by chlorinated pesticides, PCBs and PBDEs in Atlantic spotted dolphin (*Stenella frontalis*) in western South Atlantic

Juliana Leonel [a,*], Satie Taniguchi [a], Dalton K. Sasaki [a], Mauro J. Cascaes [a], Patrick S. Dias [a], Silvina Botta [b], Marcos C. de O. Santos [c], Rosalinda C. Montone [a]

2.3. Chemical analysis

The analytical procedure employed was based on MacLeod et al. (1985). Briefly, after the addition of anhydrous Na_2SO_4, approximately 0.5 g of blubber tissue was extracted with methylene chloride and n-hexane (1:1) using a Soxhlet apparatus. Prior to extraction, 2,2′,4,5′,6-pentachlorobiphenyl (PCB 103) and 2,2′,3,3′,4,5,5′,6-octachlorobiphenyl (PCB 198) were added to samples, blanks and reference material (SRM 1945 from the National Institute of Standards and Technology) as surrogates for chlorinated pesticides, PCBs, and PBDEs. Extracts were initially cleaned using 5% deactivated silica:alumina column chromatography eluted with a 1:1 mixture of *n*-hexane and methylene chloride. The fraction was further purified by high-performance liquid chromatography to remove excess lipids and concentrated to a volume of 1.0 mL in hexane. An internal standard (2,4,5,6-tetrachlorometaxylene) was added prior to gas chromatographic analysis. Chlorinated pesticides were analysed through gas chromatography (GC) using an electron capture detector. PCBs and PBDEs were quantitatively analysed through a gas chromatograph coupled to a mass spectrometer (GC–MS) in a selected ion mode. Target persistent organic pollutants (POPs) were quantified with calibration curves generated from five standard solutions. Lipid weights were determined gravimetrically.

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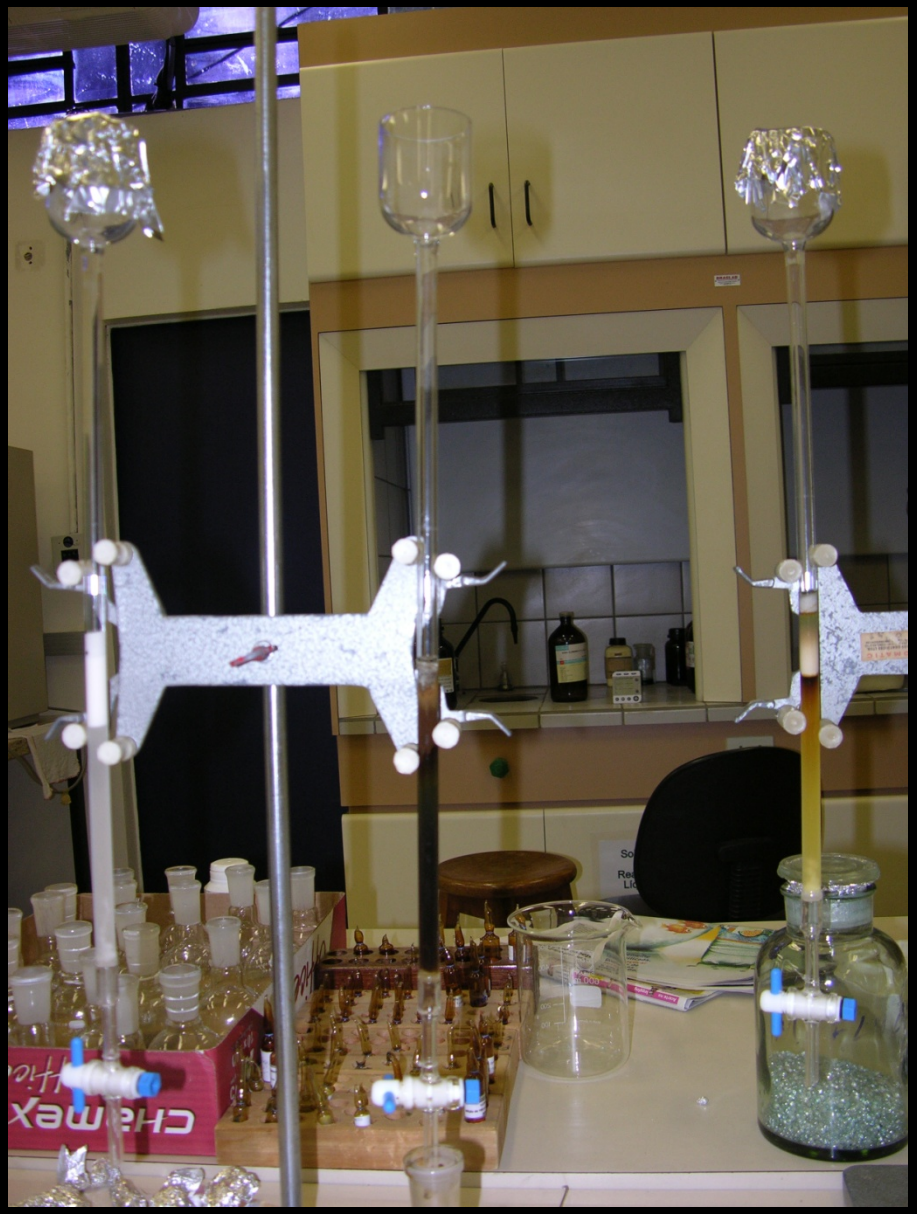


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Marine Pollution Bulletin 64 (2012) 2603–2614

Contents lists available at SciVerse ScienceDirect

Marine Pollution Bulletin

journal homepage: www.elsevier.com/locate/marpolbul

Baseline

Overview of the 20th century impact of trace metal contamination in the estuaries of Todos os Santos Bay: Past, present and future scenarios ☆

Vanessa Hatje [a,*], Francisco Barros [b]

Sediment samples were divided into two parts, the first used for the determination of particle-size distribution, and the second for chemical analyses. Before chemical analyses, sediments were wet sieved to separate the fraction smaller than 63 μm, freeze-dried, homogenized and comminuted in a ball mill. An extraction using 20 ml of 1 M HCl, shaking for 12 h at room temperature, was carried out with sediments and SPM samples. Sediment and SPM samples were extracted in triplicates. Elements were determined using ICP OES (Varian, VISTA-PRO). Blanks were included in each batch of analysis. The precision and accuracy of the analytical technique were assessed using a CRM, MESS-2 (National Research Council of Canada) with each batch of samples. As expected, results indi-

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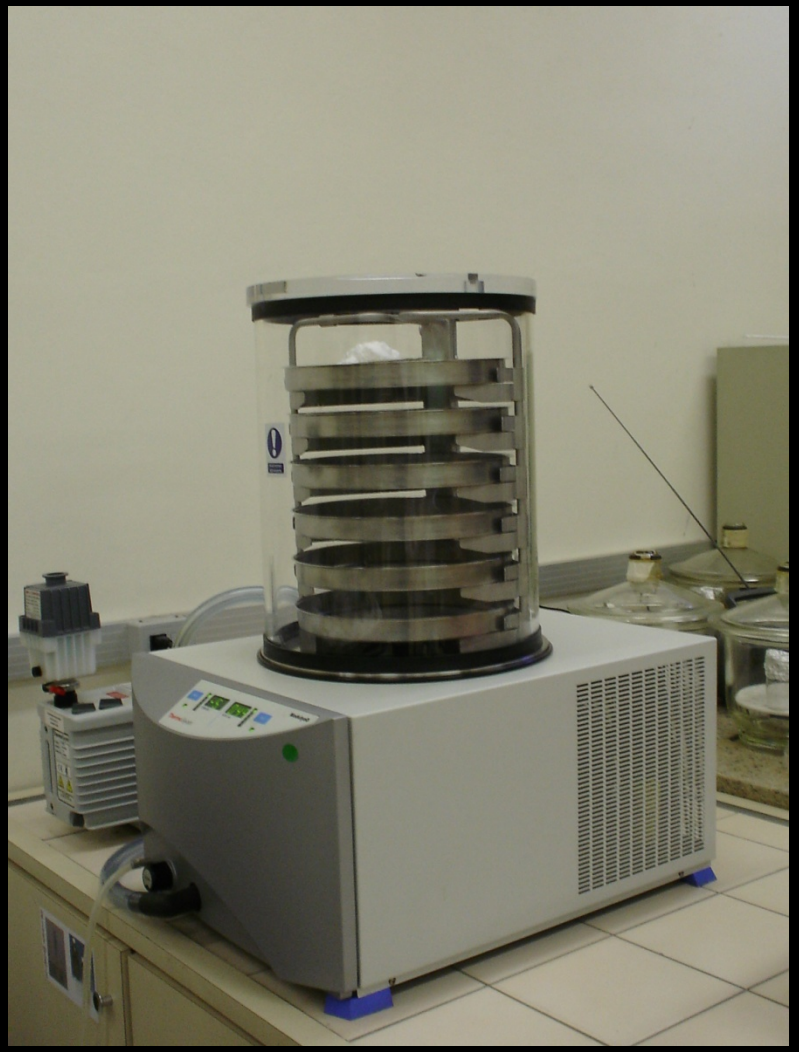

Pré -Tratamento da Amostra

-todos os procedimentos para o preparo da amostra no laboratório

- escolha do pré-tratamento depende do estado inicial da amostra

- a maioria das técnicas analíticas necessita um pré-tratamento

Pré -Tratamento da Amostra – Por quê?

- homogeneização

- dissolução de materiais sólidos

- separação de substâncias interferentes

- pré-concentração dos analitos

Pré -Tratamento da Amostra

Operações preliminares (geralmente físicas)

- moagem
- secagem
- filtração
- centrifugação
- destilação

Preservação das Amostras

- liofilização
- acidificação
- adição de mercúrio
- anticoagulantes
- refrigeração/congelamento

Homogeneização

- etapa importante

- obter uma amostra representativa

- análises em replicata testam a homogeneização.

- atenção com a contaminação

Moagem

- graal e pistilo

- liofilizador (pulverização)

- moinho de bolas

Homogeneização

- graal e pistilo

- liofilizador (pulverização)

- moinho de bolas

Métodos de Preparo de Amostras

Técnica Analítica

Espectroscópia → decomposição (digestão) da amostra → parcial ou total → aquecimento ou agitação ou reagentes ácidos e/ou alcalino

Cromatografia → extração → líquido-líquido, fase sólida, soxhlet, microondas → purificação → fracionamento

DÚVIDAS???