

Novas tecnologias na Análise de Contaminantes : do Conhecido ao desconhecido

POP'S

25

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Sistemas Analíticos, Lda.

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Sistemas Analíticos, Lda.

Espectrometria Cromatografia

Enquadramento

- Novas directivas comunitárias
- Novas directivas internacionais
- Iniciativas Globais de diminuição da poluição
- Aparição de novas tecnologias
- Aumento do consumo alimentar e do intercambio global : mais amostras
- Necessidade de harmonização

Exemplos normativos

- Directiva SANCO para controlo de pesticidas e contaminantes em alimentos
- Novos Métodos para pesticidas polares
- Normativa da FDA para controlo de metais (USP e ICH 3)
- Controlo agro-veterinario
- Mercury Initiative
- Grupos de trabalho para Poluição por plástico e Microplásticos

Necessidades

- Preparação de amostra mais expedita
- Actualização tecnológica associada aos novos (menores) limites de detecção
- Análise inequívoca
- Investigação de NOVOS contaminantes e de NOVOS metabolitos destes
- Cumprimento de normativas nos países de destino

Contaminantes

- **Metais**
- **Material de origem orgânica (POP's)**
- **Não metais**
- **Microplásticos e associados**
- **Materiais por descobrir**

Metais

- Diminuição dos limites de detecção
- Alimentos para crianças e bebés com valores de metais contaminantes muito baixos
- A amostra TEM que ser previamente digerida

- Hg, Pb, Cd, As
- Mn, Cr, Sn, etc.
- Se, Co, etc.

ANALYSIS

thermo scientific

NEW



AA

Flame
Furnace

ICP

CID
Axial
Radial
Duo

ICPMS

Quads

Triple Q

Col. Cell

HR- ICPMS

Magnet
GD
TIMS

IC

Cations
Anions
Ion
Supress

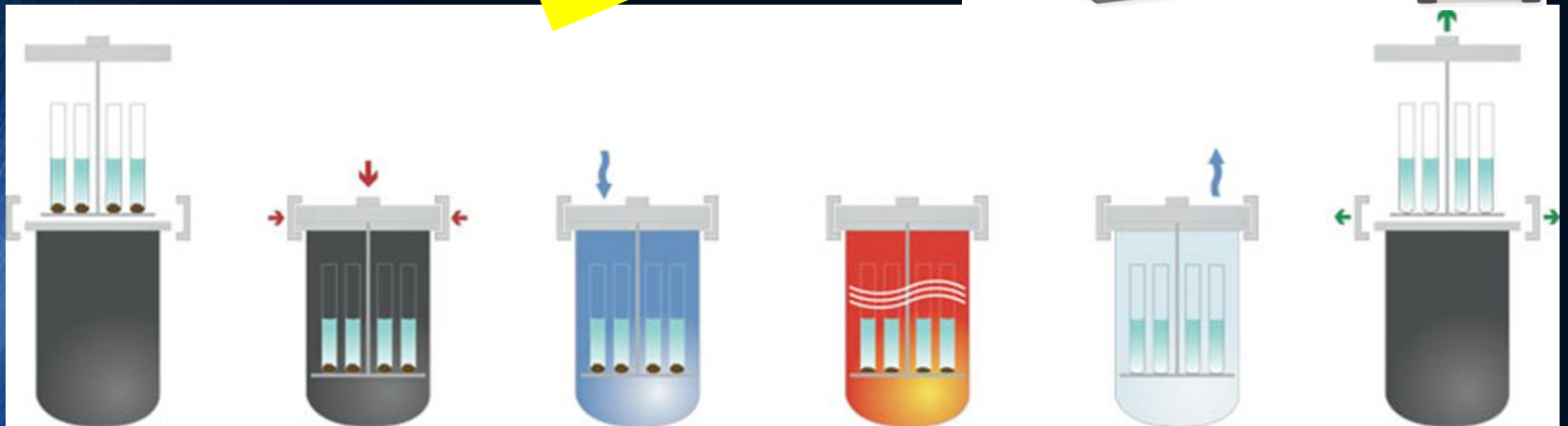
Sample Prep

- **Metais : Digestão de amostras**
- **Necessidade de fazer digestões mais rápidas e de usar um método único para todas as matrizes**
- **A Digestão por Microondas é uma das metodologias estabelecidas na digestão de alimentos**

Ultrawave : Digestão por Micro-ondas

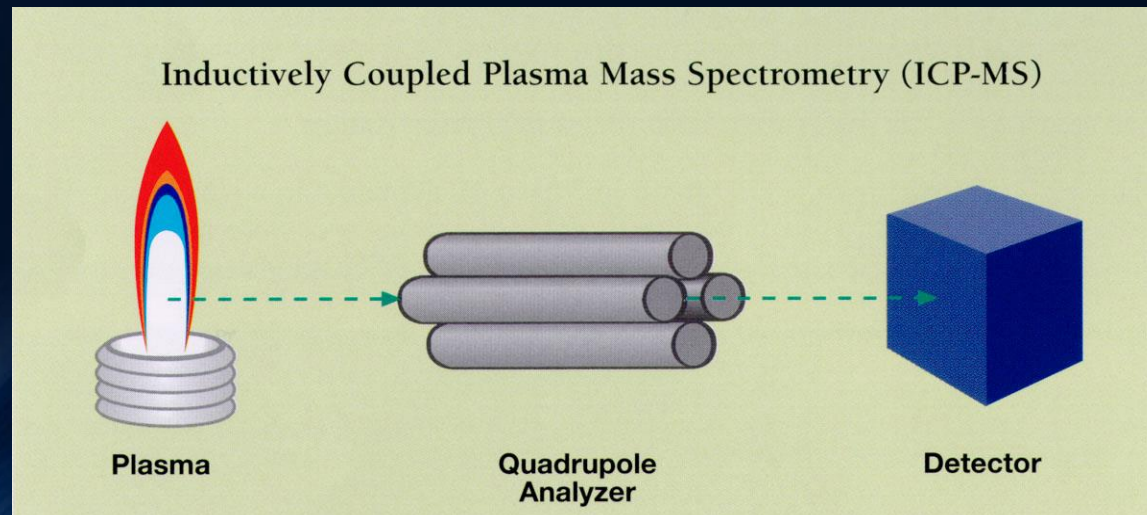
- Single Reaction Chamber
 - 4 times faster than conventional Microwave digestions
- Microwave digestions

NEW



ICP - ICPMS

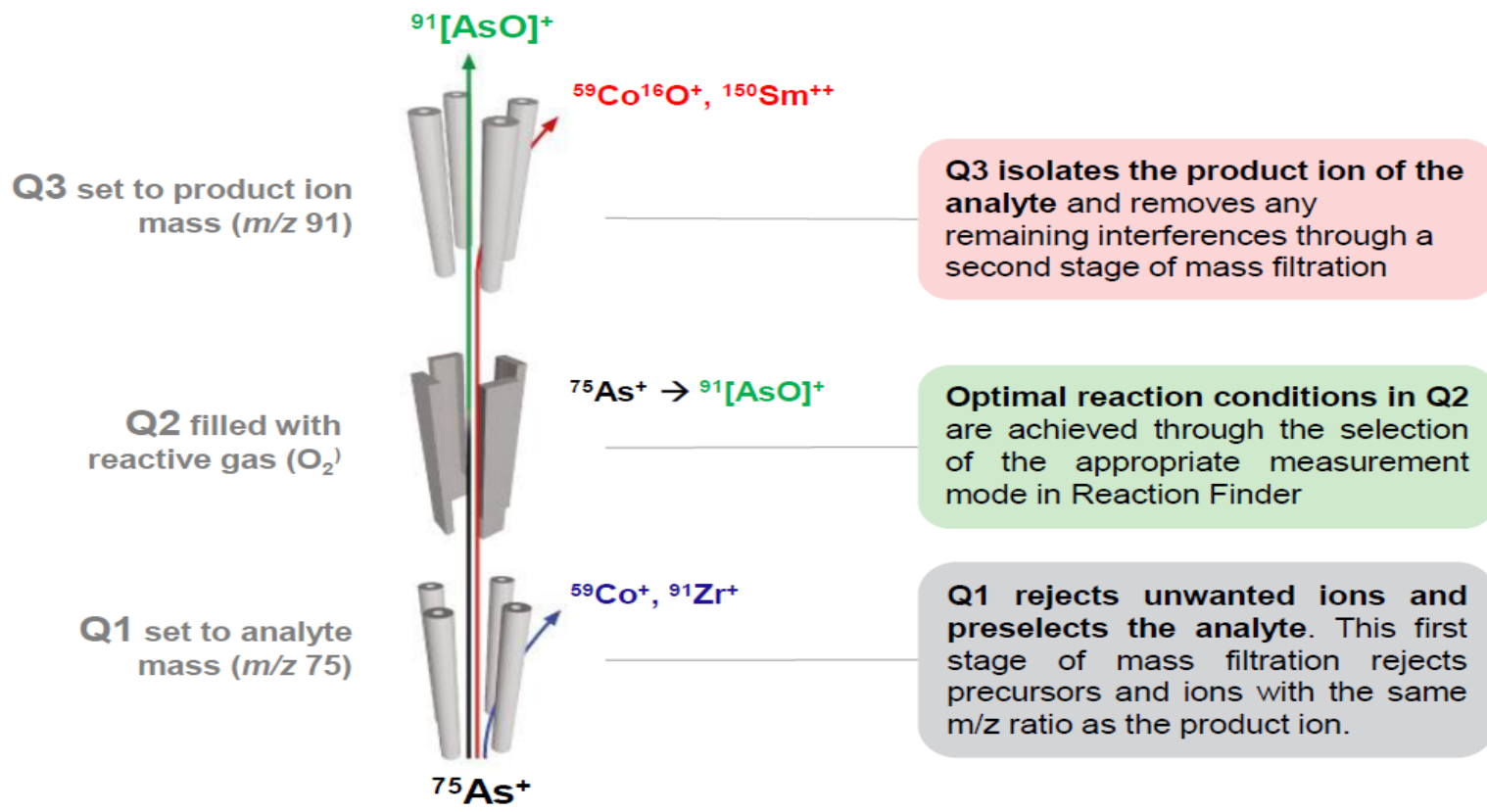
- Técnicas Multielementares : todos os metais numa análise só
- A diminuição dos limites de detecção obriga a utilização de ICPMS
- O ICP ótico pode ser complementar para a análise dos macro e semimicro nutrientes



ICPMS ICAP TQ

NEW

- ICAP TQ : Célula de colisão de ultima Geração
- Tres quadrupolos : Triple Quad Technology
- As = 75 CoO = 75 and AsO=91 Zr= 91



Easiest Method development

Without Reaction Finder

Select

- Select the Analytes to be measured

Select

- For each analyte, select the isotopes to be measured

Select

- Select the internal standard element

Select

- Select the Q1 Analyte

Select

- Select the CRC gas (None, He, H₂, O₂, NH₃)

Select

- Select the mode (KED, Single Quad Mode, Triple Quad Mode)

Select

- Select the Q3 Mass (On-mass/mass shift product ion)

Decide

- Are the suggested settings ok? If not, update them

Analyze

- Enter sample names and positions or import from LIMS and start the LabBook

With Reaction Finder

Select

- Select the Analytes to be measured

Select

- Select the internal standard element

Decide

- Are the suggested settings ok? If not, update them

Analyze

- Enter sample names and positions or import from LIMS and start the LabBook



Análise Directa de Mercurio

**NEW:
Triple Cell**

Peixe
Leite
Leite em pó
Solos



Organic Contaminant and Pollutants

- LCMSMS
- GCMSMS
- High Resolution Mass Spectrometry

THE ORBITRAP

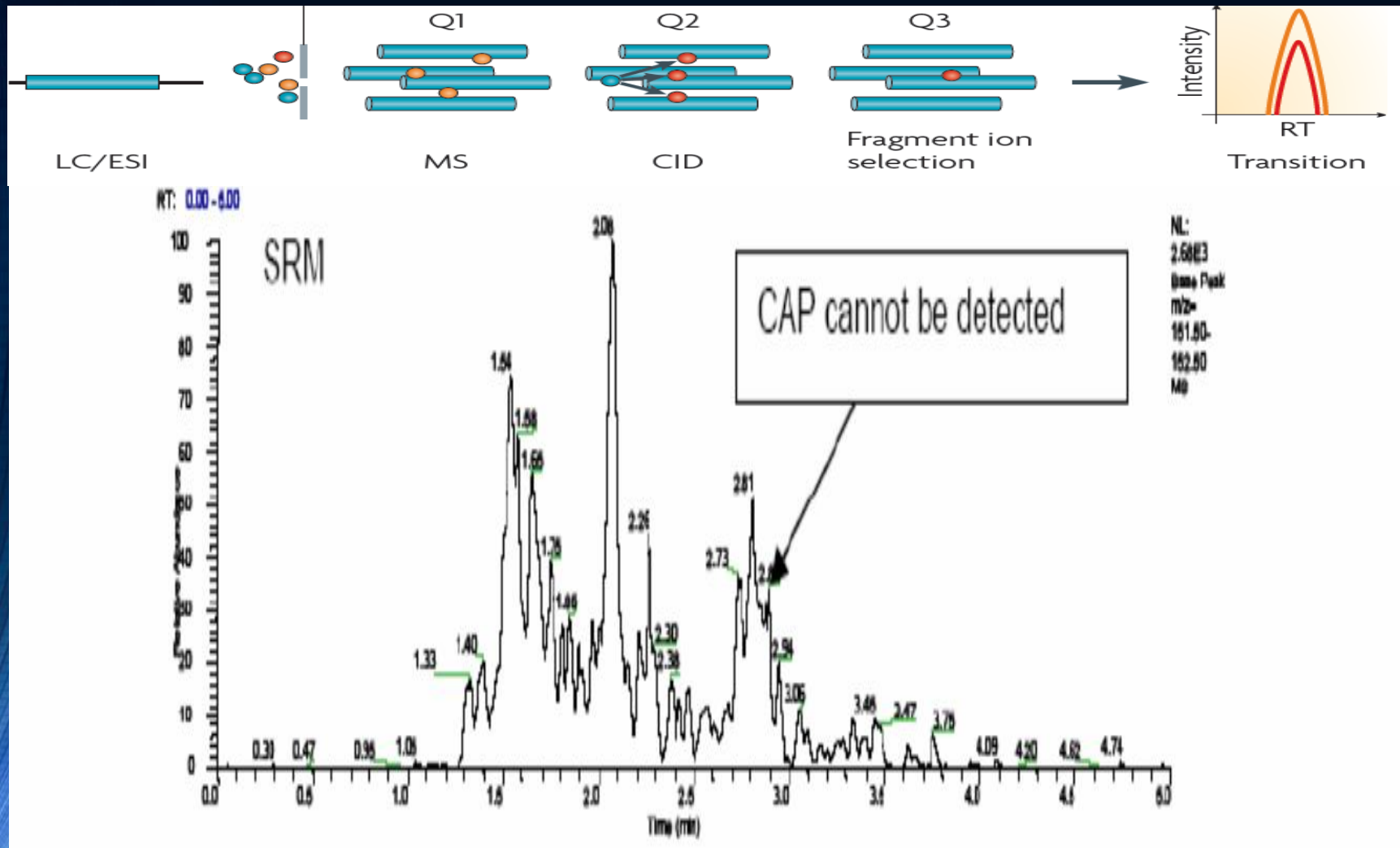
NEW

Sample Prep : Extract FASTER

- Higher Pressure / Higher Temperature extraction
 - ASE 350
 - Milestone Microwave Extraction Workflow

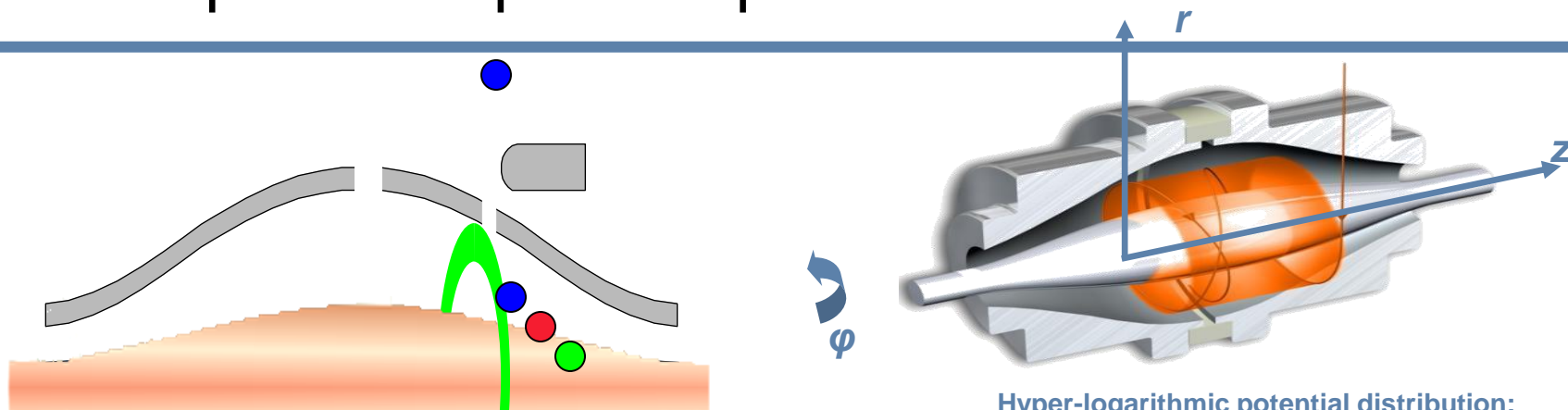


Triple Quad Technology limitations



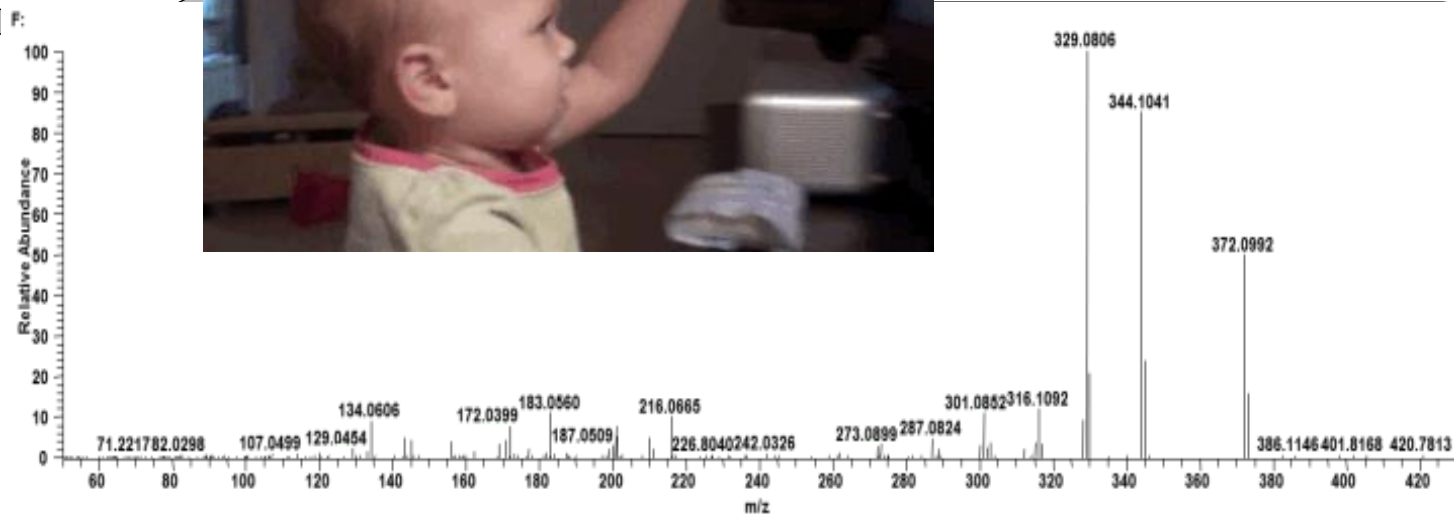
From : Chloramphenicol in Meat/ Pork Liver Reference : FDA Research Center

Orbitrap – Principle of Operation



Hyper-logarithmic potential distribution:
"ideal Kingdon trap"

$$-r^2 / 2 + R_m^2 \cdot \ln(r / R_m)$$

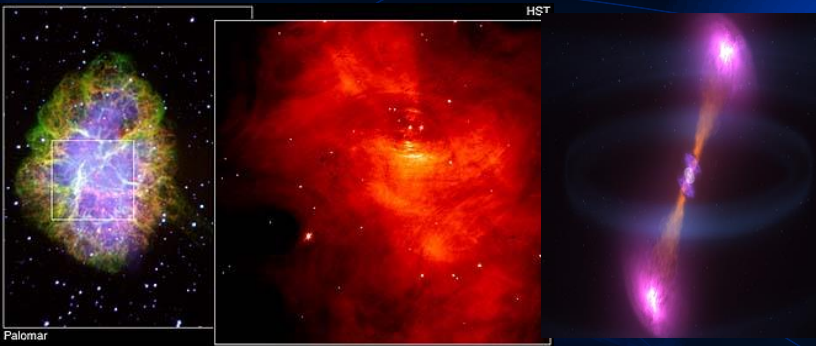


Alexander Makarov

Makarov A. *Anal. Chem.* 2000, 72, 1156-1162.

Orbitrap : LC e GC





C = 12.0000

H = 1.0078

N = 14.0031

O = 15.9949

S = 31.9721

Exact Mass

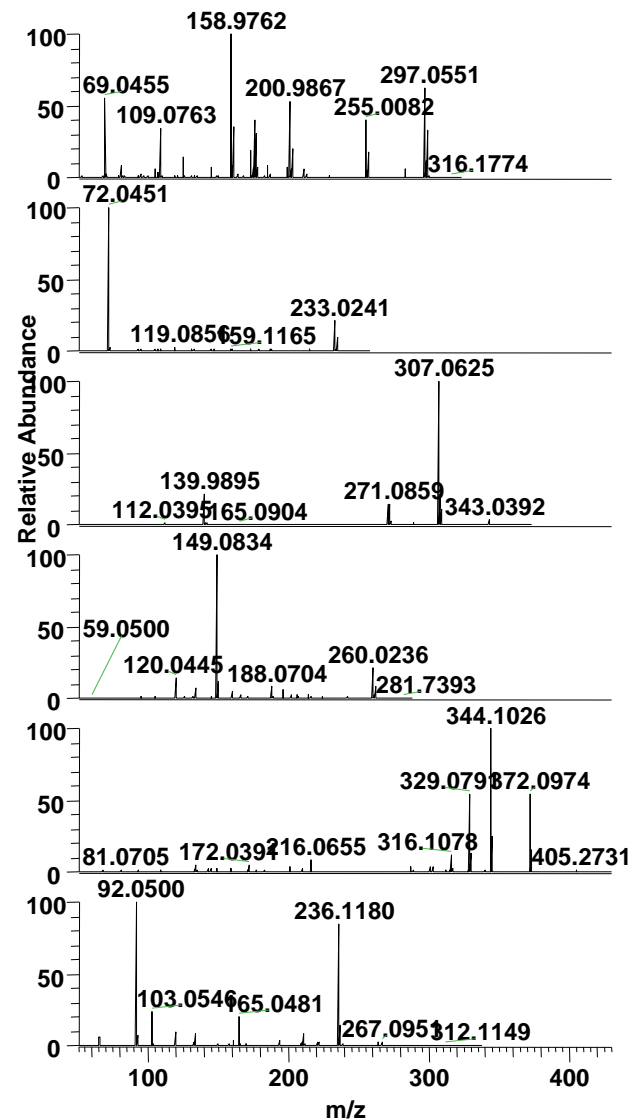
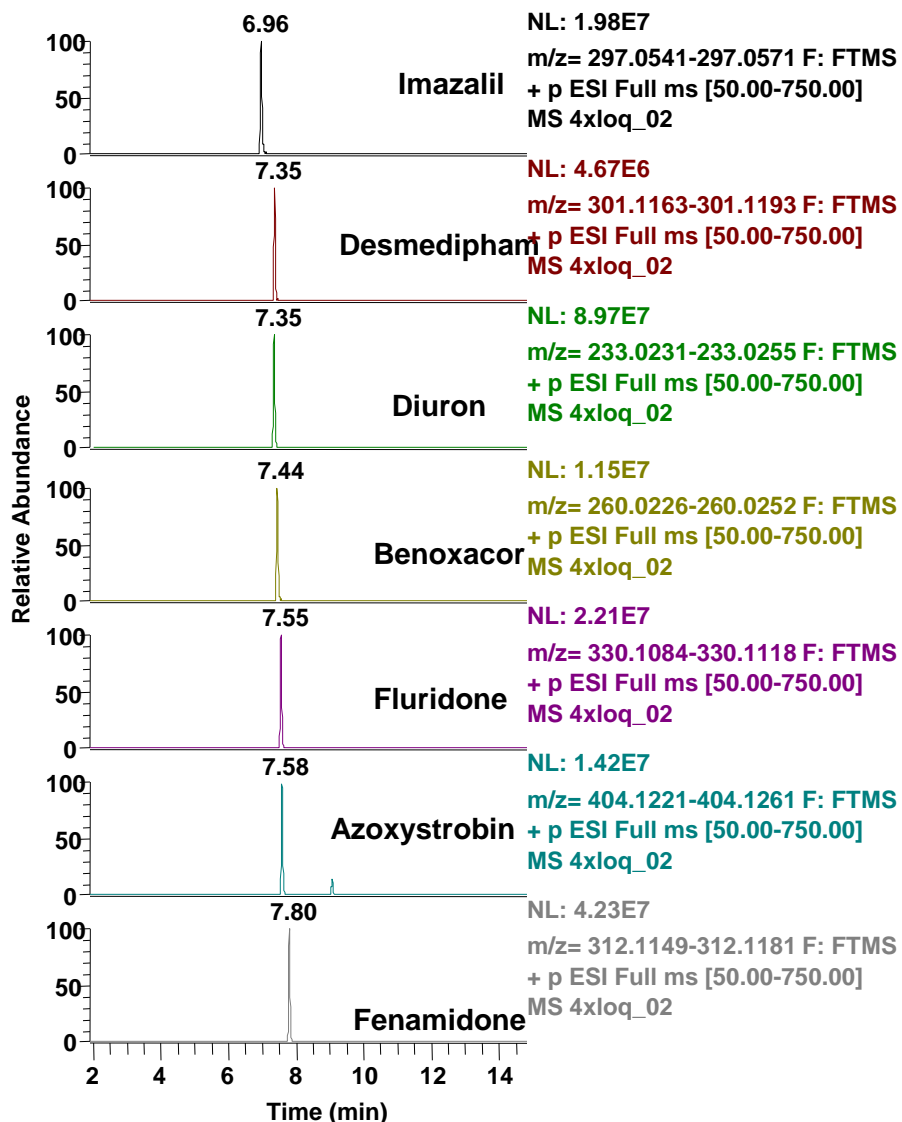
Measured Mass	Tolerance	Suggestions
32.0	+/- 0.3	$^{16}\text{O}_2$ $^{12}\text{C}^1\text{H}_3^{16}\text{O}^1\text{H}$ $^{14}\text{N}_2^1\text{H}_4$ ^{32}S

PORQUE Alta resolução?

Confirmação Inequivoca

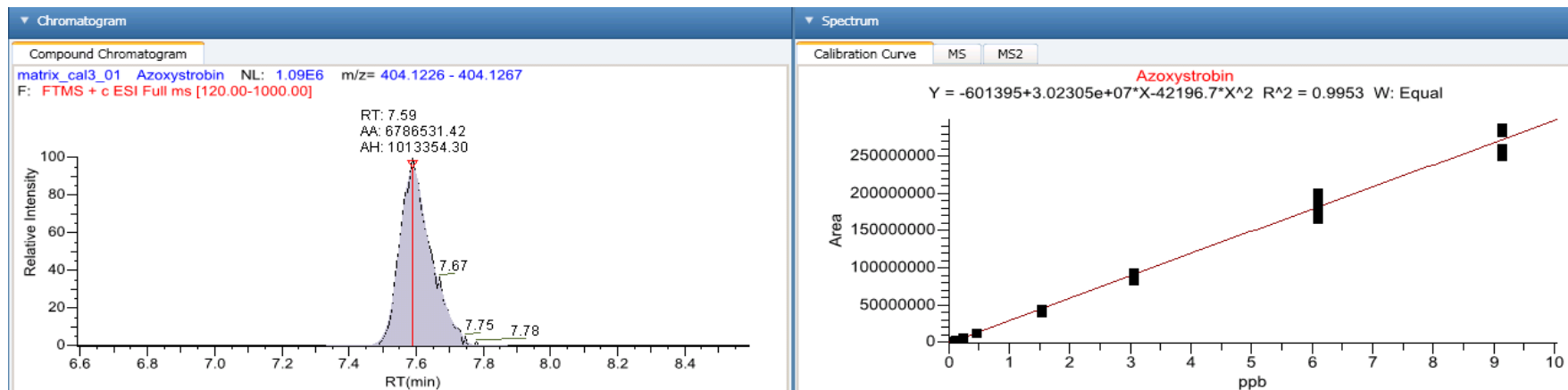
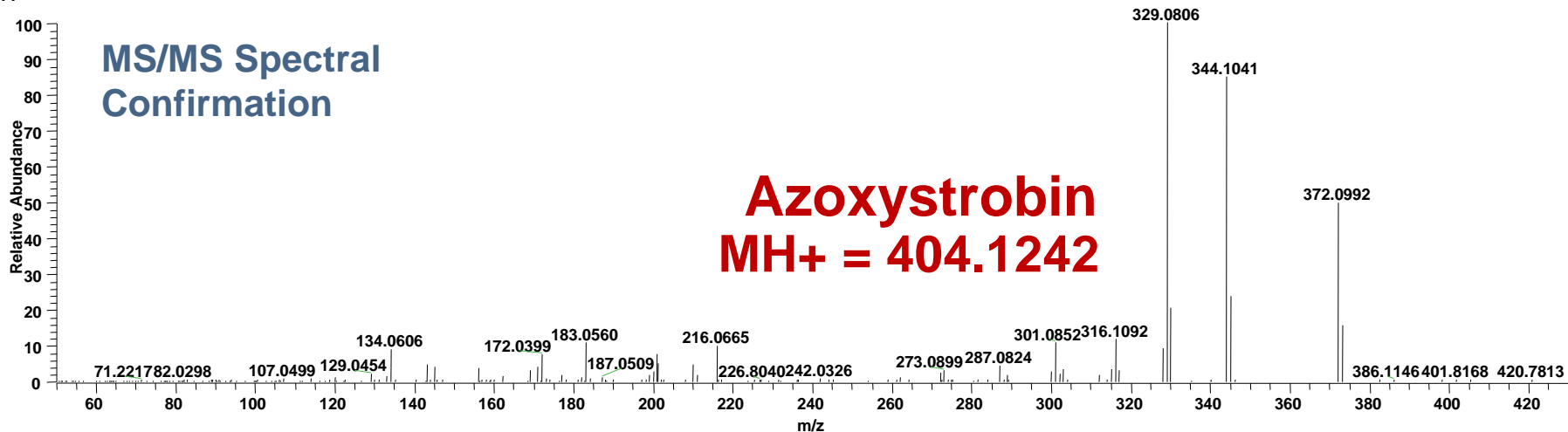
- **Massa Exacta (Resolução MAIOR que 70000)**
 - **Espectro MSMS em ALTA Resolução**
 - **Sistemas Cromatograficos separativos**
- Nota : Multiplos compostos (mais de 600)

Identification and MS/MS Confirmation



Quantification of Azoxystrobin 1.5 ppb (MSMS)

F:



Quantitation comparable to most sensitive instruments

Why High Res MS?

- **Only Technology to provide:**
- Unequivocal ID
- Target Compounds Qual AND Quan
- Screen untarget compounds
- Retrospective Analysis
- No method development : Full Scan Mass spectra fingerprint analysis.



- **iSQ 9000 is a GCMSMS Triple Quad**
- **NOVA extremely sensitivity source (10 times)**
- **Change columns and lenses without vacuum brake**
- **AUTOSRM : Automatic method development of Collision energies for MSMS, or even SIM ions**
- **Pesticide analyser : Ready to go Workflows**
- **Dioxin analyser : Workflows Ready to Go...**

Cromatografia Iónica Hifenizada

- **IC- ICPMS**
- Especificação de Arsénio e Crómio

- **IC – MSMS IC- HRMS**
- Única tecnologia para pesticidas polares
- Análise de ClO_4 em leite e bebidas

Micro Plastics : The problem

BPA is one of the most studied chemicals found in plastic. It is usually found in plastic packaging or food storage containers and can leak out into food. Some evidence has shown that BPA can interfere with reproductive hormones, especially in women (*1).

Phthalates, a type of chemical used to make plastic flexible, have been shown to increase the growth of breast cancer cells. However, this research was carried out in a petri dish, so the results can't be generalized to humans (*2).

POPs are a class of chemicals that remain in the environment and have harmful effects on the human health such as pesticides (DDT), industrial chemicals such as polychlorinated biphenyls (PCB) etc...

Evitar o consumo de Microplasticos

Controlo de :

- Fraude
- Fraca qualidade no fabrico



Micro Raman



Micro FTIR

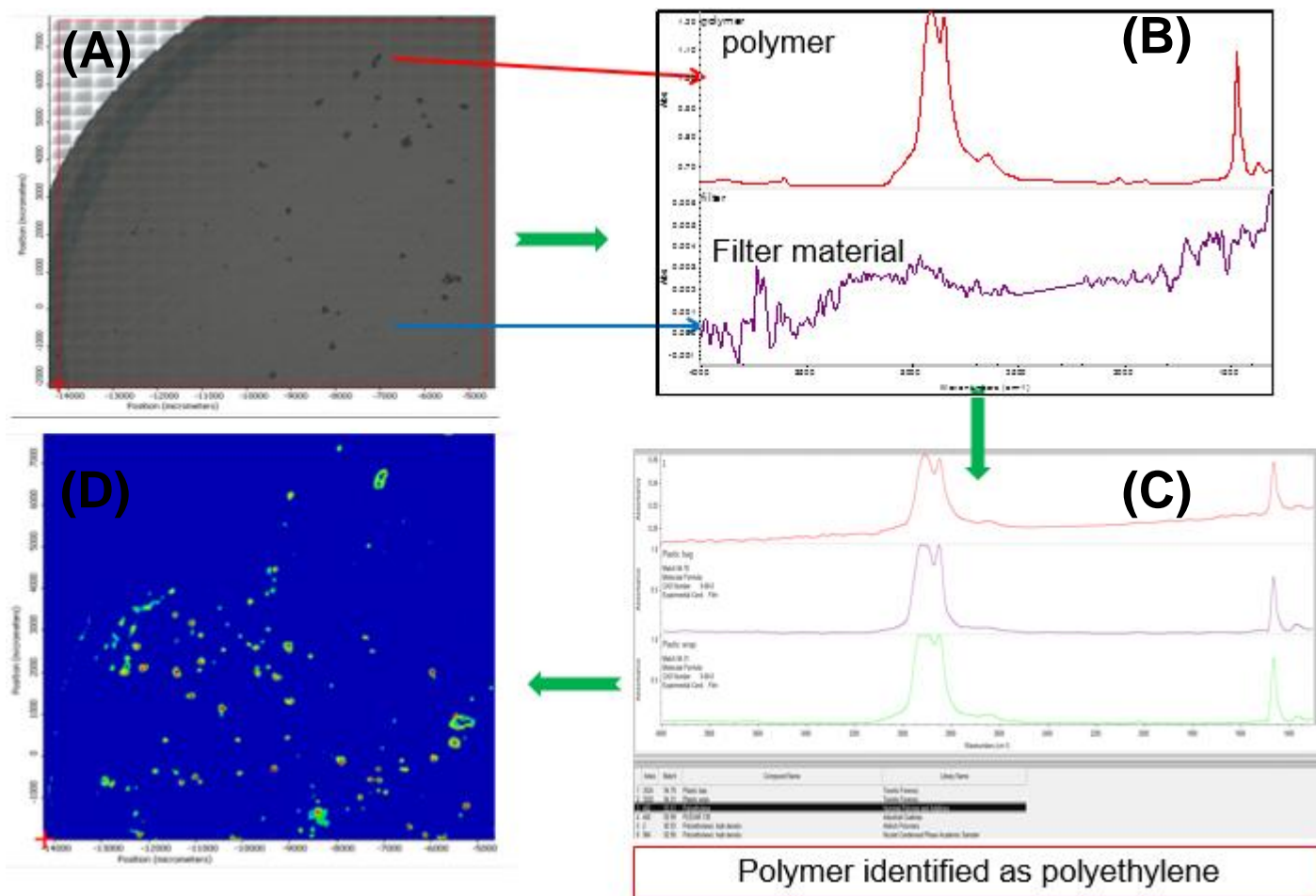
Microplásticos até 10 Micron : Medir e identificar

(A) Visual image of the filter showing particles

(B) Spectra of one of the particles and the filter paper

(C) Library searching to identify the material of the particles

(D) Correlation map to localize the particles with the same chemistry



Agradecimentos

- Grupo de Aplicações Thermo Scientific Bremen
- Milestone
- Grupo de Aplicações de Micro FTIR e Micro Raman Thermo Scientific Europe

Conclusões

Ao poluir águas límpidas com lodo, não encontrarás água potável para beber.

Esquilo – Filósofo Grego 525 BC - 456 BC