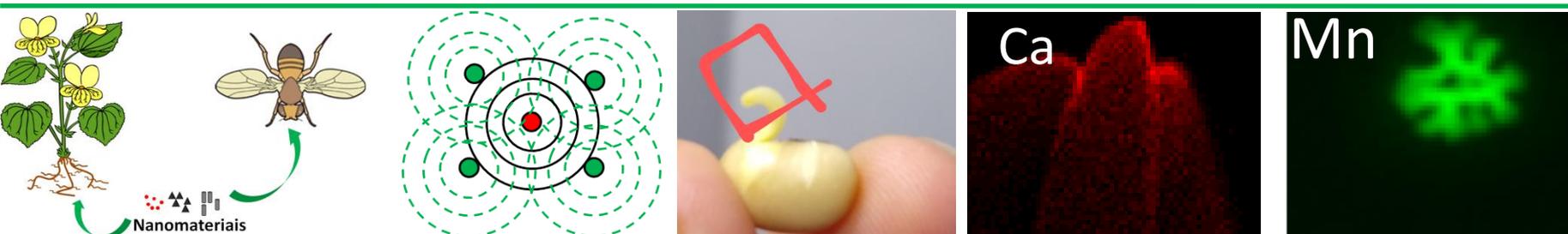




# Microanálise 2D por XRF

Prof. Hudson W.P. Carvalho

Divisão de Desenvolvimento de Técnicas e Métodos Analíticos e Nucleares- DVTEC  
Laboratório de Instrumentação Nuclear

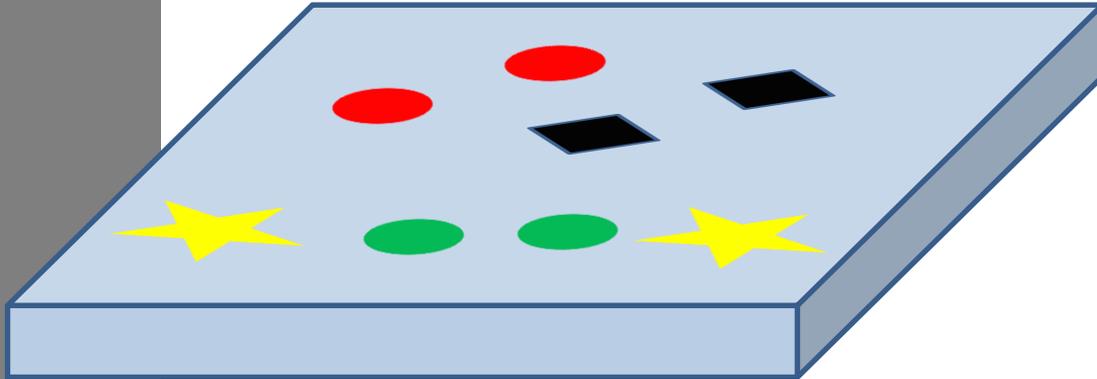


# Conteúdo

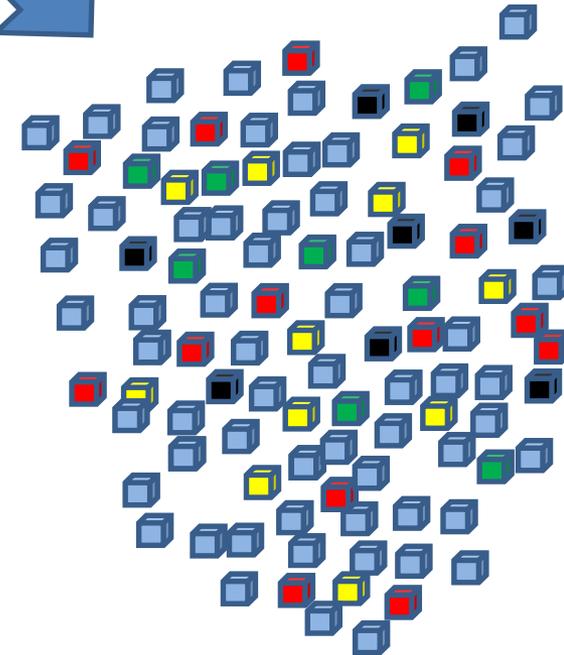
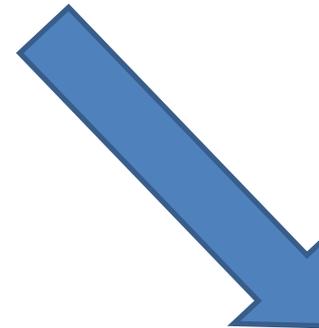
- O que se busca na microanálise?
- Como produzir microfexes de raios X
- Principais parâmetros das análises
- Exemplos de aplicações

# Determinação elemental por XRF

Amostra heterogênea



Amostra homogênea

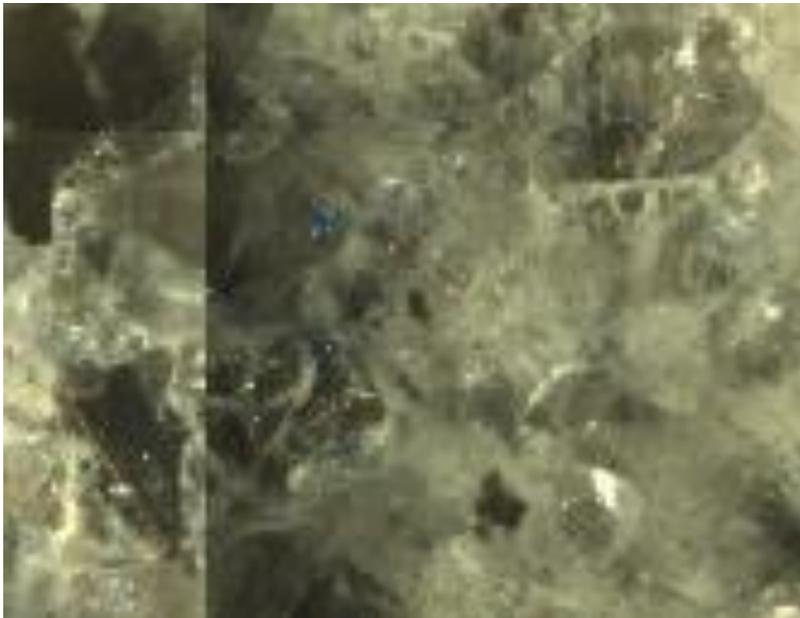


- ☐ Normalmente o analista se interessa pela concentração média dos elementos na amostra homogeneização da amostra
- ☐ Nesse caso, a determinação elemental é precedida pela homogeneização da amostra

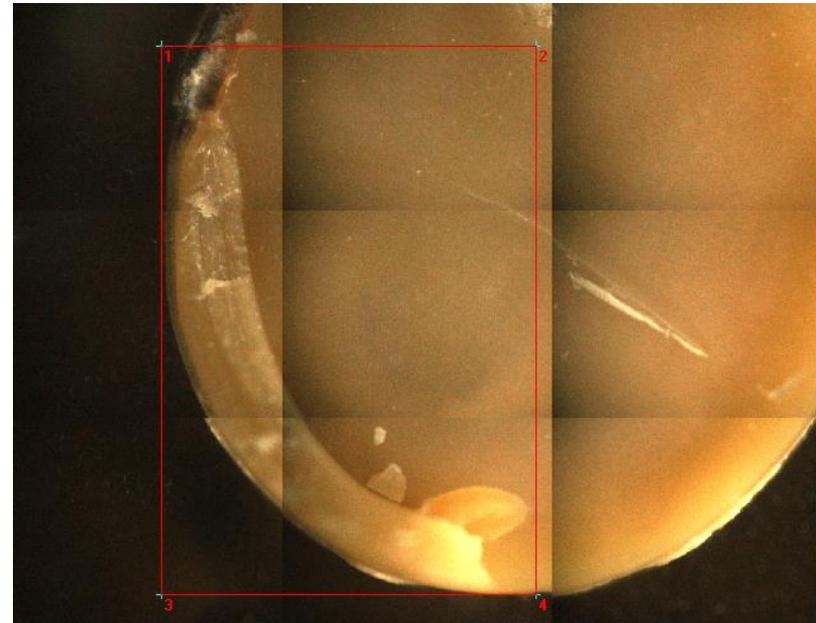
# O que se busca na microanálise?

- ❑ A microanálise se interessa pela heterogeneidade
- ❑ Por padrões de distribuição espacial estáticos ou dinâmicos
- ❑ Concentrações locais ao invés de médias

## Granito



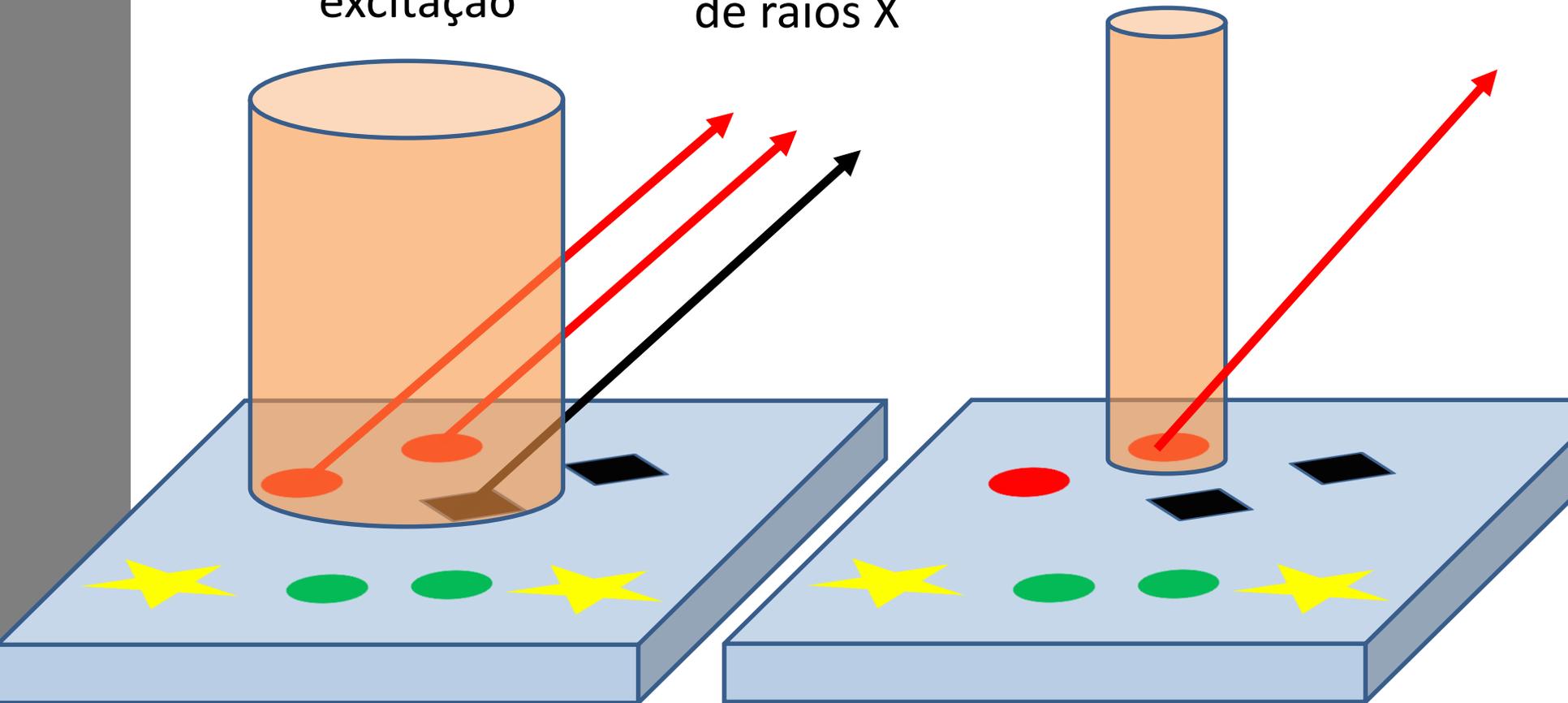
## Semente de soja

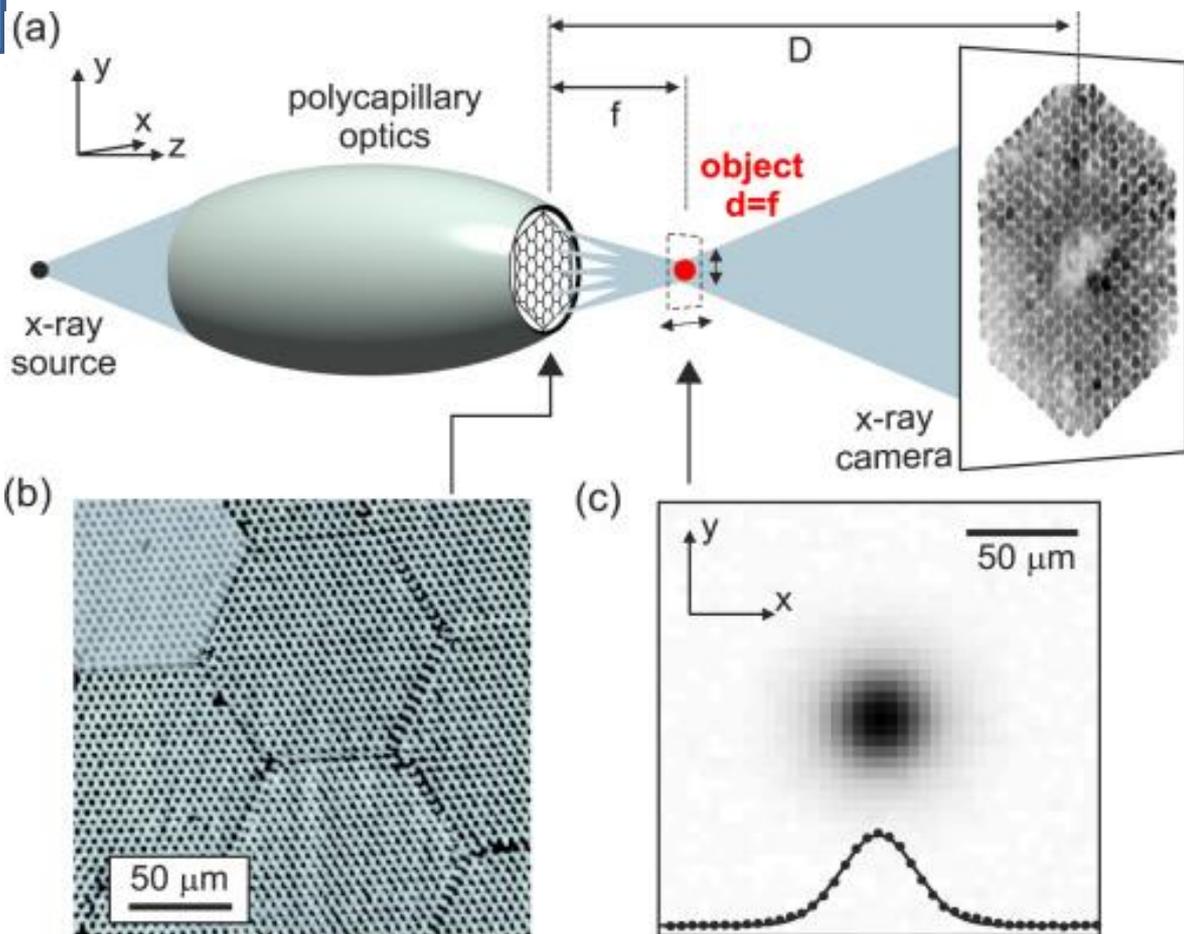
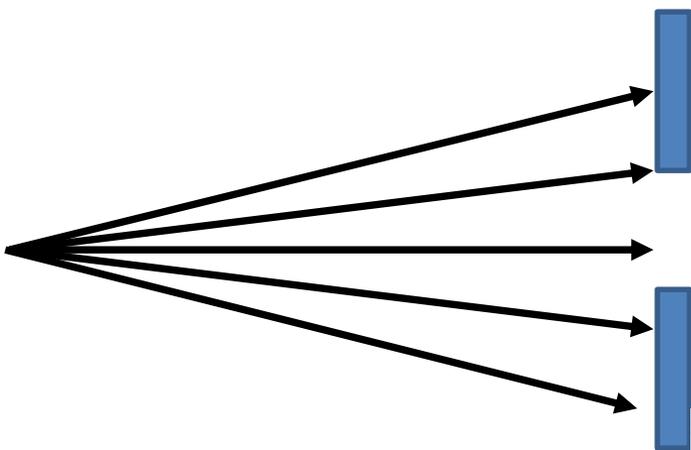


# Como obter um microfeixes de raios X

Feixe de  
excitação

Fluorescência  
de raios X





# Como produzir um micro feixe?

Aperture



Mono capillary



Poly capillary



Minimum diameter

About 10  $\mu\text{m}$

10  $\mu\text{m}$

High energy: 30  $\mu\text{m}$   
Low energy: ???

Beam collimation

Almost parallel

Almost parallel

Divergent

Beam intensity

Very low

High

High

Cost

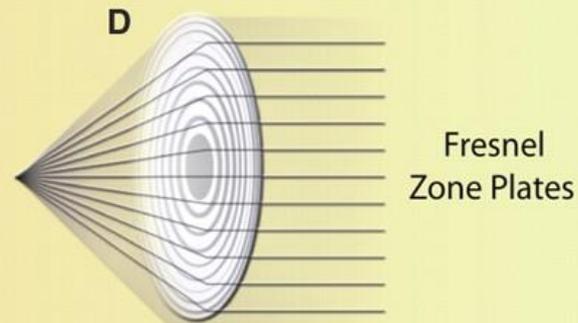
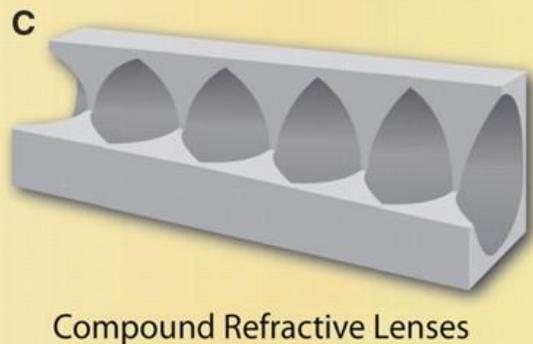
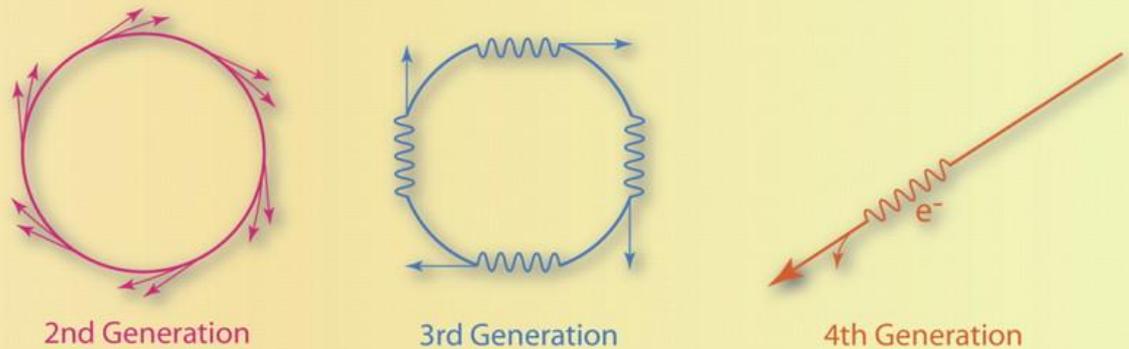
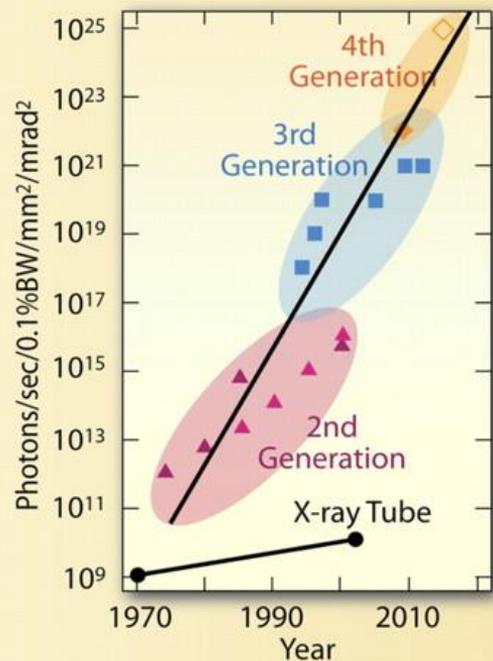
Low

Medium

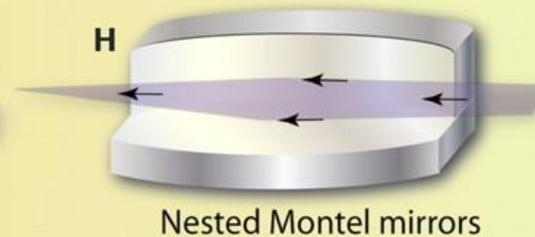
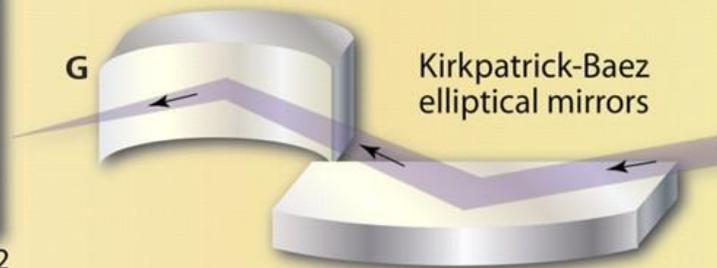
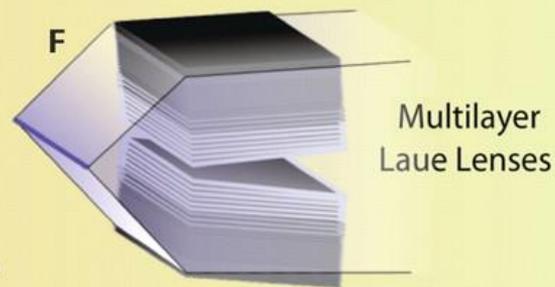
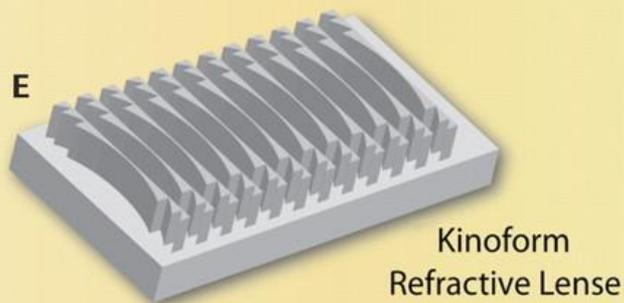
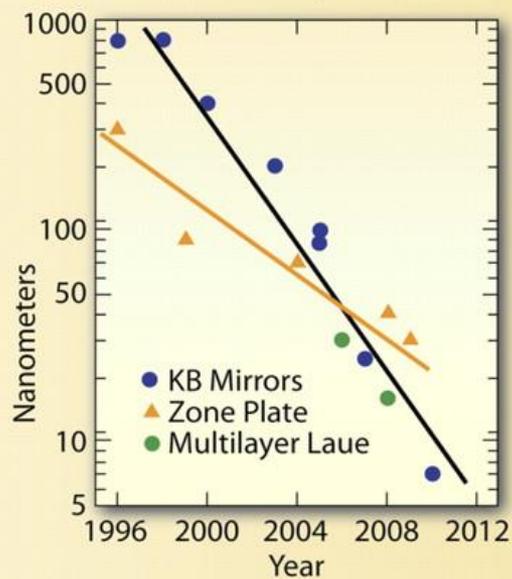
High

**HORIBA**  
Scientific

### A Time-average 10 keV X-ray Brilliance



### B 10 keV Spot Size



to focus

ce



# Parâmetros analíticos

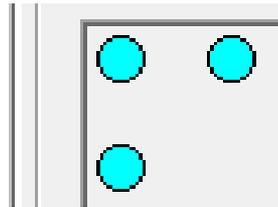
❑ Corrente, tensão, tempo de aquisição, vácuo?, filtro?

❑ Resolução lateral

**32x25**

X beam spacing: 69  $\mu\text{m}$

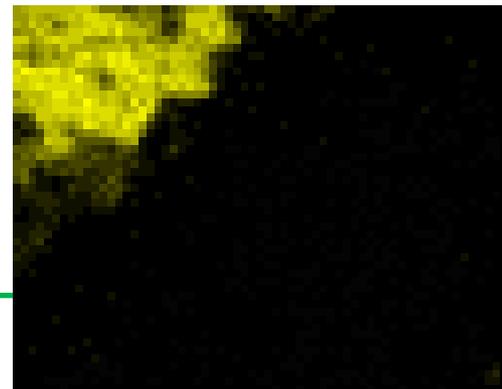
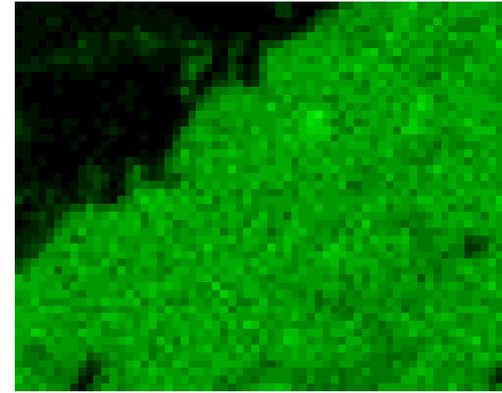
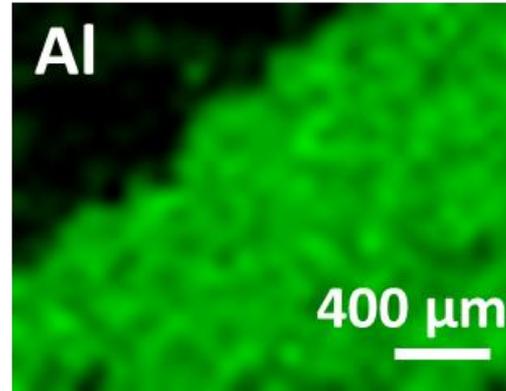
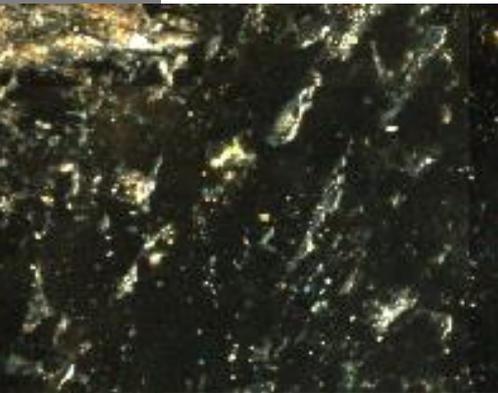
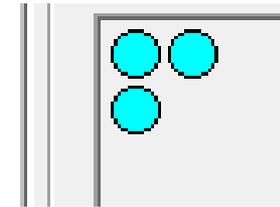
Y beam spacing: 69  $\mu\text{m}$



**64x50**

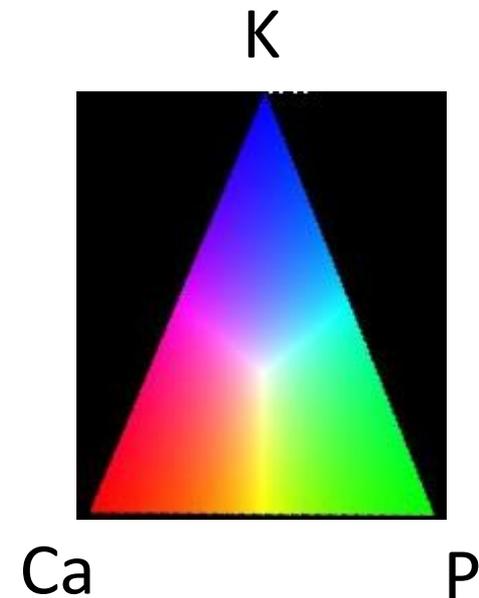
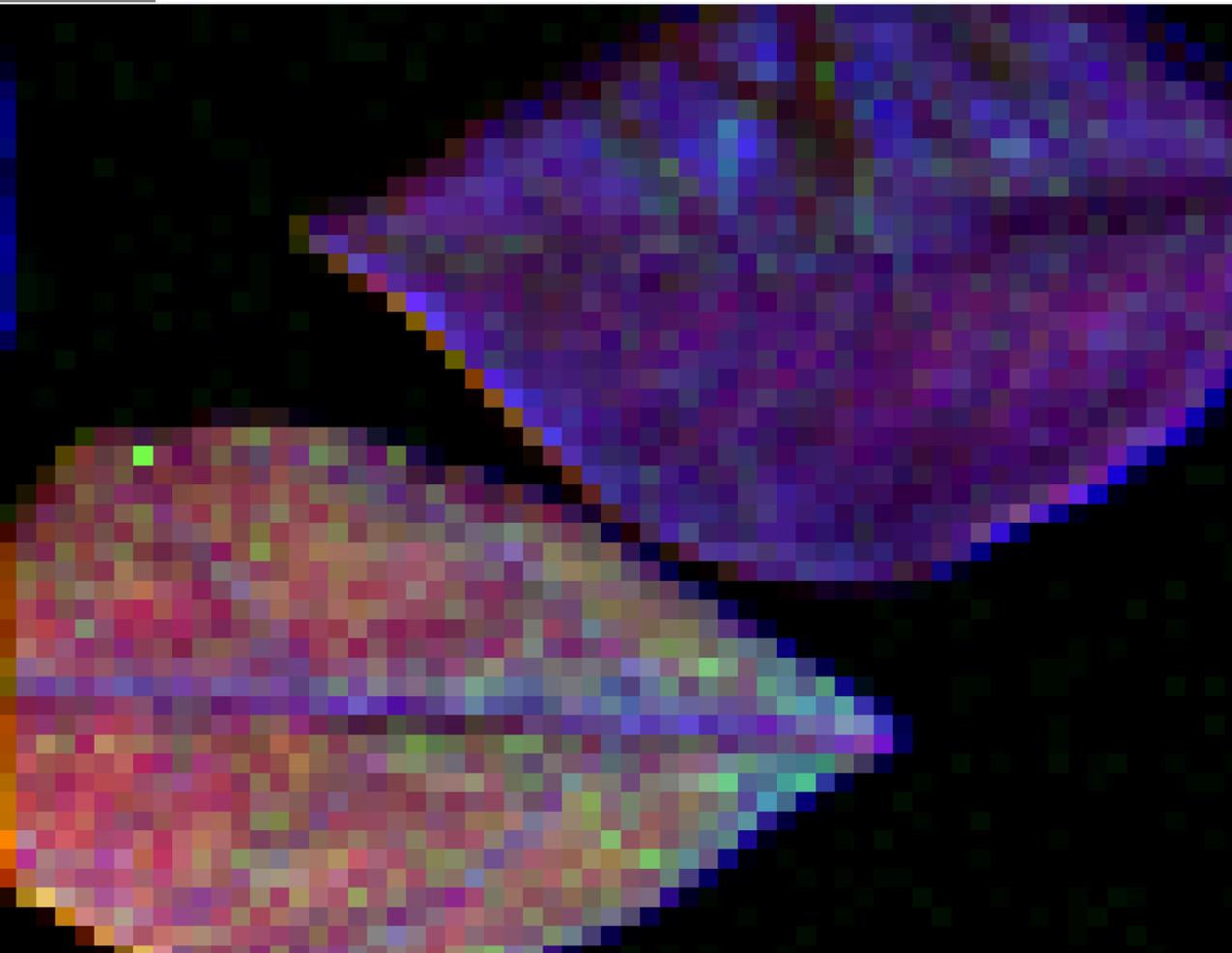
X beam spacing: 34  $\mu\text{m}$

Y beam spacing: 34  $\mu\text{m}$



# Nutrição de plantas

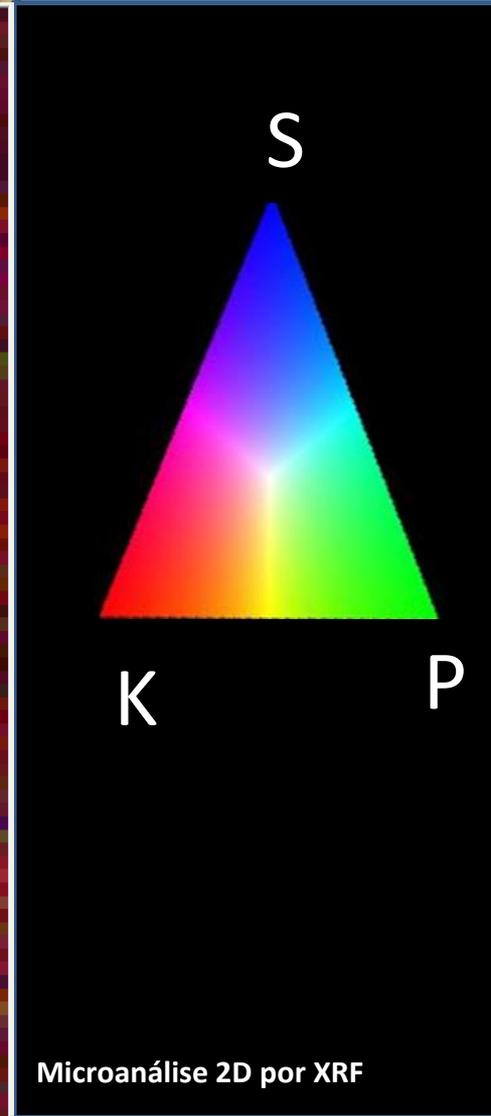
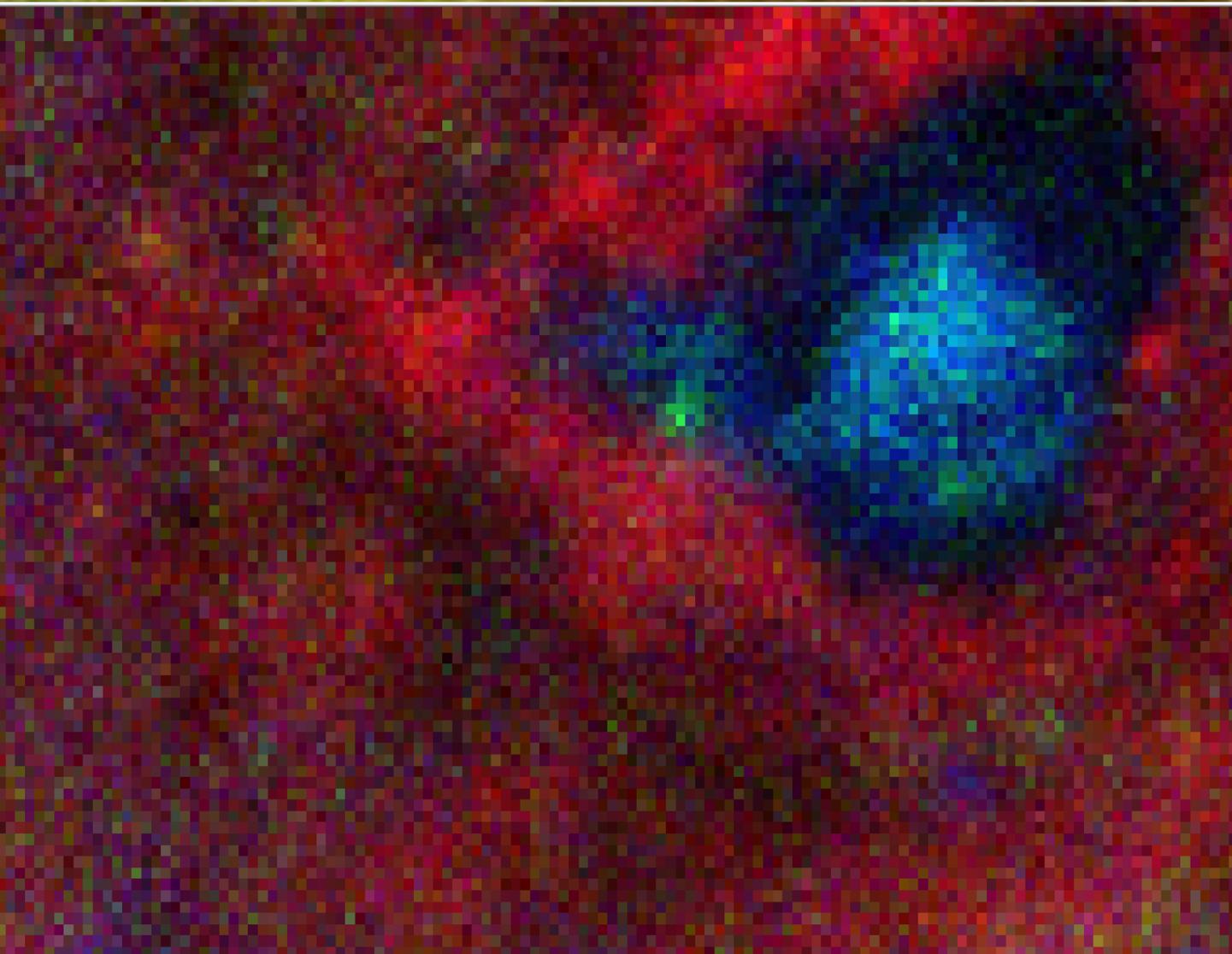
- Distribuição espacial de nutrientes em uma folha de laranjeira



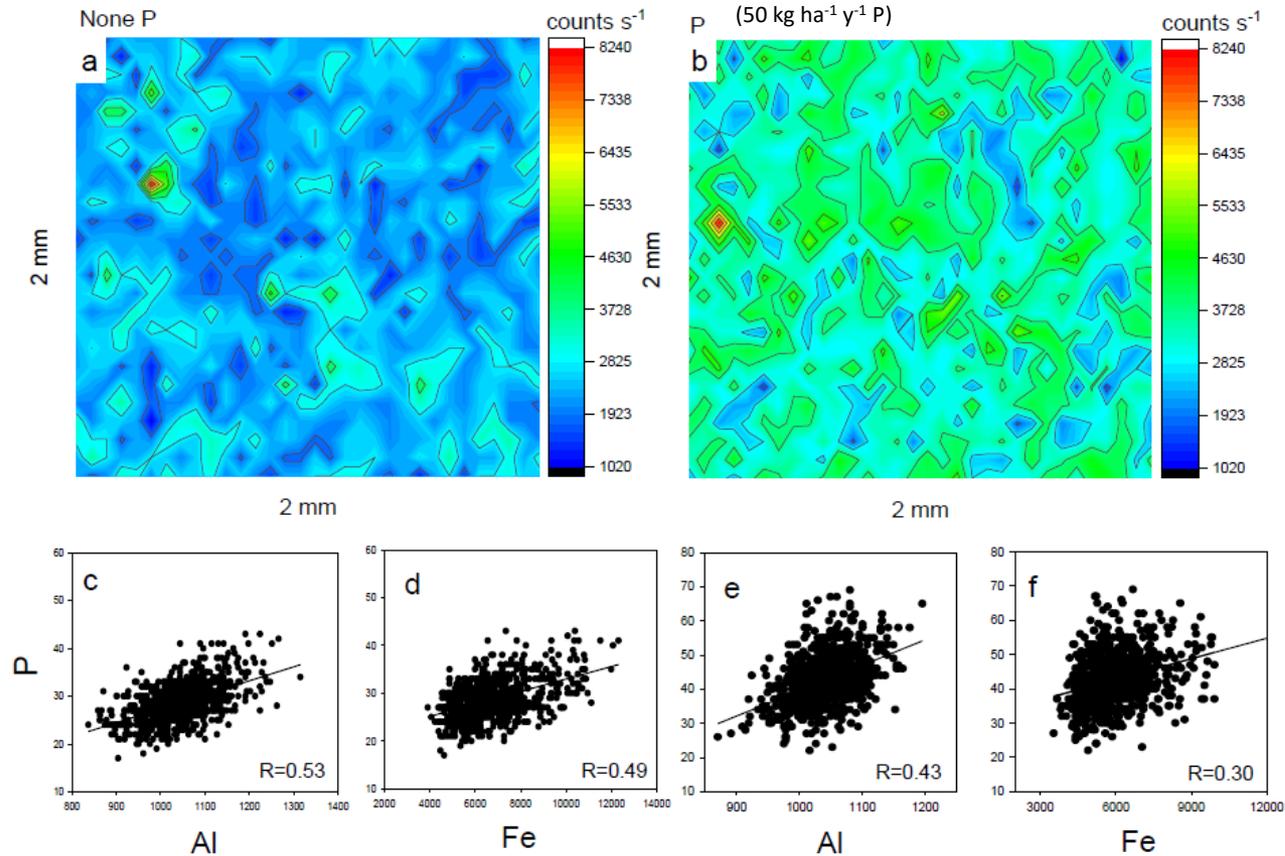
C. R Carvalho,  
Mestrado, USP-ESALQ, 2017

# Fitopatologia

- ❑ Folha acometida por um espécie de ferrugem



## Correlação elementar

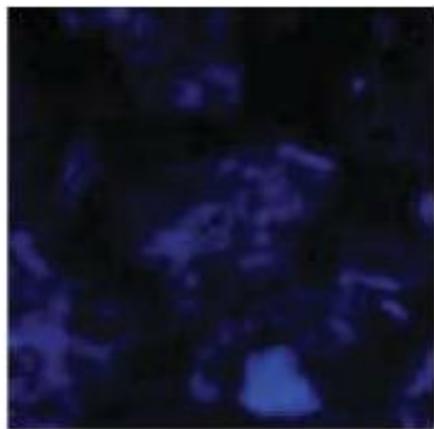


**Fig. 2.** Quantitative spatial distribution of P (a; b) and scattering plots of elements distribution maps between P and Al and between P and Fe (c; d; e; f).



J.A. Antonangelo *et al.* 2018

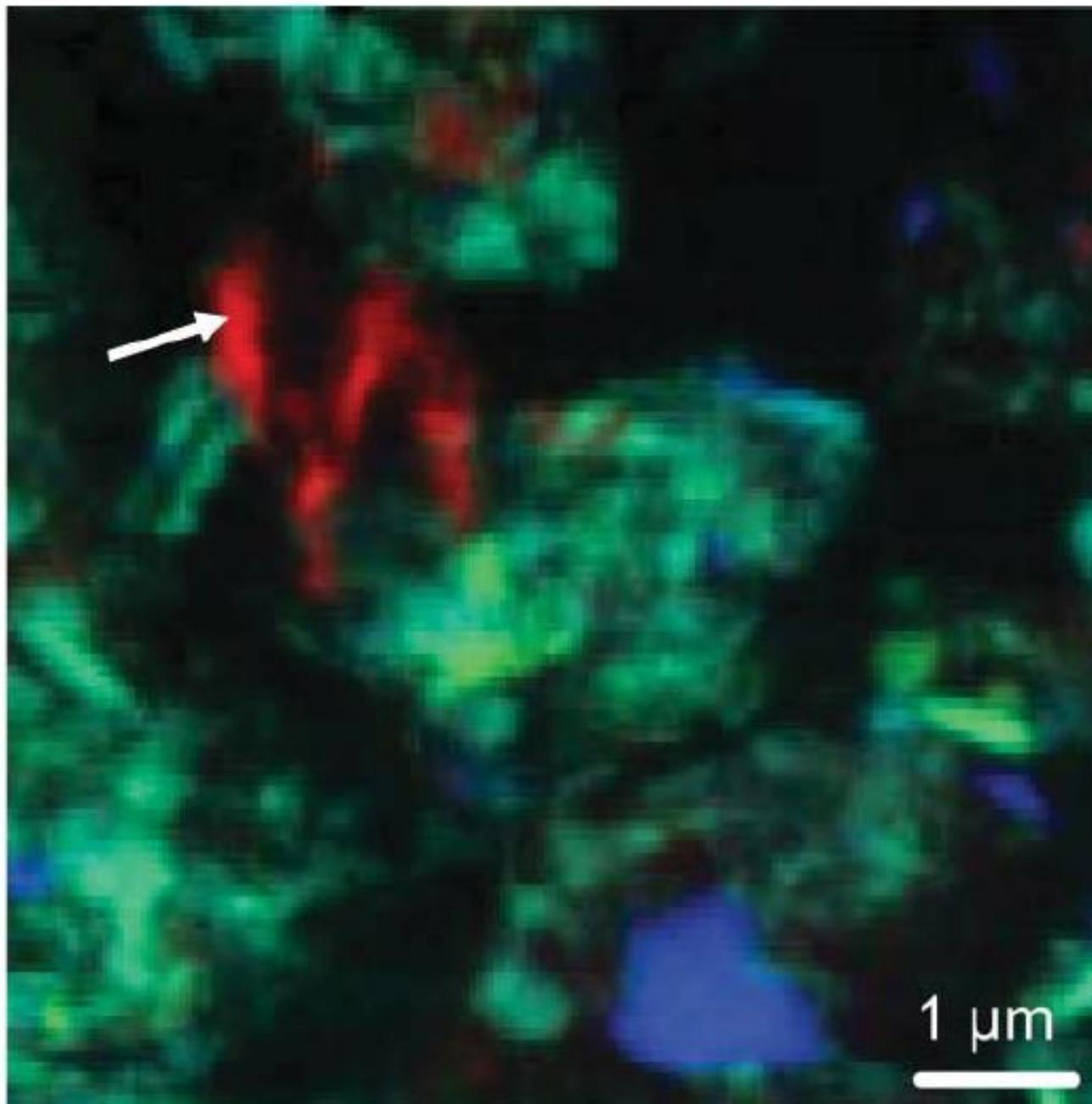
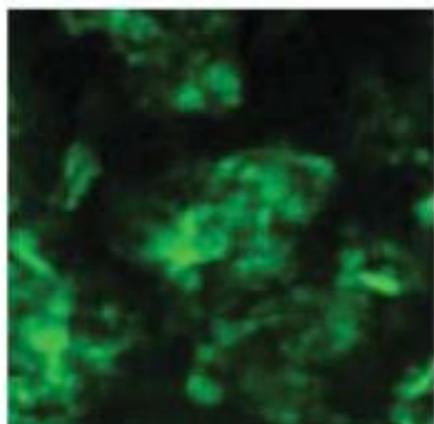
Si



P



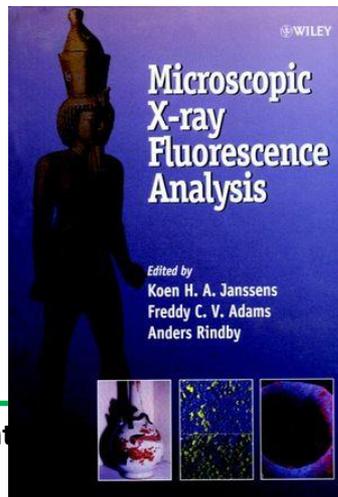
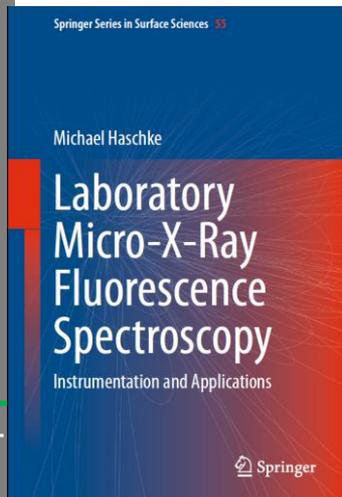
Al



# Sumário

- ❑ Adequada para a investigação de heterogeneidades
- ❑ Análises pontuais, em linha ou bidimensionais
- ❑ Resolução lateral da ordem de milímetros a nanômetros
- ❑ Resultados qualitativos e quantitativos
- ❑ Mais comum em síncrotron, mas vem se popularizando em laboratórios

# Bibliografia



## Laboratory Microprobe X-Ray Fluorescence in Plant Science: Emerging Applications and Case Studies

Eduardo S. Rodrigues<sup>1</sup>, Marcos H. F. Gomes<sup>1</sup>, Nádia M. Duran<sup>1</sup>, João G. B. Cassanji<sup>1</sup>, Tatiana N. M. da Cruz<sup>1</sup>, Analdar Sant'Anna Neto<sup>2</sup>, Susilaine M. Savassa<sup>1</sup>, Eduardo de Almeida<sup>1</sup> and Hudson W. P. Carvalho<sup>1\*</sup>

<sup>1</sup> Nuclear Instrumentation Laboratory, Center for Nuclear Energy in Agriculture, University of São Paulo, Piracicaba, Brazil, <sup>2</sup> Physiology of Trees Laboratory, Department of Forest Science, College of Agriculture Luiz de Queiroz, University of São Paulo, Piracicaba, Brazil

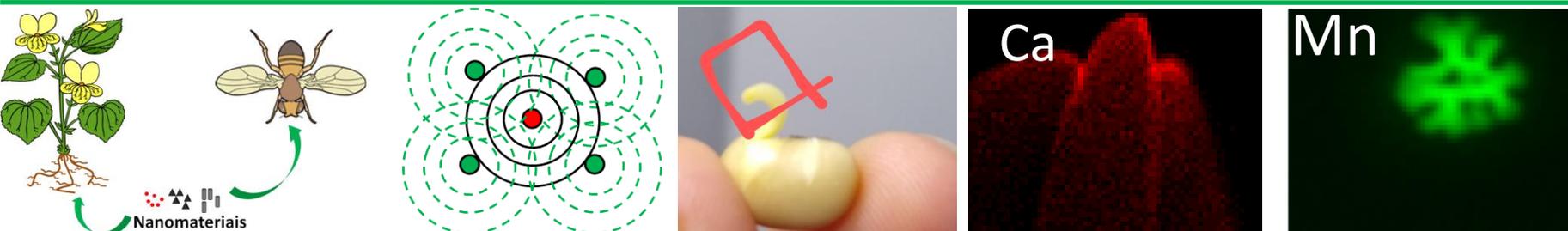


# Tomografia por XRF

Prof. Hudson W.P. Carvalho

Divisão de Desenvolvimento de Técnicas e Métodos Analíticos e Nucleares- DVTEC

Laboratório de Instrumentação Nuclear



# Conteúdo

- Radiografia e tomografia
- Microanálise por fluorescência de raios X 2D e 3D
- XRF confocal
- Como se mede uma tomografia por atenuação
- Tomografia por fluorescência de raios X
- Exemplos de aplicação

# Radiografia e Tomografia

□ Do plano para o espaço

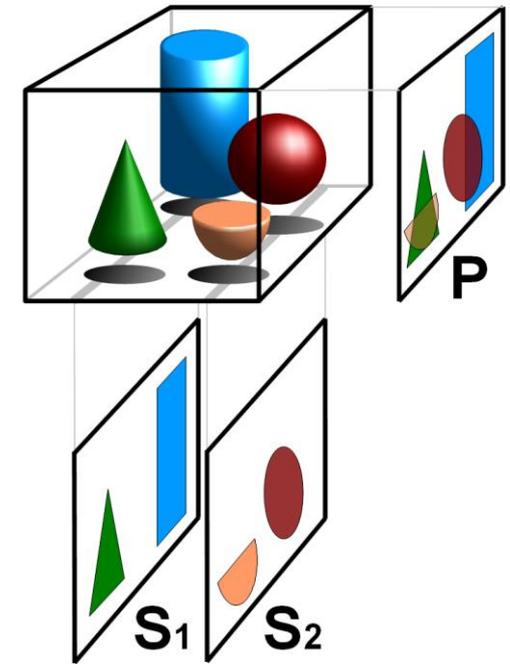


Prêmio Nobel 1901



Prêmio Nobel 1979

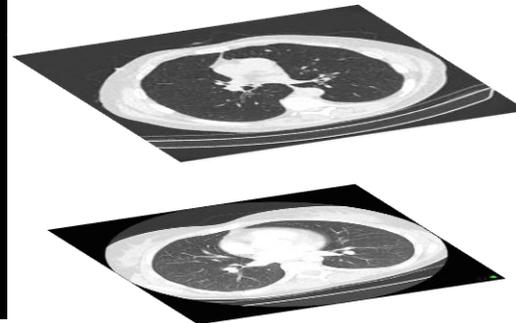
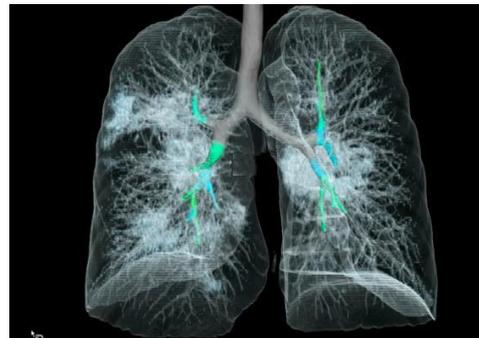
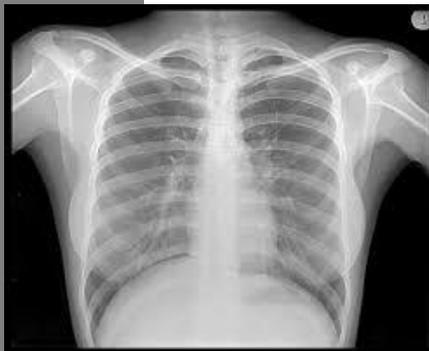
[https://upload.wikimedia.org/wikipedia/commons/0/08/Webcam\\_CT\\_transmissions.ogv](https://upload.wikimedia.org/wikipedia/commons/0/08/Webcam_CT_transmissions.ogv)



[https://en.wikipedia.org/wiki/Tomography#/media/File:TomographyPrinciple\\_Illustration.png](https://en.wikipedia.org/wiki/Tomography#/media/File:TomographyPrinciple_Illustration.png)

2D

3D



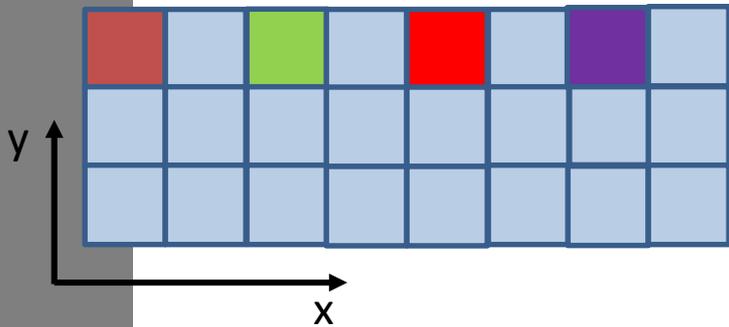
<https://radiopaedia.org/cases/normal-chest-ct-lung-window-1?lang=us>  
<https://mytmg.com/about-lung-cancer-screenings/>

[https://www.dut.ac.za/faculty/health\\_sciences/radiography/](https://www.dut.ac.za/faculty/health_sciences/radiography/)

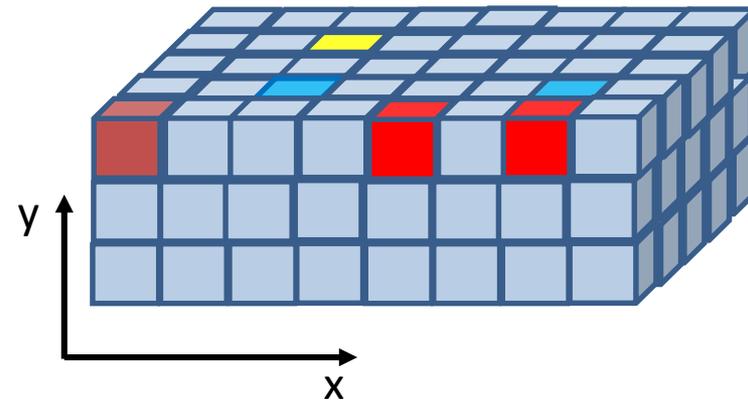
# Fluorescência de raios X em 2D e 3D

## ☐ Artefatos na XRF

### 2D



### 3D



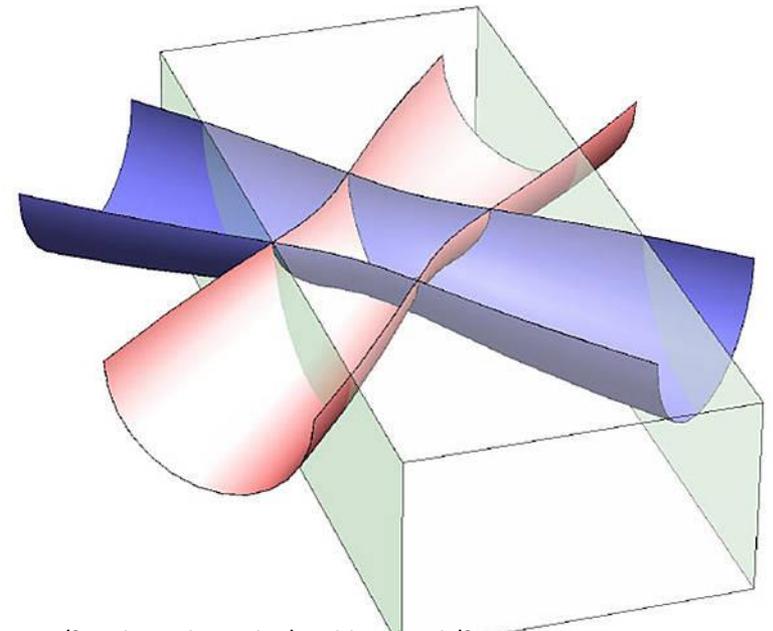
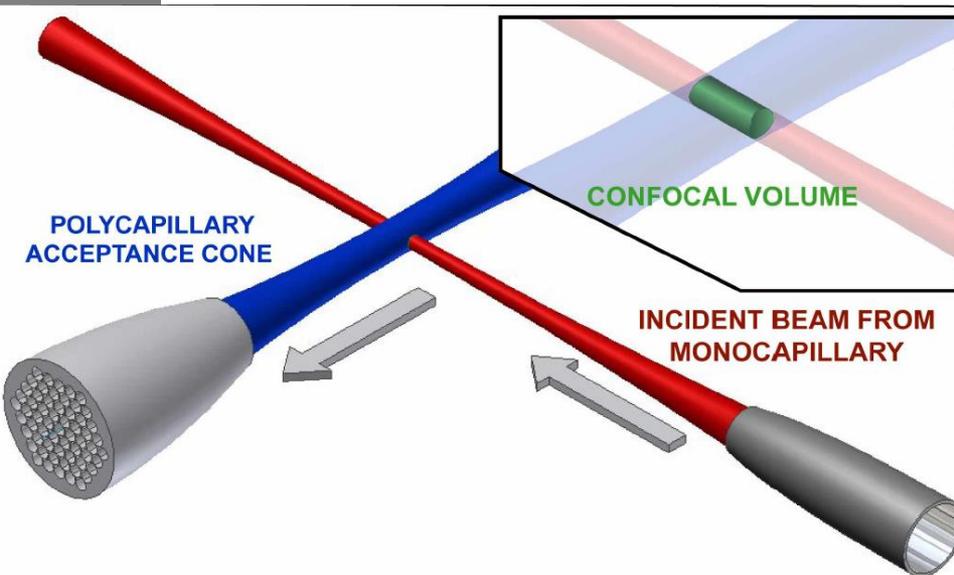
Sobreposição de elementos na dimensão horizontal

$$\text{purple square} = \text{red square} + \text{blue square}$$

$$\text{green square} = \text{blue square} + \text{yellow square}$$

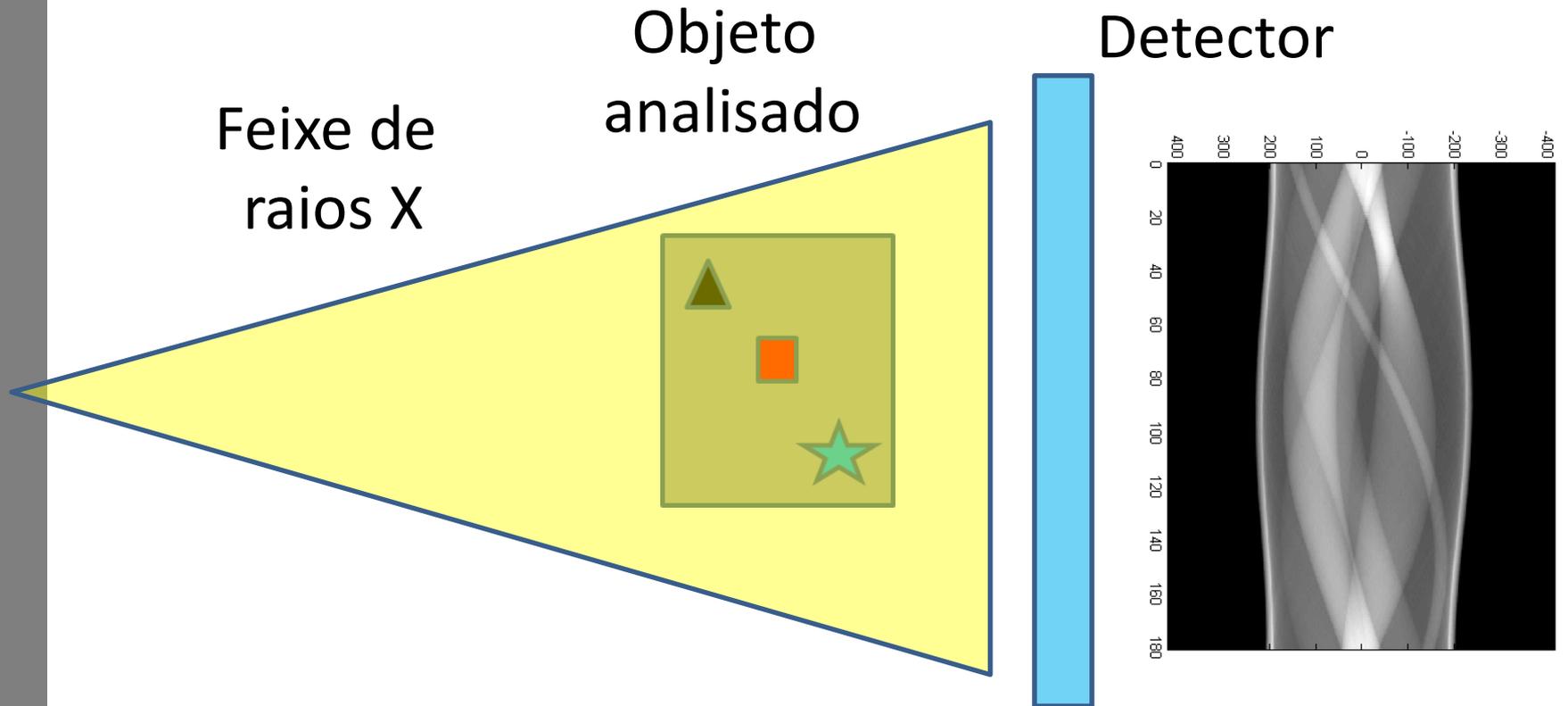
# Confocal $\mu$ -XRF

- ❑ Emprego de elementos focalizadores
- ❑ Excita elementos e detecta fótons de uma determinada região do espaço

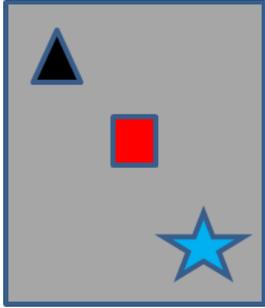


Mater. Res. Soc. Symp. Proc. Vol. 852 © 2005 Materials Research Society

# Como é feita uma tomografia?



- ❑ Registra-se uma radiografia em cada ângulo de exposição

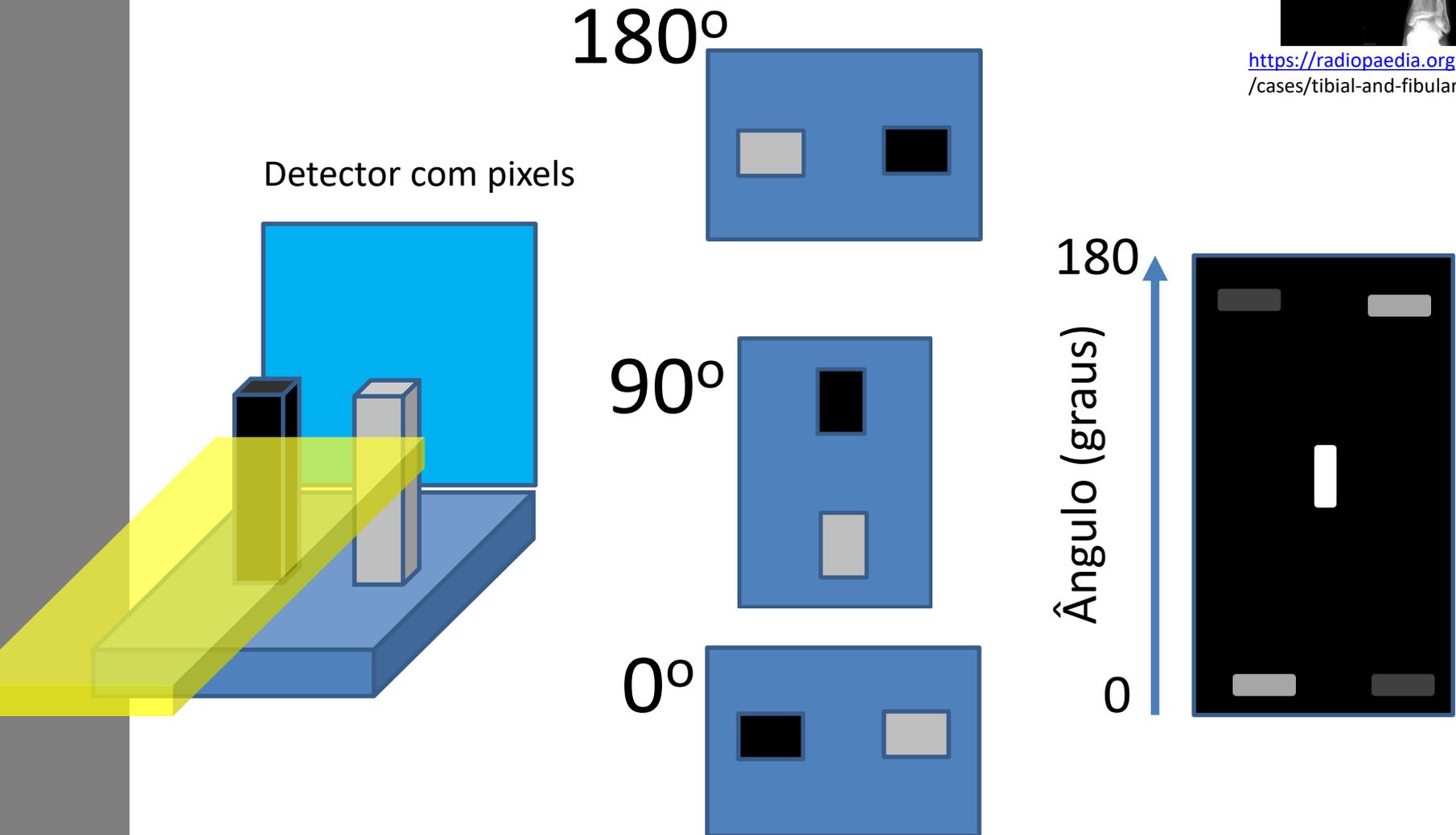


# A formação do sinograma

- A obtenção de um sinograma

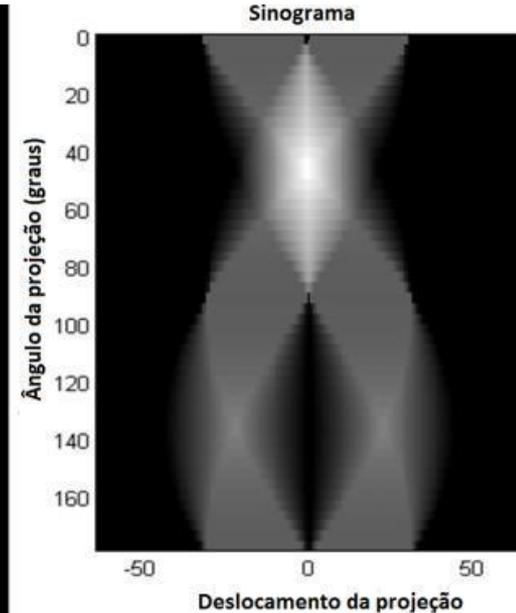
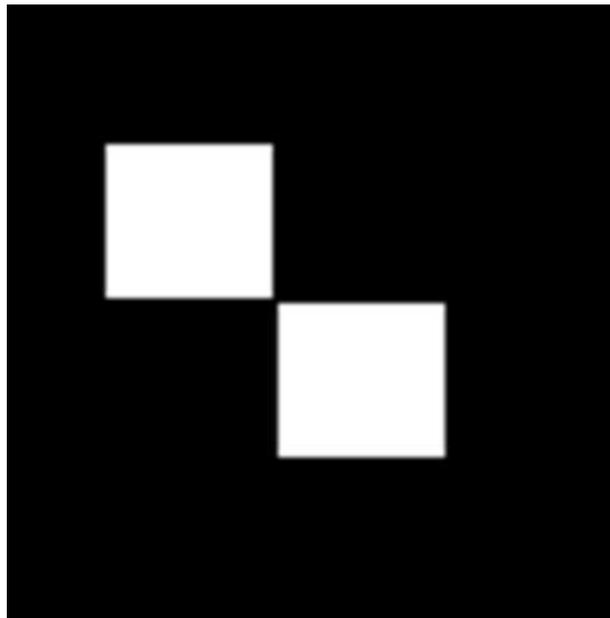
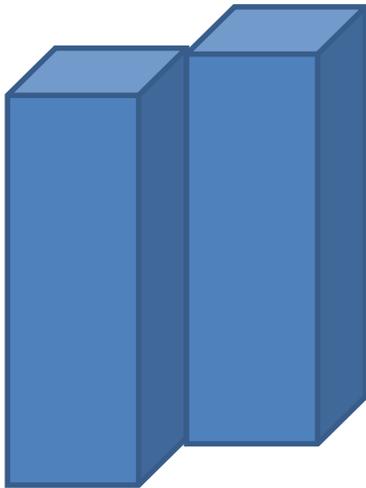


<https://radiopaedia.org/cases/tibial-and-fibular-fractures>



# A formação do sinograma

- Um algoritmo combina as projeções numa imagem 3D



ANÁLISE DA POROSIDADE DE SEDIMENTOS GEOLÓGICOS  
USANDO A TÉCNICA DE MICROTOMOGRAFIA COMPUTADORIZADA  
Haimon Diniz Lopes Alves Dissertação de Mestrado UFRJ  
Orientador: Ricardo Tadeu Lopes Rio de Janeiro Fevereiro de 2012

# Tomografia por XRF

□ Ao invés de medir apenas a atenuação, registra-se também a XRF

Translação da amostra no espaço xyz

Translação da amostra no espaço xyz e  $\Theta$

NUCL INSTRUM METH A277, 1989, 669-672

X-Ray Fluorescence Microtomography in Biological Applications

DOI: 10.5772/24535 · Prof. R.T. Lopes, UFRJ

R. Cesare, S. Mascarenhas / A new tomographic device

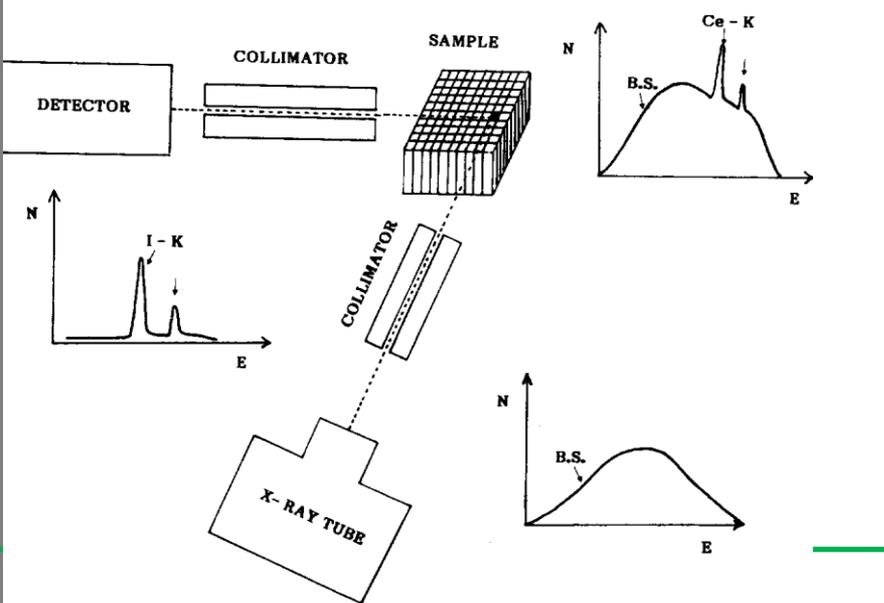
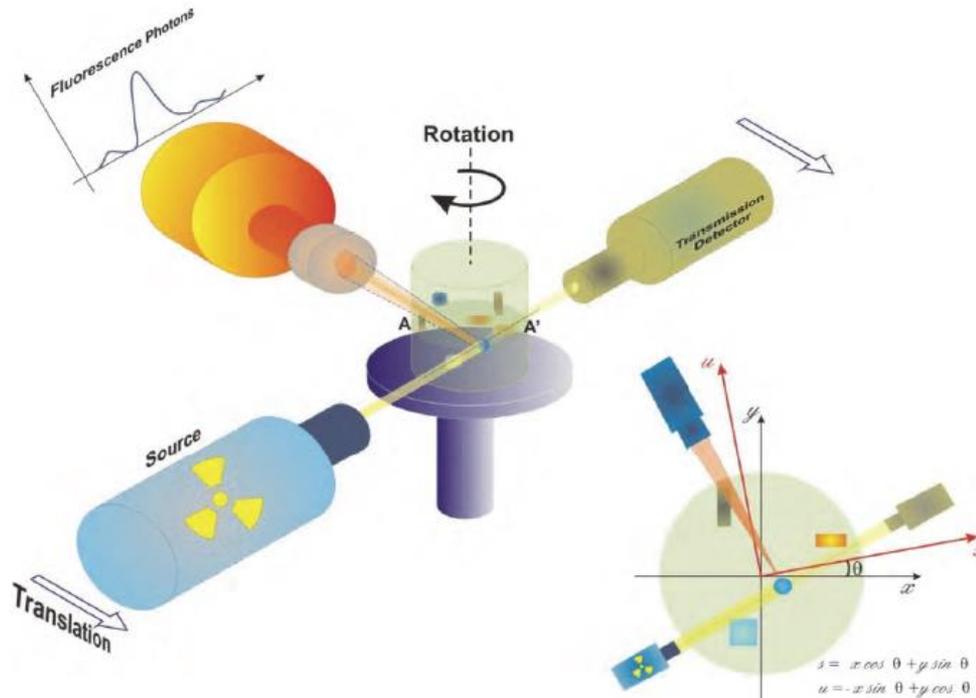
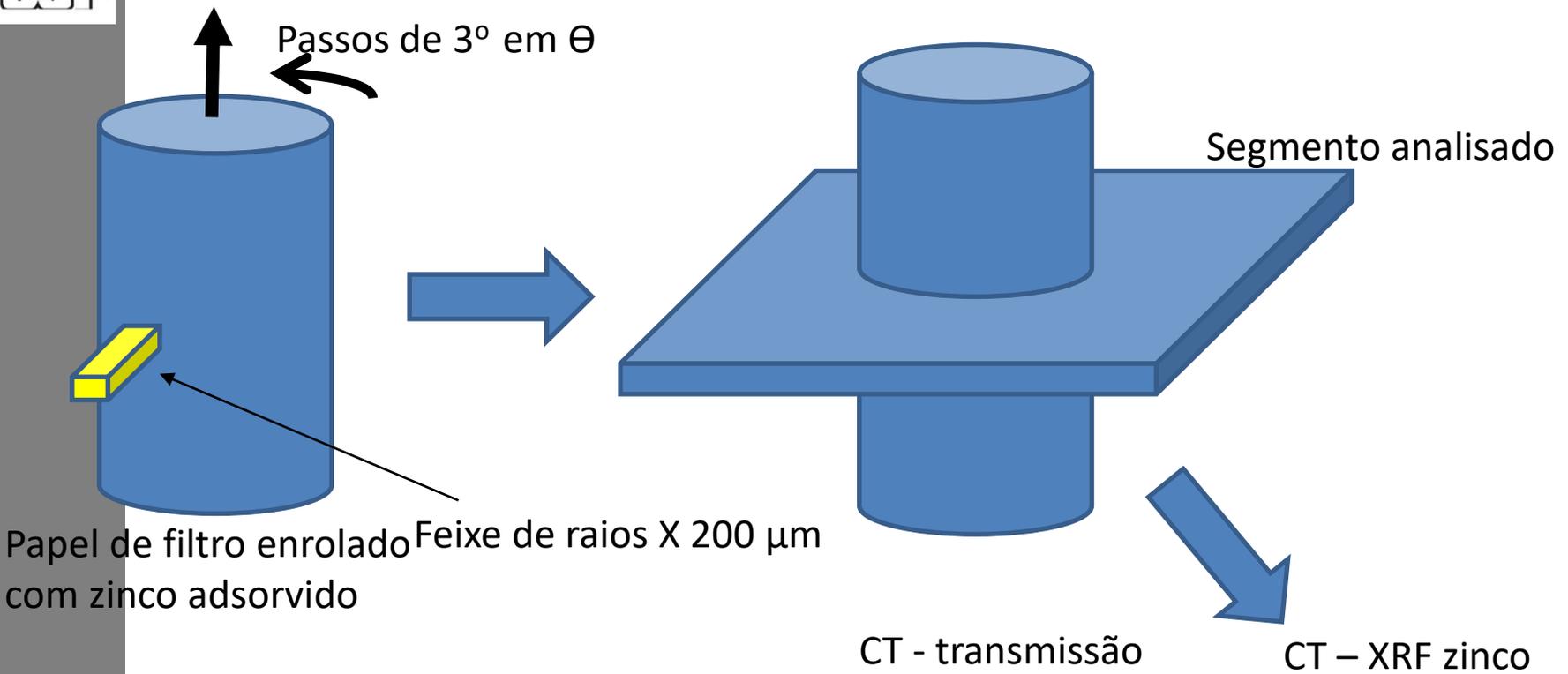


Fig. 1. Experimental setup for the fluorescence X-ray tomography.



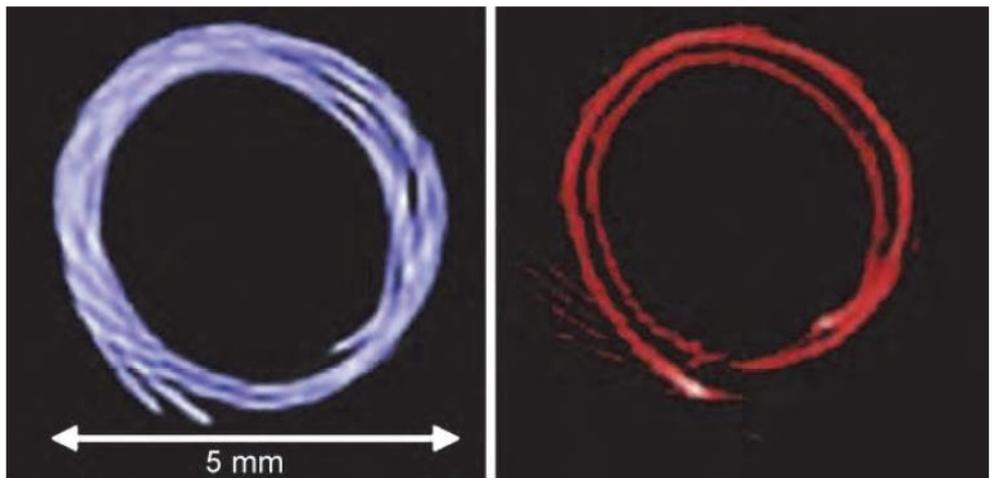
# Detalhes



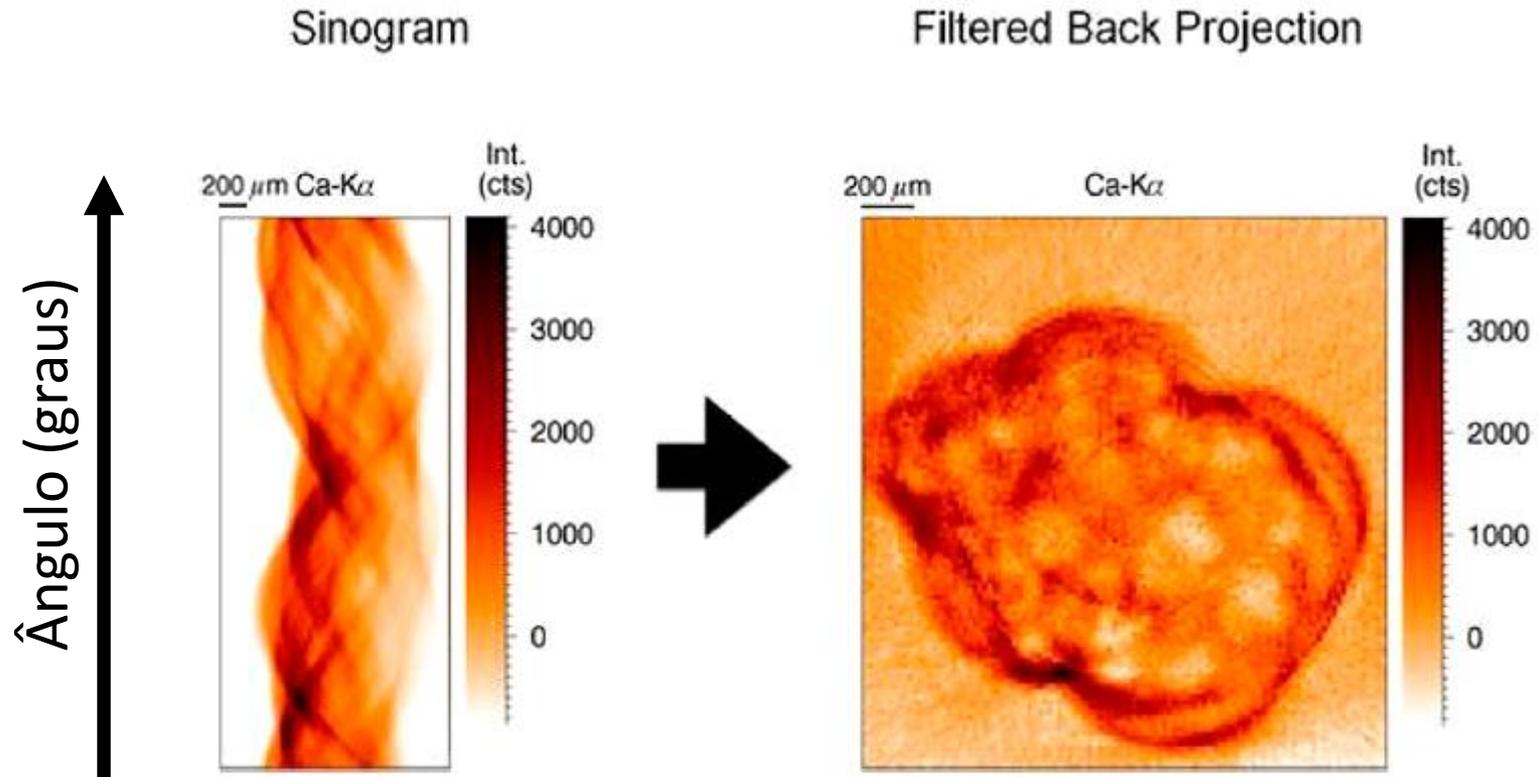
8

## X-Ray Fluorescence Microtomography in Biological Applications

Gabriela R. Pereira<sup>1</sup> and Ricardo T. Lopes<sup>2</sup>  
<sup>1</sup>Non-destructive Testing, Corrosion and Welding Laboratory, Department of Metallurgical and Materials Engineering COPPE/UFRJ Federal University of Rio de Janeiro, Rio de Janeiro/RJ  
<sup>2</sup>Nuclear Instrumentation Laboratory, Department of Nuclear Engineering COPPE/UFRJ, Federal University of Rio de Janeiro, Rio de Janeiro/RJ

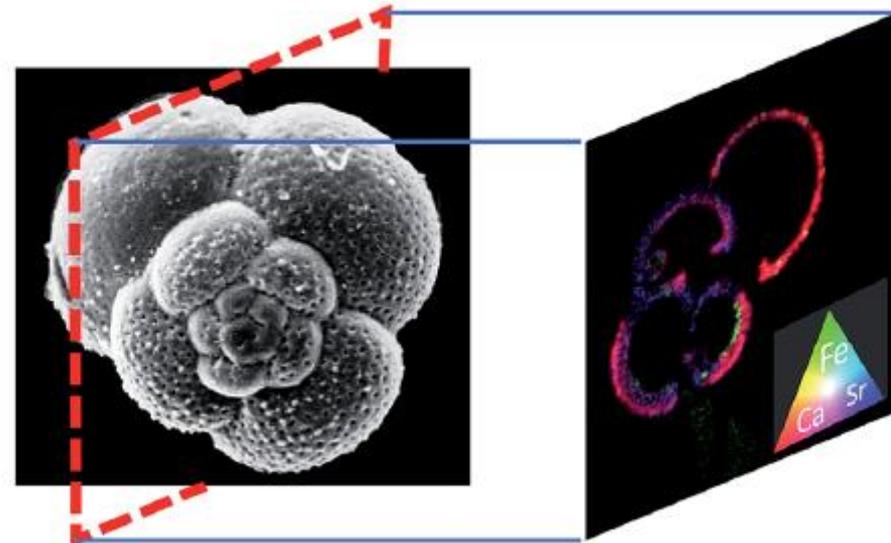
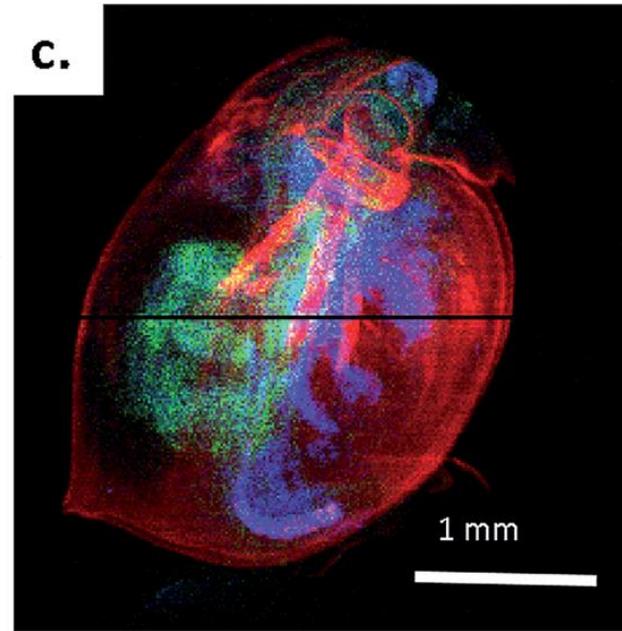
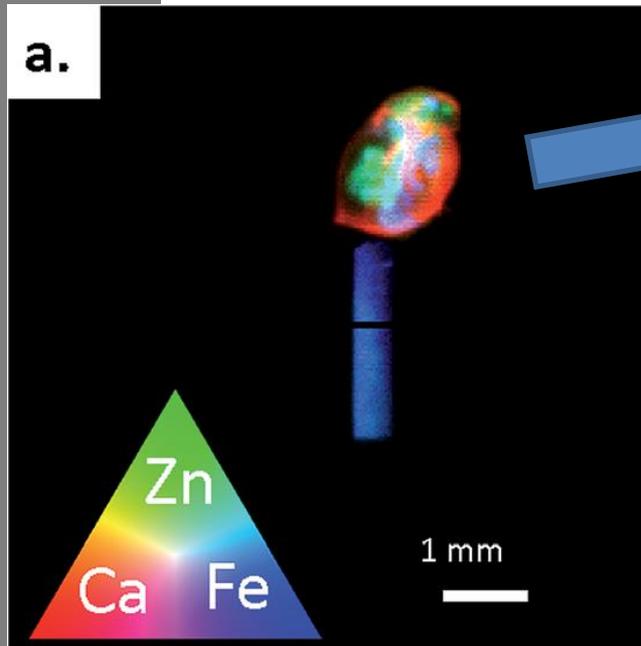


# Sinograma e projeção do elemento cálcio de um micro crustáceo



Passos de 20  $\mu\text{m}$ , 200 projeções, 14 s ponto

# Imagem por XRF 3D de *D. magna* & foraminífero



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Cite this: DOI: 10.1039/c5ja00198k

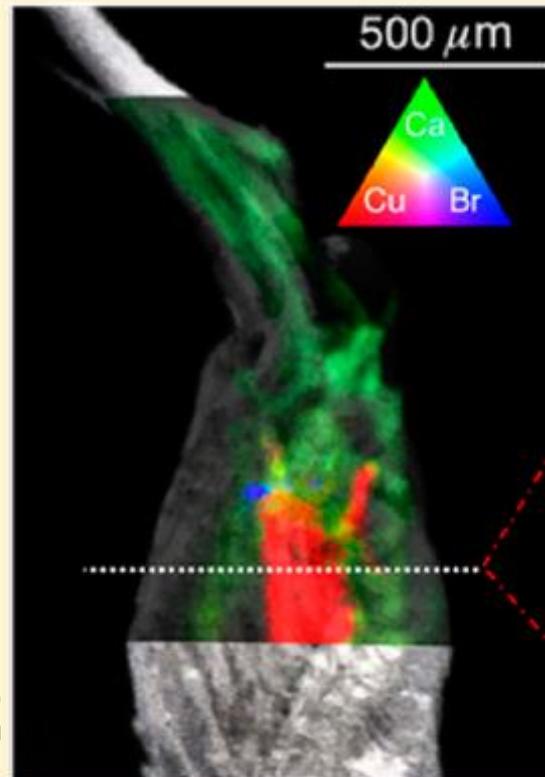
**Three-dimensional X-ray fluorescence imaging modes for biological specimens using a full-field energy dispersive CCD camera†**

Björn De Samber,<sup>a</sup> Oliver Scharf,<sup>b</sup> Günther Buzanich,<sup>b</sup> Jan Garrevoet,<sup>c</sup> Pieter Tack,<sup>b</sup> Martin Radtke,<sup>b</sup> Heinrich Rieseemeier,<sup>b</sup> Uwe Reinholz,<sup>b</sup> Roel Evens,<sup>c</sup> Karel De Schampelaere,<sup>d</sup> Gerald Falkenberg,<sup>c</sup> Colin Janssen<sup>d</sup> and Laszlo Vincze<sup>a\*</sup>

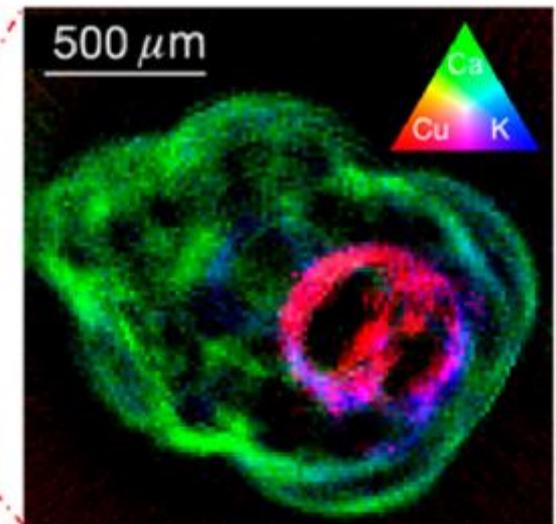
# Imagem por XRF 3D de micro crustáceo

- ❑ Cálcio na estrutura do exoesqueleto
- ❑ Cobre no sistema gástrico e hepato pâncreas

## Imagem 2 D



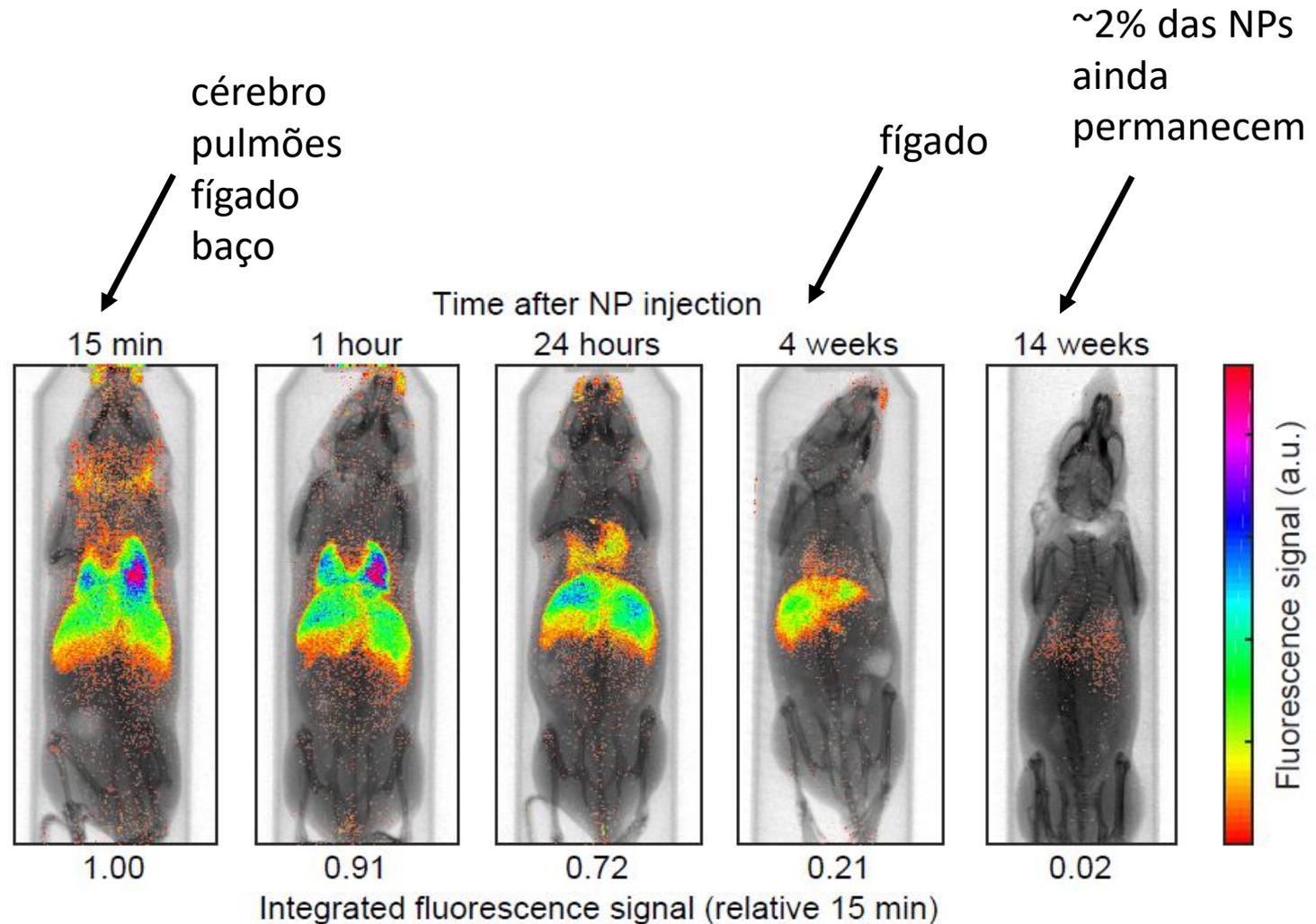
## CT – XRF 3D



# NP de Mo dentro de um animal

## Medidas in vivo: depuração de NPs

<https://iopscience.iop.org/article/10.1088/1361-6560/aad51e/pdf>



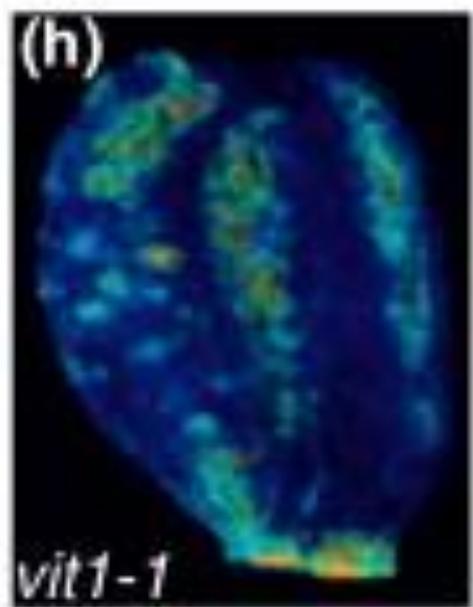
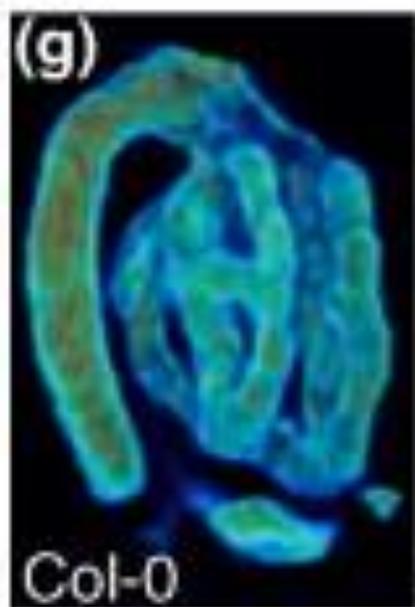
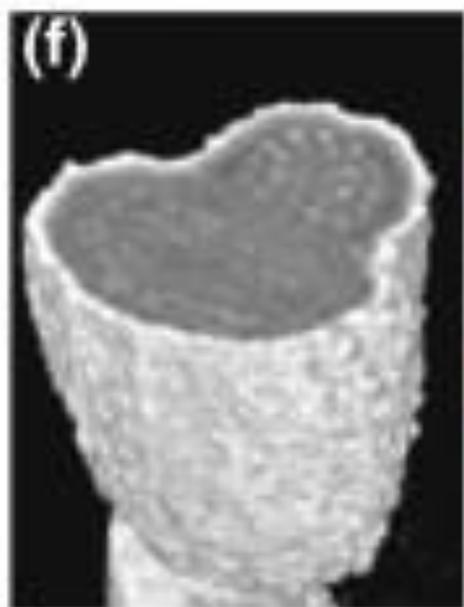
# Sumário

## Desafios envolvidos nas medidas

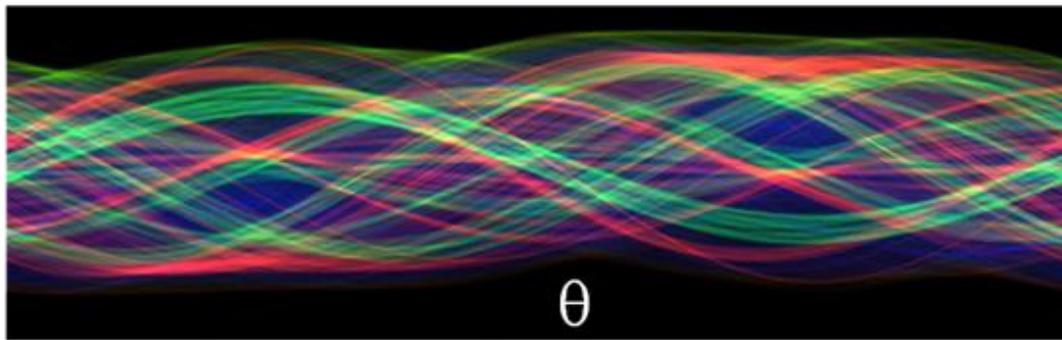
- Estabilidade de posição da amostra e porta-amostra
- Fundo espectral causado pelo espalhamento da radiação
- Correção dos efeitos auto absorção

## Características

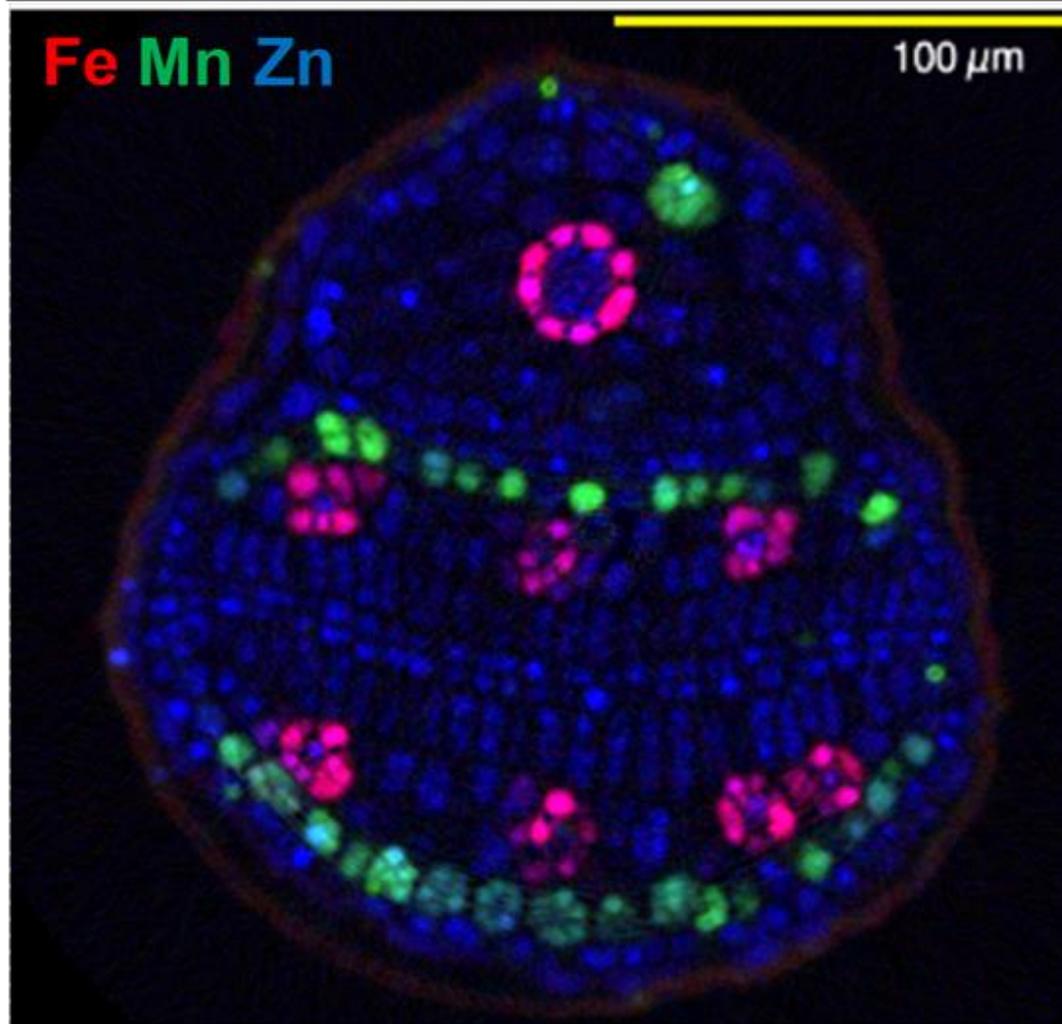
- Permite acessar a estrutura dos materiais de forma pouco invasivo (*in vivo*, condições dinâmicas...)
- Resultados qualitativos e quantitativos
- Pode ser medida em bancada ou síncrotron

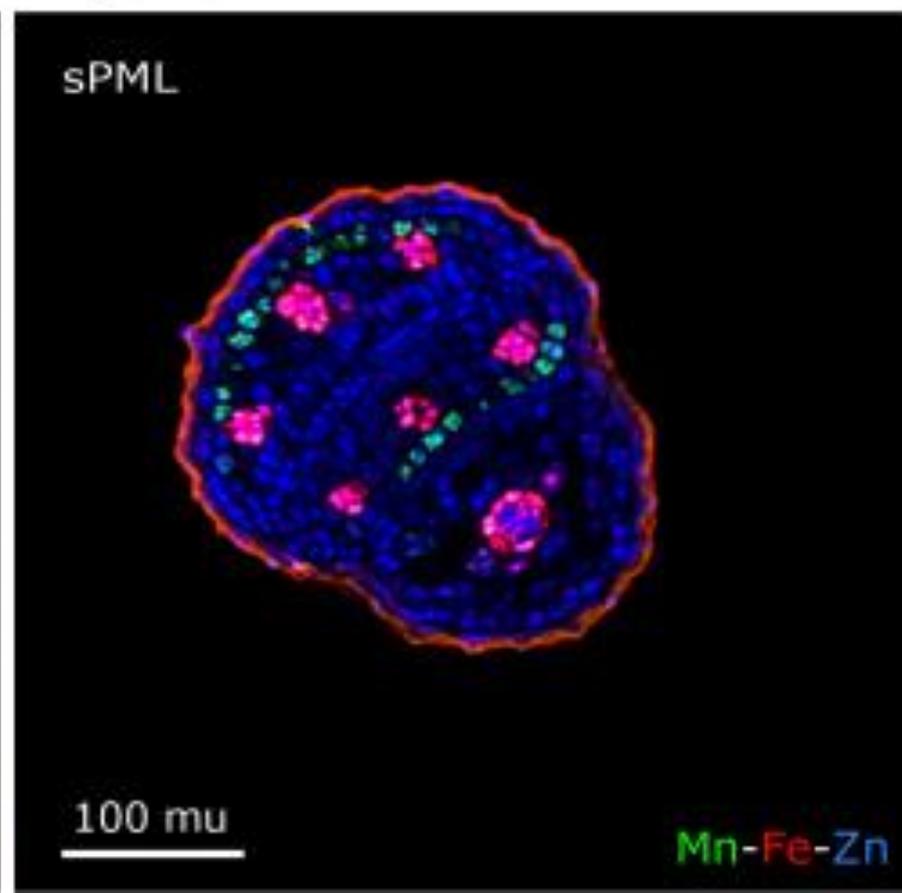
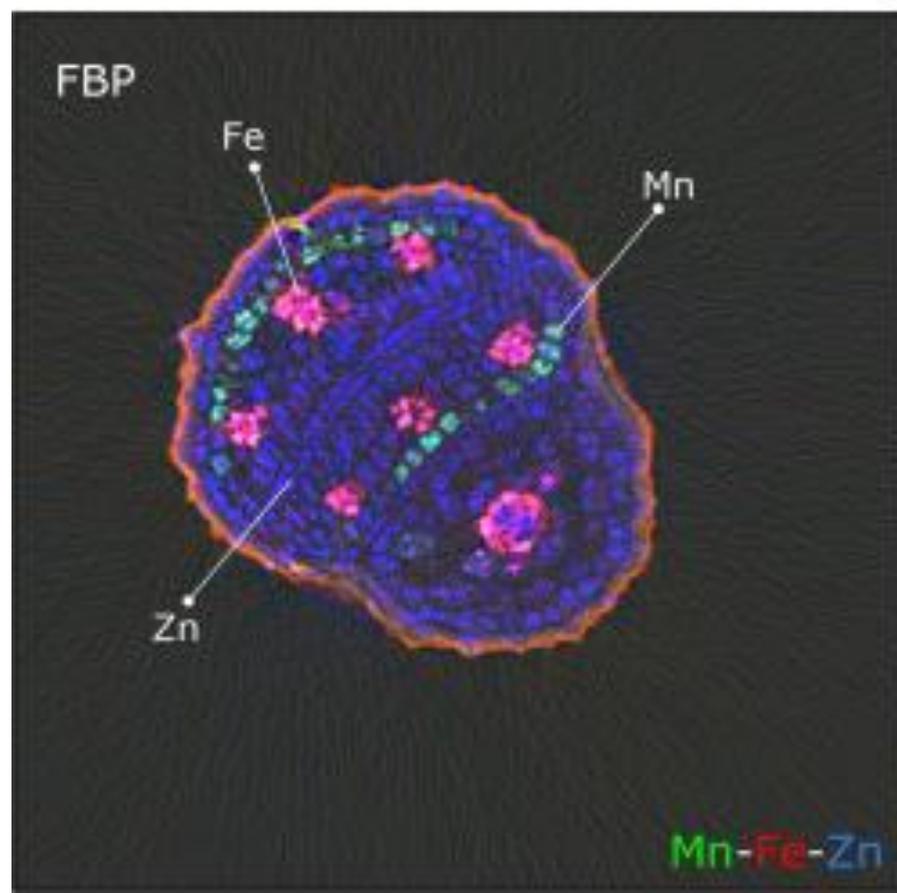
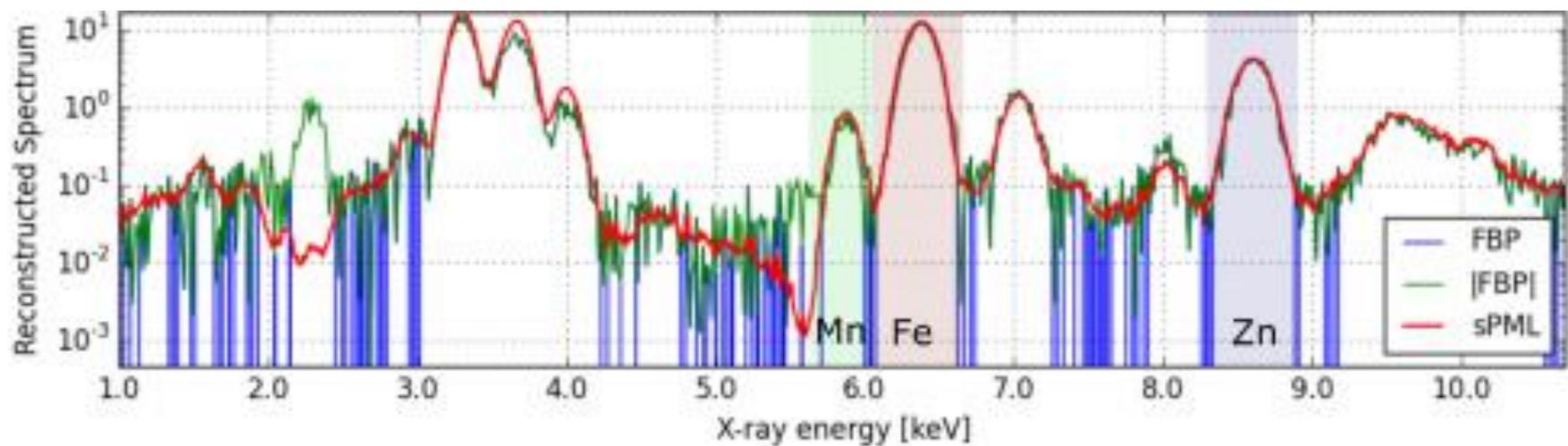


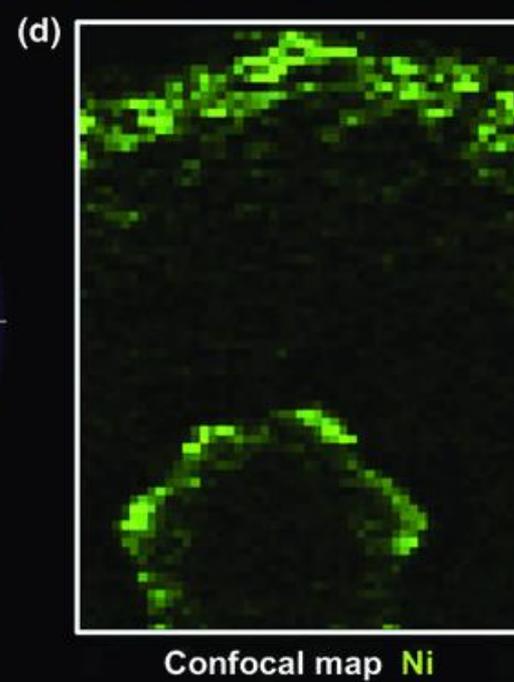
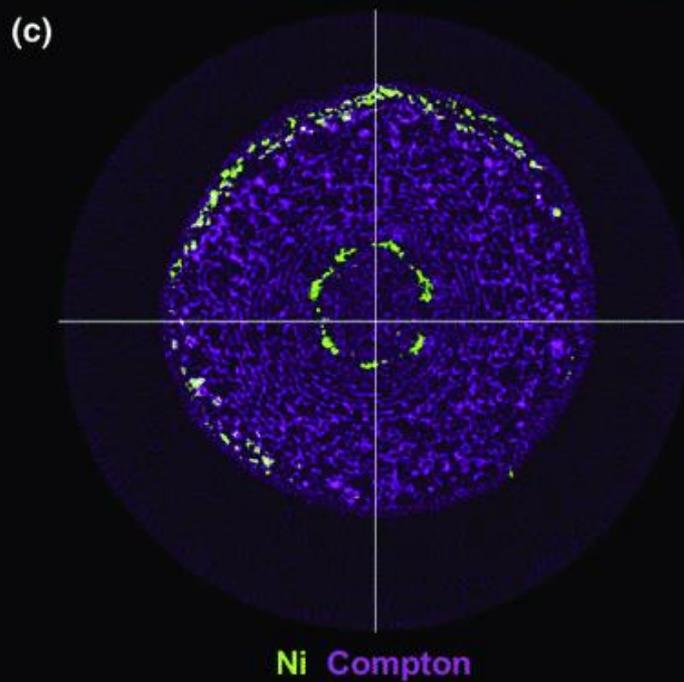
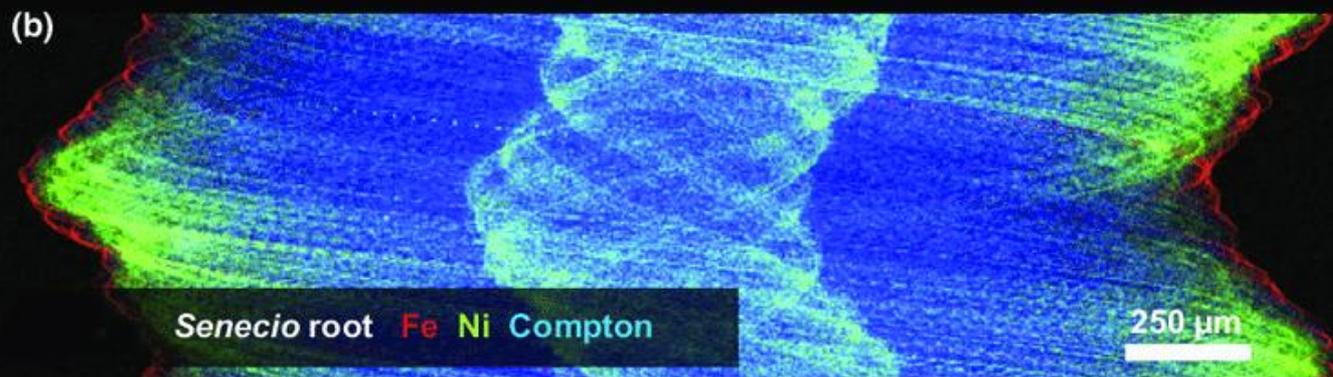
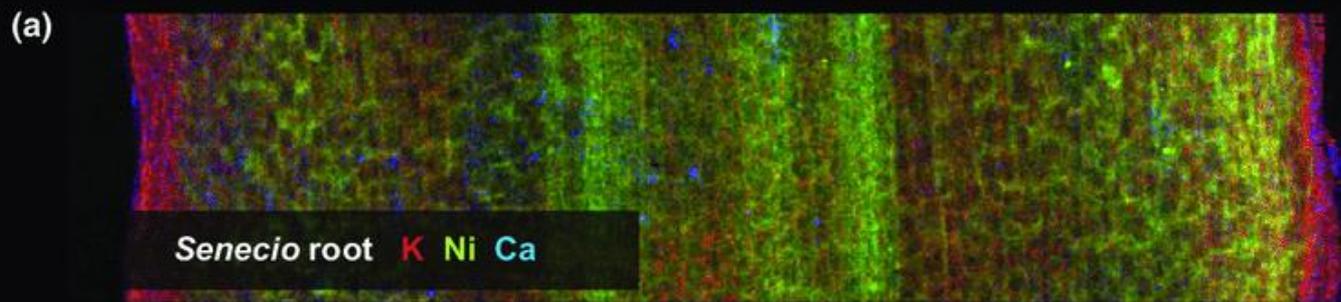
Current Opinion in Structural Biology



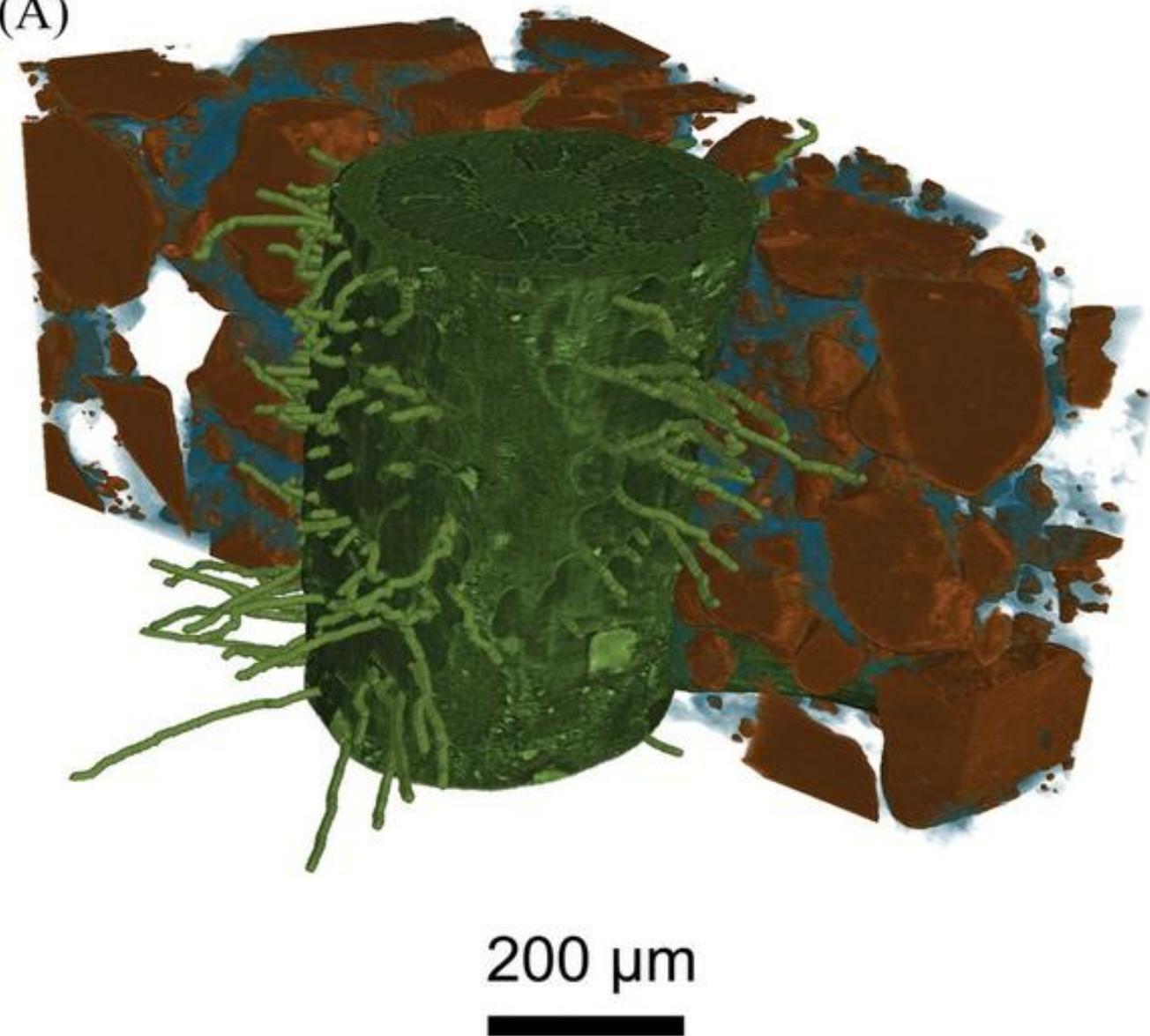
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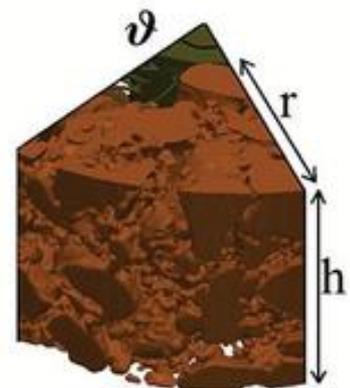




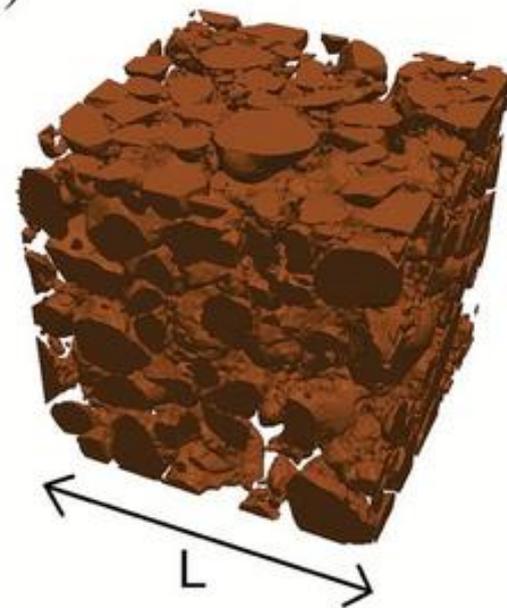
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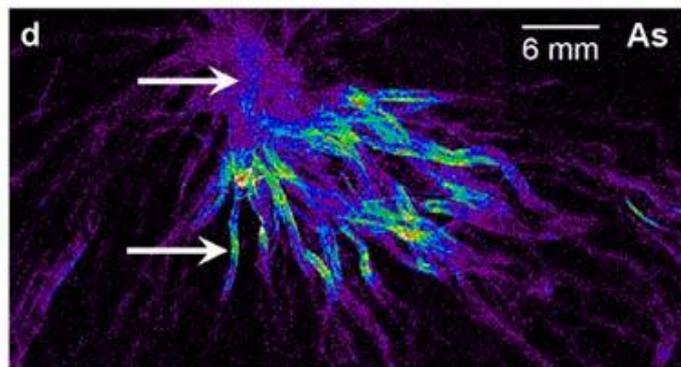
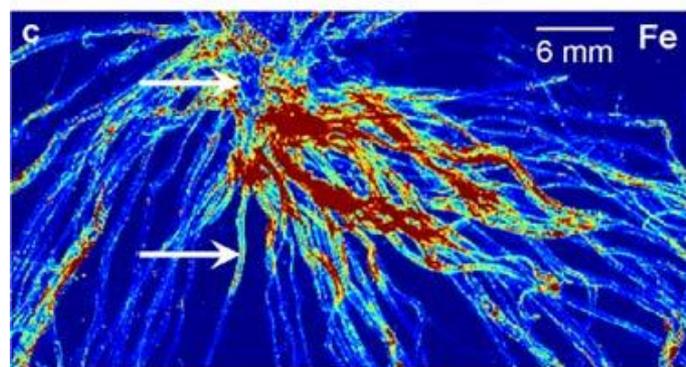
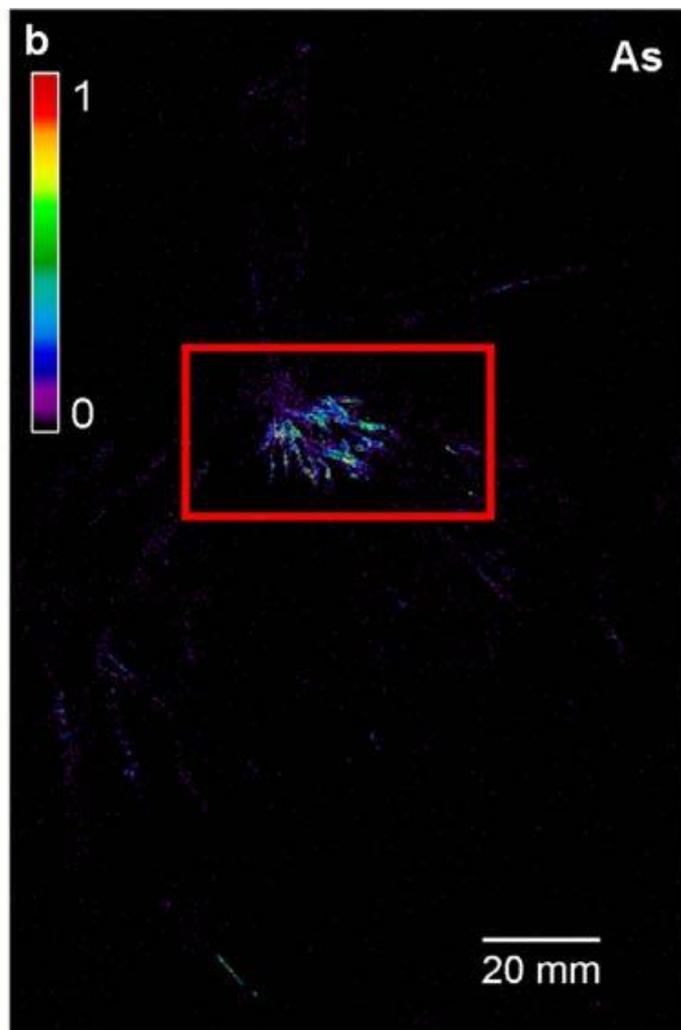
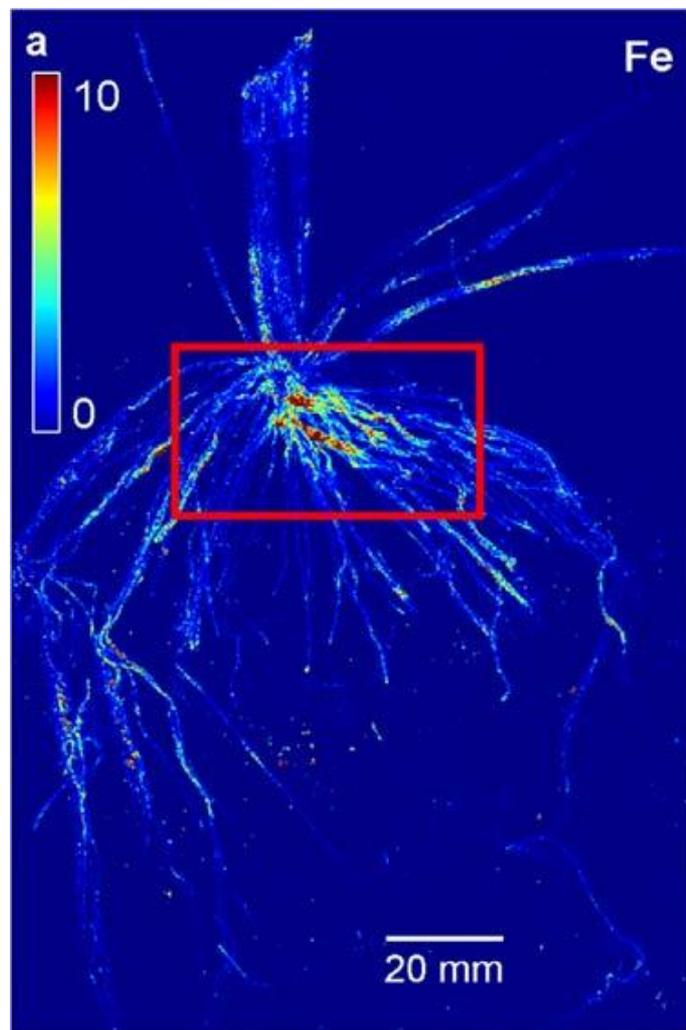


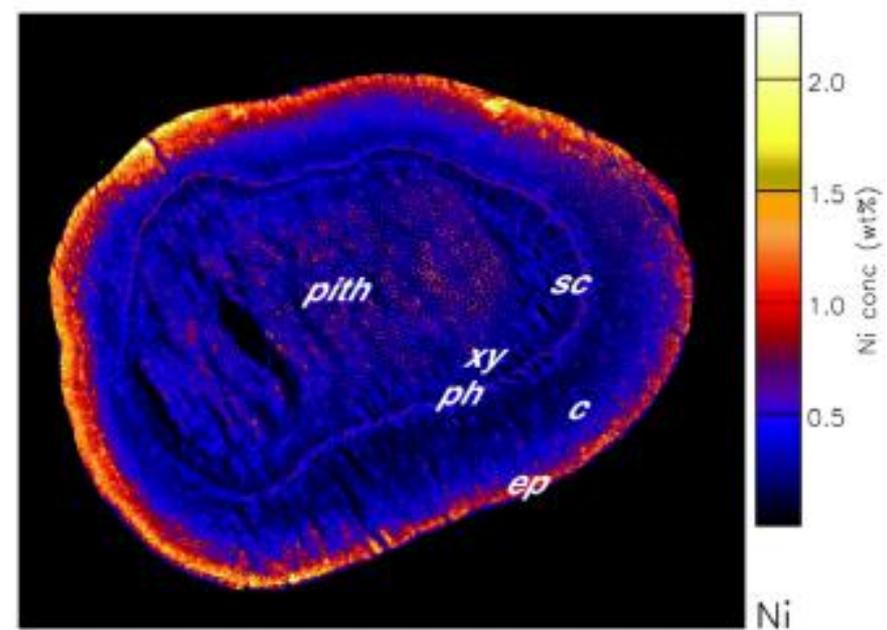
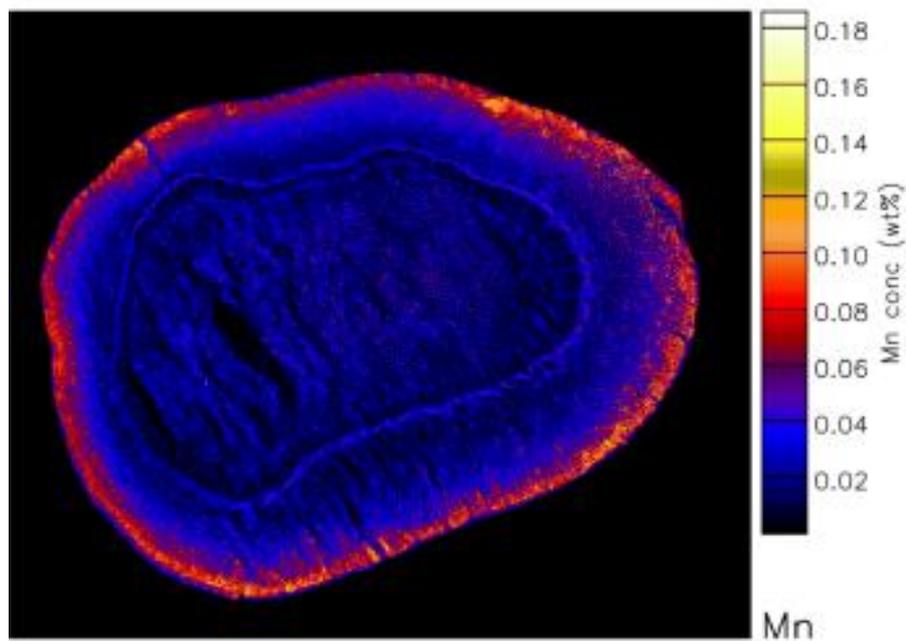
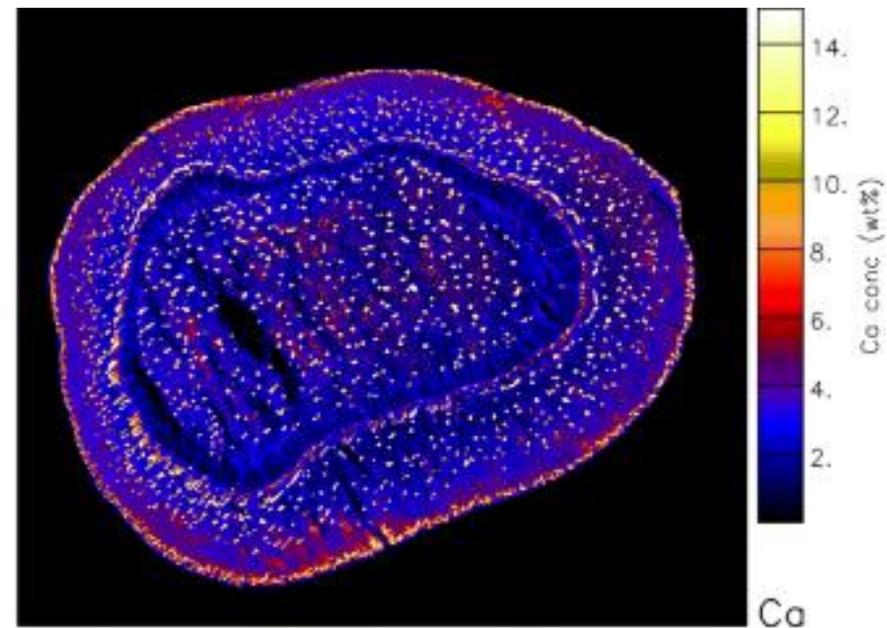
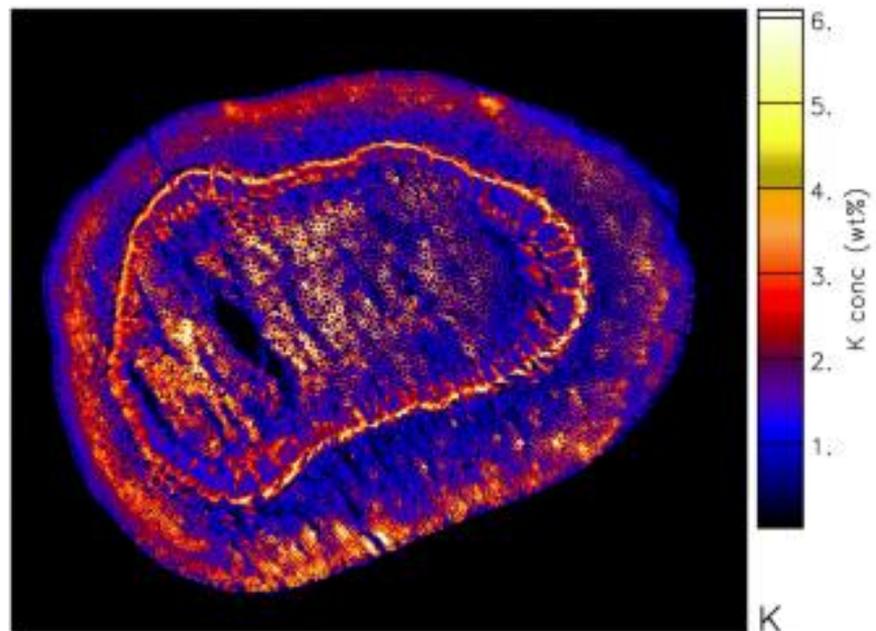
(B)



(C)







1000  $\mu\text{m}$