Elettra Sincrotrone Trieste

IAEA
International Atomic Energy Agency

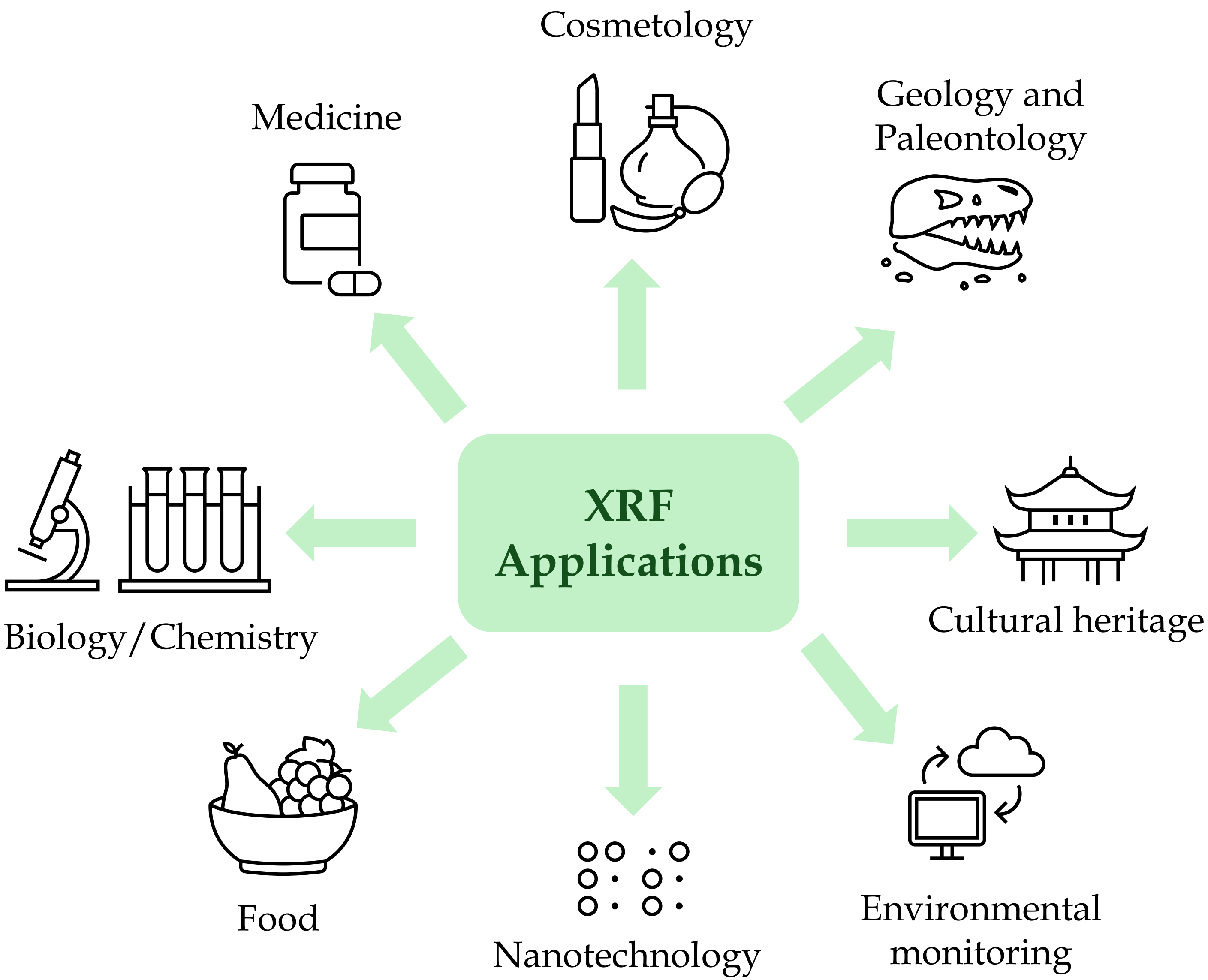
Examples of the application of XRF techniques

By Kasprzyk Paula

22th October 2024

IAEA Training Workshop, Elettra
21-25th October 2023

AGH



XRF
Applications
Medicine
Cosmetology
Geology and
Paleontology
Biology/Chemistry
Cultural heritage
Food
Nanotechnology
Environmental
monitoring

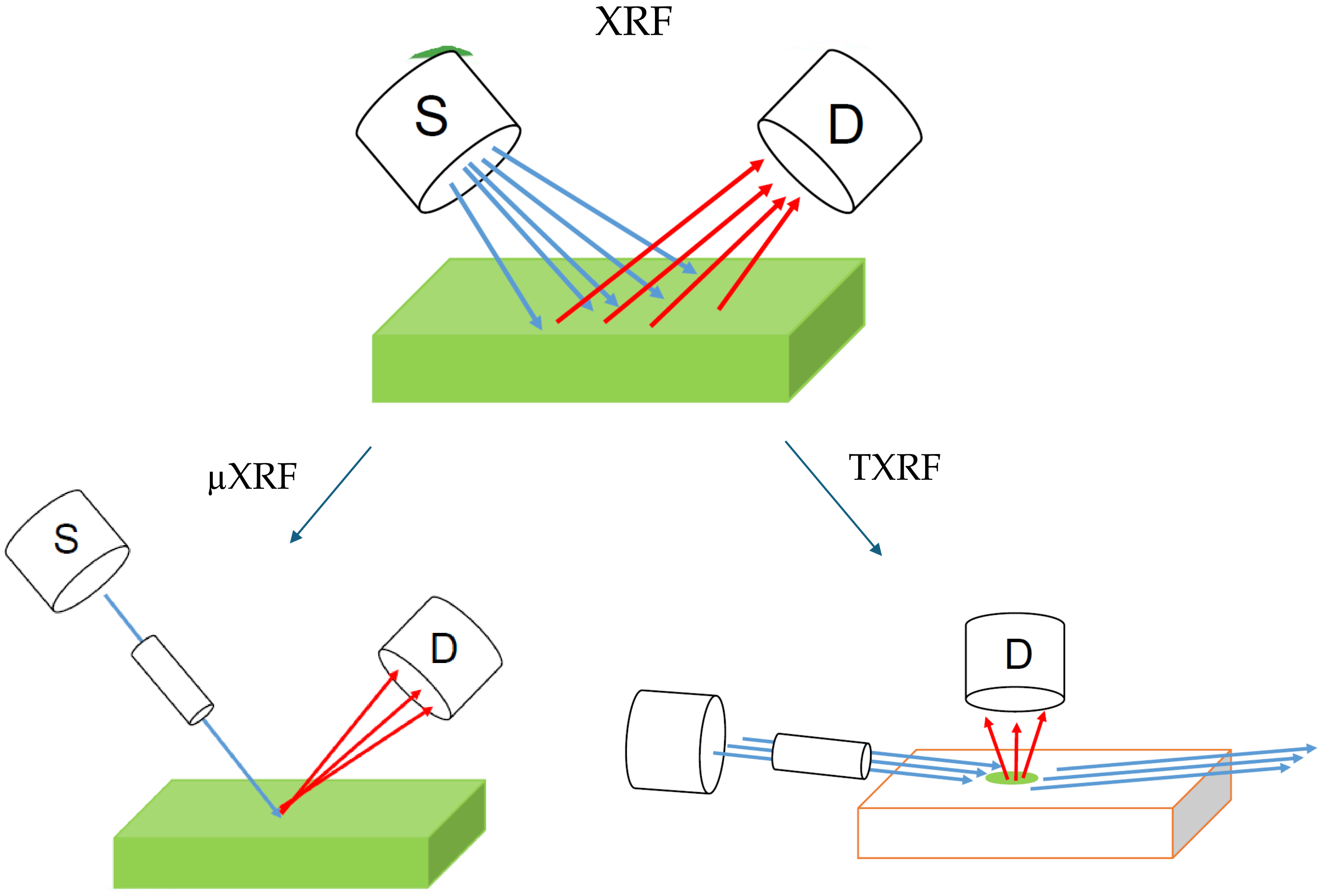
Analytical capabilities:

- Grazing Incidence X-ray Fluorescence (**GIXRF**) is a powerful technique for depth-profiling and characterization of thin layers in depths up to a few hundred nanometers.

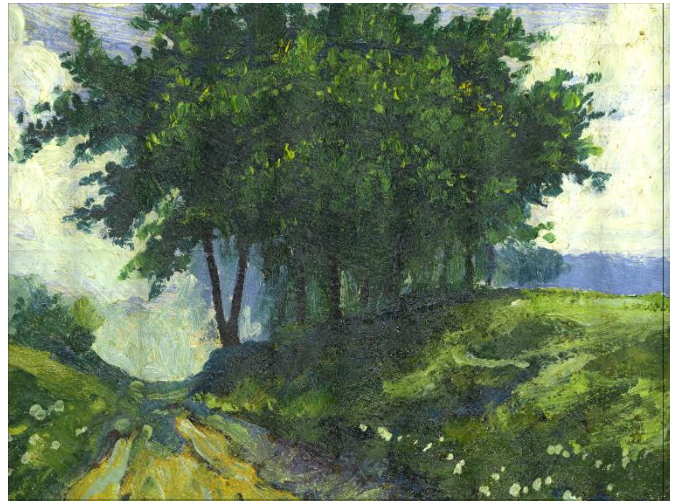
- Total Reflection X-Ray Fluorescence Analysis (**TXRF**) is an energy dispersive analysis in which almost parallel beam is impinging at angles below the critical angle on the surface of the reflector.

- Microscopic X-ray fluorescence analysis (**Micro-XRF** or **µXRF**) uses X-rays to determine the spatial distribution of major, minor and trace elements in a sample. The x-ray beam is focused to a small spot (in the µm range) onto the sample surface.

- X-Ray Reflectometry (**XRR**) is a non-destructive, highly accurate method used to determine thickness and roughness of thin layers, with thicknesses ranging from a few nanometers to some hundred nanometers, as well as the optical properties of the reflecting interfaces.

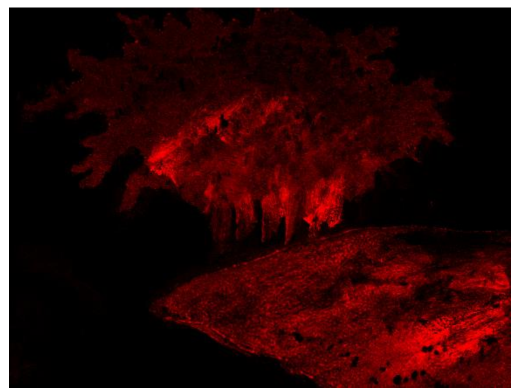

XRF
S
D
μXRF
S
D
TXRF
D

Cultural heritage



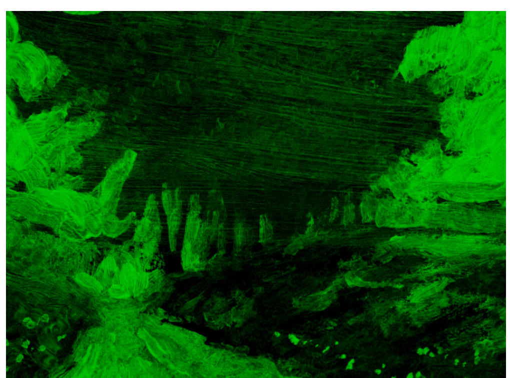
Fe

Cr

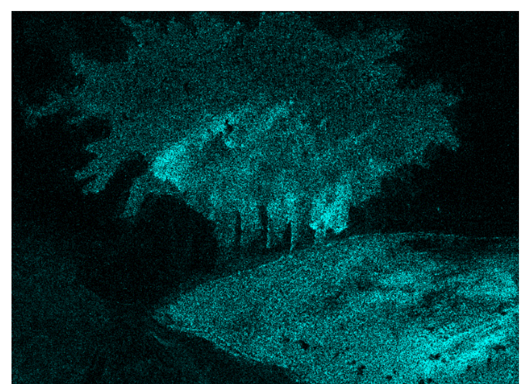


Ca

Ti



Mn



Cultural heritage

applied sciences

MDPI

Article

MA-XRF for the Characterisation of the Painting Materials and Technique of the *Entombment of Christ* by Rogier van der Weyden

Anna Mazzinghi [1,2,*], Chiara Ruberto [1,2], Lisa Castelli [2], Caroline Czelusniak [2], Lorenzo Giuntini [1,2], Pier Andrea Mandò [1,2] and Francesco Taccetti [2]

Entombment of Christ, belongs to the collection of the Uffizi gallery in Florence and was likely painted in 1460.

The research aims were to characterize the materials employed by the artist and to possibly understand his painting technique.

The detection of Ca traces associated with some areas possibly suggests the use of dyes or lakes in which a calco-potassic glass, present as a drier/siccative, is consistent with the Flemish technique during those times. ... this result may also in principle confirm the Flemish production of the painting and exclude the possibility that it was painted in Florence during the artists' journey to Italy, as the Italian glass used for this purpose were Mn-rich rather than Ca-rich.

Cultural heritage

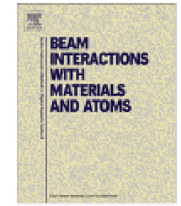
Nuclear Instruments and Methods in Physics Research B 269 (2011) 3041–3045

ELSEVIER

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

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



Characterization of the silver coins of the Hoard of Beçin by X-ray based methods

M. Rodrigues [a,b,*], M. Schreiner [a,b], M. Melcher [a,b], M. Guerra [c], J. Salomon [c,†], M. Radtke [d], M. Alram [e,f], N. Schindel [f]

Four hundred sixteen silver coins stemming from the Ottoman Empire (16th and 17th centuries) were analyzed.

The silver content of the analyzed specimen varies between 90% and 95%. These outcomes have not supported the historical interpretations, which predict that during the period studied a debasement of approximately 44% of the silver content of the coins should have occurred.

Cosmetology

Analytica Chimica Acta 1075 (2019) 27–37

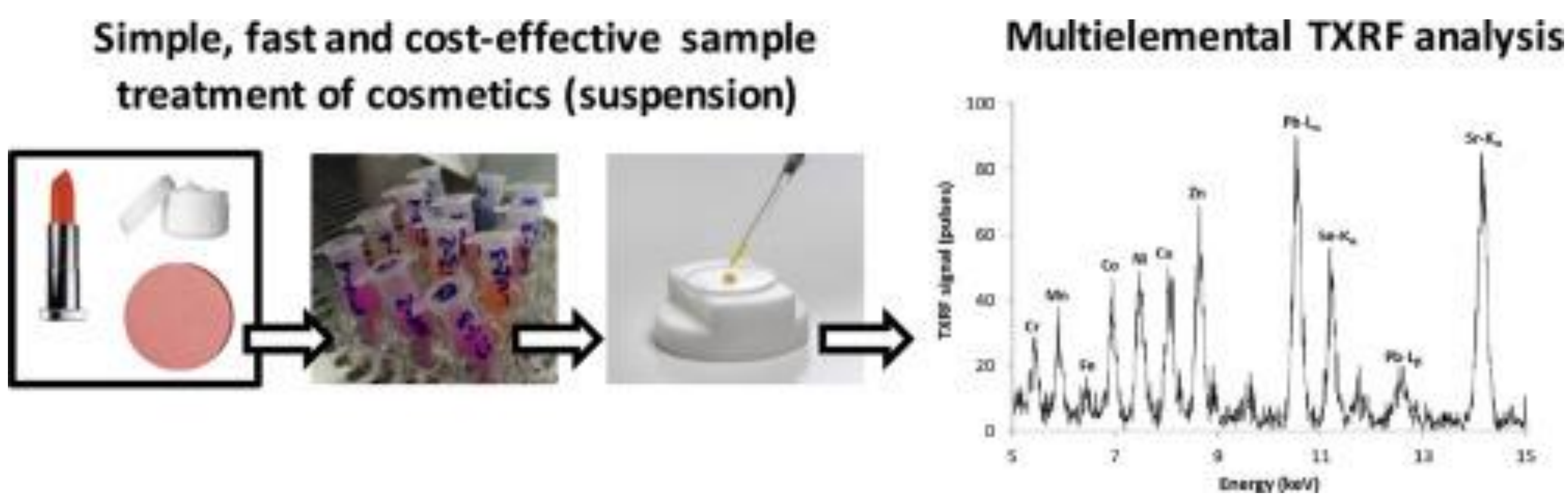
Contents lists available at ScienceDirect

Analytica Chimica Acta

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

Possibilities and drawbacks of total reflection X-ray fluorescence spectrometry as a fast, simple and cost-effective technique for multielement analyses of cosmetics

E. Marguí [a, *], R. Dalipi [b], L. Borgese [c], L.E. Depero [c], I. Queralt [d]



This paper shows a simple, rapid and cost-effective method for multielement analyses of cosmetics. Total reflection X-ray fluorescence spectrometry (TXRF) is used to determine the composition, particularly the presence of potentially toxic elements, of cosmetics.

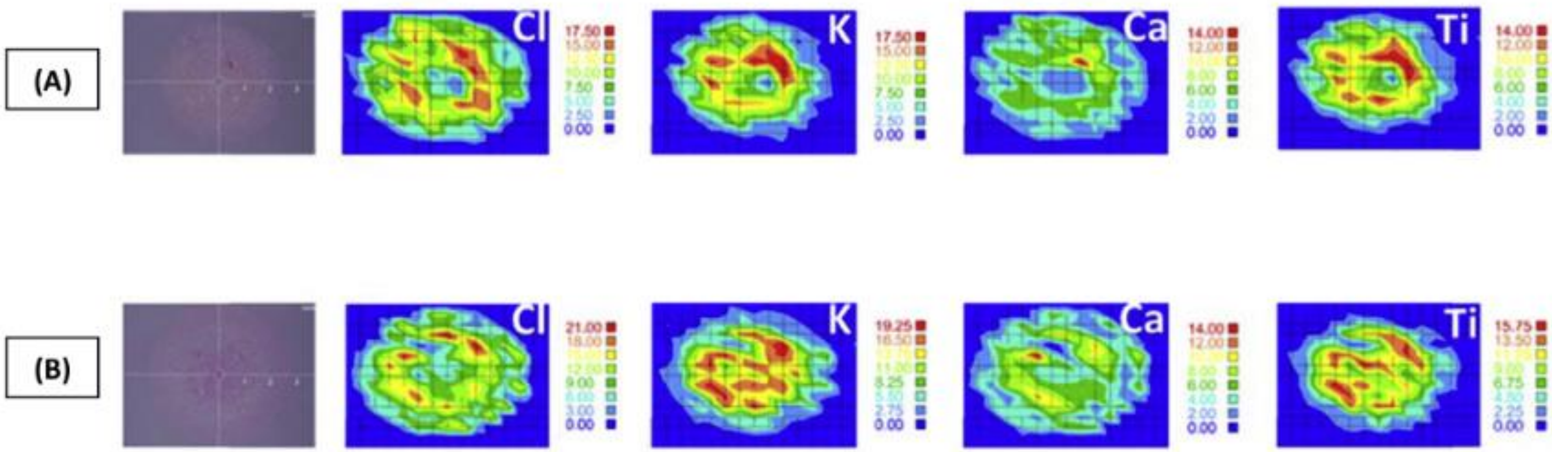
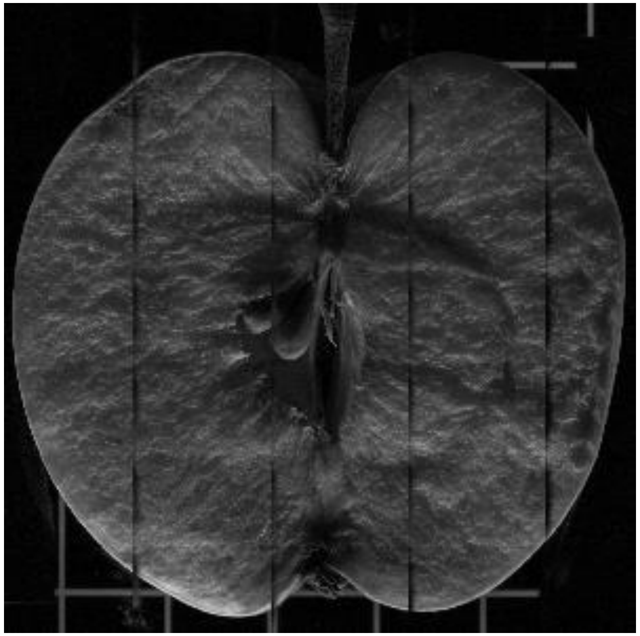
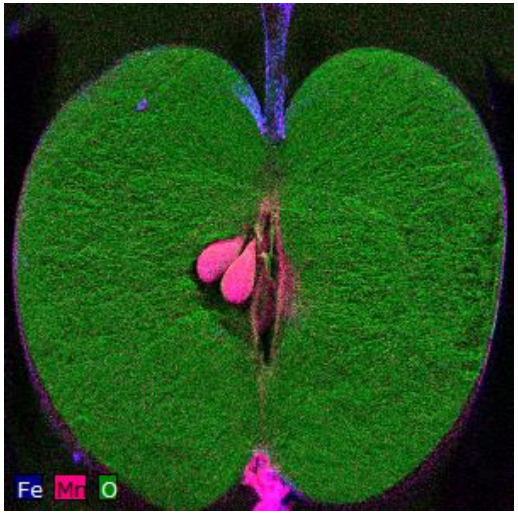


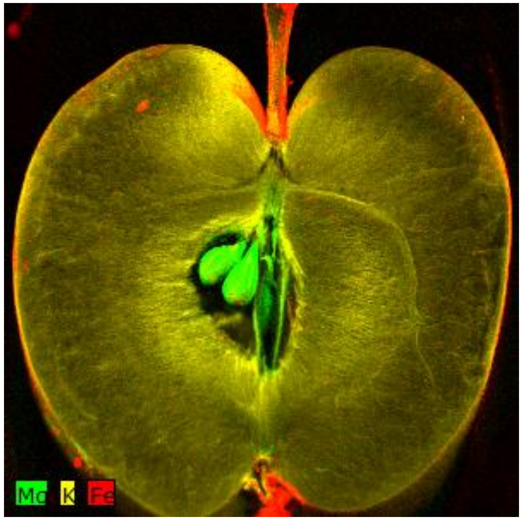
Fig. 4. 2D mappings for Cl, K, Ca and Ti in a 5 µL pink eye shadow sample spot in a quartz glass reflector, prepared by suspending 20 mg of sample in 1 mL Triton X without sonication before deposition (A) and with 5-min conventional sonication (B). The colour scale is expressed in µg cm^{-2} (measurement details: 10 kV, 1 mA, 200s, 0.3 mm, 15 × 15 map).

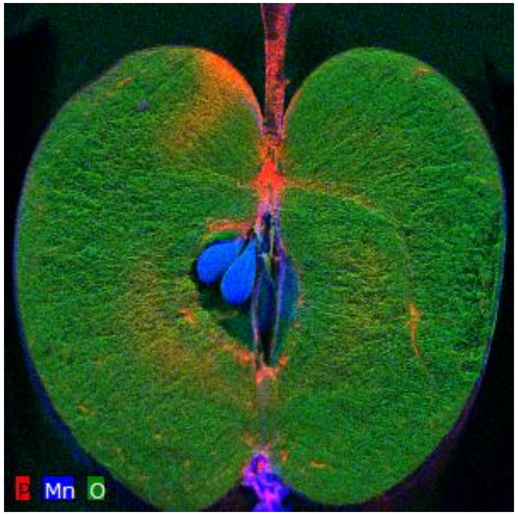
Results obtained for most elements in the lipstick and cream samples agreed with those obtained by the reference method recommended by the Food and Drug Administration.

Food









Food/Environmental

Environmental Research 125 (2013) 75–81

Contents lists available at ScienceDirect

Environmental Research

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

ELSEVIER

Selenium inhibits the phytotoxicity of mercury in garlic (*Allium sativum*)

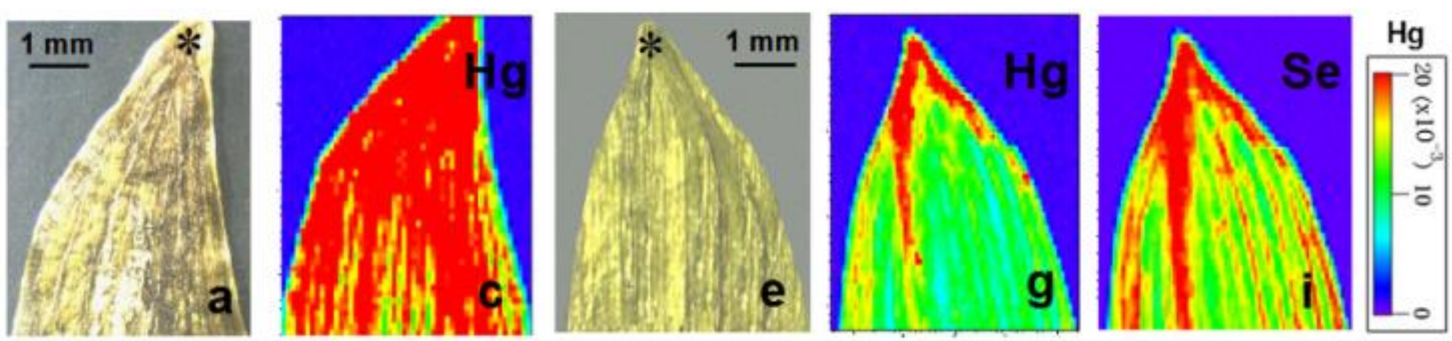
CrossMark

Jiating Zhao [a], Yuxi Gao [a,*], Yu-Feng Li [a], Yi Hu [a], Xiaomin Peng [a], Yuanxing Dong [b], Bai Li [a], Chunying Chen [a], Zhifang Chai [a,1]

Mercury (Hg) is one of the most hazardous pollutants in the environment. Its toxicity can be magnified in organisms at higher trophic levels due to its accumulation and transformation through food chain.

The distributions of selenium and mercury were examined with micro-synchrotron radiation X-ray fluorescence (μ-SRXRF), and the mercury speciation was investigated with micro-X-ray absorption near edge structure (μ-XANES).

Elemental imaging using μ-SRXRF also shows that Se could inhibit the accumulation and translocation of Hg in garlic.



Medicine/Biology

DOCTORAL THESIS

Biomolecular and elemental micro-analysis of the skeletal muscle in the quest of new tissue markers of neuromuscular diseases.

International Journal of *Molecular Sciences*

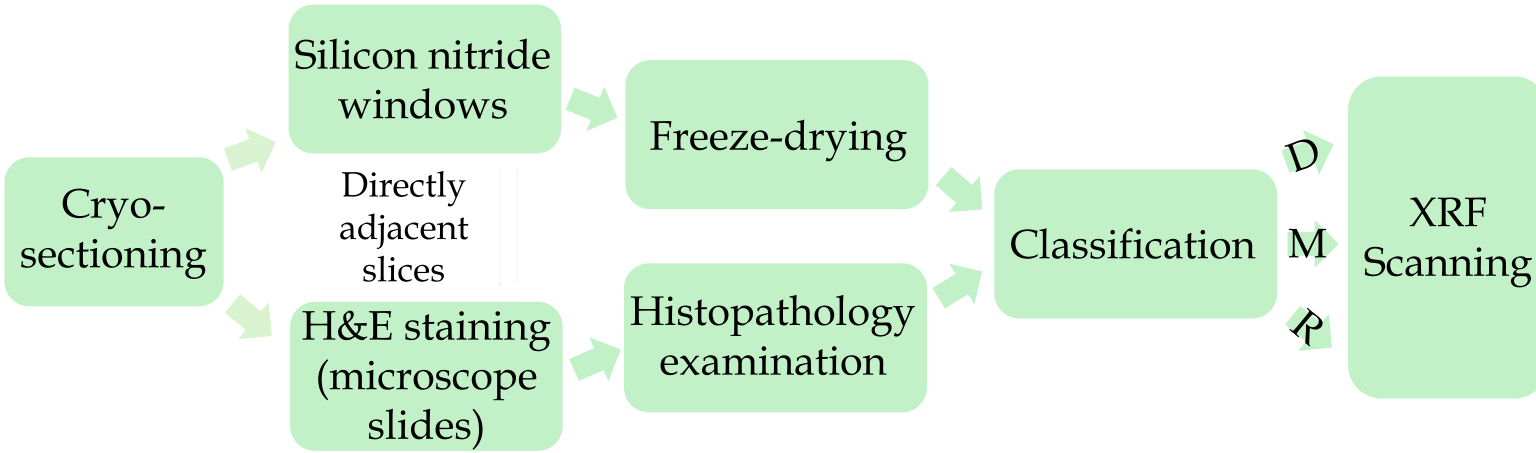
MDPI

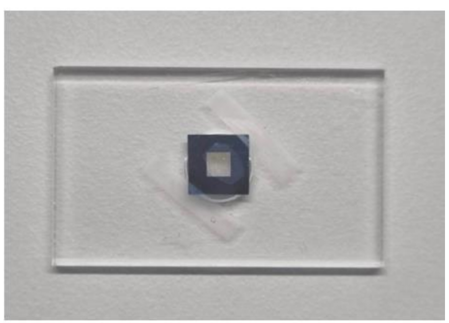
Article

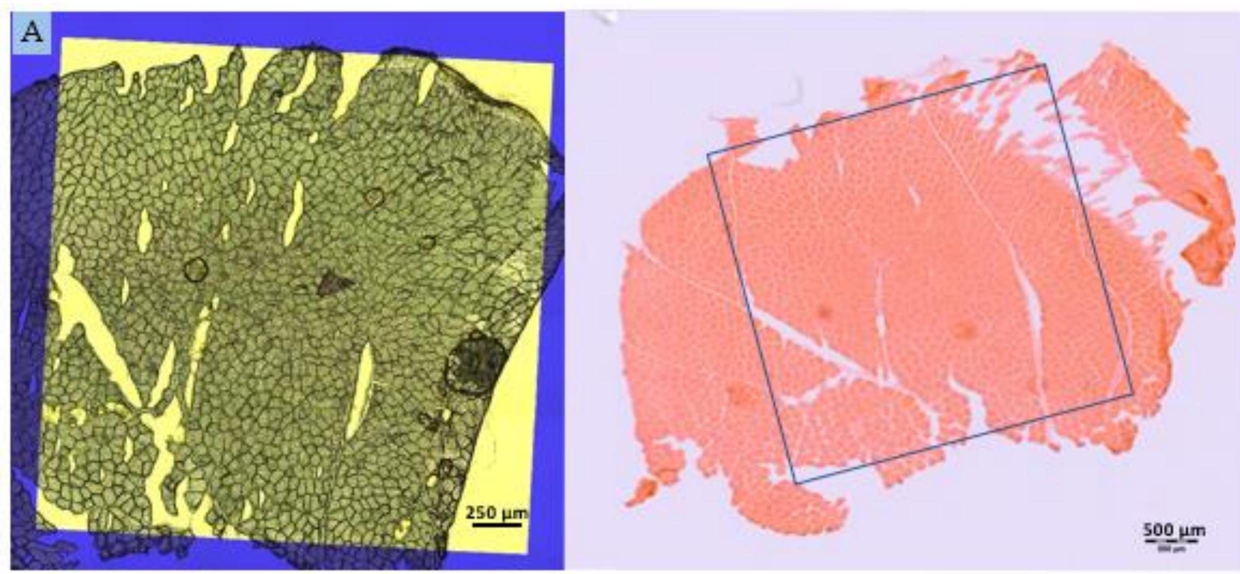
Elemental Composition of Skeletal Muscle Fibres Studied with Synchrotron Radiation X-ray Fluorescence (SR-XRF)

Paula Kasprzyk [1,*], Paweł M. Wróbel [1], Joanna Dudała [1], Kalotina Geraki [2], Magdalena Szczerbowska-Boruchowska [1], Edyta Radwańska [3], Roger M. Krzyżewski [4], Dariusz Adamek [3] and Marek Lankosz [1,*]

AGH

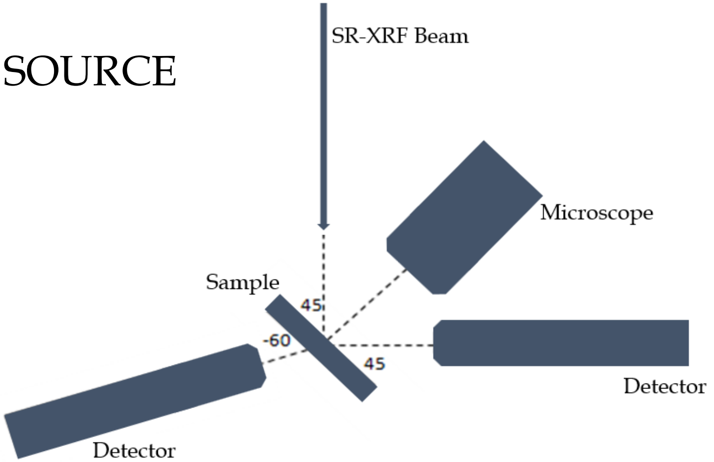

Cryo-sectioning
Silicon nitride windows
Directly adjacent slices
H&E staining (microscope slides)
Freeze-drying
Histopathology examination
Classification
D
M
R
XRF Scanning




A
250 µm
500 µm

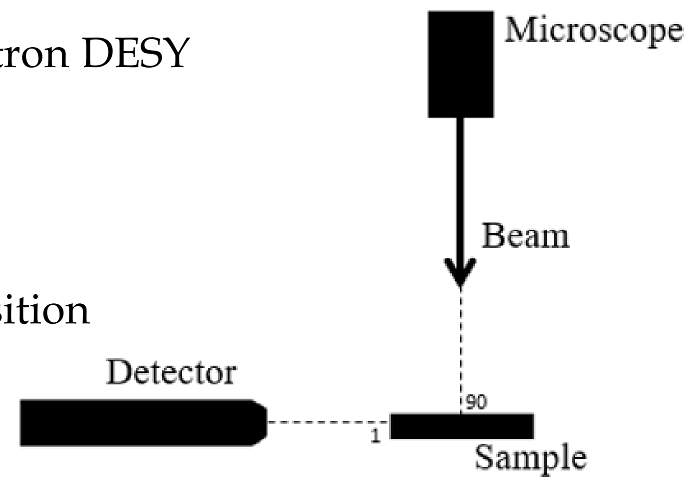
I18 beamline Synchrotron DIAMOND LIGHT SOURCE

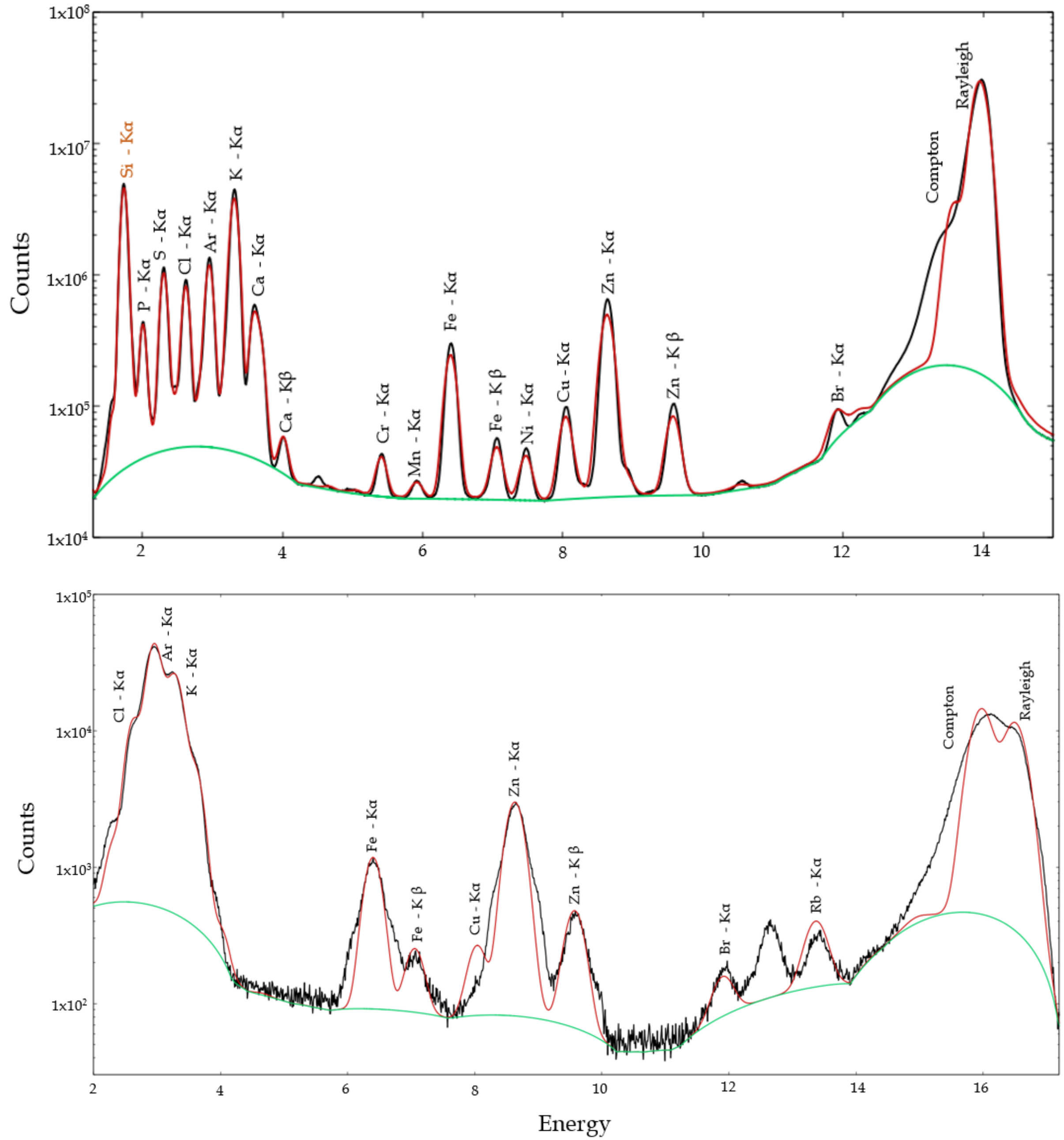
- beam size - 5 x 5 μm^2,
- energy of excitation - 13,95 keV,
- acquisition time per pixel - 4 s,
- geometry - 45°/45°,
- measurement of muscle fiber composition

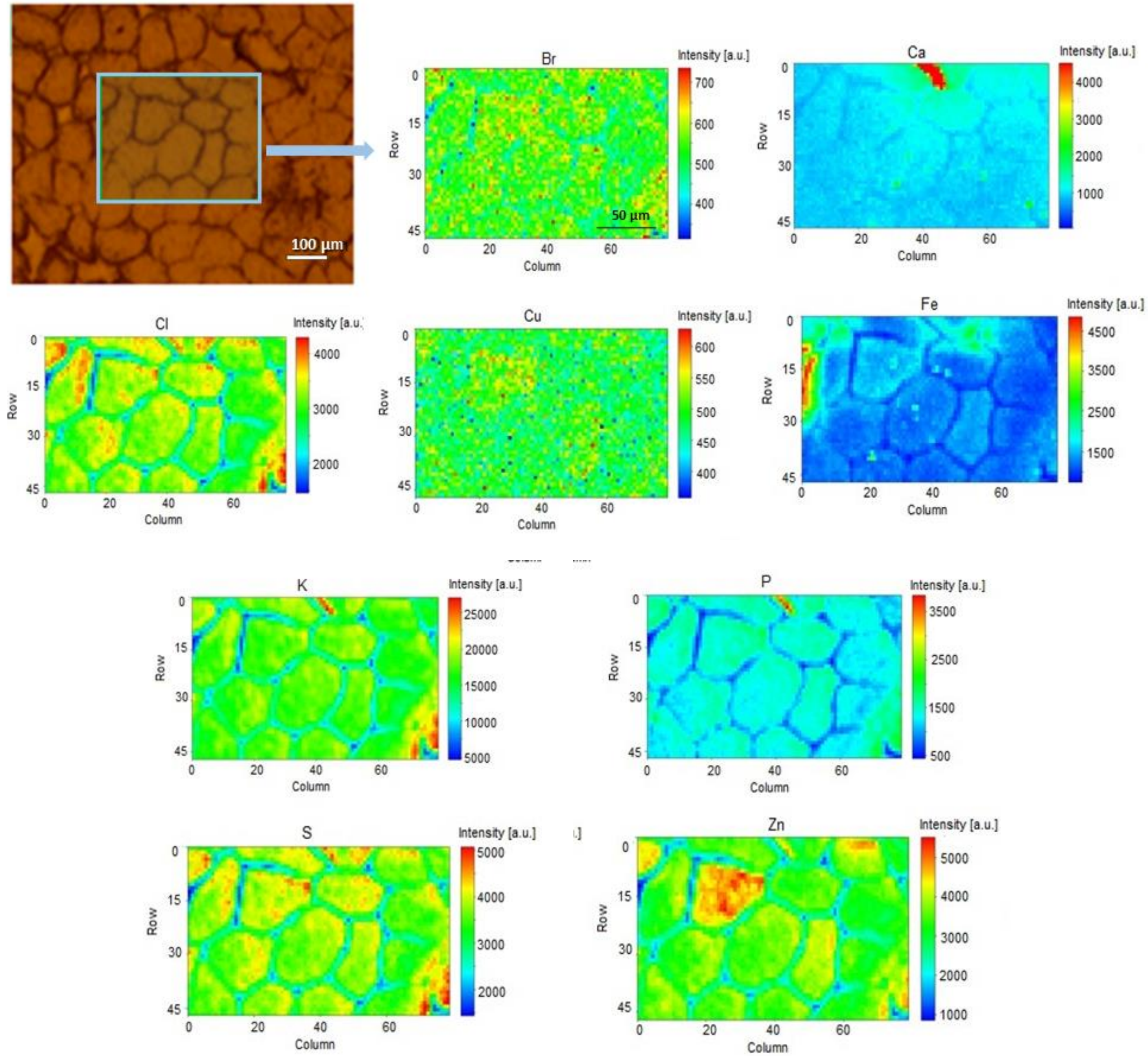


P06 beamline Deutsches Elektronen-Synchrotron DESY

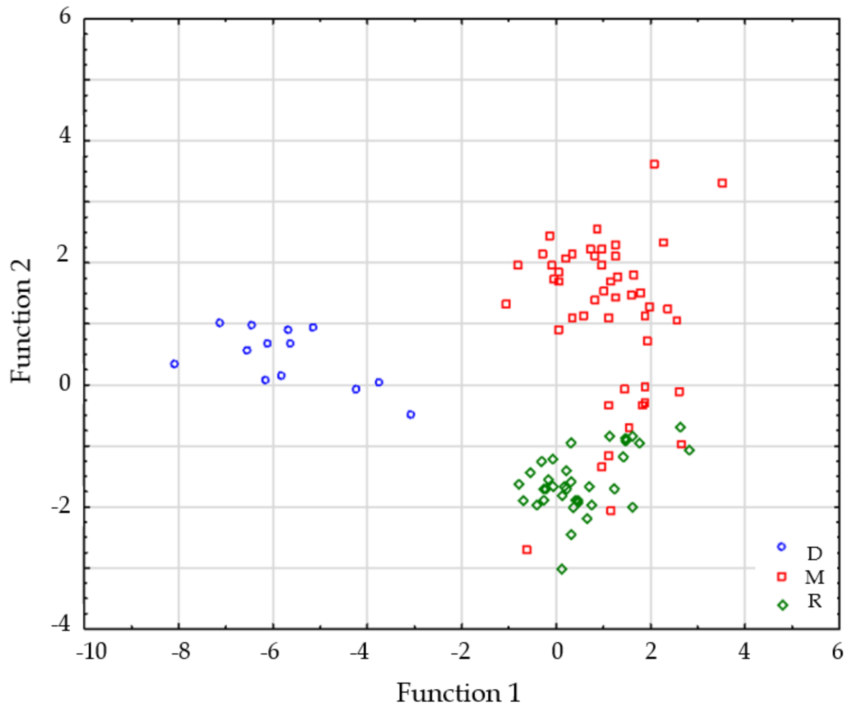
- beam size - 300 x 300 nm^2,
- energy of excitation - 16,5 keV,
- acquisition time per pixel - 4 s,
- geometry - 90°/1°,
- measurement of connective tissue composition

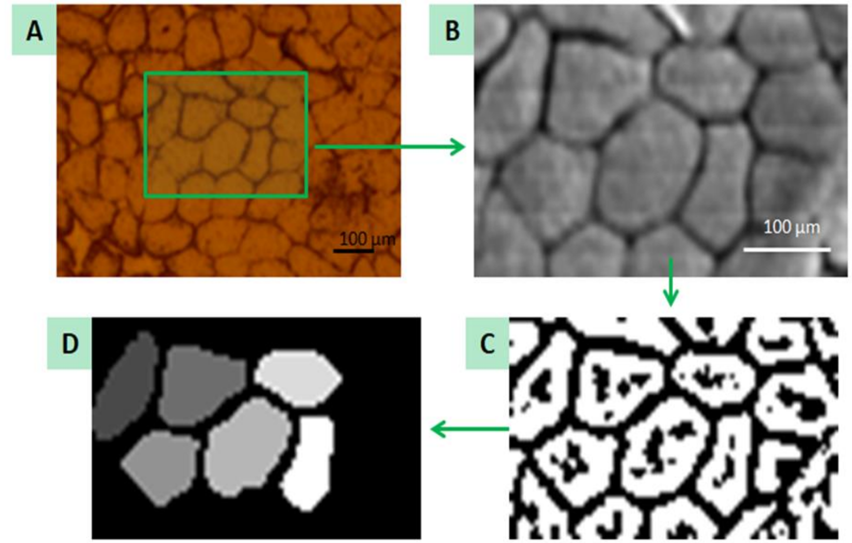



1x10^8
1x10^7
1x10^6
1x10^5
1x10^4
Counts
Si - Kα
P - Kα
S - Kα
Cl - Kα
Ar - Kα
K - Kα
Ca - Kα
Ca - Kβ
Cr - Kα
Mn - Kα
Fe - Kα
Fe - K β
Ni - Kα
Cu - Kα
Zn - Kα
Zn - K β
Br - Kα
Compton
Rayleigh
2
4
6
8
10
12
14
1x10^5
1x10^4
1x10^3
1x10^2
Counts
Cl - Kα
Ar - Kα
K - Kα
Fe - Kα
Fe - K β
Cu - Kα
Zn - Kα
Zn - K β
Br - Kα
Rb - Kα
Compton
Rayleigh
2
4
6
8
10
12
14
16
Energy



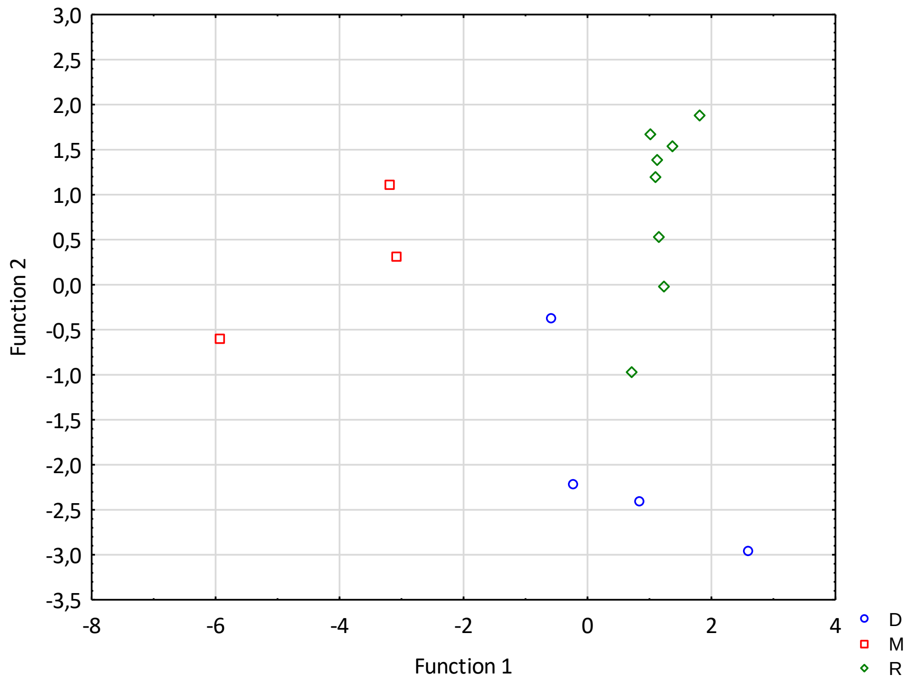
100 µm
Br
Intensity [a.u.]
700
600
500
400
50 µm
Row
Column
0
15
30
45
20
40
60
Ca
4000
3000
2000
1000
Cl
2000
Cu
550
450
Fe
4500
3500
2500
1500
K
25000
20000
15000
10000
5000
P
500
S
5000
Zn

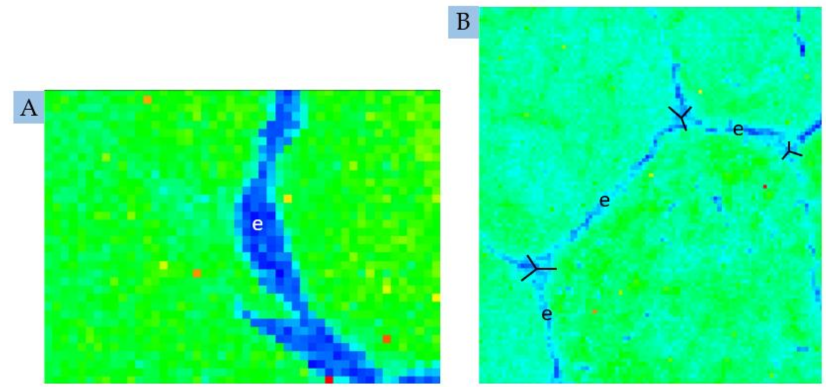


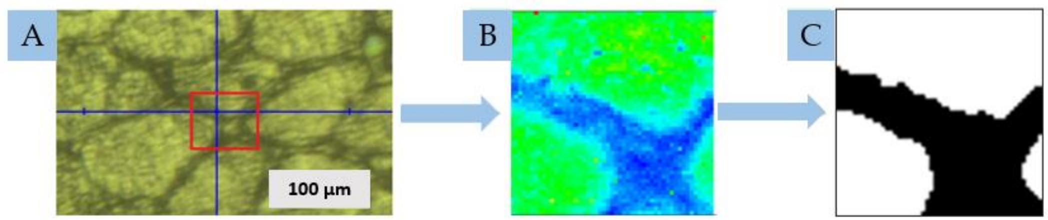


Element	Ca	K	P	Cu	Cl	Cr
Partial Wilks' lambda	0.536	0.696	0.754	0.929	0.905	0.536

	Group	%	Assignment			Total
			D	M	R	
Diagnosis	D	100%	13	0	0	13
	M	88%	0	43	6	49
	R	97%	0	1	37	38
	Total	93%	13	44	43	100







	Group	%	Assignment			Total
			D	M	R	
Diagnosis	D	75%	3	0	1	4
	M	100%	0	3	0	3
	R	87.5%	1	0	7	8
	Total	87%	4	3	8	15

Biology/Chemistry

Evaluation of changes in tissue material during fixation and formation processes.

Paula Kasprzyk[(1)], Paweł M. Wróbel[(1)], Łukasz Chmura[(2)], Konrad Kozlowski[(1)], Katarzyna Wątor[(3)], Magdalena Szczerbowska-Boruchowska[(1)]

Not published yet

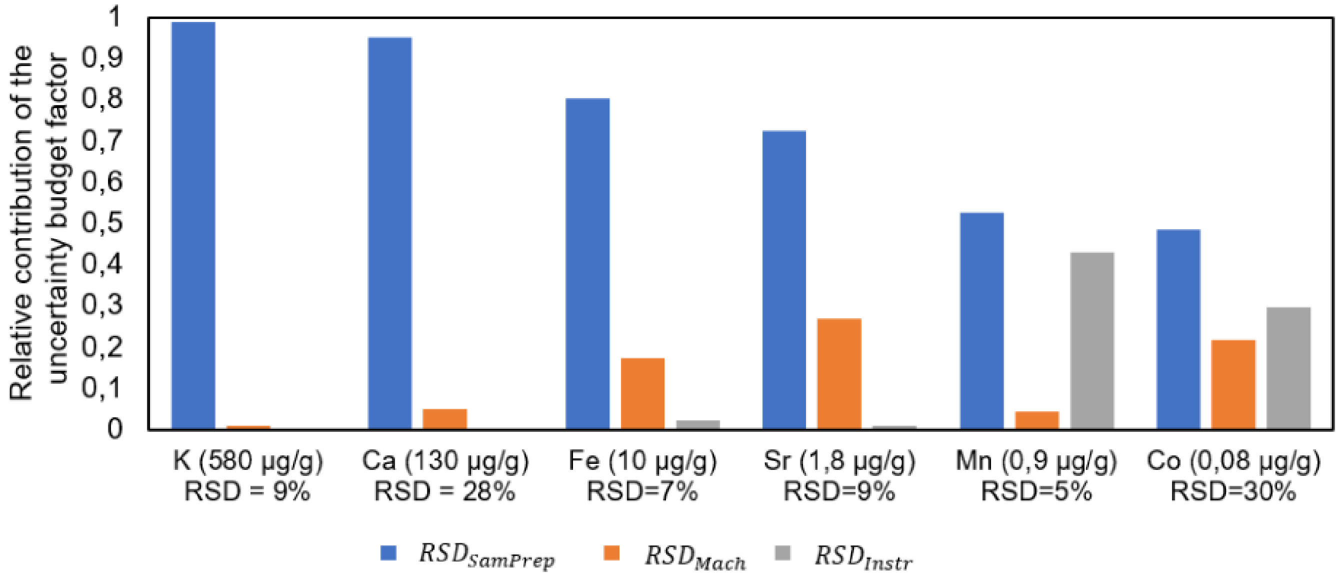
Question: How a standard preparation procedure for paraffin-embedded tissues affects the elemental composition of the tissues?
Is this effect being reproducible?

- uncertainity budget of TXRF
- optimization of sample deposition
- validation
- comparison of samples composition
- influence fixation on composition – time dependence

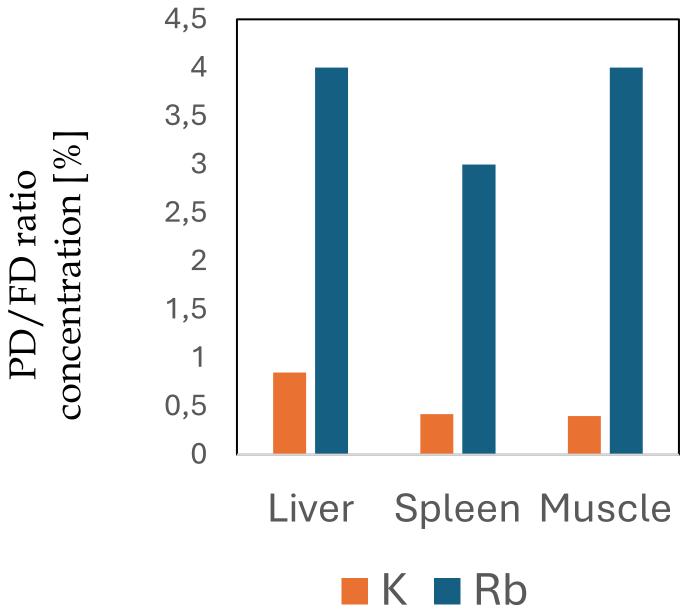
UNCERTAINTITY BUDGET

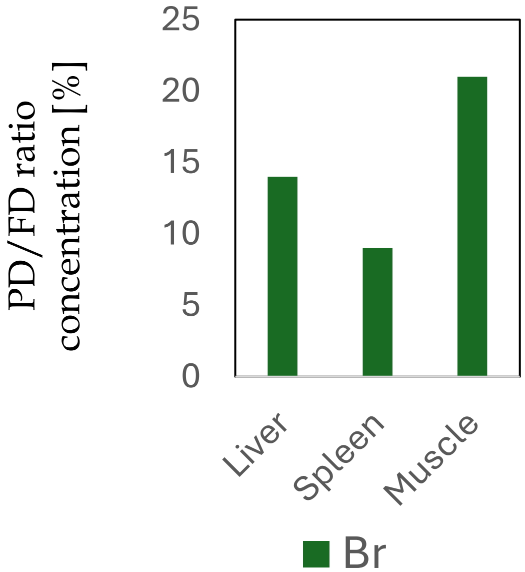
$$RSD_{Total} = \sqrt{RSD^2_{Instr} + RSD^2_{Mach} + RSD^2_{SamPrep}}$$

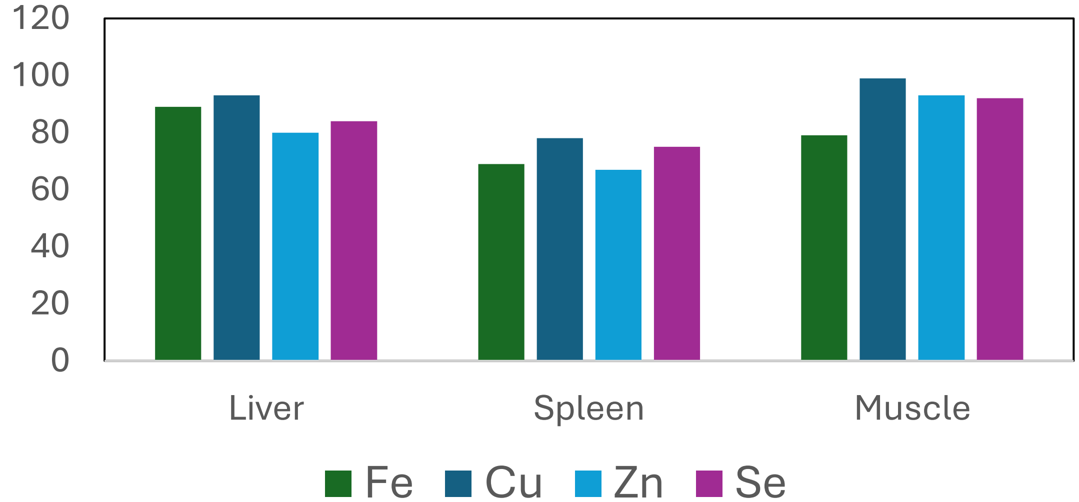
- number of repetitions $N = 10$
- $RSD^2 = \frac{1}{\bar{C}^2}\frac{\sum_{i=0}^{N}(C_i-\bar{C})^2}{N-1}$
- RSD^2_{Instr} - sample at one fixed position
- $RSD^2_{Mach} + RSD^2_{Instr}$ - one sample at ten different positions in sample changer
- RSD^2_{Total} - samples at different positions



Comparison of FD and PD samples concentration







Thank you for your attention