Interdisciplinary challenges in a digital multidisciplinary world

Karla Balzuweit

Departamento de Física e Centro de Microscopia da Universidade Federal de Minas Gerais

karla@fisica.ufmg.br

karlaweit@gmail.com

Laboratório de Microanálise

- Multiuser Fapemig project
- Microprobe – Jeol JXA 8900

- Universidade Federal de Minas Gerais
  - Chemistry department
  - Geology department
  - Physics department
- CDTN - CNEN – Centro de Desenvolvimento de Energia Nuclear

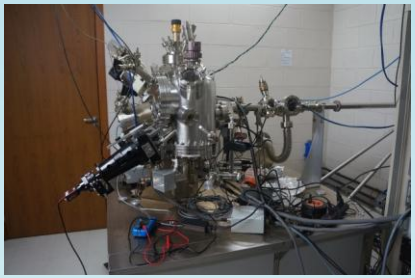
Centro de Microscopia Universidade Federal de Minas Gerais

- **multidisciplinar**  **interdisciplinar**

- Reference of microscopy in the State of Minas Gerais and the country.
- Human resources training in several levels.
- Dissemination of science in various levels.
- Support to other Institutions, Universities, Research Centers and Private Sector form the state of Minas Gerais and the country.

www.microscopia.ufmg.br

Centro de Microscopia da UFMG



LCPNano

- Physics department multiuser facility
- Simple research equipments for undergraduate and graduate students

http://lcpnano.ufmg.br/

ANTECIPA

ASSOCIAÇÃO NACIONAL DE PESQUISA
EM TECNOLOGIA E CIÊNCIA DO PATRIMÔNIO

Fundada em dezembro de 2015, durante o evento IPERION-BR Primeiro Encontro da Rede de Laboratórios Associados, com a participação dos principais laboratórios brasileiros que realizam pesquisa na área de Ciência da Conservação.

- Preservação do patrimônio cultural
- Cooperação com universidades, centros de pesquisas, bem como outras entidades.
- Fórum de intercâmbio entre pesquisadores, programas de Pós-Graduação, entidades e organizações.
- Promoção e apoio à pesquisa transdisciplinar, a produção científica e tecnológica e a sistematização, difusão e intercâmbio de informações e conhecimentos do campo expandido da Ciência do Patrimônio.
- Promoção da cooperação técnica com entidades e organismos voltados à preservação do Patrimônio Cultural, no Brasil e no exterior, a formação técnica e a organização de eventos técnico-acadêmico-científicos.

http://lacicor.eba.ufmg.br/antecipa/

UFMG

IPERION HS

centro de microscopia da UFMG

www.iperionhs.eu

IPERION HS was a consortium of **24 partners from 23 countries** that contributes to establishing a pan-European research infrastructure on heritage science. It offers training and access to a wide range of high-level scientific instruments, methodologies, data and tools for advancing knowledge and innovation in heritage science.

IN2PAST.BR

DF UFMG DEPARTAMENTO DE FÍSICA

UFMG

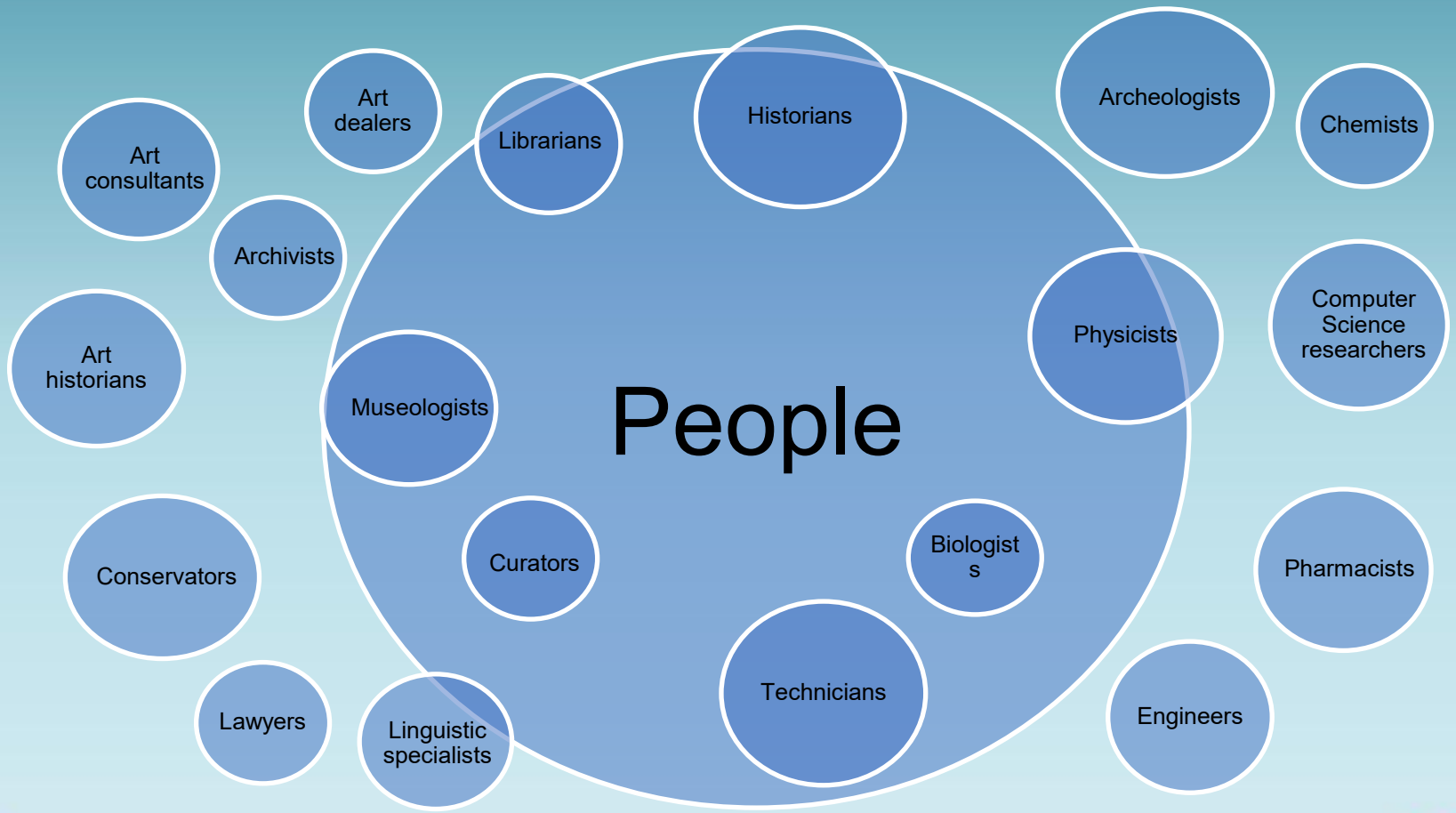
E-RIHS
EUROPEAN RESEARCH INFRASTRUCTURE FOR HERITAGE SCIENCE

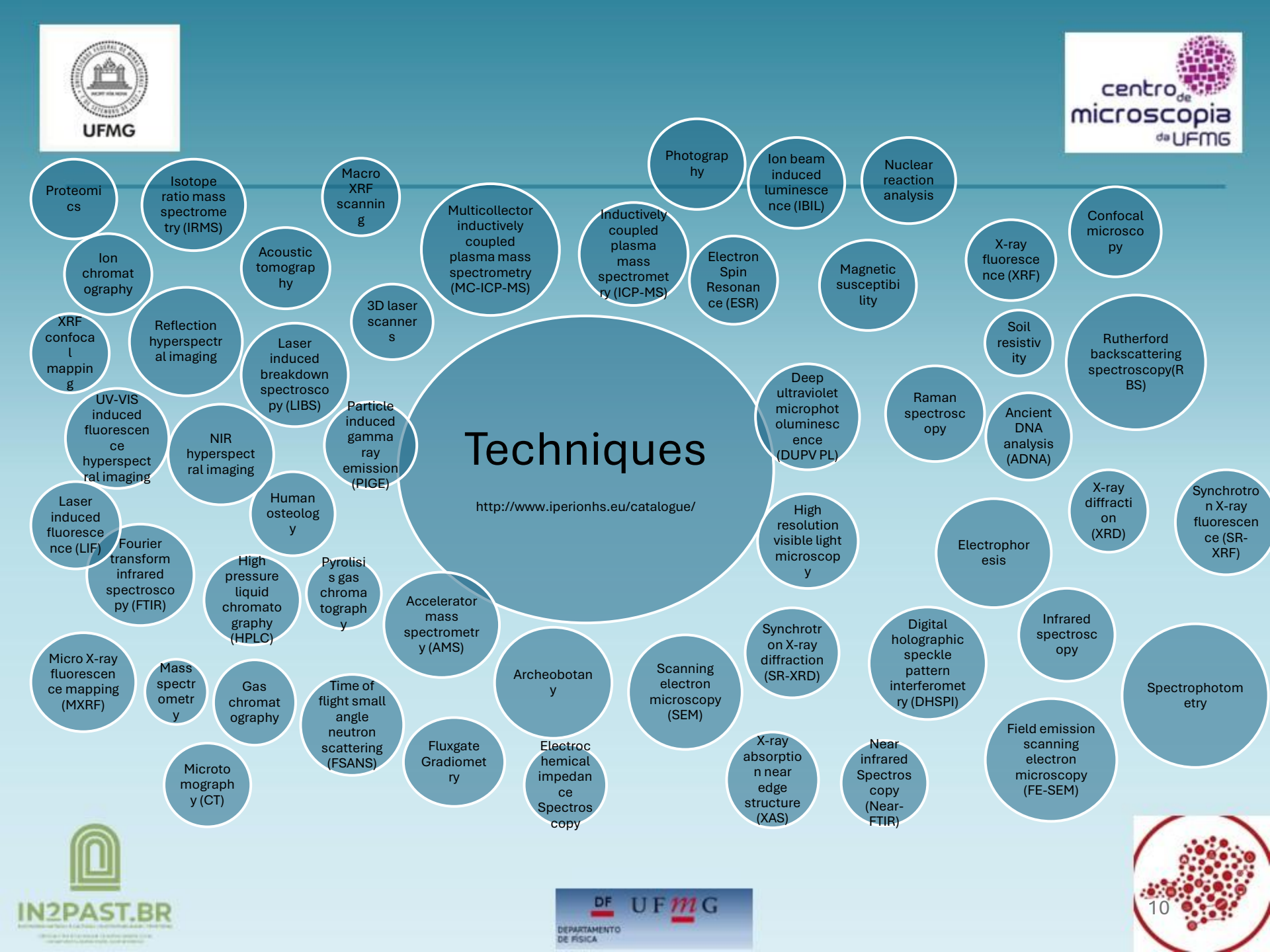
centro de microscopia da UFMG

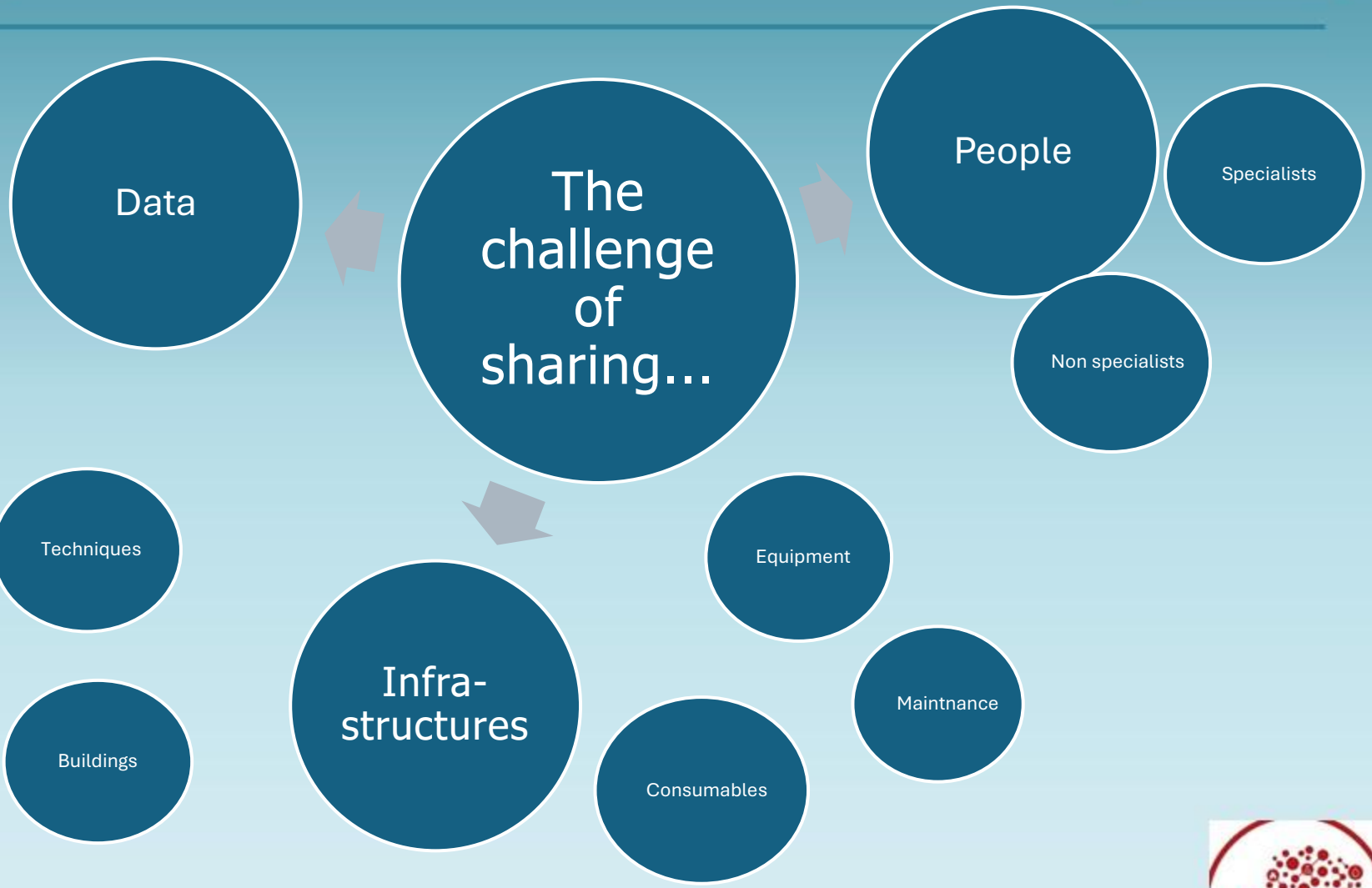
- ESFRI Roadmap Project since 2016 (GSO since 2015). Step 1 to E-RIHS ERIC submitted in 2021, Step2 to be submitted soon...
- Distributed research infrastructure (~ 100 facilities) involving 28+ countries
- 4 access platforms providing world class expertise, digital data, reference collections, laboratories and mobile instruments, building a clear identity for heritage science and linking across communities at the global level

IN2PAST.BR

DF UFMG
DEPARTAMENTO DE FÍSICA

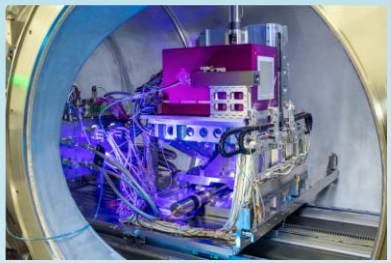

People
Art dealers
Librarians
Historians
Archeologists
Chemists
Art consultants
Archivists
Art historians
Museologists
Physicists
Computer Science researchers
Conservators
Curators
Biologists
Pharmacists
Lawyers
Linguistic specialists
Technicians
Engineers


Techniques
http://www.iperionhs.eu/catalogue/
Proteomics
Isotope ratio mass spectrometry (IRMS)
Macro XRF scanning
Multicollector inductively coupled plasma mass spectrometry (MC-ICP-MS)
Photography
Ion beam induced luminescence (IBIL)
Nuclear reaction analysis
Inductively coupled plasma mass spectrometry (ICP-MS)
Electron Spin Resonance (ESR)
Magnetic susceptibility
X-ray fluorescence (XRF)
Confocal microscopy
Ion chromatography
Acoustic tomography
3D laser scanners
Reflection hyperspectral imaging
XRF confocal mapping
Laser induced breakdown spectroscopy (LIBS)
Soil resistivity
Rutherford backscattering spectroscopy(RBS)
UV-VIS induced fluorescence hyperspectral imaging
NIR hyperspectral imaging
Particle induced gamma ray emission (PIGE)
Deep ultraviolet microphotoluminescence (DUPV PL)
Raman spectroscopy
Ancient DNA analysis (ADNA)
Laser induced fluorescence (LIF)
Human osteology
Fourier transform infrared spectroscopy (FTIR)
High pressure liquid chromatography (HPLC)
Pyrolisis gas chromatography
Accelerator mass spectrometry (AMS)
High resolution visible light microscopy
Electrophoresis
X-ray diffraction (XRD)
Synchrotron X-ray fluorescence (SR-XRF)
Micro X-ray fluorescence mapping (MXRF)
Mass spectrometry
Gas chromatography
Time of flight small angle neutron scattering (FSANS)
Archeobotany
Scanning electron microscopy (SEM)
Synchrotron X-ray diffraction (SR-XRD)
Digital holographic speckle pattern interferometry (DHSPI)
Infrared spectroscopy
Spectrophotometry
Microtomography (CT)
Fluxgate Gradiometry
Electrochemical impedance Spectroscopy
X-ray absorption near edge structure (XAS)
Near infrared Spectroscopy (Near-FTIR)
Field emission scanning electron microscopy (FE-SEM)


Data
The challenge of sharing...
People
Specialists
Non specialists
Techniques
Buildings
Infra-structures
Equipment
Maintnance
Consumables

Infra-structurs

Sirius – Brazilian new synchrotron



https://www.lnls.cnpem.br/

MOLAB – Micro XRF

http://www.iperionch.eu/molab/

Challenges

Data interoperability - Interdisciplinarity

- Definitions
- Language – Culture
- Vocabulary (Different regions can have different definitions and/or different vocabulary
- **Good practices, standards, protocols, policies**
- **Data and metadata format**
- **Diferent types of data/files due to differences in equipment: different techniques, new, old, analog, digital, commercial, home-made, etc.**
- **Open source software , proprietary software, “black boxes”…**
- **Storage**
- **…….**

Words and expressions have meaning and context!

Data too!!

Physics – vol4. – waves and optics - Halliday Resnick – older versions

Lead Oboe (English version) → Oboé de chumbo – Brazilian portugues version

https://www.evanstonsymphony.org/content/oboesjpg

Lead → lead[1] /lēd/ → Leading Oboé

Lead → lead[2] /led/ → Plumbun

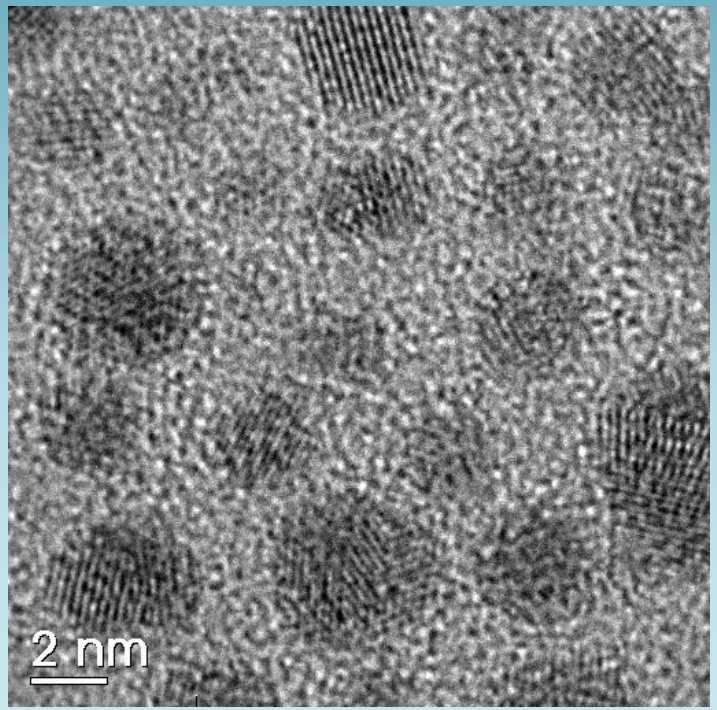
https://pt.wikipedia.org/wiki/Chumbo

Specialized knowledge/ more sophisticated algorithms

Digital images or data

- Channel - 1 D data - spectra
- Pixel - 2D data - 2D image
- Voxel - 3D, 4D, 5D, 6D, etc
  multi or hyperspectral image

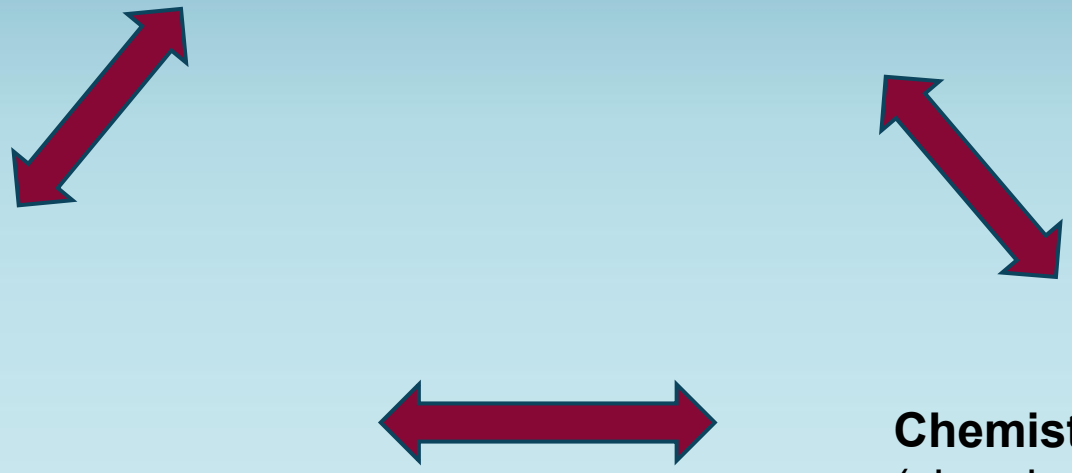
Digital image / corresponding xy intensity table 1024x1024 pixels, 256 grey tones



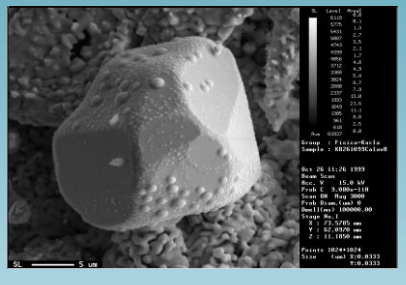
811	788	751	743	704	704	716	750	778	786	788	806	848	882	889	861	879	887	917
761	759	733	708	693	714	718	702	744	769	773	783	837	864	900	916	915	912	894
753	727	731	718	717	758	762	753	748	790	820	821	841	861	885	929	906	907	902
779	742	738	764	759	754	789	806	793	814	839	848	860	883	881	876	902	890	875
763	743	745	813	819	781	769	837	839	853	850	851	866	861	852	888	882	887	900
757	751	767	828	842	836	858	882	874	862	833	827	826	842	831	825	847	876	906
796	807	826	842	854	892	901	899	889	840	840	816	792	782	799	804	832	877	901
820	865	874	889	868	909	928	895	881	861	840	817	773	762	809	815	844	885	923
810	813	856	880	879	885	928	925	896	892	864	826	771	746	773	813	839	893	904
826	826	854	905	891	913	931	928	888	878	855	814	806	791	776	823	852	889	898
876	891	931	913	911	931	953	932	888	863	846	828	814	784	779	817	833	857	893
900	914	951	946	934	956	947	945	918	872	825	800	791	767	797	836	863	891	904
864	867	928	945	939	937	927	946	928	869	822	822	788	786	825	874	870	896	918
839	851	883	909	918	940	930	915	890	843	860	856	818	808	825	876	889	890	911
839	868	866	886	864	886	888	874	860	841	854	867	824	814	849	891	897	881	886
805	836	828	864	835	836	834	816	829	826	826	834	862	861	912	928	927	903	901
782	803	807	826	821	808	791	777	829	861	869	859	881	927	940	945	960	953	925
818	788	787	767	791	800	785	794	848	884	876	863	892	944	965	973	988	939	882
779	745	746	754	783	810	821	831	843	862	897	894	910	949	955	956	957	904	863
798	753	747	749	766	803	861	859	854	870	904	920	943	936	951	940	924	873	850
839	784	797	766	765	791	833	815	840	884	902	905	934	962	988	971	919	864	848
839	803	810	838	798	802	804	790	826	888	892	873	902	946	967	947	902	853	835
849	817	831	859	847	837	813	797	816	850	862	847	858	895	901	891	853	842	803
836	860	850	841	853	853	847	829	827	817	827	833	822	850	842	837	843	849	840
826	837	860	836	839	865	840	809	802	814	835	837	821	821	822	835	838	864	845
823	835	859	854	865	869	848	813	785	813	841	843	827	819	828	820	830	851	857
829	832	869	897	899	882	879	860	837	847	852	850	828	844	831	829	814	833	873
867	846	888	916	908	896	909	882	873	876	867	859	863	861	819	841	821	852	874
844	835	872	906	915	921	916	913	906	881	881	878	880	883	844	869	850	854	856
816	844	878	894	908	944	931	911	917	925	908	871	883	883	876	863	866	896	886
835	861	914	922	899	927	932	952	958	941	923	891	907	894	869	861	870	899	880
850	846	881	926	917	901	917	960	962	938	956	919	926	897	897	866	892	920	889
897	868	865	892	919	923	916	914	925	944	953	948	932	946	953	915	928	963	954
911	879	898	889	894	921	911	889	925	942	948	970	972	986	1011	978	958	964	954
882	883	867	874	847	870	893	894	924	938	955	983	1018	1030	1048	993	979	951	934
881	878	866	856	840	846	874	892	916	938	939	991	1021	1020	1027	951	915	908	918
900	888	899	872	839	845	858	876	889	911	914	954	1017	1060	1024	951	900	878	891
908	916	898	880	845	851	856	845	866	907	901	909	962	995	987	954	933	901	914
928	923	890	892	852	857	864	848	840	882	917	924	952	949	940	949	966	935	906
921	917	885	872	854	861	852	864	854	887	919	933	936	928	921	943	951	931	907
936	926	901	900	873	872	867	860	880	907	932	940	953	943	954	969	938	888	882
945	977	956	957	920	878	854	868	868	888	924	943	949	962	958	960	934	865	883
931	930	957	958	920	901	899	933	905	919	905	917	939	962	920	923	926	922	921
964	971	941	930	922	934	967	988	956	938	910	904	944	980	926	896	893	914	956
994	1002	971	974	950	955	960	968	971	935	904	910	912	946	910	888	876	904	939
987	997	1002	1008	994	964	987	960	936	934	919	892	898	908	892	862	855	933	956
1006	1000	1015	1020	1042	1004	997	968	930	934	924	896	882	886	873	874	866	927	965
1015	1031	1030	1060	1074	1056	1033	987	962	902	899	861	842	834	866	910	875	893	924
1014	1065	1057	1058	1045	1048	1053	1019	993	920	865	843	821	835	858	898	888	907	929
977	1013	1098	1060	1034	1007	1018	1013	975	910	878	848	807	847	869	881	887	920	958
969	991	1047	1017	1008	980	999	959	940	884	845	841	817	849	856	864	883	926	957
940	959	987	998	990	996	1000	950	904	870	822	831	823	827	853	860	872	931	945
933	924	942	974	1001	995	1023	959	903	882	844	817	824	855	873	884	938	934	948
928	925	946	996	1005	994	991	931	913	880	843	810	813	848	887	942	965	957	959
933	937	935	973	968	968	952	892	870	837	810	785	803	823	867	935	945	951	970
896	909	911	932	927	909	924	873	841	818	777	791	806	823	873	911	935	935	984
885	900	876	888	870	886	875	892	856	841	830	827	811	852	869	911	938	935	997
902	901	875	872	875	863	885	887	869	873	884	881	861	860	902	924	947	990	981
881	896	882	883	863	847	879	863	900	901	885	884	874	864	907	905	897	997	1010
865	889	883	890	898	889	886	887	908	886	887	855	893	923	915	893	974	968	
853	855	848	874	929	962	947	930	894	900	903	886	904	939	954	926	903	949	975
854	840	875	906	934	978	955	932	919	935	927	903	916	965	948	950	931	930	1001
877	877	897	909	947	960	956	936	936	953	963	942	961	970	940	957	942	927	954
888	920	912	947	959	959	975	978	983	972	970	965	954	961	947	930	906	898	928
902	914	945	979	975	961	936	953	948	973	955	926	948	962	942	901	926	904	923
944	938	950	971	985	951	945	955	937	960	940	910	942	951	953	915	913	888	907
960	943	926	962	952	927	915	921	938	954	939	935	918	917	911	903	897	880	906
970	933	945	946	964	903	866	892	930	960	935	934	924	901	870	910	888	891	894
967	929	933	968	965	928	905	920	922	922	903	897	925	920	883	923	884	850	879
972	946	922	971	976	954	948	929	950	926	892	907	921	902	912	905	875	851	870
1005	986	925	941	969	941	940	940	964	924	903	911	891	873	878	905	854	836	836
1024	996	959	944	955	946	951	977	990	943	921	887	867	862	893	878	864	861	861

Nanopartículas de prata - Karla Balzuweit, Letícia Coelho, José Francisco de Sampaio, Luiz O. Ladeira, Sérgio de Oliveira
Microscopia Eletrônica de Tranmissão - JEOL 3010 - Laboratório de Microscopia Eletrônica (LME) do Laboratório de Luz Síncrotron (LNLS)

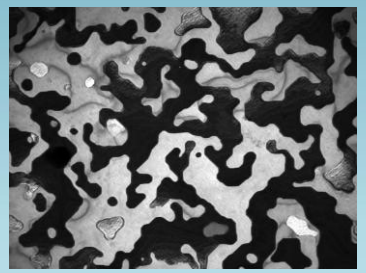
ELECTRON MICROSCOPY

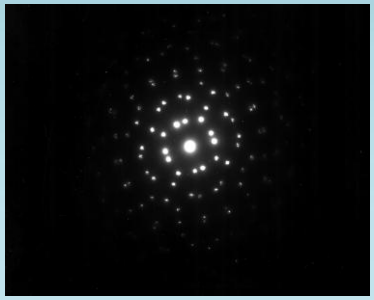


Electron Microscopy



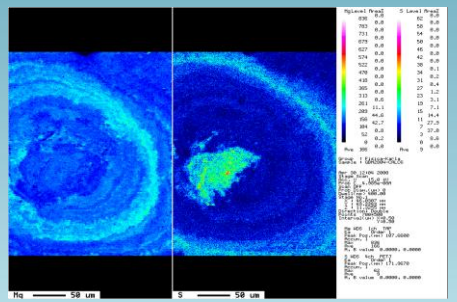
Electron images

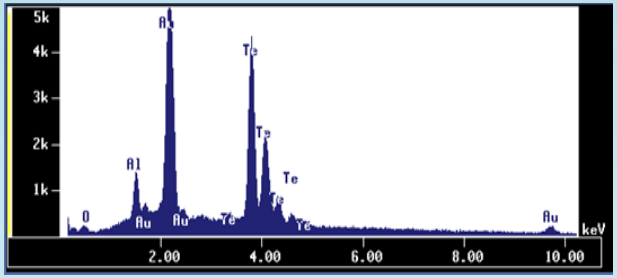




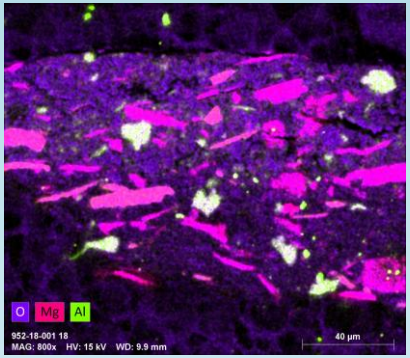
Electron diffraction

X-ray photon “images”





X-ray photon spectra



X-ray photon 3D hypercube

Imagens e espectros obtidos por Karla Balzuweit em diferentes microscópios eletrônicos do NCEM-LBNL, Laboratório de Microanálises – LMA e Centro de Microscopia da UFMG

Electron Microscopes

Microscópio Eletrônico de Varredura de bancada LCPNano-UFMG - www.lcpnano.ufmg.br/

Microscópio Eletrônico de Varredura de alta resolução
CM-UFMG – www.microscopia.ufmg.br

Microscópio Eletrônico de Transmissão e Varredura
CM-UFMG
www.microscopia.ufmg.br

Kratos - 1.500kV- NCEM – LBNL
http://ncem.lbl.gov/ncem.html

Atual:
https://foundry.lbl.gov/about/facilities/the-national-center-for-electron-microscopy-ncem/

Microscópio Eletrônico de Transmissão e Varredura com corretor de aberração esférica

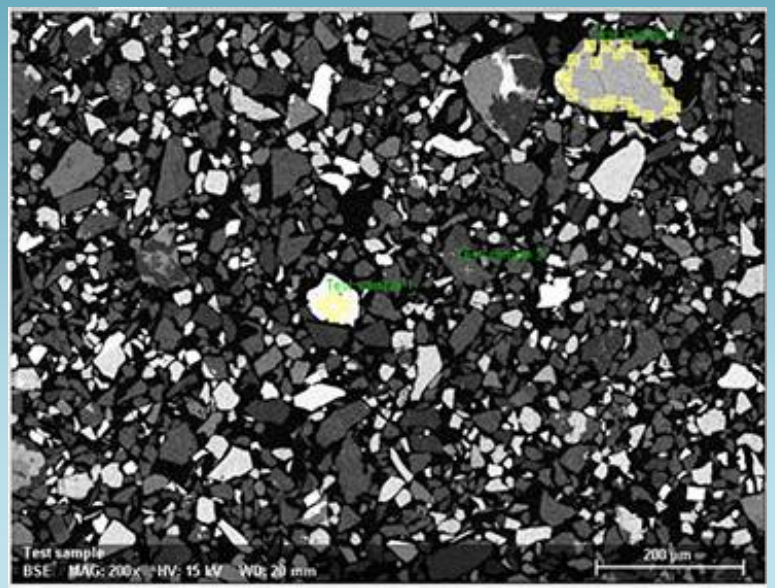
Ohio state University
https://cemas.osu.edu/fei-probe-corrected-titan3tm-80-300-stem

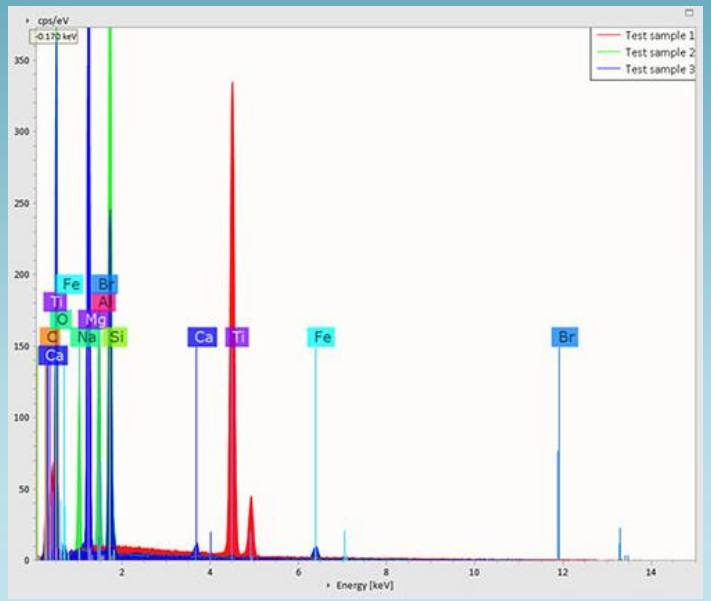
Analytical Electron Microscopy (AEM)

- EDS (Energy Dispersive Spectrometer)
  - •Qualitative, semi-quantitative and "quantitative" analysis of chemical elements
  - • energy resolution ~140eV (Mn)
  - •X-ray photons
- WDS (Wavelength Dispersive Spectrometer)
  - •Qualitative, semi-quantitative and "quantitative" analysis of chemical elements
  - •Energy resolution ~10eV
  - •X-ray photons
- EELS (Electron Energy Loss Spectrometer)
  - •Qualitative, semi-quantitative and "quantitative" analysis of chemical elements, band structure, valence, plasmons.
  - •Resolution depends of the filament:
  - o ΔE ~1-2eV (thermoionic),
  - o ΔE ~0,2eV (FEG)
  - oΔE~0,1eV (FEG with monochromator)
  - •Energy loss electrons

20

Point and multipoint spectra

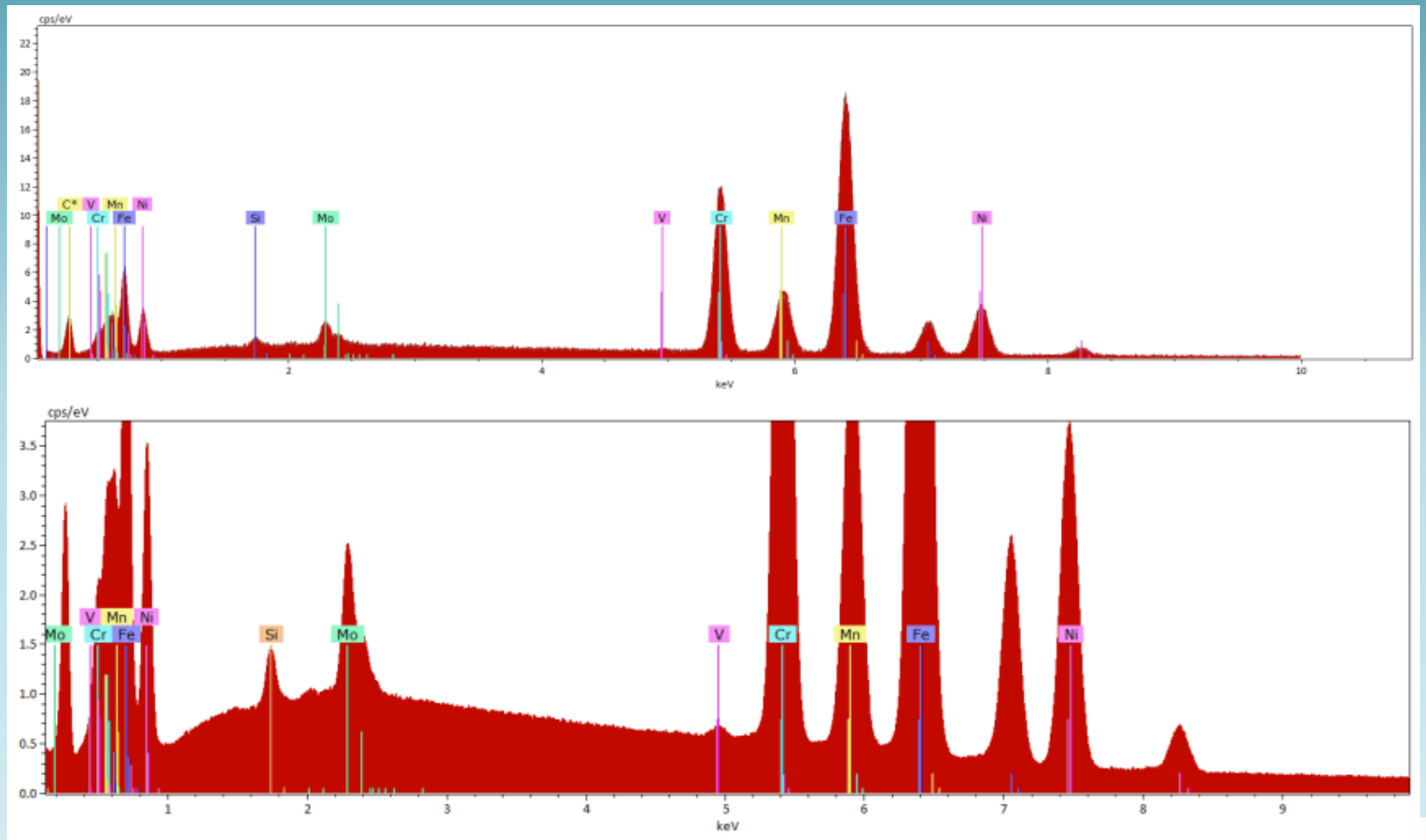




Cortesia – Dr. Tobias Salge and Dr. Ralf Terborg – Bruker Nano GmbH. And Orkun Tunçkan- Univ. Anadolu -TK

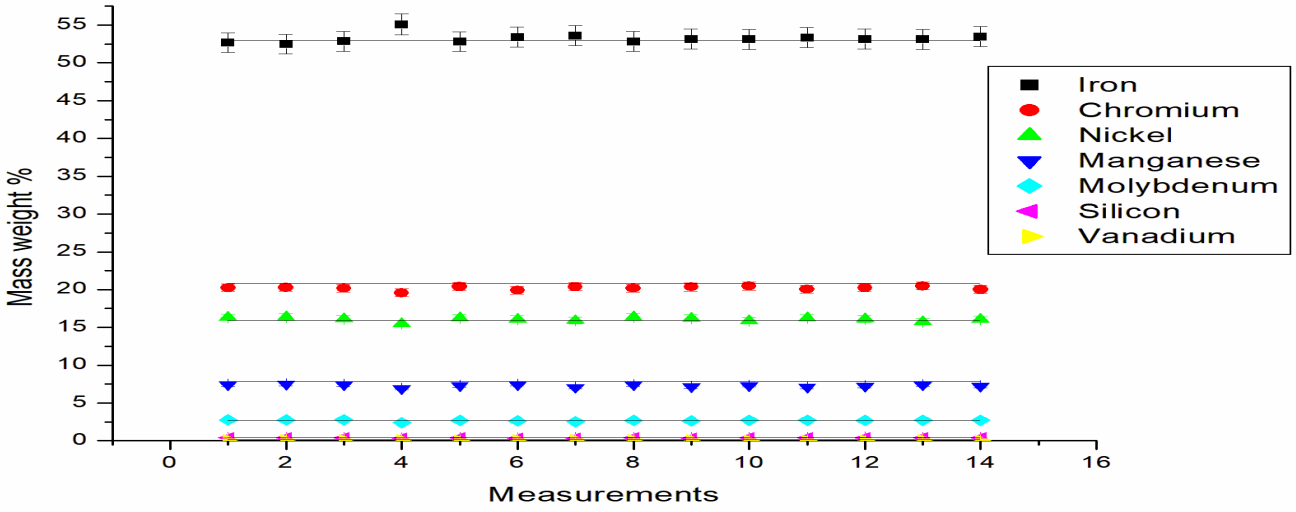
All	cps/eV	Results [Mass-%(norm.)]	Sort: Element						
EDS	Test sample 1	0.00	C 3.51	O 38.14	Ti 58.34				
EDS	Test sample 2	0.08	C 7.89	O 43.86	Na 6.94	Mg 0.01	Al 4.69	Si 28.72	Br 7.90
EDS	Test sample 3	0.08	C 8.82	O 42.80	Mg 23.88	Si 16.85	Ca 1.68	Ti 0.35	Fe 5.62

Interlaboratorial test



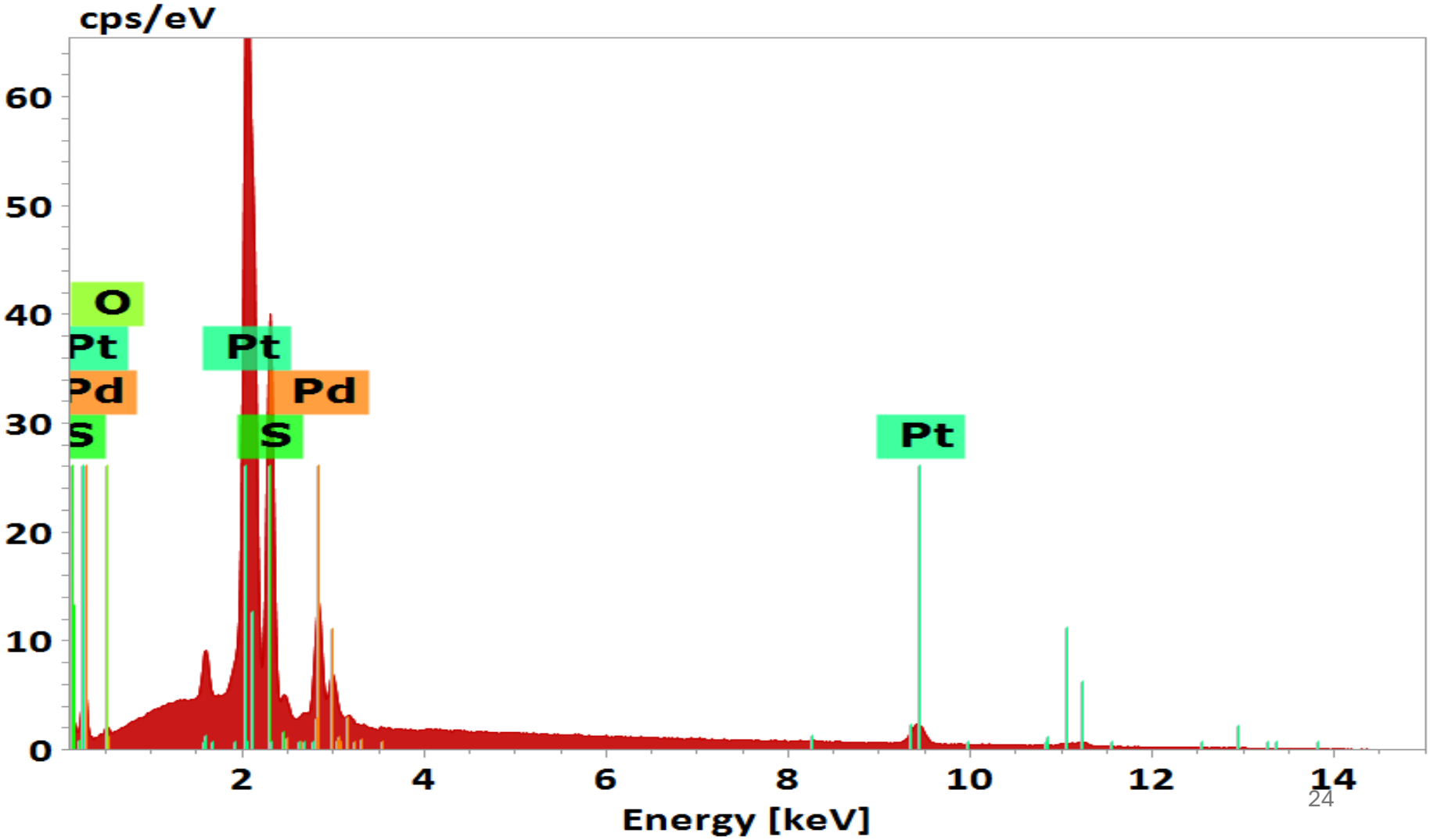
Interlaboratorial test

Element	Iron	Chromium	Nickel	Manganese	Molybdenum	Silicon	Vanadium
SEM	52 (1)	20,2 (0,5)	16,3 (0,4)	7,5 (0,2)	2,8 (0,1)	0,39 (0,04)	0,17 (0,03)
Microprobe	52,96 (0,01)	20,85 (0,64)	15,90 (0,10)	7,87 (0,10)	2,66 (0,30)	0,38 (0,01)	0,17 (0,02)

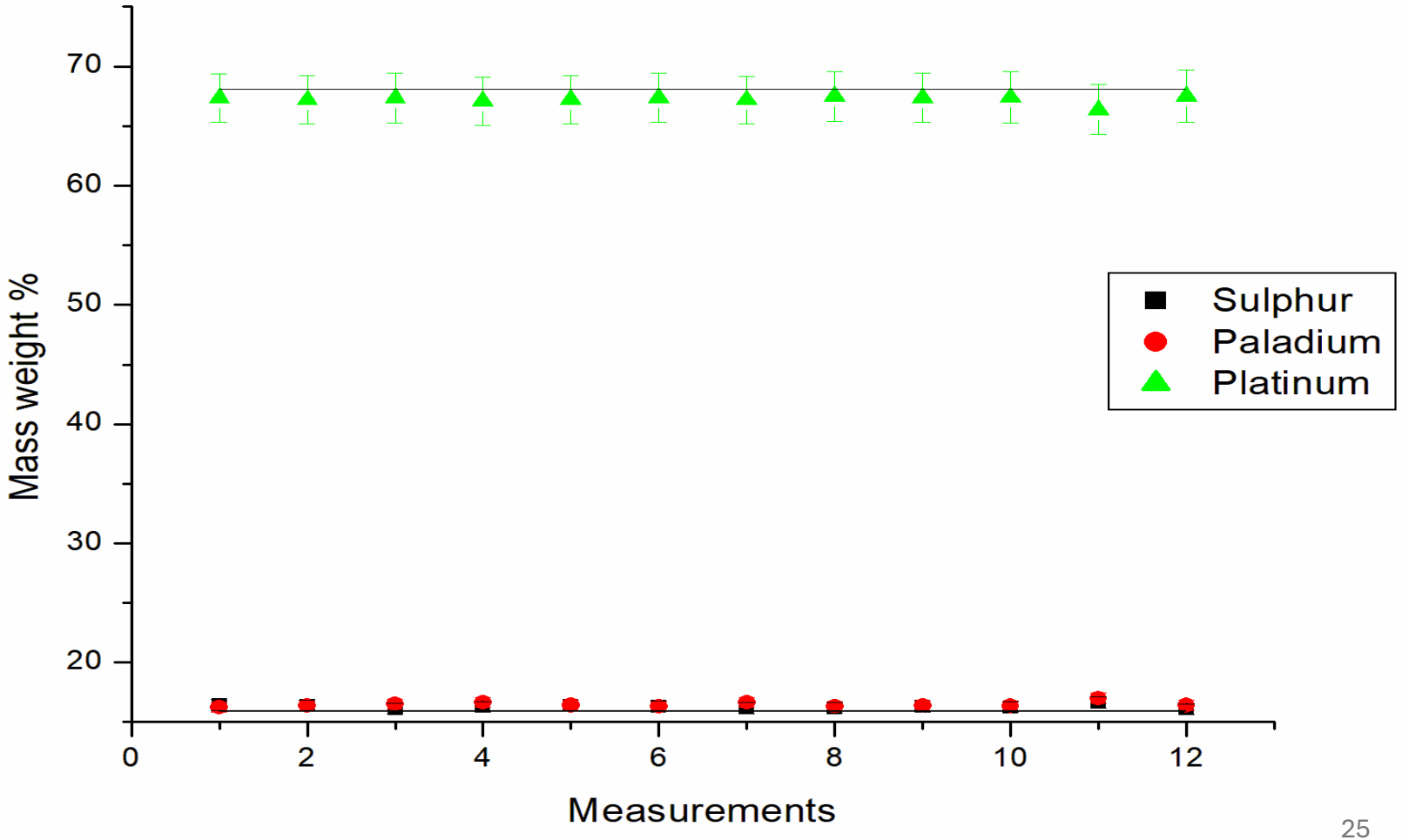


Luis Rodrigues Garcia, Márcio de Almeida Flores, Karla Balzuweit – Centro de Microscopia da UFMG

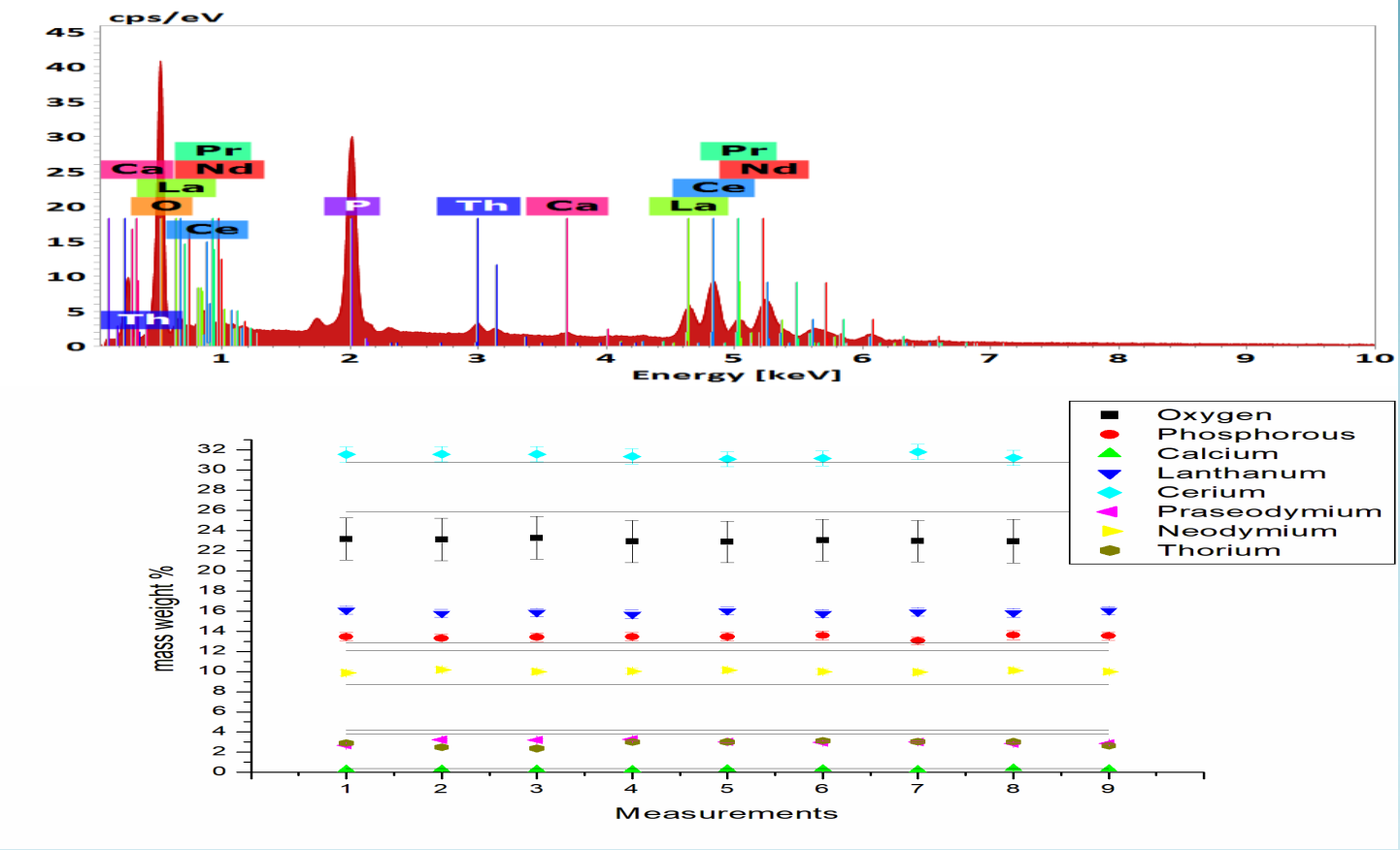
Pt-Pd-S - standard



Pt-Pd-S - standard



Rare earth standard



Monte Carlo Simulation

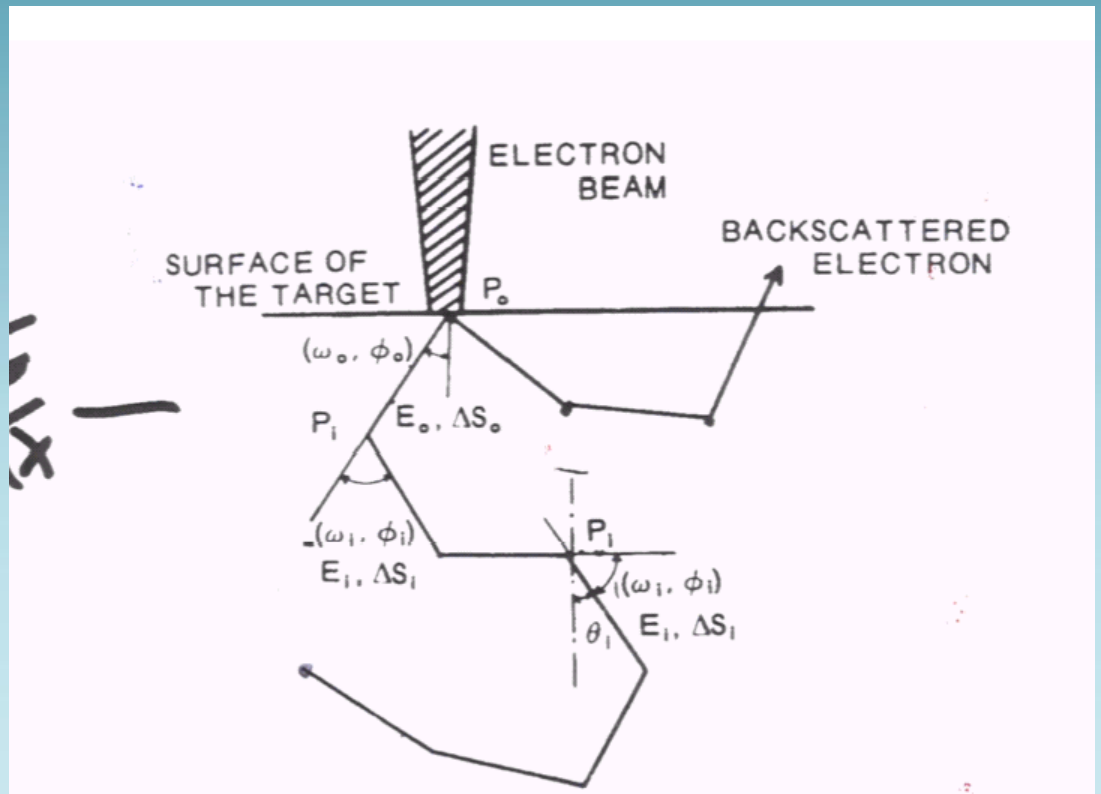


FIGURE 2. Simplified model of electron trajectories (Shinoda *et al*

27

Lectures of Phil Russell - www.phys.appstate.edu/nanotech/PHY4880/2002/lectures/

Different elements

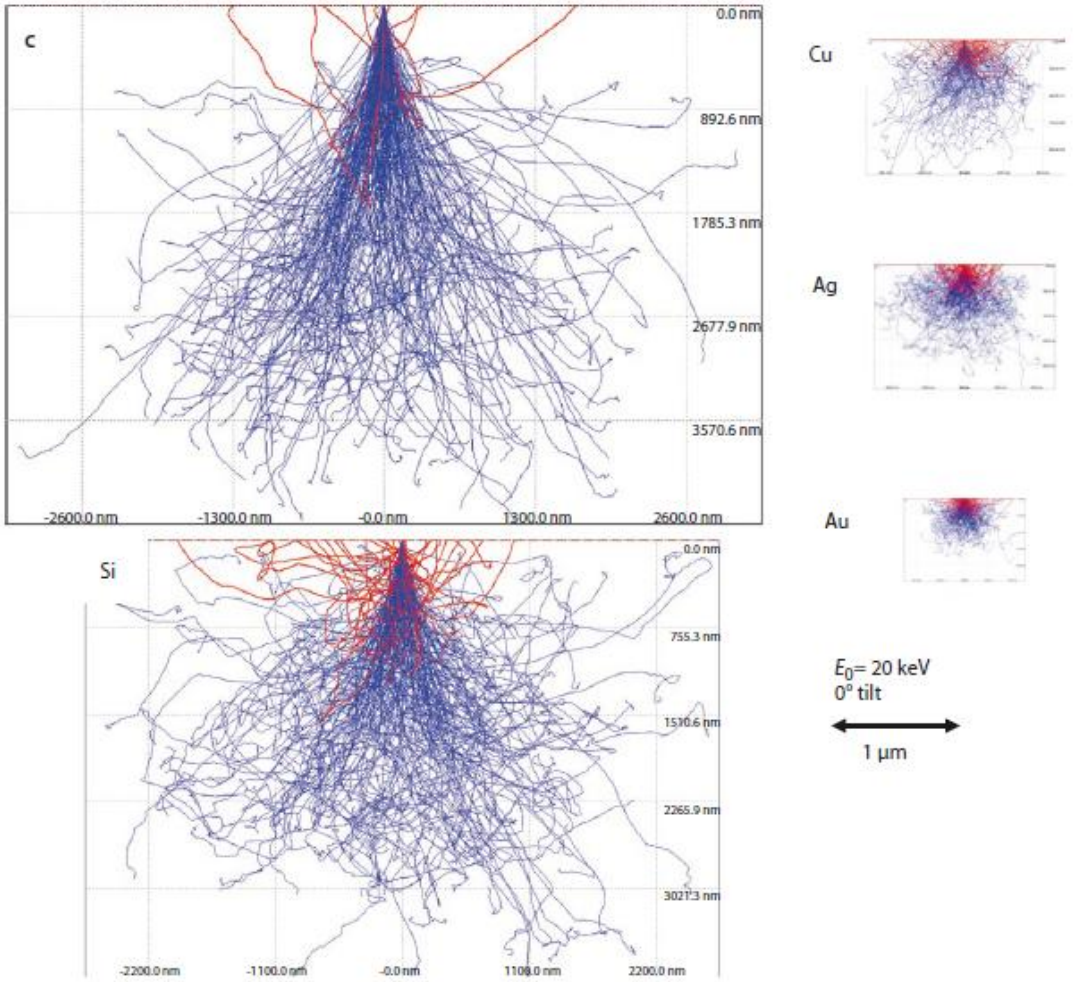


Fig. 1.8 Monte Carlo simulations for an incident beam energy of 20 keV and 0° tilt for C, Si, Cu, Ag, and Au, all shown at the same scale (CASINO Monte Carlo simulation)

- C, Si, Cu, Ag, Au
- 20kV

- Red - electrons which "escape" the sample.
- Blue – electrons absorbed

Goldstein , 4th ed.

Varying high tension of the electron beam

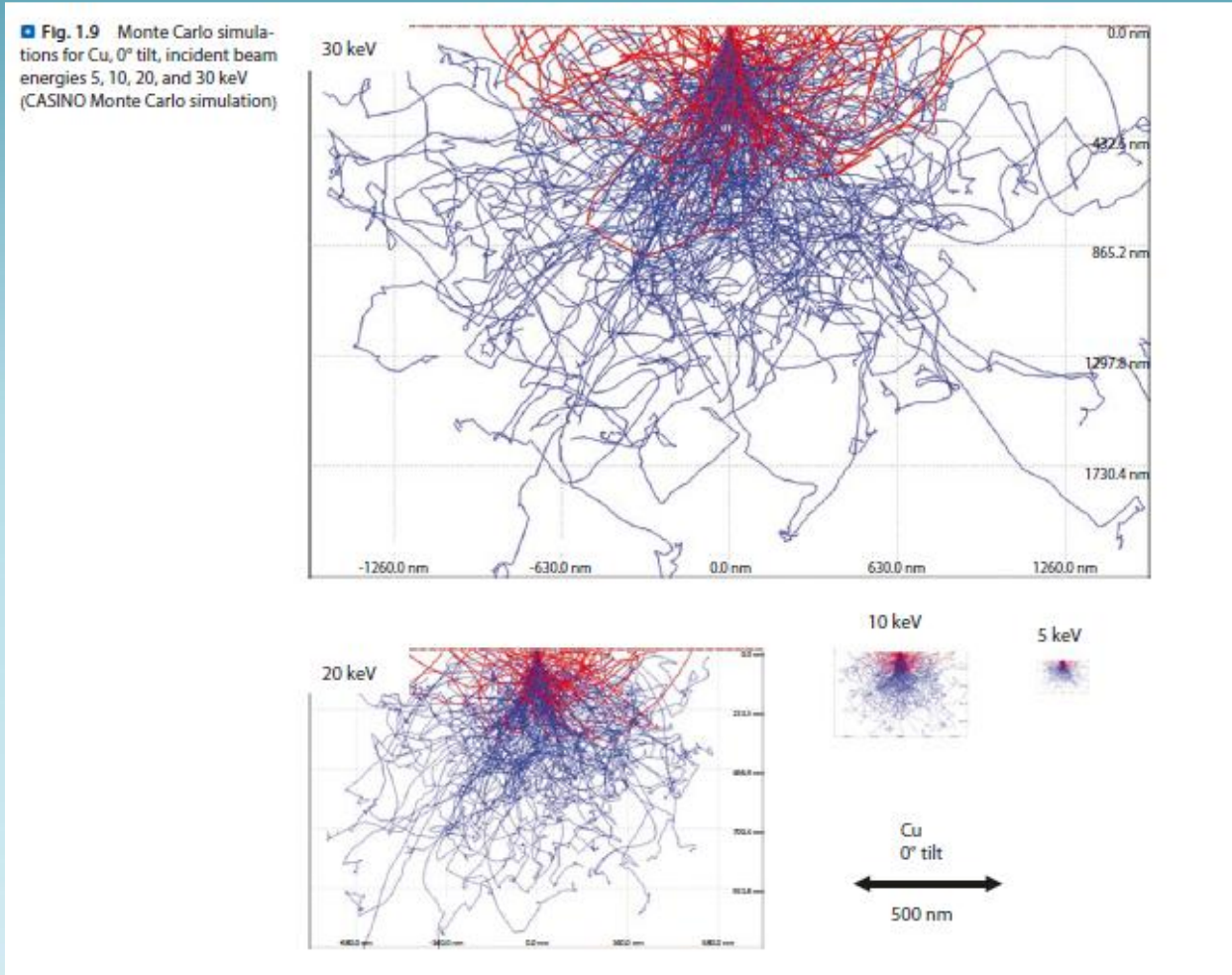
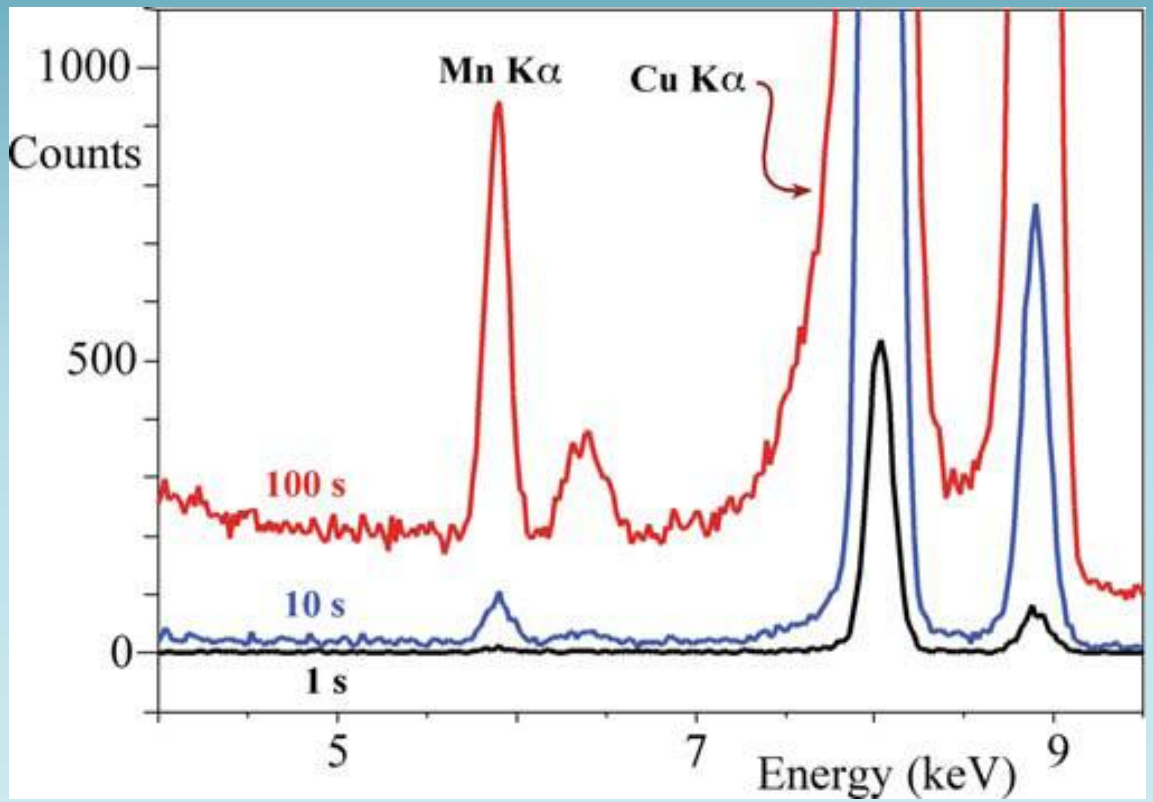


Fig. 1.9 Monte Carlo simulations for Cu, 0° tilt, incident beam energies 5, 10, 20, and 30 keV (CASINO Monte Carlo simulation)

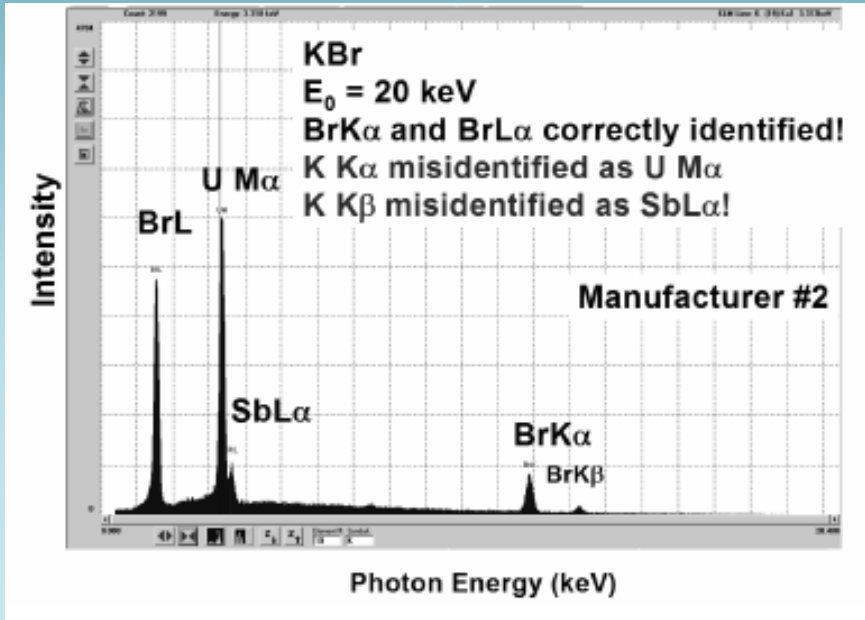
Goldstein , 4th ed.

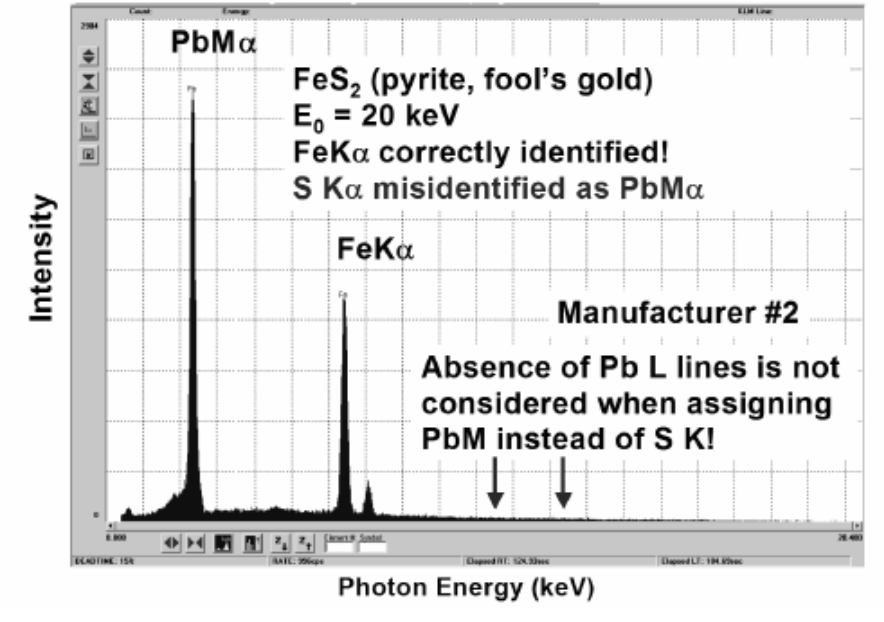
Increasing acquisition time



Charles Lyman – Lehigh University

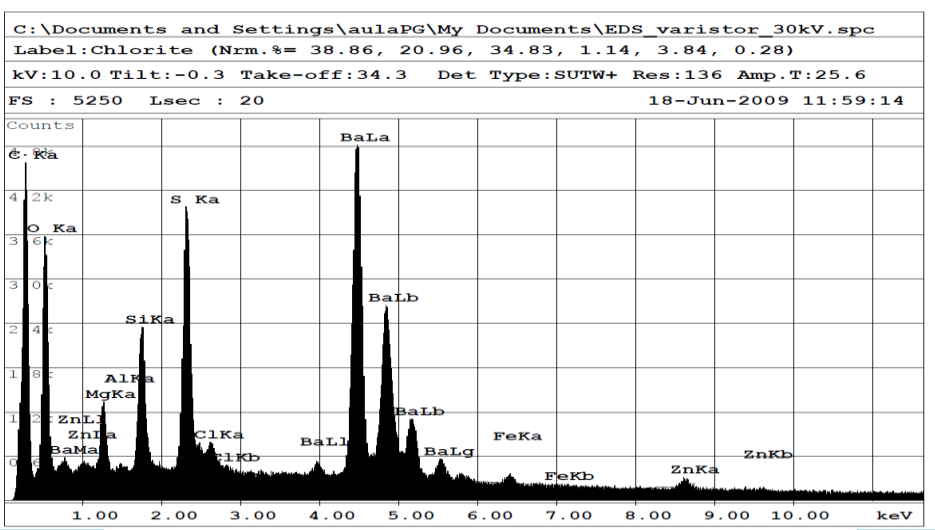
Errors in the automatic identification of elements

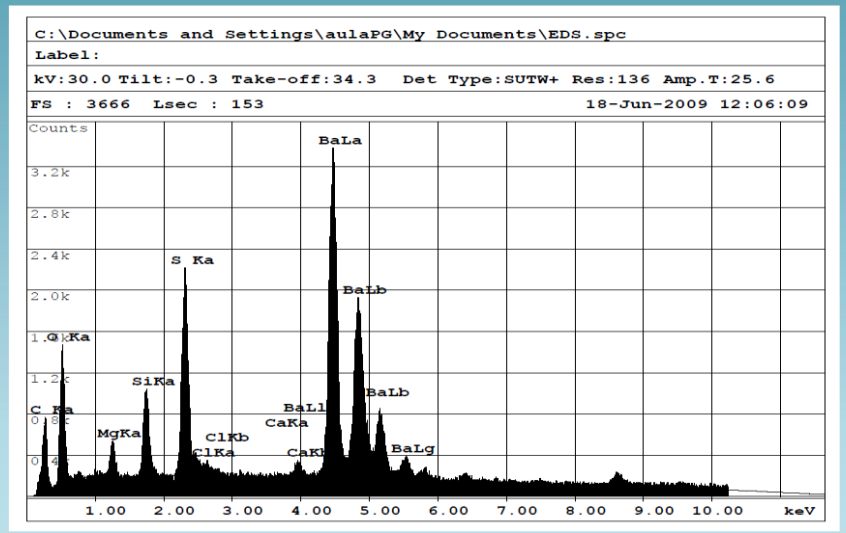




Charles Lyman – Lehigh University

Same point measured – detector had not been adjusted





EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : User c:\edax32\eds\genuser.sec

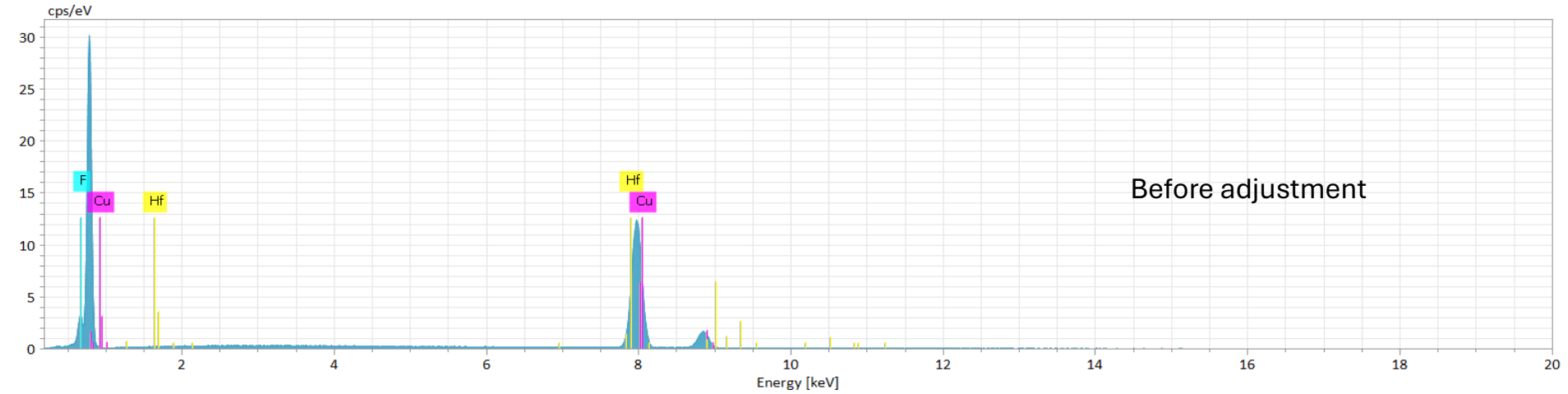
Element	Wt %	At %	K-Ratio	Z	A	F
C K	12.51	47.35	0.0552	1.3248	0.3330	1.0000
O K	4.68	13.29	0.0313	1.2922	0.5171	1.0001
ZnL	0.24	0.17	0.0013	1.0359	0.5261	1.0003
MgK	0.98	1.83	0.0068	1.2708	0.5463	1.0006
AlK	0.09	0.15	0.0007	1.2138	0.6241	1.0012
SiK	2.28	3.69	0.0203	1.2332	0.7190	1.0017
S K	5.19	7.36	0.0537	1.2136	0.8490	1.0037
ClK	0.72	0.92	0.0074	1.1620	0.8821	1.0062
BaL	71.27	23.58	0.6075	0.8454	1.0077	1.0005
FeK	2.04	1.66	0.0219	1.1088	0.9682	1.0000
Total	100.00	100.00				

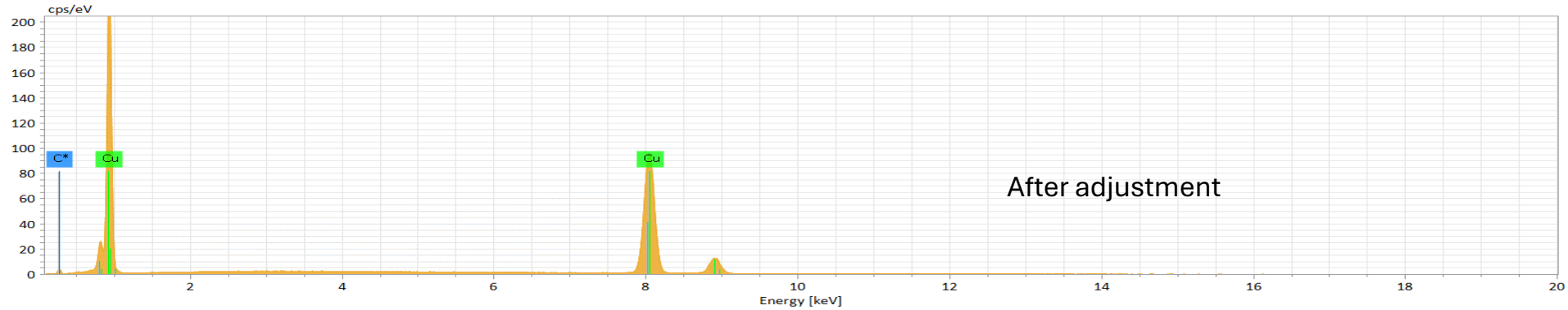
EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : User c:\edax32\eds\genuser.sec

Element	Wt %	At %	K-Ratio	Z	A	F
C K	28.57	51.81	0.0669	1.0877	0.2152	1.0002
O K	24.33	33.13	0.0540	1.0722	0.2071	1.0001
MgK	2.25	2.02	0.0055	1.0333	0.2361	1.0018
SiK	3.58	2.78	0.0143	1.0341	0.3836	1.0045
S K	6.65	4.52	0.0397	1.0275	0.5764	1.0072
ClK	0.48	0.29	0.0029	0.9842	0.6181	1.0111
CaK	0.09	0.05	0.0008	1.0169	0.8514	1.0460
BaL	34.05	5.40	0.3074	0.7986	1.1304	1.0000
Total	100.00	100.00				

Karla Balzuweit – Centro de Microscopia da UFMG

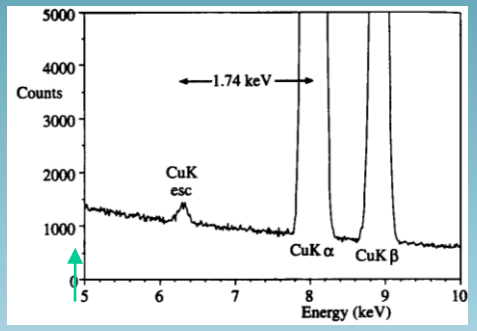
Energy scale bar adjustment (“calibration”)



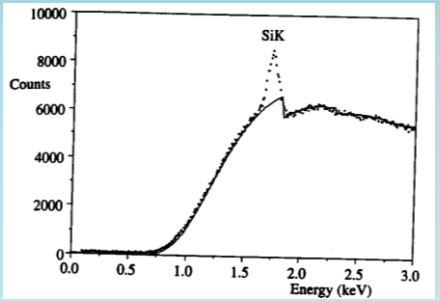


EDS artefacts

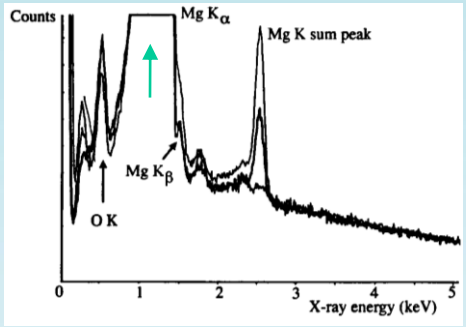
- Pico escape do silício
  - **Si Ka 1.74 keV**
  - **Subtrair 1,74eV de outros picos**
  - **Independente da taxa de contagem**



- Fluorescência do silício
  - **Fóton gerado dentro do detector na "dead layer"**
  - **Detectado pela região ativa do detector**

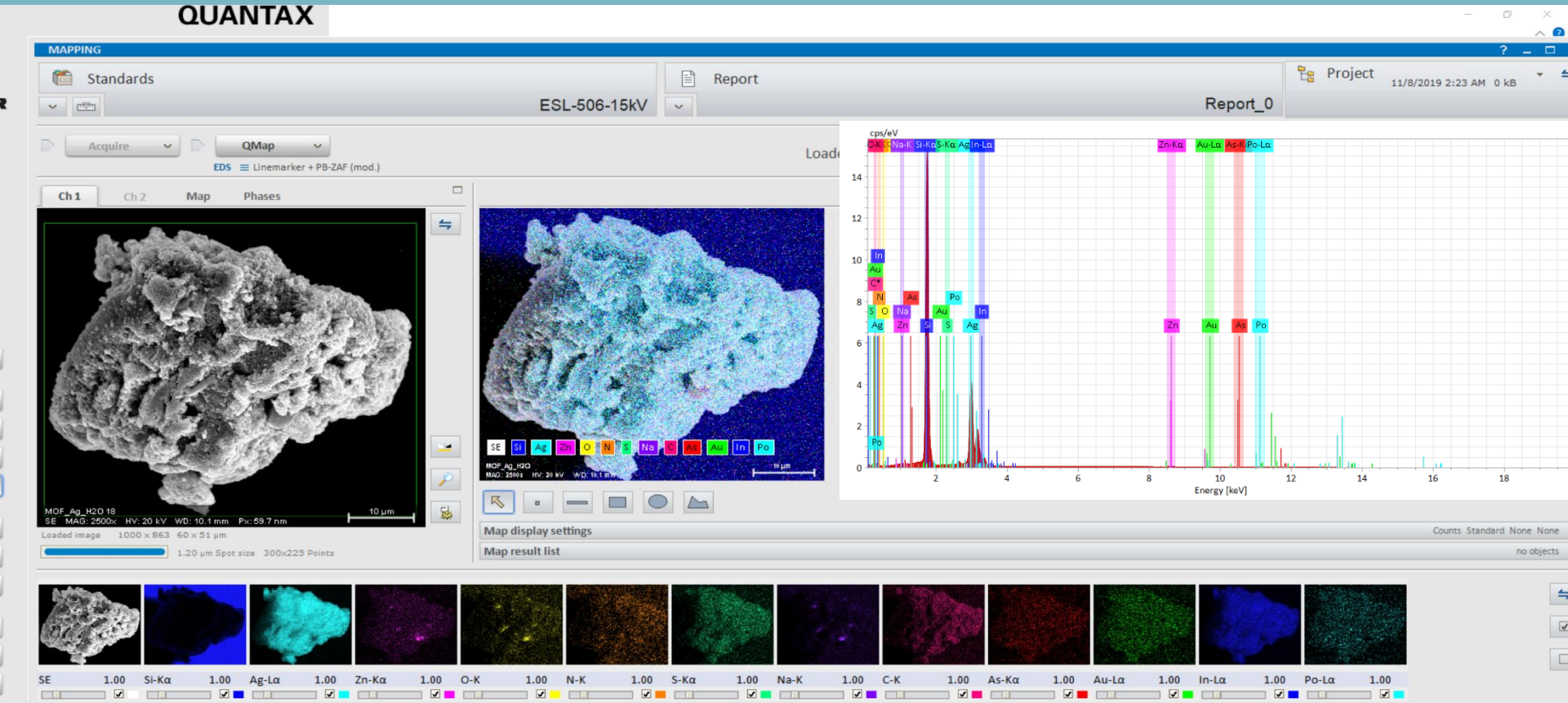


- Pico soma
  - **Dois fótons chegam simultaneamente ao detector**
  - **Contabiliza-se um pico com o dobro da energia (o elemento não existe na amostra)**
  - **Contagens altas**



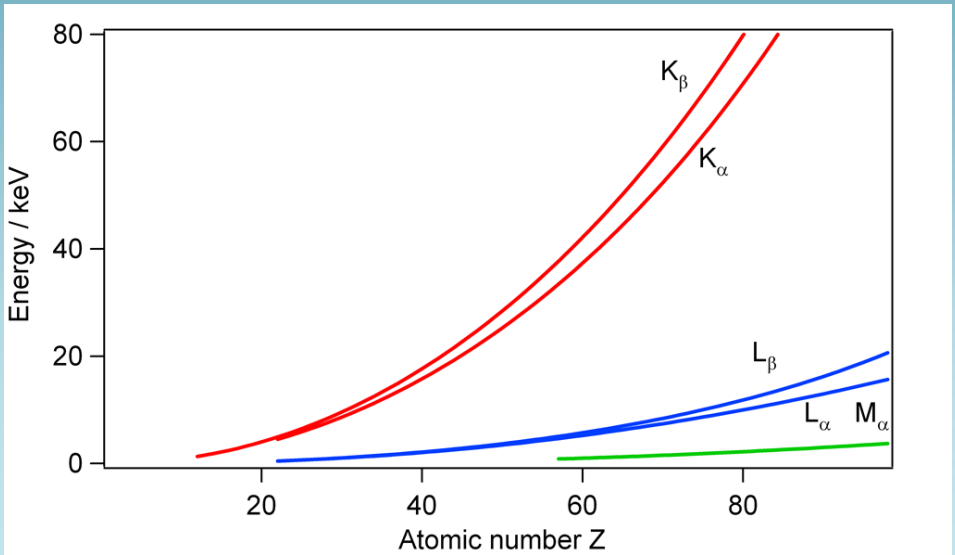
Charles Lyman – Lehigh University

“Measuring” the background



Karla Balzuweit – Centro de Microscopia da UFMG

Line Energy and Z: Moseley's law



Transmission Electron Microscopy Williams and Carter 2009

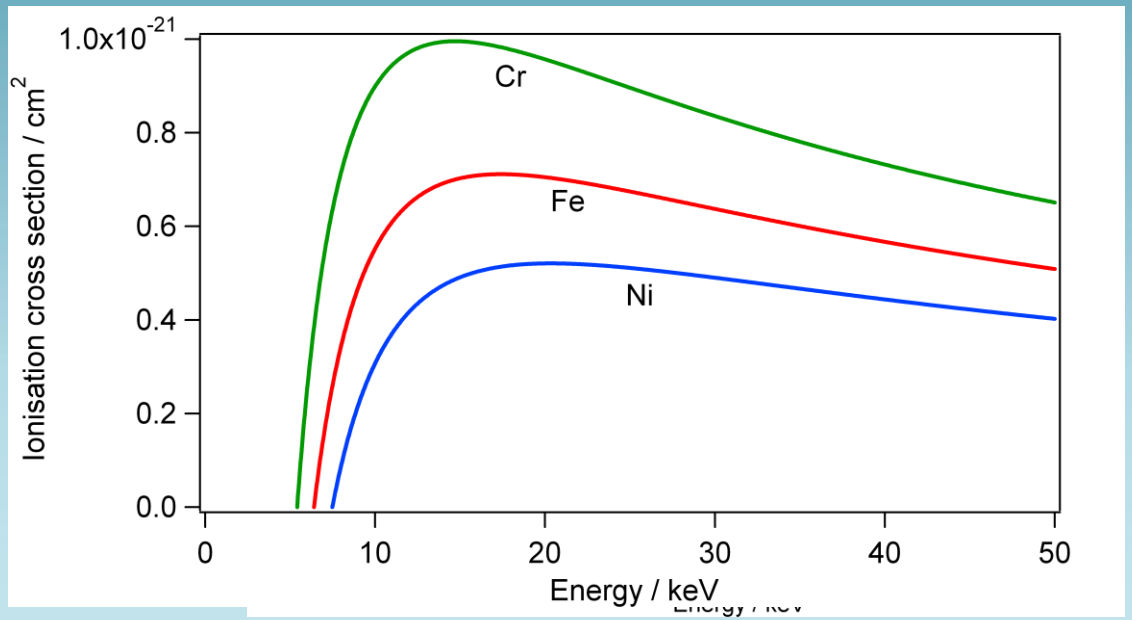
X-rays are **characteristic** because their specific energies are characteristic for the particular element which is excited.

$$E = c_1 (Z - c_2)^2$$

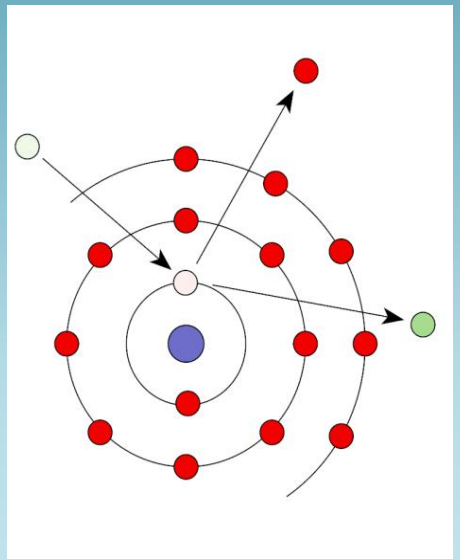
c_1, c_2 depend on the type of transition

Moseley's law is an **empirical** description of the relationship between X-ray line energy and the atomic number of the emitting atom.

Probability of Ionization: Ionisation cross-section for electrons

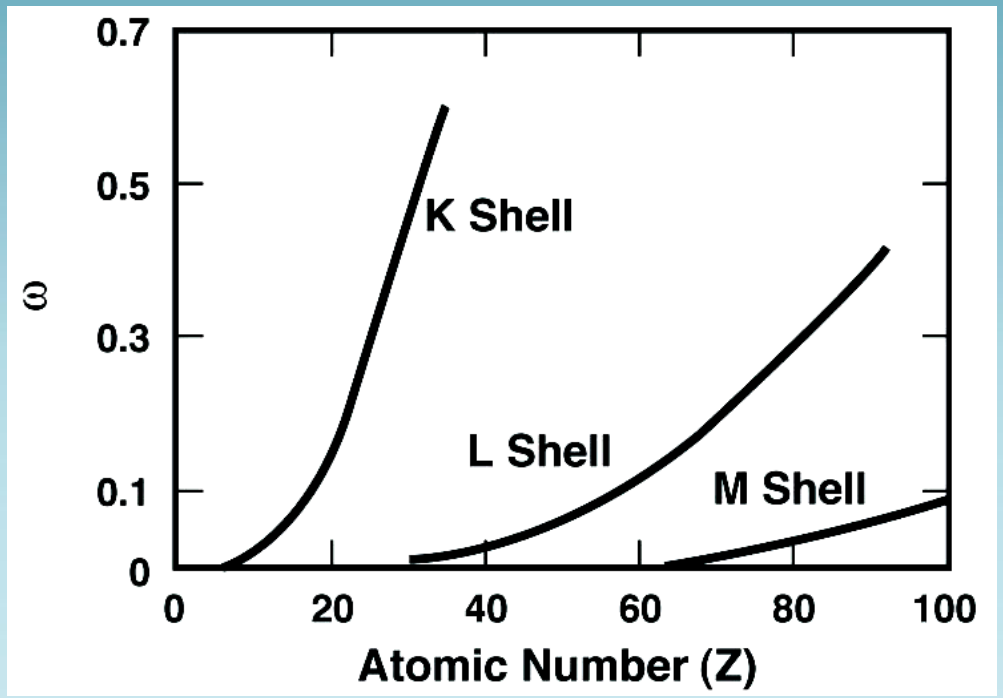


Ionisation cross section for electrons



- Ionisation cross section ~ probability of excitation
- maximum ionisation cross section at ~ 2,5 x E_{bind}

Fluorescence yield (ω)



Charles Lyman Lectures – PASI 2006

ω increases with com Z:

ω_K – typical values:
0.03 carbon (12) K-series @ 0.3 keV
0.54 germanium (32) K-series @ 9.9 keV
0.96 gold (79) K-series @ 67 keV

Production of X-rays is quite inefficient for low Z (O, N, C, etc.).

Production of X-rays is quite inefficient for L and M transitions:

ω_L e ω_M are always < 0.5:
ω_L = 0.36 for gold - Au (79)
ω_M = 0.002 for gold - Au (79)

Data Correction strategies

- ZAF: 1st generalized algebraic procedure; assumes a linear relation between concentration and x-ray intensity;
- Phi-rho-Z: based upon depth profile (tracer) experiments;
- Monte Carlo: based upon statistical probabilities of electron-sample interactions, particularly for unusual specimen geometries.

Z A F

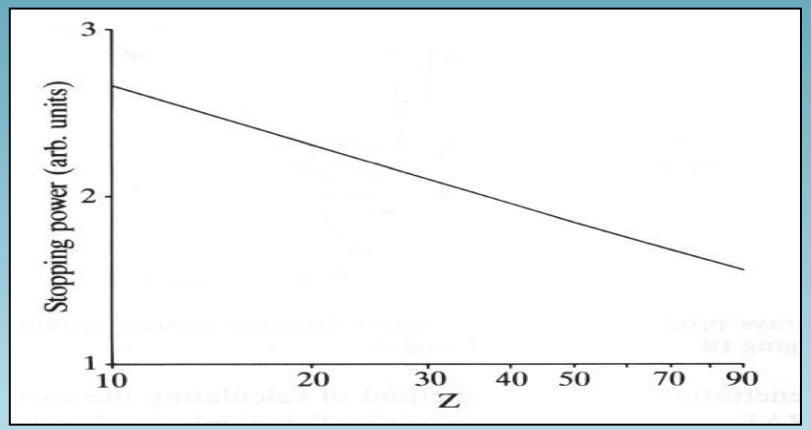
$$C_i^{unk} = \frac{I_i^{unk}}{I_i^{std}} \frac{ZAF_i^{unk}}{ZAF_i^{std}} C_i^{std}$$

Z – Atomic number
Electron backscattering
Stopping power
A – Absorption
F – Fluorescence

Heinrich, 1991, Strategies of electron probe data reduction, in Electron Probe Quantitation, Ed. Heinrich and Newbury, Plenum, New York, 9-18.

Z correction

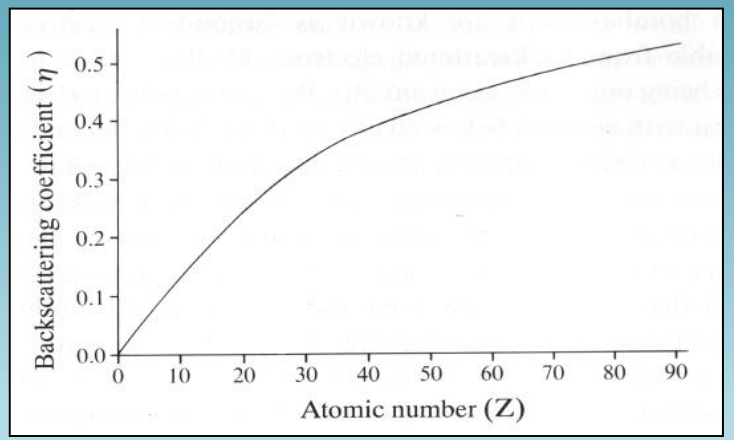
Stopping Power Correction + Backscatter Correction



$$S = \frac{Z}{A} \ln\left[\frac{1.166 \cdot E_{mean}}{J}\right]$$

where J=11.5+ Z and $E_{mean}= (E_0+E_c)/2$

(J is the mean ionization energy; J, Z and A are of the target, E mean is of the X-ray)



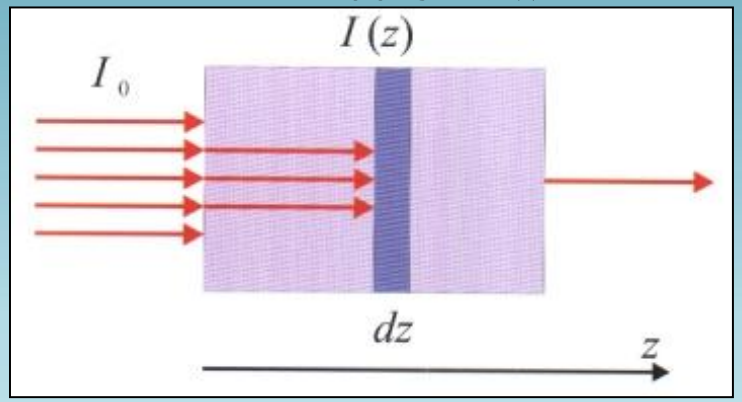
$$R = \frac{1}{1 + [0.008 \cdot (1 - W) \cdot Z]}$$

where $W = E_c/E_0$ (the inverse of overvoltage), and Z is of the target, and W is of the X-ray

$$k_Z = R_{std}/R_{unk} * S_{unk}/S_{std}$$

Absorption / Fluorescence correction

Beers Law



$$I = I_0 \exp^{-(\mu/\rho)(\rho Z)}$$

$$K_A = f(\chi_s) / f(\chi_a)$$

$\chi = \mu/\rho \text{ cosec } \psi$

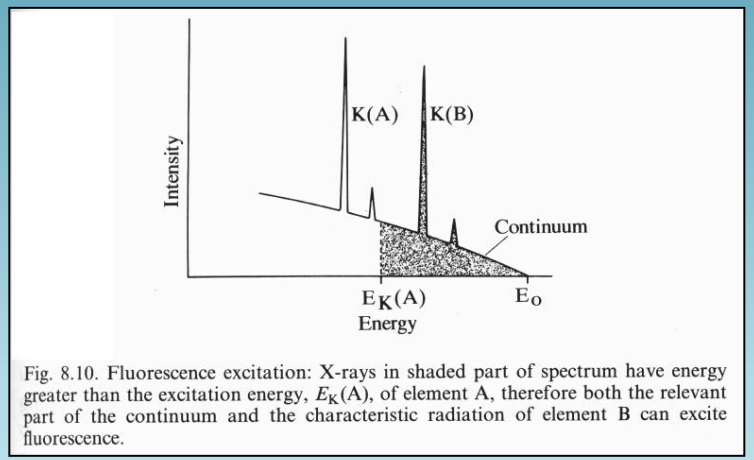


Fig. 8.10. Fluorescence excitation: X-rays in shaded part of spectrum have energy greater than the excitation energy, $E_K(A)$, of element A, therefore both the relevant part of the continuum and the characteristic radiation of element B can excite fluorescence.

$$F = \frac{1}{1 + \square \frac{I_f}{I_p}}$$

"I_f/I_p is the ratio of emitted X-rays from fluorescence, compared to the X-ray intensity from inner shell ionization. In a compound, this term is summed overall all the elements that could fluorescence the element of interest."

TEM EDS correction factors

- Correção ZAF
- Fator de Cliff – Lorrimer = Aproximação de filme fino
  - exceto para amostras grossas eelementos com diferença em energia maior que 5 a 10KeV
  - A (absorção) pode ser desprezada e F (fluorescência) normalmente são desprezado
  - elementos leves (seus raios-x tendem a ser mais absorvidos)

$$\frac{C_A}{C_B} = k_{AB}\frac{I_A}{I_B}$$

$$\frac{C_A}{C_B} = k_{AB}\frac{I_A}{I_B} \qquad \frac{C_B}{C_C} = k_{BC}\frac{I_B}{I_C} \qquad k_{AB} = \frac{k_{AC}}{k_{BC}}$$

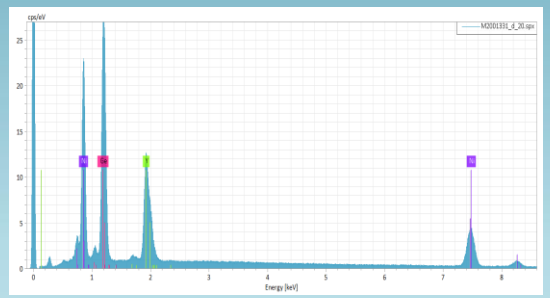
$$C_A + C_B = 100\%$$

$$C_A + C_B + C_C = 100\%$$

42

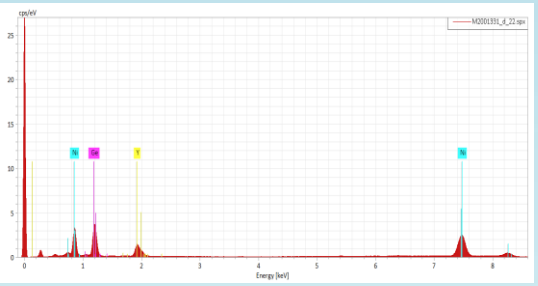
Data acquisition
Good practices / Protocols
Knowledge

EDS analisys



Element	Mass [%]	Mass Norm. [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Germanium	41.00713	41.27993	2.310389	5.634116
Nickel	32.94032	33.15946	1.039468	3.15561
Yttrium	25.3917	25.56062	1.008355	3.971198
	99.33916	100		

Flat sample



Element	Mass [%]	Mass Norm. [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Germanium	58.5193	45.55074	3.290781	5.623411
Nickel	36.6049	28.49282	1.143193	3.123059
Yttrium	33.34639	25.95644	1.321829	3.963934
	128.4706	100		

Inclined sample

ISO 22309/ASTM1508

- ✓ Energy scale bar correctly adjusted with reference material
- ✓ 1.000.000 countns
- ✓ Correct working distance (10mm)
- ✓ Enough high tension (ionization tension) – 15kV
- ✓ Matrix correction – ZAF

Karla Balzuweit – Centro de Microscopia da UFMG

Standards and norms related to microanalysis in electron microscopy (access is paid)

1) **ISO 22029:2012 (en) Microbeam analysis — EMSA/MAS standard file format for spectral-data exchange.**
   ISO 22493 - Microbeam analysis — Scanning electron microscopy — Vocabulary.
2) ISO 23833 - Microbeam analysis — Electron probe microanalysis (EPMA) — Vocabulary.
3) ISO 15932 - Microbeam analysis — Analytical electron microscopy — Vocabulary.
4) ISO 15632 – Microbeam analysis – Selected instrumental performance parameters for the specification and checking of energy dispersive X-ray spectrometers for use in electron probe analysis.
5) ISO 22309 - Microbeam analysis – Quantitative analysis using energy dispersive X-ray spectrometry (EDS) for elements with an atomic number of 11 (Na) or above.
6) ASTM E1508-12a – Standard Guide for Quantitative Analysis by Energy Dispersive Spectroscopy.
7) ISO 17470 - Microbeam analysis — Electron probe microanalysis — Guidelines for qualitative point analysis by wavelength dispersive X-ray spectrometry.
8) ISO 16700 - Microbeam analysis — Scanning electron microscopy — Guidelines for calibrating image magnification.
9) ISO/TS 21383 - Microbeam analysis — Scanning electron microscopy — Qualification of the scanning electron microscope for quantitative measurements.

Single EDS spectra metadata format:
ISO 22029:2012(en) Microbeam analysis — EMSA/MAS standard file format for spectral-data exchange

HMSA proposal for microscopy 4D datasets*:

HSMA file stores the experimental data
XML file stores the experimental conditions (metadata)
https://www.microscopy.org/resources/scientific_data/HMSA_Specification-20191120.pdf

*3D EDS-SEM mapping included

https://www.microscopy.org/resources/scientific_data/)
.

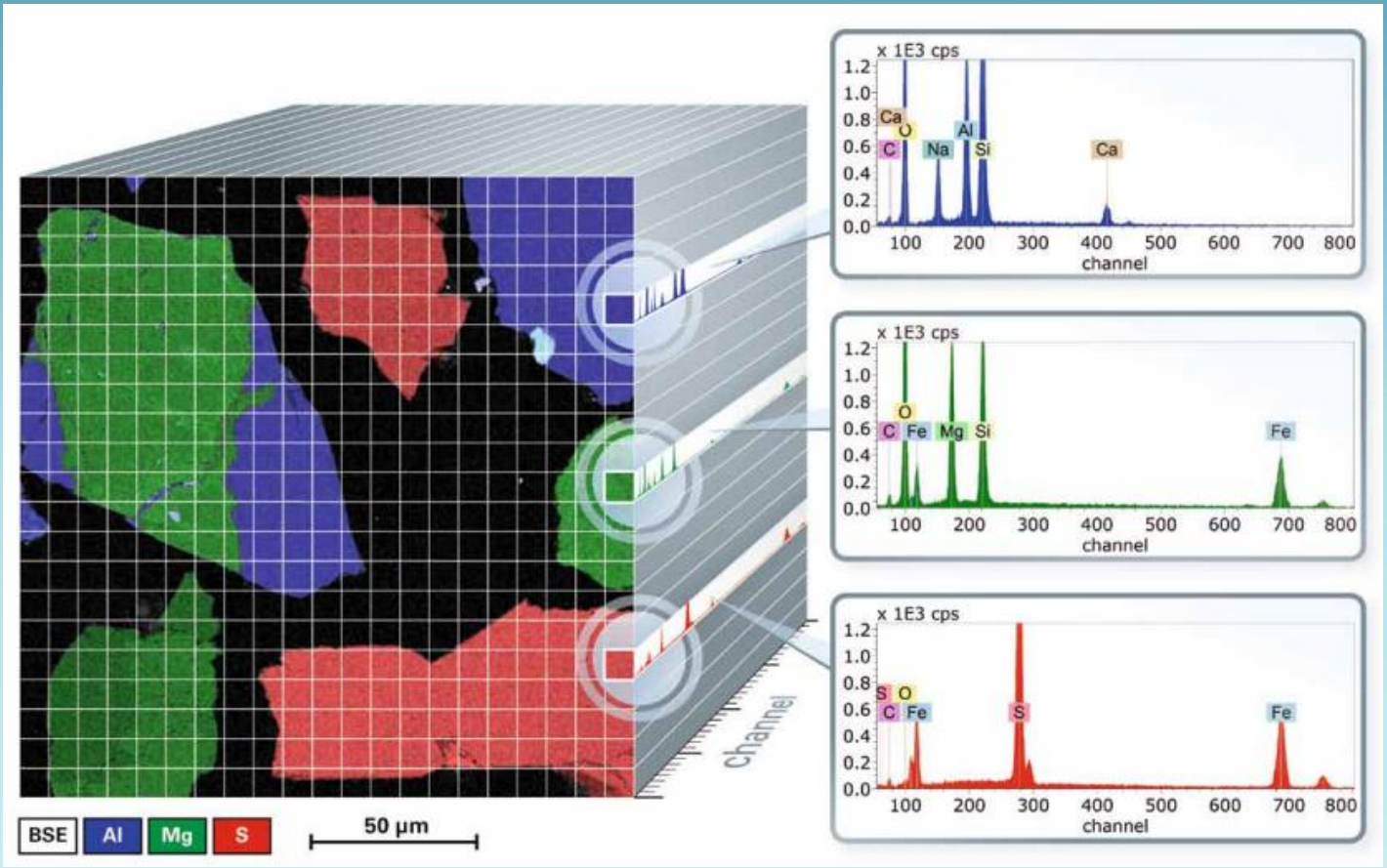
Quantitative / Semiquantitative EDS analysis
Scanning Electron Microscope
Electron Dispersive x-ray Spectrometer

- History of the sample
- Sample preparation method/process

- **Quantification process**
- Matrix correction
- Different models
- Background curve adjusted

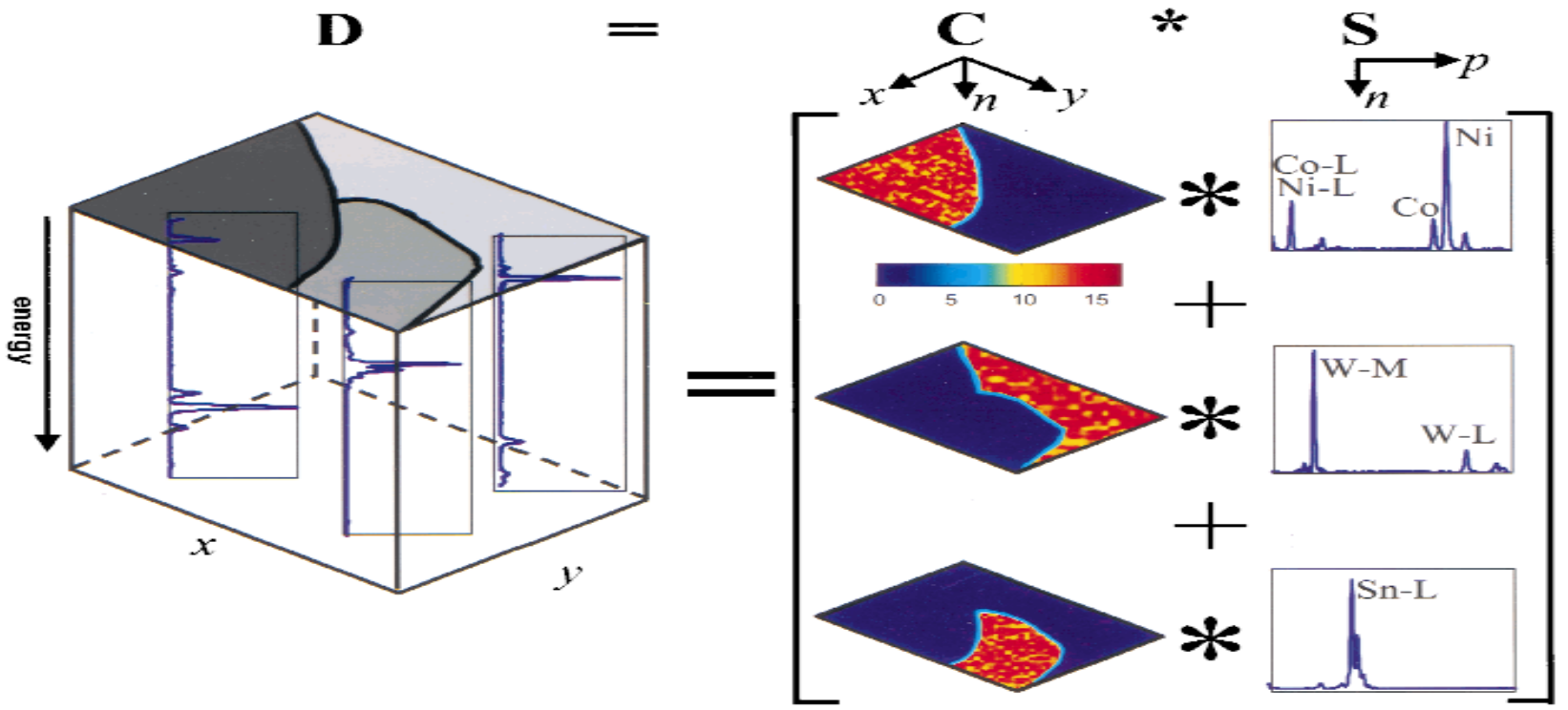
- **Data acquisition according to laboratory good practices**
- ISO 22309/ASTM1508 ($$)
- Equipment manual
- Microscope adequately aligned
- Correct choice of acquisition parameters
- Energy scale bar adjusted / “calibrated”

EDS hypermap



Cortesia – John Friel et al – Bruker Corp. and University of Pennsilvania

Dimensionality reduction



Microsc. Microanal. 9, 1–17, 2003
DOI: 10.1017/S1431927603030058

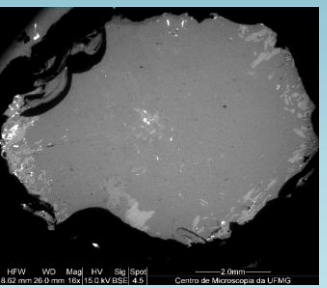
Automated eXpert Spectral Image Analysis

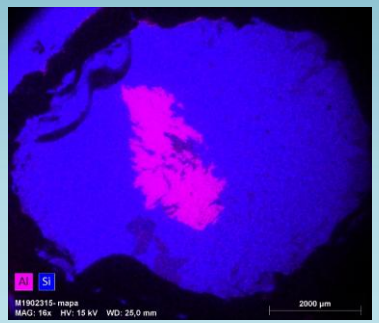
- 2003 – Software Sandia National Laboratories (https://www.osti.gov/biblio/1230701)
- Redução de dados: características espectrais e distribuição espacial
- Modelos:
  - Singular Value Decomposition (SVD),
  - Principal Component Analysis (PCA)
  - PCA with factor rotation
  - Alternating Least Squares-based Multivariate Curve Resolution (MCR-ALS)

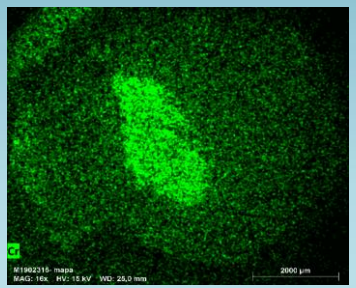
- Técnicas:
  - Energy Dispersive X-ray Spectroscopy, X-ray Fluorescence, Laser_Induced Fluorescence Spectroscopy, Time-of-Flight Secondary Ion Mass Spectroscopy

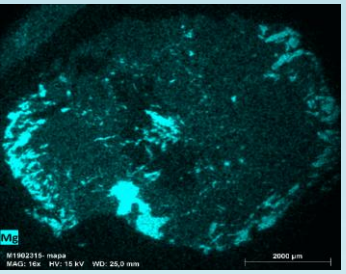
Alexandrite embedded in emerald

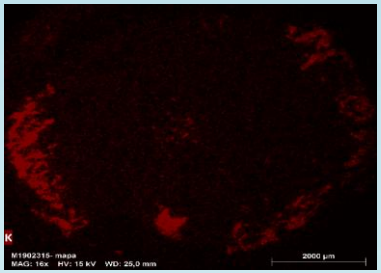
SEM-EDS hypermaps

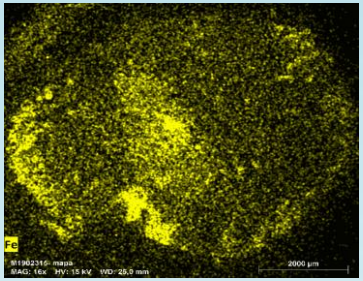




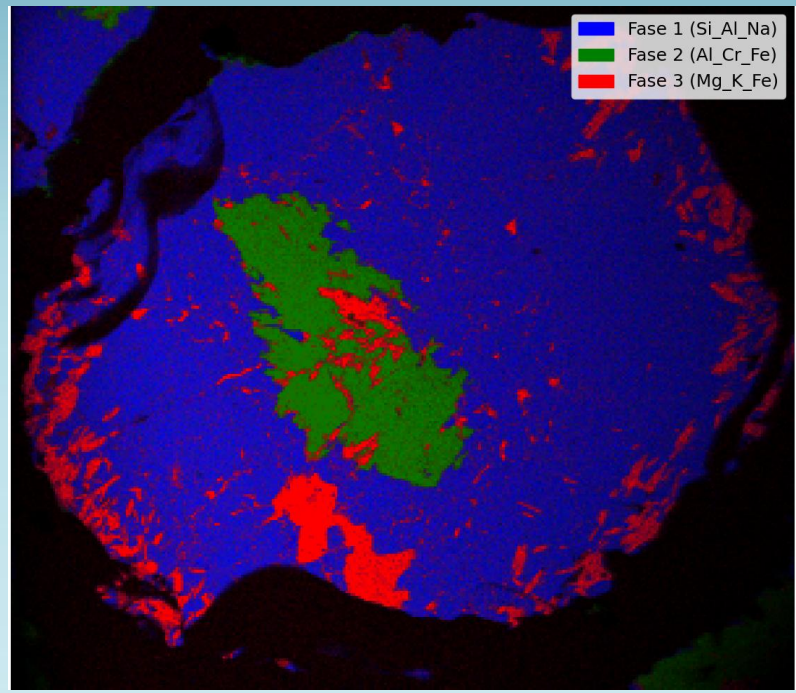








Machine learning phase separation



Storage????

Type of data?

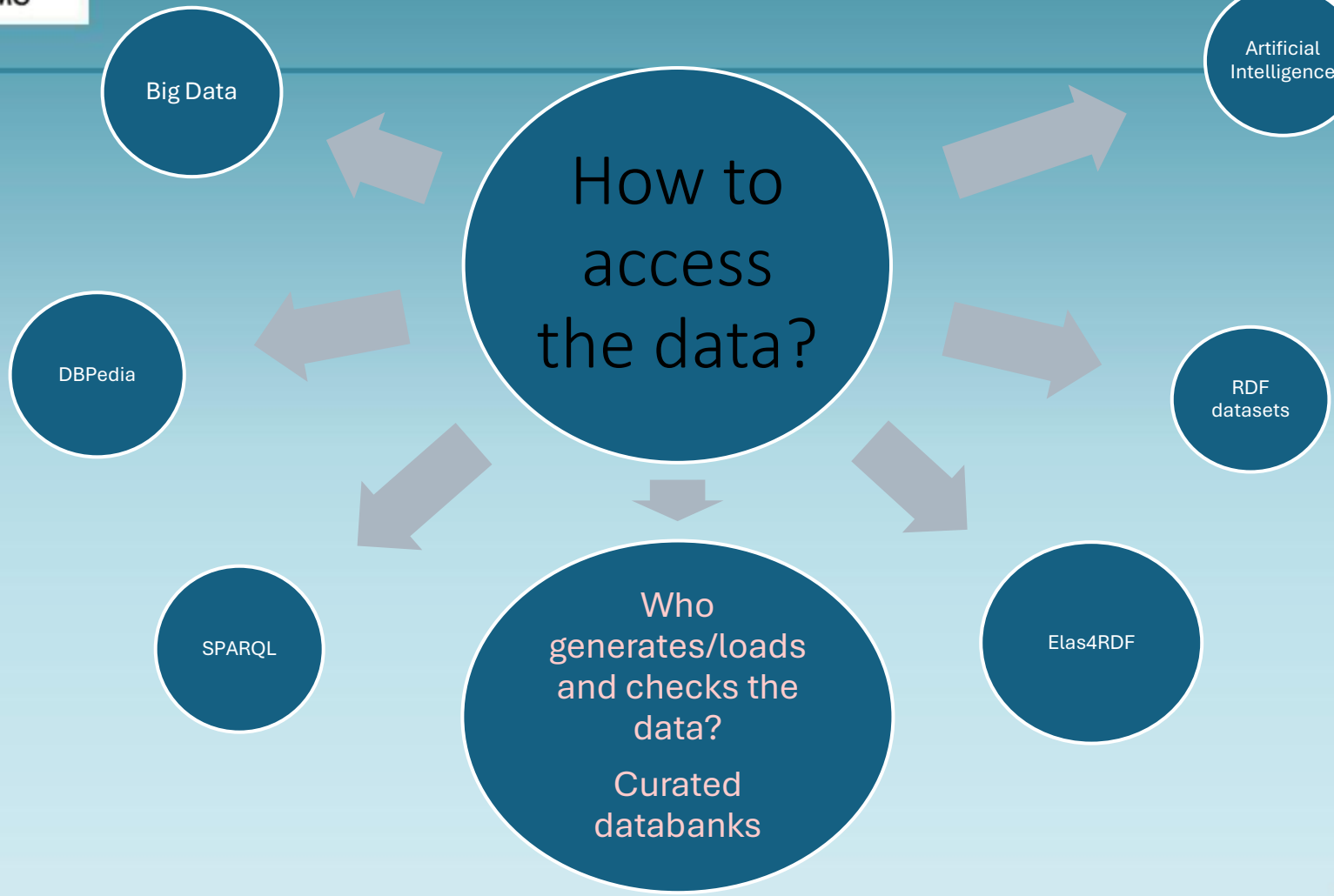
- 1D - spectra
- 2D – image (x,y)
- 3D – hypercube (x,y plus spectra)
- 4D – hypercube (x, y, plus image
- 5D – hypercube plus time?

- 1 byte = 8 bits 0 or 1
- 2^8 = 256 combination = 256 “grey levels”
- Or 3 x 2^8 = 2 x 256 combinations = 256 “color levels” (RGB or HSB ou CMYK, ou …)

- 4 bytes = 4x8 = 32 bits
- 2^{32} = 4.294.967.296 = “billion colors” ….

- 8 bytes = 8x8 = 64 bits
- 2^{64} = ……

- 1024*2 bytes = 2048 bytes = 2kb
- 2024*2048 bytes = 4 194 304 bytes = 4Mb
- 2024*2048*2048 = 8 589 592 bytes = 8.5Gb
- 2024*2048 2048*2048 = 8 796 093 0222 bytes = 8 796Tb


How to access the data?
Big Data
Artificial Intelligence
DBPedia
RDF datasets
SPARQL
Elas4RDF
Who generates/loads and checks the data?
Curated databanks

Challenges

- Adjusted and aligned equipment
- Calibrated equipment (?)
- Correct acquisition parameters
- Correct data treatment

- Good practices
- Knowledge of the technique
- Understand the data

Adjustment

Knowledge

Calibration

Storage

Training

Acknowledgments

UFMG

centro de microscopia da UFMG

E-RIHS
EUROPEAN RESEARCH INFRASTRUCTURE FOR HERITAGE SCIENCE

IPERION HS

Finep
INOVAÇÃO E PESQUISA

ResInfra
EU-LAC

FAPEMIG

CAPES

CNPq
Conselho Nacional de Desenvolvimento Científico e Tecnológico

SisNANO
Sistema Nacional de Laboratórios em Nanotecnologias

IN2PAST.BR

DF UFMG
DEPARTAMENTO DE FÍSICA

Microssonda Eletrônica

  - Tensão de aceleração - 1kV a 50kV
  - **Amostras grossas**

- Detecção de elétrons retroespalhados
- Detecção de elétrons secundários
- Detecção de fótons característicos por espectroscopia de energia dispersiva (EDS)
- Detecção de fótons característicos por espectroscopia de comprimento de onda (WDS)
- Channelling
- Catodoluminescência
- Corrente induzida pelo feixe de elétrons (EBIC)