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Laboratorij za kemijska mjerenja
(LKM)

XRF metoda

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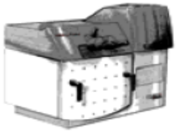
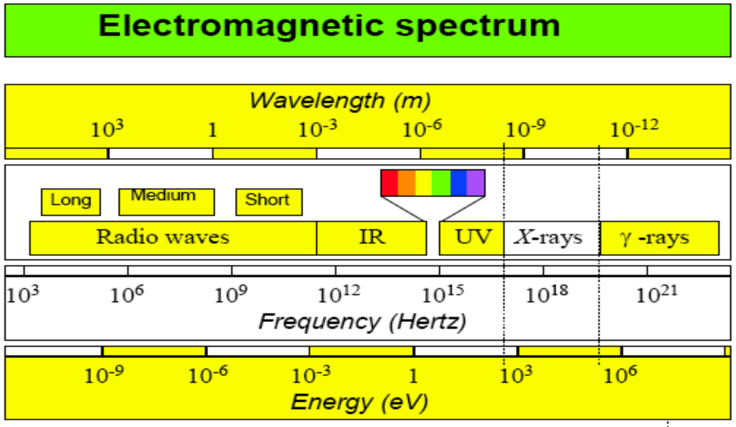
- Nerazarajuća metoda
- Kvalitativna i kvantitativna ispitivanja materijala
- U LKM-u namijenjena je ispitivanju legura koje sadrže plemenite kovine
- Zašto XRF naziv
- Javlja se X zračenje i fluorescencija-svjetlucanje
- Zasniva se na spektrometriji pomoću koje dobivamo naše rezultate

- XRF metoda je čista fizikalna metoda
- Spektrometrija se može koristiti za određivanje identiteta, strukture i okoliša atoma i molekula analizom zračenja koje oni emitiraju ili apsorbiraju
- Potrebno je poznavanje zračenja
- X-Ray spektrometrija zahtjeva raspravu o različitim doprinosima te metode
- Zabluda u koji se može upasti je dobivene rezultate na uređaju smatrati isključivo konačnim rezultatima,često oni zahtjevaju neku korekciju ili obradu

Physics of X-Rays



X-Rays as a part of the electro-magnetic spectrum



For the transformation between wavelength [in nm] and erengy [in keV] the following relation can be used.

$$E_{[keV]} = 1.241 / \lambda_{[nm]}$$

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Physics of X-Rays

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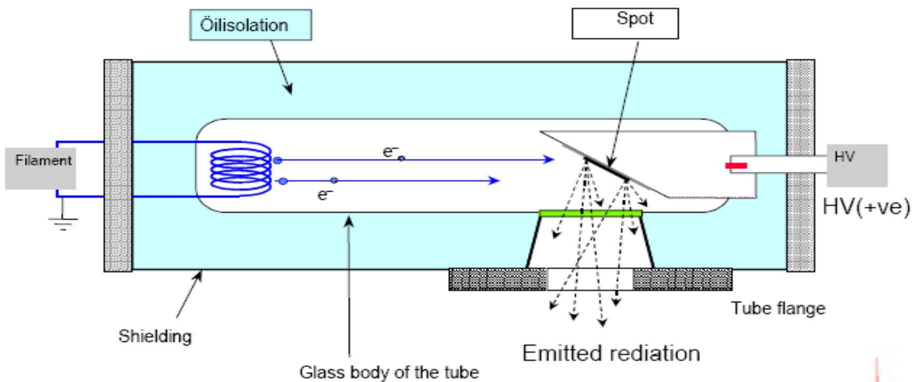
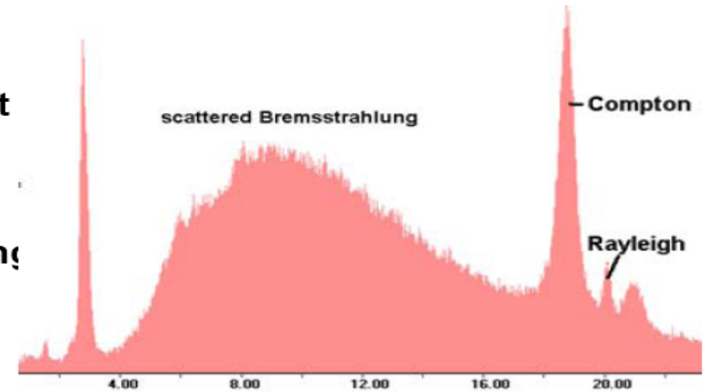
Generation of Y-Rays in a X-Ray tube



- Emission of electrons from the filament
- Acceleration of electrons to the anode
- De-acceleration of electrons on the anode
- Emission of continuous Bremsstrahlun and characteristic fluorescence radiation of the anode material



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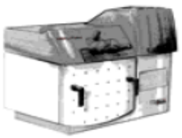
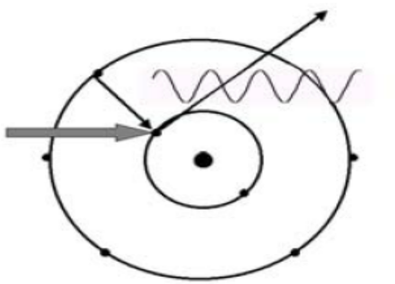


Physics of X-Rays

Exciting radiation

The excitation radiation of a tube consist in characteristic and continuous radiation

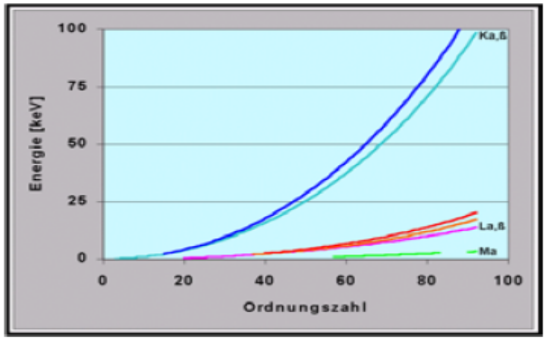
Characteristic radiation



Electrons in the tube ionise a atom of the target-material.

This vacancy will filled up from more outer electrons. The difference energy can be emitted as X-Rays.

The energy of the emitted radiation depends on the type of the atom - it is the characteristic radiation.



The energy of the characteristic radiation is proportional to the square of the atomic number.

$$E \approx Z^2$$

Moseley's law

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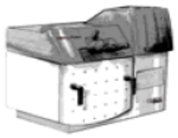
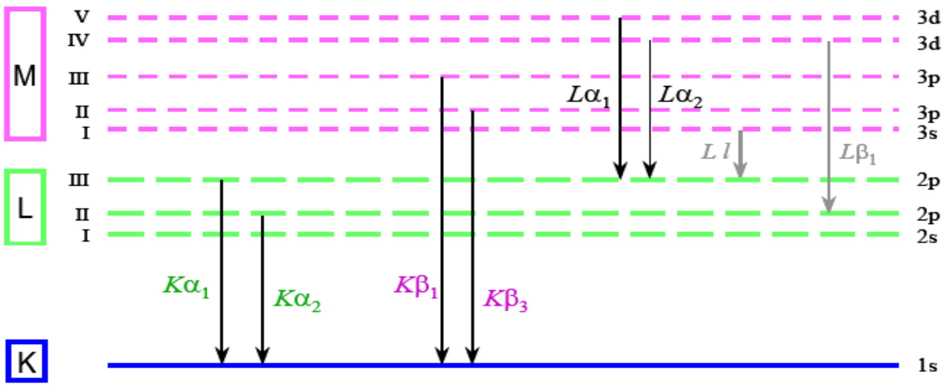
Physics of X-Rays



Nomination of X-Rays



Linie		Übergang	Energie [keV]	Wellenlänge [nm]
Siegbahn	IUPAC			
$K\alpha_1$	K - L3	2p - 1s	8.044	0.15405
$K\alpha_2$	K - L2	2p - 1s	8.026	0.15444
$K\beta_1$	K - M3	3p - 1s	8.904	0.1393
$K\beta_3$	K - M2	3p - 1s	ditto	0.1392
K Kante			8.993	0.1381
$L\alpha_1$	L3 - M5	3d - 2p	0.932	1.3303
$L\beta_1$	L2 - M4	3d - 2p	0.952	1.3023
L_1 *Kante*			1.060	1.1696

For the notation after Siegbahn:

- The name of the shell with the primary vacancy gives the leading letter
- The intensity of the single lines determines the following greeke letter (The intensities depends on the transition probability)

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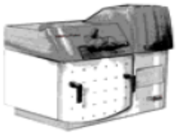
Physics of X-Rays



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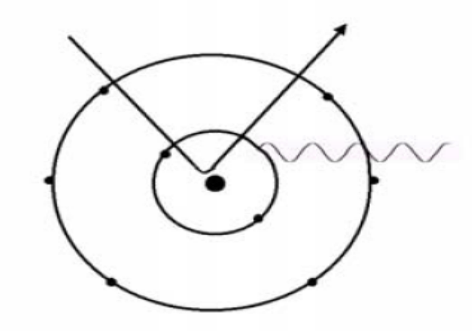
Bremsstrahlung



The distribution of the continuous spectrum is described by Kramer's-formula:

$$I_E \bullet dE = k \bullet i \bullet z \bullet [E-E_o/E]dE$$

with:

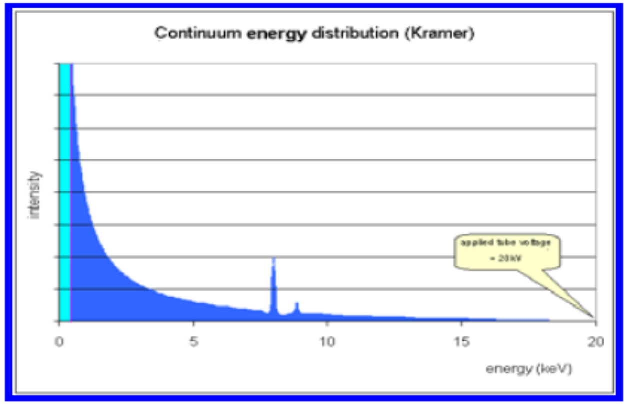
- $I_E \bullet dE$ – intensity distribution
- k – Proportional factor
- i – tube current
- Z – Atomic number of the target
- Eo – Excitation voltage

The incident radiation will be scattered on the nucleus of the atom.

The loss of energy can happen in different steps and will emitted as electro-magnetic radiation.

The tube voltage corresponds to the high energetic end of the spectrum.

The excitation spectrum of the tube looks as follows



Besides the K- and L-lines of the Rh-anode this spectrum shows the continuous Bremsstrahlung too. This radiation is important for the effective excitation of all elements in the sample. The low energy part of this radiaton will absorbed in the tube window.

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Physics of X-Rays

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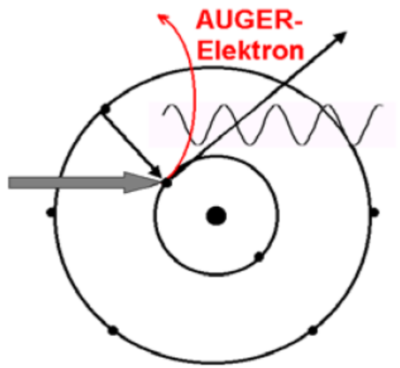
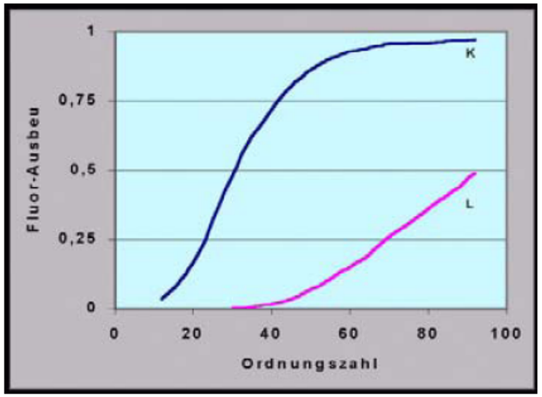
Interaction of radiation with matter

X-Rays are absorbed and scattered in matter.

X-Ray absorption can happen by different processes:

Ionisation of electron shells in the atom





In case of filling the vacancy the free energy can emitted as characteristic radiation or as AUGER-electrons.

The probability for an emission of a X-Ray photon (fluorescence yield) depends on the atomic number.

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Physics of X-Rays

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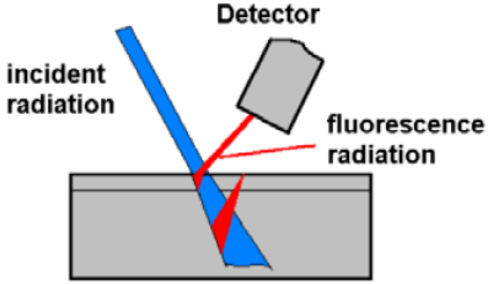
Information depth



The fluorescence radiation is excited in the sample and will absorbed on the way to the sample surface.

Because the excitation radiation every times has the highest energy the information depth is determined by the absorption of the fluorescence radiation in the sample.

The absorption depends on

- the energy of the fluorescence radiation i.e. of the element of interest (Moseley's law)
- the composition of the sample itself (the absorption depends on the mean mass-attenuation coefficient of the sample).

The absorption is described by the Lambert-Beer's law:

$$I = I_o \exp(-\mu^* \rho^* d)$$

With a small change you get:

$$d\,[cm] = \ln(I / I_o) / \mu\rho$$

For a estimation of the information depth it can assumed:

- The ratio I / I_o is 0.05
- The measuring angle of the fluorescence radiation in the Eagle is 60°

From this the following easy relation is concluded:
d [cm] ≈ 4 / μρ

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CRM-legura zlata referentni materijal

Elementi % Au 75.09 Ag12.46 Cu12.44

Metoda: XRF X-Ray spectrometry

Uzorak: platinsko zlato Au Ag Cu Zn Pt Pd

Veličina koja se mjeri: težina %

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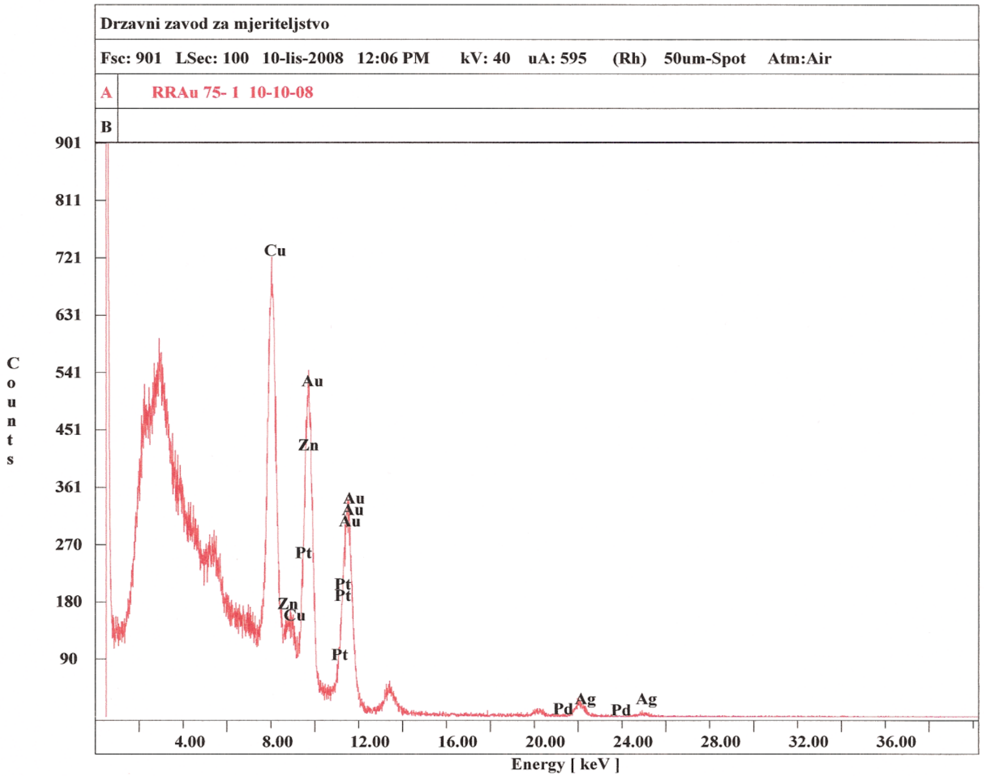

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Elem:	Net	Wt%	At%	I-Error%	BG	Wt-Error
CuK	168.8482	11.46	25.85	0.94	42.16000	0.21
ZnK	0.00000	0.00	0.00	0.00	40.01000	0.00
PtL	10.29025	4.66	3.42	8.07	29.36000	0.38
AuL	130.9600	67.76	49.30	1.03	25.87000	1.29
PdK	0.47000	0.83	1.12	25.08	0.46000	0.21
AgK	7.33000	15.29	20.31	3.92	0.47000	0.65

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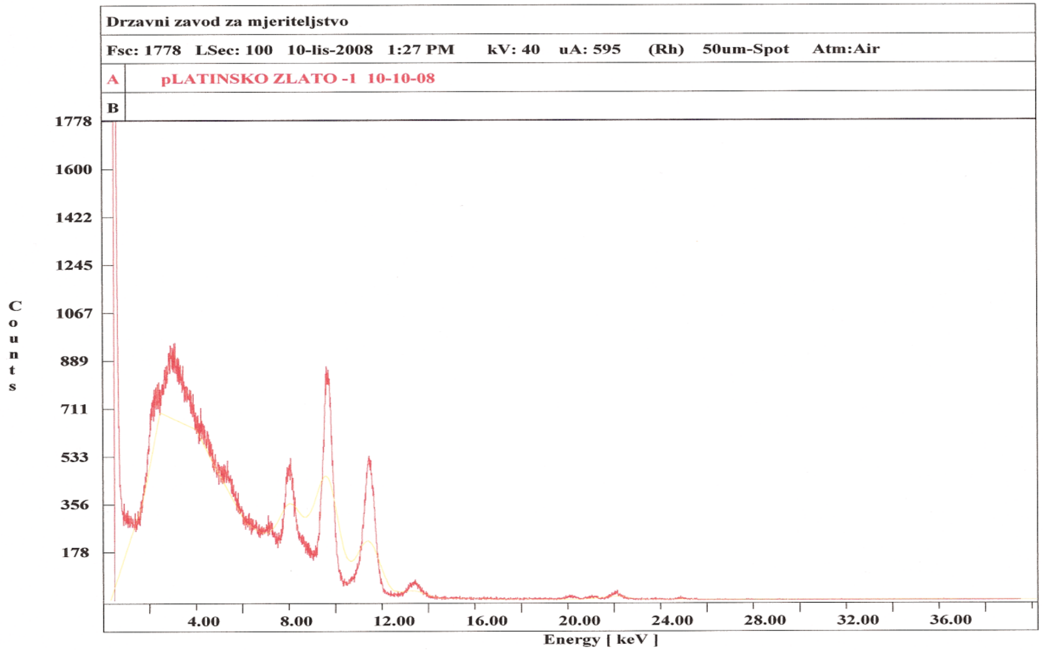

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Elem:	Net	Wt%	At%	I-Error%	BG	Wt-Error
CuK	68.95000	3.13	8.16	1.89	50.09000	0.08
ZnK	0.00000	0.00	0.00	0.00	48.23000	0.00
PtL	87.13999	26.03	22.11	1.45	36.50000	0.56
AuL	167.0800	56.33	47.38	0.90	30.47000	1.04
PdK	2.75000	3.43	5.34	8.41	1.30000	0.29
AgK	7.60000	11.07	17.01	4.19	1.26000	0.50

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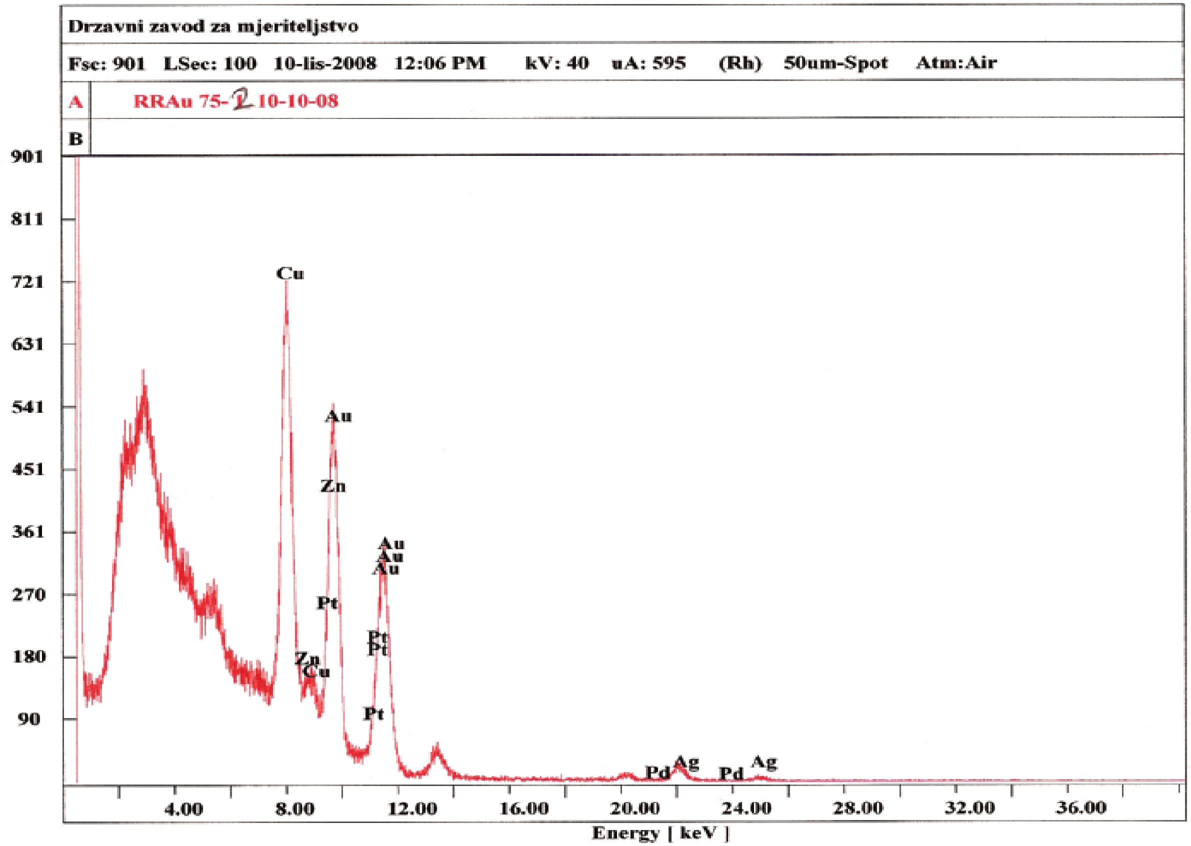

RRAu 75- 1 10-10-08 12:06 PM 10-lis-2008

Elem:	Net	Wt%	At%	I-Error%	BG	Wt-Error
CuK	130.9700	9.56	22.07	1.06	30.11000	0.18
ZnK	0.00000	0.00	0.00	0.00	29.82000	0.00
PtL	35.84000	17.52	13.18	2.51	22.66000	0.52
AuL	101.5700	55.96	41.68	1.17	19.46000	1.11
PdK	0.37000	0.70	0.97	29.23	0.40000	0.21
AgK	7.23000	16.26	22.11	3.93	0.42000	0.69

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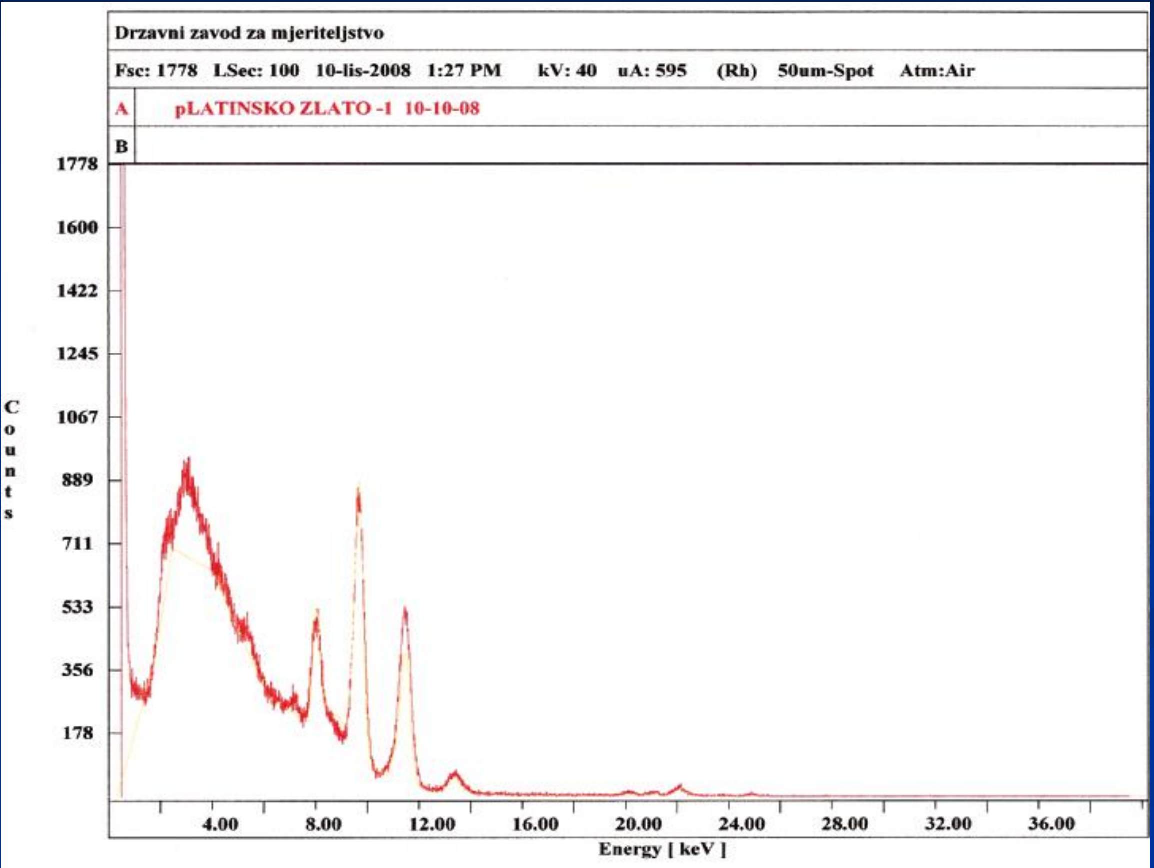

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Elem:	Net	Wt%	At%	I-Error%	BG	Wt-Error
CuK	91.22404	3.95	10.09	1.67	70.13000	0.09
ZnK	5.27537	0.26	0.65	22.03	64.88000	0.06
PtL	44.44269	12.47	10.38	2.65	47.32000	0.39
AuL	212.5400	68.42	56.39	0.81	40.47000	1.23
PdK	3.01000	3.56	5.43	8.16	1.51000	0.30
AgK	8.20000	11.34	17.06	4.05	1.41000	0.49

Nakon pet mjerenja CRM i uzorka učinjen je izračun vrijednosti koja se mjeri (koristeći sve doprinose mjernih nesigurnosti i korekcija)

ZAKLJUČAK

Dobar rezultat ispitivanja XRF metodom postiže se dobrim poznavanjem svih mogućnosti uređaja, poznavanjem i razlučivanjem grafičkih prikaza ne oslanjajući se isključivo na digitalne rezultate i poznavanjem svojstava materijala kojeg ispitujemo.

HVALA NA PAŽNJI !