Determination of Pb Content Using XRF Analysis

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Outline

- Introduction
- Aims and objectives
- Materials and method
- Results and discussion
- Conclusion
- Acknowledgement

X-ray fluorescence (XRF)

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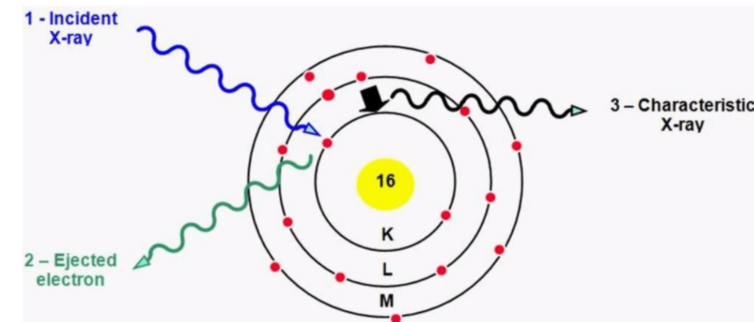
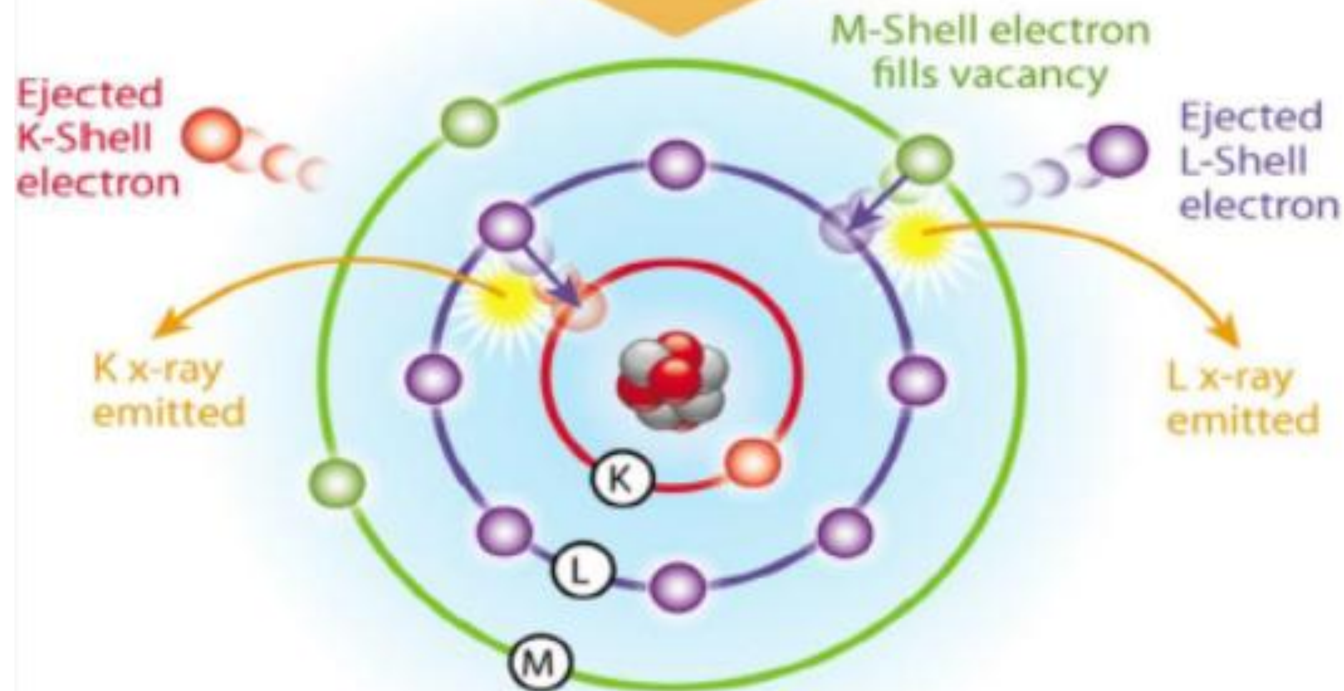
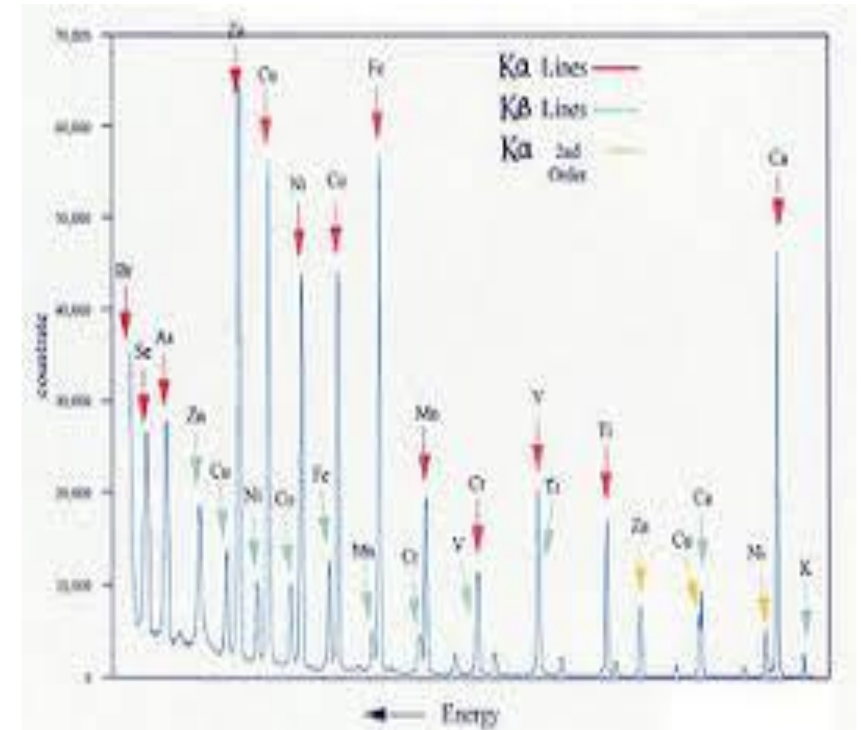
- XRF is a non-destructive analytical technique used to determine the elemental composition
- Analyzes for both solid and liquids materials
- It measures the fluorescent X-ray emitted from a sample when it is excited by a primary X-ray source.
- Each of the elements present in a sample produces a set of characteristic fluorescent X-rays "a fingerprint“
- It is used for: Qualitative and quantitative analysis
- Used in research fields such in geochemistry, forensic science, biological studies, etc.
- It is easy to use and reliable.



Working principle of XRF





Aims and Objectives

Aims

- Determine the content of Pb in thin layer samples using XRF analysis.

Objectives

- Use KORVUS HEX Deposition System to prepare samples and determine the Pb content using the thickness standard curve.
- Prepare a reference sample for calibration of XRF spectrometer.
- Determine Pb content of thin layers by XRF analysis using software WinAxil from Canberra.
- Complete a calibration curve for thin layer samples.

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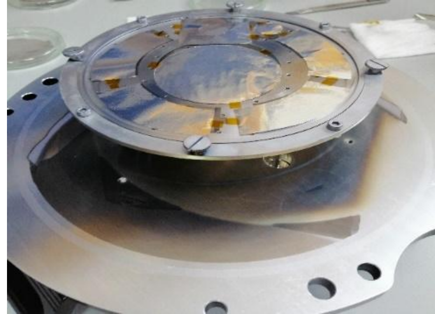
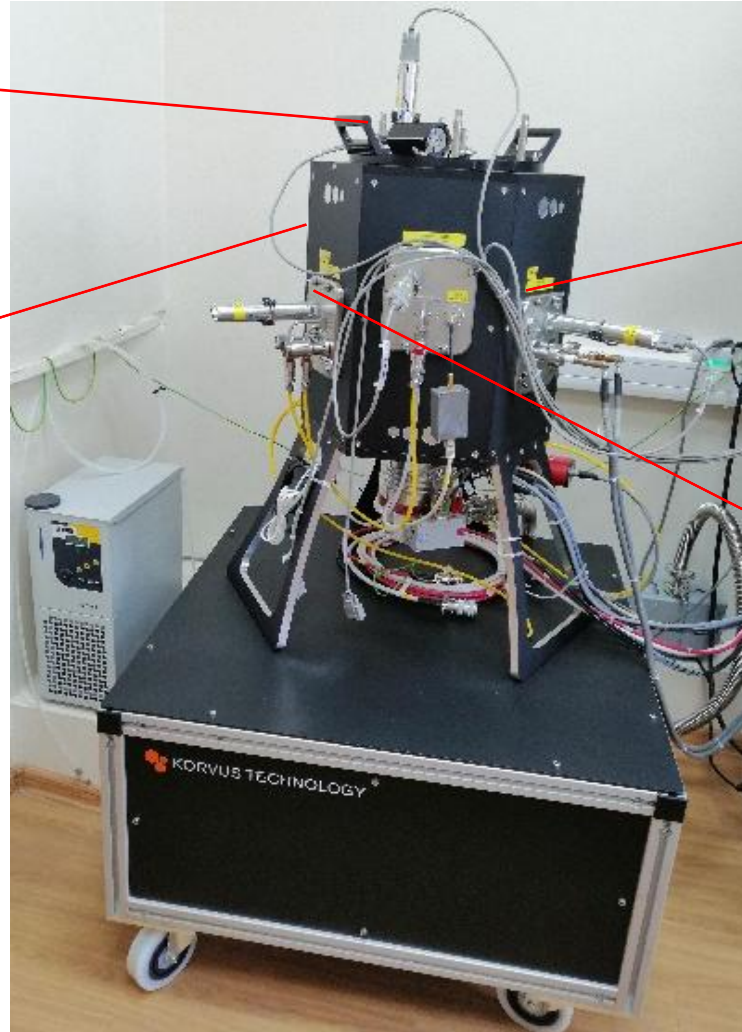
Materials and methods:

Sample preparation:
KORVUS HEX Deposition System

Lid



3. Magnetron sputtering



Types of deposition systems

1. Thermal evaporation

2. E-beam evaporator (TAU)

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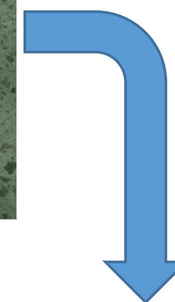
Materials and methods:
Sample preparation

Cut and clean the foil

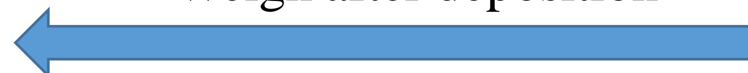
UV-O Cleaner

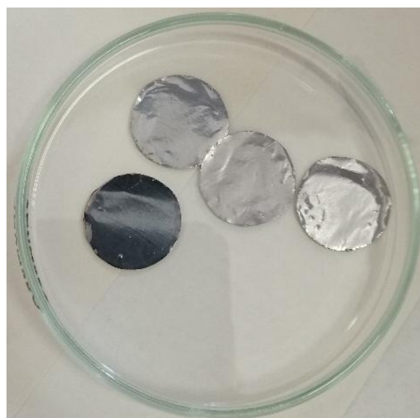
Weigh



KORVUS HEX Deposition System

Weigh after deposition

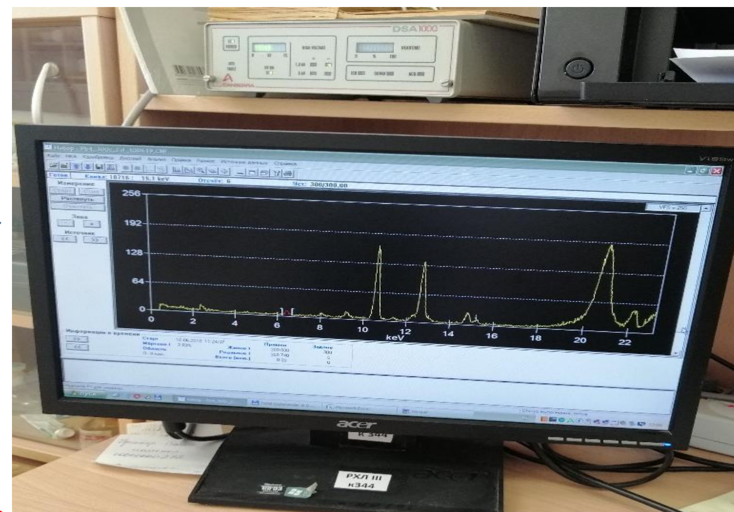




Pb on Al backing

Materials and method:

X-ray fluorescence instrument



- **Computer**
  Spectra

Sample
- Pb on Al backing

X-ray source
- Ringshaped Cd-109 (E= 22.16keV, $T_{1/2}$ = 453 days)

XRF detector
- Si(Li) detector (area 30 mm2 surface, 3 mm thickness
- Be window thickness 25 um, energy resolution (FWHM) – 145eV at 5.9 keV energy)

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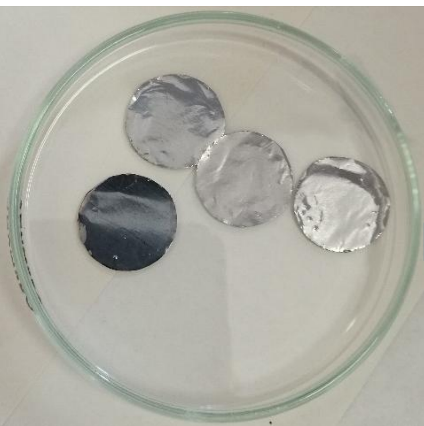
Materials and method:

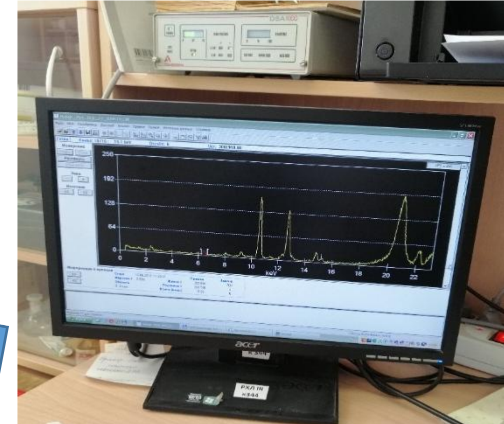
X-ray fluorescence analysis



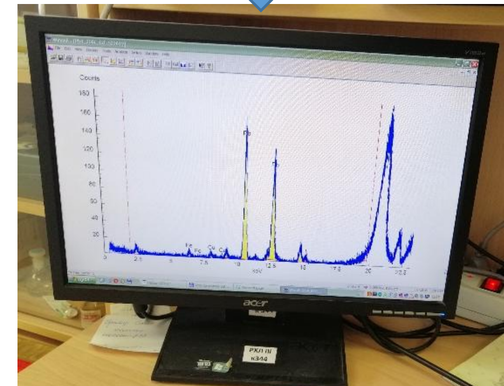
Samples

Cover and lable samples

Place on top of the detector



Spectra with peaks



WinAxil analysis

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Results:

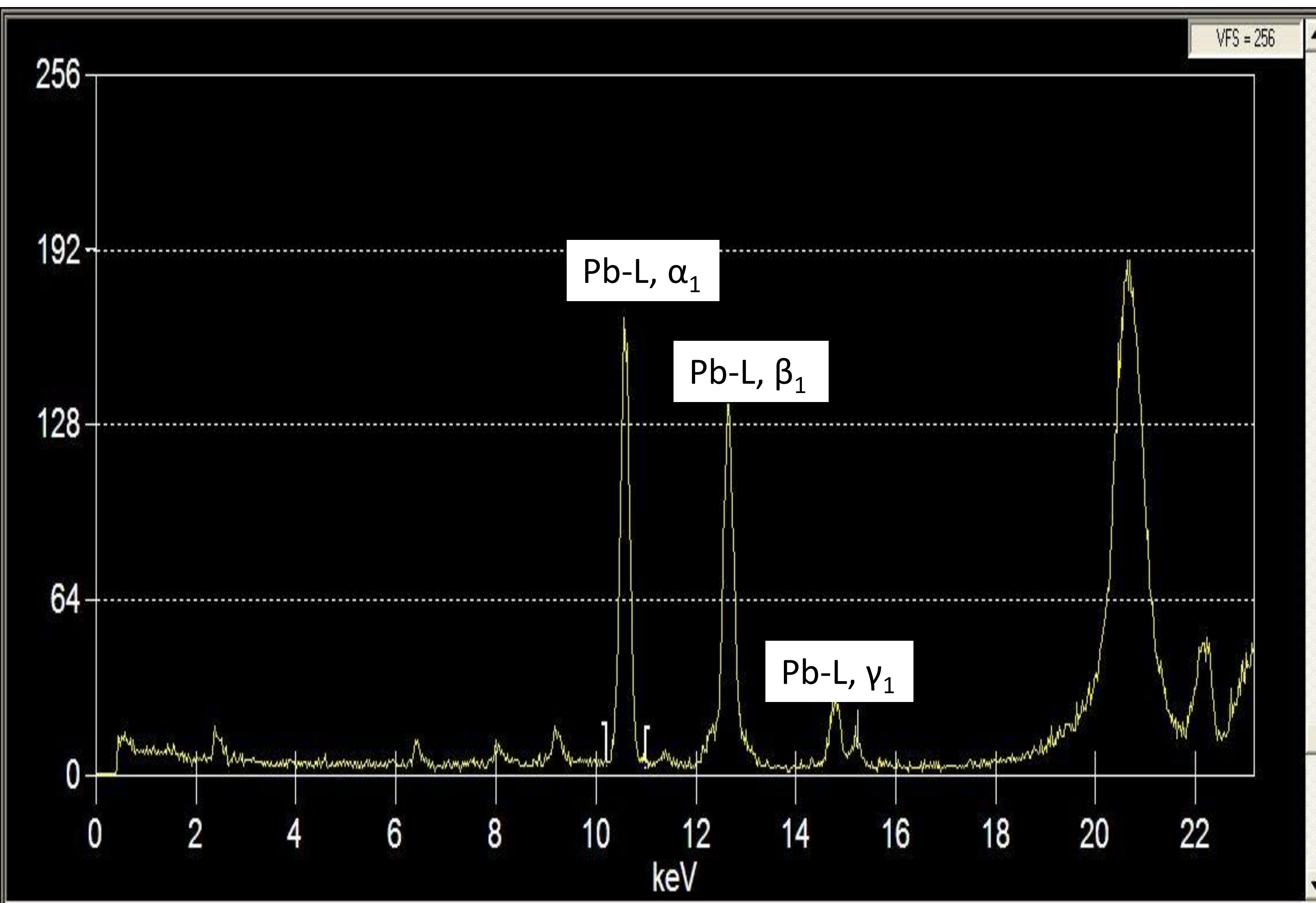
Thermal Deposition System

Table 1: Mass of Pb calculated using the thickness obtained for calibration curve

Sample	Thickness (μg/cm^2)	Mass of Pb on thin film (μg)
1 dot	115 2	290 4
2 dot	308 6	781 16
3 dots	413 10	1049 24
4 dots	510 3	1299 8

Results:

Spectra: Genie 2000



- Sample : 4 dots
- Time for analysis : 300s

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Results:
Spectra: WinAxil

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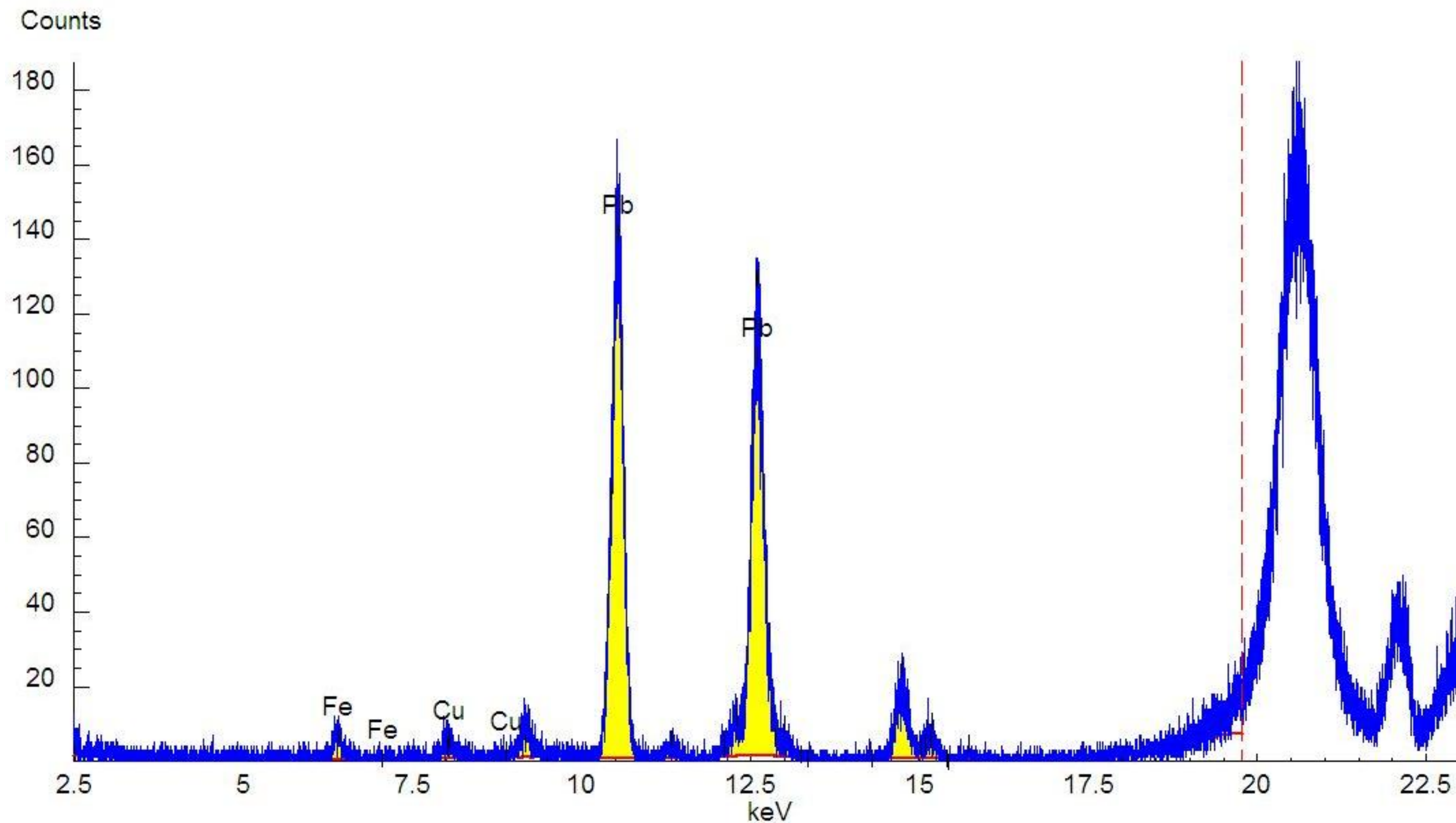

Results:

Spectra: WinAxil

Winaxil - [pb4_300c_cd_100619]

File Edit View Format Window Help

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WinAxil 4.0.1 Spectrum Evaluation Short Report: 10.06.2019  12:04:53

Spectrum: D:\XRFA\спектры\Густова\2019\0610\Pb\Pb4_300c_Cd_100619.wax
Sample:
Fitting region: 1389 - 14025(ch)  1.94 - 19.79(keV)
Continuum: Smooth Filter using 24 iterations
Chi-square: 0.991  4 iterations  Last change: 0.000

El Line      E(keV)      Area ± StdDev      Chi-Square

Fe Ka        6.399        717 ± 28           1.4
Cu Ka        8.041        644 ± 29           2.4
Pb La       10.542      20898 ± 142          0.9
```

- Sample : 4 dots

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Results: XRF

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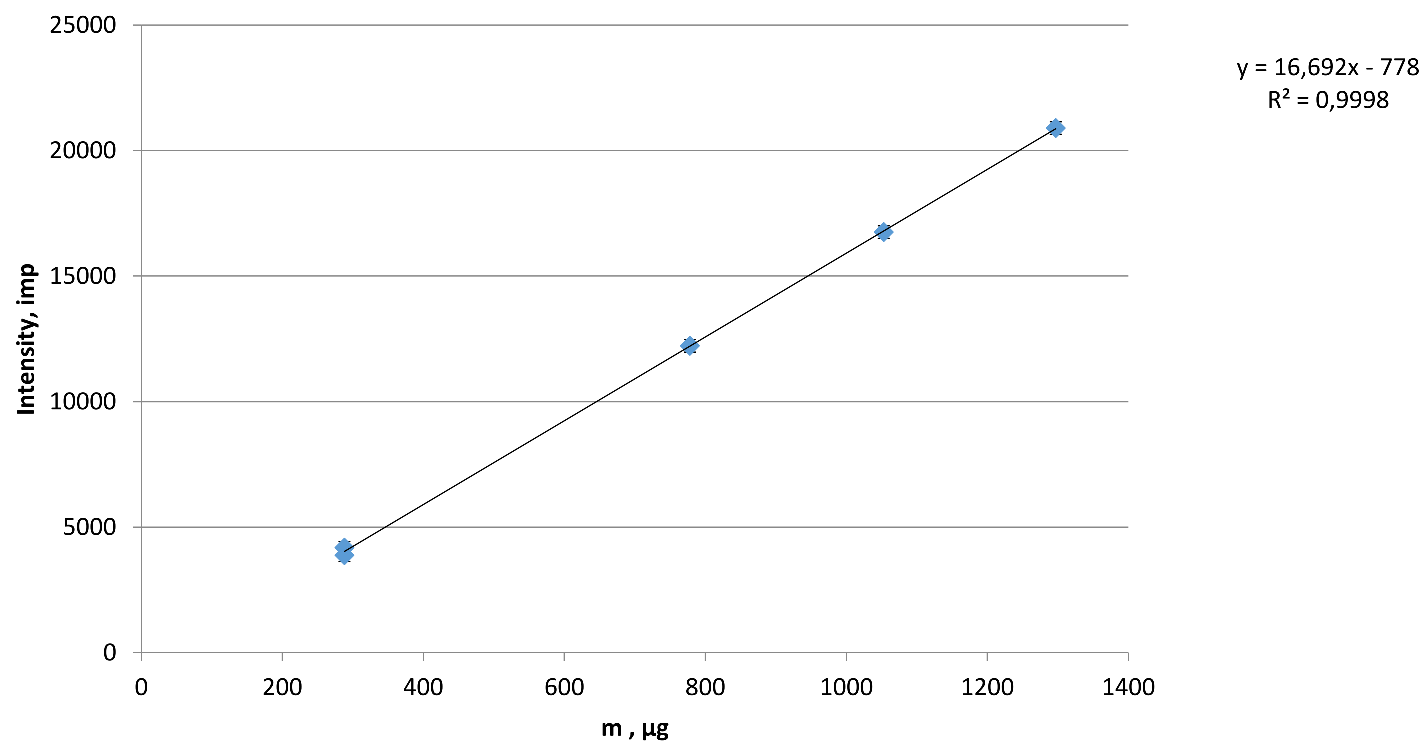


Figure 1: Linear plot of intensity against mass

Results: XRF

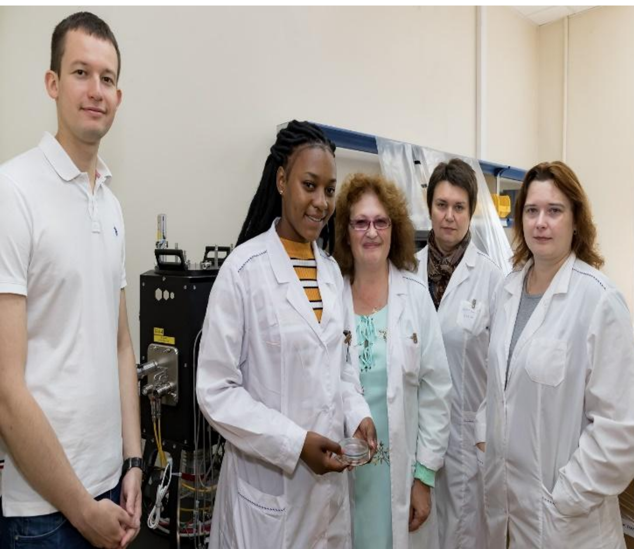
Calibration curve for THIN LAYER samples by ^{109}Cd source

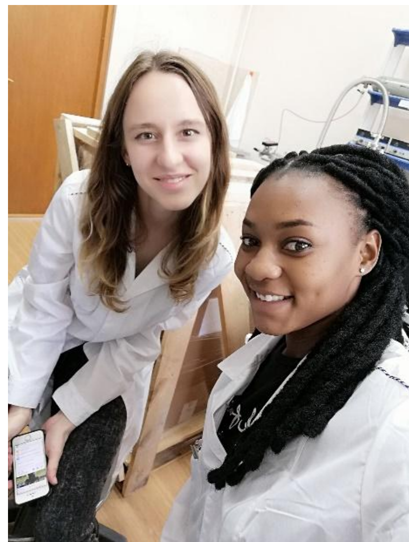
Z	El	Energy (keV)	Intensity (imp)
50	Sn	3.444	273
51	Sb	3.605	421
56	Ba	4.464	1569
57	La	4.651	1889
58	Ce	4.840	2235
59	Pr	5.034	2614
60	Nd	5.230	3019
61	Pm	5.431	3458
62	Sm	5.636	3929
63	Eu	5.846	4435
64	Gd	6.059	4972
65	Tb	6.275	5541
66	Dy	6.495	6145
67	Ho	6.720	6787
68	Er	6.948	7463
69	Tm	7.181	8179
70	Yb	7.414	8920
71	Lu	7.654	9709
72	Hf	7.898	10537
73	Ta	8.145	11403
74	W	8.396	12309
75	Re	8.651	13256
76	Os	8.910	14247
77	Ir	9.173	15280
78	Pt	9.441	16362
79	Au	9.711	17480
80	Hg	9.987	18653
81	Tl	10.266	19869
82	**Pb**	**10.549**	**21132**
83	Bi	10.836	22443
88	Ra	12.338	29792
89	Ac	12.650	31418
90	Th	12.966	33099
91	Pa	13.291	34863
92	U	13.613	36645

Conclusions

- The method for preparing samples of thin layer materials for XRFA (Thermal evaporation deposition) has been studied.
- Theoretical and practical basics of X-ray fluorescence method of analysis have been studied.
- Reference samples for Pb were prepared for calibrating the XRF spectrometer
- The dependence of intensity on the mass of the sample was obtained. This indicates that Pb samples with thin layer thickness of up 500 $\mu g/cm^2$ are thin layers.
- These reference samples of Pb were used to complete a calibration curve for thin film layers samples.

Acknowledgements





Thank you.
Any Questions ?

Спасибо.

Любые вопросы?