X-ray fluorescence analysis of archaeological finds and art objects

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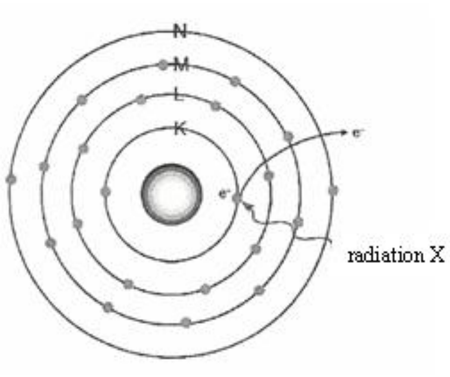
Introduction

X-ray fluorescence analysis (XFA) is a nondestructive analytical method often used to study archeological finds.

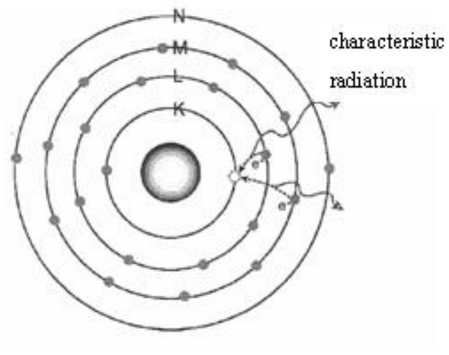
XFA is based on waking and detecting so called characteristic radiation. Energy of characteristic radiation is typical for all elements, so we can find out which elements are in the examined object and their amount, but we can't find out chemical composition.

Theory

The researched object is irradiated by radiation X and the characteristic radiation is emitted. How the characteristic radiation arises on surface K is described on the pictures. The characteristic radiation on other surfaces arises similarly.

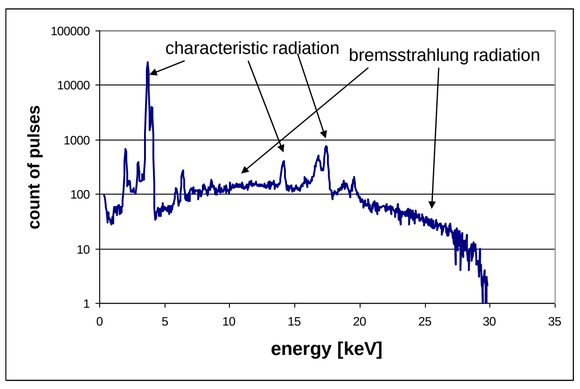


The absorption of a photon of radiation X by electron at the level K



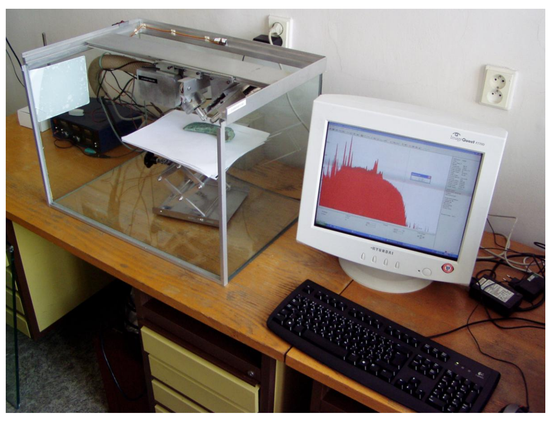
The emission of characteristic radiation series K

We do not detect only characteristic radiation; we also detect so called bremsstrahlung. The bremsstrahlung radiation arises when accelerated electrons are slowing down or changing their direction. It can have all energies from 0 to accelerating voltage (usually near to 30 kV).

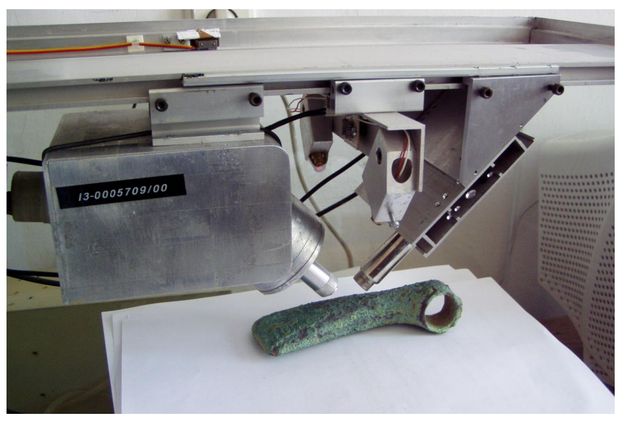


A spectrum with marked characteristic and bremssttrahlung radiation

Apparatus



The apparatus with the computer

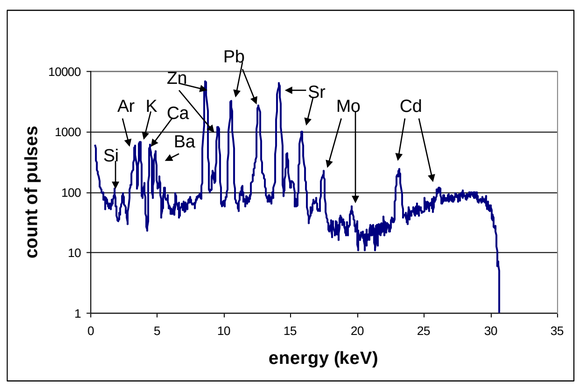


A detail, on the left is the source of X-rays, on the right is detector and in the middle is examined object.

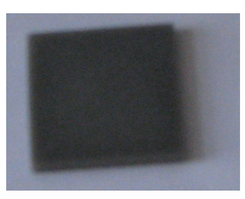
We use an X-ray tube with a molybdenum anode and a silicon detector. The whole apparatus is in the glass box because the glass shades off the X-rays.

Quantitative analysis and standards

To perform quantitative analysis are needed standards. Standards are small subjects whose composition is known with accuracy of hundredths of percents. The standards also have to be homogenous. We analyze the standard and count how many impulses would be from specific element, if the whole subject (100%) is made from that element. A program WinAxil for quantitative analysis can calculate with more data, such as time or chemical form of specific element, and its results are more accurate.



The spectrum of a standard with marked peaks of individual elements



A standard, one side has approximately 5 cm

Detection limits

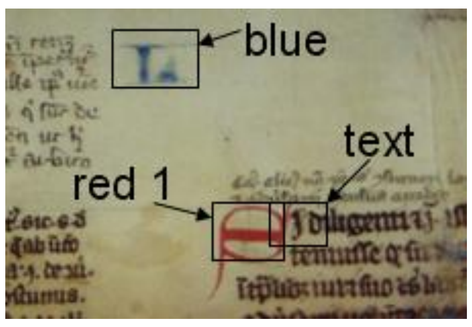
Thanks to the standards we can also find out so called detections limits. Those are minimal concentrations of specific element when we can find out the peak of that element in the spectrum.

In the table there are detection limits for Si and Pb. The acquisition time was 5 minutes. The column „Peaking time" means time, given to the detector to evaluate one impulse. If that time is bigger, the counted value is accurate, but the detector can count two impulses as one.

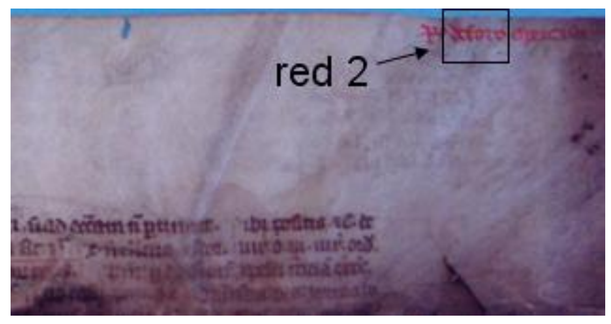
Detector	Current	Peaking time	Detection limit	
			Si	Pb
Si-PiN	100 μA	12.8 μs	5.8%	0.018%
	10 μA	12.8 μs	1.37%	0.0087%
	20 μA	3.2 μs	1.12%	0.0074%
SDD	3 μA	6.4 μs	0.54%	0.043%
	100 μA	0.8 μs	0.15%	0.0097%

You can see that the SDD detector can better detect lighter elements, such as silicon.

Measurement of the old manuscript

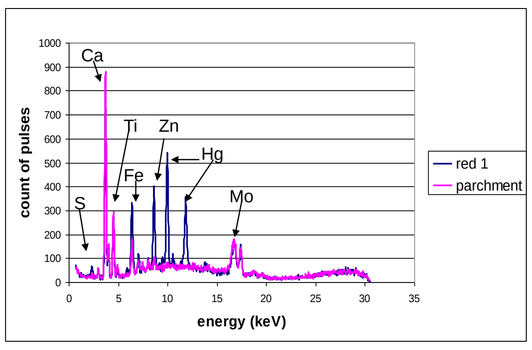


First side of the manuscript



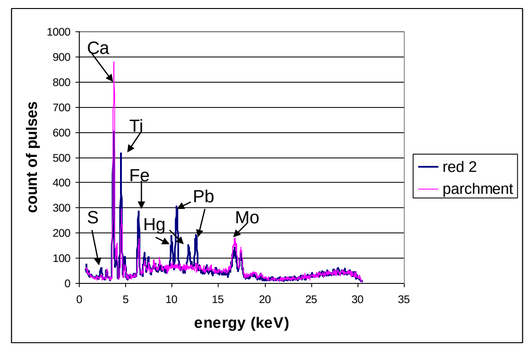
Second side of the manuscript

Because XFA is nondestructive we have used it to analyze the old manuscript. XFA was used to analyze five places on the manuscript – blue pigment, text, two red pigments and the parchment. It is important to analyze the parchment, because all the elements, which are in the parchment, we can also see in the spectrums of pigments and the text.



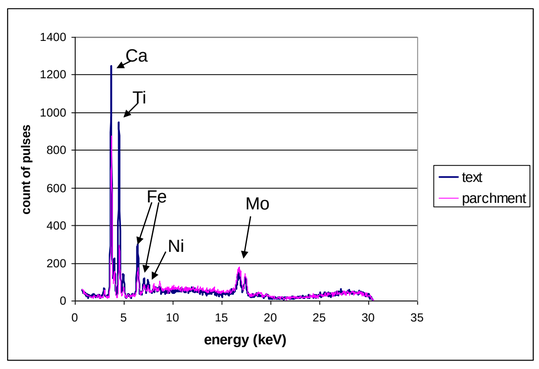
In the first red there are mercury and sulphur, so that is vermilion (HgS)

The first red color under the microscope

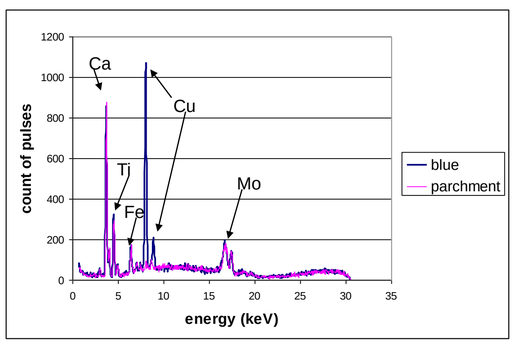


In the second red there are mercury, sulphur and lead, so that is vermilion with minium (Pb_3O_4) or with white lead.

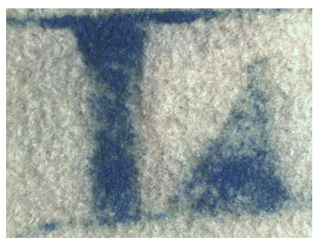
The second red color under the microscope



The text under the microscope



In the blue color there is cuprum, so that is azurite $2CuCO_3.Cu(OH)_2$.



The blue color under the microscope

Acknowledgements

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References

T. Trojek: Využití rentgenfluorescenční analýzy při studiu památek [Doktorská práce (Ph.D.)] (Praha: ČVUT, Fakulta jaderná a fyzikálně inženýrská, KDAIZ 2006)

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