XRF - X Ray Fluorescence

X-ray fluorescence is a method to determine the qualitative and quantitative element composition of solids, liquids and powders using X-rays. It is based on X-rays hitting atoms, causing a secondary radiation that is characteristic for each element in the periodic system.

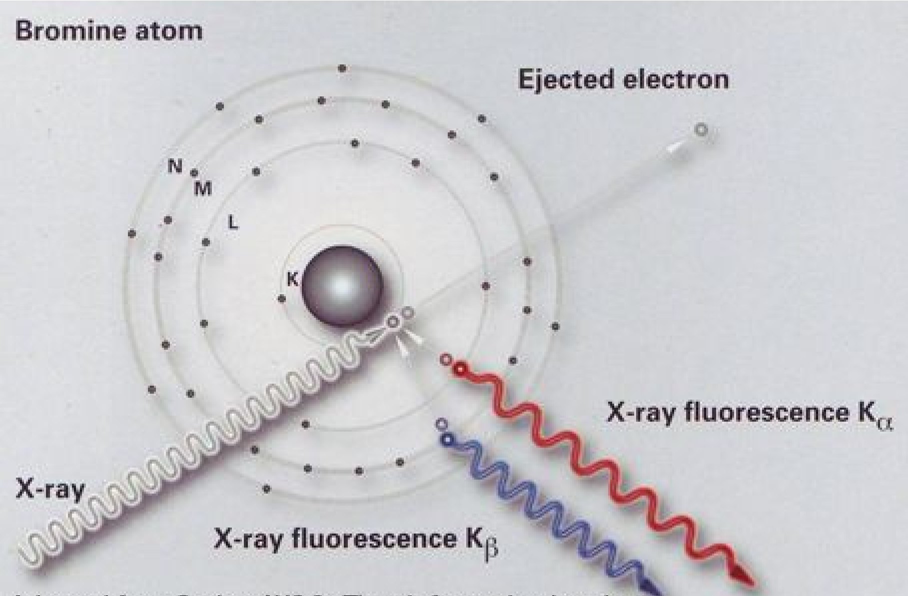
The method is based on Bohr's atomic model.

According to this model, a positively charged nucleus is surrounded by electrons located in specific areas. These areas are called "shells" and are indicated by the letters K (the innermost), L, M, N, ...

The binding energy between the electrons and the nucleus depends on:

1) the shell in which the electrons are located.

2) the atomic number (Z) of the element in the periodic system (elements with higher atomic numbers have higher binding energies for the respective shells) – e.g. the binding energy of the electrons of Silver K shell is greater than the binding energy of the electrons of Aluminium K shell.



Adapted from Bruker AXS S8 Tiger information brochure

The collision of X-rays with atoms can result in the removal of electrons from various shells leaving vacant positions behind. These vacancies are subsequently filled by electrons from higher energy shells and the excess energy of these electrons is emitted as secondary X-ray fluorescence radiation.

When e.g. an electron from the L shell substitutes a vacancy in the K shell, Kα radiation is emitted. Kβ radiation is emitted when an electron from the M shell moves to the K shell. When an electron from the M shell substitutes a vacancy in the L shell Lα is emitted. Each element has characteristic energies for the secondary X-ray fluorescence.

An analysis:

Subject of the analysis: Canon lens 50 mm., F/1.2

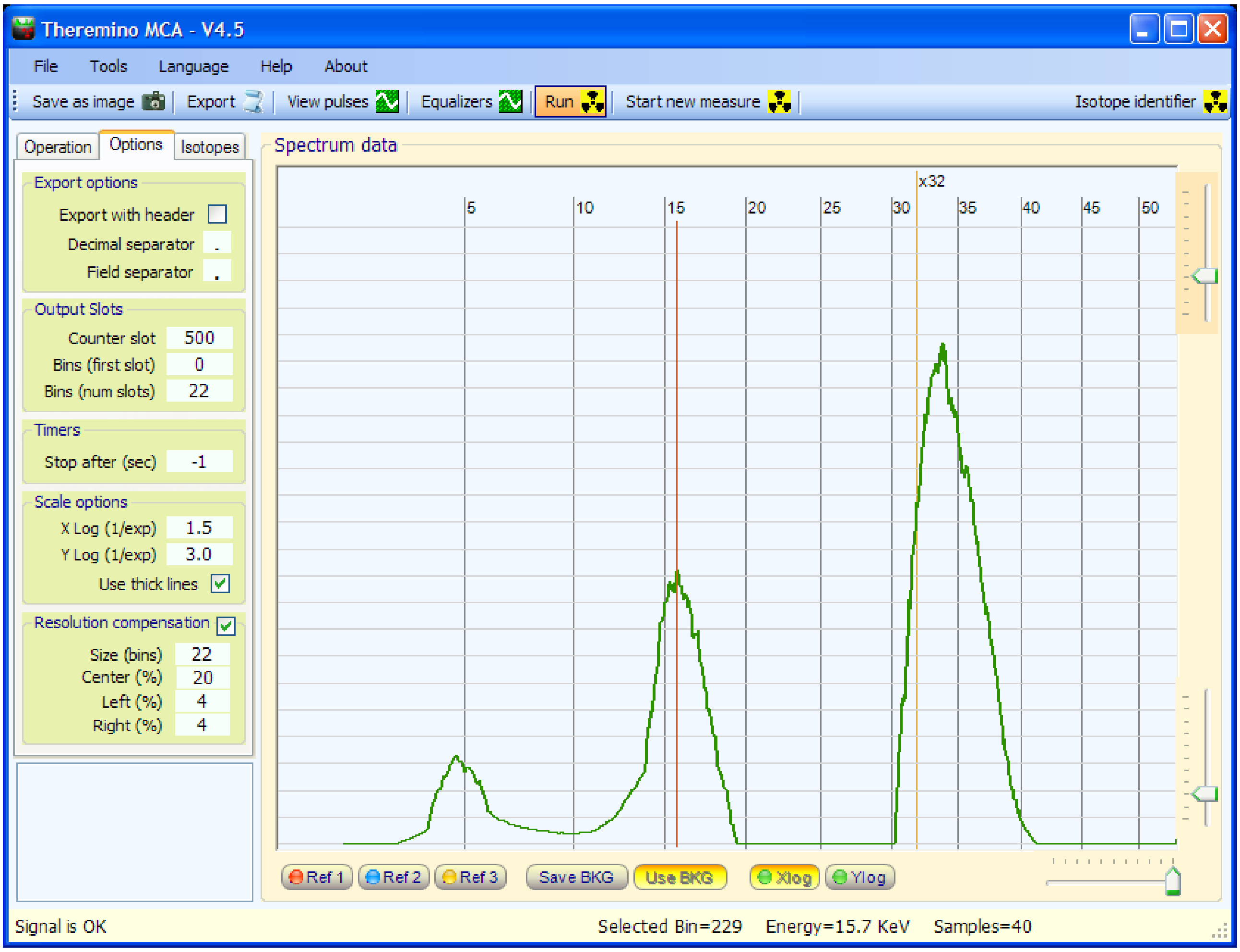
This is a very fast lens and this could partially due to a peculiar anti-reflective coating - the purpose of the analysis is to discover the composition of is this coating.

7 uCi of Am241 (not 8 as in the above image) has been used, arranged around a hole in a lead plate, behind this hole a RAP47 25.4 x 1 mm probe was placed.

Please note that good results may be achieved using just one uCi of Am241, but in this case looking carefully for the geometric relationships between probe, source and target material.

Results of the Analysis

The results of the analysis are graphed, on the x axis the energy of the X-rays emitted from the material on test, and on the y axis the intensity of the emitted rays are placed.



Three peaks were detected at energies of: 4.7 keV - 15.7 keV - 33.9 keV

Finding the unknown elements:

4.7 keV (Titanium: K-alpha1 = 4.51 keV, K-alpha2 = 4.93 keV) the peak is located between this two values.

15.7 keV (Zirconium: K-alpha1 = 15.77 keV, K-alpha2 = 17.77 keV) no K-alpha2 contribution in this case.

33.9 keV (Barium: K-alpha1 = 32.19 keV, K-alpha2 = 36.38 keV) the peak is located between this two values.

Comments on found elements:

Barium is a standard component of good glasses.

Titanium comes from the body of the lens, made of magnesium and titanium (the values of magnesium 1.25 and 1.30 keV are not detectable at room temperature).

Zirconium: A quick look to Wikipedia let us understand that zirconium oxide is often used in anti-reflective coatings, we have so reached already the goal of our research.

Software settings

The signal from the photomultiplier tube was detected, filtered and amplified by a Theremino Pmt Adapter and then sent to the software Theremino MCA 4.5.

We applied the "background subtraction" and the "compensation of the resolution." to the resulting signal

The analysis, carried out smoothly, took only 120 seconds (after the equipment calibration).

Calibration

To calibrate this device It's possible to use the same Am-241 element that we has been using to hit the atoms: it has a well defined peak at 59.5 keV, unfortunately its minor peaks are usually difficult to identify in a correct way.

To overcome this problem we can XRF some element: for the lowest values of energy, X-rays from iron (6.4 keV) and copper (8.8 keV) can be used.

A third point can be provided by silver X-rays (22.16 keV).

Strontium can supply the values (14.16 keV) for a fourth point. (Sr can be found in some phosphorescent paints of relatively recent formulation, as $SrAl_2O_4$).

Periodic Table with XRF energy

This Periodic Table shows, for each element, the characteristic energy of the K and L shells.

We printed here just the items of greatest interest to XRF.

The full periodic table can be found here: [PeriodicTable.pdf]

(Tables courtesy of George Dowell - GEOelectronics)

Legend

K Alpha 1		K Beta 1
	Elemental Symbol	
	Atomic Number	
	Elemental Name	
L Alpha 1		L Beta 1

Fe K / Cd K / Fe L / Cd L

Symbol	Atomic Number	Name	K Alpha 1	K Beta 1	L Alpha 1	L Beta 1
K	19	Potassium	3.31	3.59		
Ca	20	Calcium	3.69	4.01	0.34	0.34
Sc	21	Scandium	4.09	4.46	0.39	0.40
Ti	22	Titanium	4.51	4.93	0.45	0.46
V	23	Vanadium	4.95	5.43	0.51	0.52
Cr	24	Chromium	5.41	5.95	0.57	0.58
Mn	25	Manganese	5.90	6.49	0.64	0.65
Fe	26	Iron	6.40	7.06	0.70	0.72
Rb	37	Rubidium	13.39	14.96	1.69	1.75
Sr	38	Strontium	14.16	15.83	1.81	1.87
Y	39	Yttrium	14.96	16.74	1.92	2.00
Zr	40	Zirconium	15.77	17.67	2.04	2.12
Nb	41	Niobium	16.61	18.62	2.17	2.26
Mo	42	Molybdenum	17.48	19.61	2.29	2.40
Tc	43	Technetium	18.41	20.59	2.42	2.54
Ru	44	Ruthenium	19.28	21.66	2.56	2.68
Cs	55	Cesium	30.97	34.98	4.29	4.62
Ba	56	Barium	32.19	36.38	4.47	4.83
Lanthanide Series	57 - 71					
Hf	72	Hafnium	55.76	63.21	7.90	9.02
Ta	73	Tantalum	57.52	65.21	8.15	9.34
W	74	Tungsten	59.31	67.23	8.40	9.67
Re	75	Rhenium	61.13	69.30	8.65	10.01
Os	76	Osmium	62.99	71.40	8.91	10.35

Symbol	Atomic Number	Name	K Alpha 1	K Beta 1	L Alpha 1	L Beta 1
Co	27	Cobalt	6.93	7.65	0.77	0.79
Ni	28	Nickel	7.48	8.26	0.85	0.87
Cu	29	Copper	8.05	8.90	0.93	0.95
Zn	30	Zinc	8.64	9.57	1.01	1.03
Ga	31	Gallium	9.25	10.26	1.10	1.12
Ge	32	Germanium	9.89	10.98	1.19	1.22
As	33	Arsenic	10.54	11.73	1.28	1.32
Se	34	Selenium	11.22	12.50	1.38	1.42
Rh	45	Rhodium	20.21	22.72	2.70	2.83
Pd	46	Palladium	21.18	23.82	2.84	2.99
Ag	47	Silver	22.16	24.94	2.98	3.15
Cd	48	Cadmium	23.17	26.09	3.13	3.32
In	49	Indium	24.21	27.27	3.29	3.49
Sn	50	Tin	25.27	28.48	3.44	3.66
Sb	51	Antimony	26.36	29.72	3.61	3.84
Te	52	Tellurium	27.47	30.99	3.77	4.03
Ir	77	Iridium	64.87	73.55	9.19	10.71
Pt	78	Platinum	66.82	75.74	9.44	11.07
Au	79	Gold	68.79	77.97	9.71	11.44
Hg	80	Mercury	70.82	80.26	9.99	11.82
Tl	81	Thallium	72.86	82.56	10.27	12.21
Pb	82	Lead	74.96	84.92	10.55	12.61
Bi	83	Bismuth	77.10	87.34	10.84	13.02
Po	84	Polonium	79.30	89.81	11.13	13.44

Peaks as seen from a HPGe probe

An HPGe, liquid nitrogen cooled probe, would supply peaks with much great resolution. Scintillator crystals on the contrary, as the CsI(Tl) used here, have much wider peaks; the HPGe are however very expensive, like a small car, and the cold liquid nitrogen would be a big problem for many.

These graphs, achieved with an HPGe Nitrogen cooled probe, may however be used as a reference: