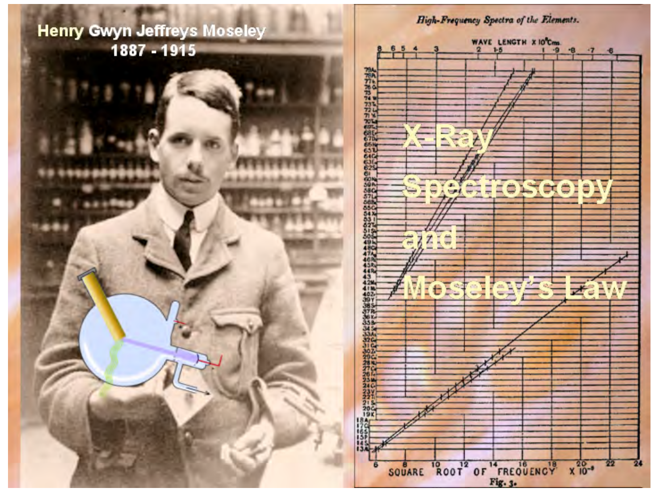
5 X-Ray-Spectroscopy and Moseley's Law



Location: room Jordan 305

A. Short Description

X-ray spectroscopy can be used to study inner shell phenomena of atoms, states of highly ionized atoms produced by accelerators or to determine material properties. There are two principal methods : Using a semiconductor detector or a Bragg-type spectrometer (see expt. Nr. 17). Semiconductor detectors are advantageous as they are simple, portable and yield sufficient resolution to distinguish adjacent elements. They have a good intrinsic efficiency, and an acceptable energy resolution in the range of a few percent, conversly the Bragg method yields much higher resolution, but very poor detection efficiency. In our experiment a high resolution silicon detector is used to measure the K-lines (or also L-lines) of several elements to verify Moseley's law. The excitation of inner shell vacancies is performed by using the 59.5 keV γ-line of a strong ^{241}Am source (best range $30 < Z < 65$), or by using a small special X-ray tube with a rhodium anode which has its maximum of emission near 10 keV. The second method is preferable for lower Z elements ($10 < Z < 30$). By observing the characteristic L - lines, one gets access to the elements with Z up to 92, but the L–spectra yield more lines, and are more complicated. Moseley's relation has a broad field of application in material analysis — see experiment Nr. 18.

Remarks

Henry Gwyn Jeffreys Moseley (November 23, 1887 – August 10, 1915) was an English physicist and a graduate of Trinity College Oxford. His main contributions to science were the quantitative justification of the previously empirical concept of atomic number, and Moseley's law. This law advanced chemistry by immediately sorting the elements of the periodic table in a more logical order. Moseley could predict the existence of several then-unknown elements. Moseley's law also advanced basic physics by providing independent support for the Bohr model of the Rutherford/Antonius Van den Broek nuclear atom, containing positive nuclear charge equal to atomic number.

As Niels Bohr once said in 1962, "You see actually the Rutherford work [the nuclear atom] was not taken seriously. We cannot understand today, but it was not taken seriously at all. There was no mention of it any place. The great change came from Moseley."

Moseley fought at Gallipoli, Turkey, where he was killed in action by a sniper in 1915, shot through the head while in the act of telephoning an order. Many speculated that he should have won the Nobel Prize, but was unable to because it is only awarded to the living. It is speculated that because of Moseley's death in the war, the British and other world governments began a policy of no longer allowing their scientists to enlist for combat.

B. Moseley's Formulae

Moseley's formulae for K-alpha and L-alpha lines, in his original semi-Rydberg style notion (squaring both sides for clarity):

$$f(K_\alpha) = (3.29 \times 10^{15}) \cdot \frac{3}{4} \cdot (Z - 1)^2 \qquad Hz$$

$$f(L_\alpha) = (3.29 \times 10^{15}) \cdot \frac{5}{36} \cdot (Z - 7.4)^2 \qquad Hz$$

the simple notation of Moseley's law being:

$$\sqrt{f} = k_1 \cdot (Z - k_2).$$

Here f is the frequency of the main or K-X-ray emission line, and k_1 and k_2 are constants that depend on the type of line, k_1 is given in units of the fundamental Rydberg frequency f_{Ry} = (3.29×10^{15}), $k_1 = \frac{3}{4} = 1 - \frac{1}{4}$ for the K-alpha-lines and $k_1 = \frac{5}{36}$ for the L-alpha lines.
The constant k_2 describes the screening of the nuclear charge and it was obtained empirically to be 1 for the K-alpha transitions, and 7.4 for the L-alpha transitions.

C. Necessary Knowledge

Physics:

- Basic atomic physics of inner shells, concepts and terms, characteristic numbers, Roentgen fluorescence yield, binding energy in the shell model of the atom, Auger electron emission
- Production of X-rays
- Bremsstrahlung; Synchrotron radiation

- Characteristic X-rays, selection rules, K- and L-series
- Absorption and scattering of X-rays
- Applications of characteristic X-ray spectroscopy
- Some quantitative considerations

Measuring Technique :
- Principles of X-ray detectors, especially silicon detectors
- Classical method of X-ray wavelength determination
- Function of the specific ^{241}Am source
- Function of an X-ray tube
- Required electronic units and function
- Multichannel-Analyzer

Mathematics : Method of least squares, line separation, background subtraction

References

[1]	**Moseley H. G. J. :** Phil. Mag. **26**, 1024 (1913) and Phil. Mag. **27**, 703 (1914)	Original papers
[2]	**... :**	......
[3]	**Deslattes, R.D., E.G. Kessler Jr., P Indelicato, L. de Billy, E. Lindroth and J. Anton :** X-ray transition energies : new approach to a comprehensive evaluation Re. Mod. Phys. **75**, 35 (2003)	Most recent review article on all X-ray transitions of the elements
[4]	**Jelen, Christian :** Moseley's Gesetz und Röntgen Spektroskopie Thesis of high school teachers, University of Stuttgart 2000	Original thesis on this teaching lab experiment
[5]	**Bergmann-Schäfer:** Experimentalphysik, Bd. IV, Teil 1, S. 129 ff Walter De Gruyter, Berlin, 1975	Basics of x-ray Radiation
[6]	**Firestone, R. B. :** Table of Isotopes Vol. II John Wiley & Sons, New York, 1996	Current reference on nuclear data
[7]	**Mayer-Rimini:** Ion Beam Handbook for Material Analysis Ch. 5	

Experimental Tasks

- Get the variable X-ray source from the TA
- Turn on first the NIM power, then turn on very slowly the high voltage bias of the detector (- 500 Volts) and watch the signals from the main amplifier. In case they are not regular stop and ask.
- Important: Check the main settings of the main amplifier to obtain good resolution. Time constant 2 - 3 μs should work best. Set amplification so that the 59.5 keV line of ^{241}Am has an amplitude of nearly 8 Volts.
- Set MCA to 2048 or 4096 channels (both channel select and conversion gain)
- Check pole zero cancellation very carefully with the oscilloscope
- Calibration of the detector using three lines Cu K_α, Ba K_α and 59.5 keV ^{241}Am line, you can also use the Cs-137 (36 keV X-ray), the Mn-54 (5.4 keV X-ray) and the Co-57 (14.4 keV γ-ray) source; measure the calibration spectrum with all lines at once, but keep the total count rate within reasonable limits (1 kHz)
- Determine detector resolution in keV for at least two lines (iron K_α and 122 keV line of ^{57}Co)
- Measure the K_α and K_β lines of the six elements of the variable source
- Verify Moseley's law with a graph and table (+ uncertainties)
- Determine 1 – 2 unknown samples (ask TA which ones)
- Alternative task: Measure several L-spectra for various elements and explain them
- In addition, you can determine more elements than six, which have appropriate K lines

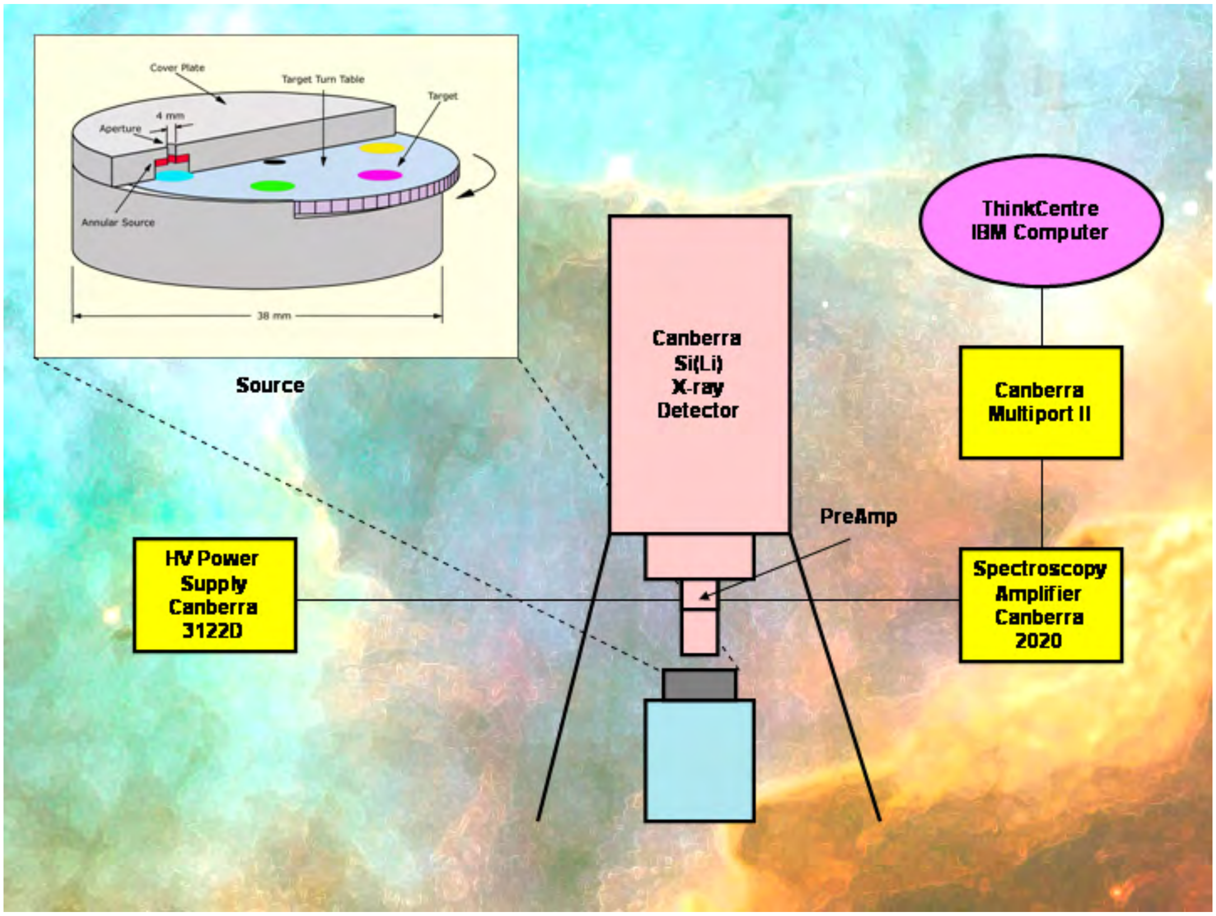
WARNINGS

- Make sure that the detector was cold for 3 hours or more prior to HV application
- No HV without preamp power on ! Internal FET can die !
- HV turn on and off very slowly. No abrupt switch off !
- Remove protection cap from detector before starting measuring
- Never touch the ultra thin Be window of the detector

- Watch count rates ! Too high count rate can latch up preamp or deteriorate resolution
- Be careful with the ^{241}Am source, it is strong and under special regulations (transuranium element)
- No measurement of spectra during LN_2 filling

D. Information Regarding the Experimental Setup

Schematic Drawing, Block Diagram



Legend:

Detector :	Si(Li)–Semiconductor Detector Canberra EURISYS Mod. ESLX 30-150-ER No 0251 (year 2002) Crystal dimensions: useful surface 30 mm^2; external diameter 6.2 mm; length 5.5 mm; volume 0.15 cm^3; dead layer $\simeq$ 0.2 μm/Si; distance from cap 3 mm Energy resolution 150 eV at 5.9 keV
Bias :	Canberra bias voltage supply Canberra # 2122 D, neg. -500 Volts
Preamp :	Preamplifier Canberra # PSC 854, "‘reset" - type
SpecAmp :	Main spectroscopy amplifier CANBERRA # 2020, (important for "‘reset" - type preamp)

MCA,ADC :	Canberra multiport II (ADC and MCA)
Computer :	Computer IBM think centre; CANBERRA MCA software Genie 2000 (dongle !)

The preamplifier works with a so-called optical feedback, which means that most of the time the feedback resistor is nearly infinite, but when the output signal reaches a certain amplitude, it is switched to a low value by a flash of a light emitting diode. The main amplifier has to deal with these strong negative switching signals and only a few have this property, the Canberra 2020 being one of them. The advantage is better resolution and higher count rates. The proper settings for pole zero cancellation and base line recovery are very important.

Gamma source for the excitation of characteristic X rays

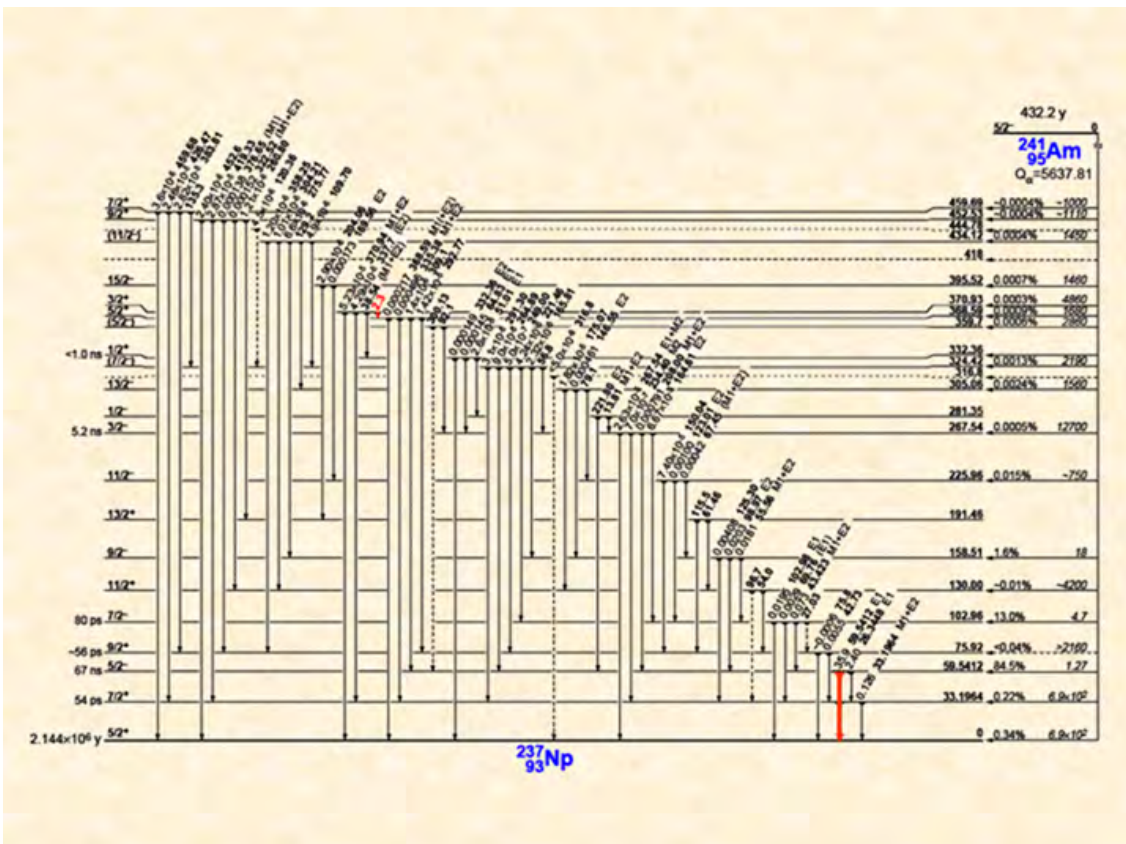


Figure 15: Decay scheme of ^{241}Am from [6].

In the block scheme the scheme of the so-called variable X-ray source is also given. The ^{241}Am source is encapsulated in ring form and radiates downwards onto samples of material. This americium-241 decay yields, besides α-particles, a strong 59.5 keV γ-transition which is used for the excitation. The alphas cannot escape from the source which is shielded in the upwards direction by tungsten to prevent the direct beam of 59.5 keV radiation being 'seen' by the detector. The produced characteristic X-rays are emitted upwards through the opening of the ring and reach the detector. The source assembly includes a target revolver with six different samples covering the range 29 < Z < 65 (copper Cu, rubidium Rb, molybdenum Mo, silver Ag, barium Ba, terbium Tb). For other materials the source is disassembled carefully and set on top of material to be investigated.

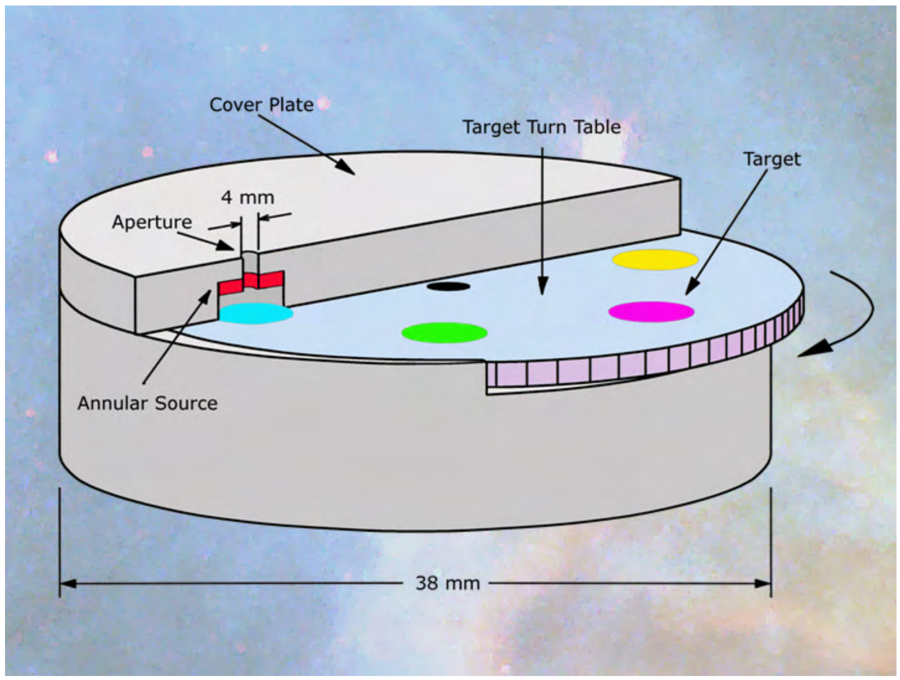


Figure 16: Variable X-ray source, an encapsulated ^{241}Am ring source radiating on six elementary targets (Cu, Rb, Mo, Ag, Ba, Tb) on a revolving disk to obtain characteristic X-rays for elements in the range 29 < Z < 65.

Calibration sources for energy calibration of the detector

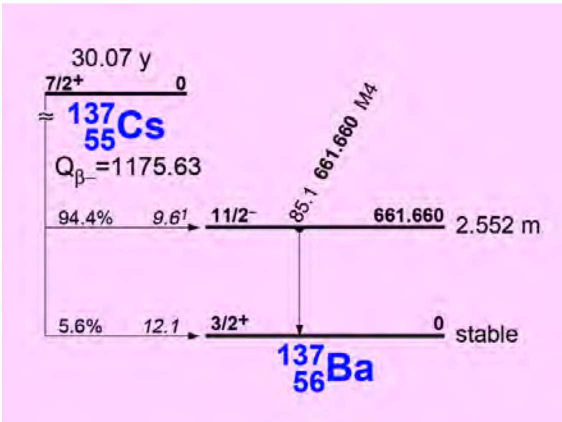
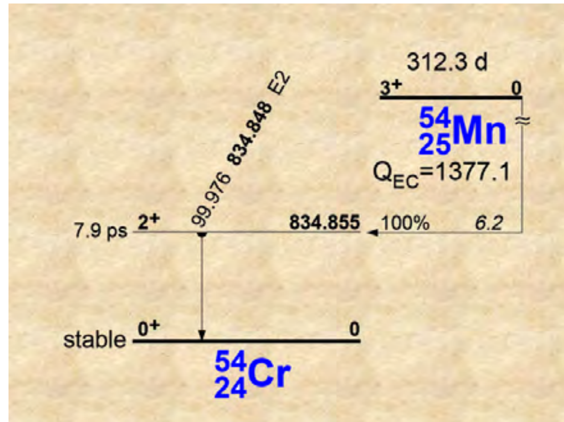


Figure 17: Decay scheme of ^{137}Cs from [6].



Decay scheme of ^{54}Mn from [6].

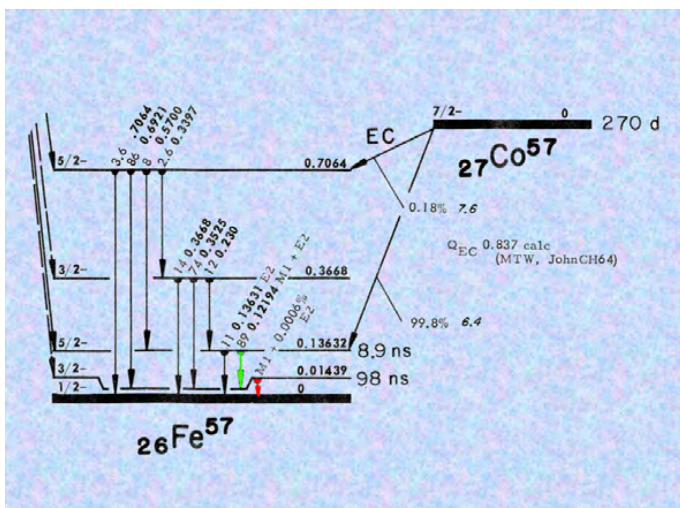


Figure 18: Decay scheme of ^{57}Co from [6].

The Cs-137 source delivers characteristic X rays of barium ($K_{\alpha_1} = 31.8$ keV, $K_{\beta_1} = 36.38$ keV), the Mn-54 source those of chromium ($K_{\alpha_1} = 5.4$ keV), and the Co-57 source those of iron ($K_{\alpha_1} = 6.4$ keV) in addition to the 14.4 keV Mößbauer-line.

X-ray tube for the excitation of characteristic X-rays

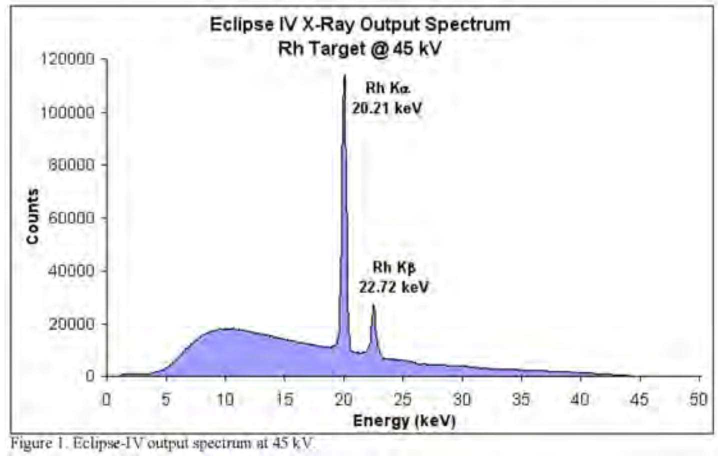


Figure 19: X-ray tube Eclipse IV. Emission spectrum of an Eclipse IV tube.

The Eclipse IV X-ray tube from Oxford Instruments, resp. AmpTek is designed as a compact mobile X-ray source for material analysis. The maximum voltage applied is 45 kV, and it can be set in a range from 10 to 45 kV. The X-ray tube is pressurized under sulfur-hexafluoride to isolate the high voltage. The anode current can also be varied in the range 0 – 50 μA by varying the current through the tungsten cathode. The anode material is rhodium, the exit window is made from a beryllium foil and the tube is operating in transmission yielding the spectrum shown in Fig. 1b. Here the characteristic rhodium lines sit on a broad Bremsstrahlungs-spectrum ranging from about 4 to 40 keV. The radiation is emitted in a cone with an angle of 130 degrees, therefore a collimator is indispensable.

E. Data Tables on the Periodic System and X-ray transitions

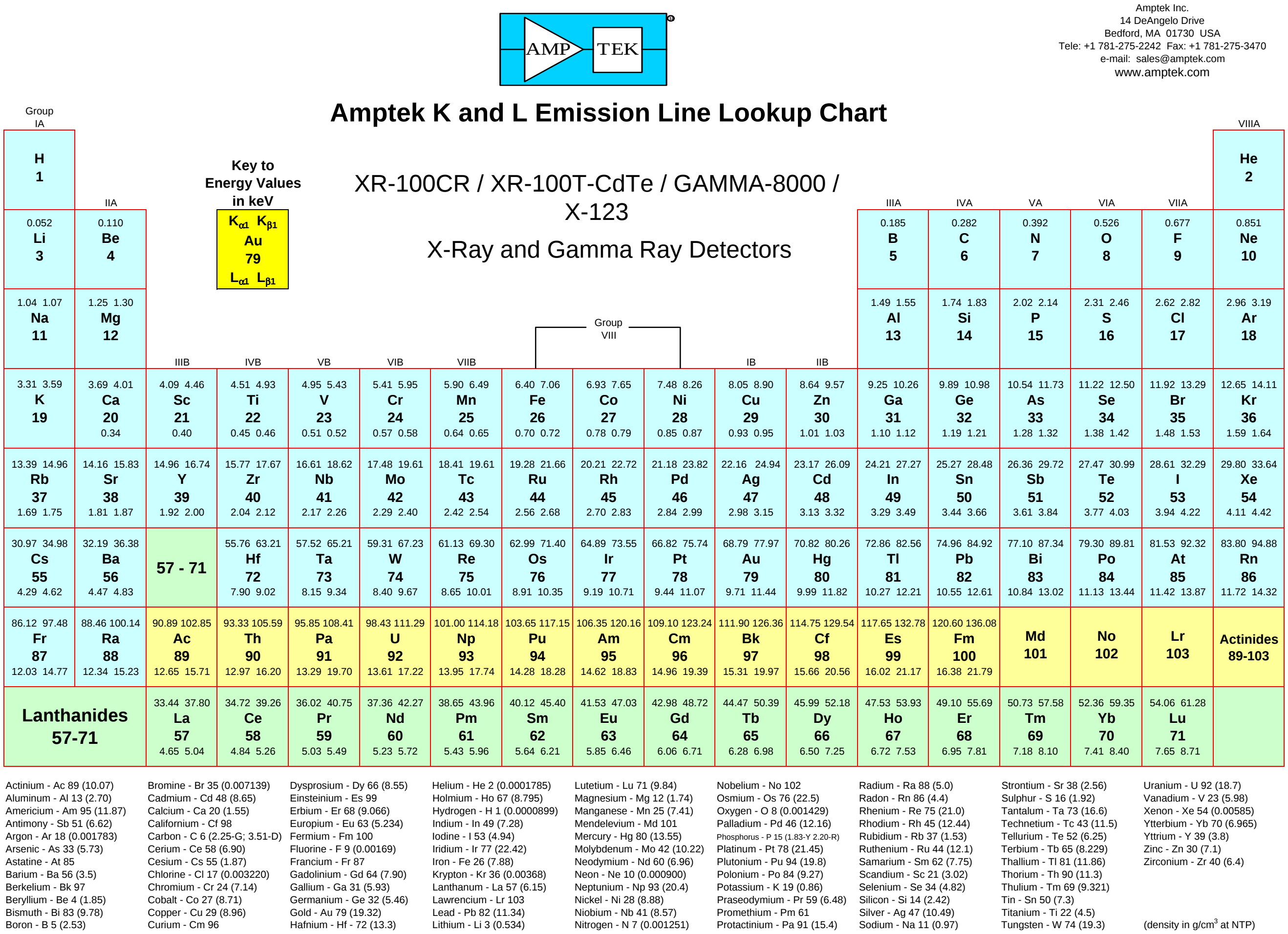


Figure 20: X-ray chart from Amptek

Z	Element	$K_{\alpha 2}$	$K_{\alpha 1}$	$K_{\beta 1}$	$K_{\beta 2}$	ω_K
10	Ne	848.61	848.61	857.89		
11	Na	1040.98	1040.98	1071.12		
12	Mg	1253.437	1253.688	1302.20		
13	Al	1486.295	1486.708	1557.57		
14	Si	1739.394	1739.985	1835.96		
15	P	2012.70	2013.68*	2139.1		
16	S	2306.700	2307.885	2464.07		0.049
17	Cl	2620.846	2622.44	2815.60		0.065
18	Ar	2955.566	2957.682	3190.49		0.081
19	K	3311.1956	3313.948	3589.63		0.099
20	Ca	3688.128	3691.719	4012.76		0.120
21	Sc	4085.9526	4090.735	4460.44		0.143
22	Ti	4504.9201	4510.899	4931.827		0.170
23	V	4944.671	4952.216	5427.320		0.198
24	Cr	5405.5384	5414.805	5946.823		0.229
25	Mn	5887.6859	5898.8010	6490.585		0.261
26	Fe	6391.0264	6404.0062	7058.175		0.293
27	Co	6915.5380	6930.3780	7649.445		0.327
28	Ni	7461.0343	7478.2521	8264.775		0.359
29	Cu	8027.8416	8047.8227	8905.413		0.393
30	Zn	8615.823	8638.906	9572.03	9658.05	0.43
31	Ga	9224.835	9251.674	10264.19	10366.42	0.46
32	Ge	9855.42	9886.52	10982.19	11100.97	0.49
33	As	10507.50	10543.2674	11725.73	11864.34	0.52
34	Se	11181.53	11222.52	12496.03	12652.29	0.55
35	Br	11877.75	11924.36	13291.56	13469.60	0.58
36	Kr	12595.424	12648.002	14112.815	14315.0	0.60
37	Rb	13335.88	13395.49	14961.42	15185.54	0.63
38	Sr	14098.03	14165.20	15835.89	16084.68	0.65
39	Y	14882.94	14958.54	16738.08	17015.6	0.67
40	Zr	15690.645	15774.914	17666.578	17970.3	0.70
41	Nb	16521.28	16615.16	18622.68	18952.9	0.71
42	Mo	17374.29	17479.372	19608.34	19965.27	0.73
43	Tc	18250.9*	18367.2*	20619.0*	21005.4*	0.75
44	Ru	19150.49	19279.16	21656.75	22074.3	0.77
45	Rh	20073.67	20216.12	22723.59	23173.0	0.78
46	Pd	21020.15	21177.08	23818.69	24299.4	0.79
47	Ag	21990.30	22162.917	24942.42	25456.71	0.80
48	Cd	22984.05	23173.98	26095.44	26644.07	0.82
49	In	24002.03	24209.75	27275.55	27861.20	0.83

Figure 21: Table of the X-ray K-series energies from NIST

Z	Element	$K_{\alpha 2}$	$K_{\alpha 1}$	$K_{\beta 1}$	$K_{\beta 2}$	ω_K
50	Sn	25044.04	25271.36	28486.26	29109.64	0.84
51	Sb	26110.78	26358.86	29725.53	30389.84	0.85
52	Te	27201.99	27472.57	30995.97	31700.76	0.86
53	In	28317.52	28612.32	32295.05	33041.7	0.86
54	Xe	29458.250	29778.78	33624.23	34414.7*	0.87
55	Cs	30625.40	30973.13	34987.3	35821.7	0.88
56	Ba	31816.615	32193.262	36377.445	37257.7	0.88
57	La	33034.38	33442.12	37801.45	38730.3	0.89
58	Ce	34279.28	34720.00	39257.77	40233.1	0.89
59	Pr	35550.59	36026.71	40748.67	41774.4	0.90
60	Nd	36847.502	37360.739	42270.90	43335*	0.90
61	Pm	38171.55	38725.11	43825.5*	44937*	0.91
62	Sm	39523.39	40118.481	45413.0	46575	0.91
63	Eu	40902.33	41452.63	47038.4	48256.6	0.92
64	Gd	42309.30	42996.72	48696.9	49960.6	0.92
65	Tb	43744.62	44482.75	50382.9	51724*	0.92
66	Dy	45208.27	45998.94	52119.7	53510*	0.93
67	Ho	46699.98	47547.10	53877.1	55325*	0.93
68	Er	48221.61	49127.24	55673.52	57214*	0.93
69	Tm	49772.67	50741.475	57508.76	59095*	0.93
70	Yb	51354.60	52389.48	59367.1	60985*	0.94

Z	Element	$K_{\alpha 2}$	$K_{\alpha 1}$	$K_{\beta 1}$	$K_{\beta 2}$	ω_K
71	Lu	52965.57	54070.39	61283*	62967*	0.94
72	Hf	54612.0#	55790.8#	63234*	64980*	0.94
73	Ta	56277.6	57533.2	65223.3	67013.5#	0.94
74	W	57981.77	59318.847	67245.0	69101.3	0.94
75	Re	59718.57	61141.00	69310.3	71232.1	0.95
76	Os	61487.27	63001.07	71413.9	73403.2	0.95
77	Ir	63287.29	64896.2	73561.7	75619.3	0.95
78	Pt	65123.3	66832.9	75749.1	77878.3	0.95
79	Au	66990.73	68804.5	77979.80	80186.2	0.95
80	Hg	68895.1	70819.5	80254.2	82545	0.95
81	Tl	70832.5	72872.5	82576.7	84948.5	0.95
82	Pb	72805.42	74970.11	84939.08	87366.9	0.96
83	Bi	74816.21	77109.2	87344.1	89861.8	0.96
84	Po	76864.4*	79292.9*	89797*	92400*	0.96
85	At	78944*	81514*	92304*	94991*	0.96
86	Rn	81066*	83783*	94867*	97639*	0.96
87	Fr	83232*	86105*	97478*	100326*	0.96
88	Ra	85436	88476	100136	103045	0.96
89	Ac	87676*	90884.8*	102847*	105868*	0.96
90	Th	89957.04	93347.38	105601.51	108718	0.96
91	Pa	92283.4	95866.4	108417.3	111625*	0.96
92	U-238	94650.84	98431.58	111295.08	114607#	0.96

Figure 22: Table of the X-ray K-series energies from NIST

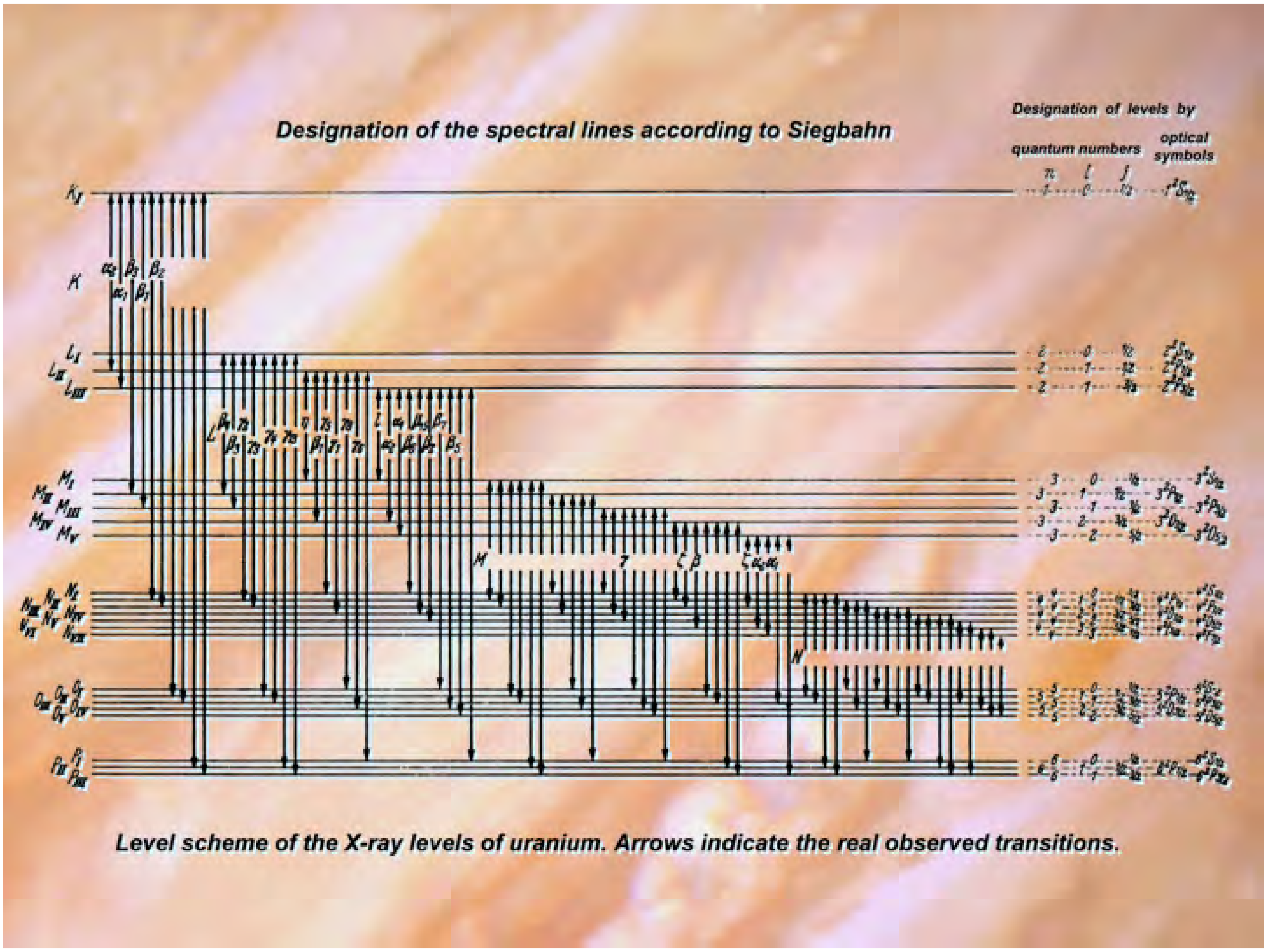


Figure 23: X-ray transitions for the L-series in uranium. The designation of the observed lines is according to Siegbahn.

Most intense L-X-ray lines of some heavy elements

Element	Tantalum	Tungsten	Platinum	Gold	Mercury	Lead	Thorium	Uranium
Symbol (Z)	Ta (73)	W (74)	Pt (78)	Au (79)	Hg (80)	Pb (82)	Th (90)	U (92)
Line*	**Energies in electron-volts [eV]**							
α_1 (L_3M_5)	8146.17	8398.242	9442.39	9713.44	9988.91	10551.6	12967.937	13614.87
α_2 (L_3M_4)	8087.93	8335.34	9361.96	9628.05	9897.68	10449.59	12809.49	13438.97
β_1 (L_2M_4)	9343.19	9672.575	11070.84	11442.45	11822.7	12613.8	16201.556	17220.15
β_2 (L_3N_5)	9651.89	9964.133	11250.66	11584.75	11924.2	12622.8	16024.6	16428.44
β_3 (L_1M_3)	9487.62	9818.91	11230.89	11610.5	11995.4	12793.4	16423.855	17455.17
β_4 (L_1M_2)	9212.47	9525.23	10854.41	11204.81	11563.1	12305.9	15639.54	16575.51
γ_1 (L_2N_4)	10895	11286	12942	13381	139830	14764	18978	20167
γ_2 (L_1N_2)	11217	11610	13270	13709	14162.3	15218.2	19302	20484
γ_3 (L_1N_3)	11277.68	11680.49	13361.5	13809.1	14264.8	15218.2	19503	20712.95
γ_5 (L_2N_1)	10570	10948	12552	12974	13410	14307	18364	19506
l (L_3M_1)	7173.2	7387.8	8268.2	8494.03	8721.32	9184.56	11118.06	11618.41
η (L_2M_1)	8428.09	8724.42	9975.2	10308.41	10651.4	11349.4	14510.327	15399.81

* Siegbahn notation (modern notation)

F. Discussion of Results

1. Discuss the energy spectra of the ^{57}Co, ^{137}Cs, ^{54}Mn and ^{241}Am sources.
2. Construct the energy calibration.
3. Explain the energy spectra of the different elements.
4. Plot the energy of the X-rays as a function of nuclear charge number Z.
5. Plot the energy resolution as a function of energy.
6. Plot the detector response function as a function of energy.
7. Check Moseley's law and compute the shielding constants.

8. Identify the unknown materials based on Moseley's law.

9. Explain the obtained X-ray L-spectra

G. Example Questions

1. How are X-rays created ?

2. What is the difference between X-rays and γ-rays ?

3. What is the Auger effect ?

4. What is a fluorescence yield ω_K ?

5. Explain Moseley's formula.

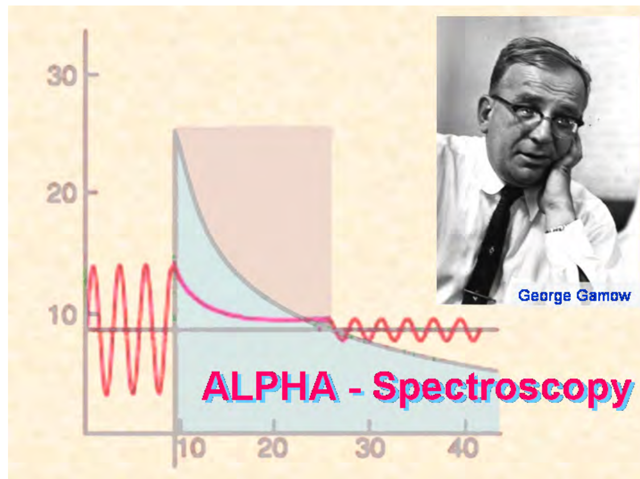
6. How is the material analysis performed ?

7. Why does the silicon-detector have to be cooled ?

8. Which other methods of X-ray spectroscopy are known ?

II . C. NUCLEAR PHYSICS EXPERIMENTS

6 Alpha Spectroscopy



Location: room Jordan 308

A. Short description

α-spectroscopy is related to the α-decay of nuclei which is described by the Gamow theory and the tunneling effect. These effects are also important for fusion reactions, for example in the nucleosynthesis of the chemical elements. Using a modern high resolution silicon detector (resolution 11 keV at about 5 MeV) one can explore the fine structure of specific α decays or determine the line energy of unknown samples. Through this the identity of the unknown nucide can be determined. Furthermore the relative intensities within an alpha-spectrum can be measured as well.

In this experiment a small vacuum chamber is evacuated by an oilfree forepump and it houses the detector, the optional foil and the α emitter. To reach high resolution the α sources used have to be very thin.

Energy loss in matter is used to determine the thickness of foils and thin layers, an important application of particle spectroscopy. The energy loss of α particles in matter is described by the Bethe-formula and more recently simulated by the TRIM or the SRIM codes.