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X-ray fluorescence and Moseley's law (Item No.: P2524715)



Binding energy, photoelectric effect, structure of electron shells, characteristic X-rays, gamma-spectrometry, X-ray spectrometry

Overview

Short description

Principle

Photons originating from nuclear transitions are called γ -quanta and photons originating from electron transitions of high energy are called X-rays. If matter is irradiated with photons of high energy, part of them are absorbed by electrons transferring the energy and momentum of the photon to the electron which is called photoelectric effect. If the energy is sufficient, also the innermost i.e. strongest bound electrons of an atom can be removed from the atom by this process. The states of the missing electrons are filled with other electrons under emission of characteristic X-rays or Auger-electrons. The emission of characteristic X-rays caused by irradiation with photons of high energy is called X-ray fluorescence. The binding energy of the innermost electrons increases with atomic number and so does the energy of the characteristic X-rays which is measured in this experiment. The energy resolution of a scintillation counter is sufficient for such examinations. The dependence of X-ray energy on atomic number was examined by Moseley.



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Equipment

Position No.	Material	Order No.	Quantity
1	Radioactive source Am-241, 370 kBq	09090-11	1
2	Radioactive source Cs-137, 37 kBq	09096-01	1
3	Multichannel analyser	13727-99	1
4	Gamma detector	09101-00	1
5	PHYWE High Precision Power Supply 1.5 kV DC	09107-99	1
6	High-voltage connecting cable	09101-10	1
7	measure Software multi channel analyser	14452-61	1
8	Tin-II chloride 250 g	31991-25	1
9	Silver foil, 150 x150 x 0.1 mm, 25 g	31839-04	1
10	Absorption material, lead	09029-01	1
11	Plastic sack, flat, DIN A5, 100pc	46444-01	1
12	Alligator clips, bare, 10 pcs	07274-03	1
13	Support rod, stainless steel, I = 250 mm, d = 10 mm	02031-00	3
14	Support base DEMO	02007-55	1
15	Universal clamp	37715-00	1
16	Right angle clamp expert	02054-00	3
17	Support rod with hole, stainless steel, 10 cm	02036-01	1
18	Spring balance holder	03065-20	1
19	Barium sulphate 500 g	30035-50	1
20	lodine resublimed 25 g	30093-04	1
21	Screened cable, BNC, l = 750 mm	07542-11	1

Tasks

- 1. Perform an energy calibration of the setup using the $59.5 \, keV$ line of ^{241}Am and the $59.5 \, keV$ keV line of ^{137}Cs .
- 2. Record spectra of the fluorescence radiation exited with the radiation of the $^{241}\mathrm{Am}$ source for different specimen.
- 3. Plot the energy of the fluorescence peak vs. the function $(Z-1)^2$ of the specimen'snuclear mass number Z and calculate the Rydberg constant from the slope of theobtained graph.

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Set-up and Procedure

Set up the experiment as shown in Fig. 1. Before turning on the operating unit for the scintillation counter, connect the high voltage cable correctly to operating unit and photomultiplier and read the instructions in the manual of the gamma-detector. Set the voltage of the operating unit to $1.0\,\mathrm{kV}$. Connect the MCA to the computer's USB port and start the "measure" program. Select the Gauge "Multi Channel Analyzer" and you will receive the start window, as shown in Fig. 2.

1. First adjust the detector: Start with the ^{241}Am source. Select "Spectra recording" (see Fig. 2), click the "Continue" button and in the spectra recording window (see Fig. 3) set the "Gain" to "Level 4" and the "Offset" to 1% and choose "Channel number" as x-Data. Place the source in a distance to the detector such that the counting rate is slightly below 1000 cts/s. Adjust the voltage on the detector's operating unit so that the 59.5 keV peak moves to the right end of the spectrum (around channel 3500). Leave this setting unchanged throughout the measurement – for low drift turn on the detector some time before measurement. "Cancel" the measurement.

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Please se	lect the measu recording	rement mod	de	
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Now calibrate the MCA so that the corresponding energy of each channel is known: Start the MCA gauge of "measure" again and select "Settings and Calibration" (see Fig. 2). The window shown in Fig.4 will appear – click on the "Calibrate" button. Set the "Gain" to "Level 4", the "Offset" to 1% and select "2-point calibration". Move one bar to the $59.5 \, keV$ peak and type the energy value in the appropriate field, as shown in Fig. 5. Then remove the ^{241}Am and bring the ^{137}Cs in direct vicinity to the detector. Click on the "Clear diagram" button and move the other bar to the then appearing $32.2 \, keV$ peak of the ^{137}Cs and type the energy value in the appropriate field and finally click on the "Apply" button and then the "Save" button of the window seen in Fig. 4 and enter a name for your calibration.

Detector calibration		
Detector / Calibration:	User calibration	•
Comment:	none	
Mode:	2-point calibration	
Gain:	Level 1, Offset 0%	
Equation:	1 <channel> keV + 0 keV</channel>	
Calibrate	Save Delete	
Device information		
Port U	SB	
Device version: 1.	02-3TEST	
Perform hardware	adjustment	



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2. Now choose the program part "Spectra recording" again with "Gain" "Level 4" and 1% "Offset". Put 3 cm of lead shielding between the $340 \text{ kBq}^{241} \text{ Am}$ source and the detector with the source close to the detector. Check the spectrum that gets recorded now and the counting rate – the detector should "see" as few of the source's radiation as possible – the presence of the source shouldn't increase the background rate a lot. Then put the fluorescent specimen in the vicinity of the detector so that it is exposed to the source's radiation and so that the fluorescence radiation can reach the detector. Move it until the counting rate gets maximal. The counting rate should be distinctly higher with specimen than without. Reset the spectrum and in the now recorded spectrum the fluorescence peak should be clearly visible and much lower than the 60 keV peak. Save the recorded spectra with the "Accept data" button. If the specimen is powder and is packed in a plastic bottle, simply the whole package can be used. Glass packages absorb too strongly and show a broad fluorescence spectrum themselves – specimen in glass bottles have to be removed from the bottle and may be put into e.g. plastic bags. Metal pieces need no packaging.



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Theory and evaluation

Fig. 6 shows the decay schemes of the used nuclides. The proportions of the energy scale are not displayed correctly and the term scheme of ^{237}Np is strongly simplified – in the experiment only the $59.5 \, keV$ of ^{237}Np is of importance. It is to be kept in mind, that the exited states of the daughter nuclides can also disintegrate by inner conversion in the case of ^{137}Ba leading to a strong $32 \, keV$ X-ray line.



In the recorded spectra use "Display options" to change the displayed area to 10 to $60 \, keV$ and on the "Channels" chart change the "Interpolation" from "Bars" to "Straight lines". Then use the "Smooth" tool to generate a new diagram where the position of the fluorescence peak is more clearly visible. With the "Survey" function read out the corresponding energy of the peak. Fig. 7 shows results combined into one graph with "Measurement" > "Assume channel".



Create a measurement with "Measurement" > "Enter data manually..." as seen in Fig. 8. Then change the x-channel with "Analysis" > "Channel modification..." to $(Z-1)^2$ as seen in Fig. 9. The number of digits beyond the point can be changed with the "Information" button. The slope of the graph can be evaluated with the "Regression" tool.

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Number of channels.	1 - Nur	nber of value	as: 5
Data	1.000.00000.000		
Fitle	Symbol	Unit	Digits
Nomic number	Z		0
Fitte	Symbol	Unit	Digits
^p eak energy	E	keV	1

7 := Atomic number	1		
		Calculate	
[0#]	-	Cancel	
		Help	
peration			
• f := (2-1) ²	<u>.</u>		
<u>differentiate</u>			
integrate			
grogressive average value			
estination channel			
add new y-channel	7.8-0	7.8-	
invervine	1/08		
Manie ummer	(2-1)*		
into new measurement	Symbol:	(Z-1)*	
 as x-channel 	Unit	1	
as y-channel	our		

The fluorescence peak that dominates here is from K_{α} radiation. The corresponding energy is the energy difference between the state of the innermost electron in the atom, the lowest energy state, and the energy of an electron state one shell above that. K_{α} radiation is thus the radiation emitted when an innermost electron is lost by photo effect and its state is filled up with an electron of the next shell and the energy difference is emitted as photon. Moseley's law now states that this energy is connected to the ionization energy of atomar hydrogen, the Rydberg constant R_{∞} , in a simple manner:

$$E_{K_lpha}=rac{3}{4}hR_\infty\cdot(Z-1)^2$$

with atomic number Z and Planck's quantum h.

ice

Fig. 10 shows that this approximation is astonishingly well valid – though one would expect strong deviations because of the complicated shell structure of heavy atoms – e.g. Cer is a lanthanoid and has an electron in the f-shell and Barium belongs to the second main column of the table of elements and has none.

The slope in Fig. 10 reads $0.0107\,{
m keV}$ and with $h=6.626\cdot10^{-34}\,{
m Js}=4.136\cdot10^{-18}\,{
m keVs}$ is then

$$R_\infty\,{=}\,(3.45\pm0.3)\,{\cdot}\,10^{15}\,1/{
m s}$$

Literature values for
$$R_\infty$$
 :

$$R_\infty/c_0 = 1.097 \cdot 10^7 \; 1/{
m s}$$

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 $egin{aligned} R_\infty &= 3.290 \cdot 10^{15} \, \mathrm{1/s} \ h \cdot R_\infty &= 13.6 \, \mathrm{eV} \ h \cdot R_\infty &= 2.178 \cdot 10^{-18} \, \mathrm{J} \end{aligned}$



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