Qualitative X-ray fluorescence analysis of powder samples

Physics | Modern Physics | Production & use of X-rays

Difficulty level	Group size	Preparation time	Execution time
hard	2	45+ minutes	45+ minutes

This content can also be found online at:

http://localhost:1337/c/5f7dd0072c24d60003b46120

General information

Application

Setup

Most applications of X rays are based on their ability to pass through matter. Since this ability is dependent on the density of the matter, imaging of the interior of objects and even peaple becomes possible. This has wide usage in fields such as medicine or security.

PHYWE
Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
www.phywe.de

Other information (1/2)

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Prior knowledge

The prior knowledge for this experiment is found in the Theory section.

Main principle

Various powder samples are subjected to polychromatic X-rays. The energy of the resulting fluorescence radiation is analysed with the aid of a semiconductor detector and a multichannel analyser. The energy of the corresponding characteristic X-ray fluorescence lines is determined. The elements of the samples are identified by comparing the line energies with the corresponding table values.

Other information (2/2)

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Learning objective

The goal of this experiment is to get to investigate the spectra of fluorescence radiation.

Tasks

1. Calibrate the semiconductor energy detector with the aid of the characteristic radiation of the molybdenum X-ray tube.
2. Record the fluorescence spectra that are produced by the samples.
3. Determine the energy values of the corresponding fluorescence lines and compare them with the literature values in order to identify the powder components.

PHYWE
Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
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Safety Instructions

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When handling chemicals, you should wear suitable protective gloves, safety goggles, and suitable clothing. Please refer to the appendix for detailed safety instructions.

Theory

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When X-rays interact with matter, they lose energy. In the range of energy that is available during this experiment, the photoelectric effect plays the most important role. This means that on one of the lower shells of the atom, an electron is ejected due to the absorbed photon energy. The now free space is taken by an electron from one of the higher shells. The energy that is produced during this process can be used for the ejection of another electron from one of the higher shells (Auger effect) or for the generation of a photon (fluorescence radiation). Since the energy of the energy levels that are involved in this process is atom-specific, the type of the emitting atom can be determined based on the energy of the fluorescence radiation.

In order to determine the type of atom, the experimental energy values are then compared to the corresponding table values (e.g. “Handbook of Chemistry and Physics”- CRC-Press, Inc. USA). During the assignment of the fluorescence lines, it must be taken into consideration that the relaxations that follow the primary ionisation process can only take place if they fulfil the quantum-mechanical selection rules $\Delta \mathrm{j} = 0, \pm 1$ and $\Delta \mathrm{l} = \pm 1$ (j = total angular momentum, l = orbital angular momentum).

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Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
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info@phywe.de
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Equipment

Position	Material	Item No.	Quantity
1	XR 4.0 expert unit, 35 kV	09057-99	1
2	XR 4.0 X-ray goniometer	09057-10	1
3	XR4 X-ray Plug-in Cu tube	09057-51	1
4	XR 4.0 X-ray material upgrade set	09165-88	1
5	XR 4.0 X-ray Chemical set for edge absorption	09056-07	1

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Robert-Bosch-Breite 10
37079 Göttingen
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info@phywe.de
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Setup and Procedure

Setup (1/2)

- Screw the adapter ring onto the inlet tube of the energy detector and connect the signal and supply cables to the corresponding ports of the detector with the aid of the right-angle plugs.
- Connect the signal and supply cables to the corresponding ports in the experiment chamber of the X-ray unit. In Figure 1, the port for the signal cable is marked in red and the port for the supply cable is marked in green. Connect the external X RED ports of the x-ray unit (see Fig. 2) to the multi-channel analyser (MCA). Connect the signal cable to the “Input” port and the supply cable to the “X-Ray Energy Det.” port of the MCA.

Fig. 1: Connectors in the experiment chamber

Fig. 2: Connection of the multi-channel analyser

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37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
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Setup (2/2)

- Secure the energy detector in the holder of the swivel arm of the goniometer. Lay the two cables with sufficient length so that the goniometer can be swivelled freely over the entire range.
- Connect the multi-channel analyser and computer with the aid of the USB cable.

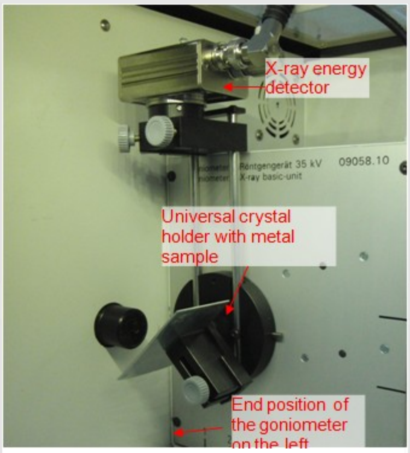

Fig. 3: Goniometer set-up

Procedure (1/4)

- Bring the goniometer block and the detector to their respective end positions on the right.
- Insert the tube with the 1mm-aperture into the exit tube of the x-ray tube.
- With the X-ray unit switched on and the door locked, bring the detector to the 0° position. Then, shift the detector by some tenths degree out of the zero position in order to reduce the total rate.
- Operating data of the x-ray tube: Select an anode voltage U_A = 25 kV and an anode current I_A = 0.02 mA and confirm these values by pressing the “Enter” button.
- Switch on the x-radiation

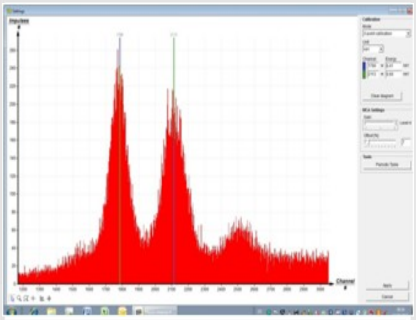

Fig. 4: calibration of the multi-channel analyser

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Fax: 0551 604 - 107
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Procedure (2/4)

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- In the MEASURE program, select “Multi channel analyser” under “Gauge”. Then, select “Settings and calibration”. After the “Calibrate” button has been clicked, a spectrum can be measured. The counting rate should be < 300 c/s. Energy calibration settings: - 2-point calibration, - Unit = keV, Gain = 2 – Set the offset so that low-energy noise signals will be suppressed (usually a few per cent are sufficient), See Fig 4.
- Measuring time: 5 minutes. Use the timer of the X-ray unit.
- Make the two coloured calibration lines congruent with the line centres of the two characteristic X-ray lines. The corresponding energy values (see e.g. P2544705) $\mathrm{E}(\mathrm{L_3M_5}/\mathrm{L_3M_4})$ = 8,41keV and $\mathrm{E}(\mathrm{L_2N_4})$ = 9,69 keV are entered into the corresponding fields, depending on the colour. (Note: Since a separation of the lines $\mathrm{L_3M_4}$ and $\mathrm{L_3M_5}$ Lines is not possible, the mean value of both lines is entered as the energy of the line).
- Name and save the calibration.

Procedure (3/4)

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Spectrum recording

- Insert the tube with the 2-mm-aperture.
- Bring the goniometer block and the detector to their respective end positions on the left. Bring the detector to the 90° position in the 1:2 coupling mode.
- Insert the metal sample with the universal crystal holder (sample at 45°).
- Operating data of the x-ray tube: Adjust an anode voltage $\mathrm{U_A}$ = 35 kV and an anode current so that the counting rate is $\leq$ 300 c/s.
- Measuring time: 3 minutes (use the timer of the X-ray unit).

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Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
Fax: 0551 604 - 107

info@phywe.de
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Procedure (4/4)

Evaluation of the measurement curves

- In order to determine the line energy, switch from the bar display to the curve display. To do so, click “Display options” and then “Interpolation and straight lines”.
- Extend the relevant line section with the aid of the zoom function
- Then, select the curve section with Open the window “Function fitting Then, select “Scaled normal distribution” and confirm.
- Find the line centroid of the normal distribution with “Peak analysis” or determine it with the function “Survey”

Evaluation

Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
www.phywe.de

Evaluation (1/4)

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Evaluation of the K_α and K_β fluorescence lines of the elements with an atomic number 30 < Z < 38

Figure 5 shows an overview of all of the fluorescence spectra of the elements with an atomic number 30 < Z <38. Within this group of elements, only the high-intensity characteristic K_α and K_β radiation can be clearly identified. The energy of the characteristic fluorescence radiation of the sample elements oxygen and sulphur of the oxide or sulphate compounds or of potassium are below the sensitivity limit of the energy detector and partly only have a very low intensity, which is why they cannot be identified in this experiment.

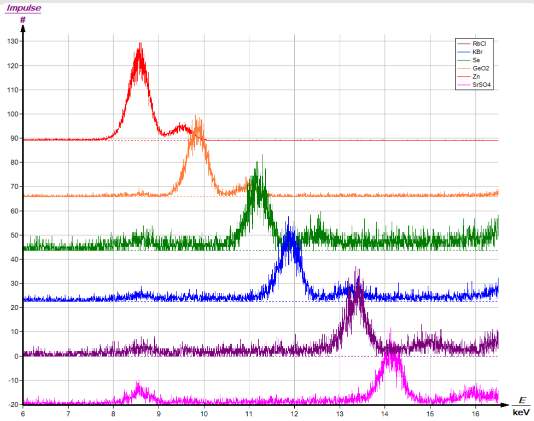

Fig. 5: Total representation of the K_α and K_β fluorescence lines of the elements with an atomic number 30 < Z < 38.

Evaluation (2/4)

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Figure 5 clearly shows that at the higher atomic numbers of the elements, the energy of the characteristic lines as well as the energetic spacing between the K_{α_1} and K_{β_2} lines increases.

Table 1 shows the evaluation of the spectra of Fig. 5.

Atomic number Z	Element	$E_{exp.}$ / keV	$E_{lit.}$ / keV	Transition
30	Zn	8.60 / 9.52	8.64 / 9.57	K_α / K_β
32	Ge	9.86 / 10.95	9.89 / 10.98	K_α / K_β
34	Se	11.19 / 12.46	11.22 / 12.49	K_α / K_β
35	Br	11.88 / 13.24	11.92 / 13.29	K_α / K_β
37	Rb	13.34 / 14.93	13.39 / 14.96	K_α / K_β
38	Sr	14.12 / 15.82	14.16 / 15.83	K_α / K_β

Table 1 shows the evaluation of the spectra of Fig. 5.

PHYWE
Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
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Evaluation (3/4)

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Evaluation of the L fluorescence lines of elements with a higher atomic number

Figure 6 shows the spectra of the powder samples with a higher atomic number. The ionisation energy of the K level of these elements is above the available excitation energy of the primary radiation of 35 keV max. This is why only the L fluorescence lines can be excited in this case.

Table 2 shows the evaluation of the spectra of Fig.6.

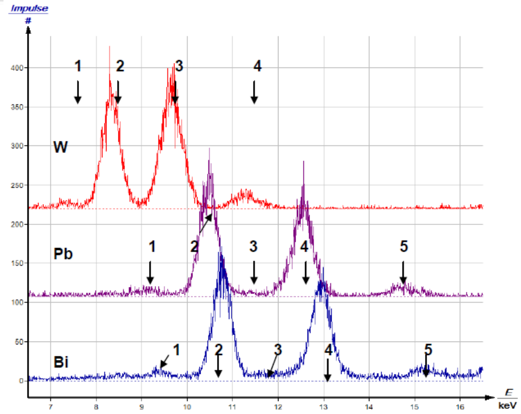

Fig. 6: L fluorescence lines of tungsten, lead and bismuth

Evaluation (4/4)

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The L_{β_1} lines, which are slightly widened, also include parts of the $L_{\beta_{2,3,4}}$ lines that are of a lower inten-sity. The L_{γ_1} line clearly becomes asymmetric towards the high-energy edge. This is due to the lower-intensity $L_{\gamma_{2,3}}$ lines with a slightly higher energy. The spectra of lead and bismuth include the additional low-intensity line (line 3). The fluorescence line at the end of the spectra (E = 17.36 keV) is caused by the primary molybdenum K_α radiation that is scattered at the samples.

Element	Line	$E_{exp.}$/ keV	$E_{lit.}$ / keV	Transition
W, Z = 74	1	7.40	7.39	$L_3M_1 - L_l$
	2	8.36	8.35 / 8.42	$L_3M_{4,5} - L_{\alpha 1,2}$
	3	9.67	9.69	$L_2M_4 - L_{\beta 1}$
	4	11.26	11.28	$L_2N_4 - L_{\gamma 1}$
Pb, Z = 82	1	9.15	9.18	$L_3M_1 - L_l$
	2	10.49	10.45 / 10.55	$L_3M_{4,5} - L_{\alpha 1,2}$
	3	11.30	11.35	$L_2M_1 - L_\eta$
	4	12.55	12.61	$L_2M_4 - L_{\beta 1}$
	5	14.73	14.76	$L_2N_4 - L_{\gamma 1}$
Bi, Z = 83	1	9.36	9.42	$L_3M_1 - L_l$
	2	10.77	10.73 / 10.83	$L_3M_{4,5} - L_{\alpha 1,2}$
	3	11.67	11.71	$L_2M_1 - L_\eta$
	4	12.95	13.02	$L_2M_4 - L_{\beta 1}$
	5	15.20	15.25	$L_2N_4 - L_{\gamma 1}$

Table 2: Elements of the powder samples with L fluorescence radiation

PHYWE
Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
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Appendix

Security Information (1/7)

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Hazard symbol, signal word	Hazard statements	Precautionary statements
Rubidium chloride (RbCl)		
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Germanium(IV) oxide (GeO_2)		
	H302: Harmful if swallowed H332: Harmful if inhaled	--

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Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
www.phywe.de

Security Information (2/7)

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Hazard symbol, signal word	Hazard statements	Precautionary statements
Silver (I) chloride (AgCl)		
	H400: Very toxic to aquatic life	P273: Avoid release to the environment.
Zinc		
-	--	--

Security Information (3/7)

PHYWE

Hazard symbol, signal word	Hazard statements	Precautionary statements
Selenium		
 	H301: Toxic if swalloed H331 Toxic if inhaled H373: Causes damage to organs through prolonged or repeated exposure H413: May cause long lasting harmful effects to aquatic life	--.

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Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
Fax: 0551 604 - 107

info@phywe.de
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Security Information (4/7)

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Hazard symbol, signal word	Hazard statements	Precautionary statements
Potassium bromide (KBr)		
	H315: Causes skin irritation H319: Causes serious eye irritation H335: May cause respiratory irritation	P261: Avoid breathing dust/fume/gas/mist/vapours/spray. P305 + P351 + P338: IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. .
Strontium sulphate ($SrSO_4$)		
-	--	--

Security Information (5/7)

PHYWE

Hazard symbol, signal word	Hazard statements	Precautionary statements
Lead (IV) oxide (PbO_2)		
	H272: May intensify fire; oxidiser H302: Harmful if swallowed H332: Harmful if inhaled H360: May damage fertility or the unborn child H373: Causes damage to organs through prolonged or repeated exposure H410: Very toxic to aquatic life with long lasting effects	P201: Obtain special instructions before use. P220: Keep/Store away from clothing/…/combustible materials. P273: Avoid release to the environment. P308 + P313: IF exposed or concerned: Get medical advice/attention.

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Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
Fax: 0551 604 - 107

info@phywe.de
www.phywe.de

Security Information (6/7)

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Hazard symbol, signal word	Hazard statements	Precautionary statements
Tungsten(IV) oxide (WO_2)		
	H335: May cause respiratory irritation	--

Security Information (7/7)

PHYWE

Hazard symbol, signal word	Hazard statements	Precautionary statements
Bismuth(III) oxide (Bi_2O_3)		
	H315: Causes skin irritation H319: Causes serious eye irritation H335: May cause respiratory irritation	P261: Avoid breathing dust/fume/gas/mist/vapours/spray. P305 + P351 + P338: IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

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37079 Göttingen
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www.phywe.de