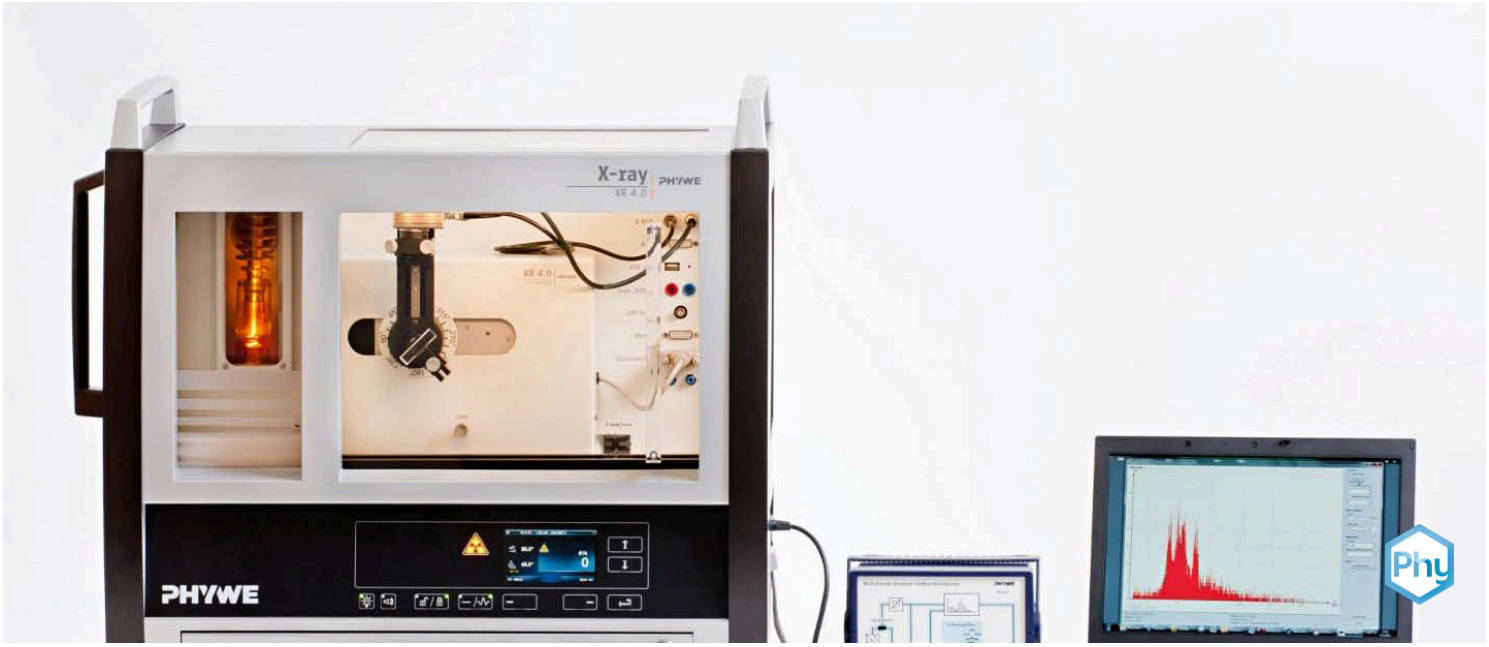
Qualitative X-ray fluorescence analysis of solutions



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/5f7f1b1c95a34b000395000a

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General information

Application

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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.

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Other information (1/2)

Prior knowledge

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

Main principle

Various saturated solutions are subjected to polychromatic X-rays. The energy of the resulting fluorescence radiation is analysed with the aid of a semiconductor detector and a multi-channel 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)

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 tungsten X-ray tube.
2. Record the fluorescence spectra of saturated potassium bromide and lead chloride solutions.
3. Determine the energy values of the correspond-ing fluorescence lines and comparison with the corresponding table values.

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Other information (2/2)

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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.

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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	Lead-II chloride, 500 g	31117-50	1
6	Potassium bromide, 100 g	30258-10	1

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Setup and Procedure

Setup (1/2)

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- 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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Setup (2/2)

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- 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.

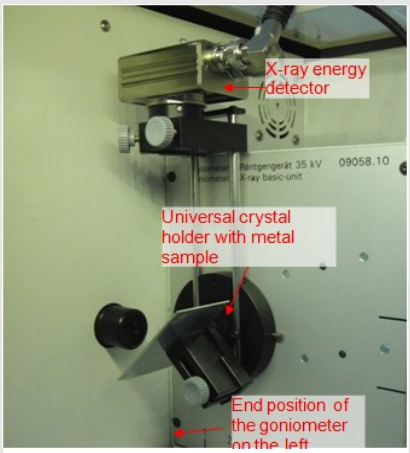

Fig. 3: Goniometer set-up

Procedure (1/4)

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- 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 tungsten 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

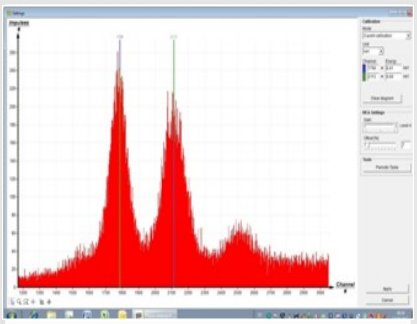

Fig. 4: calibration of the multi-channel analyser

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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) $\mathrm{E(L_3M_5/L_3M_4)}$ = 8,41keV and $\mathrm{E(L_2N_4)}$ = 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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Sample preparation

In order to produce high-intensity fluorescence lines, saturated solutions must be prepared. To do so, add approx. 0.5 g of $\mathrm{PbCl_2}$ into 50 ml of water, and approx. 32 g of KBr into another 50 ml of water. Then, fill the plastic cuvettes to 3/4 with the saturated solutions.

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 molybdenum X-ray tube: Adjust an anode voltage $\mathrm{U_A}$ = 35 kV and an anode current so that the counting rate is $\leq$ 200 c/s.
- Measuring time: 10 minutes (use the timer of the X-ray unit).

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Procedure (4/4)

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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”

Procedure (4/4)

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Evaluation

Evaluation (1/4)

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Fig. 5 shows the fluorescence spectrum of potas-sium bromide. Only the high-intensity characteristic K_α- and K_β-radiation of bromide (line 3 and 4) can be clearly identified. The energy of the characteristic fluorescence radiation of potassium is close to the sensitivity limit of the energy detectors, which is why it cannot be identified in this experiment. The lines 1 (E = 7.41 keV), 2 (E = 8.04 keV) and 5 (E = 16.35 keV), which are just visible, are caused by nickel and copper. The scattered primary radiation generates fluorescence radiation on the material components nickel and copper of the detector housing. This fluorescence radiation is also detected by the detector.

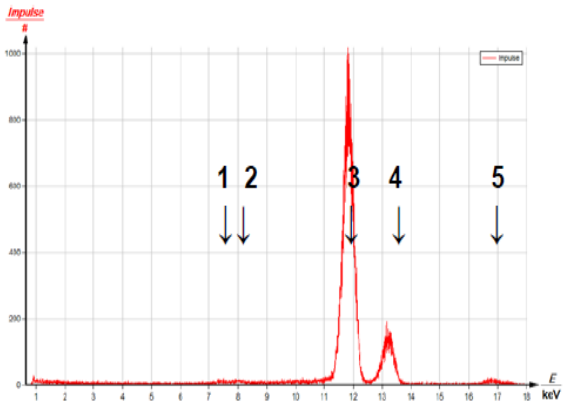

Fig. 5: K_α and K_β fluorescence lines of bromine.

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Evaluation (2/4)

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Fig. 6 shows the fluorescence spectrum of the lead chloride solution. The lines 1, 3, and 7 can be reassigned to the elements nickel, copper, and molybdenum. The other lines are part of the characteristic L-radiation of lead. The energy of the fluorescence radiation of chlorine is below the detection sensitivity of the detector.

Table 1 shows the evaluation of the spectra of Fig. 6.

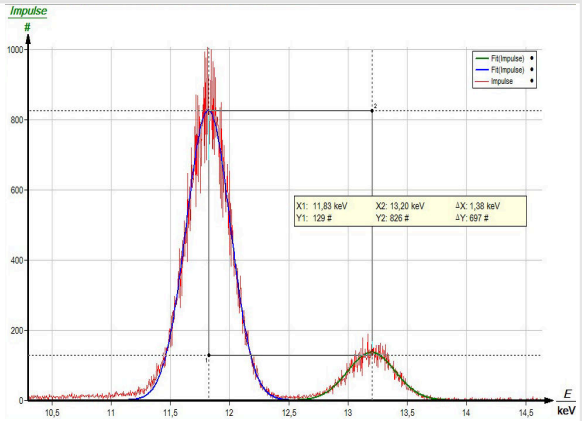

Fig. 6: Zoomed representation with a fitted normal distribution of the K_α- and K_β-line of bromide.

Evaluation (3/4)

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Atomic number Z	Element	Line	$E_{exp.}$ / keV	$E_{lit.}$ / keV	Transition
35	Br	3	11,83	11,92	$KL_{2,3}$ - K_α
		4	13,20	13,29	KM_3 - K_β
82	Pb	3	9,18	9,18	L_3M_1 - L_l
		4	10,52	10,45 / 10,55	$L_3M_{4,5}$ - $L_{\alpha 1,2}$
		5	12,58	12,61	L_2M_4 - $L_{\beta 1}$
		6	**14,84**	**14,76**	L_2N_4 - $L_{\gamma 1}$

Table 1: Assignment of the characteristic fluorescence lines of potassium bromide and lead chloride

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Evaluation (4/4)

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The L_{γ_1}-line (line 6) clearly becomes asymmetric towards the high-energy side. This is due to the low-intensity $L_{\gamma_{2,3}}$-lines of lead with a slightly higher energy, which cannot be separated clearly in this experiment.

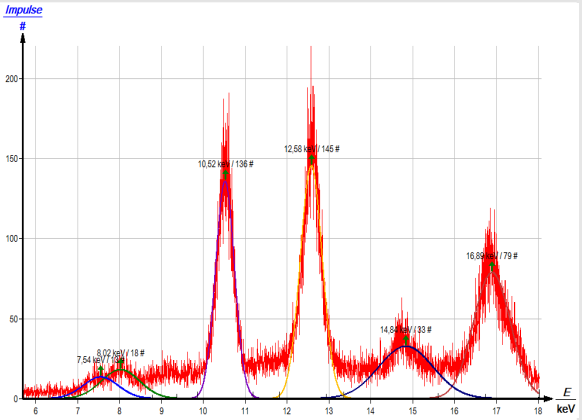

Fig. 7: L-fluorescence lines of lead.

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