Energy-dispersive measurements of K- and L-absorption edges

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

Difficulty level	Group size	Preparation time	Execution time
hard	1	10 minutes	10 minutes

This content can also be found online at:

http://localhost:1337/c/5fbd1610db521300037a902f

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

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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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37079 Göttingen

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Fax: 0551 604 - 107

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Other information (1/2)

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Prior knowledge

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

Main principle

Thin powder samples are subjected to polychromatic X-rays. The energy of the radiation that passes through the samples is analysed with the aid of a semiconductor detector and a multi-channel analyser. The energy of the corresponding absorption edges is determined, and the resulting Moseley diagrams are used to determine the Rydberg frequency, the screening constant, and the principal quantum numbers .

Other information (2/2)

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Learning objective

The goal of this experiment is to get to investigate the K- and L_absorbtion edges of different materials.

Tasks

1. Calibrate the semiconductor energy detector with the aid of the characteristic radiation of the calibration sample.
2. Record the energy spectra of the polychromatic X-rays that pass through the powder samples.
3. Determine the energy of the corresponding K- and L-absorption edges.
4. Determine the Rydberg frequency, screening constants, and principal quantum numbers with the aid of the resulting Moseley diagrams.

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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 (1/2)

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When X-rays interact with matter, they lose energy due to Compton scattering, pair production, and photoelectric effects. In the range of energy that is available during this experiment, the photoelectric effect plays the most important role. Figure 1 shows the schematic course of the transmission T as a function of the radiation energy E. At certain energy levels, the absorption (decrease in the transmission) increases drastically. In this case, the energy of the X-ray quanta is only sufficient for removing electrons from certain energy levels of the absorber atoms. As a result, the measurement of the absorption edges in X-ray spectra enables the determination of the energy levels of the inner atomic shells. If relativistic and spin-orbit coupling effects are neglected, the binding energy $\mathrm{E_n}$ of an electron on the nth shell of an atom can be described in an approximative manner by Bohr's atom model:

$$\mathrm{E_n} = \frac{\mathrm{m_e} e^4}{8\varepsilon_0^2 \mathrm{h}^2} (\mathrm{Z} - \sigma)^2 \frac{1}{\mathrm{n}^2} \quad (1)$$

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37079 Göttingen

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Theory (2/2)

With the introduction of the Rydberg frequency

$$\mathrm{R} = \frac{\mathrm{m}_e e^4}{8\varepsilon_0^2 \mathrm{h}^3} = 3,29 \cdot 10^{15} \mathrm{s}^{-1}$$

(1) leads to

$$\mathrm{E_n} = -\mathrm{R} \cdot \mathrm{h}(\mathrm{Z} - \sigma)^2 \frac{1}{\mathrm{n}^2} \qquad (2)$$

The function $\sqrt{\mathrm{E}} = \mathrm{f}(\mathrm{Z})$ provides a so-called Moseley diagram, which can be used to determine either R, n or σ.

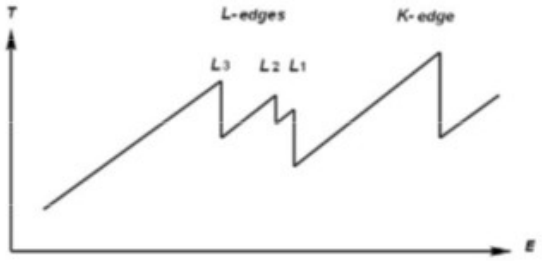

Fig. 1: Schematic course of the transmission T as a function of the quantum energy E in the range of absorption edges.

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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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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 2, 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. 3) 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. 2: Connectors in the experiment chamber

Fig. 3: 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.
- 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 2:1 coupling mode (Fig. 4).

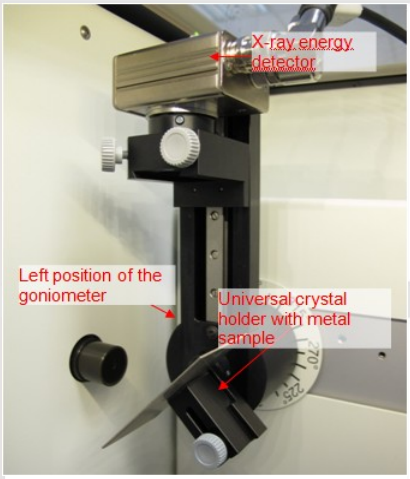

Fig. 4: Goniometer set-up

Procedure (1/6)

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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

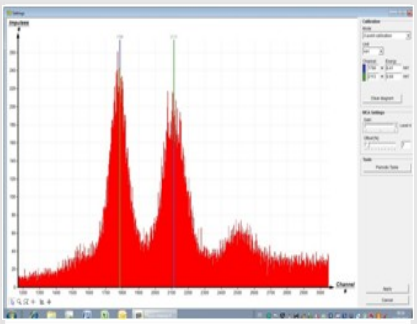

Fig. 5: calibration of the multi-channel analyser

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Procedure (2/6)

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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 5.
- 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 (3/6)

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Sample preparation

The thickness of the powder samples is very important for obtaining clear absorption edges. If the samples are not thick enough, the edge effect cannot be made visible. On the other hand, samples that are too thick do absorb the entire primary beam intensity. This is why the following method is recommended for preparing samples in the thickness range between 0.2 and 0.4 mm: Use an office punch and punch several sheets of paper of a suitable thickness (approximately 3 layers of standard writing or printing paper that are glued together). Then, seal the hole on one side with some transparent adhesive tape (Fig. 6a). The result is a little “pot”. Fill the sample powder into the pot with a spatula and smooth the surface. Seal the pot with another piece of transparent adhesive tape (Fig. 6b and c). Then, fasten the strip of paper that has been cut to size in front of the tube with the 1mm-aperture with some transparent adhesive tape (Fig. 6d).

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Procedure (4/6)

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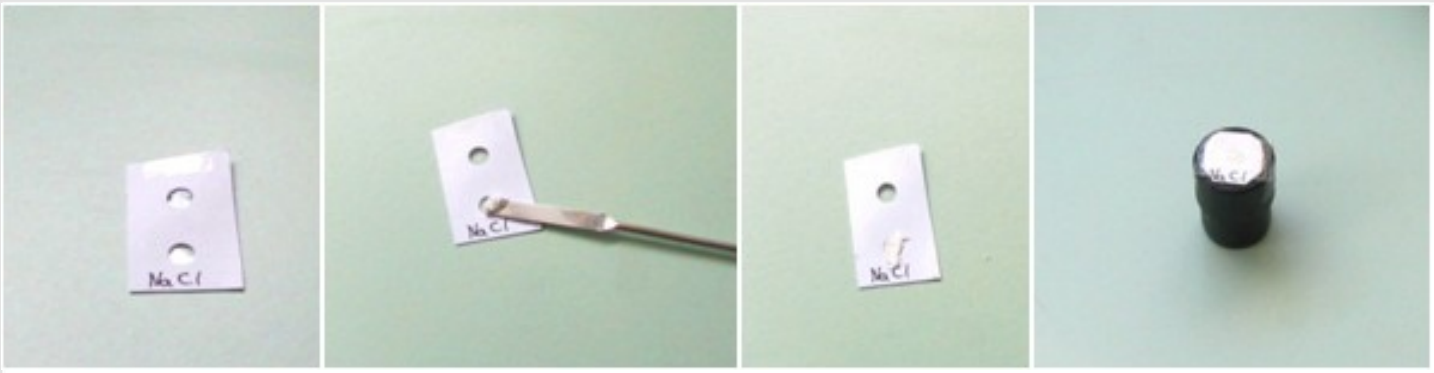

Fig. 6a-d: Sample preparation; a: perforated paper (3 layers) with adhesive tape; b: filling in the powder; c: smoothing the surface; d: fastening on the diaphragm tube

Procedure (5/6)

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Spectrum recording

- The goniometer block and detector (0° position) are now in their respective end positions on the right.
- Adjust an anode voltage U_A = 35 kV and an anode current so that the counting rate is leq 400 c/s.
- Measuring time: 3 to 5 minutes (use the timer of the X-ray unit).

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Procedure (6/6)

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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”.
- Then, smoothen the curve to a medium extent with
- Extend the relevant edge section with the zoom function
- Determine the extreme values I_{max} and I_{min} of the edge intensity with the function “Survey”
- On the measurement curve, find the energy value that belongs to the intensity middle of the edge (see Fig. 7).

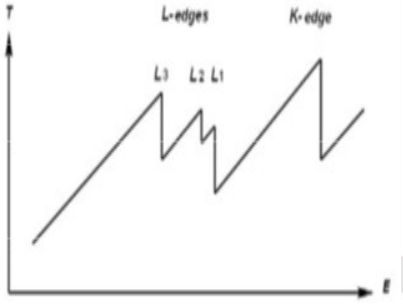

Fig. 7: Determination of the position of the absorption edges.

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Evaluation

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Evaluation of the K-absorption edges (1/3)

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Figure 8 shows the X-ray spectra with the K-absorption edges for various elements.

Column C of Table 1 shows the energy values of the K-edges, which have been determined with the aid of the spectra. Column E shows the corresponding literature values (taken from the "Handbook of Chemistry and Physics", CRC-Press, Inc., USA).

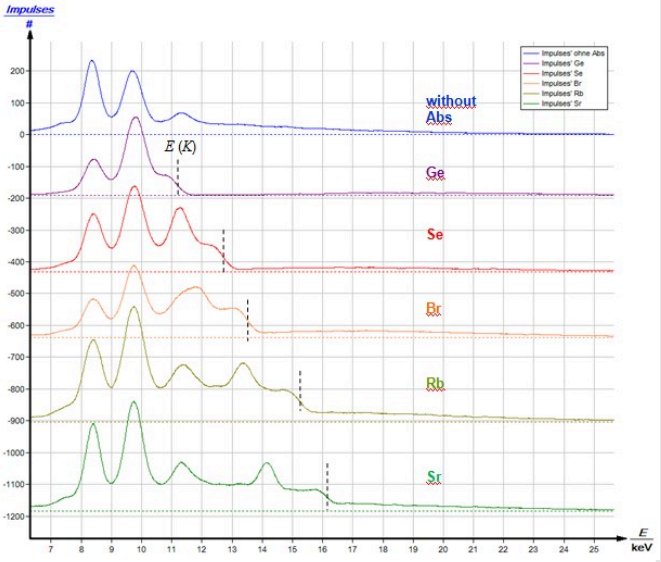

Fig. 8: X-ray spectra with the K-absorpt edges.

Evaluation of the K-absorption edges (2/3)

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A	B	C	D	E
Element	Z	E(K) exp. [keV]	Σ	E(K) lit. [keV]
Ge	32	11.09	2.67	11.103
Se	34	12.63	2.72	12.658
Br	35	13.44	2.72	13.474
Rb	37	15.13	2.75	15.200
Sr	38	16.04	2.74	16.105

Table 1: K-edge absorption

Figure 9 shows the corresponding Moseley diagram. Equation (2) is converted in order to calculate the Rydberg constant R using the Moseley diagram:

$$\sqrt{\mathrm{E_n}} = \frac{1}{\mathrm{n}}\sqrt{\mathrm{R}\cdot\mathrm{h}\cdot\mathrm{Z}-\sigma}$$

Note: convert the energy values of the K-absorption edges form keV to eV before evaluating the straight line. Form the slope of the regression line in Fig. 9, R is calculated:

$$3,6\sqrt{\mathrm{eV}} = \frac{1}{\mathrm{n}}\sqrt{\mathrm{Rh}} \quad (3)$$

$$\Rightarrow \quad \mathrm{R} = 3.13\cdot 10^{15}\,1/\mathrm{s}$$

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

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Column D of Table 1 shows the values of the screening constant σ that were calculated with the aid of (2). However, in accordance with Bohr's atom model, σ should be 1 for the K shell, since the attenuation of the nuclear field is only caused by a 1s electron that is not involved in the ionisation process. A screening value of $\sigma \approx 2.7$ emphasises the limited validity of Bohr's atomic model. More detailed calculations (Hartree) shows that the electrons of higher shells (e.g. the 3p electron) also have secondary maxima near the nucleus in the radial charge density distribution.

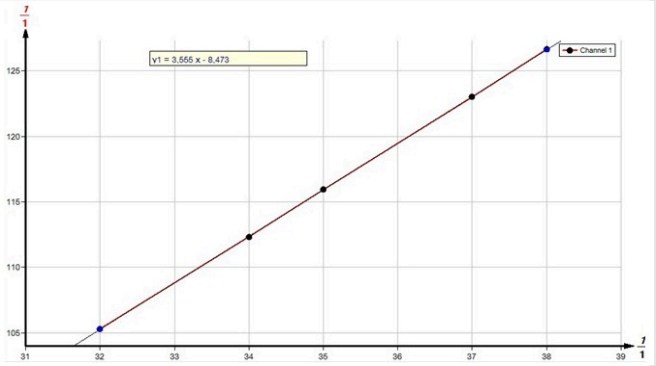

Fig. 9: Moseley diagram for the K-edge absorption.

Evaluation of the L-absorption edges (1/3)

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Figure 10 shows the X-ray spectra with the L-absorption edges for various elements. An L_1 edge cannot be proven here, due to the fact that the intensity is too low.

Columns C and D of Table 2 show the energy values of the L-edges that were determined based on the spectra. For comparison, columns E to G show the corresponding literature values.

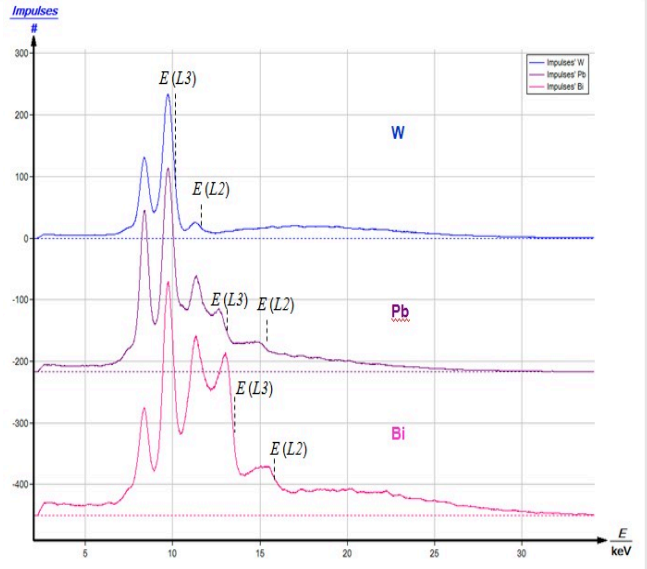

Fig. 10: X-ray spectra with the L-absorpt edges.

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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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Evaluation of the L-absorption edges (2/3)

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Figure 11 shows the corresponding Moseley diagrams. The gradients of these diagrams are used to determine the principal quantum numbern.

$L_1 : 1,9\sqrt{eV} = \frac{1}{n}\sqrt{Rh}$

with Rh = 13.6 eV (Ek of the hydrogen atom), the following results:

n = 1.94 ≈ 2; accordingly, the straight line for L_2 leads to: n = 2.16 ≈ 2.

A	B	C	D	E	F	G
Element	Z	E(L_2) exp. [keV]	E(L_3) exp. [keV]	E(L_1) lit. [keV]	E(L_2) lit. [keV]	E(L_3) lit. [keV]
W	74	11.76	10.15	12.100	11.544	10.207
Pb	82	15.33	13.00	15.861	15.200	13.035
Bl	83	15.70	13.37	16.388	15.711	13.419

Table 2: L-edge absorption

Evaluation of the L-absorption edges (3/3)

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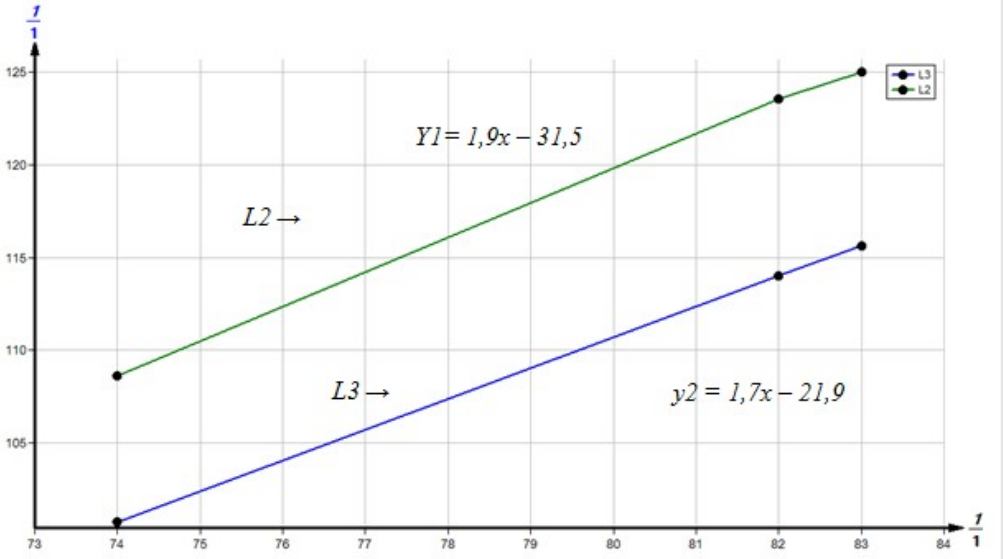

Fig. 11: Moseley diagram for the L-edge absorption.

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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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Appendix

Security Information (1/6)

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

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37079 Göttingen

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

info@phywe.de
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Security Information (2/6)

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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	--.

Security Information (3/6)

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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 ($SrSO_4$)		
-	--	--

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37079 Göttingen

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Security Information (4/6)

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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.

Security Information (5/6)

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

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37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
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Security Information (6/6)

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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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Fax: 0551 604 - 107
info@phywe.de
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