Solid-state physics

Applied solid-state physics

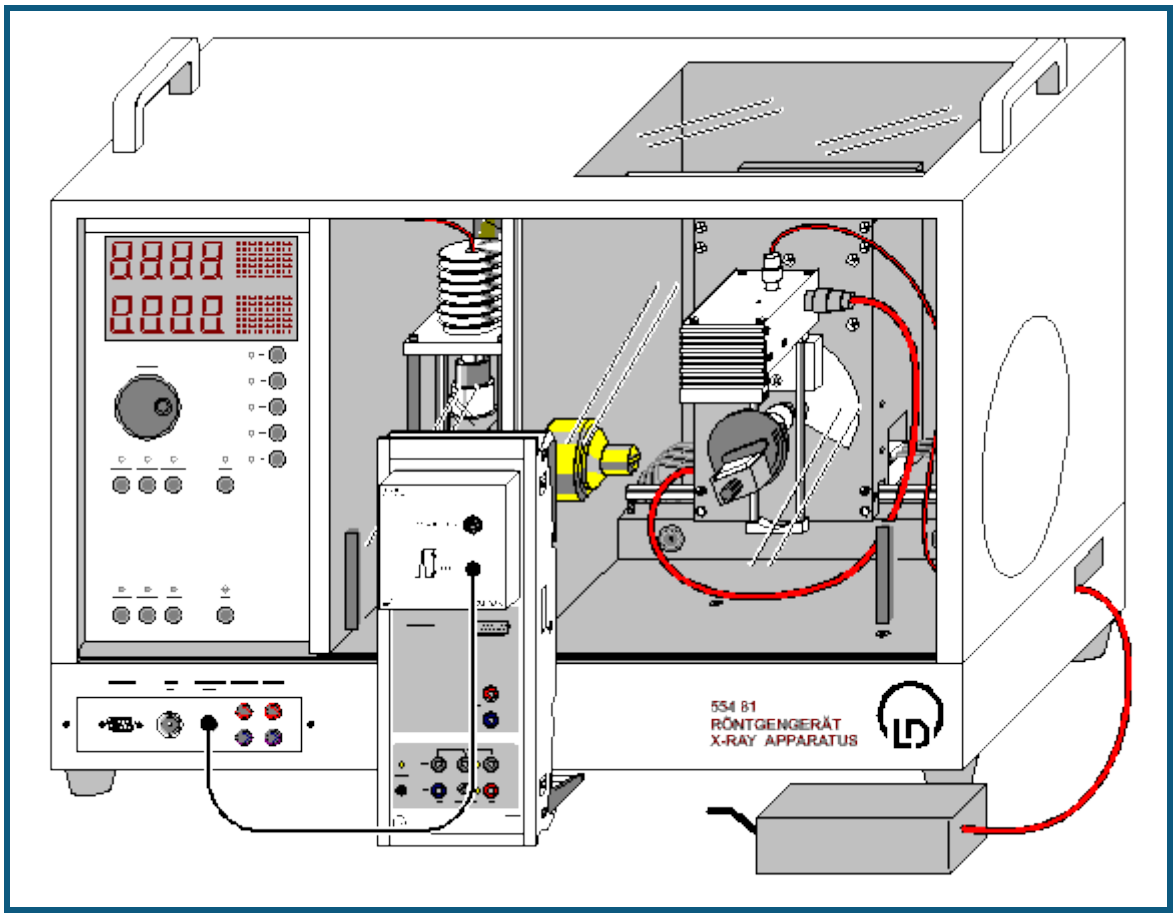
X-ray fluorescence analysis

Determination of the chemical composition of a brass sample by X-ray fluorescence analysis

Description from CASSY Lab 2

For loading examples and settings, please use the CASSY Lab 2 help.

Determination of the chemical composition of a brass sample (x-ray fluorescence)



can also be carried out with Pocket-CASSY

Safety notes

The X-ray apparatus fulfils all regulations on the design of an X-ray apparatus and fully protected device for instructional use and is type approved for school use in Germany (BfS 05/07 V/Sch RöV or NW 807 / 97 Rö).

The built-in protective and shielding fixtures reduce the dose rate outside the X-ray apparatus to less than 1 µSv/h, which is of the order of magnitude of the natural background radiation.

- Before putting the X-ray apparatus into operation, inspect it for damage and check whether the voltage is switched off when the sliding doors are opened (See instruction sheet of the X-ray apparatus).
- Protect the X-ray apparatus against access by unauthorized persons.

Avoid overheating of the anode in the X-ray tube.

- When switching the X-ray apparatus on, check whether the ventilator in the tube chamber starts rotating.

The goniometer is positioned solely by means of electric stepper motors.

- Do not block the target arm and the sensor arm of the goniometer and do not use force to move them.

When handling heavy metals or allergen substances from the target set, observe their operating instructions.

Experiment description

In this experiment, the quantitative analysis of the chemical composition of a brass sample containing lead is carried out. The components in this alloy were already identified in the experiment Non-destructive analysis of the chemical composition.

For calculating the mass ratios, the fact is made use of that the height of a peak is proportional to the number n of radiating atoms. In the reference spectrum this number n_0 is determined by the density of the substance ρ, its atomic weight A, the radiated area S and the effective thickness d of the irradiated layer:

$n_0 = S \cdot d \cdot \rho / A$.

For the number of atoms of each type in the alloy, to first approximation the expression

$n = n_0 \cdot H/H_0 = V \cdot \rho/A \cdot H/H_0$

can be used. In this H and H_0 are the peak heights in the spectrum to be analyzed and in the reference spectrum is V= S·d the irradiated volume. Using this information, the mass ratio C_i of the element number i in the alloy is

$$C_i = \frac{n_i \cdot A_i}{\sum_j n_j \cdot A_j} = \frac{\rho_i \cdot \frac{H_i}{H_{0i}}}{\sum_j \rho_j \cdot \frac{H_{0j}}{H_{0j}}}$$

Equipment list

1	Sensor-CASSY	524 010 or 524 013
1	CASSY Lab 2	524 220
1	MCA box	524 058
1	X-ray apparatus with x-ray tube Mo	554 801 or 554 811
1	Target set of alloys	554 848
1	Target set for K-line fluorescence	554 844
1	Target set for L-line fluorescence	554 846
1	X-ray energy detector	559 938
1	HF cable, 1 m	501 02
1	PC with Windows XP/Vista/7/8	

Experiment setup (see drawing)

- Guide the connection cable for the table-top power supply through the empty channel of the x-ray apparatus and connect it to the mini-DIN socket of the x-ray energy detector.
- Secure the sensor holder with the mounted x-ray energy detector in the goniometer sensor arm
- Connect the signal output of the x-ray energy detector to the BNC socket SIGNAL IN of the x-ray apparatus by means of the BNC cable included
- Feed enough connection cable through to make complete movement of the sensor arm possible
- Press the SENSOR button and set the sensor angle with the rotary adjuster ADJUST manually to 90°
- Set the distances between the slit aperture of the collimator and the axis of rotation as well as between the axis of rotation and the window of the x-ray energy detector both to 5 to 6 cm
- Press the TARGET button and adjust the target angle manually using the rotary button ADJUST to 45°.
- Connect Sensor-CASSY to the computer and connect the MCA box
- Connect the SIGNAL OUT output in the connection panel of the x-ray apparatus to the MCA box by means of the BNC cable.

Carrying out the experiment

☐ Load settings

- Connect the table-top power supply to the mains (after approx. 2 min the LED will glow green and the x-ray energy detector will be ready for use)
- Place target 3 (brass containing lead) from the target set of alloys onto the target table
- Set the tube high voltage U = 35 kV, emission current I = 1.00 mA and switch the high voltage on
- Start the spectrum recording with ⏱
- Finally, record the spectra for the targets Cu, Zn and Pb from the target sets for K-lines and L-lines fluorescence as reference spectra

Energy calibration

The energy calibration is carried out using the spectra of copper and lead (reference spectra).

- Open in the Settings EA (right mouse button) the Energy calibration, select **Global for all spectra of this input** and enter on the right-hand side the energies of the Cu K_α-line (8.04 keV) and of the Pb L_α-line (10.56 keV).
- In the context menu of the diagram select Calculate peak center, mark the Cu K_α-line and enter the result in the left-hand side of the Energy calibration (e.g. with drag & drop from the status line)
- Then determine the center for the Pb L_α-line and also enter it on the left-hand side.
- Switch the display to energy (e.g. with Drag & Drop of E_A into the diagram)

Evaluation

For identifying and labeling the lines in the brass spectrum:

- In the context menu of the diagram select Set Marker → X-ray Energies → Fe
- Then enter the lines of zinc (**Zn**) and lead (**Pb**)

It becomes apparent that the second-highest peak in the spectrum consists of two lines which are not resolved: Zn K_α and Cu K_β. The Cu K_β-line is in part superimposed with the Zn K_α-line.

The mass ratios of the alloy components are calculated by comparing the heights of the strongest lines in the fluorescence spectrum of brass and the reference spectra. These lines are: the copper K_α, the zinc K_α and the lead L_α.

For the determination of the heights of the Cu K_α and the Zn K_α, the brass fluorescence spectrum must be resolved in the energy range from 7.5 keV to 9.1 keV. For this the spectrum in this range is fitted with three Gaussian curves of equal width at the known energies of the Cu K_α-line (E = 8.04 keV), the Cu K_β-line (8.91 keV) and the Zn K_α-line (8.64 keV). The best way to do this is using the fit Gaussians of specified Energies. When selecting the area, it must be observed that all the three required energy lines are contained in the area (do not include the Pb L_l-line).

The result is a fitted contour of the fluorescence spectrum. The determined heights H are found in the status line and are to be entered in the **Mass Proportion** diagram (e.g. by drag & drop) together with the densities ρ for Cu (ρ = 8.96 g/cm^3), Zn (ρ = 7.10 g/cm^3) and Pb (ρ = 11.34 g/cm^3).

The same applies to the heights H_0 for the three reference spectra. When the three densities and the six heights have been entered, the three mass ratios are automatically calculated.

The determined mass ratios of the alloy components in the brass sample correspond well to the known chemical composition (CuZn39Pb3).

Element	Stated	Experimental
Copper	58 %	61.6 %
Zinc	39 %	35.6 %
Lead	3 %	2.9 %

Additional information

The example of the copper-zinc alloy (brass) shows how the secondary fluorescence modifies the spectrum shape. During the irradiation of such a sample with x-ray photons, the K-lines of both copper and zinc are excited. But because the K_β-line of zinc (E = 9.57 keV) lies above the K-edge of copper (E = 8.99 keV), it can also "secondarily" excite the copper L-lines.

Therefore, in the fluorescence radiation emitted, the intensity of the copper lines is higher at the cost of the Zn K_β-line, and the ratio of the Zn K_α and the K_β-lines does not correspond to the ratio found in pure zinc. For this reason, the mass ratio of the alloy components determined from the K_α-lines indicates a slightly too high proportion of copper.