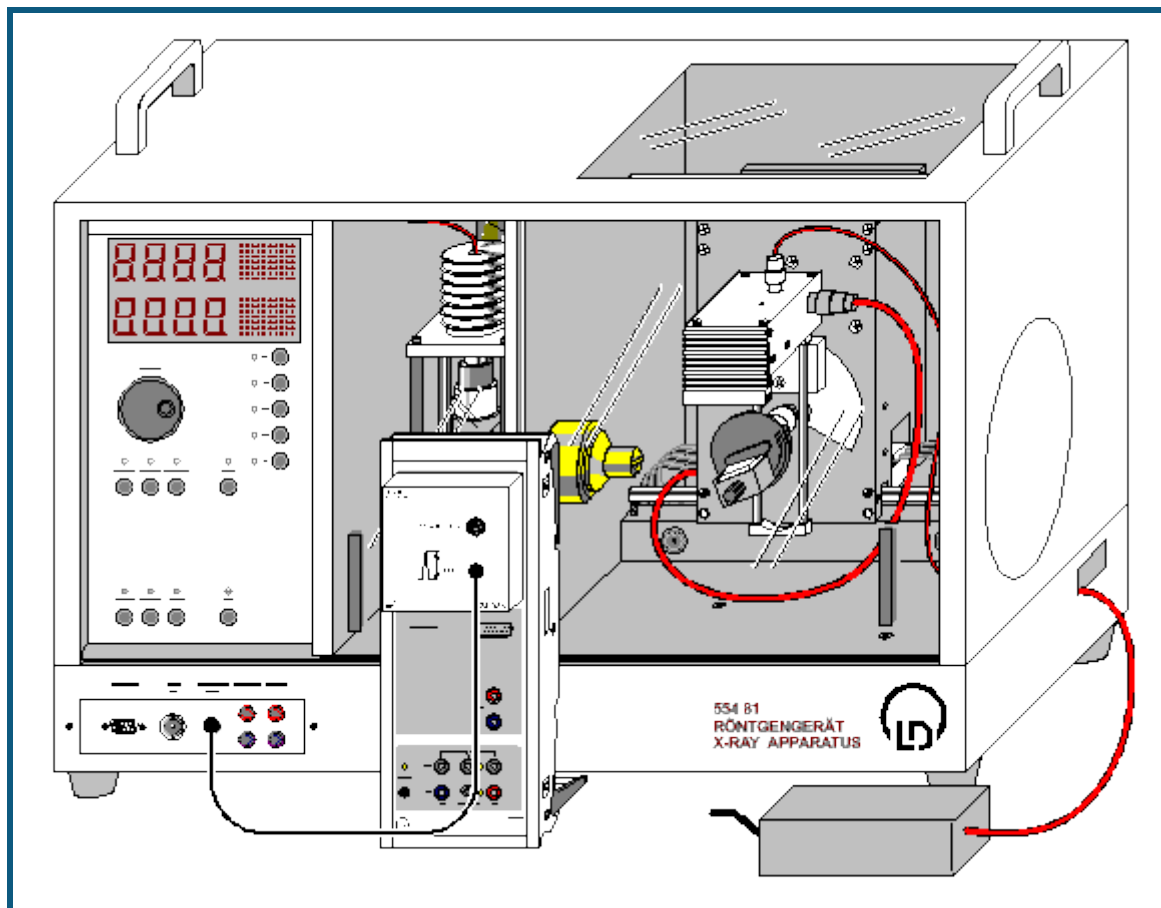# Solid-state physics

Applied solid-state physics
X-ray fluorescence analysis

Application of X-ray fluorescence for the non-destructive analysis of the chemical composition

Description from CASSY Lab 2

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

Non-destructive analysis of the chemical composition (x-ray fluorescence)

can also be carried out with Pocket-CASSY

Experiment description

When a sample is irradiated with high energy x-ray photons it will emit characteristic x-ray lines whose energy depends on the atomic number of the element of the sample material. This dependency is the topic of the experimental examples of Moseley's law (K-line and L-line x-ray fluorescence)

If a sample consists of a chemical compound or mixture, its fluorescence spectrum will also be complex. Because the inner electron shells between which the x-ray transitions occur are not involved in the chemical bonds, the characteristic lines are largely independent of the chemical bonds in the element. This means that the x-ray fluorescence spectra of a chemical compound are, to a first approximation, a superposition of the spectra of its components.

For the qualitative analysis of the chemical composition of a sample, initially all the peaks found in the fluorescence spectrum are correlated to the elements. This is done by means of the values for the energies of the characteristic

lines found in the table. For the correlation, the "pattern" of each of the spectral series is also considered: for example, together with the K_α-line there must also be the K_β-line with a lesser (approx. one fifth to one tenth) intensity in the spectrum. The L_α-line appears accompanied by the L_β-line of a similar intensity and the L_γ-line of a lesser intensity.

Information about the relative concentrations of individual elements in the compound can be gained from the relative intensities of their fluorescence lines.

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	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 the calibration target (galvanized steel plate) from the scope of delivery for the x-ray energy detector onto the 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 ⏱
- Then record spectra for the four targets in the target set of alloys

Energy calibration

The energy calibration of the spectra is made using the spectrum of the calibration target (Fe+Zn).

- 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 Fe K_α-line (6.40 keV) and of the Zn K_α-line (8.64 keV).
- In the context menu of the diagram select Calculate peak center, mark the Fe 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 Zn K_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)
- In the context menu of the diagram select Set Marker → X-ray Energies → Fe and Set Marker → X-ray Energies → Zn for identification and labeling of the lines.

It becomes apparent that four of the measured peaks have been caused by the fluorescence of the main components Fe and Zn of the galvanized steel plate.

Evaluation

For the identification of the components of the alloys:

- Select spectrum and mark a suitable section

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- In the context menu of the diagram select <u>Set Marker → X-ray Energies</u> and an element symbol and determine a suitable element by means of the displayed markers for its x-ray energies.
- Place the markers by clicking on the element symbol and determine further components of the alloy.

The results of the qualitative investigation of the alloys by means of their x-ray fluorescence spectra correspond to the known chemical composition:

- Target 1: Stainless steel X5CrNi18-10 – contains 72 % Fe, 18 % Cr, 10 % Ni.
- Target 2: Brass CuZn36 – contains 64 % Cu, 36 % Zn.
- Target 3: Brass CuZn39Pb3 – contains 58 % Cu, 39 % Zn, 3% Pb.
- Target 4: Praseodymium-samarium-cobalt magnet. These magnets can, in addition to Co, Sm, Pr, also contain Fe, Cu and Zr. Also the K-lines of bromium can be found, which originate from the fire-retardant in the plastic support.