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

Objects of the experiment

■ Recording of fluorescence spectra for several alloys.

■ Qualitative analysis of the fluorescence spectrum: determination of the elements present in the alloy.

Principle

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 relationship (Moseley's law) has already been treated together with the x-ray fluorescence spectra of several elements in the LD Physics Leaflets P6.3.5.4 and P6.3.5.5.

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 of the element*. This means that the x-ray fluorescence spectra of a chemical compound are, in a first approximation, a superposition of the spectra of its components.

For the qualitative analysis of the chemical composition of a sample, initially all 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 $K\alpha$–line there must also be the $K\beta$–line with a lower (approx. one fifth to one tenth) intensity in the spectrum. The $L\alpha$–line appears accompanied by the $L\beta$–line of a similar intensity and the $L\gamma$–line of a lower intensity.

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

Apparatus

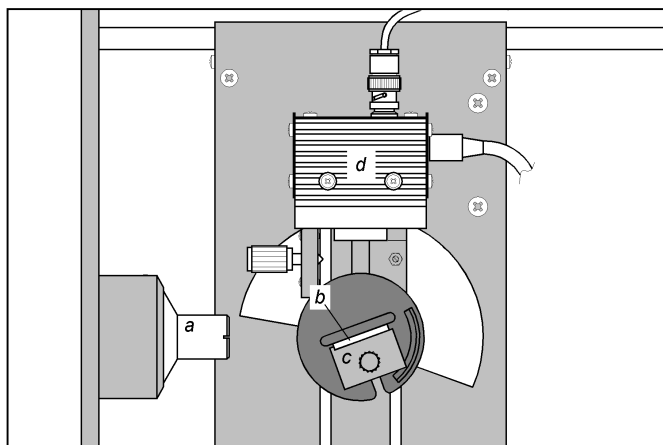|	
1 set of x-ray apparatus with Mo x-ray tube and goniometer	554 811
or	
1 set of x-ray apparatus with Cu x-ray tube and goniometer	
1 x-ray energy detector	559 938
1 alloys target set	554 848
1 CASSY sensor	524 010
1 MCA box	524 058
1 CASSY Lab	524 200
1 HF cable, 1 m	501 02
1 PC with Windows 98/NT or higher	

Fig. 1: Setup of the experiment: *a* – collimator, *b* – target, *c* – target table , *d* – detector.

Setup

The experimental set-up is shown in fig. 1.

- Push the connection cable for the table-top device through the empty channel of the x-ray device 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 device by means of the BNC cable included.

This x-ray apparatus fulfils all regulations governing the construction of x-ray apparatus for use in schools and fully protected devices for instructional use, and it is type approved for school use in Germany (NW 807 / 97 Rö).

The built-in protection and screening measures reduce the local dose rate outside the x-ray apparatus to less than 1 µSv/h, a value which is of the same order of magnitude as the natural background radiation.

■ Before commissioning the x-ray apparatus, inspect it for damage and check that the high voltage is switched off when the slide doors are opened (see the operating instructions for the x-ray apparatus).

■ Protect the x-ray apparatus from access by unauthorised people.

Overheating of the anode in the x-ray tube is to be avoided.

■ When switching on the x-ray apparatus, check if the fan in the tube chamber is rotating.

The goniometer is positioned exclusively 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.

- 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 twist adjuster ADJUST manually to 90°.
- Connect sensor CASSY to the computer and connect the MCA box.
- Connect the SIGNAL OUT output in the connection panel of the x-ray device to the MCA box by means of the BNC cable.
- 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.
- Place the calibration target from the scope of delivery for the x-ray energy detector onto the table.
- Press the TARGET button and adjust the target angle manually using the twist button ADJUST to 45°.

Carrying out the experiment

- Connect the table-top device to the mains (after approx. 2 min the LED of the x-ray energy detector will glow green and the device will be ready for use).
- Call CASSY Lab and set the measuring parameters "Multi-channel measurement, 512 channels, negative pulses, amplification = -2.5, measuring duration = 180 s".
- Set the tube high voltage $U = 35$ kV, emission current $I = 1$ mA and switch the high voltage on.
- Start the spectrum recording by clicking on 🕐 or pressing F9.
- Then record spectra for the targets in the alloy target set.
- Save the entire measurement under a suitable name.

Example of a measurement

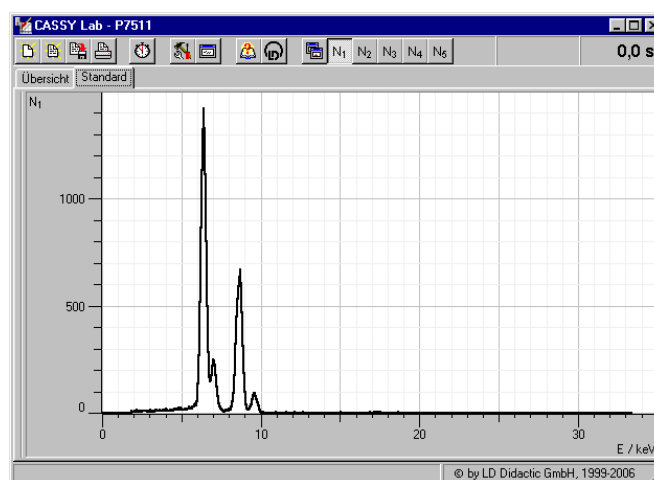The recorded spectra are shown in fig. 2a-e.

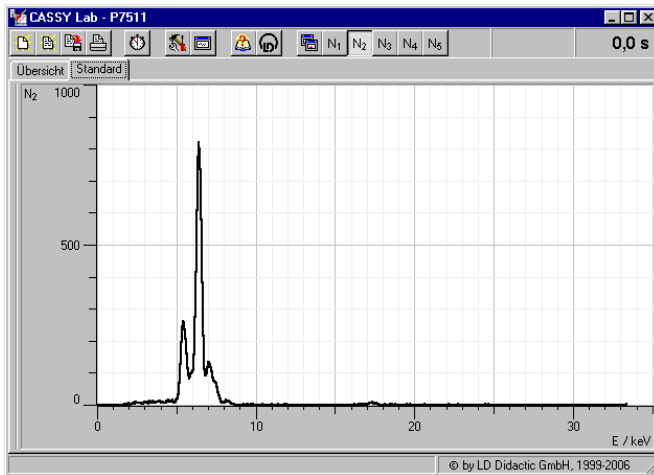Fig. 2a: Fluorescence spectrum of the calibration target.
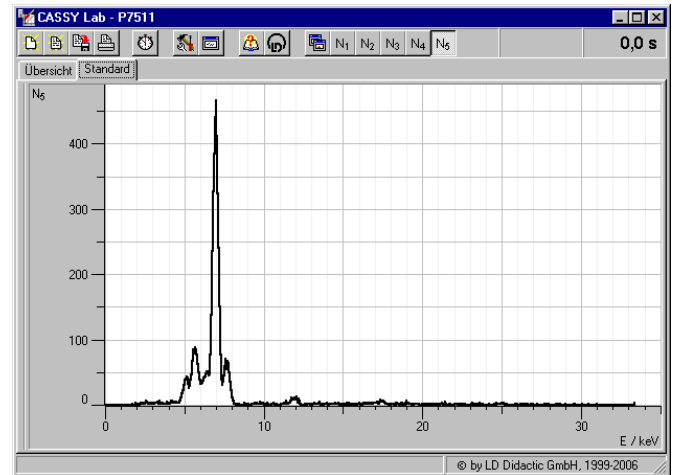
Fig. 2b: Fluorescence spectrum of target 1.
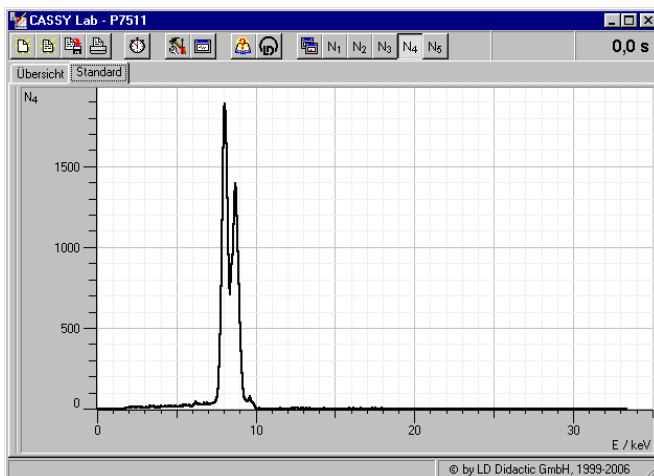

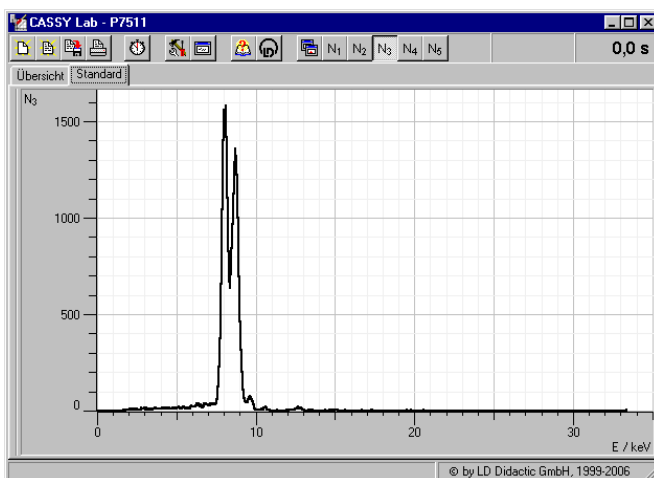Fig. 2e: Fluorescence spectrum of target 4.

Fig. 2c: Fluorescence spectrum of target 2.

Evaluation

a)   Energy calibration of the spectra

The energy calibration is made using the spectrum of the calibration target (Fe+Zn) (fig. 3a).

- Open the "Energy Calibration" dialogue window by pressing Alt+E, select "Global Energy Calibration" and enter the energies for the Fe Kα-line (6.40 keV) and the Zn Kα-line (8.64 keV).

- In the popup menu of the diagram window select the menu item "Other Evaluations" $\rightarrow$ "Calculate Peak Centre", select the Fe Kα-line and enter the result in the "Energy Calibration" dialogue window.

- Then determine the centre of the Zn Kα-line and enter it.

For identification and labelling of the lines

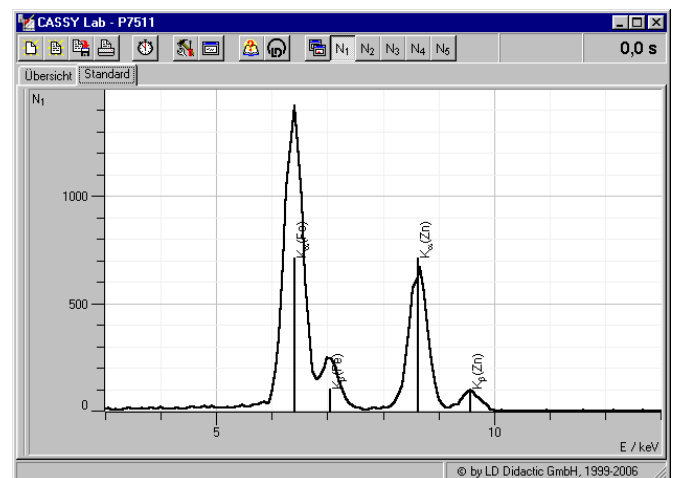- In the pop-up menu of the diagram window select the menu item "X-ray Energies" and click on the element symbol Fe.

Fig. 2d: Fluorescence spectrum of target 3.

Fig. 3a   Fluorescence spectrum of the calibration target with identified lines.

- Close the window by clicking the "OK" button.
- Call again the menu item "X-ray Energies" and click on the element symbol Zn.

It becomes apparent that four of the measured peaks have been caused by the fluorescence of the main components Fe and Zn of the galvanised steel plate (see fig. 3a).

b) Identification of the lines in the spectra

For the identification of the alloy components

- Select spectrum and mark a suitable section.
- In the popup menu of the diagram window select the menu item "X-ray Energies" and click on the element symbols and determine a suitable element by means of the displayed markers for the energies in the table.
- Close the window by clicking the "OK" button.
- Call again the menu item "X-ray Energies" and in this way determine further components of the alloy.
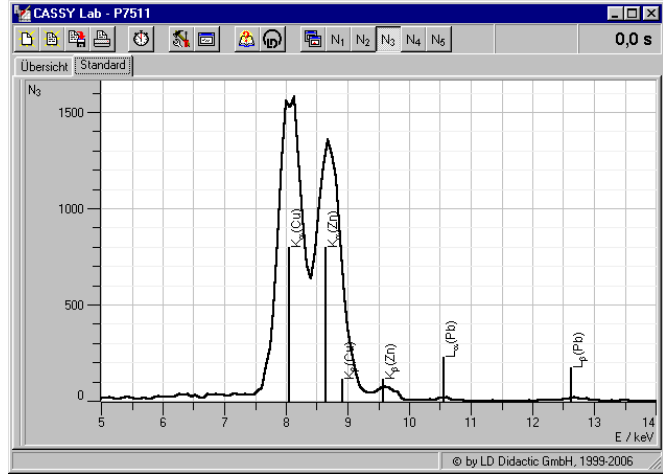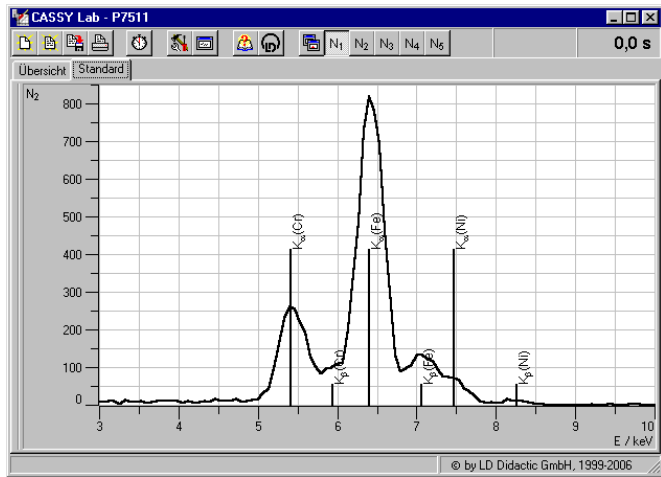

Fig. 3b Determination of the components of target 1: chromium, iron and nickel ⇒ stainless steel.

Fig. 3c Determination of the components of target 2: copper and zinc ⇒ brass.

Fig. 3d Determination of the components of target 3: copper, zinc and lead ⇒ lead containing brass.
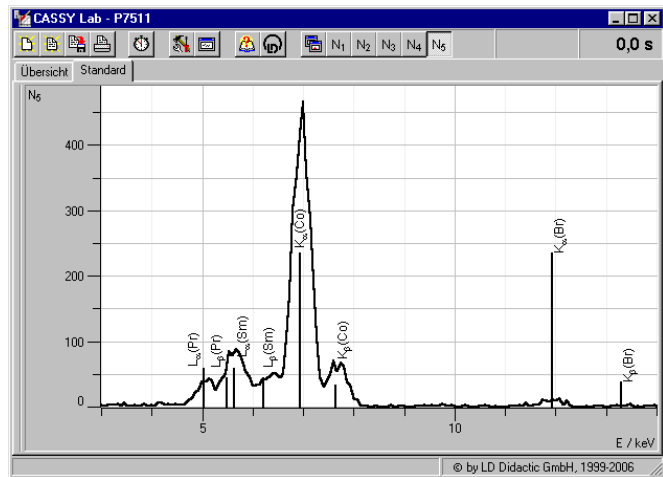

Fig. 3e Determination of the components of target 4: praseodymium, samarium, cobalt. The fluorescence lines of bromine originate from the plastic support (fire retardant)
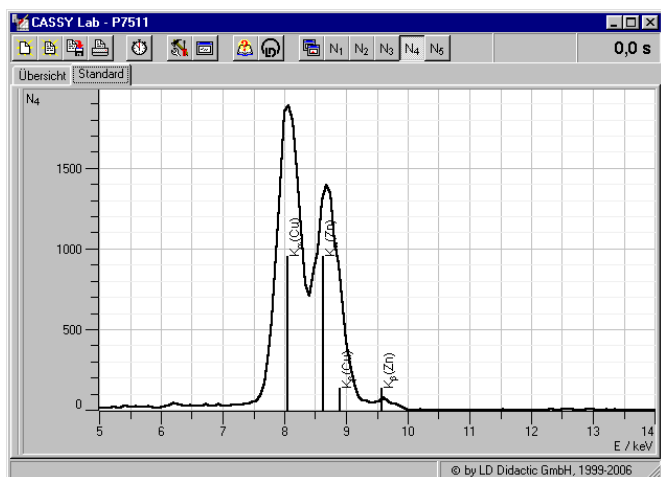
Results

The results of the qualitative investigation of the alloys correspond well 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.