5.4.06-00 Monochromatization of copper X-rays

What you can learn about ...

➔ **Bremsstrahlung**
➔ **Characteristic radiation**
➔ **Energy levels**
➔ **Absorption**
➔ **Absorption edges**
➔ **Interference**
➔ **Diffraction**
➔ **Bragg scattering**

Principle:

Polychromatic X-rays are to be energy analyzed using various monocrystals and a suitably selected thin metal foil having an absorption edge which drastically reduces the intensity of an unwanted line.

What you need:

|		
X-ray basic unit, 35 kV	09058.99	1
Goniometer for X-ray unit, 35 kV	09058.10	1
Plug-in module with Cu X-ray tube	09058.50	1
Counter tube, type B	09005.00	1
Lithium fluoride crystal, mounted	09056.05	1
Potassium bromide crystal, mounted	09056.01	1
Diaphragm tube with nickel foil	09056.03	1
Recording equipment:		
XYt recorder	11416.97	1
Connecting cable, *l* = 100 cm, red	07363.01	2
Connecting cable, *l* = 100 cm, blue	07363.04	2
or		
Software X-ray unit, 35 kV	14407.61	1
RS232 data cable	14602.00	1
PC, Windows® 95 or higher		

Complete Equipment Set, Manual on CD-ROM included
Monochromatization of copper X-rays   P2540600

Tasks:

1. The intensity of the X-rays emitted by the copper anode is to be graphically recorded as a function of the Bragg angle, using LiF and KBr monocrystals successively as analyzers.

2. The energy values of the characteristic copper lines are to be calculated.

3. The LiF monocrystal is to be used to filter out a characteristic line and the appertaining monochromatization graphically recorded.

4. Step 1 is to be repeated, using a nickel filter.

Copper X-ray intensity as a function of the glancing angle ϑ; LiF (100) monocrystal as analyzer (Diameter of diaphragm tube d = 1 mm).
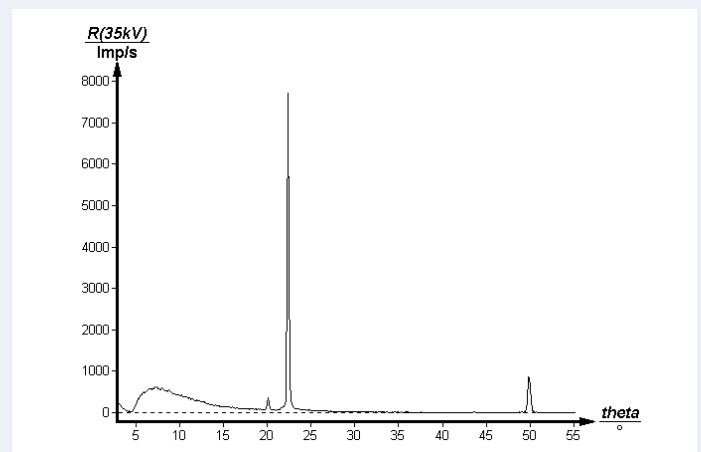
Copper X-ray monochromatization with Ni filter; LiF (100) monocrystal as analyzer (Diameter of diaphragm tube d = 2 mm).

Related topics
Bremsstrahlung, characteristic radiation, energy levels, absorption, absorption edges, interference, diffraction, Bragg scattering.

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Set-up and procedure
Set up the experiment as shown in Fig. 1. Fix the diaphragm tube with 1 mm diameter aperture in the X-ray outlet tube.
With the X-ray basic unit switched off, connect the goniometer and the counter tube to the appropriate sockets in the base plate of the experimenting area. Set the goniometer block with mounted analyzing crystal to the middle position and the counter tube to the right stop.
The following settings are recommended for the recording of the spectra:
— Auto and Coupling mode
— Gate time 2 s; Angle step width 0.1°
— Scanning range 3°-55° using the LiF monocrystal, and 3°-75° using the KBr monocrystal
— Anode voltage U_A = 35 kV; Anode current I_A = 1 mA

Fig. 1: Experimental set-up for copper X-ray monochromatization
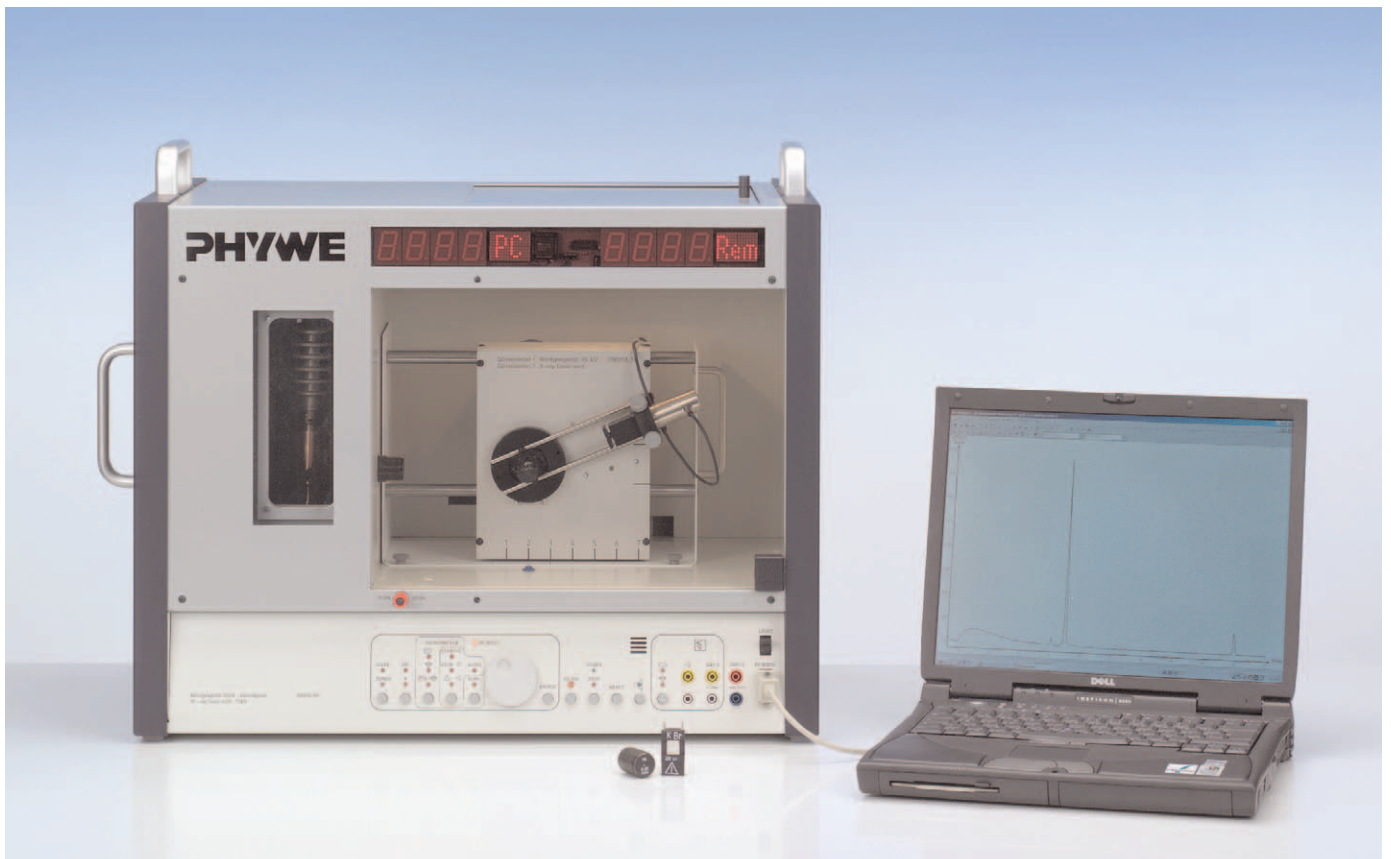
When the spectra are to be recorded with an XY recorder, connect the Y axis to the analog output (Imp/s) of the X-ray unit and, correspondingly, the X input to the analog output for the angular position of the crystal (select the analog signal for the crystal angle with the selection button for this output).
When a PC is to be used for recording purposes, connect it via the SUB-D socket of the X-ray basic unit.

Note
Never expose the counter tube to primary radiation for a longer length of time.

Theory and evaluation
The X-rays emitted from an X-ray tube are polychromatic. Lines characteristic for the anode material, whose energies are not dependent on the anode voltage, are superimposed on the continuum of the bremsspectrum (see experiment 5.4.02-00).
Monoenergetic radiation is required for many X-ray investigations (for example, structure analysis by means of the Debye-Scherrer method). Such radiation can be prepared by using crystal filters or absorption filters.

Monochromatizing by means of crystals
Monocrystals can be used for this purpose. When X-rays of wavelength λ impinge on a monocrystal under glancing angle ϑ, constructive interference after scattering only occurs when the paths of the partial waves on the lattice planes differ by one or more wavelength. This situation is explained by the Bragg equation:

$$2d \sin \vartheta = n\lambda \qquad (1)$$

(d = the interplanar spacing; n = the order of diffraction)

When the value of d is known, and the glancing angle ϑ is measured, the X-ray energy can be calculated by using the following relationship:

$$E = \frac{n \cdot h \cdot c}{2d \cdot \sin\vartheta} \qquad (2)$$

Planck's constant	h	=	$6.6256 \cdot 10^{-34}$ Js
Velocity of light	c	=	$2.9979 \cdot 10^{8}$ m/s
Lattice constant LiF (100)	d	=	$2.014 \cdot 10^{-10}$ m
Lattice constant KBr (100)	d	=	$3.290 \cdot 10^{-10}$ m
and the equivalent	1 eV	=	$1.6021 \cdot 10^{-19}$ J

Fig. 2 shows the copper X-ray intensity as a function of the glancing angle ϑ, with the LiF crystal used as analyzer.

In conjunction with (2), computation yields:
($\vartheta = 22.6°$, $n = 1$); $E_{K\alpha}$ = 8.01 keV
($\vartheta = 20.3°$, $n = 1$); $E_{K\beta}$ = 8.87 keV
($\vartheta = 50.1°$, $n = 1$); $E_{K\alpha}$ = 8.02 keV
($\vartheta = 43.8°$, $n = 2$); $E_{K\beta}$ = 8.89 keV

If only a narrow, strip-like portion of the polychromatic spectrum (for example, the K_α line is required, the analyzer crystal must be brought into the appropriate glancing angle position. Further analysis, carried out using the independently rotating counter tube detector and the corresponding analog output for the angular position of the counter tube, indicates that the scattered portion consists solely of one intense, sharp line of energy $E_{K\alpha}$ (Fig. 3).

Fig. 2: Copper X-ray intensity as a function of the glancing angle ϑ; LiF (100) monocrystal as analyzer (Diameter of diaphragm tube d = 1 mm)

Fig. 4: Copper X-ray monochromatization with Ni filter; LiF (100) monocrystal as analyzer (Diameter of diaphragm tube d = 2 mm)
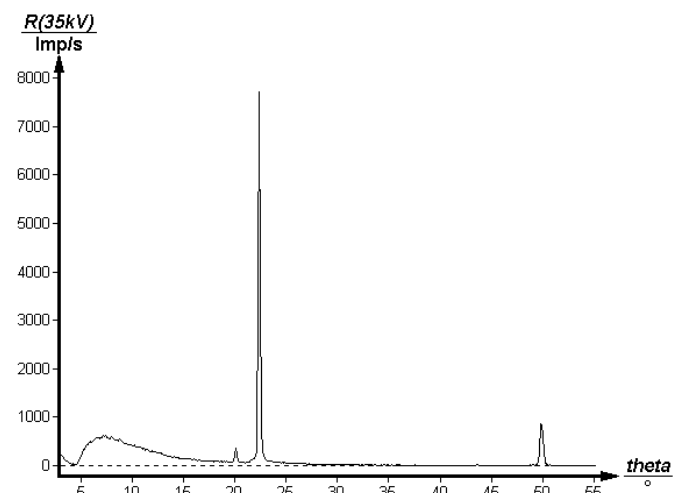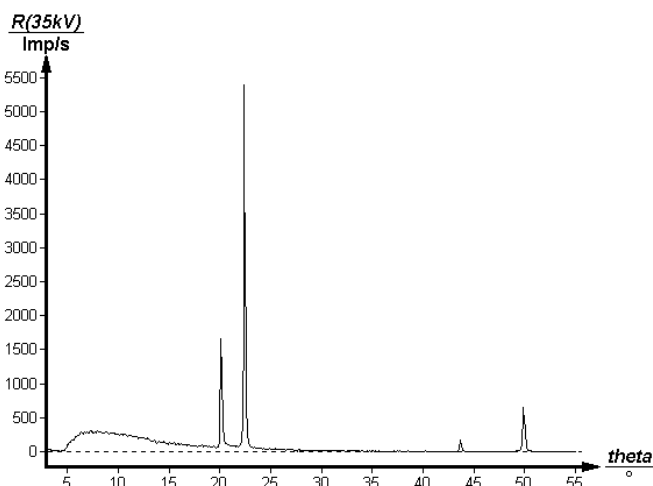
Monochromatizing by means of absorption

If a thin metal foil of thickness x is brought into the path of an X-ray beam of intensity I_0, the attenuation of the intensity can be described by the absorption law:

$$I(E,x) = I(E,0) \, e^{-\mu(E) \cdot x} \qquad (3)$$

(where μ [cm^{-1}] = the linear absorption coefficient)

Although the absorption coefficient is dependent upon wavelength or energy, it generally shows no dramatic change within an energy interval of several keV. Thus, a very similar attenuation of normal absorption can be expected. An entirely different, discontinuous characteristic absorption appears, when the energy of the X-ray quanta barely suffices to ionize the atoms of the absorbing material in the inner shells. This absorption edge can be used to pinpointedly eliminate certain wavelength ranges from the original spectrum. For example, thin nickel foils (Z for nickel = 27) are used to eliminate the K line from the copper X-ray spectrum (Z for copper = 29), because the energy of the K level of nickel lies slightly below the energy of the K_β line.

E_K (Ni) = 8.33 keV;
$E_{K\beta}$ (Cu) = 8.90 keV (literature value).

The energy of the Cu K_α line is already too low to produce ionization in nickel K shells, thus, due to normal absorption, this line is only slightly attenuated by the Ni filter.
Fig. 4 shows the result of the energy analysis of the nickel filtered copper X-rays. The LiF crystal was used as analyzer. A comparison with the unfiltered spectrum (Fig. 2) indicates that the K_β radiation intensity was actually reduced.

Using the KBr crystal as analyzer produces similar results, as shown in Fig. 5 and Fig. 6. It can also be clearly seen here, that the Cu K_β radiation is only observable in the 1st order of diffraction, whereas the Cu K_α radiation (despite slight attenuation) is still clearly recordable up to the 3rd order of diffraction.

Fig. 5: Copper X-ray intensity as a function of the glancing angle ϑ; KBr (100) monocrystal as analyzer
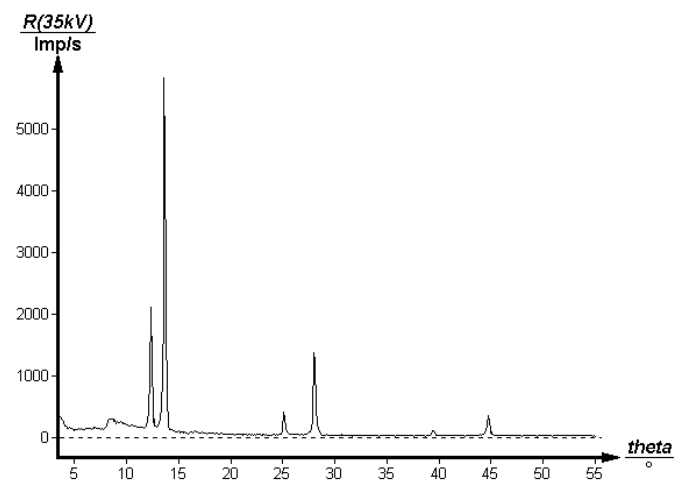

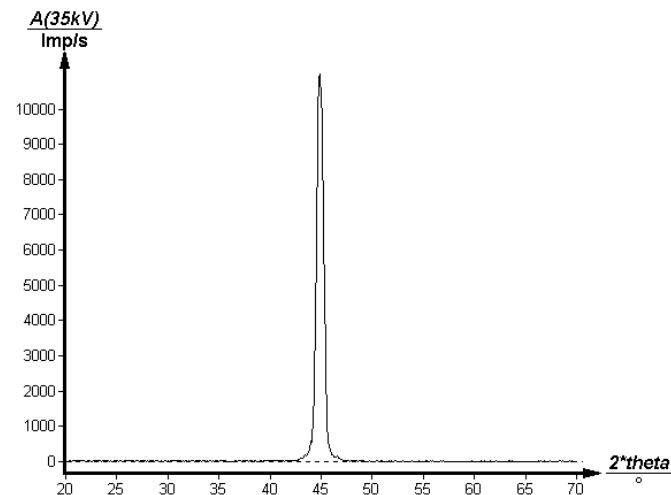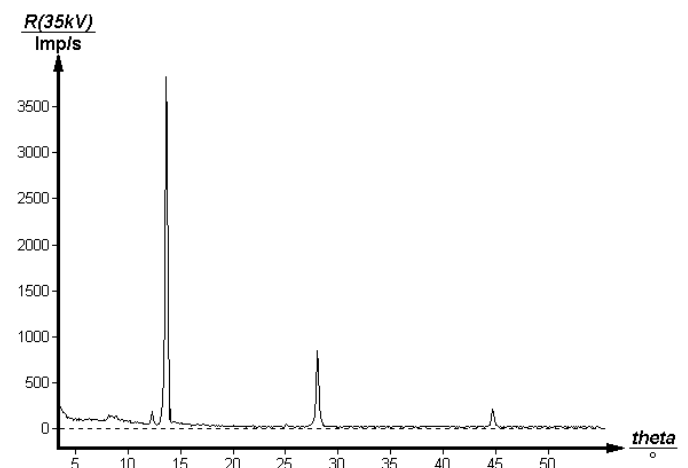Fig. 3; Copper X-ray monochromatization; Reflected energy interval $\Delta E = E_{K\alpha}$

Fig. 6: Copper X-ray monochromatization with Ni filter; KBr (100) monocrystal as analyzer