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# COLLIMATION OF X-RAYS USING TOTAL REFLECTION. PART III. A HIGH-SPEED MONOCHROMATOR FOR THE STUDY OF SMALL-ANGLE X-RAY SCATTERING

by

G. Damaschun

Translated from the German

March 1967

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SUMMARY

A monochromator employing total reflection of X-rays is described for small-angle X-ray cameras, and the spectral influence of radiation by the reflector is calculated. It is shown that the monochromator can be easily connected to a Kratky camera[1] without lengthening the recording time.


## 1. FORMULATION OF THE PROBLEM

With the aid of small-angle X-ray scattering, fluctuations of electron density in the substances to be studied have been demonstrated over ranges between 10 and 1000Å[2].

In 1954, Kratky[1] described a simple camera design which made it possible to measure small-angle scattering, without disturbance from diaphragm scattering, up to angles of $5.10^{-4}$ radians. Fiedler[3], Henke and Schulze[4], Knapp[5], and Eins and Unangst[6] have described technical variations of this camera. In 1964, Kratky and Leopold[7] described an improvement of this diaphragm system. The disadvantage of the Kratky camera in comparison with other designs, e.g., that of Jagodszinski[8] is that additional measures for monochromatization are always necessary. A monochromatic radiation is indispensable for correct interpretation of the finer details of continuous small-angle scattering. The author gave a simple method[9] for achieving adequate monochromaticity of radiation in the Kratky geometry without substantial loss of intensity.

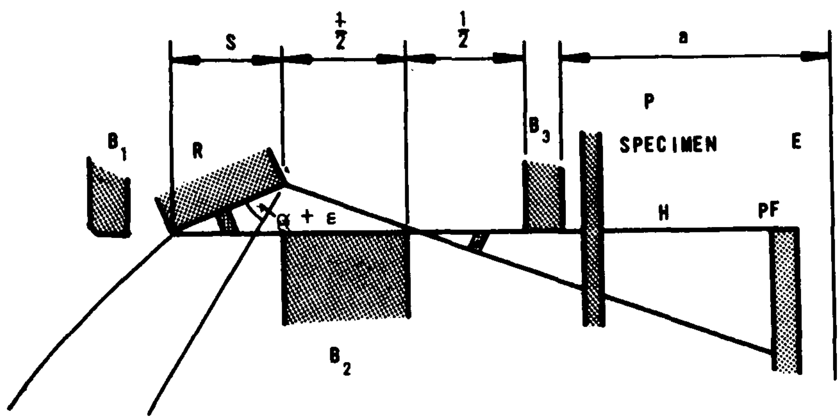
## 2. MONOCHROMATIZATION BY TOTAL REFLECTION

The principle of the monochromator described below is based on the change of spectral distribution of the radiation emitted by the X-ray tube in total reflection of X-ray radiation on a sufficiently smooth surface, when in the interval studied, the reflectivity $R = R(\vartheta, \lambda)$ is a function of wavelength and glancing angle.

In Figure 1, the entrance slit of the Kratky aperture system is replaced by a totally reflecting surface R, which is inclined at angle $\alpha$ to the principal plane of the camera. The geometry of this arrangement is investigated in the variations of Henke and Schulze[4] (Figure 2). It corresponds to a low aperture dispersion two-slit camera. It is assumed that the glancing angle $\vartheta$ and the angular divergence $\epsilon$ are small angles and that $\vartheta$ is approximately equal to $\sin \vartheta$ and also approximately equal to tan $\vartheta$, and that $\epsilon$ approximately equals sin $\epsilon$ and tan $\epsilon$. Because lateral divergence can be neglected in the usual slit collimators for small-angle X-ray scattering[10], the calculation can be unidimensional.

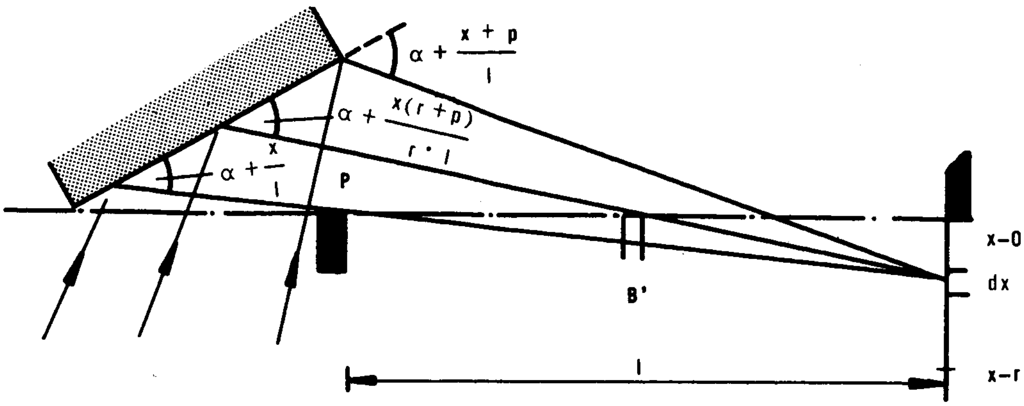
The irradiation intensity b for a line element at distance x from the principal plane is:

$$b(x, \lambda) = \frac{d\dot{W}(x, \lambda)}{dx} = \text{const} \int_{\alpha + \frac{x}{l}}^{\alpha + \frac{x+p}{l}} R(\lambda, \vartheta)\, d\vartheta. \qquad (1)$$




R = Totally reflecting surface.
H = Principal plane of the camera.
PF = Primary ray interceptor.
E = Recording plane.
B = Stops; stop $B_1$ prevents illumination from passing through the forward edge of the reflector block.
P = Specimen.

Figure 1. Principle of the Kratky Frame Camera with the Totally Reflecting Monochromator




The stop B' is eliminated in the two-slit camera.

p = Width of the entrance slit.

r = Width of the exit slit.

$l$ = Length of the stop system.

$\alpha$ = Angle between the principal plane and the reflector.

Figure 2. Determination of Irradiation Intensity in the Two-Slit Camera and in the Kratky Camera

The radiation power $dW(\lambda)$ falling on the specimen is given by integration as:

$$d\dot{W}(\lambda) = \text{const} \int_0^r \int_{\alpha + \frac{x}{l}}^{\alpha + \frac{x+p}{l}} R(\lambda, \vartheta)\, d\vartheta\, dx. \tag{2}$$

If $R(\lambda, \vartheta)$ does not change in the interval $\lambda$, $\vartheta$ being studied, which is the trivial case, for example, without a mirror, then:

$$b(x) = \text{const}\, \frac{p}{l}, \tag{3}$$

$$d\dot{W} = \text{const}\, \frac{p \cdot r}{l}. \tag{4}$$

For wavelengths $\lambda < 2\mathring{A}$ and a reflector material with low order number, $R = R(\vartheta, \lambda)$ can be replaced to a close approximation by:

$$R(\vartheta) = \begin{cases} 1 \text{ for } \vartheta \leq \vartheta_0(\lambda) \\ 0 \text{ for } \vartheta > \vartheta_0(\lambda) \end{cases} \tag{5}$$

$$\vartheta_0(\lambda) = \frac{\lambda\, \vartheta_0(\lambda_E)}{\lambda_E}$$

In this, $\vartheta_0(\lambda)$ is the glancing angle of total reflection, $\lambda_E$ is the selected wavelength, specifically the $K_\alpha$ radiation of the anode material necessary for the study, and $\vartheta_0(\lambda_E)$ is the glancing angle of total reflection for the selected wavelength. It is presupposed that:

$$\vartheta_0(\lambda_E) = \vartheta_E > \frac{p + r}{l}$$

If the specimen is to be completely illuminated by radiation of wavelength $\lambda_E$, then

$$\alpha = \vartheta_E - \frac{p + r}{l} \tag{6}$$

must hold. The length of the reflector must then be

$$s = p\left(\vartheta_E - \frac{p + r}{l}\right)^{-1} \tag{7}$$

When this is substituted into Equation (1), there results

$$b(x, \lambda) = \text{const} \cdot \int_{\frac{x}{l} + \vartheta_E - \frac{p + r}{l}}^{\frac{x}{l} + \vartheta_E - \frac{r}{l}} R(\lambda, \varpi)\, d\varpi \tag{8}$$

When Equation (5) is used, it follows that:

$$b(x,\lambda) = \begin{cases} \dfrac{p}{l} & \text{for } \lambda \geq \dfrac{\lambda_E x}{\vartheta_E l} + \lambda_E - \dfrac{\lambda_E r}{\vartheta_E l} \\ \dfrac{\vartheta_E \lambda}{\lambda_E} - \vartheta_E - \dfrac{x}{l} + \dfrac{p+r}{l} & \text{for } \dfrac{\lambda_E x}{\vartheta_E l} + \lambda_E - \dfrac{\lambda_E r}{\vartheta_E l} > \lambda > \dfrac{\lambda_E x}{\vartheta_E l} + \lambda_E - \dfrac{\lambda_E (p+r)}{\vartheta_E l} \\ 0 & \text{for } \lambda \leq \dfrac{\lambda_E x}{\vartheta_E l} + \lambda_E - \dfrac{\lambda_E (p+r)}{\vartheta_E l} \end{cases}$$

This function is represented in Figure 3. The radiation power falling on the specimen is obtained from Equation (2) by integration along the intersections at which $\lambda$ = constant. Because the expressions resulting can generally be represented only in sections by analytical functions and can be determined more easily by graphic methods, only the solution for the practically important case of p = r = d is given:

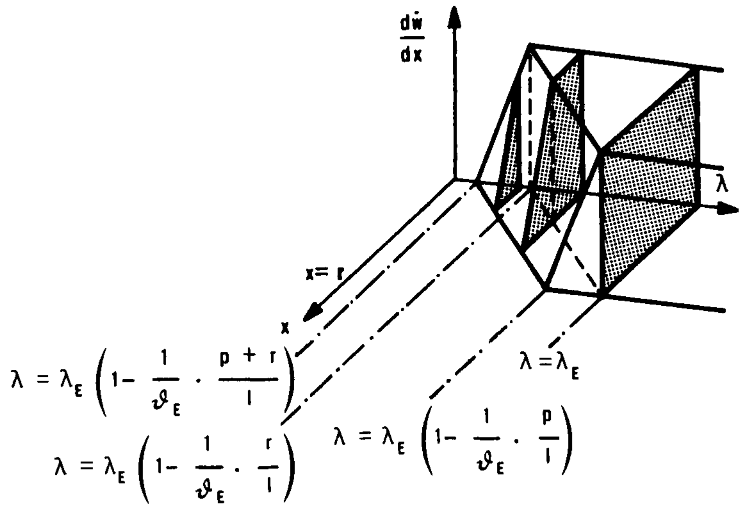
$$\frac{d\dot{W}(\lambda)}{\text{const } \dfrac{d^2}{l}} = \begin{cases} 1 & \text{for } \lambda \geq \lambda_E \\ 1 - \dfrac{1}{2}\left(\dfrac{\vartheta_E l\, \lambda}{\lambda_E d} - \dfrac{\vartheta_E l}{d}\right)^2 & \text{for } \lambda_E\left(1 - \dfrac{d}{\vartheta_E l}\right) \leq \lambda \leq \lambda_E \\ \dfrac{1}{2}\left(\dfrac{\vartheta_E l\, \lambda}{\lambda_E d} - \dfrac{\vartheta_E l}{d} + 2\right)^2 & \text{for } \lambda_E\left(1 - \dfrac{2d}{\vartheta_E l}\right) \leq \lambda \leq \lambda_E\left(1 - \dfrac{d}{\vartheta_E \cdot l}\right) \\ 0 & \text{for } \lambda \leq \lambda_E\left(1 - \dfrac{2d}{\vartheta_E \cdot l}\right) \end{cases}$$

For the Kratky stop arrangement (Figure 2), in analogy to Equations (1), (3), and (4) there results:

$$b(x,\lambda) = \frac{d\dot{W}(x,\lambda)}{dx} = \text{const} \int_{a + \frac{x(r+p)}{rl}}^{a + \frac{p+x}{l}} R(\lambda, \vartheta)\, d\vartheta \tag{1a}$$

$$b(x) = \text{const} \left(\frac{p}{l} - x \frac{p}{r \cdot l}\right) \tag{3a}$$

$$d\dot{W} = \text{const}\ \frac{1}{2}\,\frac{p \cdot r}{l} \tag{4a}$$




Note: For a given wavelength, the area of the dotted surfaces is proportional to the radiation power.

Figure 3. Radiation Intensity $b(x, \lambda) = (d\dot{W}(x, \lambda))/dx$ as a Function of Wavelength and Distance x from the Principal Plane in a Two-Slit Camera with a Plane, Totally Reflecting Monochromator

Expressions corresponding to Equations (8), (9), and (10) can be set up because of Equations (5), (6), and (7). Figure 4 shows the function corresponding to Equation (9a). For the case of practical importance, in which p = r = d:

$$d\dot{W}(\lambda)_{\text{Kratky}} = \frac{1}{2}\, d\dot{W}(\lambda)_{\text{two-slit collimator}}$$

i.e., the spectral influence is the same.

A totally reflecting mirror cuts off all radiation with a wavelength $\lambda \leq \lambda_{min}$. The mirror must have a sufficiently smooth surface to prevent reflection losses[11]. Plates of optically polished glass are suitable. For intensity reasons, generally the $CuK_\alpha$ radiation is used for the study of small-angle X-ray scattering. For the glass type used by us, the glancing angle of total reflection is $\vartheta_0(1.54\ Å)$ $4.5 \cdot 10^{-3}$ radians.

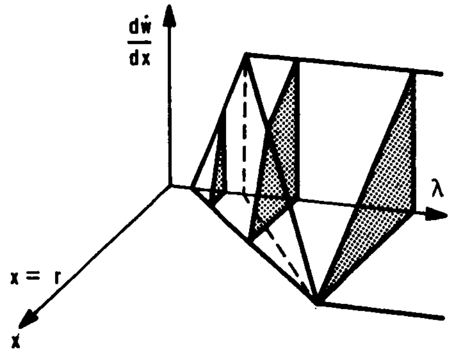



Figure 4. Radiation Intensity as a Function of λ and x is a Kratky Camera with a Plane, Totally Reflecting Monochromator

Figure 5 shows the transmission curve for an angular divergence $\epsilon = 2\,d/l = 10^{-3}$ radians. The radiation emitted by the X-ray tube must be multiplied by these values to obtain the spectral distribution behind the mirror.

The major part of the retardation spectrum, which causes error in evaluation, is completely suppressed by the mirror. The $CuK_\beta$ radiation is cut in half in this case. The drop in the transmission curve can be made even steeper by a $K_\beta$ filter. Radiation of wavelength longer than $\lambda_E$ is retained in the same ratio to the characteristic radiation as in a geometrically limited entrance slit.

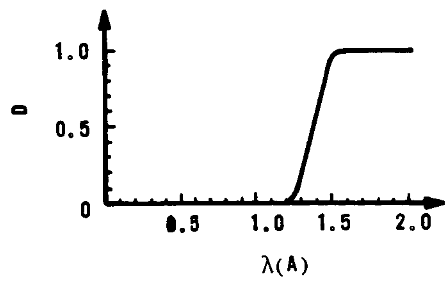



Figure 5. Monochromator Function D(λ) for a Plane Mirror in Conjunction with a Two-Slit Camera of a Divergence $\epsilon = 2\,d/l = 10^{-3}$ Radians at a Glancing Angle of Total Reflection $\vartheta_0(1.54\ \text{Å}) = 4.5 \cdot 10^{-3}$ of the Mirror Material. The Spectral Distribution Impinging on the Mirror Must be Multiplied by This Function to Obtain the Spectral Distribution Behind the Camera.

## 3. DESIGN COMBINATION WITH THE KRATKY FRAME CONSTRUCTION

An optically polished glass block is used as a totally reflecting plate. Equation (5) holds to a close approximation for glass[11, 12]. The glass plate can be rigidly connected without adjustment in very simple fashion by using the frame principle of the Kratky camera. Figure 6 shows a combination with the Kratky U-frame. The frame also has the cut shown in the figure. The edge of the glass block turned toward the tube focus lies on the plane of the principal section, and the other corner is raised by p spacer blocks. To obtain a stable arrangement, the glass block is compressed against the frame with a spring. In front of the reflector is a stop $B_1$ mounted at the height of the principal plane. It prevents irradiation of the front edge of the glass block. The stop system is then adjusted in itself and merely needs to be brought to the proper location relative to the tube focus.

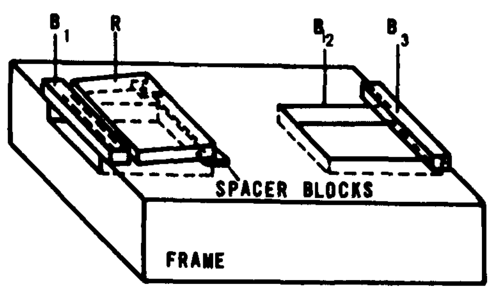



Figure 6. Combination of Totally Reflecting Monochromator with the Frame Design of the Kratky Small-Angle X-Ray Camera

## 4. EXPERIMENTAL RESULTS

The monochromator described was tested in practice on a small-angle camera of the Kratky type, developed in the institute. Figure 7 shows the principle and the design of the arrangement. The angular divergence is $\epsilon = 0.9 \cdot 10^{-3}$ radians. With the aid of a difference filter calibrated with $CuK_\beta$ radiation, the quotient M(= radiation power of the characteristic radiation/total radiation power) was measured. For recording, ORWO-RF 44 film was used. The values of Table 1 pertain only to the photographic recording, because the sensitivity of the film depends on the wavelength.

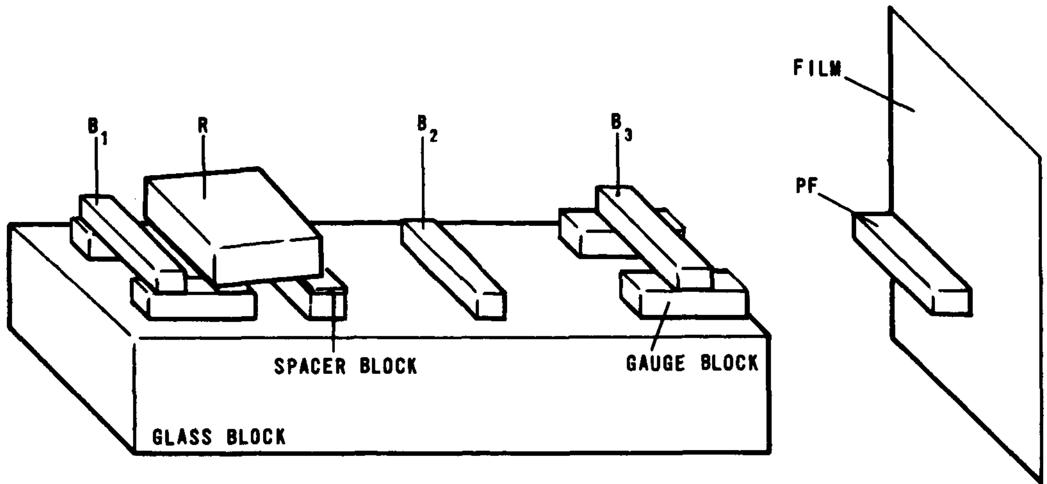



Figure 7a. Experimental Arrangement for Testing the Totally Reflecting Monochromator

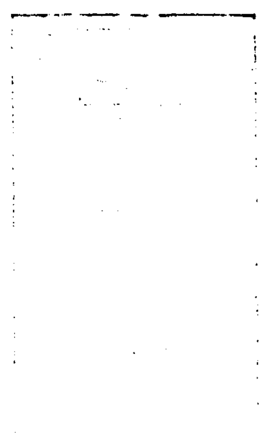


Figure 7b. Small-Angle X-Ray Camera with Totally Reflecting Monochromator

The radiation after the reflector and after a 7μ Ni filter can no longer be differentiated within the limits of error by absorption tests from pure characteristic radiation. The cutoff on the shortwave side was also shown by Reichmann[13] by spectral resolution of the radiation with a crystal.

The method described thus permits easy experimental preparation of small-angle X-ray photographs with sufficiently monochromatic radiation without lengthening the recording time.

I am deeply indebted to Professor Dr. W. Schütz for his stimulating and effective participation in the investigations.

Table 1. Degree of Monochromaticity of the Radiation of an X-ray Tube with Copper Anode in a Small-Angle X-ray Camera, with Photographic Recording

| Radiation Quality after 40 cm Air Path | M |
|---|---|
| 35 kv direct current | 0.71 |
| 35 kv direct current, 7μ Ni filter | 0.78 |
| 35 kv direct current, 21μ Ni filter | 0.71 |
| 35 kv direct current, after total reflection ($\epsilon = 0.9 \cdot 10^{-3}$ radians) | 0.91 |
| 35 kv direct current, after total reflection and 7μ Ni filter | 1.0 (±0.02) |

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13. ABSTRACT

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| Monochromatic radiation | | | | | | |
| Spectral distribution | | | | | | |
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