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The double circular-disc sector used for electron diffraction. By MIEKO TAKAGI, *Tokyo Institute of Technology, Oh-Okayama, Tokyo, Japan*

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Since the electron-diffraction method is increasing its importance in the structure determination of crystals and of amorphous substances, as well as of gas molecules, there is a growing demand for accuracy of intensity measurement of scattered electrons. In the study of gas molecules rotating sectors of various shapes have been successfully utilized to reduce the difficulties arising from the wide range of the scattering intensity of electrons due to the rapid intensity decrease with scattering angle (Finbak, Hassel & Ottar, 1941; Hassel & Viervoll, 1947; Karle & Karle, 1949). Sectors of the type s^n ($n=1, 2, 3$ and 4) are used to realize an effective exposure time of scattered electrons proportional to s^n , where $s=4\pi\sin\theta/\lambda$, λ is the wave length and 2θ is the scattering angle.

To make these sectors, however, necessitates delicate work by hand which requires much skill and time. However, the most essential requirement in using a sector is not that its shape is cut in accordance with the exactly multiple powers of s , but that it has a shape which is accurately known. Therefore, if we contrive a simpler shape of sector which can be finished with ordinary cutting machines, we will not only save the laborious work but improve the precision of the sector.

In this paper a new design of sector is described which satisfies the above requirement. This sector, the double circular-disc sector, is composed of two circular discs. Fig. 1(a) shows schematically the shape of the sector.

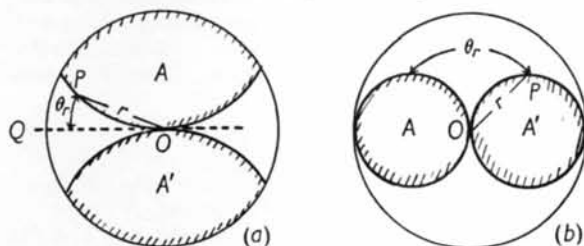


Fig. 1.

The two circular discs A and A' , having the same radius a , are in contact at the centre of rotation, O . OQ is the tangent to A and A' at O . The effective time of exposure for scattered electrons at a point P , at distance r from O , is proportional to the angle θ_r which is given by

$$a \sin \theta_r = \frac{1}{2} r. \quad (1)$$

When θ_r is small, $\sin \theta_r$ is nearly proportional to r , thus θ_r is approximately a s^1 sector. At larger value of θ_r , however, the deviation from an s^1 sector is not negligible and equation (1) should be applied.

Fig. 2 is a photograph of the sector. The two discs A and A' , of radius of about 4 cm., were cut from a brass plate. The maximum value of θ_r is about 30° . These discs are fixed on an inner race of ball bearing, which is mounted on the holder H with balls between them, and the holder is connected to a shaft S by a spring belt B made of 18-8 stainless steel. The shaft projects from the vacuum through a Wilson seal and is driven by a motor. The sector is rotated at speeds about 200-500 revolutions per minute. The box under the sector

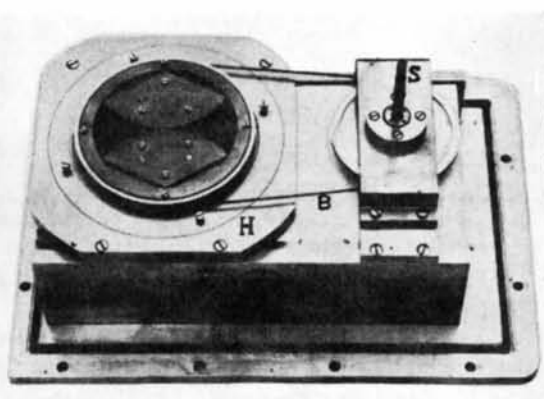


Fig. 2.

carries eight photographic plates 8×12 cm. in size. The upper limit of s on a diffraction photograph is about 30 \AA^{-1} for 50 kV. electrons with the camera length 33 cm.

The accuracy of the exposure time, which is limited by the accuracy of cutting the discs and by the vibration due to the rotation, is lower at inner part than at outer part of the sector. In order to attain an accuracy of 1% at a point 0.5 cm. from the centre, the fluctuation of the radius of the discs, Δr_1 , and the radial amplitude of the vibration, Δr_2 , should be less than 0.01 mm. and 0.02 mm., respectively*. The cutting accuracy of 0.01 mm. is easily attained with a lathe. It is more difficult to keep Δr_2 within the limit, especially when the speed of rotation is high; thus, particular care was required in designing the support of the sector. The values of Δr_2 for the present sector was kept under 0.02 mm. for rotation of about 200 revolutions per minute.

In the case of diffraction by crystals and amorphous materials by both reflexion and transmission methods, the background intensities, caused mainly by multiple

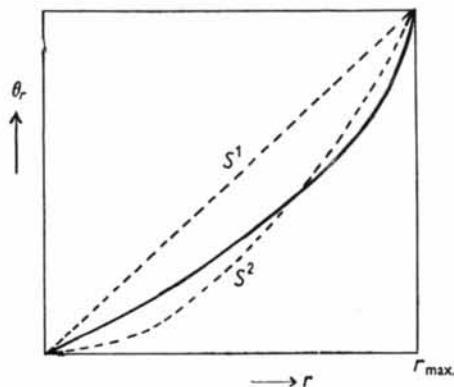


Fig. 3. Aperture of sectors as the function of r . Broken lines: s^1 and s^2 sectors; full line: circular-disc sector of Fig. 1(b). The curves are normalized at r_{\max} .

* These requirements are more severe for sectors of higher powers of s .

scattering, are superposed on the singly-scattered intensities; and since the variation of the background intensities with scattering angle is in general moderate, the decrease of the total intensity with scattering angles is not so rapid as in the case of diffraction by gas molecules. Thus, the s^1 , or approximately s^1 , sector is useful in the electron-diffraction study of solid and liquid substances.

We can suggest here that another double circular-disc sector, as shown in Fig. 1(b) may be useful for samples for which the intensity decrease is more rapid than inverse s^1 . The discs A and A' touch at O , the centre of rotation. The aperture θ_r of the sector as the function of r , is shown in Fig. 3 in comparison with those for the s^1 and s^2 sectors. The double circular-disc sector of this type approximates to the s^1 sector at small values of r and to the s^2 sector at large values of r .

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Crystallographic properties of diammonium phosphate, $(\text{NH}_4)_2\text{HPO}_4$. By J. P. SMITH, J. R. LEHR and W. E. BROWN, *Division of Chemical Development, Tennessee Valley Authority, Wilson Dam, Ala., U.S.A.*

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A rapidly growing interest in diammonium phosphate as a nitrogen-phosphorus fertilizer material prompted a study of its crystallographic properties, which were but partly described in the literature.

Diammonium phosphate forms colorless monoclinic crystals, holohedral class $2/m$. Crystals from the system $\text{NH}_3\text{-H}_3\text{PO}_4\text{-H}_2\text{O}$ usually are tabular to platy on (001), the common forms being {001}, {110} and {101}. The presence of nitrate ion causes elongation along b to produce blade-like crystals. Sulfate ion causes thickening along c to produce equant, distorted bipyramids, the forms {001}, {110} and {101} being equally developed. The presence of ferrocyanide ion along with sulfate ion similarly causes thickening along c to give equant crystals but changes the habit to parallelepipedons with equally developed {001} and {110}, sometimes modified by prominent {201}. Certain aliphatic surfactants introduce strain, with resultant warping of the tabular crystals on (001).

The crystals are biaxial positive, $2V = 80\text{-}85^\circ$ (calc. = 85°),

$$N_\alpha = 1.508, N_\beta = 1.518, N_\gamma = 1.530,$$

$Bx_a = Z = b$, and $X \wedge a = 43.5^\circ$ in acute β . Interfacial angle measurements yield $\beta = 113.5^\circ$. No dispersion was observed.

Rotation and Weissenberg photographs ($\text{Cu } K\alpha$, $\lambda = 1.54 \text{ \AA}$) show the unit cell to be monoclinic with the dimensions

$$a = 8.03, b = 6.68, c = 11.02 \text{ \AA}, \beta = 113^\circ 38'.$$

The only systematic absences apparently are $h0l$ with h odd and $0k0$ with k odd. The most probable space

group is $C_{2h}^5\text{-}P2_1/a$. With $4[(\text{NH}_4)_2\text{HPO}_4]$ per unit cell, the calculated specific gravity, 1.61, agrees quite well with the value 1.62 reported by Schiff (1859) and the value 1.60 calculated from refractive indices through the Gladstone-Dale relationship (Larsen & Berman, 1934).

The comparison in Table 1 shows that the X-ray data

Table 1. Comparison of X-ray and optical goniometric results

$a : b : c$	X-ray	Mitscherlich	Brooke
	1.2021 : 1 : 1.6497	1.1981 : 1 : 1.6546	—
(100) \wedge (001)	$113^\circ 38'$	$113^\circ 14'$	—
(110) \wedge (110)	$95^\circ 23'$	$95^\circ 30'$	$95^\circ 45'$
(110) \wedge (001)	$74^\circ 20'$	$74^\circ 37'$	$74^\circ 10'$
(101) \wedge (001)	$70^\circ 21'$	$70^\circ 16'$	$70^\circ 28'$

yield axial ratios and interfacial angles in good agreement with the optical goniometric data of Mitscherlich and of Brooke as reported by Groth (1908).

The powder diffraction pattern reported by Hanawalt, Rinn & Frevel (1938) is suitable as a means of identification, although these authors listed only the stronger lines.

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