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THE RELATIVE INTENSITIES OF REFLECTION OF X-RAYS FROM THE PRINCIPAL ATOMIC PLANES OF POWDERED SODIUM CHLORIDE*

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ABSTRACT

Measurements have been made on the relative intensities of reflection of the alpha doublet of characteristic molybdenum x-rays, from powdered sodium chloride, using the modified Bragg apparatus described in a previous communication. Determinations were made with (a) filtered rays and (b) rays reflected from a calcite crystal. In a separate investigation the lines were obtained on photographic plates and their intensities were found photometrically. All three series gave results that are in agreement with the measurements of Bragg, James and Bosanquet on large crystals, after their results are corrected for their determinations of the "extinction" in those crystals. Our experiments thus indicate that results on measurements with 325 mesh powder are as free from the effects of "extinction" as are the results of the workers just mentioned.

REFLECTIONS, or more properly diffractions, from crystals are complicated by the phenomenon of "extinction," i.e., an additional absorption at angles at which reflection takes place. It has been pointed out a number of times that the least ambiguous results in measurements of intensities of reflection should be obtained from powders rather than from single large crystals. Darwin[1] has given a detailed discussion of the phenomenon of extinction, analyzing it into two effects, which will be discussed further below.

The work to be described was undertaken with the purpose of developing a technique for obtaining accurate measurements on intensities of reflection from crystal powders. Three series of determinations of the relative intensities of reflection from the principal atomic planes of sodium chloride were made using three different methods. The results have been corrected to indicate the "reflecting power" of the crystal at the observed points, and compared with the corresponding values of

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[1] Darwin, Phil. Mag. **43**, 800 (1922).

Bragg, James and Bosanquet[2] obtained from corrected measurements from single crystals of rock salt.

Two experimental arrangements were tested. The first of these is given diagrammatically in Fig. 1. X-rays from the water cooled Coolidge tube, which was furnished with a molybdenum target, passed through the slit S_1, and the Zirconium filter (Zr), and then to the slit S_2, the upper and lower halves of which could be adjusted as to width separately, the rays from the upper half going to the reference crystal and to the ionization chamber E' to which a Bumstead electroscope was attached. The rays which passed through the lower half of slit S_2 were further limited by slit S_3 and passed on to the pellet of powdered sodium chloride

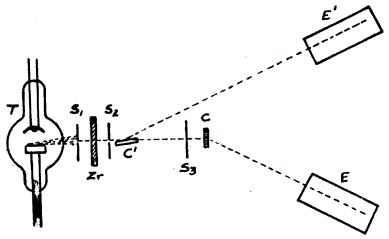

Fig. 1.

C from which the "reflected" rays were caught in the ionization chamber E and measured by a second Bumstead electroscope. The use of the reference crystal in intensity measurements has been described elsewhere.[3] The pellet of powdered salt, C, was continuously oscillated through a small angle by means of an electric motor and a cam designed to yield a uniform angular motion. This produced a greater "randomness" in the orientation of the particles.

A convenient form of flat pellet of the powder (ground to pass through a 325 mesh sieve) was obtained by compressing, in a vice, the sifted material between two closely fitting plungers in a steel cylinder.

With the apparatus as described a series of measurements was made by one of the authors (S. J. B.) on pellets of varying thicknesses of sodium chloride. The averages of several runs are given in column 2 of Table I. The intensity did not vary appreciably with thickness if it was near the optimum value given by the relation t (optimum) $= 1/\mu \sec\theta$, where t is the thickness of the pellet, μ the linear absorption coefficient of the wave-length in question, and θ the incident angle of the rays.

[2] Bragg, James and Bosanquet, Phil. Mag. **41**, 308 (1921); **42**, 1 (1921).
[3] MacInnes and Shedlovsky, Phys. Rev., **27**, 130 (1926).
[4] Glockler and Traub, Phys. Zeits., **22**, 345 (1921).

A second series of measurements was later made by the same investigator using filtered x-rays and substituting a photographic plate for the ionization chambers. The intensities were then calculated from accurate photometric determinations made through the kindness of the Mt. Wilson Observatory, using the relations given by Glockler and Traub.[4] On account of the difficulties of the photographic method, the results were not as reproducible nor as trustworthy as those obtained by other methods. The values are, however, given in column 4 of Table I.

On account of the difficulty experienced with the experimental arrangements just described in correcting for general radiation, which was but

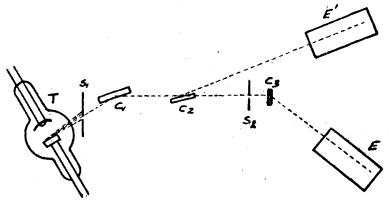

Fig. 2.

partly removed by the zirconium screen, a radically different experimental procedure was adopted. A diagram of the modified apparatus is shown in Fig. 2. X-rays from the molybdenum target of the tube T, after passing through the slit S_1, were reflected from an exceptionally good calcite crystal C_1, which was oriented so as to reflect the alpha doublet of the rays through the slit S_2, from which the resulting substantially monochromatic rays passed to the powdered crystals C_3. The reference crystal C_2 was used as in the experiments already described. It was of interest to see to what extent we were successful in obtaining monochromatic x-rays by this apparatus. For that purpose a rock salt crystal was substituted for the powder and the rays passing through slit S_2 were analyzed. The results were as follows in arbitrary units of intensity: α doublet 100.0, β line 0.00, half wave-length, 2.4, one third wave-length, 1.0. The general radiation was smaller than the experimental error in reading the electroscopes. A third series of intensity measurements with the modified method was made on sodium chloride powder and are given in column 3 of Table I.

The radiation reflected from each plane appears on a photographic plate as a halo, since the numerous particles correctly situated for reflection send out the reflected rays in the form of a hollow cone. If the slit limiting the incident beam were a point, the halo would be a circle. In the case of a rectangular slit, such as was used, the halo is plano convex

near the center and becomes circular at greater angles. The exact shapes of the haloes depend on the size of the slit, the thickness of the powder, the distance from the powder to the plate, and the reflecting angle. The radius of a halo is given by the expression $l \sin 2\theta$, where l is the length of the arm of the ionization chamber and 2θ the chamber angle, or twice the reflecting angle of the rays. The energy measured in the ionization chamber is the fraction of the halo defined by the height of the chamber slit a, that is: $a/2\pi l \sin 2\theta$. In addition, the number of contributing planes from each crystal face must be taken into account. Thus, since $a/2\pi l$ is a constant of the apparatus, the measured values should, therefore, be multiplied by (sin 2θ/number of reflecting planes).

Furthermore, our results with molybdenum rays and the powder and those of Bragg, James and Bosanquet with rhodium rays and single crystals can be more readily compared if both are converted into the corresponding reflecting powers which depend on the crystal lattice alone. This involves multiplying by two terms which occur in the Darwin[5]-Compton[6] equation: first, a polarization correction $(1+\cos^2 2\theta)$; and second, another function of the angle of reflection, which is sin 2θ for a single crystal and sin θ for a powder.

TABLE I

Relative intensities of reflection of x-rays from sodium chloride. [100] = 100

Reflecting plane	Filtered source S.J.B.	Reflected source L.H.	Photographic measurements S.J.B.	Bragg, James & Bosanquet.
[100]	100.	100.	100.	100.
[111]	6.1	4.5	6.8	5.3
[110]	61.	59.	73.	59.5
[222]	39.5	37.5	51.	46.
[200]	28.	29.	24.	31.
[311]	1.4	1.5	——	1.4

Table I gives a summary of the relative, completely corrected, reflecting powers obtained by the different methods outlined in this article, and also the corresponding values given by Bragg, James and Bosanquet, whose nomenclature for indicating the reflecting plane and order has been followed. The first order reflection from the cube face [100] has been arbitrarily given the value 100.

DISCUSSION OF RESULTS

The photographic measurements agree with the others in order of magnitude. They are, however, the least trustworthy because of the aforementioned difficulties with the photographic method.

[5] C. G. Darwin, Phil. Mag. **27**, 675 (1914).

[6] A. H. Compton, Phys. Rev. **9**, 29 (1917).

The other measurements of the relative intensities are in good agreement among themselves and with the values of Bragg, James and Bosanquet, who have used an entirely different method for obtaining their results and also a different wave-length of x-rays.

This latter agreement indicates that our reflections from powders of 325 mesh were as free from "extinction" effects as those obtained from single crystals of rock salt by the method of Bragg, James and Bosanquet.[2] They roughened the surface of their crystals by grinding, reducing what Darwin[1] terms "primary extinction" as much as possible, and corrected for "secondary extinction"* with the aid of measurements made with thin crystal slips of varying thicknesses.

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* "Primary extinction" occurs in homogeneous (perfect) crystals and is due to the diminishing of the energy in the incident beam by interference with doubly reflected rays, which are parallel to, but of exactly opposite phase to the incident beam. " 'Secondary extinction' may be calculated by allowing for the ordinary absorption of the incident beam and in addition subtracting from it the amount of reflection . . . .from homogeneous blocks oriented at the proper angle in the conglomerate crystal." (Darwin, ibid., p. 817).