Acta Cryst. (1962). **15**, 1044

Anomalous dispersion corrections for molybdenum.*

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(*Received* 16 *December* 1961)

A nearly perfect dodecahedron of a single crystal of molybdenum (maximum dimension approximately 200 microns, minimum dimension approximately 180 microns) was examined by means of Mo $K\alpha$ radiation and a single-crystal orienter mounted on an XRD-5 unit, and the relative intensities of 229 reflections from the crystal were measured. Balanced filters of yttrium and zirconium foils were employed, and pulse height analysis was used to discriminate further against unwanted radiation. The 229 reflections were reduced to the 17 independent reflections listed in Table 1 and are shown graphically in Fig. 1. The observed structure factors squared were obtained from the intensities by applying the Lorentz-polarization factors and a spherical absorption correction ($\mu R = 2{\cdot}00$) in the usual manner and averaging the results of the *hkl* permutations over the independent reflections. To calculate structure factors squared and relate them to the observed data, the author considered the following five parameters; real and imaginary anomalous dispersion corrections, a temperature correction, a scale factor, and a secondary extinction correction.

Table 1. *Observed and calculated relative structure factors squared for a single crystal of molybdenum*

hkl	F_o^2	kF_c^2	*hkl*	F_o^2	kF_c^2
110	1532	1507	420	1315	1295
200	1931	1838	332	1251	1195
211	1954	1966	422	1140	1093
220	1918	1956	510, 431	1016	1007
310	1848	1879	521	827	852
222	1813	1782	440	737	784
312	1637	1659	530, 433	695	708
400	1603	1526	442, 600	649	669
330, 411	1414	1408			

Structure factors squared were calculated in the following manner: Anomalous dispersion corrections and a temperature factor were applied to the theoretical scattering curve to yield a calculated scattering curve: i.e.

$$f_c = [(f_{\text{theo}} + \Delta f')^2 + (\Delta f'')^2]^{\frac{1}{2}} \exp(-B \sin^2\theta/\lambda^2) . \quad (1)$$

For a body-centered cubic structure the structure factor is

$$F = 2f_c . \quad (2)$$

Secondary extinction corrections were made in the manner of Pinnock, Taylor & Lipson (1956), i.e.

$$(F_c)^2 = (F)^2/[1 + (g/\mu)Lp(F)^2] , \quad (3)$$

where g is the secondary extinction coefficient, μ is the linear absorption coefficient and Lp is the Lorentz-polarization factor. The calculated structure factors squared were then scaled to the observed structure factor squared,

$$(F_o)^2 = k(F_c)^2. \quad (4)$$

Equation (4) was programmed for an IBM 704 computer so that for given values of f_{theo}, $\sin^2\theta/\lambda^2$, Lp, and $(F_o)^2$ a least-squares analysis could be performed to obtain $\Delta f'$, $\Delta f''$, B, g/μ, and k along with their standard deviations.

The theoretical scattering curve employed for molybdenum was that of Ibers (1959) based on the Thomas–Fermi–Dirac statistical model. The values of $\sin^2\theta$ for Mo $K\alpha$ radiation were computed by using a value of $a_0 = 3{\cdot}146$ Å for molybdenum (Donnay & Nowacki, 1959). Lorentz-polarization values were taken from Vol. II of the *International Tables for X-ray Crystallography*. Another quantity, B in equation (1), is also known and can be employed to reduce the number of parameters from five to four. If a value of 380 °K. is used for the Debye characteristic temperature of molybdenum (James, 1948), the value of B is calculated to be 0·260 Å^2.

Use of the 229 reflections and the complete least-squares matrix gave the following results:

$$\Delta f' = -1{\cdot}68 \pm 0{\cdot}43, \quad \Delta f'' = 0{\cdot}39 \pm 1{\cdot}81,$$
$$g/\mu = 0{\cdot}000200 \pm 0{\cdot}000018, \quad k = 2{\cdot}23 \pm 0{\cdot}14 .$$

The computed values of the structure factor squared are given in Table 1, and are shown graphically in Fig. 1.

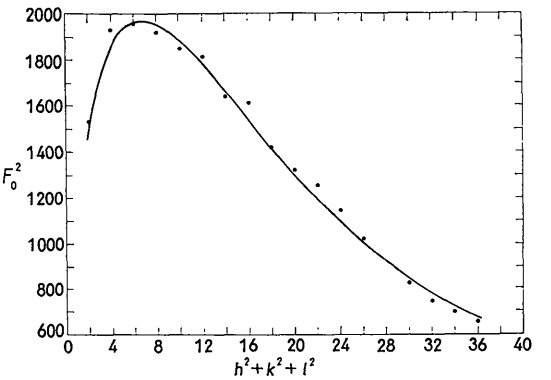

Fig. 1. Observed (.) versus calculated (—) relative structure factors squared for a single crystal of molybdenum.

The results given above for the anomalous dispersion corrections are in good agreement with the values of $\Delta f' = -1{\cdot}7$ and $\Delta f'' = 0{\cdot}9$ given by Dauben & Templeton (1955) for molybdenum and Mo $K\alpha$ radiation. The author concludes that for this particular case the theoretical values of anomalous dispersion are completely verified by the experimental results.

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* Work performed under the auspices of the United States Atomic Energy Commission.