where $f(a, b, c, \ldots)$ is the function, E_f is the probable error in the function, and E_a etc. are the probable errors of the independently measured quantities, the probable errors in the bond lengths and angles have been calculated and are listed in Table 5. Finally, Table 6 shows the comparison of some 630 calculated and observed structure factors for the final three-dimensional IBM calculation.*

Fig. 2 shows the general features of the packing. The three N–Cl distances indicate weak hydrogen bonds, and the calculations of N–Cl–N angles show that the four atoms lie nearly in a plane. An interesting point in this structure is the close approach of both the ring oxygen and the ring nitrogen to the carbonyl oxygen of the next molecule. It seems at least possible that a bifurcated hydrogen bond exists here. The plane of atoms 1, 3, 5, and 6 was calculated and found to be

$$0{\cdot}009075x+0{\cdot}1987y+0{\cdot}1577z-1 = 0\,.$$

Of the ring atoms, the oxygen was found to lie furthest from this plane, at a distance of 0·06 Å. Thus the ring is very nearly planar.

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* Table 6 has been deposited as Document No. 4852 with the ADI Auxiliary Publications Project, Photoduplication Service, Library of Congress, Washington 25, D.C., U.S.A. A copy may be secured by citing the Document number and by remitting $2·50 for photoprints, or $1·75 for 35 mm. microfilm. Advance payment is required. Make checks or money orders payable to: Chief, Photoduplication Service, Library of Congress.

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A Method for Converting Experimental X-ray Intensities to an Absolute Scale

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A relationship between the density of a sample and an integral over the scattered X-ray intensity is derived. The relationship is shown to be useful in adjusting experimental X-ray intensities to an absolute scale.

In nearly all X-ray work the scattered intensity is observed on an arbitrary relative scale. It is often desirable to obtain the intensities on an absolute scale. In the case of an X-ray amorphous scatterer, this has usually been done by assuming that the observed intensity converges at large scattering angles to the sum of the squares of the atomic form factors and incoherent scattering. This procedure is not quite satisfactory, as it requires accurate measurements of the low intensity scattered at large angles. In addition, the observed intensity will still have appreciable undulations in this region, making a curve-fitting procedure somewhat ambiguous. The purpose of this paper is to develop an analytical method, primarily intended for liquid scattering, but which may also be adapted for crystalline powder patterns. The equations are derived for a system containing one kind of atoms, but the generalization to complex systems is straightforward.

A pair distribution function $\sigma(r)$ is defined by

$$\sigma(r) = \int_V \int_f \varrho(\mathbf{p})\varrho(\mathbf{p}+\mathbf{r})\,df\,dV\,, \tag{1}$$

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where $\varrho(\mathbf{p})$ is the electron density at point $\mathbf{p}$, $\int_V dV$ is the integration over the whole volume V of the irradiated sample and $\int_f df$ is an integration over the surface of a spherical shell of radius $r = |\mathbf{r}|$ with the center at $\mathbf{p}$. The total coherent X-ray intensity scattered from the sample will then be:

$$I_t(s) = \int_0^\infty \sigma(r) \frac{\sin sr}{sr} dr , \qquad (2)$$

where s is taken to be $4\pi \sin \theta/\lambda$, 2θ being the scattering angle and λ the wavelength of the radiation.

Since the scattering at zero (and very small) angle, $I_0(s)$, cannot easily be experimentally observed, it should be subtracted from $I_t(s)$. The zero-angle scattering is essentially the scattering from the whole irradiated sample acting as one particle of uniform mean density $\varrho_0 = Nz/V$, where N is the total number of atoms and z is the number of electrons on each atom. Hence $I_0(s)$ is obtained by substituting $\varrho(\mathbf{p})\varrho(\mathbf{p}+\mathbf{r}) = \varrho_0^2$ into equation (1), performing the integration and substituting into equation (2):

$$I_0(s) = \int_0^\infty 4\pi r^2 \varrho_0^2 V \frac{\sin sr}{sr} dr . \qquad (3)$$

(Strictly these equations assume an infinitely large sample. The subsequent error on going to an actual sample size is negligible, however.)

Also the scattering which arises from distances within the same atom will be subtracted. This scattering is given by:

$$Nf^2(s) = N \int_0^\infty \int_V \int_f \varrho_n(\mathbf{p})\varrho_n(\mathbf{p}+\mathbf{r}) \frac{\sin sr}{sr} df dV dr$$
$$= \int_0^\infty \sigma_{\text{at.}}(r) \frac{\sin sr}{sr} dr . \qquad (4)$$

By $\varrho_n(\mathbf{p})$ (and $\varrho_m(\mathbf{p})$) should be understood the contribution to the electron density from atom n (and m respectively) at the point $\mathbf{p}$; $f(s)$ is the atomic form factor and $\sigma_{\text{at.}}(r)$ the pair distribution function involving only distances within the same atom. Hence it is possible to write:

$$I_t(s) - I_0(s) - Nf^2(s) = \alpha N I_e(s) - N(f^2(s) + I_i(s))$$
$$= \int_0^\infty [\sigma(r) - \sigma_{\text{at.}}(r) - 4\pi r^2 \varrho_0^2 V] \frac{\sin sr}{sr} dr, \qquad (5)$$

where αN is a factor which will bring the experimental intensity $I_e(s)$ (corrected for polarization) to an absolute scale and $NI_i(s)$ is the incoherent part of the scattered intensity. A Fourier transform gives:

$$\sigma(r) - \sigma_{\text{at.}}(r) - 4\pi r^2 \varrho_0^2 V$$
$$= \frac{2rN}{\pi} \int_0^\infty [\alpha I_e(s) - f^2(s) - I_i(s)]\, s \sin sr\, ds . \qquad (6)$$

The two first terms on the left side of equation (6) can be written:

$$\sigma(r) - \sigma_{\text{at.}}(r) = \sum_{m \neq n}\sum \int_V \int_f \varrho_n(\mathbf{p}) \varrho_m(\mathbf{p}+\mathbf{r}) df dV ; \qquad (7)$$

the summation being taken over all m different from n. Supposing r is small, then:

$$\sigma(r) - \sigma_{\text{at.}}(r) = 4\pi r^2 \sum_{m \neq n}\sum \int_V \varrho_n(\mathbf{p}) \varrho_m(\mathbf{p}) dV . \qquad (8)$$

If the electron shells of any two atoms m and n do not penetrate into each other, then the product $\varrho_n(\mathbf{p})\varrho_m(\mathbf{p})$ will be zero. Substitution of equation (8) into equation (6) gives:

$$-2\pi^2 \varrho_0^2 V + 2\pi^2 \sum_{m \neq n}\sum \int_V \varrho_n(\mathbf{p}) \varrho_m(\mathbf{p}) dV$$
$$= \int_0^\infty N s^2 [\alpha I_e(s) - f^2(s) - I_i(s)] \frac{\sin sr}{sr} ds . \qquad (9)$$

By letting r go to zero and assuming that there is no overlap of electron shells from different atoms, equation (9) can be written:

$$-2\pi^2 \varrho_0^2 \frac{V}{N} = -2\pi^2 z^2 \frac{N}{V}$$
$$= \alpha \int_0^\infty s^2 I_e(s) ds - \int_0^\infty (f^2(s) + I_i(s)) s^2 ds . \qquad (10)$$

This equation can in principle be used for finding α, since the left-hand side of the equation can be determined separately from the density of the sample. In applying equation (10), the infinity in the upper integration limit must be substituted by the highest experimentally accessible value, s_0. Therefore the usefulness of the equation depends upon how fast the difference of the integrals on the left-hand side converges. This will be considered in more detail in the following.

An expression similar to (10) can be derived from the equations of the atomic center pair distribution:

$$-2\pi^2 \frac{N}{V} = \alpha \int_0^\infty s^2 \frac{I_e(s)}{f^2(s)} ds - \int_0^\infty s^2 \left(1 + \frac{I_i(s)}{f^2(s)}\right) ds . \qquad (11)$$

This equation is exact in so far that there is no overlap of two different atoms. In this case, however, the integrals will converge more slowly toward each other.

For an artificial atomic electron-density distribution, $U_g(a)$, where a is the distance from the center of the atom, and $g(s)$ is the corresponding atomic form factor, one gets by analogy:

$$-2\pi^2 \frac{N}{V} \left[\int_0^\infty U_g(a) da\right]^2$$
$$= \int_0^\infty \alpha \frac{I_e(s)}{f^2(s)} s^2 g^2(s) ds - \int_0^\infty \left(1 + \frac{I_i(s)}{f^2(s)}\right) s^2 g^2(s) ds , \qquad (12)$$

since

$$\int_0^\infty U_g(a)da = z\,. \tag{13}$$

The atomic electron-density distribution $U_g(a)$ is related to the atomic form factor by:

$$U_g(a) = \frac{2a}{\pi}\int_0^\infty sg(s)\sin sa ds\,. \tag{14}$$

Equation (12) is exact for electron-density distributions $U_g(a)$ which are zero for all values of a larger than half the closest approach of two atoms. With this limitation on $g(s)$, it may be used as a weighting function for speeding up the convergence of the right-hand side of equation (12).

An interesting choice for $g(s)$ is $g(s) = f(s)$ when s is smaller than s_0 and $g(s) = 0$ when s is larger than s_0. For this function the left-hand side of (12) will be identical with the left-hand side of (10). To see this one may observe that

$$g(s) = \int_0^\infty U_g(a)\frac{\sin sa}{sa}da \tag{15}$$

and

$$f(s) = \int_0^\infty U_f(a)\frac{\sin sa}{sa}da\,. \tag{16}$$

When s goes to zero, one has

$$g(0) = f(0) = \int_0^\infty U_g(a)da = \int_0^\infty U_f(a)da = z\,. \tag{17}$$

For this choice of $g(s)$ equation (12) can be written:

$$-2\pi^2\varrho_0^2\frac{V}{N} = \alpha\int_0^{s_0} I_e(s)s^2ds - \int_0^{s_0}(f^2(s)+I_i(s))s^2ds\,. \tag{18}$$

Hence this equation does not depend on integration limits except through the discarded overlap term. The assumption about no overlap of electron shells will be better fulfilled for larger values of s_0, however, since the distortion effect of finite integration limits broadens the electron-density distribution. For small values of s_0 the term $\varrho_n(\mathbf{p}).\varrho_m(\mathbf{p})$ is by no means negligible compared with $\varrho_n^2(\mathbf{p})$. Correction for the overlap involves information about the pair distribution function. An approximate correction can be calculated if it is assumed that each atom has a fixed number of nearest neighbors at a fixed distance, coordination number and distance being estimated from other sources.

For the highest values of s_0 easily obtained by standard experimental procedure, the error due to overlap will be negligible. This is demonstrated by an actual application of equation (18) to the experimental X-ray scattering from vitreous boron oxide. The experimental data were obtained by the present author on a Geiger-counter diffractometer with crystal monochromatized Mo $K\alpha$ radiation. Table 1 shows how the normalization factor α varies with the upper integration limit s_0; α is taken to be unity at the largest observed value of s. The dependence on integration limit in this case is surprisingly small, considering that other errors are also involved, such as inaccurate atomic form factors due to incomplete knowledge about the electronic states in the solid.

Table 1. *The factor α, converting relative intensities to absolute intensities, for different values of the integration limit s_0 in the case of vitreous boron oxide*

s_0	α	s_0	α
3	0·803	7	0·992
4	0·934	8	1·017
5	1·038	9	1·010
6	0·994	10	1·000

Note added in proof, 12 *October* 1956.—Through private channels it has been brought to the attention of the author that a similar derivation has been utilized by Norman (1954).

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