

the largest arc which is not resolvable. An equation similar to (40), but having an equality sign, applies to this arc. Two distinct cases arise as in § 5, characterised by the same conditions, and we assume as before that ψ_0 is small. In the first, substitution of Δ from the above equality into equation (25) with $\mu = 0$ (which is the same as (37) with $\nu = 0$) gives values of u and v , from which, by equation (7), we obtain for the lower extremity

$$\sin(\chi + \psi_0) = \cos \psi_0 \tan \varphi_0 \tan \theta + \cos \psi_0 / \cos \theta. \quad (41)$$

The corresponding equation for the second case is

$$\cos(\chi + \psi_0) = \frac{1}{\cos \theta} \left\{ \frac{\tan^2 \psi_0 + \tan^2 \varphi_0}{1 + \tan^2 \varphi_0 + \tan^2 \psi_0} \right\}^{\frac{1}{2}}. \quad (42)$$

An arc which is longer than would be expected from (41) or (42) therefore cannot be a single reflexion.

Finally, it is perhaps worth pointing out that the equations relevant to specimens with fibre orientation may be deduced from the equations given in this paper for the elliptical distribution, with $\varphi_0 = \psi_0$.

Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.

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On the crystal structure of guanidinium bromate. By J. DRENTHE, W. DRENTHE, AAFJE VOS and E. H. WIEBENGA, *Laboratorium voor Anorganische en Fysische Chemie der Rijks Universiteit, Groningen, The Netherlands.*

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In an early X-ray investigation of guanidinium iodide by Theilacker (1935) an improbably small value, 1.18 Å, was reported for the C-N distance in the guanidinium ion. Raman spectra indicate a distance of 1.33 Å (Kellner, 1941).

In order to obtain other X-ray evidence on the structure of the guanidinium ion we attempted to find a guanidinium salt which would allow a simple X-ray determination of its structure by heavy-atom techniques and Fourier syntheses of projections. From among several salts the bromate was selected for further investigation because of its flat unit cell.

Guanidinium bromate, $C(NH_2)_3BrO_3$, seems to be monoclinic with

$$a = 3.77, b = 33.46, c = 9.11 \text{ \AA}; \beta = 99^\circ.$$

The density is 2.16 g.cm.⁻³, requiring 8 molecules per unit cell. It forms twins with common a and b axes, but different c axes. Since reflexions hkl were observed only for $h+k = 2n$, it was assumed that the C face (001) is centred. A Patterson synthesis of the [100] projection excluded the presence of m or 2, leaving Cc as the only possible monoclinic space-group. The presence of the weak reflexions 001 and 005, however, is in contradiction with this space group. This means that the crystals are not actually monoclinic but triclinic. This again is in contradiction with the equality $F_{hkl}^2 = F_{\bar{h}\bar{k}l}^2$, which was observed without any exception on the zero-, first- and second-layer-line Weissenberg photographs about the a axis. The discrepancy can be explained either by assuming that the deviation from monoclinic symmetry is too small to cause an appreciable difference between F_{hkl}^2 and $F_{\bar{h}\bar{k}l}^2$ or by a second twinning, such that each reflexion hkl of one of the twins coincides with $\bar{h}\bar{k}l$ of the other.

Tentatively rejecting the second possibility, the position of the bromine atoms could be easily found from the [100] Patterson projection and a generalized Patterson projection (Cochran & Dyer, 1952) based on the $1kl$ reflexions. The configuration of these atoms was in agreement with the space group Cc ; in addition to this it showed centres of symmetry in the [100] projection. By application of the vector convergence method (Beever & Robertson, 1950), a triangular guanidinium ion and the three oxygen atoms of the BrO_3 -ion appeared in the [100] projection. As a first approximation it was then assumed that the whole structure belongs to the space group Cc and that its [100] projection is centrosymmetrical. A Fourier refinement of the atomic coordinates led to a reliability factor $\Sigma(|F_o| - |F_c|) / \Sigma|F_o|$ of 0.16 for the $0kl$ structure factors. Assuming a flat trigonal guanidinium ion, a C-N distance of 1.34 Å could be deduced with reasonable certainty from the final Fourier synthesis (estimated probable error 0.04 Å).

Attempts to account for the observed reflexions 001 and 005, such that for no hkl the deviation from the equality $F_{hkl}^2 = F_{\bar{h}\bar{k}l}^2$ would exceed the experimental errors, were not successful. It was concluded that a small deviation from monoclinic symmetry exists, which is masked by an approximately 50-50% twinning. Since we were not successful in obtaining single crystals, a further refinement of the atomic coordinates had to be abandoned.

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