

die kurzen Abstände von 3,09 bzw. 3,08 Å hin, die zwischen dem Wassermolekül O(4) und seinen beiden Cl-Nachbarn [Cl(3) ( $x+1, y-1, z$ ) und Cl(3) ( $-x, -y+1, -z$ )] beobachtet werden.

Herrn Dr R. E. Marsh sei an dieser Stelle herzlich gedankt für wertvolle Diskussionen und für sein Interesse am Fortgang der Arbeit.

#### Literatur

BARNET, M. T., CRAVEN, B. M., FREEMAN, H. C., KIME, N. E. & IBERS, J. A. (1966). *Chem. Comm.* p. 307.  
 CHATT, J., DUNCANSON, L. A., GATEHOUSE, B. M., LEWIS, J., NYHOLM, R. S., TOBE, M. L., TODD, P. F. & VENANZI, L. M. (1959). *J. Chem. Soc.* p. 4073.  
 DUCHAMP, D. J. (1964). A.C.A. Meeting, Bozeman, Montana, Paper B-14.  
*International Tables for X-ray Crystallography* (1962). Vol. III, p. 202. Birmingham: Kynoch Press.

JOHNSON, C. K. (1965). *ORTEP*. ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.  
 KAY, M. I. & FRAZER, B. C. (1961). *Acta Cryst.* **14**, 56.  
 LARSON, A. C. (1967). *Acta Cryst.* **23**, 664.  
 NAKAMOTO, K., FUJITA, J. & MURATA, H. (1958). *J. Amer. Chem. Soc.* **80**, 4817.  
 PAULING, L. (1968). *Die Natur der Chemischen Bindung*, 3. Aufl. Weinheim: Verlag Chemie.  
 PROUT, C. K. (1962). *J. Chem. Soc.* p. 4429.  
 SCHAEFER, W. P. (1968). *Inorg. Chem.* **7**, 725.  
 SCHAEFER, W. P. & MARSH, R. E. (1966). *Acta Cryst.* **21**, 735.  
 SHARMA, B. D. & MCCONNELL, J. F. (1965). *Acta Cryst.* **19**, 797.  
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175.  
 THEWALT, U. & MARSH, R. E. (1967). A.C.A. Meeting, Minneapolis, Minn., Paper P-1.  
 WERNER, A. (1910). *Liebigs Ann.* **375**, 1.  
 ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139.

### Short Communications

*Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

*Acta Cryst.* (1970). **B26**, 88

**The crystal structure of 2-thiohydantoin, C<sub>3</sub>H<sub>4</sub>ON<sub>2</sub>S. Erratum.** By LEWIS A. WALKER, KIRSTEN FOLTING and LYNNE L. MERRITT, JR., *Department of Chemistry, Indiana University, Bloomington, Indiana, U.S.A.*

(Received 18 June 1969)

Correction to *Acta Cryst.* (1969). **B25**, 88

The following correction should be made in our paper on 2-thiohydantoin (Walker, Folting & Merritt, 1969):  
 p. 88, col. 2, line 3: instead of  $a=6\cdot635$  read  $a=5\cdot635$ .

#### Reference

WALKER, L. A., FOLTING, K. & MERRITT, L. L. JR (1969). *Acta Cryst.* **B25**, 88.

*Acta Cryst.* (1970). **B26**, 88

**Intensity measurement of high angle integrating Weissenberg reflexions.** By J.W. JEFFERY, *Department of Crystallography, Birkbeck College, Malet Street, London W.C.1, England*

(Received 24 April 1969)

The effect of the integration limits on the measurement of intensities of  $\alpha_1, \alpha_2$  doublets is investigated and the minimum limits required to avoid errors due to the separation are defined.

Errors can arise in the photometry of high angle integrated reflexions, due to  $\alpha_1, \alpha_2$  separation, unless this factor is taken into account in setting the integrating ranges. Of course, if the doublet is treated as a single reflexion, and the greatest measurement across any doublet on a non-

integrating photograph is added to the diameter,  $b$ , of the photometer beam to give the integrating range in that direction, then there will be an area of diameter  $b$ , in the centre of the resulting integrated spot, whose uniform density will be proportional to the total energy in  $\alpha_1 + \alpha_2$ .

Table 1. Spot size and integration limits for selected conditions

$r$  = circumferential spot size for high angle reflexions;  $r'$  = the corresponding measurement for low angles;  $l$  = spot size in the axial direction. Limits for case (a) are calculated for low angle reflexions; case (b) gives the optimum limits; and case (c) the limits required treating  $\alpha_1\alpha_2$  doublets as single reflexions for maximum  $\theta$  (i)  $80^\circ$ , (ii)  $85^\circ$ .

			Integration limits (mm)			
Spot size			Case	Case	Case (c)	
$r$	$r'$	$l$	(a)	(b)	(i)	(ii)
0.2	0.4	0.6	Circ. 0.9	1.4	1.5	2.3
			Long. 1.1	1.45	1.5	1.9
0.5	1.0	1.5	Circ. 1.5	2.0	1.85	2.7
			Long. 2.0	2.5	2.45	2.85

However, this may result in unnecessarily large integration ranges, with consequent long exposures, and possibly in overlapping of neighbouring reflexions. What is required are integrating ranges which enable  $\alpha_1 + \alpha_2$  to be measured as a single reflexion just up to the point where  $\alpha_1$  and  $\alpha_2$  can be measured separately.

The situation at the changeover point is illustrated in Fig. 1.  $l$  is the longitudinal non-integrated spot extension (assumed equal for  $\alpha_1$  and  $\alpha_2$ ) and  $r$  the extension in the perpendicular (circumferential) direction. The inner rectangles represent the area of uniform density for the individual members of the doublet and the overlap of these two and the areas clear of overlap top and bottom must just be large enough to take the photometer beam. If the  $\alpha_1\alpha_2$  separation,  $s$ , is less than the critical value (*i.e.* for smaller  $\theta$ ) the central, overlap, area will be larger and can be photometered; if  $s$  is larger, the two individual areas will be larger and can be photometered separately. For a standard Weissenberg camera the sideways (longitudinal)

separation will be  $s/2$ , as shown for zero layers, and not very different for non-zero layers.

For the critical separation,  $s = b + r$  and the circumferential integration range =  $2(b + r)$ , the longitudinal range =  $s/2 + l + b = 3/2b + l + r/2$ .

The critical value of  $\theta$  can be calculated as follows:

$$s = 2R\Delta\theta \text{ where } R \text{ is the camera radius.}$$

$$\Delta\lambda = 2d \cos \theta \Delta\theta = \lambda \cot \theta \Delta\theta$$

$$\therefore \Delta\theta = \frac{\Delta\lambda}{\lambda} \tan \theta = \frac{s}{2R}$$

$$\therefore \tan \theta = \frac{s}{2R} \cdot \frac{\lambda}{\Delta\lambda} = \frac{b+r}{2R} \cdot \frac{\lambda}{\Delta\lambda} \quad (1)$$

Because of the back reflexion focusing effect,  $r$  for good crystals will be smaller than the corresponding measurement,  $r'$ , for low angle reflexions. If we take  $r = r'/2 = 0.2$ ,  $b = 0.5$  and  $2R = 57.3$  mm, for Cu  $K\alpha$  radiation we have

$$\tan \theta = \frac{0.7}{57.3} \cdot \frac{1.54}{3.82 \times 10^{-3}} = 4.93,$$

$$\therefore \theta = 78.5^\circ.$$

If the integration range required for low angle reflexions ( $r' + b$ ) were to be chosen, it would still be possible to measure all reflexions with  $\theta > 78.5^\circ$  accurately, by taking  $\alpha_1$  and  $\alpha_2$  separately, but there would be a large number with  $\theta < 78.5^\circ$  which would not have sufficient uniform areas to measure either separately or overlapping. When  $\theta$  becomes small enough for adequate overlap area to occur for this integration range, we have:

$$r' + b - (s + r) = b \quad \therefore s = r' - r = r$$

and this is assuming that the focusing effect is still keeping  $r = r'/2$ .

$$\therefore \tan \theta = \frac{r}{2R} \cdot \frac{\lambda}{\Delta\lambda} = 1.41$$

$$\therefore \theta = 54.5^\circ.$$

Some 40% of the observable reflexions would be affected and since  $r$  would have increased the number would be even greater. However, most would only be slightly affected, because the plateau of density at the centre of an integrated spot slopes very gradually at the edges. However, the problem is obviously a serious one and the correct integrating limits should be applied whenever possible. Table 1 gives the integrating limits for  $b = 0.5$  mm and two sets of values of  $r$ ,  $r'$  and  $l$ .  $l$  can be taken as the same for high and low angle reflexions. The limits are given (a) as derived for low angle reflexions, (b) the correct values for all reflexions as derived above and (c) treating all reflexions as single (*i.e.* producing an adequate overlapping uniform area for all reflexions) for  $\theta$  up to (i)  $80^\circ$ , (ii)  $85^\circ$ .

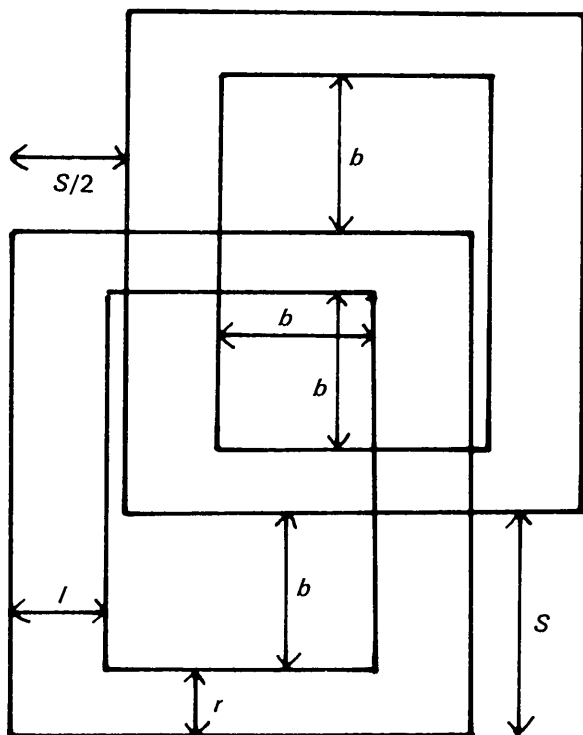


Fig. 1. Diagram of an integrated  $\alpha_1\alpha_2$  doublet at the 'change-over point' between making a single measurement and treating  $\alpha_1$  and  $\alpha_2$  separately.