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## Variations and Covariances of the Anisotropic Temperature Parameters\*

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It is proposed that, in reporting results of crystal structure refinements that include anisotropic temperature parameters, the covariances involving the temperature parameters be expressed in terms of the following six parameters: the lengths of the three principal axes of the vibrational ellipsoids and the orientational angles about these three axes. Equations are presented for deriving the covariances that relate to these new parameters from the covariances given in terms of the primary parameters  $B_{kl}$ .

In reporting the results of a crystal structure analysis that includes adjustments of anisotropic thermal parameters, it is the common practice to list the magnitudes  $U_{ii}$  ( $i=1,2,3$ ) of the principal axes of a vibrational ellipsoid and the direction cosines of these axes relative to a crystal coordinate system with axes  $a_j$ . Thus, twelve quantities – three  $U_{ii}$ 's and nine direction cosines – are presented, of which only six are independent, because of the constraints on the cosines. Along with these twelve derived parameters are often presented their associated standard deviations (and sometimes covariances as well). The three standard deviations  $\sigma(U_{ii})$  of the magnitudes of the principal axes are readily interpretable. It is more difficult, however, to understand the meaning of the nine standard deviations associated with the direction cosines, not only because of the constraints among them but also because of the difficulty of visualization. We suggest here an alternative approach to presenting the uncertainties in the orientations of the principal axes. In particular, we suggest that these uncertainties be represented as uncertainties in the angular displacements  $\alpha_i$  ( $\alpha_1 \equiv \alpha$ ,  $\alpha_2 \equiv \beta$ ,  $\alpha_3 \equiv \gamma$ ) of the ellipsoid around its principal axes. For example, the quantity  $\sigma(\gamma)$  in Fig. 1 represents the uncertainty of orientation of the ellipsoid with respect to a rotation about the eigenvector  $c$ . Note that while  $\alpha = \beta = \gamma = 0$ , the variances and covariances relating to these angles need not be zero.

To derive expressions for variances and covariances

that involve  $\alpha$ ,  $\beta$ , and  $\gamma$ , we presume that the temperature parameters  $B_{kl}$  have been transformed to the coordinate system in which the coordinate axes are parallel to the eigenvectors of the  $B_{kl}$ . This is achieved by a similarity transformation that diagonalizes  $B_{kl}$  (see, for example, Rollett & Davies, 1955; Waser, 1955; Busing & Levy, 1958) by means of a matrix  $G_{jl}$ ,

$$U_{ij} = \sum_k \sum_l G_{ik} G_{jl} B_{kl} = V_i \delta_{ij}. \quad (1)$$

The  $\delta_{ij}$  are Kronecker deltas, and the  $U_{ij}$  are the diagonalized values of the temperature parameters; we have found it convenient to introduce the new symbols,  $V_i = U_{ii}$ .† Variances and covariances are attached to all parameters  $U_{ij}$  including those that are zero. The covariance matrix associated with the  $U_{ij}$  is related to that associated with the  $B_{kl}$  by

$$\begin{aligned} \text{cov}(U_{ij}, U_{kl}) &= \sum_{a,b,c,d} \frac{\partial U_{ij}}{\partial B_{ab}} \frac{\partial U_{kl}}{\partial B_{cd}} \text{cov}(B_{ab}, B_{cd}) \\ &= \sum_{a,b,c,d} G_{ia} G_{jb} G_{kc} G_{ld} \text{cov}(B_{ab}, B_{cd}). \quad (2) \end{aligned}$$

† Important aspects of these matters, including the transformation to main axes in oblique crystal systems, were recently summarized by Cerrini (1971). The matrices  $G_{ik}$  in (1) depend, of course, on whether covariant or contravariant (or mixed) tensor components are chosen; but our results do not depend on the details of the  $G_{ik}$ , and we also do not use index positions to distinguish between covariant, contravariant, or mixed components.

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To find the covariances between the new parameters  $\alpha_i$  and  $\alpha_j$ , and those between  $\alpha_i$  and  $V_j$ , we must transform  $\text{cov}(U_{ij}, U_{kl})$  to the new parameters  $\alpha_i$  and  $V_j$ , by equations that are analogous to the first portion of (2). Among the quantities needed are the partial derivatives of the  $V_j$  with respect to the  $U_{kl}$ , which are equal to unity when the three indices are equal, and zero when any two of them are different,

$$\partial V_j / \partial U_{kl} = \delta_{jk} \delta_{jl} \quad (3)$$

Hence, the variances and covariances for the parameters  $V_i$  are equal to the analogous quantities that pertain to the diagonal components  $U_{ii}$ ,

$$\text{cov}(V_i, V_j) = \text{cov}(U_{ii}, U_{jj}) \quad (4)$$

To obtain the covariances that involve the parameters  $\alpha_k$  we proceed indirectly, by deriving expressions for the derivatives  $\partial U_{ij} / \partial \alpha_k$  rather than  $\partial \alpha_k / \partial U_{ij}$ . Consider the operator  $\mathbf{R}_i(\alpha_i)$  which rotates about the principal axis  $a_i$  of the ellipsoid ( $a_1 \equiv a, a_2 \equiv b, a_3 \equiv c$ ), by an angle  $\alpha_i$  ( $\alpha_1 \equiv \alpha, \alpha_2 \equiv \beta, \alpha_3 \equiv \gamma$ ). The derivatives of the  $U_{ij}$  are defined by the expression

$$\frac{\partial \mathbf{U}}{\partial \alpha_k} = \lim_{\alpha_k \rightarrow 0} \frac{1}{\alpha_k} [\mathbf{R}_k(\alpha_k) \mathbf{U} \mathbf{R}_k^{-1}(\alpha_k) - \mathbf{U}] \quad (5)$$

For example, consider the partial derivatives of the  $U_{ij}$  with respect to  $\alpha$ , a rotation around the  $a$  axis:

$$\begin{aligned} \left( \frac{\partial U_{ij}}{\partial \alpha} \right) &= \lim_{\alpha \rightarrow 0} \frac{1}{\alpha} \left[ \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos \alpha & -\sin \alpha \\ 0 & \sin \alpha & \cos \alpha \end{pmatrix} \begin{pmatrix} V_1 & 0 & 0 \\ 0 & V_2 & 0 \\ 0 & 0 & V_3 \end{pmatrix} \right. \\ &\quad \times \left. \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos \alpha & \sin \alpha \\ 0 & -\sin \alpha & \cos \alpha \end{pmatrix} - \begin{pmatrix} V_1 & 0 & 0 \\ 0 & V_2 & 0 \\ 0 & 0 & V_3 \end{pmatrix} \right] \\ &= \begin{pmatrix} 0 & 0 & 0 \\ 0 & 0 & (V_2 - V_3) \\ 0 & (V_2 - V_3) & 0 \end{pmatrix} \quad (6) \end{aligned}$$

The equations that involve  $\beta$  and  $\gamma$  are analogous and can be written down by suitable cyclic changes in (6).

The substance of our developments can also be obtained by applying first order perturbation methods, as used *e.g.* in quantum mechanics (see *e.g.* Pauling & Wilson, 1935; Franklin, 1968), to the eigenvalues and eigenvectors of the temperature parameter ellipsoid. If we let the  $U_{ij}$  be varied by  $\epsilon_{ij}$  ( $=\epsilon_{ji}$ ), the change of the eigenvalues  $U_{ii} = V_i$  turns out to be  $\delta V_i = \epsilon_{ii}$ . Moreover, an expansion of the change  $\delta \mathbf{x}^{(i)}$  of the (normalized) eigenvector  $\mathbf{x}^{(i)}$  in terms of the original eigenvectors,  $\delta \mathbf{x}^{(i)} = \sum e_{ik} \mathbf{x}^{(k)}$ , with  $e_{kk} = 0$ , leads to  $e_{ik} = \epsilon_{ik} / (V_i - V_k)$ . This result is equivalent to the statement that *e.g.*  $\mathbf{x}^{(1)}$  is rotated about  $\mathbf{x}^{(2)}$  by  $\delta \beta = \epsilon_{12} / (V_1 - V_2)$  and about  $\mathbf{x}^{(3)}$  by  $\delta \gamma = -\epsilon_{13} / (V_1 - V_3)$ .

We now use the relationships (6) to express co-

variances that pertain to the  $U_{ij}$  in terms of covariances that involve the  $\alpha_k$  and then invert the resulting equations. For example, we find that

$$\begin{aligned} \text{cov}(U_{23}, U_{12}) &= \sum_{i,k} \frac{\partial U_{23}}{\partial \alpha_i} \frac{\partial U_{12}}{\partial \alpha_k} \text{cov}(\alpha_i, \alpha_k) \\ &= (V_2 - V_3)(V_1 - V_2) \text{cov}(\alpha, \gamma) \quad (7) \end{aligned}$$

so that

$$\text{cov}(\alpha, \gamma) = \text{cov}(U_{23}, U_{12}) / (V_2 - V_3)(V_1 - V_2).$$

In this way we bypass the singular aspects of (6) and of the analogous expressions for  $\beta$  and  $\gamma$  (their Jacobians vanish). The results are

$$\begin{aligned} \text{cov}(V_1, \alpha) &= \text{cov}(U_{11}, U_{23}) / (V_2 - V_3) \\ \text{cov}(V_1, \beta) &= \text{cov}(U_{11}, U_{13}) / (V_3 - V_1) \\ \text{cov}(V_1, \gamma) &= \text{cov}(U_{11}, U_{12}) / (V_1 - V_2) \\ \text{cov}(V_2, \alpha) &= \text{cov}(U_{22}, U_{23}) / (V_2 - V_3) \\ \text{cov}(V_2, \beta) &= \text{cov}(U_{22}, U_{13}) / (V_3 - V_1) \\ \text{cov}(V_2, \gamma) &= \text{cov}(U_{22}, U_{12}) / (V_1 - V_2) \\ \text{cov}(V_3, \alpha) &= \text{cov}(U_{33}, U_{23}) / (V_2 - V_3) \\ \text{cov}(V_3, \beta) &= \text{cov}(U_{33}, U_{13}) / (V_3 - V_1) \\ \text{cov}(V_3, \gamma) &= \text{cov}(U_{33}, U_{12}) / (V_1 - V_2) \\ \text{cov}(\alpha, \alpha) &= \text{cov}(U_{23}, U_{23}) / (V_2 - V_3)^2 \\ \text{cov}(\alpha, \beta) &= \text{cov}(U_{23}, U_{13}) / (V_2 - V_3)(V_3 - V_1) \\ \text{cov}(\alpha, \gamma) &= \text{cov}(U_{23}, U_{12}) / (V_2 - V_3)(V_1 - V_2) \\ \text{cov}(\beta, \beta) &= \text{cov}(U_{13}, U_{13}) / (V_3 - V_1)^2 \\ \text{cov}(\beta, \gamma) &= \text{cov}(U_{13}, U_{12}) / (V_3 - V_1)(V_1 - V_2) \\ \text{cov}(\gamma, \gamma) &= \text{cov}(U_{12}, U_{12}) / (V_1 - V_2)^2. \end{aligned}$$

If the units of the  $V_i$  are  $\text{\AA}^+2$ , the units of  $\text{cov}(V_i, V_j)$  are  $\text{\AA}^+4$ , those of  $\text{cov}(V_i, \alpha_j)$ ,  $\text{\AA}^+2$ . radian, and those of  $\text{cov}(\alpha_i, \alpha_j)$ , (radian)<sup>2</sup>.

The covariances between the  $V_i$  and  $\alpha_i$ , and the positional coordinates of the atom,  $x_i$ , can be derived in a similar manner if desired. Using the covariances between the original temperature parameters  $B_{ab}$  and the coordinates  $x_k$  as starting point, we first go to the  $U_{ij}$  by a relationship analogous to (2),

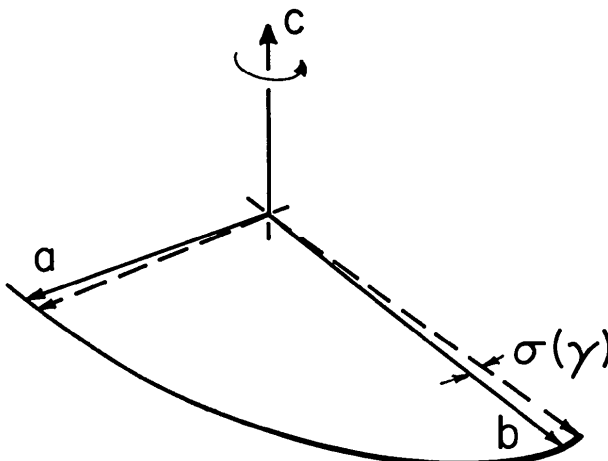


Fig. 1. The standard deviation of  $\gamma$  in relationship to the eigenvectors of the temperature ellipsoid.

$$\text{cov}(U_{ij}, x_k) = \sum_{ab} G_{ia} G_{jb} \text{cov}(B_{ab}, x_k) \quad (8)$$

and thence

$$\text{cov}(V_i, x_k) = \text{cov}(U_{ii}, x_k).$$

To obtain the covariances between  $\alpha$ ,  $\beta$ ,  $\gamma$  and  $x_k$  we employ transformations similar to (7), which give

$$\begin{aligned} \text{cov}(\alpha, x_k) &= \text{cov}(U_{23}, x_k)/(V_2 - V_3) \\ \text{cov}(\beta, x_k) &= \text{cov}(U_{31}, x_k)/(V_3 - V_1) \\ \text{cov}(\gamma, x_k) &= \text{cov}(U_{12}, x_k)/(V_1 - V_2). \end{aligned}$$

In summary, we have derived the transformations that yield covariances of the temperature parameters in terms of the quantities  $V_i$  and  $\alpha_j$ . This system of variables is preferable to two others: (1) to that of the  $V_i$  and the directional cosines of the eigenvectors of the temperature parameters, because the directional cosines are not independent of each other, offsetting the favorable aspect of their geometrical significance; (2) to the system of the  $U_{ij}$ , which are independent of each other (when  $i \leq j$ ), but where the off-diagonal parameters have no intuitively obvious interpretation. In contrast, the  $V_i \equiv U_{ii}$  and  $\alpha_j$  are both independent of each other and subject to easy geometric interpretation.\*

We note that the advantage of using the  $V_i$  and  $\alpha_j$  is one of visualization, and not necessarily mathematical. Either of the covariance matrices, that for the  $U_{ij}$  (with  $i \leq j$ ) and that for the  $V_i$  and  $\alpha_j$ , contain all available covariance information regarding temperature parameters.

### An example

As a numerical example, we choose a carboxylate oxygen atom in the structure of L-N-acetylhistidine monohydrate (Kistenmacher & Marsh, 1971). This compound forms triclinic crystals, space group  $P1$ , with two molecules in the unit cell. The data were collected using Cu  $K\alpha$  radiation out to  $2\theta = 154^\circ$ ; least-squares refinement led to an  $R$  index of 0.029 for 2152 reflections. The standard deviations in the positions of the nonhydrogen atoms are about 0.003 Å. The atom we selected has moderately anisotropic

\* When the variances (*i.e.* the diagonal terms of the covariance matrix) of the variables  $V_i$  and  $\alpha_j$  are dominant, the situation regarding the uncertainties of the temperature parameters is particularly easy to visualize, because essentially all information can be conveyed by the standard deviations of the  $V_i$  and  $\alpha_j$ . The more common case is, however, that there are strong correlations between the errors in at least some of the  $V_i$  and  $\alpha_j$ . The proper assessment of these errors requires not only a knowledge of the standard deviations but of the (off-diagonal) covariances as well. To report only standard deviations in this case would be an oversimplification that could easily mislead and must be guarded against. The problem of variances and covariances has been considered recently by Johnson (1971). He shows for idealized data that at least approximately the covariance matrix of the temperature parameters,  $\text{cov}(B_{ij}, B_{kl})$ , is related to the positional covariance matrix  $\text{cov}(x_i, x_j)$ .

thermal motion; the amplitudes of vibration are approximately equal along the minor and intermediate principal axes of motion, but considerably larger in the direction of the major axis. We report all intermediate results for the purposes of checking computer programs, and for the same reasons include more figures than we necessarily believe to be significant.

$$\begin{aligned} \text{Cell dimensions:}^* \quad a &= 8.865 \text{ (2) \AA} & \alpha &= 102.24 \text{ (1)^\circ} \\ b &= 9.097 \text{ (2)} & \beta &= 90.30 \text{ (1)} \\ c &= 7.346 \text{ (2)} & \gamma &= 117.73 \text{ (1)} \end{aligned}$$

Anisotropic temperature parameters, in the form  $\exp(-b_{11}h^2 \dots - b_{23}kl)$ :

$$\begin{aligned} b_{11} &= 8.968 \times 10^{-3} \\ b_{22} &= 6.945 \\ b_{33} &= 24.172 \\ b_{12} &= 7.071 \\ b_{13} &= -8.135 \\ b_{23} &= 2.435 \end{aligned}$$

The variance-covariance matrix for these parameters is shown at the top of Table 1.

To transform the coefficients  $b_{ij}$  to main axes it is convenient to write the exponent of the temperature

\* The standard deviations of the cell dimensions were not used in the present calculations. The angles  $\alpha$ ,  $\beta$ ,  $\gamma$  are, of course, not related to the angles used to define the orientational variances and covariances of the eigenvectors of the temperature ellipsoid.

Table 1. Covariance matrices of temperature coefficients

	$10^{10} \times \text{cov}(b_{ij}, b_{kl})$					
	$b_{11}$	$b_{22}$	$b_{33}$	$b_{12}$	$b_{13}$	$b_{23}$
$b_{11}$	430.5	-7.5	-199.7	351.3	-7.3	-55.2
$b_{22}$	-7.5	377.2	-166.7	341.9	70.8	121.1
$b_{33}$	-199.7	-166.7	1314.5	-191.1	-124.1	230.4
$b_{12}$	351.3	341.9	341.9	1107.4	149.4	-12.7
$b_{13}$	-7.3	70.8	-124.1	149.4	1804.6	696.7
$b_{23}$	-55.2	121.1	230.4	-12.7	696.7	1711.3

	$10^8 \times \text{cov}(U_{ij}, U_{kl})$					
	$U_{11}$	$U_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
$U_{11}$	36.08	-10.25	-14.71	-2.20	2.18	0.69
$U_{22}$	-10.25	45.03	-17.90	-2.89	0.29	-2.50
$U_{33}$	-14.71	-17.90	100.97	0.43	1.43	-4.08
$U_{12}$	-2.20	-2.89	0.43	23.75	-1.15	1.80
$U_{13}$	2.18	0.29	1.43	-1.15	36.78	-3.20
$U_{23}$	0.69	-2.50	-4.08	1.80	-3.20	38.98

	$10^4 \times \text{cov}(V_i, \alpha_j)$					
	$V_1$	$V_2$	$V_3$	$\alpha_1$	$\alpha_2$	$\alpha_3$
$V_1$	0.00361	-0.00103	-0.00147	-0.00141	0.00435	0.1507
$V_2$	-0.00103	0.00450	-0.00179	0.00513	0.00058	0.1986
$V_3$	-0.00147	-0.00179	0.01010	0.00837	0.00286	-0.0297
$\alpha_1$	-0.00141	0.00513	0.00837	1.639	0.131	2.536
$\alpha_2$	0.00435	0.00058	0.00286	0.131	1.458	1.569
$\alpha_3$	0.1507	0.1986	-0.0297	2.536	1.569	1119

factor in the form  $-(\frac{1}{4}) \sum_{ij} B_{ij} h_i h_j (\mathbf{a}_i \cdot \mathbf{a}_j^*)$ , where a factor  $\frac{1}{2}$  must not be overlooked when going from the  $b_{ij}$  to the  $B_{ij}$  when  $i \neq j$ . Similarly, when  $i=j, k=l$ , each element of the covariance matrix  $\text{cov}(B_{ij}, B_{kl})$  follows directly from the related element of  $\text{cov}(b_{ij}, b_{kl})$ , but when  $i=j$  and  $k \neq l$ , or when  $i \neq j$  and  $k=l$  an additional factor  $\frac{1}{2}$  must be used, and a factor  $\frac{1}{4}$  when  $i \neq j$  and  $k \neq l$ . In addition to transforming to main axes, we divide by  $8\pi^2$ , which yields the diagonal matrix  $U_{ij}$ . The values of the  $U_{ii}$  and of the directional cosines of these eigenvectors relative to the crystallographic axes are:

	<i>a</i>	<i>b</i>	<i>c</i>
$U_{11} = 0.02139 \text{ \AA}^2$	$U_{11} = -0.0586$	$0.8801$	$0.0204$
$U_{22} = 0.02285$	$U_{22} = 0.9359$	$-0.4692$	$0.3441$
$U_{33} = 0.07161$	$U_{33} = 0.3474$	$0.0730$	$-0.9387$

Note that these eigenvectors should form a right-handed system of axes for a proper connection with the sense of rotation around these eigenvectors.

The covariance matrix of the  $U_{ij}$  follows from that of the  $B_{ij}$  by equations (1) and (2). The result is given in the middle of Table 1. The final covariances between the  $V_i$  and  $V_j$ , the  $V_i$  and  $\alpha_j$ , and the  $\alpha_i$  and  $\alpha_j$ , are shown in the bottom matrix of Table 1. The units for the different terms just enumerated are, respec-

tively,  $\text{\AA}^4$ ,  $\text{\AA}^2 \cdot \text{radian}$ , and  $\text{radian}^2$ . Considering just the square roots of the diagonal terms of this matrix we find

$$\begin{aligned} V_1 &= 0.0214 \pm 0.0006 \text{ \AA}^2 & \sigma(\alpha_1) &= \pm 0.7^\circ \\ V_2 &= 0.0228 \pm 0.0007 & \sigma(\alpha_2) &= \pm 0.7 \\ V_3 &= 0.0716 \pm 0.0010 & \sigma(\alpha_3) &= \pm 19. \end{aligned}$$

Of special note is the relatively large value of the standard deviation associated with  $\alpha_3$ , which reflects the near equality of  $V_1$  and  $V_2$ . We emphasize, however, that these standard deviations alone do not give the complete story, because the covariances are by no means negligible.

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## The Crystal Structure of Benzamide

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The structure of benzamide has been refined using counter-measured intensity data (1037 planes). Libration corrections have been applied to the full-matrix least-squares refined parameters. The principal interatomic distances are C–C (mean benzene ring) 1.391 (5), C–O 1.249 (3), C–N 1.342 (3) and C–C (carboamide) 1.501(4) Å.

#### Previous work

Earlier determinations of the crystal structure of benzamide have been reported, by Penfold & White (1959) and Blake & Small (1959). Both of these were based upon two-dimensional photographically recorded data and are consequently, by present day standards, of limited accuracy. The results reported here are of a recent refinement making use of more extended experi-

mental data collected at the Chemistry Department, University of Birmingham.

#### Experimental

An evenly developed crystal of mean dimension 0.2 mm was grown from benzene solution and used for the experimental measurements. The crystal was mounted on the three-circle diffractometer of Small &