Therefore, the hydrogen peaks in a difference Fourier leaving out hydrogen atoms should be only about 60% as high in a noncentric projection as in a centric projection.

The second function to be proposed is a Patterson synthesis using as coefficients:

$$(|\mathbf{F}_{PH1}|-|\mathbf{F}_P|)^2+(|\mathbf{F}_{PH2}|-|\mathbf{F}_P|)^2-(|\mathbf{F}_{PH1}|-|\mathbf{F}_{PH2}|)^2 . \quad \text{II}'$$

The Rossmann function gives the self-vectors of the heavy atoms of derivative one and the self-vectors of the heavy atoms of derivative two as positive peaks and the cross-vectors between one and two as negative peaks. In practice there may be many sites in each derivative, in which case the negative peaks may be obscured by the positive peaks. In Function II the self-vectors have been removed from the Rossmann function and the cross-vectors remain as positive peaks. This function does not involve any additional complications in scaling over the original Rossmann function, although the background will be higher.

Function II may be simplified to give:

$$(|\mathbf{F}_{PH1}|-|\mathbf{F}_P|)(|\mathbf{F}_{PH2}|-|\mathbf{F}_P|) . \quad \text{II}''^{*}$$

It is easy to show that Function II″ is a cross-correlation between derivative one and derivative two, just as the Patterson is a self-correlation.

It should be pointed out that the algebraic sign of the coefficients of Functions I and II can, and frequently will, be negative. This sign must be included in the synthesis. The uses of these two functions and the results of a study of the heavy atom chemistry of triclinic lysozyme will be submitted for publication in the near future.

Reference

ROSSMANN, M. B. (1960). *Acta Cryst.* **13**, 221.

---

[*] This algebra was kindly shown by Dr R. E. Dickerson.

Acta Cryst. (1963). **16**, 318

Refinement of the crystal structure of 6-amido-3-pyridazone.

By PAUL CUCKA, *American-Standard, Research Division, Monroe and Progress Streets, Union, New Jersey, U.S.A.*

(*Received 2 October* 1962)

The structure of 6-amido-3-pyridazone was determined some years ago (Cucka & Small, 1954; I in the following), but the refinement was left incomplete because of the lack of computing facilities. The present article reports the results of a refinement of the structure carried out on an IBM 7090 computer using the least squares program written by Martin, Busing & Levy (1962).

The unit cell and intensity data were the same as used in I. Weights were assigned to the structure factors according to the scheme

$$|F_o| \leq 4|F_{\min}| \qquad \sqrt{w}=|F_o|/4|F_{\min}|$$
$$|F_o| \geq 4|F_{\min}| \qquad \sqrt{w}=4|F_{\min}|/|F_o|$$

$F_{\min}$ being the smallest observed F; zero weights were assigned to unobserved reflections and to the very strong $\bar{2}01$ reflection. Trial parameters were taken from the previous work (I), including estimates for the hydrogen atom positional parameters. Scattering factors for carbon, nitrogen and oxygen were taken from Berghuis *et al.*

Table 1. *Atomic coordinates*

Atom	x/a	$\sigma(x/a)$	y/b	$\sigma(y/b)$	z/c	$\sigma(z/c)$
C_1	0·2943	0·0009	0·4218	0·0005	0·5398	0·0010
C_2	0·2031	0·0011	0·4712	0·0006	0·3618	0·0011
C_3	0·1351	0·0010	0·4154	0·0005	0·1952	0·0011
C_4	0·1479	0·0009	0·3073	0·0005	0·2008	0·0010
C_5	0·0647	0·0008	0·2443	0·0005	0·0196	0·0009
N_1	0·2311	0·0007	0·2578	0·0004	0·3579	0·0007
N_2	0·3021	0·0007	0·3165	0·0004	0·5212	0·0008
N_3	0·0733	0·0007	0·1435	0·0004	0·0346	0·0009
O_1	0·3611	0·0007	0·4643	0·0003	0·7047	0·0007
O_2	0·9904	0·0006	0·2900	0·0004	0·8645	0·0007
H_1	0·196	0·012	0·543	0·008	0·356	0·015
H_2	0·068	0·012	0·436	0·008	0·096	0·016
H_3	0·364	0·013	0·289	0·008	0·613	0·016
H_4	0·385	0·013	0·616	0·007	0·842	0·016
H_5	0·513	0·012	0·401	0·007	0·907	0·015

Table 2. *Bond lengths*

C_1–C_2	1·4270 ± 0·0095 Å	C_2–H_1	0·932 ± 0·105 Å
C_1–O_1	1·2435 ± 0·0077	C_3–H_2	0·822 ± 0·093
C_1–N_2	1·3694 ± 0·0084	N_2–H_3	0·803 ± 0·099
C_2–C_3	1·3397 ± 0·0097	O_1–H_4	2·138 ± 0·096
C_3–C_4	1·4037 ± 0·0091	O_1–H_5	1·901 ± 0·093
C_4–C_5	1·5015 ± 0·0084	N_3–H_4'	0·882 ± 0·096
C_4–N_1	1·3049 ± 0·0078	N_3–H_5'	1·024 ± 0·094
C_5–N_3	1·3100 ± 0·0084		
C_5–O_2	1·2374 ± 0·0071		
N_1–N_2	1·3539 ± 0·0068		

Table 3. *Bond angles*

∠ O_1–C_1–C_2	127·8 ± 1·3°	∠ C_3–C_4–N_1	122·7 ± 1·2°
O_1–C_1–N_2	119·3 ± 1·0	C_3–C_4–C_5	119·7 ± 1·1
C_2–C_1–N_2	114·0 ± 1·0	C_5–C_4–N_1	117·6 ± 1·0
C_1–C_2–H_1	120·4 ± 13·5	C_4–C_5–N_3	118·1 ± 1·0
C_1–C_2–C_3	120·4 ± 1·2	C_4–C_5–O_2	118·5 ± 1·0
H_1–C_2–C_3	119·1 ± 13·7	O_2–C_5–N_3	123·4 ± 1·2
C_2–C_3–H_2	127·4 ± 15·6	N_1–N_2–C_1	126·8 ± 1·2
C_2–C_3–C_4	119·6 ± 1·2	N_1–N_2–H_3	118·9 ± 14·1
H_2–C_3–C_4	112·0 ± 12·1	C_1–N_2–H_3	113·7 ± 13·1
C_4–N_1–N_2	116·2 ± 1·0		

Table 4. *Deviations of atoms from molecular plane*

Atom	Deviation from plane	Estimated error
C_1	0·0035 Å	0·0082 Å
C_2	0·0022	0·0110
C_3	0·0269	0·0104
C_4	0·0001	0·0095
C_5	−0·0347	0·0067
N_1	0·0138	0·0062
N_2	0·0218	0·0072
N_3*	−0·1112	0·0058
O_1	−0·0347	0·0088
O_2*	−0·0021	0·0061

* Not included in determination of molecular plane.

Table 5. *Thermal parameters and their standard deviations*

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}	$U_{\perp}$
C_1	0·0485	0·0396	0·0490	0·0027	−0·0132	0·0048	0·234 Å
δ	0·0037	0·0034	0·0034	0·0031	0·0029	0·0027	
C_2	0·0863	0·0406	0·0511	0·0090	−0·0192	0·0053	0·293
δ	0·0049	0·0035	0·0037	0·0036	0·0035	0·0033	
C_3	0·0817	0·0489	0·0433	0·0109	−0·0333	0·0003	0·295
δ	0·0049	0·0041	0·0032	0·0037	0·0033	0·0030	
C_4	0·0516	0·0365	0·0474	0·0028	−0·0041	0·0041	0·229
δ	0·0038	0·0035	0·0033	0·0028	0·0028	0·0027	
C_5	0·0452	0·0494	0·0309	−0·0042	−0·0116	−0·0010	0·216
δ	0·0037	0·0037	0·0026	0·0032	0·0026	0·0027	
N_1	0·0524	0·0393	0·0324	−0·0010	−0·0119	−0·0012	0·229
δ	0·0032	0·0026	0·0023	0·0026	0·0024	0·0022	
N_2	0·0553	0·0421	0·0409	0·0050	−0·0140	0·0027	0·240
δ	0·0034	0·0031	0·0027	0·0026	0·0024	0·0023	
N_3	0·0582	0·0443	0·0450	0·0003	−0·0173	−0·0041	0·250
δ	0·0034	0·0032	0·0029	0·0026	0·0025	0·0025	
O_1	0·0911	0·0484	0·0443	−0·0044	−0·0280	−0·0041	0·301
δ	0·0036	0·0028	0·0025	0·0026	0·0024	0·0022	
O_2	0·0707	0·0533	0·0417	−0·0077	−0·0222	0·0031	0·269
δ	0·0034	0·0028	0·0024	0·0024	0·0024	0·0020	

(1955), and McWeeny's (1951) scattering factors were used for hydrogen. The refinement was based on F, *i.e.* the function minimized was $\Sigma w(F_o - sF_c)^2$, where s is a scale factor.

The atomic coordinates after 5 cycles of refinement are given in Table 1. Bond lengths and angles are given in Tables 2 and 3. The least squares plane through the atoms C_1, C_2, C_3, C_4, C_5, N_1, N_2 and O_1 is given by

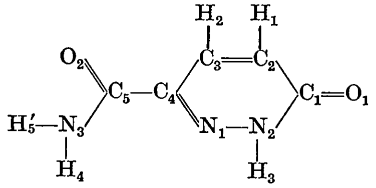
$$0{\cdot}8577 \pm 0{\cdot}0004x + 0{\cdot}0657 \pm 0{\cdot}0010y - 0{\cdot}5100 \pm 0{\cdot}0008z = 0{\cdot}6011 \pm 0{\cdot}0043$$

where the coefficients of x, y and z are the direction cosines of the perpendicular to the molecular plane, and x, y and z are in Å units. The distances of the atoms from this plane are given in Table 4. The thermal parameters U_{ij} relative to the crystal axes are given in Table 5; the final column of this table lists the root mean square amplitudes of the thermal motion in the direction perpendicular to the molecular plane. In all tables the ± limits are equal to one standard deviation as estimated by least squares methods.

The final value of R was 0·131 including unobserved reflections, and 0·106 omitting reflections with zero weight. The correlation matrix shows negligible interactions, except for the interaction between the scale and thermal parameters, where the coefficients reach a value of 0·31 in some cases.

Discussion

The bond lengths and the hydrogen atom positions clearly indicate that the structure has the keto form, I.



(I)

The evidence with regard to the hydrogen atom positions was improved somewhat by running a refinement in which the thermal and positional parameters were allowed to vary and in which a further set of variables a_i was included where the a_i were factors multiplying the hydrogen scattering factors. After three cycles of refinement starting with all $a_i = 1$ the values of the a_i's were all somewhat greater than one, but not significantly so. There are, therefore, regions of positive electron density at the hydrogen atom positions.

The hydrogen bond lengths are 2·911, 2·847 and 2·868 Å for the bonds involving the O_1–N_3', O_1–N_3'', and O_2–N_2' pairs of atoms.

The molecular ring is planar to within the error of measurement but the atoms C_5, O_1 and N_3 deviate slightly but significantly from the molecular plane. This becomes more obvious when the least squares plane is found going through the ring atoms only, when the deviations of the O_1 and C_5 atoms are found to exceed 0·05 Å.

The thermal parameters indicate that there is somewhat greater amplitude of motion perpendicular to the molecular plane than in the plane (the amplitude in the molecular plane can be approximately found from the U_{22} values since the molecule is nearly parallel to the b axis), as would be expected from the easy cleavage parallel to the molecular plane.

The writer is grateful to Drs Martin, Busing and Levy for making their least squares program available to him, and to Mrs J. Root for assistance in the computation.

References

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A.L. (1955). *Acta Cryst.* **8**, 478.

Cucka, P. & Small, R. W. H. (1954). *Acta Cryst.* **7**, 199.

Martin, K. O., Busing, W. R. & Levy, H. A. (1962). Private communication.

McWeeny, R. (1951). *Acta Cryst.* **4**, 513.