motion (maximum principal axes of 10–14 Å^2) results in mean positions for these atoms which are somewhat displaced toward the more distant neighbors. Because of the known presence of absorption errors, no detailed analysis of thermal motion was made. The ellipsoids are reasonable in size and orientation, however, varying from nearly spherical for Ni(1) (3·3 × 3·7 × 3·9) and P(6) (3·0 × 3·6 × 4·2) to highly elongated for nitrogen atoms [4·4 × 6·1 × 14·4 for N(9)] and *para* carbon atoms [3·5 × 4·5 × 10·3 for C(35)].

Thanks are due to Dr L. R. Melby for pointing out the interesting features of this material and for supplying crystals. The author is further grateful to Drs Weiher, Melby, and Benson for a copy of their paper prior to publication and for discussions concerning the magnetic behavior of this and similar materials. Dr Charles T. Prewitt, Mr George McCowan, and Mr Dan Usavage worked closely with the author in the design and construction of the precision Weissenberg camera described.

References

CAVALCA, L., NARDELLI, M. & FAVA, G. (1962). *Acta Cryst.* **15**, 1139.
DAVISON, A., EDELSTEIN, N., HOLM, R. H. & MAKI, A. H. (1963). *J. Amer. Chem. Soc.* **85**, 2029.
DEWAR, M. J. S. & SCHMEISLING, H. N. (1960). *Tetrahedron,* **11**, 96.
EISENBERG, R., IBERS, J., CLARK, R. J. H. & GRAY, H. B. (1964). *J. Amer. Chem. Soc.* **86**, 113.
FREEMAN, A. J. (1959). *Acta Cryst.* **12**, 261.
HOERNI, J. A. & IBERS, J. A. (1954). *Acta Cryst.* **7**, 744.
LIDE, D. R., JR. & MANN, D. E. (1957). *J. Chem. Phys.* **27**, 868.
MCWEENY, R. (1951). *Acta Cryst.* **4**, 513.
PARRISH, W. (1960). *Acta Cryst.* **13**, 838.
PAULING, L. (1960). *Nature of the Chemical Bond,* 3rd Ed. Ithaca: Cornell Univ. Press.
SUTTON, L. E. (1958). *Tables of Interatomic Distances and Configurations in Molecules and Ions.* London: The Chemical Society.
WEIHER, J. F., MELBY, L. R. & BENSON, R. E. (1964). *J. Amer. Chem. Soc.* **86**, 4329.

Acta Cryst. (1966). **20**, 118

The Crystal and Molecular Structure of Hydrolyzed Cocarboxylase*

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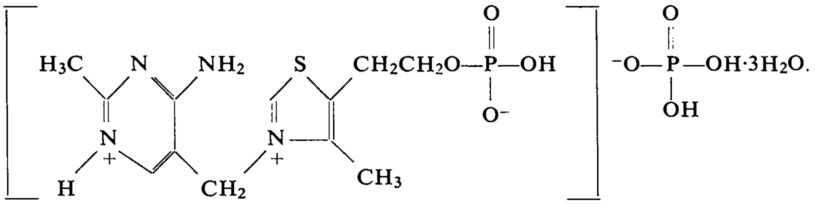
(*Received* 29 *April* 1965)

A material thought to be cocarboxylase was actually found to be hydrolyzed cocarboxylase with the formula



It crystallizes in space group $P\bar{1}$ with two molecules in the unit cell and the following unit-cell parameters:

$$a = 8{\cdot}75\ \text{Å} \qquad b = 10{\cdot}10\ \text{Å} \qquad c = 13{\cdot}25\ \text{Å}$$
$$\alpha = 101^\circ\ 53' \qquad \beta = 108^\circ\ 54' \qquad \gamma = 98^\circ\ 44'$$

The parameters of the organic portion of the molecule are similar to those found in vitamin B_1. The phosphate groups are hydrogen bonded to other phosphate groups and to the water molecules. Only the amino group and the adjacent nitrogen atom of the organic portion are involved in hydrogen bonding. The extra proton was found to be on the nitrogen atom opposite the amino group, as in vitamin B_1, but it does not appear to be involved in any hydrogen bonding.

The structure was solved by obtaining phases directly from the structure factor magnitudes by means of the symbolic addition procedure.

Introduction

Cocarboxylase, the pyrophosphoric acid ester of vitamin B_1, plays a vitally important role in metabolic processes in the form of biochemical decarboxylation of many α-keto acids. It may be obtained naturally from yeast or rice polishings but is more readily produced synthetically (Weijlard & Tauber, 1938).

The structural formula for cocarboxylase is:

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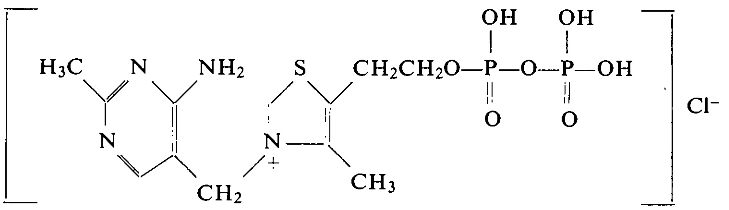
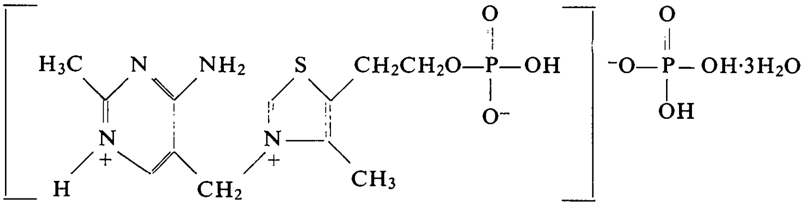
* Presented at the February, 1965, meeting of the American Crystallographic Association.



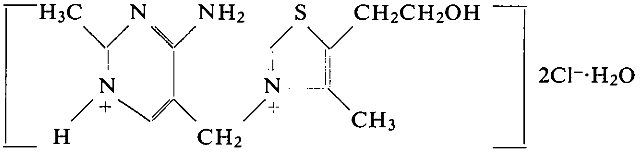
(1)

Although the material under consideration in this study was assumed to have the above formula, determination of the crystal structure showed that it was instead hydrolyzed cocarboxylase, which was found to be:



(2)

It was, nevertheless, an interesting determination from a crystallographic standpoint, especially in the arrangement of the hydrogen bonding. The organic portion closely resembles that for cocarboxylase and the structure provides an interesting comparison to the previously determined structure of the vitamin B_1 molecule (Kraut & Reed, 1962) whose formula is



(3)

Experimental

Material purchased from the Bios Laboratories, Inc., which was labelled 'cocarboxylase'†, was recrystallized from an aqueous solution at room temperature. The crystals were in the form of thin, colorless platelets. Multiple-film equi-inclination Weissenberg photographs using Cu $K\alpha$ radiation were taken with the crystal mounted parallel to the a axis. Visual estimates of the intensities were made with the aid of a calibrated film strip. A total of 2926 independent reflections were observed, 2398 of which were read to have intensities greater than zero.

Cell parameters were determined from precession photographs taken with the crystal mounted on the a^* axis. The space group is triclinic, $P\bar{1}$, with the following unit cell dimensions:

$a = 8{\cdot}75 \pm 0{\cdot}02$ Å $\quad \alpha = 101°53' \pm 20'$

$b = 10{\cdot}10 \pm 0{\cdot}02 \quad \beta = 108\ 54 \pm 20$

$c = 13{\cdot}25 \pm 0{\cdot}03 \quad \gamma = 98\ 44 \pm 20$

$Z = 2 \quad \varrho_{\text{X-ray}} = 1{\cdot}567$ g.cm^{-3} .

The intensities were corrected for Lorentz and polarization factors but no correction was made for absorption. The data were placed on an absolute scale by means of a K curve (Karle & Hauptman, 1953) and both structure factor magnitudes, $|F|$, and normalized structure factor magnitudes, $|E|$, were computed. For a triclinic space group,

$$E_h^2 = F_h^2 / \sum_{j=1}^{N} f_{j\mathrm{h}}^2 \qquad (4)$$

where N is the number of atoms in the unit cell, $f_{j\mathrm{h}}$ is the atomic scattering factor for the jth atom and the F_h^2 have been placed on an absolute scale and corrected for average thermal motion.

The statistical averages listed in Table 1 indicate a centrosymmetric space group. The theoretical values were computed on the basis of a random distribution of atoms in the unit cell.

The distribution of normalized structure factors is given in Table 2.

Table 1. *Statistical averages*

	Experimental	Centro-symmetric	Non-centro-symmetric
$\langle\lvert E\rvert\rangle$	0·802	0·798	0·886
$\langle\lvert E^2-1\rvert\rangle$	0·925	0·968	0·736
$\langle\lvert E\rvert^2\rangle$	1·018	1·000	1·000

Table 2. *Distribution of normalized structure factors*

	Experimental	Centro-symmetric	Non-centro-symmetric
$\lvert E\rvert > 3$	0·2 %	0·3 %	0·01 %
$\lvert E\rvert > 2$	3·2	5·0	1·8
$\lvert E\rvert > 1$	31·5	32·0	37·0

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† Subsequent chemical analysis showed that the original material did not possess any Cl^- but did have a free orthophosphate ion, hence the original material was already hydrolyzed.

Phase determination

The phases for hydrolyzed cocarboxylase were determined directly from the structure factor magnitudes by application of the symbolic addition procedure (Karle & Karle, 1963, 1964, 1965; Karle, Britts & Gum, 1964). It was necessary to assign three phases in order to specify the origin in space group $P\bar{1}$. Positive signs were assigned to three linearly independent reflections having large $|E|$ magnitudes and these reflections were also involved in a large number of interactions in the Σ_2 relationship,

$$sE_{\mathbf{h}} \sim s\sum_{k} E_{\mathbf{k}}E_{\mathbf{h}-\mathbf{k}} \tag{5}$$

where s means 'sign of'. In addition to the three origin specifying phases, seven other reflections were assigned 'letter' phases in order to facilitate the symbolic addition procedure. These reflections were also involved in many Σ_2 interactions and had large $|E|$ magnitudes. The starting set of phases for initiating the symbolic addition procedure is listed in Table 3.

Table 3. *Starting set for application of* Σ_2

h	k	l	$\lvert E\rvert$	Phase
3	$\bar{2}$	8	3·15	+
4	6	1	3·25	+
2	7	$\bar{1}\bar{0}$	2·76	+
3	5	$\bar{8}$	2·98	t
2	5	$\bar{1}\bar{0}$	3·56	s
1	$\bar{1}$	3	2·16	x
6	$\bar{4}$	5	3·05	m
2	$\bar{1}\bar{2}$	4	3·23	n
0	$\bar{1}\bar{0}$	4	2·72	r
6	2	$\bar{2}$	2·40	p

The unknown letters were assigned one at a time as needed. The need to assign an additional letter arises when the phases of a group of reflections are not related to those already determined by (5), or when the indications from (5) are too weak to permit acceptance. The reliability of the sign indications from (5) is given by the probability formula

$$P_+(E_{\mathbf{h}}) \sim \tfrac{1}{2} + \tfrac{1}{2} \tanh\,(\sigma_3/\sigma_2^{3/2}) \sum_{\mathbf{k}} |E_{\mathbf{h}}| E_{\mathbf{k}} E_{\mathbf{h}-\mathbf{k}}\,, \tag{6}$$

where $P_+(E_{\mathbf{h}})$ is the probability that the sign of $E_{\mathbf{h}}$ is positive and

$$\sigma_n = \sum_{j=1}^{N} Z_j^n\,; \tag{7}$$

Z_j is the atomic number of the jth atom in a unit cell containing N atoms. Values of $P_+(E_{\mathbf{h}}) < 0·95$ should be avoided. In most space groups, it is not usually necessary to assign more than three or four letters throughout the entire procedure. In $P\bar{1}$, however, the phase determination is not as readily initiated since there are fewer relationships among the $E(hkl)$ and hence generally fewer combinations contributing to Σ_2 at first. Therefore more unknown symbols may be required initially, although ultimately relationships which reduce the number of unknowns occur among the symbols. In this determination it was eventually possible to express the seven original letter phases in terms of only three: s, p, and x. Relationships such as the following:

$$r = s = t$$
$$p = mn = mnrt$$
$$x = pn = m$$

occurred most frequently throughout the phase determination, in addition to less frequently occurring indications that $p = s = +$. These relationships occur among the many terms contributing to a particular sum in Σ_2.

The most probable combination of signs was, therefore, one derived by letting $p = +$, $s = +$, and $x = -$.

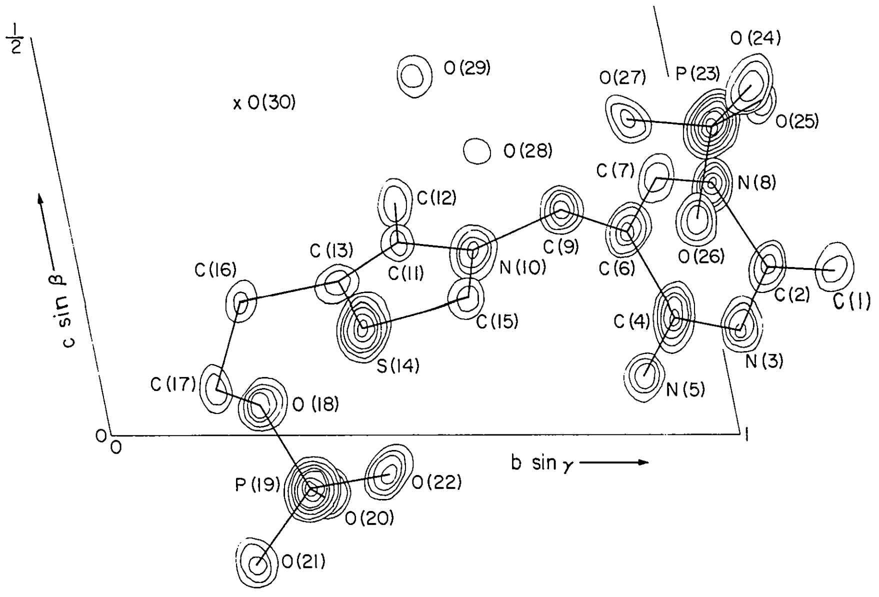

Fig. 1. Sections projected along the a axis from a three-dimensional E map computed from 320 phases determined by the symbolic addition procedure. Contours are on an arbitrary scale and are equally spaced.

The symbol x was chosen to be negative, even though there were no indications to this effect, in order to avoid all positive phases. An E map, a three-dimensional Fourier map with E values rather than F values for coefficients (Karle, Hauptman, Karle & Wing, 1958) was computed with the use of 320 terms ($|E| > 1{\cdot}5$). Sections from this map, projected along the a axis, are illustrated in Fig. 1. Usually 10–12 terms per atom in the asymmetric unit are sufficient to obtain a well resolved E map.

The first and most probable choice of phases, *i.e.* with $p = +$, $s = +$, and $x = -$, proved to be the correct combination, although it was not immediately apparent upon initial inspection of the E map. For cocarboxylase, we had expected four heavy atoms, one S, two P and one Cl, and we also expected the two P atoms to be nearby each other to form the pyrophosphate group. Closer inspection of the E map revealed that we had hydrolyzed cocarboxylase instead of cocarboxylase. Twenty-nine of the 30 atoms in the asymmetric unit were located from this initial map. The remaining water oxygen atom, O(30), was found in a difference map.

The refinement

The coordinates of the 30 atoms in the asymmetric unit were subjected to a least-squares refinement with the ORFLS program (Busing, Martin & Levy, 1962) which was adapted in our laboratory to the IBM 7030 machine (STRETCH). The function which was minimized was $\Sigma w(F_o - F_c)^2$ with unit weights. Individual scale factors were used for each layer. The atomic form factors were obtained from *International Tables for X-Ray Crystallography* (1962).

Several cycles of refinement using all the intensity data and isotropic temperature factors reduced the R index only to 21·3%. A difference map computed at this stage showed regions of strong positive and negative electron density at right angles to each other around all the heavier atoms, which was characteristic of considerable anisotropic motion. Since the number of variables for an anisotropic refinement exceeded the capacity of the program, several cycles of refinement were performed varying the parameters alternately of one-half the atoms at a time. The R value for the observed reflections decreased rapidly to 13·0%.* Further refinement was not attempted although the magnitudes of the shifts indicated that the refinement was not complete. A listing of the fractional coordinates and anisotropic temperature factors may be found in Table 4. An electron density map based on these coordinates is shown in Fig. 2.

A difference map computed at this stage (Fig. 3) revealed the locations of 15 hydrogen atoms. Five hydrogen atoms on the organic group and the six hydrogen atoms of the water molecules were not found. The coordinates of the hydrogen atoms were not refined, but the bond lengths and angles involving these hydrogen positions were all reasonable.

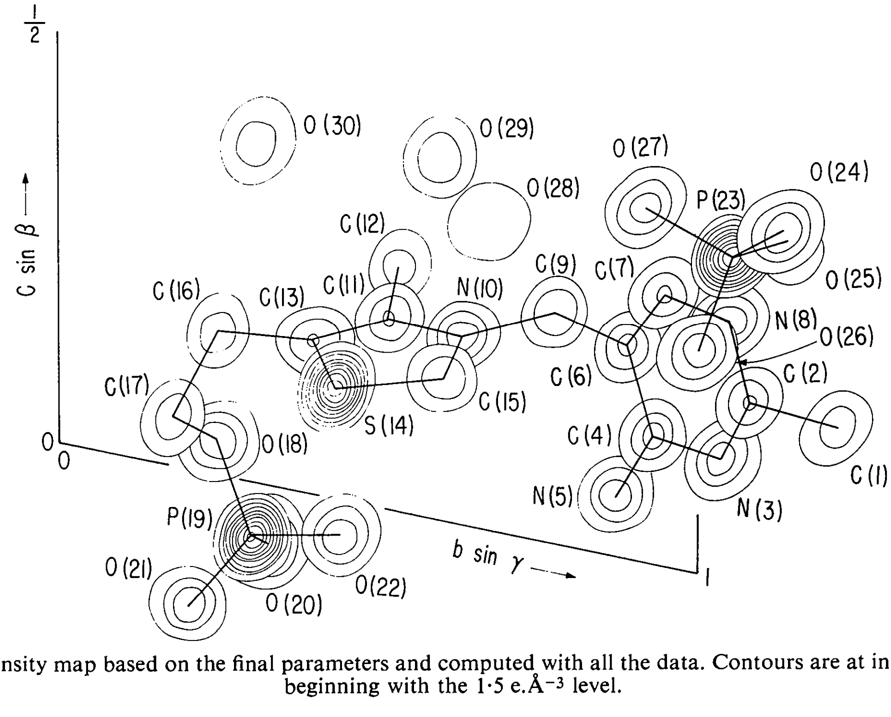

Fig. 2. Electron density map based on the final parameters and computed with all the data. Contours are at intervals of 3·0 e.Å^{-3}, beginning with the 1·5 e.Å^{-3} level.

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* Observed and calculated structure factors have been deposited as Document number 8418 with the ADI Auxiliary Publications Project, Photoduplication Service, Library of Congress, Washington, D.C. 20540. A copy may be secured by citing the Document number and by remitting $3·75 for photoprints, ot $2·00 for 35 mm microfilm. Advance payment is required. Make checks or money orders payable to: Chief, Photoduplication Service, Library of Congress, Washington, D.C., U.S.A.

The structure

The structure determination of hydrolyzed cocarboxylase shows that the pyrophosphate group has been cleaved. The organic portion of the molecule is terminated by an orthophosphate group. In addition to another free orthophosphate group, there are three molecules of water per asymmetric unit. The configuration of the organic portion of the molecule is very similar to that of vitamin B_1 found by Kraut & Reed (1962). The similarity can be seen by comparing Fig. 2 of this paper with Fig. 2(*a*) of Kraut & Reed.

Equations for least-squares planes fitted through the pyrimidine ring and the thiazolium ring are, respectively:

$$-6{\cdot}0001x+6{\cdot}5735y+6{\cdot}7516z=3{\cdot}8601 \qquad (8)$$

and

$$3{\cdot}8605x-2{\cdot}6782y+9{\cdot}3402z=2{\cdot}3853 \qquad (9)$$

where the x, y, and z refer to the triclinic axes and the value on the right is the plane-to-origin distance in Å (Schomaker, Waser, Marsh & Bergman, 1959). For the pyrimidine ring, the r.m.s. deviation of ring atoms from the plane is 0·018 Å with a maximum deviation of 0·028 Å. For the thiazolium ring, the r.m.s. deviation is 0·006 Å with a maximum deviation of 0·007 Å. In addition, the five atoms attached to the two rings are nearly coplanar with the rings. The planes of the pyrimidine and thiazolium rings are at a dihedral angle of 90° which is significantly different from the value of 76° found in vitamin B_1 (Kraut & Reed, 1962).

The bond distances and angles are shown in Fig. 4. The values for the organic portion of the molecule are in general agreement with those found in vitamin B_1.

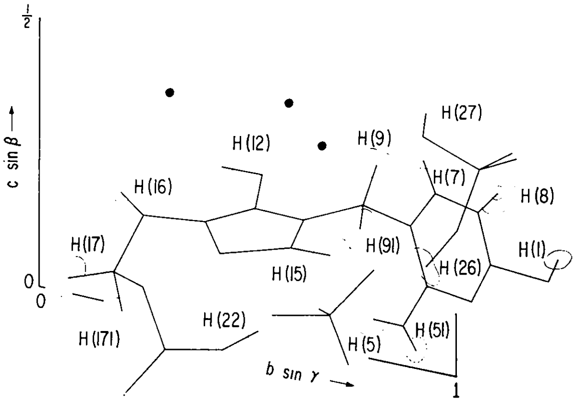

Fig. 3. A difference map showing the location of fifteen hydrogen atoms. The contours are at 0·5 e.Å^{-3} and 1·0 e.Å^{-3} levels except for the dashed contour for H(51) which is at the 0·25 e.Å^{-3} level.

Table 4. *Fractional coordinates* for hydrolyzed cocarboxylase*

The thermal parameters are of the form $[T=\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$. Each thermal parameter is multiplied by 10^4.

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0·9162	1·2148	0·2094	17	112	85	25	37	55
C(2)	0·8009	1·0952	0·2192	49	67	48	36	30	29
N(3)	0·6549	1·0376	0·1448	15	57	32	24	21	27
C(4)	0·5554	0·9298	0·1559	34	53	34	30	24	17
N(5)	0·4109	0·8712	0·0755	21	83	45	36	27	33
C(6)	0·6120	0·8866	0·2562	59	56	34	51	37	29
C(7)	0·7662	0·9466	0·3303	32	71	44	36	33	28
N(8)	0·8641	1·0518	0·3133	37	62	29	33	11	21
C(9)	0·5001	0·7762	0·2785	84	58	49	34	49	28
N(10)	0·5072	0·6321	0·2265	41	61	38	36	13	24
C(11)	0·4120	0·5158	0·2328	27	86	49	36	30	36
C(12)	0·2905	0·5319	0·2956	143	101	120	43	122	46
C(13)	0·4394	0·3966	0·1881	56	88	48	28	22	42
S(14)	0·5936	0·4320	0·1331	167	68	65	50	58	28
C(15)	0·6146	0·6049	0·1757	130	87	54	39	29	28
C(16)	0·3610	0·2505	0·1750	177	69	80	48	27	35
C(17)	0·2315	0·1771	0·0565	66	89	70	68	40	35
O(18)	0·0940	0·2472	0·0410	81	119	40	91	44	45
P(19)	0·0299	0·3028	0·9330	29	70	37	20	22	13
O(20)	0·8617	0·3298	0·9275	24	102	51	44	3	31
O(21)	0·0338	0·2012	0·8345	142	104	46	75	52	8
O(22)	0·1610	0·4411	0·9566	98	83	62	48	65	34
P(23)	0·2939	1·0528	0·3944	25	76	38	38	6	27
O(24)	0·4711	1·1320	0·4350	28	128	72	55	27	58
O(25)	0·1798	1·1363	0·4276	70	110	69	77	23	15
O(26)	0·2288	0·9991	0·2654	166	117	25	55	36	24
O(27)	0·2827	0·9163	0·4329	87	119	99	37	22	82
O(28)	0·0038	0·6761	0·3800	312	335	253	44	211	60
O(29)	0·7455	0·5954	0·4393	266	112	104	69	41	33
O(30)	0·7301	0·3122	0·4158	278	173	161	114	152	86
Standard error:									
C	0·0015	0·0010	0·0009	25	11	8	12	12	7
N	0·0012	0·0008	0·0006	19	8	5	9	8	5
O	0·0013	0·0010	0·0008	23	12	8	13	11	8
P	0·0004	0·0003	0·0002	8	3	2	3	3	2
S	0·0004	0·0003	0·0003	8	3	2	3	3	2

* Coordinates are chosen so that they may be substituted directly into the equations representing the least squares planes.

At this stage of the refinement, differences of the order of 0·035 Å occur in the C(2)–N(8), N(10)–C(11) and C(13)–S(14) bonds. The only larger difference, 0·075 Å, occurs in the C(11)–C(12) bond.

Phosphate groups

Several structure determinations on organic phosphates have been performed in recent years and the results are tabulated in Table 5. The results of this investigation as well as the results of the other investigations shown in Table 5 are in very good agreement with each other. The P–O bonds are classified as to whether they are bonded to an organic group or to a hydrogen atom or are either P=O or $P-O^-$ bonds. It appears that there are distinct differences between these types of bonds. Excluding the values of Dunitz & Rollett (1956) which are consistently lower than the other determinations, the average P–OR bond length is 1·599 Å, the average P–OH bond length is 1·568 Å and the average P–O is 1·505 Å. It also appears that the unprotonated O–P–O angle is several degrees larger than the tetrahedral value.

Hydrogen bonding

The hydrogen bonding illustrated in Fig. 5 was deduced both from the location of many of the hydrogen atoms (Fig. 3) and from the distances between possible donors and acceptors. The extra proton has been found on N(8) opposite the amino group of the pyrimidine ring. This is the same location for the extra proton as in vitamin B_1. However, in this case, the extra proton does not enter into any hydrogen bonding. The distance between N(8) and the nearest oxygen atoms, O(25) or O(30), is 3·26 Å.

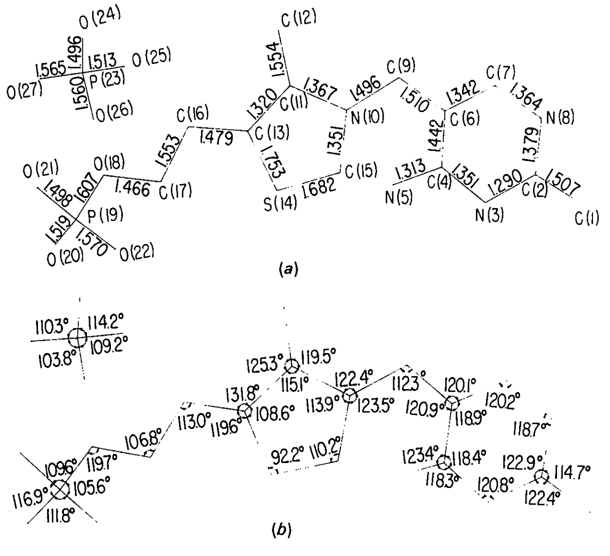

Fig. 4. Bond distances and angles in hydrolyzed cocarboxylase. Four angles omitted in the phosphate groups are: O(18)–P(19)–O(20), 104·8°; O(21)–P(19)–O(22), 107·5°; O(24)–P(23)–O(26), 108·5°; and O(25)–P(23)–O(27), 110·3°. The standard deviations for bond lengths are 0·011 to 0·017 Å and the standard deviations for bond angles are 0·6° to 1·1°.

The six water molecules around a center of symmetry are hydrogen-bonded to each other in the form of a puckered six-membered ring. Each water molecule is hydrogen-bonded to an oxygen atom of the phosphate ion. The phosphate ion is hydrogen-bonded to a phosphate ion across a center of symmetry and to a phosphate group which is attached to the organic portion. The latter phosphate group forms hydrogen bonds across another center of symmetry. The hydrogen bonds between the water molecules and phosphate groups form a continuous network throughout the crystal.

There are only two hydrogen bonds involving the organic portion of the crystal and both of these involve the amino group at N(5). One bond is formed to O(20′) of the phosphate group and the other is formed across a center of symmetry to N(3) of the pyrimidine ring. The N(3)–N(5)″ distance is 3·14 Å, a rather large value

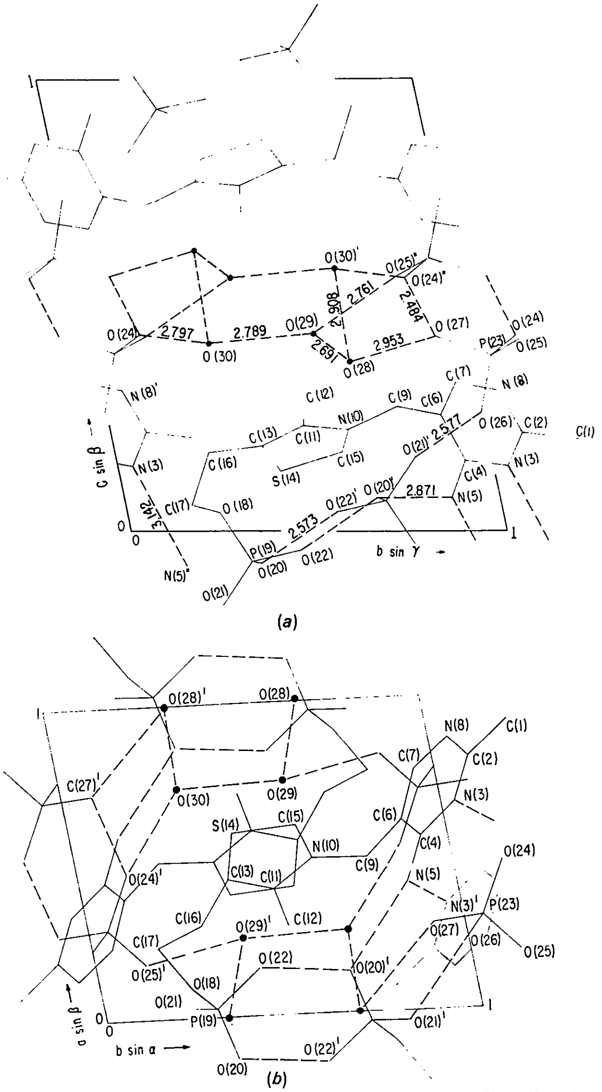

Fig. 5. Packing of molecules and hydrogen bonding in the crystal of hydrolyzed cocarboxylase. (*a*) Projection along the *a* axis. (*b*) Projection along the *c* axis.

for a hydrogen bond. However similarly large values for $-NH_2 \cdots$ ring N bonds have been observed in various other crystals (Fuller, 1959).

The distances and angles of the hydrogen bonds are listed in Table 6. The bond lengths are of the order of magnitude found between similar groups in other crystals. The angles around the oxygen atoms of the water molecules deviate considerably from a trigonal or tetrahedral arrangement.

The only other close approach between atoms is 3·36 Å between O(27) and O(30′).

We are grateful to Dr Jerome Karle for his continuing interest, and we wish to express our appreciation to Mr Stephen Brenner and Dr Donald Mitchell who performed all the calculations.

References

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). ORFLS, Oak Ridge National Laboratory, Oak Ridge, Tennessee, U.S.A.

Dunitz, J. D. & Rollett, J. S. (1956). *Acta Cryst.* **9**, 327.

Fuller, W. (1959). *J. Phys. Chem.* **63**, 1705.

Iitaka, Y. & Huse, Y. (1965). *Acta Cryst.* **18**, 110.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

Karle, I. L. & Karle, J. (1963). *Acta Cryst.* **16**, 969.

Karle, I. L. & Karle, J. (1964). *Acta Cryst.* **17**, 1356.

Karle, I. L. & Karle, J. (1965). *Acta Cryst.* **18**, 345.

Karle, J. & Hauptman, H. (1953). *Acta Cryst.* **6**, 473.

Karle, I. L., Hauptman, H., Karle, J. & Wing, A. B. (1958). *Acta Cryst.* **11**, 257.

Karle, I. L., Britts, K. & Gum, P. (1964). *Acta Cryst.* **17**, 496.

Kraut, J. (1961). *Acta Cryst.* **14**, 1146.

Kraut, J. & Jensen, L. H. (1963). *Acta Cryst.* **16**, 79.

Kraut, J. & Reed, H. J. (1962). *Acta Cryst.* **15**, 747.

Schomaker, V., Waser, J., Marsh, R. E. & Bergman, G. (1959). *Acta Cryst.* **12**, 600.

Trueblood, K. N., Horn, P. & Luzzati, V. (1961). *Acta Cryst.* **14**, 965.

Weijlard, J. & Tauber, H. (1938). *J. Amer. Chem. Soc.* **60**, 2263.

Table 5. *Comparison of bond lengths in organic phosphates*

Bond	Hydrolyzed[a] cocarboxylase		Spermine[b] phosphate hexahydrate	2-amino[c] ethanol phosphate	Adenosine-[d] 5′-phosphate	Calcium[e] thymidylate	Dibenzyl[f] phosphoric acid
P–OR	1·607			1·591	1·610	1·587	1·545 1·566
P–OH	1·570	1·560 1·565	1·589	1·557	1·566		1·545
P–O$^-$	1·498	1·496	1·518	1·503	1·495	1·486	1·469
P=O	1·519	1·513	1·517 1·529	1·493	1·514	1·514 1·474	
∠O–P–O	116·9°	114·2°		117·4°	118·2°		117·2°

(*a*) Present investigation
(*b*) Iitaka & Huse, 1965
(*c*) Kraut, 1961
(*d*) Kraut & Jensen, 1963
(*e*) Trueblood, Horn & Luzzati, 1961
(*f*) Dunitz & Rollett, 1956

Table 6. *Hydrogen bond distances and angles*

Bond	Length	Angle	Degrees
N(5)″H...N(3)′	3·142 Å	N(5)″...N(3)′...C(4)′	124·0°
		C(4)″...N(5)″...N(3)′	117·0
N(5)H...O(20)′	2·871	C(4)...N(5)...O(20)′	128·9
		N(5)...O(20)′...P(19)′	125·0
(phosphate to phosphate)			
O(22)H...O(20)′	2·573	P(19)...O(22)...O(20)′	119·4
		P(19)′...O(20)′...O(22)	121·0
O(26)H...O(21)′	2·577	P(23)...O(26)...O(21)′	118·8
		P(19)′...O(21)′...O(26)	113·3
O(27)H...O(24)″	2·484	P(23)...O(27)...O(24)″	121·8
		P(23)″...O(24)″...O(27)	132·0
(water to phosphate)			
O(28)H*...O(27)	2·953	P(23)...O(27)...O(28)	132·3
O(30)H*...O(24)′	2·797	P(23)′...O(24)′...O(30)	152·3
O(29)H*...O(25)″	2·761	P(23)″...O(25)″...O(29)	129·5
(water to water)			
O(29)H*...O(28)	2·691	O(27)...O(28)...O(29)	141·1
O(28)H*...O(30)′	2·908	O(27)...O(28)...O(30)′	70·1
O(30)H*...O(29)	2·789	O(29)...O(28)...O(30)′	87·1
		O(25)″...O(29)...O(28)	87·6
		O(25)″...O(29)...O(30)	150·3
		O(28)...O(29)...O(30)	102·4
		O(24)″...O(30)′...O(28)	99·4
		O(24)′...O(30)...O(29)	122·8
		O(28)′...O(30)...O(29)	100·4

* The positions of these hydrogen atoms were assumed.