dehydration of crystalline powders of the hydrate showed, as expected, that the water content of the crystal is strongly bound. Several hours of heating at a temperature of about 750 °C. were required to entirely convert a sample to the anhydrous form, which was identified by means of powder diffraction patterns. Additional information was obtained from a differential thermal analysis experiment, which gave evidence that the water content is lost in two stages, the first in the range of temperature between 200 and 300 °C., and the second between 600 and 700 °C. Further data are required for an understanding of the process. However, an explanation is suggested by the structural arrangement. It may be that, at the lower temperature, O_{w2} is expelled from its channels as H_2O, leaving a framework structure of $In(OH)HPO_4$. At the higher temperature, the framework collapses to free the second H_2O, and to recrystallize as $InPO_4$.

It is of interest to recall that $InPO_4$ and $TlPO_4$ are isostructural with $VCrO_4$ and $CuCrO_4$ (Brandt, 1943) and also with an unstable form of $CrPO_4$ (Sullivan & McMurdie, 1952), this on the basis of comparisons with their published d-values. The anhydrous structure type is unrelated to the known forms of $AlPO_4$ or $FePO_4$. However, the only crystals which could be found to be identifiable as isotypes of $InPO_4.2\ H_2O$, $TlPO_4.2\ H_2O$ and $TlAsO_4.2\ H_2O$ proved to be three minerals, phosphates and arsenates of Fe and Al. They are scorodite, $FeAsO_4.2\ H_2O$ (Hiriyama & Sakurai, 1949); strengite, $FePO_4.2\ H_2O$ (Kokkorros,

1938) and very probably mansfieldite, $Al_2O_3.As_2O_5.$ $4\ H_2O$ (Allen, Fahey & Axelrod, 1948).

References

ALLEN, V. T., FAHEY, J. J. & AXELROD, M. (1948). Amer. Min. 33, 126.
BRANDT, K. (1943). Ark. Kemi. Min. Geol. A, 17, No. 6.
CRUICKSHANK, D. W. J. (1949). Acta Cryst. 2, 65.
HIRIYAMA, K. & SAKURAI, K. (1949). X-rays, 5, 85. (Structure Report (1949), 12, 251.)
KOKKOROS, P. (1938). Prakt. Akad. Athen. 13, 337.
MOONEY, R. C. L. (1956). Acta Cryst. 9, 113.
ROGERS, D. & MOFFATT, R. (1956). Acta Cryst. 9, 1037.
SULLIVAN, B. & McMURDIE, H. F. (1952). J. Res. Nat. Bur. Stand. Wash. 48, 159.
WELLS, A. F. (1950). Structural Inorganic Chemistry, 2nd ed. Oxford: Clarendon Press.
Tables of Interatomic Distances in Molecules and Ions (1958). SUTTON, L. E., Editor, London, The Chemical Society, Burlington House, W. I.

The Crystal Structure of 2-Amino-Ethanol Phosphate

BY JOSEPH KRAUT

Department of Biochemistry, University of Washington, Seattle, Washington, U.S.A.

The crystal structure of 2-amino-ethanol phosphate has been solved by Patterson superpositions and refined in three dimensions by difference syntheses and least-squares. The refined parameters include hydrogen positions and isotropic temperature factors, as well as heavy atom positions and anisotropic temperature factors. The refinement was based on 1239 reflections, and was taken to a final R factor of 0·065 for 1000 observed reflections.

Bond lengths and angles are normal. An extensive system of hydrogen bonds is found, the arrangement of which further supports the belief that at least two of the oxygens of the phosphate group are attached by double bonds.

Introduction

2-amino-ethanol phosphate,

$$NH_3^+-CH_2-CH_2-O-PO_3H^-,$$

is a moiety of the brain lipid phosphatidyl ethanolamine, and is also found free in brain tissue. This

study of its structure was undertaken for several reasons. First, it provided a conveniently small, though biochemically important molecule with which to acquire initial experience in the application of automated vector-coincidence methods. A long range study of the possibility of using vector coincidence as an essentially automatic structure-solving tool is

being undertaken in this laboratory. Second, there is no accurate structural information available on the phosphoric acid monoester group. Finally, it was presumed that a zwitterionic compound such as this would contain an interesting hydrogen-bonding system.

Experimental

A sample of 2-amino-ethanol phosphate was purchased from the California Corporation for Biochemical Research and recrystallized from ethanol–water as long prisms. The unit-cell parameters and space group were established by precession photography: space group $P2_1/c$,

$$a=9\cdot04\pm0\cdot02, \quad b=7\cdot75\pm0\cdot02, \quad c=8\cdot86\pm0\cdot02,$$
$$\beta=102° 27' \pm 8'.$$

The prism axis was found to correspond to the unit-cell c axis. The density measured by flotation was $1\cdot562$ and the calculated density $1\cdot546$ g.cm.$^{-1}$, assuming four molecules per unit cell.

Intensity data were collected by planimetering densitometer tracings of reflections on two sets of equi-inclination Weissenberg photographs taken with Cu $K\alpha$ radiation. The first set consisted of reciprocal lattice levels of constant l, from 0 to 7, obtained with a crystal measuring approximately $0\cdot10\times0\cdot13\times0\cdot13$ mm., on a linearly integrating Nonius camera. The second set of photographs contained reciprocal lattice levels of constant k, from 0 to 3, from a crystal measuring approximately $0\cdot26\times0\cdot51\times0\cdot43$ mm. and were taken with a non-integrating Supper camera. Corrections for absorption were not made. All data were put on a common scale and a single scale factor was used throughout the refinement. Of the total of about 1500 reflections within the Cu $K\alpha$ limit, 1239 were examined and of these 233 were too weak to be observed. Six reflections were found to exhibit severe extinction and the corresponding ΔF terms were consequently omitted from difference-Fourier and least-squares computations.

Determination of the structure

A sharpened, origin-removed three-dimensional Patterson was prepared and the approximate coordinates of the phosphorus atom were readily determined by inspection. The four term minimum function based on these coordinates was then computed from the Patterson. That is, if $M(u, v, w)$ represents the minimum function, $P(u, v, w)$ the Patterson function and x, y and z the phosphorus atom coordinates, then

$$M(u, v, w)$$
$$= \min \{P(u-x, v-y, w-z), P(u+x, v+y, w+z),$$
$$P(u+x, v-y-\tfrac{1}{2}, w+z-\tfrac{1}{2}),$$
$$P(u-x, v+y-\tfrac{1}{2}, w-z-\tfrac{1}{2})\}.$$

It is not difficult to show that, since the set of translations imposed on the Patterson function is related by the symmetry elements of space group $P2_1/c$, the resulting function $M(u, v, w)$ must have the symmetry of that space group. This function should, furthermore, reproduce the structure of the crystal, albeit somewhat obscured by the occurrence of fortuitously overlapping Patterson peaks. A three-dimensional model of the superposition map was constructed using the ten highest peaks in the asymmetric unit; it was a relatively simple matter then to pick out the eight peaks representing the structure.

An F_c calculation with these initial atomic positions gave $R=0\cdot27$. Comparison with the final set of refined coordinates shows that most of the atoms had been placed within $0\cdot1$ Å, the largest deviation having been in the position of O5, which was $0\cdot17$ Å in error.

Refinement

The following atomic scattering factors were used throughout the refinement: carbon, nitrogen and oxygen of Berghuis et al. (1955), hydrogen of McWeeny (1951) and phosphorus of Viervoll & Øgrim (1949).

One cycle of refinement based upon coordinate shifts calculated from a three-dimensional F_o synthesis decreased R to $0\cdot20$. At this point an attempt was made to refine the structure further by means of ΔF maps of the (010) and (001) projections, and R was reduced to $0\cdot12$ and $0\cdot14$ for these zones before it became necessary to take account of anisotropy of thermal motion. However, a recalculation of F_c for the full three-dimensional data, using the 'refined' parameters, gave an overall R of $0\cdot21$; i.e., R was increased by $0\cdot01$ by refinement of the projections!

Refinement in three dimensions was then resumed. Six cycles of three-dimensional ΔF syntheses were required to reduce R to $0\cdot09$, using individual anisotropic temperature factors. Hydrogen atoms were now visible in the difference map, as well as indications of further adjustments required in the thermal parameters. Fortunately, however, at this juncture an IBM-709 was installed at the University of Washington's Research Computer Laboratory.

With the aid of a compatibility program, Busing & Levy's (1959) least-squares program ORXLS was adapted for the IBM-709. Least-squares refinement was based on the observed amplitudes, and a weighting scheme similar to Hughes' (1941) was used. That is, σ was taken to be either unity or $F/20$, whichever was larger. Two cycles were run on the heavy atom parameters; changes during the second cycle were negligible. Next, the heavy atoms were fixed and three cycles of refinement of the hydrogen parameters, with individual isotropic temperature factors, were computed.

The hydrogen parameters, initially taken from the last ΔF map, refined much less decisively than those for the heavier atoms, although the magnitudes of the changes did decrease with succeeding cycles.

Table 1. *Positional parameters of heavy atoms and their estimated standard deviations*

	x/a	$\sigma(x/a)$	y/b	$\sigma(y/b)$	z/c	$\sigma(z/c)$
P1	0·2332	0·0001	0·0231	0·0001	0·1130	0·0001
O2	0·3701	0·0003	−0·0766	0·0004	0·1899	0·0005
O3	0·1686	0·0003	−0·0136	0·0005	−0·0549	0·0004
O4	0·1063	0·0004	0·0023	0·0006	0·2058	0·0004
O5	0·2865	0·0003	0·2189	0·0004	0·1333	0·0004
C6	0·1797	0·0006	0·3514	0·0007	0·0742	0·0006
C7	0·2641	0·0006	0·5206	0·0007	0·0708	0·0006
N8	0·3478	0·0004	0·5715	0·0005	0·2284	0·0004

Table 2. *Thermal parameters of heavy atoms and their estimated standard deviations*

β as given here is defined by

$$T = \exp\{-10^{-4}(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)\}$$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
P1	028 ± 01	070 ± 02	058 ± 02	−006 ± 01	−005 ± 01	003 ± 01
O2	045 ± 04	086 ± 07	181 ± 07	004 ± 04	−023 ± 04	022 ± 05
O3	048 ± 03	163 ± 07	076 ± 04	−006 ± 04	015 ± 03	−040 ± 04
O4	064 ± 04	280 ± 11	064 ± 04	−037 ± 05	008 ± 03	022 ± 05
O5	065 ± 04	083 ± 06	102 ± 05	010 ± 04	−018 ± 03	001 ± 04
C6	088 ± 06	099 ± 09	110 ± 07	015 ± 06	−022 ± 05	−007 ± 06
C7	115 ± 07	091 ± 08	078 ± 06	005 ± 06	003 ± 05	007 ± 06
N8	045 ± 04	081 ± 07	076 ± 05	002 ± 04	009 ± 04	007 ± 04

Furthermore, the changes were not large in comparison with the relatively large estimated standard deviations. Finally, R decreased only by 0·01 during the entire process. These considerations cast some doubt on the authenticity of the apparent refinement. One circumstance, however, leads one to believe that the refinement of the hydrogen parameters was, in some degree, meaningful: although H13 was, through a card-punching error, initially misplaced by 0·7 Å, the refinement procedure eventually returned it to the vicinity of its correct original position. Consequently, the final hydrogen parameters resulting from least-squares refinement are presented in Table 3.

An additional cycle of least-squares refinement of the hydrogens was attempted after converting their isotropic temperature factors to the more general matrix form. At the conclusion of the computation, however, the resulting matrices failed to pass the test for positive-definiteness.

The final value of the estimated standard deviation of an observation of unit weight, calculated from the expression

$$(\Sigma w(\Delta F)^2/(m-n))^{\frac{1}{2}}$$

(Peterson & Levy, 1957) was 0·94. This is reasonably close to the ideal value of unity and indicates satis-factory agreement between the weighting scheme used and the distribution of actual experimental error in the observations.

The refined parameters and their estimated standard deviations are given in Tables 1, 2 and 3. A final round of F_c calculations based on these parameters resulted in an R factor of 0·065, excluding unobserved reflections, or 0·076 if all reflections are included except the six showing extinction. Table 4 contains the observed and calculated structure factors.

Discussion

Fig. 1 shows a single molecule within the structure and the arrangement of hydrogen bonds between it and adjacent molecules. In terms of overall conformation, the molecule has assumed a *gauche* arrangement about the C6–C7 bond instead of the more usual zig-zag arrangement of linear molecules.

Tables 5 and 6 contain the bond distances and angles calculated from the parameters of Tables 1 and 3. The standard deviations were estimated using the simplifying assumption of isotropic error in the positional parameters and orthogonal coordinates. Equation (10) of Darlow (1960) was used to obtain the standard deviations of the bond angles.

Table 3. *Hydrogen atom parameters and their estimated standard deviations*

	x/a	$\sigma(x/a)$	y/b	$\sigma(y/b)$	z/c	$\sigma(z/c)$	B	$\sigma(B)$
H9	0·281	0·006	0·552	0·007	0·288	0·006	1·2	1·1
H10	0·366	0·006	0·695	0·007	0·220	0·006	0·9	1·0
H11	0·439	0·007	0·533	0·008	0·277	0·007	1·8	1·2
H12	0·017	0·011	−0·018	0·012	0·152	0·010	6·9	2·3
H13	0·098	0·008	0·404	0·011	0·154	0·009	4·1	1·9
H14	0·123	0·006	0·314	0·008	−0·032	0·006	1·2	1·1
H15	0·168	0·007	0·597	0·009	0·028	0·007	2·0	1·3
H16	0·335	0·007	0·516	0·008	0·006	0·007	1·7	1·2

The phosphate group may be compared with that of the diester dibenzyl phosphoric acid, studied by Dunitz & Rollett (1956). The two groups show about the same deviations from tetrahedral symmetry. There is, however, a small but possible significant increase of approximately 0·03 Å in the lengths of the P–OC and the P–O bonds in 2-amino-ethanol phosphate.

The hydrogen bonding system about the phosphate group contains some interesting features: the two hydrogen bonds from O2 to N8' and N8" respectively

Table 4. *Observed and calculated structure factors*

Reflections are grouped in subtables of constant l. Within each subtable the columns contain, respectively, h, k, $10F_o$ and $10F_c$. The symbol * denotes the minimum observable for reflections too weak to be observed, and x denotes reflections presumed to exhibit extinction and therefore not included in the refinement

$l = 0$

h	k	10Fo	10Fc		h	k	10Fo	10Fc		h	k	10Fo	10Fc
0	2	774x	1174		3	6	256	271-		6	7	106	111-
0	4	524	573		3	7	100	98		6	8	65	64-
0	6	172	157		3	8	117	112-		7	0	183	174-
0	8	64	53		3	9	55	59		7	1	19	18-
1	0	108	117		4	0	100	83-		7	2	244	295-
1	1	228	244-		4	1	8*	3-		7	3	64	55
1	2	174	185		4	2	64	55-		7	4	40*	6
1	3	343	376		4	3	241	221		7	5	123	120
1	4	157	151		4	4	267	260		7	6	34*	17
1	5	33*	34		4	5	38*	7-		7	7	86	89
1	6	193	196		4	6	184	185		8	0	205	212
1	7	118	112-		4	7	72	69		8	1	77	74-
1	8	92	82		4	8	137	109		8	2	223	228
1	9	153	150-		4	9	48	47		8	3	78	75
2	0	715x	1201-		5	0	90	75		8	4	132	114
2	1	246	275-		5	1	188	173-		8	5	63	66
2	2	380	405-		5	2	210	200		8	6	27*	21
2	3	398	439-		5	3	253	246-		9	0	85	74
2	4	389	392-		5	4	59	57-		9	1	80	74
2	5	87	83-		5	5	255	266-		9	2	167	179
2	6	169	170-		5	6	59	53		9	3	42	40
2	7	404	34-		5	7	59	97-		9	4	72	83
2	8	91	82-		5	8	29*	8		9	5	27*	2
2	9	28*	23		6	0	139	91-		10	0	91	107-
3	0	49	28-		6	1	151	138		10	1	9*	1
3	1	296	300		6	2	163	156-		10	2	129	137-
3	2	222	221-		6	3	46	38-		10	3	116	123-
3	3	130	117		6	4	204	221-		10	4	43	39-
3	4	136	124-		6	5	40*	20		11	0	29	12
3	5	210	205		6	6	115	119-		11	1	29	27-
										11	2	60	71-

$l = 2$

h	k	10Fo	10Fc		h	k	10Fo	10Fc		h	k	10Fo	10Fc
0	0	152	180-		3	9	80	71		6-	3	177	176
0	1	281	308-		3-	0	720x	912-		6-	4	45*	24
0	2	83	88-		3-	1	130	130		6-	5	183	184
0	3	145	139		3-	2	295	283-		6-	6	104	99
0	4	87	84		3-	3	327	344		6-	7	75	78
0	5	113	105-		3-	4	278	262-		6-	8	27*	34
0	6	213	200		3-	5	98	94		7	0	383	393
0	7	156	151-		3-	6	109	108-		7	1	92	85-
0	8	98	102		3-	7	79	69		7	2	348	366
0	9	160	151-		3-	8	40*	43-		7	3	87	88
1	0	596x	806-		3-	9	28*	15		7	4	175	189
1	1	244	249-		4	0	241	221		7	5	66	72
1	2	235	234-		4	1	181	164-		7	6	34*	39
1	3	340	362-		4	2	292	299-		7	7	96	105
1	4	356	353-		4	3	196	183-		7-	0	198	164-
1	5	61	55-		4	4	42*	37-		7-	1	75	72-
1	6	169	165-		4	5	248	248-		7-	2	228	228-
1	7	45*	13-		4	6	45*	28		7-	3	73	67
1	8	96	90-		4	7	75	80-		7-	4	225	236-
1	9	31*	29		4	8	34*	14-		7-	5	44*	1
2	0	144	133-		4-	0	481	512		7-	6	138	149-
2	1	355	386		4-	1	198	195-		7-	7	85	88
2	2	240	240-		4-	2	229	228		8	0	65	56
2	3	240	249		4-	3	60	47-		8	1	84	86
2	4	106	97-		4-	4	148	147		8	2	201	210
2	5	303	310		4-	5	134	130-		8	3	65	67
2	6	235	225-		4-	6	100	105-		8	4	117	124
2	7	123	125		4-	7	98	101-		8	5	33*	25
2	8	99	91-		4-	8	37*	40-		8	6	84	89
2	9	59	54		4-	9	87	81-		8-	0	319	293-
2-	0	435	445-		5	0	124	115-		8-	1	11*	10
2-	1	166	177		5	1	184	170		8-	2	311	328-
2-	2	223	209-		5	2	141	142-		8-	3	78	73-
2-	3	274	296-		5	3	9*	5-		8-	4	95	95-
2-	4	157	149-		5	4	222	225-		8-	5	98	107-
2-	5	40*	10-		5	5	45*	34		8-	6	67	53-
2-	6	44*	24-		5	6	97	94-		9	0	196	211-
2-	7	125	130		5	7	119	117-		9	1	10*	1
2-	8	41*	15-		5	8	55	50-		9	2	233	228-
2-	9	166	161		5-	0	437	472		9	3	157	163-
3	0	53	45-		5-	1	39	35-		9	4	99	100-
3	1	36	24-		5-	2	358	369		9	5	112	141-
3	2	42	36-		5-	3	259	243-		9-	0	12*	26
3	3	196	195		5-	4	389	396		9-	1	59	62
3	4	331	322		5-	5	45*	48-		9-	2	94	82
3	5	43*	8-		5-	6	221	238		9-	3	33	29-
3	6	220	205		5-	7	83	89-		10	0	99	97-
3	7	83	82		5-	8	118	120		10	1	26	20-
3	8	118	118		6	0	83	77-		10	2	187	198-
					6	1	57	51-		10	3	32	31
					6	2	198	201-		10-	0	84	87
					6	3	36	30-		10-	1	58	39-
					6	4	45*	42		10-	2	157	147
					6	5	72	71		11-	0	40	46-
					6	6	40*	33		11-	1	8*	6-
					6	7	63	64		11-	2	82	78-
					6-	0	160	155		11-	3	63	73
					6-	1	116	109					
					6-	2	246	237					

$l = 1$

h	k	10Fo	10Fc		h	k	10Fo	10Fc		h	k	10Fo	10Fc
0	1	376x	477		3	6	186	196		6	5	49*	9-
0	2	110	117-		3	7	48*	50-		6	6	45*	34
0	3	335	419-		3	8	117	119		6	7	39*	54-
0	4	93	77-		3	9	31*	19		6-	1	19*	5-
0	5	256	246		3-	1	190	170-		6-	2	12	8-
0	6	251	248-		3-	2	231	236-		6-	3	134	102
0	7	40*	3-		3-	3	331	347-		6-	4	90	82
0	8	84	78-		3-	4	39*	34-		6-	5	71	74-
0	9	80	69		3-	5	261	246-		6-	6	74	97
1	1	117	97-		3-	6	188	195-		6-	7	41*	9-
1	2	17	33		3-	7	89	78-		6-	8	140	146
1	3	62	82		3-	8	48	50-		7	1	177	171
1	4	160	150-		3-	9	59	52-		7	2	84	78
1	5	97	95-		4	1	44	39		7	3	49	40
1	6	205	222-		4	2	237	240-		7	4	131	134
1	7	49*	43		4	3	87	80		7	5	46*	48
1	8	78	76-		4	4	44*	20-		7	6	98	109
1	9	55	47-		4	5	47*	52		7	7	30*	13-
1-	1	356	380		4	6	126	123-		7-	1	422	418-
1-	2	62	20		4	7	47*	21		7-	2	80	68-
1-	3	373	395		4	8	39*	42-		7-	3	271	286-
1-	4	36*	22		4-	1	60	49		7-	4	49*	28
1-	5	277	268		4-	2	50	35-		7-	5	101	103-
1-	6	206	217		4-	3	113	86-		7-	6	43*	41-
1-	7	47*	38		4-	4	166	174-		7-	7	86	79-
1-	8	58	65		4-	5	165	162		8	1	241	249
1-	9	83	72		4-	6	126	130-		8	2	56	45
2	1	349	385-		4-	7	48*	29		8	3	207	191
2	2	228	248		4-	8	70	76-		8	4	78	90
2	3	356	411-		4-	9	37	46		8	5	31*	23
2	4	38*	9		5	1	31	11-		8	6	31*	23
2	5	273	265-		5	2	80	60-		8-	1	57	47-
2	6	204	207		5	3	141	129		8-	2	43	30-
2	7	49*	44-		5	4	142	140-		8-	3	83	128-
2	8	78	76		5	5	67	70-		8-	4	67	79-
2	9	45	44-		5	6	136	144-		8-	5	71	74-
2-	1	61	37-		5	7	44*	6		9	1	27	16
2-	2	68	59		5	8	168	177-		9	2	110	95-
2-	3	28	32		5-	1	359	358		9	3	35	27
2-	4	204	200		5-	2	248	252		9	4	120	140-
2-	5	117	116-		5-	3	293	326		9-	1	157	155
2-	6	195	192		5-	4	74	67		9-	2	51	42-
2-	7	49*	30		5-	5	176	165		9-	3	155	123
2-	8	45*	44		5-	6	134	137		10	1	151	152-
2-	9	55	61-		5-	7	80	74		10	2	37	32-
3	1	268	253-		5-	8	37*	39		10	3	148	158-
3	2	96	85		6	1	169	165-		10-	1	44	34
3	3	357	386-		6	2	73	77		10-	2	108	92
3	4	212	211		6	3	106	100-		10-	3	58	53
3	5	45*	5		6	4	49*	49-		11-	1	37	30-
										11-	2	26	25

$l = 3$

h	k	10Fo	10Fc		h	k	10Fo	10Fc		h	k	10Fo	10Fc
0	1	438	484-		3	6	35*	40-		6	5	33*	37
0	2	11	4		3	7	82	80		6	6	84	90
0	3	141	143-		3	8	27*	1		6	7	22*	21-
0	4	128	127-		3	9	15*	16		6	1	401	408
0	5	146	148-		3-	1	350	332-		6-	2	172	163
0	6	236	242-		3-	2	157	149		6-	3	366	378
0	7	35*	7		3-	3	200	189-		6-	4	35*	34
0	8	107	106-		3-	4	218	233		6-	5	197	193
0	9	56	46-		3-	5	176	164-		6-	6	69	75
1	1	24	12		3-	6	25*	18-		6-	7	86	98
1	2	74	69		3-	7	37*	16		6-	8	20*	3-
1	3	98	81-		3-	8	49	44		7	1	217	223
1	4	105	98-		3-	9	66	56-		7	2	27	15
1	5	119	113-		4	1	85	85-		7	3	175	160
1	6	130	114		4	2	131	121-		7	4	54	57
1	7	35*	24		4	3	80	70		7	5	75	84
1	8	63	54		4	4	170	171-		7	6	24*	2-
1-	1	22*	21-		4	5	98	93-		7-	1	11*	10-
1-	2	76	66-		4	6	63	53-		7-	2	63	43
1-	3	41	28-		4	7	31*	12-		7-	3	53	43
1-	4	27	27-		4	8	168	164-		7-	4	35*	36
1-	5	31*	12		4-	1	9*	12		7-	5	89	94-
1-	6	177	178-		4-	2	140	128-		7-	6	30*	49
1-	7	112	112-		4-	3	199	194-		7-	7	64	44-
1-	8	66	64-		4-	4	31*	30-		8	1	42	37
1-	9	26	26		4-	5	178	166-		8	2	91	81-
2	1	440	451		4-	6	146	150-		8	3	38	38
2	2	161	152		4-	7	75	70-		8	4	115	125-
2	3	77	76		4-	8	27*	31-		8	5	23*	24
2	4	254	247		4-	9	17*	37-		8-	1	373	381-
2	5	186	180		5	1	240	244-		8-	2	64	54-
2	6	228	236		5	2	77	73		8-	3	250	271-
2	7	34*	24		5	3	171	166-		8-	4	33*	17
2	8	134	135		5	4	74	78-		8-	5	111	101-
2	9	41	43		5	5	88	80-		8-	6	26*	23-
2-	1	180	194-		5	6	32*	4		8-	7	16*	4-
2-	2	31	26-		5	7	108	104-		9	1	196	191-
2-	3	37	26-		5	8	18*	32-		9	2	39	33-
2-	4	27*	13		5-	1	283	263		9	3	175	177-
2-	5	85	72		5-	2	80	64-		9	4	18*	11-
2-	6	220	206		5-	3	184	162		9-	1	11*	10
2-	7	35*	3-		5-	4	190	197-		9-	2	21	14
2-	9	43	40		5-	5	242	253		9-	3	77	62-
3	1	211	217		5-	6	124	127		9-	4	29*	20-
3	2	138	139-		5-	7	97	91		10	1	190	176-
3	3	211	215		5-	8	56	66-		10-	2	32	34-
3	4	85	80		6	2	91	80		10-	3	157	147
3	5	158	157		6	3	38	28-		11-	1	22	15-
					6	4	117	114		11-	2	69	68

Table 4 (cont.)

h	k	Fo	Fc
0	0	249	247-
0	1	146	142-
0	2	43	16
0	3	229	224-
0	4	236	234-
0	5	65	50-
0	6	146	135-
0	7	38*	31-
0	8	103	86-
0	9	22*	2-
1	0	122	115
1	1	289	296
1	2	46	34
1	3	188	189
1	4	74	63
1	5	266	269
1	6	105	90-
1	7	106	107
1	8	32*	35-
1	9	42	39
1	0	207	200-
1	1	230	253-
1	2	167	153-
1	3	41	36-
1	4	31*	5
1	5	198	183-
1	6	136	120
1	7	182	159-
1	8	89	72
1	9	131	128-
2	0	398	398
2	1	52	48-
2	2	290	287
2	3	96	81
2	4	437	453
2	5	38*	40-
2	6	243	237
2	7	80	64
2	8	133	124
2	0	380	406
2	1	135	138
2	2	52	45-
2	3	89	74
2	4	75	52
2	5	50	43
2	6	39*	0-
2	7	38*	6-
2	8	33*	2
2	9	22*	28-
3	0	427	426
3	1	122	105-
3	2	403	431
3	3	91	75-
3	4	92	84

l = 4

h	k	Fo	Fc
3	5	188	183-
3	6	80	67
3	7	65	59-
3	8	27*	2
3	0	352	351-
3	1	145	148
3	2	179	160-
3	3	61	53-
3	4	163	154-
3	5	126	99
3	6	67	54-
3	7	171	165
3	8	55	43-
3	9	169	166
4	0	371	351-
4	1	152	147
4	2	292	297-
4	3	25	14
4	4	291	287-
4	5	39*	44
4	6	93	87-
4	7	90	83-
4	8	44	35-
4	0	568	599-
4	1	9*	21
4	2	241	228-
4	3	195	201
4	4	188	168-
4	5	84	67
4	6	80	60-
4	7	75	69
4	8	29*	11-
5	0	369	355-
5	1	73	70-
5	2	411	453-
5	3	100	92-
5	4	182	168-
5	5	38*	30
5	6	95	88-
5	7	42	44
5	0	265	250
5	1	119	116-
5	2	90	79
5	3	64	51-
5	4	93	92
5	5	115	108-
5	6	69	56-
5	7	115	120-
5	8	26*	15-
6	0	230	224
6	1	93	89-
6	2	190	198
6	3	87	80
6	4	80	80

h	k	Fo	Fc
6	5	63	56
6	6	28*	29-
6	7	71	67
6	0	295	280
6	1	50	41-
6	2	212	198
6	3	205	209-
6	4	188	191
6	5	91	78-
6	6	121	117
6	7	98	95-
6	8	46	53
7	0	63	40
7	1	64	61
7	2	233	222
7	3	65	46
7	4	153	154-
7	5	28*	7
7	6	108	123
7	0	12*	2
7	1	60	55
7	2	83	72
7	3	103	99
7	4	39*	25-
7	5	126	114
7	6	58	59
7	7	63	75
8	0	101	95-
8	1	11*	11
8	2	128	116-
8	3	164	159-
8	4	26*	35-
8	5	113	128-
8	0	97	82-
8	1	25	18-
8	2	140	132-
8	4	141	134-
8	5	33*	29
8	6	78	95-
9	0	41	38-
9	1	8*	8
9	2	153	154-
9	3	74	75
9	0	66	59-
9	1	11*	2-
9	2	96	98-
9	3	100	79-
10	0	98	102
10	1	64	27
10	2	160	147
10	3	45	37-
11	0	110	110
11	1	23	16-
11	2	117	135

l = 5

h	k	Fo	Fc
0	1	61	55-
0	2	38	33-
0	3	108	107-
0	4	158	191-
0	5	150	138-
0	6	46*	16
0	7	44*	20-
0	8	35*	18
1	1	428	442
1	2	136	130
1	3	154	147
1	4	181	177
1	5	175	173
1	6	172	169
1	7	43*	32
1	8	86	84
1	1	347	375-
1	2	44	34-
1	3	137	133-
1	4	109	108-
1	5	120	126-
1	6	205	199-
1	7	4*	3-
1	8	89	89-
2	1	249	254
2	2	109	102-
2	3	251	250
2	4	87	77
2	5	204	210
2	6	45*	15-
2	7	120	123
2	8	30*	19
2	1	234	249
2	2	11	0-
2	3	138	145
2	4	41*	28
2	5	102	93
2	6	80	76-
2	7	44*	60-
2	8	35*	34-
3	1	122	120-
3	2	132	126-

h	k	Fo	Fc
3	3	9*	8
3	4	144	139-
3	5	120	122-
3	6	127	120-
3	7	37*	23-
3	8	118	120-
3	1	195	197
3	2	27	21-
3	3	183	186
3	4	82	21
3	5	160	150
3	6	185	176
3	7	43*	57
3	8	85	86
4	1	353	367-
4	2	106	94
4	3	248	249-
4	4	46*	38-
4	5	148	150-
4	6	41*	17
4	7	130	127-
4	1	193	183-
4	2	124	115
4	3	109	103-
4	4	170	174
4	5	84	87-
4	6	134	135
4	7	41*	38
4	8	31*	35
5	1	148	141-
5	2	79	77
5	3	141	138-
5	4	101	92
5	5	42*	13-
5	6	71	82
5	7	45	41-
5	1	148	135-
5	2	65	65-
5	3	237	239-
5	4	45*	32-
5	5	190	187-
5	6	111	118-

h	k	Fo	Fc
5	7	87	100-
5	8	26*	34-
6	1	165	164
6	2	10	0-
6	3	103	93
6	4	42*	29
6	5	36*	33
6	6	27*	17-
6	1	37	36-
6	2	74	68-
6	3	48	20-
6	4	170	167-
6	5	89	90
6	6	108	106-
6	7	33*	30
7	1	113	107
7	2	61	58-
7	3	78	77
7	4	88	104-
7	5	26*	44
7	1	93	101
7	2	116	119
7	3	152	144
7	4	45*	56
7	5	53	75
7	6	48	62
7	7	26*	44
8	1	63	60-
8	2	51	42-
8	3	65	66-
8	1	48	40-
8	2	16	12-
8	3	33	21-
8	4	43*	58
8	5	76	102-
8	6	28*	56
9	1	198	181-
9	2	60	46-
9	3	147	127-
10	1	141	132
10	2	15	10
10	3	78	69
11	1	200	212

l = 6

h	k	Fo	Fc
0	0	238	240-
0	1	158	160
0	2	198	206-
0	3	98	88
0	4	106	100-
0	5	186	186
0	6	115	113-
0	7	76	86
0	8	46	42-
1	0	163	152
1	1	42	40-
1	2	85	73
1	3	9*	4
1	4	219	212
1	5	66	57-
1	6	80	83
1	7	26*	33
1	8	42	45
1	0	240	237-
1	1	70	72-
1	2	84	81-
1	3	102	96-
1	4	217	221-
1	5	30*	18-
1	6	118	126-
1	7	27*	29-
1	8	82	74-
2	0	292	284
2	1	58	54-
2	2	278	293
2	3	26	22-
2	4	91	85
2	5	150	142-
2	6	66	61
2	7	63	68-
2	0	130	129
2	1	158	165-
2	2	88	82
2	3	75	73-
2	4	104	96
2	5	158	160-
2	6	116	115
2	7	119	123-
2	8	72	69

h	k	Fo	Fc
3	0	256	248-
3	1	96	98
3	2	183	185-
3	3	9*	4
3	4	172	169-
3	5	29*	29
3	6	27*	6-
3	7	51	48-
3	0	551	495
3	1	101	92
3	2	234	251
3	3	66	60
3	4	232	230
3	5	44	46
3	6	117	119
3	7	27*	11
3	8	64	56
4	0	242	238-
4	1	34	34-
4	2	321	345-
4	3	57	48-
4	4	161	159-
4	5	66	62
4	6	88	95-
4	7	58	68
4	0	94	80
4	1	104	101
4	2	143	138
4	3	37	31
4	4	29*	31
4	5	98	98
4	6	29*	8
4	7	121	125
4	8	15*	20-
5	0	136	138
5	1	58	59-
5	2	108	110
5	3	116	104
5	4	38	42
5	5	76	80
5	6	44	37-
5	0	343	319-
5	1	40	31-
5	2	151	147-

h	k	Fo	Fc
5	3	54	53
5	4	113	109-
5	5	30*	19-
5	6	43	51-
5	7	23*	21
6	0	61	48
6	1	16	19
6	2	210	211
6	3	8*	0-
6	4	140	149
6	5	18*	27-
6	0	73	54-
6	1	63	60-
6	2	118	121-
6	3	56	49-
6	4	51	34-
6	5	68	84-
6	6	64	68-
7	0	99	15
7	1	16	13
7	2	7*	8-
7	3	142	144-
7	4	16*	12
7	0	72	61
7	1	11*	5-
7	2	93	91-
7	3	94	91-
7	4	29*	19
7	5	27*	22-
7	6	22*	10
8	0	7*	4
8	1	28	24
8	0	57	55-
8	1	35	34
8	2	9	2-
8	3	99	79
9	0	66	64-
9	1	10*	11
9	2	93	83-
9	3	98	86
10	1	9*	11
10	1	8*	6-
10	2	16	18-
10	3	66	63-

l = 8

h	k	Fo	Fc
0	0	35	31
0	1	25	14-
0	2	24	17-
0	3	9*	6
1	0	147	152
1	1	27	30-
1	2	156	169
1	3	9*	20-
1	0	263	274-
1	1	84	85
1	2	188	213-
1	3	72	67
2	0	59	57-
2	1	41	43
2	2	28	23-
2	3	61	47-
2	0	184	190-
2	1	33	24-
2	2	96	97-
2	0	9*	1-
3	0	12*	12

h	k	Fo	Fc
3	1	10*	1-
3	2	103	98-
3	3	9*	7-
3	0	119	116
3	1	92	94-
3	2	61	60
3	3	89	64-
4	0	139	144
4	1	34	29-
4	2	127	113
4	3	119	108
4	0	221	225
4	1	47	45
4	2	121	122
4	3	23	16
5	0	29	24
5	1	9*	12-
5	2	149	147
5	3	33	27-
5	0	12*	22
5	1	63	63

h	k	Fo	Fc
5	2	59	56
5	3	58	45
6	0	56	47-
6	1	14	15
6	0	157	173-
6	1	41	39-
6	2	114	102-
6	3	9*	17-
7	0	11*	21-
7	1	34	36-
7	2	50	49-
7	3	58	47-
8	0	184	161
8	1	9*	13
8	2	130	126
8	3	26	19-
9	0	71	55
9	1	8*	13
9	2	71	73
9	3	39	40

l = 7

h	k	Fo	Fc
0	1	37	24
0	2	110	111
0	3	82	85-
0	4	119	125
0	5	27*	15
0	6	117	116
0	7	20*	24-
1	1	134	103
1	2	95	90-
1	3	108	110
1	4	27*	28
1	5	127	129
1	6	24*	27-
1	7	89	85
1	1	264	283-
1	2	9*	0
1	3	244	254-
1	4	70	78-
1	5	214	210-
1	6	26*	15
1	7	89	80-
2	1	11*	9-
2	2	105	106-
2	3	38	39
2	4	106	99-
2	5	81	83-
2	6	95	95-
2	7	15*	21-
2	1	249	252-
2	2	29	18-
2	3	90	91-
2	5	47	49-
2	6	114	110-

h	k	Fo	Fc
2	7	21*	14
3	1	173	170-
3	2	91	84
3	3	90	93-
3	4	27*	2-
3	5	55	65-
3	6	20*	27
3	1	348	363
3	2	30	17
3	3	248	259
3	4	27*	32
3	5	160	167
3	6	25*	30-
3	7	20*	15
4	1	11*	9
4	2	64	64
4	3	9*	1
4	4	77	74
4	5	62	61
4	6	68	80
4	1	302	315
4	2	54	45-
4	3	243	248
4	4	27*	26-
4	5	149	154
4	6	82	86
4	7	64	65
5	1	211	206
5	2	19	18
5	3	128	120
5	4	41	40
5	5	38	44
5	1	129	133-
5	2	43	36

h	k	Fo	Fc
5	3	80	78-
5	4	57	70
5	5	49	59-
5	6	53	55-
5	7	15*	27
6	1	46	40
6	2	41	37-
6	3	20	24
6	4	75	81-
6	1	114	113-
6	2	27	29-
6	3	175	171-
6	4	27*	23-
6	5	126	140-
6	6	77	83-
7	1	86	95-
7	2	62	68-
7	1	11*	13
7	3	50	56-
7	3	27	13
7	4	109	117-
7	5	50	58
7	6	57	71-
8	1	75	64
8	2	84	83
8	3	107	103
8	4	65	65
8	5	54	70
9	1	27	22-
9	2	27	26
9	3	31	25-
10	1	127	130-
10	2	36	36-

l = 9

h	k	Fo	Fc
0	1	152	134
0	2	81	69-
0	3	120	110
1	1	25	27
1	2	92	83-
1	3	33	33
1	1	53	52
1	2	110	93
1	3	9*	14-
2	1	135	131-
2	2	38	36
2	3	70	68-

h	k	Fo	Fc
2	1	132	141-
2	2	36	28
2	3	152	137-
3	1	57	38
3	2	34	31
3	3	47	31
3	1	134	145-
3	2	28	20-
3	3	63	51-
4	1	185	186
4	2	41	36
4	1	127	146

h	k	Fo	Fc
4	2	9*	14
4	3	126	115
5	1	78	69
5	2	43	48-
5	3	49	45
6	1	200	193-
6	2	8*	15
6	3	141	133-
7	1	47	43-
7	2	15	5
7	3	71	71-
8	1	134	137
8	2	33	42-

l = 10

h	k	Fo	Fc
0	0	76	75
0	1	14	18-
0	2	94	94
0	3	7*	11-
1	0	101	89-
1	1	27	15
1	2	63	58-
1	3	72	68-
1	0	146	136
1	1	15	4
1	2	98	92
1	3	27	28
2	0	27	36-

h	k	Fo	Fc
2	1	8*	11
2	2	98	104-
2	0	34	37-
2	1	40	36
2	2	8*	11-
2	3	35	31
3	0	47	45
3	1	13	15-
3	0	73	69-
3	1	9*	14-
3	2	30	26-
3	3	7*	2
4	0	49	45

h	k	Fo	Fc
4	1	53	51-
4	2	8*	1-
4	3	56	51-
5	0	39	26
5	1	29	21
5	2	7*	8-
5	3	66	3-
6	0	87	73-
6	1	47	48
6	2	39	41-
6	3	60	63
7	0	111	108-
7	1	22	17-
7	2	4*	78-

l = 11

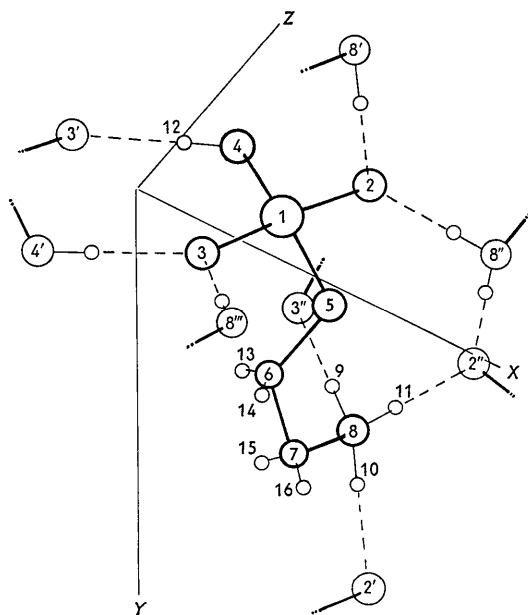| h	k	Fo	Fc
1	1	136	144
1	2	4*	41-
2	1	75	76
2	2	48	57
3	1	30	34-
3	2	16	18
4	1	48	50-
4	2	4*	42-

Fig. 1. The crystal structure of
2-amino-ethanol phosphate.

Table 6. *Bond distances and angles for hydrogen bonds,
and their estimated standard deviations*

Bond	Distance	σ
O4–O3'	2·557 Å	0·008 Å
N8–O2'	2·761	0·008
N8–O2"	2·748	0·008
N8–O3"	2·802	0·008

Angle	Degrees	σ
P1–O2–N8'	119·5°	0·2°
P1–O2–N8"	122·3	0·2
N8'–O2–N8"	117·9	0·2
P1–O3–O4'	129·6	0·2
P1–O3–N8'''	123·4	0·2
O4'–O3–N8'''	106·7	0·2
P1–O4–O3'	117·6	0·2
O2'–N8–O2"	110·9	0·2
O2'–N8–O3"	108·1	0·2
O2"–N8–O3"	113·2	0·2
H9–N8–O3"	7	4
H10–N8–O2'	5	3
H11–N8–O2"	14	4
H12–O4–O3'	13	6

Table 5. *Bond distances and angles for covalent bonds,
and their estimated standard deviations*

Bond	Distance	σ
P1–O2	1·493 Å	0·005 Å
P1–O3	1·503	0·005
P1–O4	1·557	0·005
P1–O5	1·591	0·005
O5–C6	1·429	0·007
C6–C7	1·521	0·008
C7–N8	1·491	0·007
N8–H9	0·90	0·06
N8–H10	0·98	0·06
N8–H11	0·90	0·06
O4–H12	0·86	0·09
C6–H13	1·20	0·08
C6–H14	1·01	0·06
C7–H15	1·05	0·06
C7–H16	0·95	0·06

Angle	Degrees	σ
O2–P1–O3	117·4°	0·2°
O2–P1–O4	109·8	0·2
O2–P1–O5	103·9	0·2
O3–P1–O4	109·3	0·2
O3–P1–O5	109·6	0·2
O4–P1–O5	106·2	0·2
P1–O5–C6	118·7	0·3
O5–C6–C7	108·9	0·4
C6–C7–N8	111·4	0·3
O5–C6–H13	119	4
O5–C6–H14	108	3
C7–C6–H13	95	4
C7–C6–H14	113	3
H13–C6–H14	113	5
C6–C7–H15	97	4
C6–C7–H16	113	4
N8–C7–H15	114	4
N8–C7–H16	108	4
H15–C7–H16	115	5

are almost exactly coplanar with the bond P1–O2 (mean deviation from least-squares plane, 0·03 Å) and the same is true of the hydrogen bonds from O3 to O4' and N8''' with respect to the bond P1–O3. Furthermore, the angles involved are those expected in a molecule containing a double bond. Assuming that the unshared electron pairs on the oxygens are concentrated in the hydrogen bond directions, and taken in conjunction with the already well established shortness of this type of bond in comparison with a normal P–O bond, this observation seems to leave little room for doubt that one is dealing with bonds having almost entirely double-bond character, as suggested by Pauling (1960) and others. It is also worth noting that the two planes are approximately at right angles; possibly, if this turns out to be generally true, it may have some bearing on the theory of this type of bond.

Other features of the structure are unexceptional. The covalent bond lengths and angles are within the normal range. The hydrogen bonds between the nitrogen and oxygens are of the usual length. Although the hydrogen bond from O4 to O3' is quite short, a similarly short bond has been found between oxygens of the phosphate group in phosphoric acid (2·53 Å) and in cytidylic acid *b* (2·51 Å) by Furberg (1955) and by Alver & Furberg (1959). Hydrogen bonds of this length are also found between carboxyl groups (see Fuller, 1959).

As the last four entries of Table 6 show, the hydrogen atoms do not appear to lie exactly along the hydrogen-bond directions, but in view of the large errors in estimating the angles of deviation, a great deal of significance cannot be attributed to these figures.

The parameters of the vibration ellipsoids have been calculated from the betas of Table 2, and are listed in Table 7. The quantities in the columns headed c_{ia} etc.

Table 7. *Magnitudes and direction cosines of the principal axes of the vibration ellipsoids*

	Axis i	B_i	c_{ia}	c_{ib}	c_{ic}
P1	1	2·09	−0·475	0·400	0·869
	2	1·64	0·079	0·908	−0·418
	3	0·76	0·876	0·133	0·264
O2	1	6·37	−0·406	0·128	0·971
	2	2·06	0·284	0·959	−0·070
	3	1·13	0·869	−0·253	0·229
O3	1	4·43	0·022	0·896	−0·437
	2	1·75	0·008	0·443	0·874
	3	1·47	−0·999	0·024	0·212
O4	1	7·06	−0·227	0·966	0·166
	2	1·86	0·278	0·182	−0·981
	3	1·79	0·933	0·181	0·102
O5	1	4·08	0·631	0·075	−0·890
	2	2·08	0·253	0·921	0·234
	3	1·44	0·734	−0·382	0·391
C6	1	4·81	0·708	0·180	−0·820
	2	2·38	0·137	0·914	0·344
	3	1·83	0·693	−0·364	0·458
C7	1	3·96	0·967	0·036	−0·454
	2	2·41	0·163	0·675	0·668
	3	2·00	0·195	−0·737	0·589
N8	1	2·41	−0·200	0·384	0·923
	2	1·89	0·193	0·917	−0·382
	3	1·38	0·961	−0·104	0·044

are the cosines of the angles between the ith ellipsoid axis and the a, b and c axes of the unit cell. The problem of transformation to principal axes has been discussed by Busing & Levy (1958), Waser (1955) and Rollett & Davies (1955). The extreme anisotropy of vibration of O2 and O4 should be noted, as well as the fact that O2 and O3 have their maximum vibration directions perpendicular to the hydrogen-bond planes mentioned above. Similarly, O4, O5, C6 and C7 are vibrating most strongly in directions generally perpendicular to the planes of their bonds, though not exactly so.

The hydrogen atom temperature factors, as seen from Table 3, do not differ significantly from zero, except possibly for H12. It was thought, at first, that H12 might be disordered between O4 and O3, and that as a consequence the least-squares program was attempting partially to 'erase' it be means of an artificially high temperature factor. However, no

evidence for a partial hydrogen attached to O3 could be found in a final ΔF map. It was concluded that its large B is in fact due to the strong vibration of O4, to which H12 is attached.

References

ALVER, E. & FURBERG, S. (1959). *Acta Chem. Scand.* **13**, 910.
BERGHUIS, J., HAANAPPEL, IJ. M., POTTERS, M., LOOPSTRA, B. O., MACGILLAVRY, C. H. & VEENENDAAL, A. L. (1955). *Acta Cryst.* **8**, 478.
BUSING, W. R. & LEVY, H. A. (1958). *Acta Cryst.* **11**, 450.
BUSING, W. R. & LEVY, H. A. (1959). *A Crystallographic Least Squares Refinement Program for the IBM 704*, U.S. Atomic Energy Commission Publication ORNL 59-4-37.
DARLOW, S. F. (1960). *Acta Cryst.* **13**, 683.
DUNITZ, J. D. & ROLLETT, J. S. (1956). *Acta Cryst.* **9**, 327.
FULLER, W. (1959). *J. Phys. Chem.* **63**, 1705.
FURBERG, S. (1955). *Acta Chem. Scand.* **9**, 1557.
HUGHES, E. W. (1941). *J. Amer. Chem. Soc.* **63**, 1737.
MCWEENY, R. (1951). *Acta Cryst.* **4**, 513.
PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed., chapter 9. Ithaca: Cornell University Press.
PETERSON, S. W. & LEVY, H. A. (1957). *Acta Cryst.* **10**, 70.
ROLLETT, J. S. & DAVIES, D. R. (1955). *Acta Cryst.* **8**, 125.
VIERVOLL, H. & ØGRIM, O. (1949). *Acta Cryst.* **2**, 277.
WASER, J. (1955). *Acta Cryst.* **8**, 731.