Table 4. *Pertinent interatomic distances and standard deviations*

Th—F(1)	2·41 (1) Å	F(2)—F(4)*a*	2·79 (2) Å
Th—F(2)	2·26 (1)	F(2)—F(7)*a*	2·81 (2)
Th—F(3)	2·34 (1)	F(2)—F(7)*b*	2·94 (2)
Th—F(4)*a*	2·42 (1)	F(3)—F(4)*a*	2·83 (2)
Th—F(5)	2·35 (1)	F(3)—F(4)*b*	3·15 (2)
Th—F(6)	2·36 (1)	F(3)—F(6)	2·76 (2)
Th—F(7)*a*	2·45 (1)	F(3)—F(7)*a*	2·88 (2)
Th—F(4)*b*	2·45 (1)	F(4)*a*–F(4)*b*	2·48 (2)
Th—F(7)*b*	2·44 (1)	F 4)*a*–F(5)	2·74 (2)
F(1)–F(2)	2·95 (2)	F(4)*a*–F(6)	2·86 (2)
F(1)–F(4)*a*	2·80 (2)	F(5)—F(6)	2·66 (2)
F(1)–F(5)	2·84 (2)	F(5)—F(7)*a*	2·79 (2)
F(1)–F(7)*b*	2·76 (2)	F(6)—F(7)*a*	3·73 (2)
F(1)–F(4)*b*	3·42 (2)	F(6)—F(7)*b*	2·74 (2)
F(2)–F(3)	2·94 (2)	F(7)*a*–F(7)*b*	2·54 (2)

The eighth fluorine atom, F(8), is not involved in the thorium coordination sphere, being 4·98 Å from its nearest thorium neighbor. This fluorine atom has five nitrogen atom neighbors at an average distance of 2·72 Å (range 2·65–2·87 Å), which form a much distorted square pyramid. Its nearest fluorine neighbor is at a distance of 3·35 Å.

Though the ninefold coordination of Th^{4+} is to be expected on the basis of the ratio of the thorium to fluorine ionic radii, to the best of our knowledge this is the first actinide fluoride complex in which a fluorine atom is not included in the coordination sphere of the heavy metal ion. One might expect that the composition of this material would best be described by writing the formula as $(NH_4)_3ThF_7.NH_4F$ and that the first NH_4F would be relatively easy to lose. However, it is found that the first NH_4F loss from $(NH_4)_4ThF_8$ (which is also the first endotherm above room temperature) occurs at an even higher temperature (205°) than it does from $(NH_4)_4UF_8$ (180°). In this latter compound all fluorine atoms are involved in the coordination of the U^{4+}.

The authors gratefully acknowledge the advice and assistance of D. T. Cromer on this problem.

References

Brown, D., Kettle, S. F. A. & Smith, A. J. (1967). *J. Chem. Soc.* A, p. 1430.
Burnham, C. W. (1962). *I. U. C. World List of Crystallographic Computer Programs,* Program 338.
Busing, W. R. & Levy, H. A. (1957). *Acta Cryst.* **10**, 180.
Cromer, D. T. (1965). *Acta Cryst.* **18**, 17.
Cromer, D. T. & Waber, J. T. (1965). *Acta Cryst.* **18,** 104.
Hamilton, W. C. (1965). *Acta Cryst.* **18**, 502.
International Tables for X-*ray Crystallography* (1962). Vol. III. Birmingham: Kynioch Press.
Larson, A. C. (1968). Unpublished work.
Larson, A. C., Cromer, D. T. & Roof, R. B., Jr (1964). Los Alamos Scientific Laboratory Report, LA-3043.
Pauling, L. (1960). *The Nature of the Chemical Bond,* 3rd. Ed. p. 561. Ithica: Cornell Univ. Press.
Penneman, R. A. & Rosenzweig, A. (1969). *Inorg. Chem.* **8**, 627.
Penneman, R. A., Ryan, R. R. & Rosenzweig, A. (1968). *Chem. Comm.* p. 990.
Rosenzweig, A. & Cromer, D. T. (1969). *Acta Cryst.* In the press.

Acta Cryst. (1969). B**25**, 1962

The Crystal Structure of 1-*p*-Nitrobenzeneazo-2-naphthol (Para Red) from Overlapped Twin-Crystal Data

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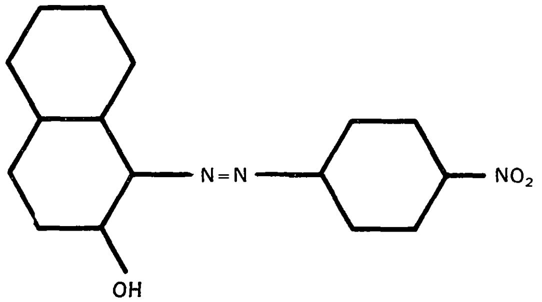
The crystal structure of the red pigment known as Para Red has been determined by the use of photographic X-ray methods. The crystals are monoclinic, space group *Pb*, with two molecules per unit cell. The dimensions of the unit cell at room temperature are $a=3·797\pm0·002$, $b=27·036\pm0·006$, $c=7·199\pm0·002$ Å, $\gamma=115°13'\pm1'$. The structure was solved first in projection and then in three dimensions but failed to refine properly. It was found that the crystals suffered from pseudo-merohedral twinning of small obliquity and that the reflexions from the two individuals overlapped at room temperature. The overlapped data were subsequently used successfully in three methods of least-squares refinement, each method having certain advantages. With structure amplitudes corrected for twinning, the final *R* value was 0·13. The almost planar molecules were arranged end-to-end in zigzag fashion.

Introduction

Para Red (paranitroaniline red), 1-*p*-nitrobenzeneazo-2-naphthol, $C_{16}H_{11}N_3O_3$, was one of the first of the synthetic organic dyes and pigments, the Para Red process for dyeing being developed in 1880 (Venkataraman, 1952, p. 4). It is currently used as a paint pigment. The structural formula was known to be



We are indebted to Mr L. Williamson, formerly of Taubmans Paints, Sydney, for supplying the sample used in this work.

The main interest in the structure determination lies in the fact that twinned crystals were used in the solution and refinement. This paper outlines the sequence of events and describes the structure, while further details of the methods used are described elsewhere (Grainger & McConnell, 1969; Grainger, 1969, referred to below as I and II respectively).

Crystal data

1-*p*-Nitrobenzeneazo-2-naphthol (Para Red)
$C_{16}H_{11}N_3O_3$

Crystallization method: slow cooling of a hot pyricine solution.

Crystal habit: prismatic needles elongated along the *c* axis, with main faces of the form {011}.

Optical properties: red, strongly light-absorbent, pleochroic. Electrical properties: pyroelectric.

Unit-cell dimensions at 20 °C (monoclinic, 'first setting'):

$$a = 3{\cdot}797 \pm 0{\cdot}002 \text{ Å}$$
$$b = 27{\cdot}036 \pm 0{\cdot}006$$
$$c = 7{\cdot}199 \pm 0{\cdot}002$$
$$\gamma = 115° \; 13' \pm 1'.$$

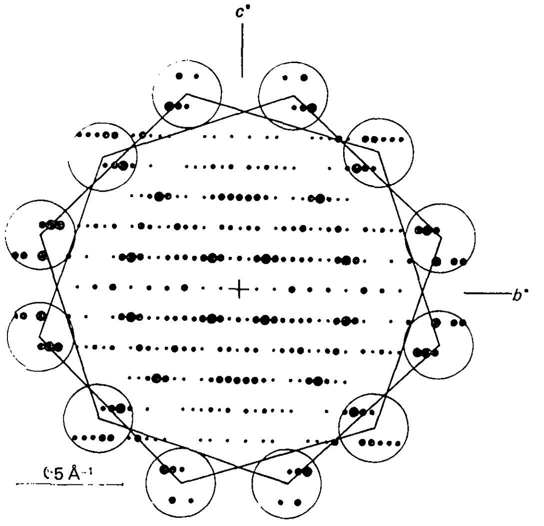


Fig. 1. 0*kl* weighted reciprocal lattice.

The errors are standard deviations.

Measured density: 1·4 g.cm^{-3}.

Calculated density: 1·47 g.cm^{-3} assuming $Z = 2$.

Conditions for reflexion: $k = 2n$ for 0*k*0 reflexions.

Space group: *Pb* (No. 7, *International Tables for X-ray Crystallographic*, 1952.)

The space group is uniquely determined for $Z = 2$.

Purity: A microanalysis performed by Dr E. Challen of the School of Chemistry of The University of New South Wales gave the following percentages after recrystallization (calculated figures in brackets): carbon 65·26 (65·50), nitrogen 14·01 (14·32), hydrogen 3·86 (3·78).

X-ray powder diffractograms indicated that the crystalline form was unchanged on recrystallization from the sample of the material used in paint manufacture.

Plane-polarized light travelling parallel to the *c* axis is strongly absorbed if the electric vector is parallel to the *b* axis, while absorption in the perpendicular direction is much less marked. These observations indicate (Bunn, 1946, p. 285) that the double bond of the azo group is probably parallel to the *b* axis. The crystal shape, the vibration directions for transmitted light, and the crystal cleavage all pointed to flat molecules roughly parallel to the (100) plane. The *b* axis was in fact chosen with these facts in mind: an alternative choice was possible making an angle of approximately 98° with the needle *a* axis.

Optical examination did not disclose the presence of embedded twinned inclusions until, after twinning had been detected by X-ray means, a microscope with a very powerful light source was employed.

The strong pyroelectric behaviour was shown by Martin's test (Bunn, 1946, p. 290), indicating a non-centrosymmetric structure, and further confirming the space group.

The cell dimensions were obtained from 0*kl* and *hk*0 Weissenberg photographs from crystals sprinkled with silicon powder for calibration purposes. Chromium radiation (unfiltered) was used to bring a greater number of strong reflexions into the high angle region. Although the *hk*0 photograph was complicated by twinning, correct indexing was possible when the twinning pattern was understood. About 80 measurements were used in a least-squares program written by Kastalsky (1967), to determine the values above. Standards used were the wavelengths in *International Tables for X-ray Crystallography* (1962), and the lattice parameter $a = 5{\cdot}43054$ Å for silicon (Parrish, 1960). Measurements on $K\alpha$, $K\alpha_1$, $K\alpha_2$ and $K\beta$ lines and spots were made as appropriate.

Structure determination

Preliminary 0*kl*, 1*kl* and 2*kl* Weissenberg photographs were taken with Cu $K\alpha$ radiation. Comparatively short exposures (about 13 hours) were given, and many reflexions were unobserved. A Wilson plot of the 0*kl* data gave the temperature factor $B = 4{\cdot}0$ Å^2.

The $0kl$ weighted reciprocal lattice (radii proportional to $|F_o|$, after applying the inverse temperature correction) is shown in Fig. 1, and revealed the presence of twelve 'benzene peaks', circled in the Figure, arising from benzene or naphthalene groups in parallel planes as nearly as could be judged, but with one rotated 30° with respect to the other (Taylor & Lipson, 1964). Since there were known to be four groups of either benzene or naphthalene in the cell, a total of 24 peaks would be possible, so the smaller number indicated the existence of pairing in the orientations of the groups.

The [100] Patterson projection (Fig. 2) showed twelve peaks near the origin corresponding to the interatomic distances in the range 2·4 to 2·8 Å, which generally bore out the conclusions derived from the weighted reciprocal lattice. The shorter 1·4 Å vectors expected from benzene rings are evident only as bulges protruding from the origin peak.

By the use of a jointed cardboard model with the *azo* nitrogen atoms arranged in the *trans* configuration and the benzene and naphthalene groups coplanar and with parallel sides, an arrangement was soon found which fitted in well with all the information at hand. In particular, the strong Patterson peaks near the origin (and the benzene peaks in the reciprocal lattice) were satisfied, and also peaks L and M of the Patterson map were identified as the strong peaks expected as a result of the many parallel vectors from the benzene ring to each of the two fused rings of the naphthalene group in parallel orientation. In addition, the Patterson peak E could be interpreted as involving the nitrogen and oxygen atoms which had not so far been taken into account.

In the space group Pb the position of the molecule along the x and y axes is arbitrary, so the chief remaining uncertainty was its position along the z axis, assuming that the orientation and molecular configuration had been correctly determined. A trial model based on packing of the glide-related molecules proved to be fairly accurate. The nitro and hydroxyl groups were assumed to lie in the plane of the molecule. The trial model was then similar to that of Fig. 3 except for a slight twist to the molecule as a whole.

Assuming the plane of the molecule to be parallel to (100), y and z coordinates for all atoms except hydrogen were derived by the use of the model, and $0kl$ structure factors were calculated, giving an R value of 0·52 after scaling. After three Fourier syntheses and structure factor cycles, R had been reduced to 0·28. The least-squares program was then used, and after a few cycles R fell to 0·19 with 160 reflexions with the temperature factor fixed at 4·0 Å^2.

The projected benzene and naphthalene rings resulting from this refinement were quite regular, and the projected bond lengths within the rings were compared with the expected lengths to deduce the orientation of the molecule with respect to the (100) plane, assuming it to be planar. From this, relative x coordinates were calculated (absolute values being unnecessary, as mentioned above) and an R value of 0·35 was obtained using these coordinates to calculate $1kl$ structure factors. Least-squares refinement finally reduced R to 0·18 for 480 $0kl$, $1kl$ and $2kl$ reflexions with anisotropic temperature factors (since the program was unable to refine isotropically).

Refinement now seemed to be at an end. Although it was confidently believed that the structure was fundamentally correct, and a three-dimensional difference Fourier synthesis revealed no significant peaks, there were disturbing features, apart from the relatively high R. The benzene and naphthalene rings were decidedly non-planar and many of their bonds were of unusual length, ranging from 1·16 to 1·76 Å.

It was at this point that in attempting to take low temperature data for a further refinement, it was found that the crystals of Para Red were twinned. Full details of this discovery and the unusual characteristics of the twinning are given elsewhere (I). Briefly, the crystals were twinned by pseudo-merohedry with a small obliquity angle at room temperature (0° 22′) which became doubled when the crystals were cooled to −140°C. This change was sufficient to reveal the twinning which at room temperature had been unnoticed since the reflexions arising from the two individuals of the twin overlapped. The twinning showed itself at low tempera-

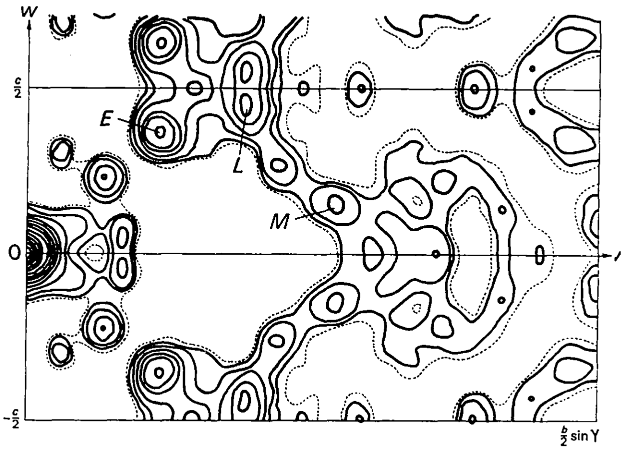


Fig. 2. [100] Patterson projection.

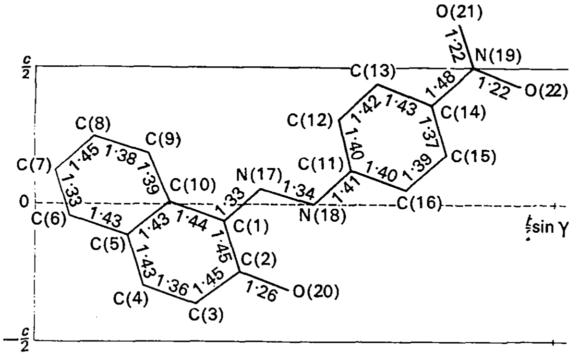


Fig. 3. [100] projection of the molecule; distances are in ångströms.

tures by splitting of the diffraction spots along the festoons on *c* axis Weissenberg photographs.

It should be noted that the solution in projection was accomplished with $0kl$ data which is not affected by the type of twinning encountered here. Trouble was experienced only with three-dimensional data.

Investigations disclosed that it is possible to derive structure amplitudes 'corrected for twinning' from the overlapped data, so it was decided to try this out in practice. Somewhat later the authors also learned of the method developed by Kennicott (1963) for refining overlapped data.

A new set of data was therefore taken at room temperature with a new crystal rotating about the *c* axis. Multiple-film equi-inclination Weissenberg photographs were taken with copper radiation, filtered for $hk0$ and $hk1$ levels, unfiltered for the $hk2$ to $hk4$ levels. Exposures were increased to approximately 90 hours. Multiple-film intensity scales were also taken, and since these were prepared with a zero-level reflexion, allowance was made for the variation of film absorption with equi-inclination angle, φ, by the application of an effective film factor $K_o{}^{\sec\varphi-1}$, where K_o is the film factor at normal incidence. This expression follows from the equation $K_n = K_o^{\sec\varphi n}$ for the *n*th layer film factor (Qurashi, 1953), which is derived directly from the exponential law for X-ray absorption. By use of a typical value for 'Ilfex' X-ray film, $K_o = 2{\cdot}93$, (the value is not critical), the effective film factor for the fourth layer was 1·12.

Of the 1530 non-equivalent reflexions accessible to copper radiation, the number measured in these *c* axis data was 943, of which 96 were unobservably weak. No corrections were made for absorption by the crystal, as the linear absorption coefficient was calculated to be 8·6 cm^{-1} for copper $K\alpha$ radiation, and absorption would amount only to about 16% for the longest path through the crystal.

In placing the intensities on a common scale with another crystal the effects of twinning were avoided by the use of only an $0kl$ photograph, for the relative intensities of $0kl$ reflexions were known to be unaffected by twinning.

In Para Red the effect of twinning in reciprocal space is the same as it would be if the two individuals were related by a mirror plane perpendicular to the b^* axis, although it could equally well be described in other ways (I). Thus pairs of reflexions of the type h,k,l and $h, -(6h+k), l$ which are related by this mirror plane may be regarded as mutually interacting and overlapping. In general this alters them both, increasing the intensity (F^2) of one and decreasing that of the other by an equal amount. Regarding as 'correct' the readings which would have been obtained with an untwinned crystal of the same total volume, the amount of 'intensity' thus transferred when twinning is present depends upon the intensity of each and the amount of twinning (II).

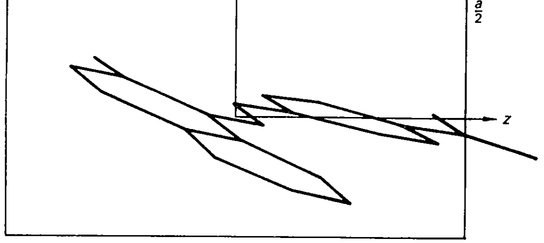


Fig. 4. Projection of molecule on to the (010) plane.

However, the sum of the intensity readings of such a 'related pair' of reflexions will be the same irrespective of the amount of twinning, and this fact was used in the 'summed data' method of refinement. In this an 'intensity' equal to this sum is regarded as an observation in a least-squares program modified according to the method of Kennicott. All atomic parameters were refined in several cycles, together with an overall scale factor, without requiring any knowledge of the amount of twinning. At first sight this method sounds ideal, but it suffered from the disadvantage that there were 197 parameters to be determined from only about 460 'observations', thereby decreasing the accuracy. However, its ability to provide an overall scale factor and to refine all parameters in a minimum of computing time without involving the amount of twinning, makes it extremely useful before passing on to the normal Kennicott method.

The normal procedure of Kennicott was next used, mainly in order to refine the twin fraction *f*, defined as the ratio of the volume of the smaller individual of the twin to the volume of the whole crystal. For this refinement the intensities of the composite or overlapped reflexions of the twin are used directly as observations, and the program compares these with the values calculated by summing the contributions from the two reflexions which are involved, adjusting the parameters to obtain a least-squares best fit. The program employs two scale factors to take account of the twin fraction, and these were initially set to correspond to a twin fraction 0·275. On refinement of all parameters it was found that the twin fraction, calculated from the scale factors, rose steadily to the value $0{\cdot}286 \pm 0{\cdot}005$.

The twin fraction for this crystal had previously been found experimentally (II) by cooling the crystal to $-140\,^\circ\text{C}$ so that sufficient spots were split to measure separately the split components. This gave $f = 0{\cdot}29 \pm 0{\cdot}04$, in excellent agreement with the above value.

Finally it was decided to obtain $|F_o|$ values 'corrected for twinning' (II), and to use these in the final refinement so as to present the final results in familiar form. The corrected intensity I_c of one of the related reflexions is given in terms of its measured intensity I_o and that of the related reflection I'_o by the relation

$$I_c = I_o + \frac{f}{1-2f}(I_o - I'_o).$$

The value $f = 0{\cdot}286 \pm 0{\cdot}005$ was used.

The corrected intensities were then converted to structure amplitudes and at the same time an estimated standard deviation for each $|F_o|$ was calculated for use in least-squares refinement. These apparently simple operations were found to involve unusual considerations. The importance of calculating individual e.s.d.'s starting from the errors in the raw film readings has been amply demonstrated (II). The calculated e.s.d.'s ranged from about 8% to 100% of $|F_o|$. In general the percentage bears no simple relationship to $|F_o|$: thus any attempts to apply weights which are a function of $|F_o|$ will be incorrect.

By the use of the corrected structure amplitudes as observations, four cycles of a normal least-squares program gave a final R of 0·13 for all 926 observations having available related reflexions. The significance of this comparatively high figure is discussed in II, where it is pointed out that this is as good as can be expected when account is taken of the unusual nature of the data. The value of Σ (e.s.d.)/Σ $|F_o|$ is, in fact, a little higher than this, *viz.* 0·17. For that part of the data which is expected to be of normal accuracy (comprising 585 reflexions) R is 0·09, and it can be argued that the overall R value is of less than usual significance in these circumstances.

In the final cycle coordinate shifts were all less than 0·2 of the e.s.d. The program was able to refine only 140 of the 197 parameters in any one cycle, so the final temperature parameter refinement was carried out in the previous cycle which gave shifts of about the same amount.

Interatomic distances and angles, thermal vibrations and the angles between planes were calculated using a version of *ORFFE* (Busing & Levy, 1961) adapted to the IBM 360/50 by Craig (1967).

The structure factor, Fourier and least-squares calculations in the early parts of the investigation were carried out on UTECOM, a DEUCE computer, with programs written by Rollett (1961). The final series of least-squares calculations were done on the IBM 360/50 with a program based on *ORFLS* (Busing, Martin & Levy, 1962), modified to deal with overlapped data in the Kennicott manner; the authors are indebted to Dr G.Cox of the Australian Atomic Energy Commission Research Establishment, Lucas Heights, for assistance in this regard. Several data reduction programs for applying Lorentz and polarization corrections and for manipulating the data in the special ways required by the twinning of Para Red were written by one of us (C.T.G).

Description of the structure of Para Red

Fig. 3 shows the [100] projection of the molecule and the numbering of the atoms. The final atomic coordinates and their e.s.d's are given in Table 1, the bond lengths and angles in Table 2. Owing to the overall high residual the e.s.d's are rather large and even these are probably optimistic since not all parameters were refined together. For this reason only two places of decimals are used for the bond lengths in Fig. 3.

Table 1. *Atomic coordinates*

The e.s.d.'s (by 10^4) are in brackets.

	x/a	*y/b*	*z/c*
C(1)	0·0080 (29)	0·1811 (3)	−0·0607 (14)
C(2)	0·1606 (35)	0·1966 (3)	−0·2469 (14)
C(3)	0·2046 (33)	0·1552 (4)	−0·3600 (15)
C(4)	0·0969 (36)	0·1037 (5)	−0·2931 (16)
C(5)	−0·0539 (32)	0·0877 (4)	−0·1098 (13)
C(6)	−0·1733 (36)	0·0327 (4)	−0·0451 (16)
C(7)	−0·3074 (34)	0·0180 (4)	0·1257 (14)
C(8)	−0·3586 (36)	0·0565 (4)	0·2505 (17)
C(9)	−0·2511 (33)	0·1090 (4)	0·1857 (17)
C(10)	−0·0969 (29)	0·1268 (4)	0·0105 (14)
C(11)	0·0195 (29)	0·3038 (3)	0·1237 (15)
C(12)	−0·0677 (33)	0·2907 (4)	0·3117 (15)
C(13)	−0·1041 (34)	0·3285 (3)	0·4390 (14)
C(14)	−0·0283 (30)	0·3808 (3)	0·3623 (13)
C(15)	0·0610 (31)	0·3939 (4)	0·1788 (13)
C(16)	0·0895 (31)	0·3556 (4)	0·0574 (16)
N(17)	−0·0331 (28)	0·2160 (3)	0·0580 (13)
N(18)	0·0526 (30)	0·2667 (3)	−0·0049 (10)
N(19)	−0·0642 (34)	0·4202 (4)	0·4955 (16)
O(20)	0·2431 (*)	0·2437 (*)	−0·3096 (13)
O(21)	−0·1628 (40)	0·4064 (4)	0·6551 (17)
O(22)	−0·0145 (36)	0·4644 (3)	0·4302 (14)

* These coordinates were not varied.

Table 2. *Intramolecular distances and angles*

The e.s.d.'s of the distances (by 10^3) are in brackets.

Atoms	Distance (Å)	Atoms	Angle (°)
1–2	1·449 (14)	10–1–2	121·6 (0·7)
2–3	1·451 (13)	1–2–3	117·5 (0·7)
3–4	1·360 (13)	2–3–4	120·5 (0·9)
4–5	1·430 (14)	3–4–5	123·0 (0·8)
5–6	1·433 (11)	4–5–10	119·4 (0·8)
6–7	1·326 (14)	5–10–1	118·0 (0·8)
7–8	1·451 (13)	10–5–6	119·0 (0·9)
8–9	1·380 (11)	5–6–7	121·6 (0·8)
9–10	1·388 (13)	6–7–8	120·7 (0·8)
10–1	1·443 (9)	7–8–9	117·3 (1·0)
10–5	1·429 (9)	8–9–10	124·0 (0·9)
2–20	1·259 (10)	9–10–5	117·4 (0·7)
1–17	1·331 (10)	16–11–12	120·5 (0·8)
17–18	1·345 (7)	11–12–13	121·7 (0·6)
18–11	1·410 (10)	12–13–14	115·0 (0·8)
11–12	1·402 (13)	13–14–15	123·4 (0·6)
12–13	1·423 (11)	14–15–16	120·1 (0·6)
13–14	1·429 (9)	15–16–11	119·3 (0·9)
14–15	1·373 (12)	13–14–19	114·8 (0·8)
15–16	1·394 (12)	14–19–21	120·2 (0·7)
16–11	1·395 (9)	14–19–22	115·6 (1·0)
14–19	1·482 (10)	21–19–22	124·1 (0·9)
19–21	1·217 (12)	1–2–20	121·2 (0·8)
19–22	1·223 (10)	10–1–17	115·4 (0·8)
20–18	2·472 (11)	1–17–18	117·1 (0·7)
		17–18–11	116·6 (0·7)
		18–11–12	122·5 (0·6)

The bond lengths do not differ significantly from the corresponding ones in related structures, 4-nitroaniline (Trueblood, Goldish & Donohue, 1961), *N*,*N*-dimethyl-*p*-nitroaniline (Mak & Trotter, 1965), *p*-nitrophenol

(Coppens & Schmidt, 1965), 1, 5-dimethylnaphthalene (Beintema, 1965), β-naphthol (Watson & Hargreaves, 1958), except that the C(6)–C(7) bond is unusually short.

The best least-squares planes for the atoms in the naphthalene and benzene groups respectively were calculated with a program of Shirley Chu adapted by B. M. Craven and D. C. Craig.

They are:

naphthalene:

$$0{\cdot}89805X-0{\cdot}14061Y+0{\cdot}34921Z=-0{\cdot}80524\ ;$$

benzene:

$$0{\cdot}91845X-0{\cdot}09173Y+0{\cdot}21639Z=-0{\cdot}48386\ ,$$

where X, Y, Z are coordinates in Å along the crystallographic axes. Relevant out-of-plane distances are given in Table 3, and it can be seen that both the naphthalene and benzene groups are substantially planar. Since the X-coordinates, which are the most important ones in this connexion, have errors of about 0·014 Å, the deviations from these planes are probably not significant More accurate studies of such aromatic structures invariably find the rings planar within experimental errors, and the present work does not contradict these.

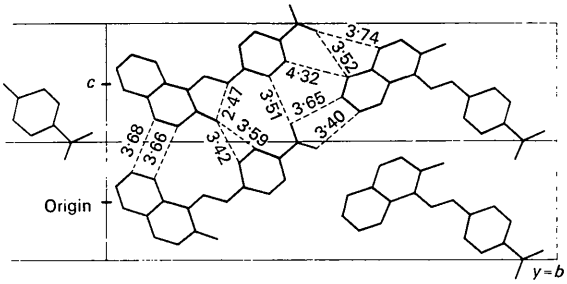


Fig. 5. Intermolecular distances on the (100) projection.

Table 3. *Distances from the least squares planes*

* These atoms not used in calculating the plane.

Atom	Distance from the naphthalene plane	Atom	Distance from the benzene plane
1	0·008 Å	11	−0·009 Å
2	0·015	12	0·012
3	0·008	13	−0·010
4	0·005	14	0·005
5	0·012	15	−0·002
6	−0·023	16	0·003
7	0·005		
8	−0·003		
9	0·001		
10	0·019	18*	−0·002
		19*	−0·010
17*	0·017	21*	−0·071
20*	−0·071	22*	−0·048

The naphthalene plane makes an angle of approximately 9° with the plane of the benzene ring. As is obvious in the (010) molecular projection shown in Fig. 4, this angle represents a twist of one with respect to the other about the axis of the molecule. The planes of the benzene ring and the nitro group are inclined at an angle of about 5°.

The packing of the molecules is shown in Fig. 5, together with the closer intermolecular contacts. Hydrogen atoms have of course not been included. The distances given have e.s.d's of about 0·013 Å. All distances are consistent with van der Waals contacts, and

Table 4. *Thermal parameters*

The b_{ij} are coefficients in the expression $\exp\left[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+2b_{12}hk+2b_{23}kl+2b_{13}hl)\right]$.

Atom	$b_{11}\times 10^3$	$b_{22}\times 10^5$	$b_{33}\times 10^4$	$b_{12}\times 10^4$	$b_{23}\times 10^4$	$b_{13}\times 10^4$
1	75	163	250	46	−2	−35
2	99	169	273	35	6	5
3	108	229	217	53	−6	36
4	109	256	181	78	−34	−66
5	111	189	212	71	−33	−132
6	130	176	224	76	−20	−162
7	124	178	155	69	−6	−32
8	110	166	432	53	3	−56
9	100	166	427	66	7	25
10	76	148	273	54	−13	−31
11	69	122	371	32	5	6
12	112	134	261	50	1	95
13	119	143	226	53	3	−25
14	82	122	245	41	−11	−43
15	116	128	171	49	2	−12
16	116	159	230	69	4	41
17	75	128	248	30	1	1
18	102	128	192	42	−1	2
19	137	162	507	83	−16	−57
20	163	217	330	62	27	200
21	266	289	396	163	−5	183
22	234	175	439	114	−5	6
Mean e.s.d.	8	12	41	8	5	40

Table 5. *Structure factor table for Para Red*

The column headings FOBS, ESD, FCAL and ANG indicate respectively: $|F_o|$, the e.s.d. of $|F_o|$, $|F_c|$, and the phase angle of F_c. The significance of the column headed T is discussed elsewhere (Grainger, 1969).

H	K	L	FOBS	ESD	FCAL	ANG	T
0	2	0	18.2	1.4	18.4	177	21
0	4	0	20.1	1.5	21.4	179	21
0	6	0	27.0	2.0	29.5	163	21
0	8	0	13.6	1.0	12.3	68	21
0	10	0	19.1	1.4	20.4	18	21
0	12	0	7.6	0.6	7.9	160	21
0	14	0	16.4	1.2	17.1	193	21
0	16	0	8.8	0.7	9.0	181	21
0	18	0	6.4	0.5	6.2	140	21
0	20	0	5.3	0.4	5.3	81	21
0	22	0	4.5	0.3	4.1	133	21
0	24	0	4.8	0.4	4.4	82	21
0	26	0	0.9	0.9	1.5	316	23
0	28	0	0.8	0.8	0.7	354	23
0	30	0	3.6	0.3	3.4	266	21
1	0	0	137.3	12.2	120.5	351	1
1	2	0	68.9	6.7	63.3	245	1
1	4	0	4.5	1.8	4.1	300	12
1	6	0	13.2	1.4	9.8	205	1
1	8	0	6.7	1.0	7.6	40	11
1	10	0	35.4	3.5	31.4	335	1
1	12	0	12.5	1.7	9.6	128	31
1	14	0	7.3	1.7	6.8	181	12
1	16	0	7.8	0.7	7.1	138	1
1	18	0	6.1	1.6	3.7	260	12
1	20	0	7.2	1.3	5.3	99	12
1	22	0	3.2	0.5	3.3	290	1
1	24	0	4.4	0.6	3.9	34	31
1	26	0	0.7	0.7	0.9	92	33
2	0	0	20.5	1.8	18.6	0	1
2	2	0	21.8	2.0	19.5	228	1
2	4	0	5.2	0.5	4.3	168	1
2	6	0	7.7	0.9	7.1	183	1
2	8	0	5.3	1.9	4.9	185	12
2	10	0	18.5	1.8	15.6	329	1
2	12	0	10.4	5.1	7.3	187	12
2	14	0	2.9	1.3	2.3	350	12
2	16	0	2.9	0.5	2.3	94	2
2	18	0	1.1	1.1	0.7	357	13
2	20	0	6.8	0.6	6.0	138	1
2	22	0	4.2	0.5	3.3	289	1
3	0	0	8.9	0.8	7.9	336	1
3	2	0	1.2	1.2	1.0	241	13
3	4	0	0.6	0.6	0.8	79	13
3	6	0	0.5	0.5	2.2	141	13
3	8	0	1.2	1.2	2.5	118	13
3	10	0	4.2	0.4	4.1	349	1
3	12	0	4.0	0.5	4.5	192	1
3	14	0	0.7	0.7	0.5	335	33
4	0	0	5.6	1.0	3.0	307	12
4	2	0	0.0	0.5	0.3	216	14
4	4	0	0.0	0.5	0.8	231	14
4	6	0	0.4	0.4	0.6	359	33
-1	2	0	37.4	7.1	31.8	137	12
-1	4	0	53.8	6.0	53.6	206	1
-1	6	0	16.5	16.5	22.4	227	13
-1	8	0	25.8	11.7	22.4	213	12
-1	10	0	11.0	1.1	11.3	80	1
-1	12	0	8.6	1.9	13.7	208	12
-1	14	0	7.2	0.9	5.7	258	1
-1	16	0	13.1	6.0	14.3	201	12
-1	18	0	12.5	1.7	13.0	156	31
-1	20	0	12.4	1.3	12.2	224	1
-1	22	0	1.5	1.5	2.6	313	13
-1	24	0	11.5	1.2	11.7	74	1
-1	26	0	9.6	1.1	10.9	326	1
-1	28	0	1.7	0.9	2.2	6	12
-1	30	0	4.5	0.6	4.9	291	31
-1	32	0	0.8	0.8	0.6	187	33
-2	2	0	12.8	1.5	12.3	262	1
-2	4	0	4.5	4.5	6.8	247	13
-2	6	0	10.5	0.8	11.2	296	21
-2	8	0	19.0	1.8	16.3	214	1
-2	10	0	10.5	1.7	10.4	250	12
-2	12	0	2.4	2.4	3.2	225	13
-2	14	0	3.1	3.1	6.3	3	13
-2	16	0	3.0	0.7	3.7	233	12
-2	18	0	6.3	1.0	5.4	241	12
-2	20	0	12.4	1.2	12.2	265	1
-2	22	0	7.5	3.0	7.5	344	12
-2	24	0	28.8	2.8	30.0	78	1
-2	26	0	7.7	0.8	7.4	332	1
-2	28	0	0.7	0.7	1.1	54	13
-2	30	0	4.4	0.4	3.7	272	1
-2	32	0	1.4	1.4	2.3	134	13
-2	34	0	3.9	0.6	4.0	80	11
-3	2	0	5.6	0.5	5.2	240	1
-3	4	0	4.8	2.0	5.8	21	12
-3	6	0	4.0	1.0	1.9	173	12
-3	8	0	5.3	1.6	4.9	235	12
-3	10	0	10.7	1.1	9.0	269	1
-3	12	0	7.3	0.8	6.5	352	1
-3	14	0	12.3	1.2	10.7	89	1
-3	16	0	2.1	1.0	2.2	35	12
-3	18	0	0.9	0.9	1.6	340	13
-3	20	0	4.2	0.4	3.8	303	1
-3	22	0	5.4	0.5	4.0	170	1
-3	24	0	28.0	2.4	23.3	95	1
-3	26	0	6.0	0.6	4.8	257	1
-3	28	0	2.5	0.6	3.3	273	12
-3	30	0	3.1	0.5	2.6	270	12
-3	32	0	0.7	0.7	1.2	175	33
-4	2	0	7.3	0.8	4.2	186	1
-4	4	0	4.3	0.5	3.5	318	1
-4	6	0	0.9	0.9	0.7	140	13
-4	8	0	0.9	0.9	0.7	329	13
-4	10	0	4.1	1.5	1.2	309	12
-4	12	0	2.4	0.4	1.9	321	21
-4	14	0	9.6	1.0	8.4	110	1
-4	16	0	1.7	0.9	1.4	216	2
-4	18	0	1.7	0.9	0.8	288	2
-4	20	0	2.5	0.6	2.1	282	12
-4	22	0	4.7	1.0	5.3	201	12
-4	24	0	7.5	0.9	7.5	103	1
-4	26	0	5.8	0.5	4.7	289	1
-4	28	0	2.7	0.2	2.0	308	1
-4	30	0	0.5	0.5	0.3	345	33
0	2	1	43.1	3.2	53.2	113	21
0	3	1	44.1	3.3	50.4	107	21
0	4	1	31.9	2.4	34.6	66	21
0	5	1	17.4	1.3	17.4	272	21
0	6	1	22.2	1.7	22.7	68	21
0	7	1	17.2	1.3	16.6	169	21
0	8	1	15.4	1.2	13.3	3	21
0	9	1	8.1	0.6	7.9	218	21
0	10	1	8.7	0.6	8.2	5	21
0	11	1	22.6	1.7	22.5	185	21
0	12	1	28.3	2.1	26.2	121	21
0	13	1	19.7	1.5	20.4	29	21
0	14	1	6.2	0.5	5.8	7	21
0	15	1	3.4	0.3	3.3	38	21
0	16	1	3.5	0.3	4.0	61	21
0	17	1	5.2	0.4	6.3	44	21
0	18	1	0.7	0.7	1.4	157	23
0	19	1	10.0	0.8	10.9	172	21
0	20	1	1.3	0.4	1.2	132	22
0	21	1	4.2	0.3	4.5	211	21
0	22	1	16.8	1.3	17.8	281	21
0	23	1	3.5	0.3	3.4	61	21
0	24	1	7.6	0.6	8.0	147	21
0	25	1	5.0	0.4	5.5	278	21
0	26	1	0.7	0.7	0.8	161	23
0	27	1	2.1	0.2	1.9	329	21
0	28	1	0.6	0.6	1.0	145	23
1	1	1	20.8	2.0	18.9	342	1
1	2	1	20.6	2.2	18.4	72	1
1	3	1	6.4	4.9	7.8	85	12
1	4	1	7.9	1.4	7.5	19	12
1	5	1	2.8	2.8	4.4	103	13
1	6	1	2.8	2.8	5.0	85	13
1	7	1	4.4	4.4	5.8	112	13
1	8	1	8.3	1.1	9.6	297	31
1	9	1	2.8	2.8	3.3	38	13
1	10	1	9.3	1.0	9.1	353	1
1	11	1	7.5	0.7	6.8	204	1
1	12	1	10.3	1.0	9.6	106	1
1	13	1	10.1	1.3	10.8	65	1
1	14	1	10.2	1.0	9.3	2	1
1	15	1	6.6	1.5	4.1	250	12
1	16	1	5.9	5.9	1.5	74	13
1	17	1	7.1	2.3	3.1	29	12
1	18	1	1.3	1.3	1.3	104	13
1	19	1	1.0	1.0	1.1	301	13
1	20	1	1.6	0.7	1.2	83	32
1	21	1	4.8	0.4	4.2	67	1
1	22	1	9.7	0.8	8.6	286	1
1	23	1	2.4	0.3	2.1	125	1
1	24	1	4.3	0.4	4.7	146	1
1	25	1	0.5	0.5	0.6	270	13
1	26	1	0.3	0.3	0.5	63	13
2	1	1	12.0	2.3	13.0	211	12
2	2	1	4.2	0.4	4.3	81	1
2	3	1	5.5	0.7	4.9	264	1
2	4	1	2.2	0.4	1.7	269	12
2	5	1	4.9	0.5	3.7	165	1
2	6	1	2.6	0.3	2.3	135	31
2	7	1	1.1	1.1	1.5	55	13
2	8	1	3.9	1.5	4.2	217	12
2	9	1	6.7	1.0	6.8	288	11
2	10	1	6.5	2.6	3.8	327	12
2	11	1	2.3	2.3	1.9	313	13
2	12	1	1.5	1.5	2.3	98	13
2	13	1	4.6	0.4	4.5	74	1
2	14	1	2.8	0.4	3.0	348	31
2	15	1	0.9	0.9	0.2	82	13
2	16	1	0.6	0.6	0.5	145	13
2	17	1	0.9	0.9	1.7	206	33
2	18	1	0.5	0.5	0.5	73	13
2	19	1	1.9	0.3	1.8	331	32
2	20	1	0.6	0.6	1.1	90	13
2	21	1	0.5	0.5	0.5	69	33
3	1	1	5.8	0.5	5.3	181	1
3	2	1	1.9	0.6	1.3	33	12
3	3	1	1.4	1.4	0.6	131	13
3	4	1	0.9	0.9	0.4	311	13
3	5	1	2.7	0.4	2.8	124	1
3	6	1	0.7	0.7	1.0	50	13
3	7	1	1.2	1.2	2.1	140	13
3	8	1	4.7	0.7	4.5	187	11
3	9	1	1.7	0.5	2.4	276	32
3	10	1	3.2	0.3	3.1	342	1
3	11	1	1.5	0.4	2.2	192	32
3	12	1	0.7	0.7	1.5	84	13
3	13	1	0.4	0.4	0.9	323	13
3	14	1	0.3	0.3	0.5	283	13
3	15	1	0.7	0.3	0.7	10	12
4	1	1	0.8	0.8	0.7	216	13
4	2	1	0.4	0.4	0.8	333	13
4	3	1	0.3	0.3	0.5	15	13
4	4	1	0.5	0.5	0.6	347	33
-1	0	1	21.1	2.8	17.9	25	31
-1	1	1	70.5	6.6	72.5	239	1
-1	2	1	66.4	6.8	79.7	151	1
-1	3	1	36.5	2.7	37.6	48	21
-1	4	1	34.2	9.4	22.6	91	12
-1	5	1	15.3	15.3	19.9	330	13
-1	6	1	21.1	2.8	19.3	92	31
-1	7	1	5.8	4.5	4.7	62	12
-1	8	1	13.2	2.7	12.1	58	12
-1	9	1	22.7	2.2	23.5	218	1
-1	10	1	10.6	1.2	12.3	231	1
-1	11	1	20.0	1.8	19.8	192	1
-1	12	1	12.8	1.2	11.2	162	1
-1	13	1	21.6	2.0	20.2	51	1
-1	14	1	8.2	1.1	7.0	230	31
-1	15	1	12.1	1.1	11.1	47	1
-1	16	1	4.7	1.3	4.4	19	12
-1	17	1	1.4	1.4	2.3	274	13
-1	18	1	4.1	1.7	1.9	281	12
-1	19	1	9.1	1.3	9.8	221	11
-1	20	1	2.8	2.2	5.1	326	12
-1	21	1	11.1	1.2	11.6	236	1
-1	22	1	25.4	2.4	24.9	285	1
-1	23	1	15.5	1.6	16.1	23	1
-1	24	1	5.8	0.6	6.1	144	1
-1	25	1	4.0	0.4	3.4	338	1
-1	26	1	1.6	0.7	1.6	143	32
-1	27	1	1.0	1.0	2.3	357	13
-1	28	1	0.0	0.5	0.8	122	14
-1	29	1	2.0	0.4	2.3	224	12
-1	30	1	0.0	0.5	0.5	219	14
-1	31	1	1.5	0.4	1.5	133	2
-1	32	1	1.5	0.3	1.4	351	2
-2	0	1	8.7	2.5	10.6	326	12
-2	1	1	21.7	2.6	22.2	233	1
-2	2	1	46.2	4.3	42.7	165	1
-2	3	1	21.8	2.4	22.7	35	1
-2	4	1	2.9	0.7	2.8	306	12
-2	5	1	19.3	1.7	16.2	74	1
-2	6	1	2.5	0.2	2.6	88	21
-2	7	1	3.1	3.1	0.8	156	13
-2	8	1	5.2	0.5	4.9	34	1
-2	9	1	13.6	2.9	12.6	210	12
-2	10	1	9.1	9.1	6.4	250	13
-2	11	1	17.2	2.8	16.1	235	12
-2	12	1	17.5	1.8	14.9	306	1
-2	13	1	17.2	1.9	17.3	68	1
-2	14	1	0.8	0.8	2.0	283	13
-2	15	1	5.2	0.7	6.1	37	11
-2	16	1	2.9	0.3	3.3	41	1
-2	17	1	2.7	0.7	3.4	10	12
-2	18	1	2.6	0.3	2.7	12	31
-2	19	1	5.8	0.5	5.9	308	1
-2	20	1	9.2	0.9	9.1	353	1
-2	21	1	7.4	0.9	8.0	172	1
-2	22	1	15.9	1.6	16.9	309	1
-2	23	1	12.9	1.2	11.5	36	1
-2	24	1	6.2	0.6	5.9	120	1
-2	25	1	0.8	0.8	1.9	77	13
-2	26	1	2.8	0.4	3.1	165	31
-2	27	1	3.2	0.3	3.2	206	1
-2	28	1	2.8	0.3	2.6	129	1
-2	29	1	0.9	0.9	1.4	248	33
-2	30	1	2.0	0.4	1.7	146	2
-2	31	1	1.9	0.3	1.8	178	32
-2	32	1	2.3	0.2	2.3	64	1
-2	33	1	0.5	0.5	0.7	30	33
-3	0	1	7.1	0.7	7.1	313	1
-3	1	1	4.7	0.5	5.1	166	1
-3	2	1	16.4	1.5	14.1	162	1
-3	3	1	3.9	0.4	3.6	127	1
-3	4	1	3.5	0.4	3.7	335	1
-3	5	1	2.9	1.6	2.8	92	12
-3	6	1	1.7	1.7	2.2	342	13
-3	7	1	2.3	1.8	2.9	249	12
-3	8	1	2.0	0.5	1.7	14	12
-3	9	1	1.9	0.3	1.5	354	21
-3	10	1	2.9	0.3	2.8	50	1
-3	11	1	8.3	0.8	7.9	218	1
-3	12	1	8.8	0.8	8.6	331	1
-3	13	1	8.7	0.8	8.2	65	1
-3	14	1	2.3	0.5	2.9	80	12
-3	15	1	1.0	1.0	1.1	113	13
-3	16	1	3.4	3.4	3.1	128	13
-3	17	1	2.4	0.7	1.3	79	12
-3	18	1	1.5	1.5	1.2	75	13
-3	19	1	0.5	0.5	1.0	274	13
-3	20	1	4.3	0.4	4.3	21	1
-3	21	1	7.5	0.7	7.4	169	1
-3	22	1	6.0	0.5	6.1	348	1
-3	23	1	1.4	0.7	1.6	343	12
-3	24	1	6.8	0.6	6.4	129	1
-3	25	1	5.7	0.5	5.6	312	1
-3	26	1	5.3	0.7	6.0	221	1
-3	27	1	1.7	0.5	2.2	186	32
-3	28	1	1.9	0.4	2.3	153	12
-3	29	1	1.5	0.5	1.4	16	32
-3	30	1	2.4	0.2	2.3	160	1
-3	31	1	1.6	0.3	1.4	165	2
-3	32	1	1.4	0.3	1.6	111	2
-3	33	1	1.3	0.2	1.3	314	1
-4	0	1	0.8	0.8	2.0	312	13
-4	1	1	0.8	0.8	1.3	188	13
-4	2	1	5.0	0.5	5.0	165	1
-4	3	1	0.7	0.7	1.6	181	13
-4	4	1	0.9	0.9	2.0	0	33
-4	5	1	0.9	0.9	1.9	301	33
-4	6	1	0.8	0.8	0.7	331	33
-4	7	1	0.8	0.8	1.2	18	33
-4	8	1	0.8	0.8	0.3	206	33
-4	9	1	0.9	0.9	1.2	45	33
-4	10	1	1.3	0.7	1.8	67	12
-4	11	1	4.4	0.4	4.5	191	1
-4	12	1	1.8	0.3	2.3	352	21
-4	13	1	0.0	0.5	0.7	37	14
-4	14	1	2.5	0.4	2.3	136	1
-4	15	1	0.9	0.9	2.0	318	33
-4	16	1	0.8	0.8	1.9	194	33
-4	17	1	0.8	0.8	0.4	239	33
-4	18	1	0.8	0.8	0.3	181	33
-4	19	1	0.9	0.9	0.9	266	33
-4	20	1	0.9	0.9	0.5	53	33
-4	21	1	1.9	0.4	1.8	191	2
-4	22	1	1.1	1.1	1.6	24	13
-4	23	1	2.4	0.3	2.4	7	1
-4	24	1	3.0	0.3	2.8	134	1
-4	25	1	4.5	0.4	4.8	329	1
-4	26	1	6.3	0.6	6.1	248	1
-4	27	1	2.6	0.2	2.8	123	1
-4	28	1	0.5	0.5	0.3	69	33
0	3	2	1.2	0.1	1.2	51	21
0	4	2	11.9	0.9	11.8	313	21
0	5	2	19.6	1.5	20.5	75	21
0	6	2	18.4	1.4	18.8	338	21
0	7	2	26.4	2.0	26.1	27	21
0	8	2	4.5	0.3	3.8	219	21
0	9	2	9.6	0.7	8.8	106	21
0	10	2	16.9	1.3	18.1	152	21
0	11	2	20.5	1.5	19.6	263	21
0	12	2	10.5	0.8	9.4	88	21
0	13	2	4.0	0.3	4.2	175	21
0	14	2	9.8	0.7	9.3	314	21
0	15	2	7.6	0.6	7.8	215	21
0	16	2	8.3	0.6	8.4	140	21
0	17	2	6.1	0.5	6.9	87	21
0	18	2	5.7	0.4	5.8	204	21
0	19	2	3.7	0.3	4.0	51	21
0	20	2	15.2	1.1	16.2	162	21
0	21	2	16.9	1.3	15.6	223	21
0	22	2	9.5	0.7	10.7	312	21
0	23	2	5.1	0.4	5.3	185	21
0	24	2	4.3	0.3	5.0	49	21
0	25	2	3.1	0.2	3.4	31	21
0	26	2	3.1	0.2	3.1	323	21
0	27	2	0.5	0.5	0.6	303	23
0	28	2	0.5	0.5	0.5	15	23
0	29	2	0.4	0.4	0.2	82	23
0	30	2	0.3	0.3	0.8	288	23
1	0	2	31.9	2.8	32.5	333	1
1	1	2	15.0	1.6	15.0	268	1
1	2	2	5.7	0.7	7.0	61	1
1	3	2	5.6	2.1	10.2	41	12
1	4	2	12.9	5.3	6.3	257	12
1	5	2	8.9	8.0	13.5	37	12
1	6	2	5.6	0.8	4.5	348	1
1	7	2	12.0	2.1	12.1	357	12
1	8	2	4.4	4.4	6.1	221	13
1	9	2	7.9	1.6	7.0	345	12
1	10	2	5.0	0.7	4.5	145	31
1	11	2	3.5	0.6	3.8	281	12
1	12	2	3.7	1.4	1.3	43	12
1	13	2	0.4	0.4	0.6	152	13
1	14	2	4.6	1.2	4.4	317	12
1	15	2	2.1	2.1	1.0	30	13
1	16	2	3.6	1.0	3.3	1	12
1	17	2	5.8	0.6	4.9	59	1
1	18	2	5.4	1.2	3.8	115	12
1	19	2	7.0	0.8	5.9	54	1
1	20	2	11.1	1.0	10.6	149	1
1	21	2	9.5	0.9	8.9	221	1
1	22	2	4.0	0.4	3.9	337	1
1	23	2	6.0	0.5	4.5	123	1
1	24	2	0.6	0.6	0.8	139	33
1	25	2	1.7	0.2	1.4	80	1
1	26	2	1.7	0.1	1.5	308	1
2	0	2	20.2	1.9	20.1	321	1
2	1	2	10.4	2.0	11.5	290	12
2	2	2	0.0	0.5	1.3	288	14
2	3	2	1.9	1.9	0.6	228	13
2	4	2	1.8	0.3	2.3	319	32
2	5	2	3.6	0.6	2.9	23	12
2	6	2	0.7	0.7	0.8	34	33
2	7	2	2.7	0.3	3.2	233	1
2	8	2	1.2	1.2	2.1	186	13
2	9	2	3.0	0.5	3.6	2	12
2	10	2	4.5	0.4	4.0	332	1
2	11	2	6.1	0.8	6.7	247	31
2	12	2	3.7	0.8	3.6	97	12
2	13	2	3.2	0.3	3.4	312	1
2	14	2	3.1	0.3	2.5	214	1
2	15	2	0.7	0.7	0.8	24	33
2	16	2	1.9	0.3	2.6	330	12
2	17	2	1.5	0.6	1.6	30	12
2	18	2	0.4	0.4	1.0	217	13
2	19	2	2.8	0.3	2.6	26	1
2	20	2	2.8	0.2	2.9	119	1
2	21	2	3.4	0.3	3.9	249	1
3	0	2	3.0	0.3	3.1	316	1
3	1	2	2.6	0.3	2.4	315	1
3	2	2	1.6	0.5	1.8	213	32
3	3	2	1.8	0.4	1.9	201	12
3	4	2	0.7	0.7	0.4	167	33
3	5	2	0.4	0.4	0.6	57	13
3	6	2	0.8	0.8	1.2	125	13
3	7	2	2.5	0.3	2.2	139	1
3	8	2	3.3	0.4	3.7	203	1
3	9	2	3.2	0.3	3.3	78	1
3	10	2	6.2	0.6	5.8	324	1
3	11	2	4.8	0.4	5.1	239	1
3	12	2	1.8	0.2	2.1	105	1
3	13	2	0.0	0.5	0.7	300	14
4	0	2	0.9	0.9	0.5	8	13
4	1	2	0.5	0.5	1.0	340	33
4	2	2	0.0	0.5	0.2	180	14
4	3	2	0.9	0.3	0.5	226	32
-1	1	2	10.3	1.0	9.9	107	1
-1	2	2	10.2	2.2	10.7	208	12
-1	3	2	8.7	0.7	9.7	305	21
-1	4	2	16.5	1.8	20.3	2	1
-1	5	2	2.5	2.5	3.2	128	13
-1	6	2	3.1	3.1	3.7	163	13
-1	7	2	8.0	2.1	9.6	37	12
-1	8	2	4.4	0.8	3.5	250	12
-1	9	2	13.1	1.3	12.3	83	1
-1	10	2	32.4	3.2	34.3	170	1
-1	11	2	34.4	3.3	31.7	272	1
-1	12	2	5.5	0.8	7.5	82	11
-1	13	2	16.3	1.9	15.6	338	1
-1	14	2	21.7	2.0	18.9	276	1
-1	15	2	12.1	1.3	11.8	207	1
-1	16	2	5.0	0.7	5.1	156	31
-1	17	2	4.4	0.5	4.1	110	1
-1	18	2	8.7	0.9	8.8	261	1
-1	19	2	1.9	0.3	2.2	5	2
-1	20	2	8.6	0.9	7.9	130	1
-1	21	2	11.3	1.0	11.4	256	1
-1	22	2	7.2	0.7	7.4	319	1
-1	23	2	3.0	0.8	4.1	179	12
-1	24	2	9.0	1.0	10.4	61	1
-1	25	2	5.5	0.9	6.0	17	12
-1	26	2	2.5	2.5	0.9	333	13
-1	27	2	1.6	1.6	0.8	88	13
-1	28	2	0.6	0.6	1.3	354	13
-1	29	2	0.5	0.5	1.9	83	13

Table 5 (*cont.*)

H	K	L	FOBS	ESD	FCAL	ANG	T
-1	30	2	0.6	0.6	0.4	317	33
-1	31	2	0.0	0.5	0.8	5	14
-1	32	2	0.0	0.5	0.9	255	14
-2	1	2	11.1	1.1	10.8	26	1
-2	2	2	21.0	2.6	21.9	151	1
-2	3	2	18.6	1.9	18.4	32	1
-2	4	2	14.3	1.9	15.7	290	1
-2	5	2	4.1	0.4	5.0	225	1
-2	6	2	8.0	0.6	8.9	198	21
-2	7	2	1.9	0.6	2.5	168	12
-2	8	2	13.6	1.9	13.1	287	11
-2	9	2	8.8	2.7	10.3	49	12
-2	10	2	17.7	2.8	16.5	165	12
-2	11	2	5.8	1.6	7.6	295	12
-2	12	2	4.5	4.5	6.5	7	13
-2	13	2	15.1	1.7	14.1	0	1
-2	14	2	14.8	1.3	12.9	292	1
-2	15	2	7.8	0.7	7.0	172	1
-2	16	2	1.8	0.3	2.0	72	32
-2	17	2	4.6	0.5	4.2	200	1
-2	18	2	0.7	0.7	1.0	73	33
-2	19	2	2.0	0.3	1.7	113	12
-2	20	2	5.3	0.5	4.7	107	1
-2	21	2	3.8	0.4	4.3	284	1
-2	22	2	1.0	1.0	1.1	184	13
-2	23	2	6.1	0.8	6.0	118	31
-2	24	2	5.9	0.6	5.3	84	1
-2	25	2	1.9	0.4	1.7	9	12
-2	26	2	1.9	0.4	1.8	326	12
-2	27	2	0.7	0.7	0.9	156	33
-2	28	2	2.6	0.3	2.3	352	1
-2	29	2	3.5	0.3	3.0	116	1
-2	30	2	1.6	0.3	1.6	46	2
-2	31	2	2.5	0.4	2.6	102	11
-2	32	2	0.3	0.3	0.7	258	13
-2	33	2	0.0	0.5	1.3	123	14
-3	1	2	4.5	0.5	3.8	84	1
-3	2	2	9.5	0.9	8.8	152	1
-3	3	2	11.8	1.2	12.6	48	1
-3	4	2	12.1	1.3	13.5	287	1
-3	5	2	7.7	0.7	6.7	198	1
-3	6	2	2.2	0.4	2.8	333	12
-3	7	2	4.1	0.3	4.0	195	1
-3	8	2	2.7	0.7	3.1	313	12
-3	9	2	2.3	0.2	2.1	59	21
-3	10	2	4.9	0.5	5.6	132	1
-3	11	2	0.0	0.5	0.4	76	14
-3	12	2	3.4	0.4	3.3	49	1
-3	13	2	1.4	1.4	2.0	43	13
-3	14	2	7.7	1.6	4.5	301	12
-3	15	2	5.7	1.7	3.7	205	12
-3	16	2	1.9	1.9	1.8	144	13
-3	17	2	2.6	0.6	3.2	239	12
-3	18	2	1.4	0.6	1.7	112	12
-3	19	2	1.8	0.4	1.7	180	12
-3	20	2	1.6	0.5	1.2	228	32
-3	21	2	2.6	0.3	2.4	237	1
-3	22	2	0.7	0.7	0.8	351	33
-3	23	2	2.0	0.4	2.0	109	2
-3	24	2	4.0	0.4	4.0	104	1
-3	25	2	0.7	0.7	1.1	250	13
-3	26	2	2.3	0.4	2.0	335	12
-3	27	2	0.6	0.6	0.9	281	13
-3	28	2	1.2	1.2	1.9	67	13
-3	29	2	1.0	1.0	1.1	124	13
-3	30	2	0.0	0.5	1.0	96	14
-3	31	2	1.9	0.2	1.6	134	1
-4	1	2	3.0	0.3	2.6	153	1
-4	2	2	2.3	0.2	1.9	123	1
-4	3	2	4.2	0.4	3.8	31	1
-4	4	2	4.7	0.4	4.3	312	1
-4	5	2	3.9	0.3	3.5	219	1
-4	6	2	1.0	0.5	1.0	49	2
-4	7	2	2.2	0.3	1.8	208	1
-4	8	2	0.8	0.8	1.6	145	33
-4	9	2	0.5	0.5	0.6	237	13
-4	10	2	0.8	0.8	1.7	202	33
-4	11	2	1.4	0.4	1.9	295	32
-4	12	2	2.1	0.2	2.2	27	21
-4	13	2	1.4	0.4	1.6	28	32
-4	14	2	0.8	0.8	0.2	293	33
-4	15	2	2.2	0.3	2.2	241	1
-4	16	2	0.8	0.8	0.6	198	33
-4	17	2	0.0	0.5	1.0	325	14
-4	18	2	0.6	0.6	0.4	64	13
-4	19	2	0.5	0.5	0.4	209	13
-4	20	2	0.8	0.8	1.0	306	13
-4	21	2	0.5	0.5	1.1	263	13
-4	22	2	1.1	0.4	1.4	49	12
-4	23	2	1.3	0.5	1.9	341	12
-4	24	2	4.4	0.4	4.1	125	1
-4	25	2	0.5	0.5	0.7	50	33
-4	26	2	2.4	0.2	2.3	243	1
-4	27	2	0.9	0.3	1.2	102	32
0	4	3	5.4	0.4	5.8	319	21
0	5	3	3.3	0.2	3.4	52	21
0	6	3	7.4	0.6	6.9	163	21
0	7	3	10.4	0.8	9.6	348	21
0	10	3	13.9	1.0	12.7	207	21
0	11	3	9.8	0.7	10.0	164	21
0	12	3	4.6	0.3	4.7	175	21
0	13	3	3.3	0.2	3.7	71	21
0	14	3	3.6	0.3	3.7	302	21
0	15	3	3.4	0.3	3.8	103	21
0	16	3	0.5	0.5	1.5	267	23
0	17	3	3.7	0.3	3.5	210	21
0	18	3	0.5	0.5	0.7	43	23
0	19	3	4.4	0.3	4.7	78	21
0	20	3	2.6	0.2	2.4	219	21
0	21	3	2.3	0.2	2.3	223	21
0	22	3	2.6	0.2	2.6	281	21
0	23	3	2.8	0.2	3.0	323	21
0	24	3	2.7	0.2	2.7	314	21
0	25	3	2.0	0.2	1.9	268	21
0	26	3	1.5	0.2	1.4	268	21
0	27	3	1.7	0.2	1.7	284	21
0	28	3	0.3	0.3	0.5	207	23
1	0	3	17.9	1.7	18.5	31	1
1	2	3	19.3	1.9	19.7	175	1
1	3	3	1.6	1.6	1.6	226	13
1	4	3	6.6	0.6	5.4	78	1
1	5	3	8.3	1.0	8.5	284	1
1	6	3	4.6	0.5	4.4	349	1
1	7	3	6.0	0.6	5.6	298	1
1	8	3	18.7	1.7	16.9	29	1
1	9	3	10.7	1.1	10.8	87	1
1	10	3	9.5	0.9	8.7	246	1
1	11	3	5.4	1.2	6.5	212	12
1	12	3	15.6	1.5	15.6	127	1
1	13	3	11.2	1.0	10.7	19	1
1	14	3	3.9	0.4	3.3	256	1
1	15	3	4.5	0.4	4.6	64	1
1	16	3	1.4	1.4	2.5	18	13
1	17	3	1.2	0.8	0.8	217	12
1	18	3	4.6	0.4	4.0	9	1
1	19	3	10.5	1.0	9.6	91	1
1	20	3	5.3	0.5	5.6	160	1
1	21	3	3.0	0.3	3.1	221	1
1	22	3	4.0	0.3	3.6	203	1
1	23	3	1.5	0.4	1.1	327	12
1	24	3	1.8	0.2	1.5	315	31
1	25	3	0.5	0.5	1.2	228	33
2	0	3	2.7	0.4	2.2	301	31
2	1	3	7.1	0.9	7.4	221	1
2	2	3	4.1	0.7	3.3	181	12
2	3	3	4.4	1.2	3.3	183	12
2	4	3	2.1	0.4	2.2	99	32
2	5	3	1.5	1.5	2.2	284	13
2	6	3	3.0	0.3	3.1	336	1
2	7	3	0.8	0.8	0.9	261	13
2	8	3	8.5	0.8	6.6	337	1
2	9	3	4.7	0.4	4.0	119	1
2	10	3	6.3	0.6	6.3	299	1
2	11	3	8.8	0.8	8.8	226	1
2	12	3	12.1	1.1	10.7	124	1
2	13	3	5.2	0.6	5.4	15	1
2	14	3	1.5	1.5	2.7	313	13
2	15	3	3.6	0.3	3.6	291	1
2	16	3	1.4	0.5	1.8	5	2
2	17	3	2.9	0.3	2.8	324	1
2	18	3	5.3	0.5	4.9	357	1
2	19	3	5.6	0.5	5.7	81	1
2	20	3	2.8	0.5	3.6	158	12
3	0	3	3.9	0.3	3.6	241	1
3	1	3	2.5	0.4	2.1	204	1
3	2	3	0.8	0.8	1.2	190	33
3	3	3	0.8	0.8	0.5	164	13
3	4	3	0.4	0.4	0.3	138	13
3	5	3	0.0	0.5	0.6	339	14
3	6	3	0.9	0.9	1.2	124	13
3	7	3	1.2	0.6	1.0	62	12
3	8	3	2.4	0.3	2.6	297	31
3	9	3	1.1	0.5	1.7	122	12
3	10	3	2.7	0.3	2.9	278	1
3	11	3	3.0	0.3	3.1	207	1
3	12	3	3.4	0.3	3.6	139	1
-1	1	3	6.4	0.8	7.5	215	1
-1	2	3	12.6	1.2	14.9	95	1
-1	3	3	6.0	0.4	5.5	105	21
-1	4	3	3.5	2.7	5.5	338	12
-1	5	3	5.2	0.8	5.2	107	12
-1	6	3	6.7	3.0	6.0	174	12
-1	8	3	7.2	3.3	5.5	343	12
-1	9	3	12.8	1.1	12.0	129	1
-1	10	3	2.3	1.2	4.5	99	12
-1	11	3	6.4	1.1	5.1	216	12
-1	12	3	2.1	0.7	1.6	237	12
-1	13	3	1.4	1.4	2.3	38	13
-1	14	3	3.9	3.9	8.6	350	13
-1	15	3	5.2	1.6	1.4	117	12
-1	16	3	4.0	1.5	3.4	196	12
-1	17	3	9.1	1.0	7.5	26	1
-1	18	3	3.7	3.7	0.6	231	13
-1	19	3	0.4	0.4	1.5	69	13
-1	20	3	0.8	0.8	0.4	342	13
-1	21	3	0.5	0.5	0.8	117	13
-1	22	3	7.1	0.6	6.3	1	1
-1	23	3	1.9	0.5	2.0	336	2
-1	24	3	0.9	0.9	2.0	105	13
-1	25	3	1.9	1.9	4.3	303	13
-1	26	3	2.9	0.7	3.8	254	12
-1	27	3	1.9	0.4	2.2	284	12
-1	28	3	0.3	0.3	1.1	141	13
-1	29	3	2.2	0.2	2.1	340	1
-1	30	3	1.8	0.2	2.1	87	31
-1	31	3	0.5	0.5	0.4	36	33
-2	1	3	3.7	0.5	4.0	275	1
-2	2	3	5.7	0.5	6.1	104	1
-2	3	3	4.2	0.5	3.7	118	1
-2	4	3	3.5	0.4	3.1	69	1
-2	5	3	2.3	0.3	2.2	283	1
-2	6	3	1.3	0.3	1.8	163	22
-2	7	3	1.6	0.4	2.4	240	12
-2	8	3	2.1	0.5	2.2	201	12
-2	9	3	2.6	0.6	3.2	122	12
-2	10	3	1.8	1.1	2.7	89	12
-2	11	3	3.1	0.5	3.3	3	12
-2	12	3	2.7	0.4	2.7	143	31
-2	13	3	6.5	0.9	6.1	74	11
-2	14	3	5.1	0.6	4.7	291	1
-2	15	3	8.5	0.9	9.0	145	1
-2	16	3	2.1	0.4	2.1	90	32
-2	17	3	7.0	0.6	7.0	37	1
-2	18	3	2.3	0.4	1.9	240	12
-2	19	3	2.0	0.4	2.7	191	2
-2	20	3	1.6	1.6	2.4	122	13
-2	21	3	0.9	0.9	2.3	252	13
-2	22	3	2.3	1.1	3.8	333	12
-2	23	3	1.7	1.7	3.5	17	13
-2	24	3	1.6	1.6	4.2	104	13
-2	25	3	4.7	0.7	5.3	314	11
-2	26	3	6.2	0.6	6.0	249	1
-2	27	3	0.8	0.8	1.0	99	13
-2	28	3	0.7	0.7	0.9	123	13
-2	29	3	0.8	0.8	1.4	33	13
-2	30	3	0.4	0.4	1.6	132	13
-2	31	3	0.6	0.6	0.8	57	13
-2	32	3	3.9	0.5	4.2	108	1
-3	1	3	2.8	0.3	2.6	322	1
-3	2	3	5.1	0.7	5.4	210	1
-3	3	3	5.0	0.8	5.1	68	12
-3	4	3	4.1	0.4	3.6	303	1
-3	5	3	3.9	0.5	4.5	174	31
-3	6	3	1.5	1.1	2.2	83	12
-3	7	3	3.8	0.4	3.1	84	1
-3	8	3	1.9	0.5	1.9	154	12
-3	9	3	5.5	0.4	5.5	169	21
-3	10	3	2.8	0.3	2.8	221	1
-3	11	3	2.9	0.5	3.7	274	12
-3	12	3	5.2	0.5	5.3	314	1
-3	13	3	3.9	0.5	3.4	75	31
-3	14	3	1.5	0.7	3.0	242	12
-3	15	3	6.3	0.8	6.9	174	1
-3	16	3	4.9	0.7	4.2	56	11
-3	17	3	1.9	0.5	2.6	346	12
-3	18	3	0.5	0.5	0.9	175	13
-3	19	3	1.3	0.7	2.5	266	12
-3	20	3	0.8	0.8	0.2	202	33
-3	21	3	1.6	0.5	1.4	275	2
-3	22	3	3.7	0.3	3.4	290	1
-3	23	3	6.0	0.5	5.0	35	1
-3	24	3	3.4	0.3	3.1	62	1
-3	25	3	2.2	0.3	2.2	301	1
-3	26	3	2.5	0.3	2.9	243	31
-3	27	3	2.0	0.3	2.2	154	1
-3	28	3	0.7	0.7	0.7	79	13
-3	29	3	0.4	0.4	0.7	143	13
-3	30	3	0.7	0.7	0.9	187	13
-4	1	3	0.6	0.6	0.8	300	13
-4	2	3	2.2	0.2	2.3	253	1
-4	3	3	2.3	0.2	2.3	70	1
-4	4	3	3.5	0.3	3.7	306	1
-4	5	3	4.8	0.4	5.0	185	1
-4	6	3	3.3	0.3	3.4	73	1
-4	7	3	0.8	0.8	0.9	305	33
-4	8	3	0.7	0.7	1.5	131	13
-4	9	3	2.2	0.3	1.7	227	31
-4	10	3	1.7	0.4	1.3	220	2
-4	11	3	3.5	0.5	4.1	274	31
-4	12	3	5.8	0.4	5.8	325	21
-4	13	3	3.5	0.5	3.9	36	31
-4	14	3	0.6	0.6	1.5	127	13
-4	15	3	2.2	0.3	2.4	173	31
-4	16	3	2.5	0.2	2.9	95	1
-4	17	3	0.8	0.8	1.6	342	33
-4	18	3	0.6	0.6	0.8	157	13
-4	19	3	0.6	0.6	1.2	292	13
-4	20	3	0.3	0.3	0.9	316	13
-4	21	3	0.3	0.3	0.4	143	13
-4	22	3	0.3	0.3	1.1	324	13
-4	23	3	2.4	0.2	2.7	41	1
0	5	4	7.1	0.5	6.4	3	21
0	6	4	7.8	0.6	7.3	199	21
0	7	4	4.4	0.3	4.3	248	21
0	8	4	3.1	0.2	3.1	227	21
0	9	4	1.9	0.1	2.0	356	21
0	10	4	7.7	0.6	6.9	163	21
0	11	4	2.4	0.2	2.3	148	21
0	12	4	9.3	0.7	8.7	104	21
0	13	4	20.2	1.5	19.1	358	21
0	14	4	15.7	1.2	15.8	276	21
0	15	4	11.4	0.9	10.3	194	21
0	16	4	3.9	0.3	3.9	352	21
0	17	4	2.4	0.2	2.5	157	21
0	18	4	1.7	0.3	1.5	274	21
0	19	4	2.5	0.2	2.3	127	21
0	20	4	1.7	0.3	1.9	131	21
0	21	4	3.0	0.2	3.1	242	21
0	22	4	2.0	0.2	2.1	338	21
0	23	4	3.1	0.2	2.9	88	21
0	24	4	0.6	0.6	0.8	254	23
0	25	4	3.0	0.2	3.0	203	21
0	26	4	0.5	0.5	0.3	235	23
0	27	4	1.1	0.2	1.1	226	21
1	0	4	2.2	0.6	2.5	74	12
1	1	4	4.9	0.6	4.2	60	1
1	2	4	3.5	0.3	3.5	83	1
1	3	4	7.3	0.7	6.7	316	1
1	4	4	5.3	0.7	5.0	3	31
1	5	4	3.3	0.3	3.3	60	1
1	6	4	2.7	2.7	6.2	235	13
1	7	4	8.5	1.1	8.6	299	31
1	8	4	5.7	0.9	5.6	32	12
1	9	4	4.0	1.3	4.1	133	12
1	10	4	9.2	1.0	9.4	90	1
1	11	4	2.7	0.6	2.6	221	12
1	12	4	10.9	1.0	9.6	114	1
1	13	4	14.6	1.3	12.8	359	1
1	14	4	9.6	0.9	8.2	280	1
1	15	4	7.1	1.0	6.8	213	11
1	16	4	4.5	0.5	4.1	157	1
1	17	4	2.1	0.4	1.8	68	32
1	18	4	0.7	0.7	1.4	268	13
1	19	4	1.7	0.4	1.8	121	12
1	20	4	3.0	0.3	3.2	129	1
1	21	4	1.5	0.3	0.9	154	2
1	23	4	0.5	0.5	1.1	32	3
1	24	4	0.6	0.6	1.9	232	13
2	0	4	0.0	0.5	0.7	46	14
2	1	4	4.1	0.4	4.2	123	1
2	2	4	1.4	0.5	1.1	94	12
2	3	4	0.8	0.8	0.7	197	13
2	4	4	2.0	0.4	2.2	266	32
2	5	4	1.3	0.6	2.3	175	12
2	6	4	5.7	0.5	5.4	245	1
2	7	4	6.8	0.6	7.2	316	1
2	8	4	5.2	0.5	4.6	359	1
2	9	4	2.0	2.0	3.1	165	13
2	10	4	2.4	0.5	3.0	58	12
2	11	4	0.7	0.7	1.3	130	13
2	12	4	1.6	0.5	2.1	58	32
2	13	4	3.9	0.4	3.5	352	1
2	14	4	3.5	0.3	3.6	316	1
2	15	4	3.5	0.3	3.2	236	1
2	16	4	2.5	0.3	2.3	168	1
2	17	4	2.2	0.2	2.1	331	1
2	18	4	3.1	0.2	3.0	13	1
3	0	4	1.6	0.5	2.2	166	32
3	1	4	1.9	0.4	1.6	80	2
3	2	4	1.2	0.6	0.7	181	2
3	3	4	1.6	0.6	2.2	119	12
3	4	4	0.7	0.7	0.8	257	13
3	5	4	1.1	0.6	1.2	168	2
3	6	4	1.7	0.3	2.1	220	2
3	7	4	2.3	0.3	2.6	302	1
3	8	4	0.3	0.3	1.0	45	13
3	9	4	0.6	0.6	0.8	202	13
3	10	4	0.3	0.3	0.9	217	13
3	11	4	1.6	0.1	1.7	119	1
-1	1	4	2.6	0.4	2.7	308	12
-1	2	4	8.0	1.3	8.2	148	12
-1	3	4	21.1	1.6	22.0	34	21
-1	4	4	10.0	1.2	10.0	299	1
-1	5	4	3.1	0.4	3.7	300	1
-1	6	4	4.0	0.4	3.7	353	1
-1	7	4	3.8	0.6	3.9	67	12
-1	8	4	1.3	0.6	1.5	1	12
-1	9	4	3.6	1.1	1.1	88	12
-1	10	4	5.2	0.7	4.8	207	31
-1	11	4	0.7	0.7	1.0	169	13
-1	12	4	12.8	1.2	12.0	41	1
-1	13	4	8.5	1.1	9.2	3	31
-1	14	4	7.2	0.9	7.1	290	1
-1	15	4	8.7	0.9	7.6	209	1
-1	16	4	6.7	1.2	5.2	34	12
-1	17	4	4.4	0.5	3.6	179	1
-1	18	4	2.3	2.3	2.9	100	13
-1	19	4	2.7	2.7	1.4	74	13
-1	20	4	1.8	1.8	3.1	151	13
-1	21	4	7.6	1.0	7.5	225	1
-1	22	4	2.0	0.7	2.8	327	12
-1	23	4	2.1	0.4	2.0	76	32
-1	24	4	1.2	0.6	0.7	168	2
-1	25	4	2.4	0.3	2.2	205	1
-1	26	4	0.8	0.8	0.8	87	13
-1	30	4	2.9	0.3	2.4	338	1
-2	1	4	5.6	1.4	5.5	165	12
-2	2	4	2.5	2.5	3.3	93	13
-2	3	4	10.3	1.0	9.7	23	1
-2	4	4	8.2	0.9	5.0	310	1
-2	5	4	1.0	1.0	1.4	231	13
-2	6	4	3.9	0.3	4.0	37	21
-2	7	4	5.2	0.5	4.8	71	1
-2	8	4	5.8	1.1	7.9	95	12
-2	9	4	4.2	1.6	6.4	112	12
-2	10	4	10.5	1.0	11.6	171	1
-2	11	4	10.3	1.1	10.6	258	1
-2	12	4	7.9	0.7	7.1	20	1
-2	13	4	2.6	0.6	2.8	33	12
-2	14	4	2.9	0.3	3.0	267	1
-2	15	4	4.1	0.4	4.1	239	1
-2	16	4	2.0	0.4	2.5	145	32
-2	17	4	2.3	0.3	2.4	102	1
-2	18	4	1.3	1.3	2.5	190	13
-2	19	4	1.2	1.2	1.0	262	13
-2	20	4	2.2	0.8	2.1	149	12
-2	21	4	8.3	0.8	7.2	228	1
-2	22	4	4.1	0.4	3.9	320	1
-2	23	4	1.9	0.4	1.6	310	2
-2	24	4	1.6	0.5	1.5	165	32
-2	25	4	0.8	0.8	1.3	58	13
-2	26	4	0.6	0.6	1.0	186	13
-2	27	4	0.7	0.7	1.5	60	13
-2	28	4	1.5	0.3	1.6	94	12
-3	1	4	2.5	0.3	2.0	187	1
-3	2	4	2.0	0.4	1.5	97	2
-3	3	4	2.8	0.3	2.2	19	1
-3	4	4	0.9	0.9	2.0	310	13
-3	5	4	2.1	0.4	1.0	228	32
-3	6	4	1.2	1.2	1.4	177	13
-3	7	4	2.1	2.1	2.5	109	13
-3	8	4	1.6	1.6	1.2	168	13
-3	9	4	5.1	0.4	4.3	93	21
-3	10	4	9.7	0.9	9.1	180	1
-3	11	4	10.6	1.0	10.1	261	1
-3	12	4	6.7	0.6	6.1	327	1
-3	13	4	2.1	0.4	2.5	34	32
-3	14	4	4.3	0.4	4.5	325	1
-3	15	4	0.8	0.8	1.5	177	13
-3	16	4	0.7	0.7	1.2	73	13
-3	17	4	0.5	0.5	1.6	35	13
-3	18	4	1.6	0.5	2.2	220	32
-3	19	4	0.7	0.7	1.4	241	13
-3	20	4	0.6	0.6	1.3	96	13
-3	21	4	3.9	0.4	3.2	262	1
-3	22	4	2.2	0.3	2.4	331	1
-3	23	4	0.6	0.6	1.1	337	13
-3	24	4	0.4	0.4	0.8	182	13
-3	25	4	1.8	0.3	1.6	50	12
-3	26	4	1.6	0.3	1.6	238	2
-3	27	4	3.0	0.3	2.6	102	1
-3	28	4	1.9	0.2	1.7	33	1
-3	29	4	0.0	0.5	0.5	94	14
-4	6	4	0.6	0.6	0.8	77	3
-4	8	4	1.6	0.3	1.9	287	12
-4	9	4	1.2	0.3	1.7	60	32
-4	10	4	1.9	0.3	2.5	161	12
-4	11	4	3.6	0.3	4.1	269	1
-4	13	4	0.4	0.4	1.1	72	13
-4	14	4	2.3	0.3	2.3	4	1
-4	15	4	1.2	0.4	1.1	137	32
-4	16	4	2.1	0.2	2.1	81	1

there is no evidence of intermolecular hydrogen bonding. The distances parallel to the *a* axis between the atoms of adjacent molecules are of course all equal to the cell dimension 3·80 Å.

Temperature parameters are given in Table 4. Calculations of the thermal ellipsoids yielded very little of value. No clear evidence was to be seen of any rigid-body motions, and the only general feature of note was the larger amplitudes of vibration of the hydroxyl and nitro groups.

The observed and calculated structure amplitudes, the calculated phase angles and the e.s.d's of the observed amplitudes are listed in Table 5.

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References

BEINTEMA, J. (1965). *Acta Cryst.* **18**, 647.

BUNN, C. W. (1946). *Chemical Crystallography*. Oxford Univ. Press.

BUSING, W. R. & LEVY, H. A. (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis*. Ed. R. PEPINSKY and others. Oxford: Pergamon Press.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS, A Fortran Crystallographic Least-Squares Program*. Oak Ridge National Laboratory, Oak Ridge, Tennessee.

COPPENS, P. & SCHMIDT, G. M. J. (1965). *Acta Cryst.* **18**, 654.

CRAIG, D. C. (1967). Private communication.

GRAINGER, C. T. (1969). *Acta Cryst.* A**25**, 427.

GRAINGER, C. T. & MCCONNELL, J. F. (1969). *Acta Cryst.* A**25**, 422.

International Tables for X-ray Crystallography (1952). Vol. I. Birmingham: Kynoch Press.

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

KASTALSKY, V. (1967). Private communication.

KENNICOTT, P. R. (1963). G. E. Research Laboratory, Schenectady, New York, Report No. 63-RL-(3321G).

MAK, T. C. W. & TROTTER, J. (1965). *Acta Cryst.* **18**, 68.

PARRISH, W. (1960). *Acta Cryst.* **13**, 838.

QURASHI, M. M. (1953). *Acta Cryst.* **6**, 668.

ROLLETT, J. S. (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis*. Ed. R. PEPINSKY and others. Oxford: Pergamon Press.

TAYLOR, C. A. & LIPSON, H. (1964). *Optical Transforms*. London: Bell.

TRUEBLOOD, K. N., GOLDISH, E. & DONOHUE, J. (1961). *Acta Cryst.* **14**, 1009.

VENKATARAMAN, K. (1952). *The Chemistry of Synthetic Dyes*, Vol. 1. New York: Academic Press.

WATSON, H. C. & HARGREAVES, A. (1958). *Acta Cryst.* **11**, 556.

Acta Cryst. (1969). B**25**, 1970

The 5-Hydroxyl Configuration in Dialuric Acid Monohydrate by Neutron Crystal Structure Determination

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The crystal structure of dialuric acid monohydrate (5,6-dihydroxyuracil) has been reinvestigated with three-dimensional neutron diffraction data, in order to obtain more accurate hydrogen atom positions than in Bolton's X-ray determination. The crystal data of Bolton (*Acta Cryst.* (1965) **19**, 1051) were assumed, *i.e.* $a=12{\cdot}714$, $b=3{\cdot}676$, $c=12{\cdot}949$ Å, $\beta=94°24'$, space group $P2_1/n$, with four formula units, $C_4H_4O_4N_2.H_2O$, in the unit cell. With 871 neutron data above background, least-squares refinement of positional and anisotropic thermal parameters for all atoms gave a final R index of 0·05. Molecular association in this crystal structure is dominated by conventional hydrogen bonds (two NH---OC bonds and three hydrogen bonds involving the water molecule) and van der Waals interactions within stacks of parallel, almost flat, dialuric acid molecules. Averaged over time, the O–H bond of the 5-hydroxyl group is found to be restricted to a range of about 50° of the full rotation about the C–O bond. In its mean position, this bond is twisted 67° from the configuration of molecular coplanarity in which the hydrogen atom is nearest the 4-position of the pyrimidine ring. There are weak hydrogen bonding interactions of the 5-hydroxyl group throughout the observed range of O–H twist, with no position seeming to be particularly favourable. For the non-hydrogen atoms, the greatest discrepancy between X-ray and neutron bond lengths is 0·014 Å for N(1)–C(6) for which the estimated standard deviation is 0·005 Å.

Introduction

An X-ray crystal structure determination of dialuric acid monohydrate (5,6-dihydroxyuracil, I) was first reported by Alexander & Pitman (1956) for the structure in projection. Bolton (1965), using three-dimensional X-ray intensity data, confirmed the earlier work and reported the location of the imine hydrogen atoms.