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The Crystal Structure of 1,5-Dinitronaphthalene

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Crystals of 1,5-dinitronaphthalene are monoclinic, space group $P2_1/a$, with two molecules in the unit cell. The structure has been determined from projections along the three principal crystallographic axes, and details of the molecular geometry and dimensions have been obtained. The nitrogen atom and all the carbon atoms lie on one plane, but the planes of the nitro groups are rotated about the C–N bonds 49° out of the aromatic plane.

Introduction

The author has recently investigated the crystal and molecular structures of aromatic nitro compounds in which the steric effects of groups *ortho* to the nitro groups prevent the attainment of completely coplanar configurations, and reduce the resonance interaction between the nitro groups and the aromatic π-electrons (Trotter, 1958, 1959*a*, *b*, *c*, *d*). In all the molecules studied so far, the nitro groups have been hindered by two ortho groups, and are rotated about the C–N bonds approximately 65° out of the aromatic planes. The structure of 1,5-dinitronaphthalene, in which there is overcrowding effectively only on one side of the nitro group, has now been determined. The structure of this molecule has been investigated previously by Sevastyanov, Zhdanov & Umansky (1947, 1948), but these authors, from one projection only, made the unlikely deduction that the molecule is completely planar.

Experimental

Crystals of 1,5-dinitronaphthalene are off-white in colour, and consist of prisms elongated along the *c*-axis. Sevastyanov, Zhdanov & Umansky (1947, 1948) quote a value of 1·62 g.cm.$^{-3}$ for the measured density, but in redetermining the density it was found that the crystals floated in carbon tetrachloride, so that the true value must be less than 1·595 g.cm.$^{-3}$. An accurate value was determined by flotation in a mixture of chloroform and carbon tetrachloride, and also in aqueous potassium iodide solution; the mean of several determinations was 1·578 g.cm.$^{-3}$.

The unit-cell dimensions and space group were determined from rotation and oscillation photographs of a crystal rotating about the *c*-axis, an *hk*0 Weissenberg film, and 0*kl* and *h*0*l* precession films. The values of the unit-cell parameters differ slightly, but significantly, from those given in the previous investigation.

Crystal data

1,5-Dinitronaphthalene, $C_{10}H_6N_2O_4$; $M=218{\cdot}2$; m.p. 217 °C.

Monoclinic,

$a=7{\cdot}76\pm0{\cdot}02$, $b=16{\cdot}32\pm0{\cdot}04$, $c=3{\cdot}70\pm0{\cdot}01$ Å, $\beta=101°\,48'\pm10'$.

Volume of the unit cell = 458·7 Å^3.

Density, calculated (with $Z=2$) = 1·579 g.cm.$^{-3}$, measured = 1·578 g.cm.$^{-3}$.

Absorption coefficient for X-rays, $\lambda=1{\cdot}542$ Å, $\mu=12{\cdot}3$ cm.$^{-1}$; $\lambda=0{\cdot}7107$ Å, $\mu=1{\cdot}52$ cm.$^{-1}$.

Total number of electrons per unit cell = $F(000)=224$.

Absent spectra: *h*0*l* when *h* is odd, 0*k*0 when *k* is odd. Space group is $P2_1/a$–C^5_{2h}.

Molecular symmetry-centre.

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* National Research Council Postdoctorate Fellow.

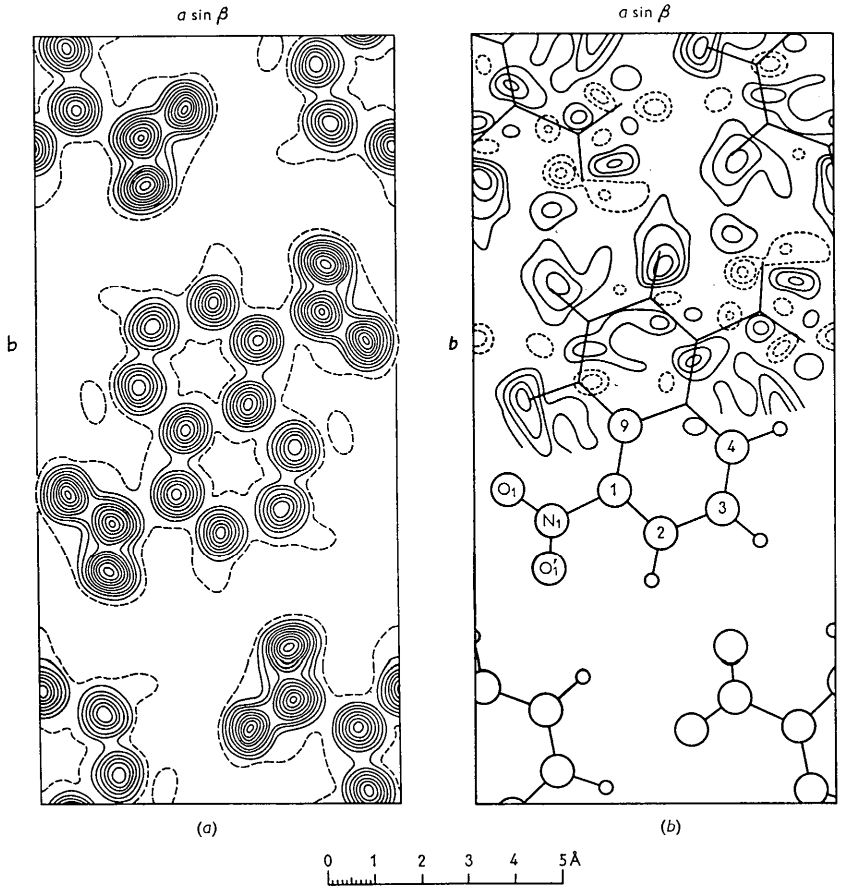
The intensities of the *hk*0 reflexions were recorded on moving-film exposures for a crystal rotating about the *c*-axis, using Cu $K\alpha$ radiation, with multiple-film technique to correlate strong and weak reflexions. The 0*kl* and *h*0*l* reflexions were recorded on precession films with Mo $K\alpha$ radiation, using multiple exposures for intensity correlation. All the intensities were estimated visually, the range being about 6000 to 1. The same crystal was used for all the photographs; the cross-section normal to the *c*-axis was $0{\cdot}30 \times 0{\cdot}20$ mm., and no absorption corrections were applied. The structure amplitudes were derived by the usual formulae for a mosaic crystal, the absolute scale being established later by correlation with the calculated structure factors. 134 independent *hk*0 reflexions, 26 *h*0*l*, and 42 0*kl* were observed, representing 83%, 60% and 50% respectively of the possible number observable with the radiations and experimental conditions used.

Structure analysis

[001] *projection*

In deriving an approximate trial structure it was assumed initially that the molecule was completely planar with C–C and C–N bond lengths 1·40 Å, N–O bond lengths 1·20 Å, and all valency angles 120°. The molecular orientation in the *c*-axis projection was determined from an examination of the *hk*0 weighted reciprocal lattice, a choice between possible orientations then being made by computing a few structure factors. The trial structure deduced was similar to that of the previous investigators. Structure factors were calculated for the stronger reflexions, and a Fourier series then computed using measured structure amplitudes and calculated signs. On the resulting electron-density map all the carbon, nitrogen and oxygen atoms were well resolved. New atomic centres were chosen and structure factors calculated for all the *hk*0 reflexions, using McWeeny's (1951) curves for carbon, nitrogen and oxygen, corrected for thermal vibration with $B = 4{\cdot}8$ Å^2 for all the atoms. The value of R, the usual discrepancy factor, was 26·8%, and it was obvious that the temperature factor used was much too large. Taking $B = 2{\cdot}8$ Å^2, R was reduced to 21·7%.

Refinement of the positional and temperature parameters proceeded by computing successive $(F_o - F_c)$ syntheses. On the first difference map, there were regions of high density in positions corresponding to



(*a*) (*b*)

Fig. 1. (*a*) Electron-density projection along the *c*-axis; contours at intervals of 1 e.Å^{-2}, with the one-electron line broken. (*b*) *upper*, $(F_o - F_c)$ synthesis, contours at intervals of 0·2 e.Å^{-2}, negative contours broken, zero contour omitted; *lower*, projection of the structure along [001].

hydrogen atoms, and a contribution from the hydrogens was included in the F_c values, coordinates being obtained by assuming that they were situated on the diagonals of the rings, with C–H = 1·08 Å. After two cycles, R had dropped to 12·2%. Observed and calculated structure factors are listed in Table 5, and the final F_o synthesis, computed with measured structure amplitudes and calculated signs, is shown in Fig. 1(*a*), the interpretation of this map being shown on the lower part of Fig. 1(*b*).

The upper part of Fig. 1(*b*) shows a difference synthesis, computed with coefficients $(F_o - F_c')$, where F_c' are structure factors calculated omitting the hydrogen atom contributions. There are several significant features on this map:

(i) maxima of 0·7–0·9 e.Å^{-2} close to the expected hydrogen positions;
(ii) electron-density fluctuations corresponding to anisotropic thermal vibrations of the nitrogen and oxygen atoms;
(iii) maxima on most of the C–C bonds, these indicating anisotropic vibration of the carbon atoms.

The hydrogen-atom contributions were included in the structure-factor calculations, but no allowance was made for the anisotropic vibrations.

The projected O–O distances suggested that the nitro groups were not in the naphthalene plane, but from this projection alone of course it was not possible to deduce the angles between the planes, since the nitro groups could be tilted out of the plane of projection in the same sense as or in the opposite sense to the aromatic rings.

z-Coordinates

Approximate z-coordinates for the carbon and nitrogen atoms were obtained by considering the bond distances in the c-axis projection, and oxygen parameters were then deduced by assuming that the nitro groups were tilted out of the [001] projection plane in the same direction as the naphthalene skeleton, so that there were only small angles between the naphthalene and nitro-group planes.

Structure factors were calculated for the $0kl$ reflexions, and, although the agreement between F_o and F_c was on the whole encouraging, there were a few poor agreements. A Fourier series was summed using those reflexions whose signs were reasonably certain (this necessitated omitting one or two fairly large terms). On the resulting electron-density map the position of the naphthalene skeleton was clearly indicated, and one of the oxygen atoms in the asymmetric unit was clearly resolved—in a position about 1 Å from its position in the trial structure. The nitro groups were clearly rotated out of the naphthalene plane about the C–N bonds by about 50°. Structure factors were recalculated with these revised coordinates, and the R value for the $0kl$ reflexions was 12·0%. A difference synthesis reduced this to 8·8%. Observed and calculated structure factors are listed in Table 5, and the final F_o synthesis is shown in Fig. 2. This map illustrates clearly the large deviations from coplanarity.

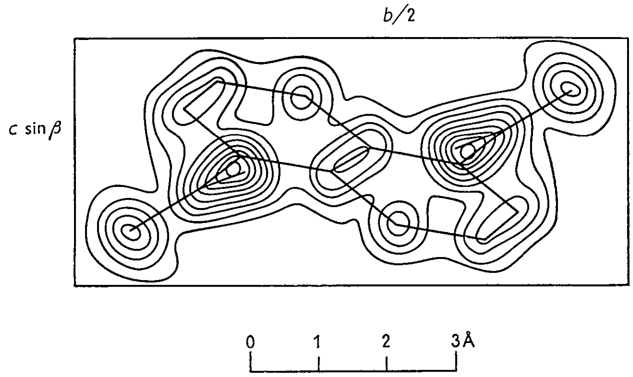


Fig. 2. Electron-density projection along the *a*-axis, with contours at intervals of 2 e.Å^{-2}.

Structure factors were calculated for the $h0l$ reflexions, and values of F_o and F_c are listed in Table 5, the R value being 16·9%. In the b-axis projection there is a great amount of overlap, and the Fourier map is not shown.

Coordinates and molecular dimensions

The final positional and temperature parameters of the carbon, nitrogen and oxygen atoms in one asymmetric unit are listed in Table 1, x, y and z being coordinates referred to the monoclinic crystal axes, and expressed as fractions of the unit-cell edges. The results of Sevastyanov, Zhdanov & Umansky are included for comparison. The coordinates of the carbon atoms can be fitted to an equation of the form

$$lX' + mY + nZ' = 0$$

where X', Y, Z' are coordinates expressed in Å units and referred to axes a', b and c, where a' is perpendicular to b and c; l, m and n were determined by the method of least squares. The equation of the nitro-

Table 1. *Positional and temperature parameters*

	Sevastyanov, Zhdanov & Umansky (1948)		Present analysis			
Atom	x	y	x	y	z	B (Å^2)
C_5	0·125	0·100	0·117	0·099	−0·017	1·9
C_6	−0·021	0·156	−0·009	0·152	−0·209	1·9
C_7	−0·203	0·120	−0·182	0·121	−0·319	1·9
C_8	−0·224	0·044	−0·213	0·041	−0·256	1·9
C_{10}	0·090	0·014	0·087	0·016	0·048	1·9
N_5	0·287	0·134	0·295	0·136	0·092	2·8
O_5	0·417	0·094	0·418	0·097	0·048	3·8
O_5'	0·306	0·202	0·307	0·200	0·271	3·8

group planes can of course be obtained directly from the coordinates of the nitrogen and oxygen atoms. The equations of the mean plane of the carbon atoms, and of the plane $N_5O_5O_5'$ are

Carbon atoms:

$$0{\cdot}2276X' - 0{\cdot}2744Y - 0{\cdot}9343Z' = 0 \, .$$

Nitro group:

$$0{\cdot}0300X' + 0{\cdot}5221Y - 0{\cdot}8524Z' - 1{\cdot}3276 = 0 \, .$$

The deviations of the atoms from these planes are listed in Table 2. The angle between the planes is 48·7°.

Table 2. *Deviations from the mean planes*

Atom	Distance from naphthalene plane	Distance from plane $N_5O_5O_5'$
C_5	−0·010 Å	−0·293 Å
C_6	+0·013	—
C_7	−0·020	—
C_8	+0·020	—
C_{10}	+0·041	—
N_5	+0·016	0
O_5	+0·736	0
O_5'	−0·849	0

The bond lengths and valency angles in the molecule were calculated from the coordinates of Table 1. Consideration of the estimated standard deviations (see later) suggested that the naphthalene skeleton did not differ significantly from symmetry *mmm*, and the mean values of the lengths and angles are shown in Fig. 3. Fig. 3 also shows the distances between those atoms which would be closer than the normal van der Waals separations in a planar molecule. (The hydrogen atoms were assumed to lie on the plane of the carbon atoms with C–H = 1·08 Å.)

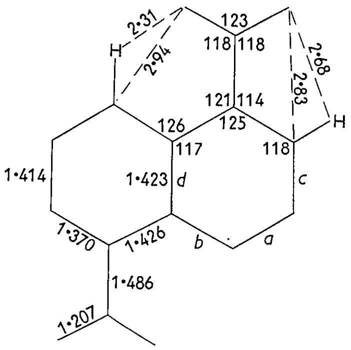


Fig. 3. Bond lengths, valency angles, and intramolecular 'overcrowded' distances.

Standard deviations

The standard deviations of the atomic positions, calculated from Cruickshank's (1949) formulae, are listed in Table 3. The corresponding standard deviations of the mean bond lengths are about 0·010 Å for bonds *a*, *b* and N–O, and 0·014 Å for bonds *c*, *d* and C–N.

Table 3. *Estimated standard deviations of atomic positions* (Å)

Atom	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	r.m.s.
C	0·010	0·012	0·012	0·011
N	0·009	0·010	0·011	0·010
O	0·008	0·010	0·010	0·009

Discussion

The naphthalene skeleton is completely planar within the limits of experimental error, the maximum deviation from the mean plane being 0·04 Å and the root mean square deviation 0·023 Å. The nitrogen atoms also lie on this plane, but the oxygen atoms of a nitro group lie one above and one below the plane at distances of 0·79 Å. The nitro groups are thus twisted out of the naphthalene plane about the C–N bonds, and the angle between the planes of a nitro group and the aromatic rings is 49°. The nitro groups are rotated out of the aromatic plane in such a way that the molecule is centrosymmetrical. In addition to these

Table 4. *Bond lengths in naphthalene and 1,5-dinitronaphthalene*

Bond	Naphthalene	1,5-Dinitronaphthalene
a	1·361 Å	1·370 Å
b	1·425	1·426
c	1·421	1·414
d	1·410	1·423
C–N	—	1·486
N–O	—	1·208

Table 5. *Measured and calculated structure factors*

hkℓ	F_o	F_c	hkℓ	F_o	F_c	hkℓ	F_o	F_c
200	26.4	-25.8	230	12.0	+11.2	440	1.8	- 0.3
400	1.3	- 0.1	240	2.4	- 0.5	450	2.7	+ 2.2
600	4.2	- 0.1	250	17.3	-14.9	460	13.8	+15.4
800	5.8	- 5.7	260	3.0	- 4.6	470	10.8	-11.8
10,0,0	10.6	+11.9	270	6.2	- 6.8	480	4.0	- 5.0
020	25.8	+23.5	280	3.1	+ 4.0	490	11.0	-12.0
040	53.0	-53.1	290	3.3	- 3.6	4,10,0	19.6	-19.3
060	8.1	+ 8.8	2,10,0	1.5	+ 2.4	4,11,0	15.9	-14.4
080	18.7	+19.5	2,11,0	4.0	+ 3.7	4,12,0	6.2	- 6.1
0,10,0	15.9	+16.5	2,12,0	20.4	+21.8	4,13,0	6.9	+ 8.1
0,12,0	10.8	-11.9	2,13,0	10.9	-12.4	4,14,0	6.4	+ 5.9
0,14,0	<1.8	- 2.9	2,14,0	2.3	+ 2.4	4,15,0	3.1	+ 3.1
0,16,0	4.4	- 6.9	2,15,0	6.9	- 6.8	4,16,0	1.8	- 0.1
0,18,0	3.2	- 3.9	2,16,0	5.7	- 8.2	4,17,0	5.9	- 5.8
0,20,0	3.3	+ 6.2	2,17,0	0.9	0	4,18,0	1.5	- 1.9
001	69.9	+70.8	2,18,0	1.6	+ 2.2	4,19,0	1.2	- 1.4
002	<4.5	- 4.2	2,19,0	1.9	- 3.1	510	<1.6	+ 1.0
003	16.1	+15.6	2,20,0	1.9	+ 2.9	520	3.1	- 2.6
			310	1.7	- 1.5	530	<1.6	+ 2.2
110	27.2	+25.1	320	23.8	-22.5	540	2.9	- 2.8
120	37.2	-35.5	330	31.7	-31.5	550	13.1	-13.4
130	6.8	+ 7.6	340	37.6	-39.5	560	5.9	+ 5.7
140	21.6	+24.3	350	25.9	+24.5	570	<1.7	+ 0.8
150	10.6	+11.9	360	1.4	- 0.6	580	3.6	+ 1.9
160	3.5	+ 3.9	370	6.0	+ 4.8	590	8.4	- 8.0
170	21.7	+23.7	380	6.8	+ 7.8	5,10,0	3.8	- 3.8
180	20.7	+21.8	390	7.0	- 7.1	5,11,0	4.3	- 6.5
190	1.1	- 3.7	3,10,0	1.3	- 4.2	5,12,0	<1.8	- 2.1
1,10,0	1.2	+ 1.9	3,11,0	2.9	- 2.6	5,13,0	<1.7	- 1.1
1,11,0	4.4	- 5.6	3,12,0	17.2	-16.4	5,14,0	<1.6	+ 0.3
1,12,0	6.4	- 7.6	3,13,0	6.3	+ 9.6	5,15,0	6.8	- 5.7
1,13,0	5.3	+ 4.5	3,14,0	10.2	-10.4	5,16,0	2.3	- 1.7
1,14,0	<1.8	- 5.1	3,15,0	8.3	+ 8.0	5,17,0	1.1	+ 1.8
1,15,0	1.7	- 3.0	3,16,0	4.7	+ 5.1	610	17.8	+18.1
1,16,0	9.0	-11.5	3,17,0	5.0	- 7.1	620	15.4	-13.6
1,17,0	<1.7	- 1.0	3,18,0	1.4	+ 2.5	630	15.3	+12.4
1,18,0	<1.6	+ 1.4	3,19,0	0.9	+ 0.5	640	6.3	- 7.2
1,19,0	1.7	+ 2.4	410	23.6	-23.5	650	<1.8	- 2.7
1,20,0	<1.1	+ 0.1	420	15.2	-12.8	660	7.1	+ 6.3
210	36.9	+36.7	430	<1.4	- 1.2	670	1.5	- 0.2
220	8.2	+ 8.6						

Table 5 (*cont.*)

hkℓ	F_o	F_c	hkℓ	F_o	F_c	hkℓ	F_o	F_c
680	1.5	− 0.5	970	0.9	− 1.2	041	28.5	−26.8
690	3.3	+ 2.4				051	< 3.6	− 1.7
6,10,0	6.3	− 7.5	$20\bar{5}$	5.8	+ 5.6	061	10.3	− 7.5
6,11,0	6.3	+ 7.1	$20\bar{4}$	<3.4	+ 0.2	071	< 3.9	+ 0.4
6,12,0	5.0	− 5.4	$20\bar{3}$	4.0	−10.1	081	20.8	+16.7
6,13,0	1.5	− 1.8	$20\bar{2}$	12.9	+ 8.0	091	21.0	+20.9
6,14,0	2.5	+ 3.8	$20\bar{1}$	18.6	+12.0	0,10,1	11.2	+11.7
6,15,0	1.2	− 1.2	201	22.9	+18.4	0,11,1	7.8	− 8.4
6,16,0	2.9	+ 2.8	202	18.5	+25.0	0,12,1	6.6	+ 7.8
710	3.3	+ 2.4	203	<3.3	+ 0.2	0,13,1	<4.6	− 0.3
720	8.5	+ 8.9	204	3.2	+ 0.1	0,14,1	<4.6	− 0.9
730	19.5	−17.5	$40\bar{5}$	<2.5	+ 0.9	0,15,1	4.9	− 4.6
740	7.2	+ 7.9	$40\bar{4}$	<3.4	− 1.9	0,16,1	7.0	− 7.7
750	9.6	− 7.9	$40\bar{3}$	5.7	− 8.0	0,17,1	6.9	− 8.2
760	7.4	− 7.5	$40\bar{2}$	13.2	−14.2	012	6.1	− 9.8
770	<1.8	+ 1.3	$40\bar{1}$	8.1	−10.4	022	15.3	+18.0
780	1.9	− 1.3	401	16.2	−13.6	032	20.8	−18.5
790	2.8	+ 1.8	402	23.0	−24.3	042	8.2	− 8.0
7,10,0	2.8	+ 3.9	403	15.9	−12.5	052	< 4.7	+ 4.3
7,11,0	<1.5	+ 1.7	404	<2.8	+ 0.2	062	14.6	−14.5
7,12,0	2.7	+ 5.0	$60\bar{4}$	<3.2	− 1.9	072	11.9	+ 9.2
7,13,0	3.3	+ 4.4	$60\bar{3}$	10.3	−10.3	082	12.4	+12.4
7,14,0	3.5	+ 3.8	$60\bar{2}$	21.3	−23.3	092	8.1	− 8.6
810	2.1	− 1.7	$60\bar{1}$	13.8	−12.8	0,10,2	11.8	+13.4
820	<1.7	− 0.4	601	10.9	−13.4	0,11,2	9.9	− 7.6
830	2.7	− 2.0	602	4.9	− 9.3	0,12,2	13.2	+12.9
840	<1.6	− 1.3	603	<3.2	+ 1.4	013	5.4	− 6.6
850	2.3	− 2.1	$80\bar{4}$	<2.9	+ 1.9	023	7.6	+ 6.3
860	<1.6	− 0.8	$80\bar{3}$	<3.4	− 1.4	033	14.3	−15.7
870	<1.5	− 0.6	$80\bar{2}$	4.3	+ 5.1	043	5.3	− 3.8
880	2.2	− 2.0	$80\bar{1}$	< 3.5	+ 2.8	053	5.3	+ 2.0
890	<1.3	+ 0.6	801	< 3.5	+ 1.4	063	7.8	− 7.0
8,10,0	2.5	− 2.2	802	< 3.1	+ 1.2	073	<5.4	− 1.6
8,11,0	<1.0	+ 0.6	$10,0,\bar{2}$	< 3.1	− 0.9	083	<5.4	+ 4.7
910	5.7	+ 5.1	$10,0,\bar{1}$	5.6	+ 6.2	093	13.5	−13.9
920	4.4	+ 2.9	10,0,1	3.0	+ 3.5	0,10,3	11.9	+11.8
930	2.5	+ 1.4				0,11,3	<5.4	− 0.2
940	<1.2	− 3.0	011	7.4	− 6.4	0,12,3	<5.4	− 2.4
950	<1.1	+ 0.7	021	29.7	+27.2	0,13,3	<5.4	− 0.3
960	2.5	+ 1.2	031	25.9	+27.2	0,14,3	6.7	− 5.6

deviations from coplanarity, however, there are significant in-plane displacements of the C–N bonds away from the *peri*-positions of the naphthalene skeleton, so that two of the exocyclic valency angles are reduced to 114°. These relatively small in-plane displacements allow the nitro groups to make much smaller angles (49°) with the aromatic plane than in molecules such as nitromesitylene, 9-nitroanthracene and 9,10-dinitroanthracene, where coplanar structures are blocked by two *ortho* groups, and the angles of twist are about 65°. The deviations from an ideal planar structure in 1,5-dinitronaphthalene are sufficient to increase the separations between the oxygen atoms and neighbouring carbon and hydrogen atoms to distances which are just a little less than the normal van der Waals contacts (Fig. 3).

The bond lengths in the molecule are compared in Table 4 with those in naphthalene (Abrahams, Robertson & White, 1949; Cruickshank, 1957), and there are no significant differences between corresponding carbon–carbon bond lengths in the two molecules. The C–N distances (1·486 Å) are similar to those in the other nitro compounds mentioned above, and to the corresponding length in nitrobenzene (Trotter, 1959*e*), while the N–O bond lengths (1·208 Å) are normal for nitro compounds.

Intermolecular distances

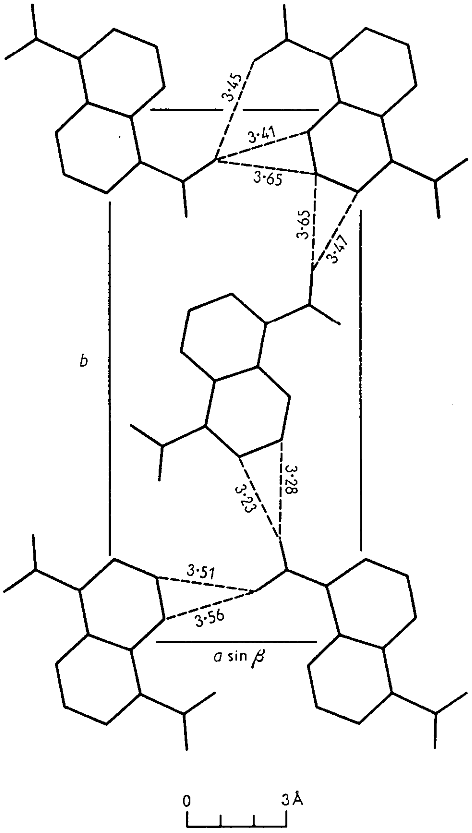


Fig. 4. Projection of the structure along [001], showing the shorter intermolecular contacts.

All the intermolecular distances correspond to normal van der Waals interactions. The perpendicular distance between the aromatic planes of molecules related by translation *c* is 3·45 Å. The shorter lateral contacts are illustrated in Fig. 4.

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