

the H-atom locations. The correct structure of the reaction product is clearly (3). The mechanism to arrive at this product will be discussed by one of us (SDB) elsewhere. The structure consists, Fig. 2, of isolated molecules separated by ordinary van der Waals distances. The molecule is almost planar (see Table 4).

An examination of Table 4 reveals that the ring system containing O(1) is planar including the exocyclic atoms but the remainder of the molecule is considerably out of this plane. Therefore, there is considerable charge delocalization within the O(1) ring which must be responsible for the relatively short C(9)–O(1) distance of 1.356 (4) Å. In spite of the fact that C(5) is also bound to this same ring and one would expect it and its neighbor O(5) also to be planar with the ring, *vis à vis* its relationship to the C(9)–C(10) double bond, it is not. C(5) is –0.10 (1) Å and O(5) –0.31 (1) Å from this plane. This situation is probably due to the tetrahedral constraints imposed by C(8) and C(7). These geometrical factors in turn no doubt contribute to the shortening of the C(5)–O(6) distance to 1.347 (4) Å and are consistent with the C(5)–O(5) distance being observed shorter than the C(2)–O(2) distance. An additional factor contributing to the non-planarity of the O(6) ring is the relatively short non-bonded C(11)···O(5) distance of 2.842 (5) Å. The above delocalization, no doubt, exists in the starting material as well and probably contributes to the formation of the unexpected reaction product.

The remaining distances and angles are normal and have the expected values.

We are grateful to the University of South Carolina for the purchase of the diffractometer.

#### References

- BLAND, N. & THORPE, J. F. (1912). *J. Chem. Soc.* pp. 856–870.
- BURKE, S. D., SAUNDERS, J. O. & MURTIASHAW, C. W. (1981). *J. Org. Chem.* **46**, 2425–2426.
- Enraf–Nonius (1980). *Data Collection Package for the CAD-4 Diffractometer* (revised for the PDP-8A, PDP-11 operation).
- FRENZ, B. A. (1980). *Enraf–Nonius Structure Determination Package*. Version 17, with local modifications for the PDP-11/40.
- JOHNSON, C. K. (1970). *ORTEP II*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- MAIN, P. (1976). *Crystallographic Computing Techniques*, edited by F. R. AHMED, pp. 97–105. Copenhagen: Munksgaard.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J. P. & WOOLFSON, M. M. (1978). *MULTAN 78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England, and Louvain, Belgium.
- STEWART, J. M. (1979). Editor, the XRAY system. Tech. Rep. TR-445. Computer Science Center, Univ. of Maryland, College Park, Maryland.

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### Crystallographic Studies of High-Density Organic Compounds: 4-Amino-5-nitrobenzo[1,2-*c*:3,4-*c'*]bis[1,2,5]oxadiazole 3,8-Dioxide\*

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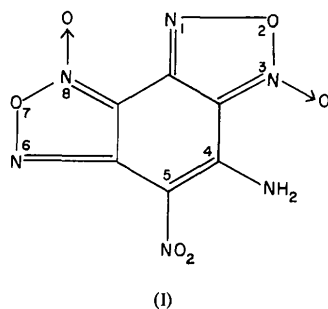
(Received 12 January 1982; accepted 6 April 1982)

**Abstract.** C<sub>6</sub>H<sub>2</sub>N<sub>6</sub>O<sub>6</sub>, *M<sub>r</sub>* = 254.1, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 13.6644 (6), *b* = 6.6969 (5), *c* = 9.5462 (8) Å, *D*<sub>calc</sub> = 1.93 Mg m<sup>-3</sup> for *Z* = 4; Cu *K*α(*λ* = 1.5418 Å) diffractometer data; 852 unique intensities; 747 intensities ≥ 3σ above background; final *R* = 0.034. The molecule is approximately planar; the largest out-of-plane deviations are associated with the

NH<sub>2</sub>, NO<sub>2</sub> and two oxide groups. Bond lengths and angles in the five-membered rings are similar to values observed in other oxadiazoles and benzoxadiazoles. Benzene annelation has no significant effect on the basic oxadiazole structure. The molecules pack in sheets approximately perpendicular to *c*, the shortest contact being a 2.940 (5) Å C···O. Hydrogen bonding by one of the amino hydrogen atoms is limited to a strong intramolecular O···H–N contact with the neighboring nitro group, while the other hydrogen enters into somewhat weaker intra- and intermolecular contacts with oxygen.

\* This name is the preferred one in line with IUPAC rules for organic nomenclature. The current *Chem. Abstr.* name is 5-nitrobenzo[1,2-*c*:3,4-*c'*]bis[1,2,5]oxadiazol-4-amine 3,8-dioxide. Our thanks to Dr K. L. Loening for this information.

**Introduction.** We are investigating the crystal structures of a number of high-density nitro-group-containing organic compounds, as a first step in an overall study of the relationships between molecular structure and crystal density in these types of substances. The title compound (I) is the first of the oxadiazoles to be investigated by us. Other nitroaromatic compounds that have been studied in this series are 3,5-diamino-2,4,6-trinitrophenol (Bhattacharjee & Ammon, 1981), 5-fluoro-2,4,6-trinitro-1,3-benzenediamine and 3,5-diamino-2,4,6-trinitrobenzamide (Ammon & Bhattacharjee, 1982).



(I) was obtained from Dr H. Adolph of the Naval Surface Weapons Center, Silver Spring, Maryland. Recrystallization from acetonitrile gave transparent yellow needles with square cross-sections. The space group and approximate cell parameters were obtained from oscillation and Weissenberg photographs with Ni-filtered Cu radiation. Accurate cell parameters were determined and intensity measurements made with a Picker FACS-I diffractometer and graphite-monochromated Cu radiation. A 0.37 × 0.07 × 0.07 mm crystal was mounted parallel to its long axis (*b*) and aligned to place this along the instrument's  $\varphi$  axis. The unit-cell parameters were determined by least squares from the  $2\theta$  values of nine reflections manually centered at  $\pm 2\theta$  (average  $|2\theta_0 - 2\theta| = 0.002^\circ$ ).

Intensities were measured with the  $2\theta$ - $\theta$  scan technique with a  $2\theta$  scan rate of  $2^\circ \text{ min}^{-1}$  with 10 s backgrounds. The scan width was calculated from  $1.9^\circ + 0.29^\circ \tan \theta$ . Four standards were monitored every 100 reflections during data collection. 869 reflections were measured to a  $2\theta$  maximum of  $127^\circ$ , which gave 852 unique reflections after averaging. Of these, 747 were  $3\sigma$  above background. The MULTAN-80 system (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) was used for the structure solution revealing all of the C, N and O atoms. The structure was refined by the method of full-matrix least squares with anisotropic temperature factors for the C, N and O atoms. The two hydrogens were located from a difference map and refined with isotropic temperature factors. The quantity minimized was  $\sum w(F_o - F_c)^2$ ,

Table 1. Fractional coordinates and temperature factors ( $\text{\AA}^2$ ) with *e.s.d.*'s in parentheses

A dagger denotes  $U_{eq}$ , the equivalent value of the anisotropic temperature factor coefficient, calculated from

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}/U_{iso}$
C(1)	-0.0135 (3)	-0.1540 (6)	0.5425 (4)	0.03 (1)†
C(2)	0.0048 (3)	0.0391 (6)	0.4834 (4)	0.032 (9)†
C(6)	-0.0591 (3)	0.1211 (6)	0.3808 (4)	0.04 (1)†
C(7)	-0.1455 (3)	0.0144 (6)	0.3376 (4)	0.044 (5)†
C(8)	-0.1669 (3)	-0.1764 (6)	0.3905 (4)	0.04 (1)†
C(9)	-0.0988 (3)	-0.2493 (6)	0.4963 (4)	0.032 (7)†
N(3)	0.0772 (3)	0.1636 (5)	0.5059 (4)	0.043 (6)†
N(5)	-0.0267 (3)	0.2963 (5)	0.3374 (4)	0.048 (3)†
N(7)	-0.2057 (3)	0.0994 (6)	0.2326 (4)	0.058 (7)†
N(8)	-0.2389 (3)	-0.2954 (7)	0.3556 (5)	0.039 (8)†
N(10)	-0.1042 (2)	-0.4212 (5)	0.5661 (3)	0.041 (6)†
N(12)	0.0359 (2)	-0.2546 (5)	0.6373 (4)	0.04 (1)†
O(3)	0.1491 (2)	0.1683 (5)	0.5825 (3)	0.040 (4)†
O(4)	0.0600 (2)	0.3310 (4)	0.4140 (3)	0.047 (8)†
O(7A)	-0.2790 (2)	0.0058 (5)	0.1920 (3)	0.058 (9)†
O(7B)	-0.1826 (2)	0.2656 (5)	0.1850 (3)	0.08 (1)†
O(10)	-0.1608 (2)	-0.5630 (4)	0.5710 (3)	0.043 (2)†
O(11)	-0.0176 (2)	-0.4283 (4)	0.6572 (3)	0.048 (7)†
H(8A)	-0.250 (4)	-0.411 (7)	0.398 (5)	0.11 (2)
H(8B)	-0.280 (3)	-0.249 (7)	0.293 (4)	0.07 (2)

$w = [1/\sigma(F)]^2$ . Scattering factors for C, N and O were calculated from the analytical expressions of Cromer & Mann (1968); the H terms were interpolated from the tabulated values of Stewart, Davidson & Simpson (1965). The final  $R$  ( $\sum |F_o| - |F_c| / \sum |F_o|$ ) and weighted  $R$  ( $[\sum w(F_o - F_c)^2 / \sum w F_o^2]^{1/2}$ ) factors were 0.034 and 0.029, respectively. Atomic coordinates and thermal parameters are listed in Table 1.\* All calculations were done on a Univac 1108 computer at the University of Maryland's Computer Science Center with the XRAY system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) of crystallographic programs.

**Discussion.** An ORTEP drawing (Johnson, 1971) of the molecule is given in Fig. 1, and bond lengths and angles are listed in Table 2. The molecule is approximately planar. With the elimination of the two oxide atoms and the amino and nitro groups, the average and maximum deviations of the benzoxadiazole atoms from their least-squares plane are 0.011 and 0.024 Å, respectively. The largest out-of-plane deviation of the eliminated atoms is 0.095 Å for the amino nitrogen.

Table 3 lists some of the bond lengths and angles common to (I) and several other oxadiazole *N*-oxide-

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36853 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

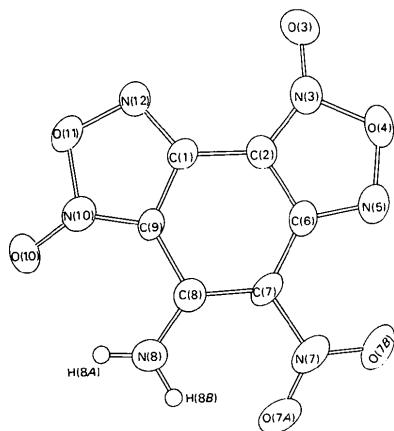


Fig. 1. An ORTEP drawing of (I) with C, N and O atoms depicted as 50% probability boundary ellipsoids. H atoms are shown as 0.1 Å radius circles.

Table 2. Bond lengths (Å) and angles (°) with *e.s.d.*'s in parentheses

C(1)–C(2)	1.433 (6)	C(9)–N(10)	1.332 (5)
C(1)–C(9)	1.400 (6)	N(3)–O(3)	1.224 (5)
C(1)–N(12)	1.315 (5)	N(3)–O(4)	1.443 (5)
C(2)–C(6)	1.422 (5)	N(5)–O(4)	1.412 (5)
C(2)–N(3)	1.312 (5)	N(7)–O(7A)	1.243 (5)
C(6)–C(7)	1.440 (6)	N(7)–O(7B)	1.243 (5)
C(6)–N(5)	1.321 (5)	N(8)–H(8A)	0.88 (5)
C(7)–C(8)	1.405 (6)	N(8)–H(8B)	0.88 (4)
C(7)–N(7)	1.417 (5)	N(10)–O(10)	1.226 (4)
C(8)–C(9)	1.457 (5)	N(10)–O(11)	1.469 (4)
C(8)–N(8)	1.309 (6)	N(12)–O(11)	1.387 (4)
C(2)–C(1)–C(9)	115.6 (3)	C(8)–C(9)–N(10)	126.9 (4)
C(2)–C(1)–N(12)	130.1 (4)	C(2)–N(3)–O(3)	136.0 (4)
C(9)–C(1)–N(12)	114.2 (3)	C(2)–N(3)–O(4)	105.7 (3)
C(1)–C(2)–C(6)	120.8 (3)	O(3)–N(3)–O(4)	118.3 (3)
C(1)–C(2)–N(3)	129.8 (4)	C(6)–N(5)–O(4)	105.4 (3)
C(6)–C(2)–N(3)	109.3 (3)	C(7)–N(7)–O(7A)	119.1 (4)
C(2)–C(6)–C(7)	120.6 (3)	C(7)–N(7)–O(7B)	118.1 (4)
C(2)–C(6)–N(5)	110.7 (3)	O(7A)–N(7)–O(7B)	122.9 (4)
C(7)–C(6)–N(5)	128.7 (4)	C(8)–N(8)–H(8A)	123 (3)
C(6)–C(7)–C(8)	121.3 (3)	C(8)–N(8)–H(8B)	116 (3)
C(6)–C(7)–N(7)	118.7 (3)	H(8A)–N(8)–H(8B)	120 (5)
C(8)–C(7)–N(7)	119.9 (3)	C(9)–N(10)–O(10)	106.2 (3)
C(7)–C(8)–C(9)	114.9 (3)	C(9)–N(10)–O(11)	136.4 (3)
C(7)–C(8)–N(8)	128.3 (4)	O(10)–N(10)–O(11)	117.4 (3)
C(9)–C(8)–N(8)	116.9 (4)	C(1)–N(12)–O(11)	104.7 (3)
C(1)–C(9)–C(8)	126.7 (4)	N(3)–O(4)–N(5)	108.9 (3)
C(1)–C(9)–N(10)	106.4 (3)	N(10)–O(11)–N(12)	108.4 (3)

containing molecules. These parameters are fairly consistent over the group of molecules and show no trend with structure. The two endocyclic oxadiazole N–O bond lengths [N(1)–O(1) and N(2)–O(1) in Table 3] are substantially different, with N(2)–O(1) being 0.05–0.13 Å larger than N(1)–O(1). These bond-length differences can be associated with differences in the relative electronegativities of the N(2)–O(2) unit and unsubstituted N(1) atom, which in turn can be translated into relative differences in the polarizations

of the *p* orbitals used by O(1) in the two N–O bonds. Thus, the enhanced electronegativity of N(2) would increase the amount of *p*-orbital character in the O(1) orbital directed toward N(2) which would produce a concomitant increase in *s*-orbital character in the hybrid orbital directed toward N(1). This imbalance of *p*-orbital character into the O(1)–N(2) bond leads to a lengthening of O(1)–N(2) and to a shortening of O(1)–N(1). Similar arguments can be developed to explain the exocyclic-bond-angle differences at N(2). In this case, the relative electronegativities of C(2) and O(1), the two oxadiazole ring atoms connected to N(2), must be considered. Since O(1) is more electronegative than C(2), the N(2) orbital toward O(1) will have more *p* character than that toward C(2), leading to a decrease in the O(1)–N(2)–O(2) angle over C(2)–N(2)–O(2). Steric effects clearly are not responsible for the angle difference, which is in the 18–21° range for all of the compounds listed in Table 3.

One may expect to see a characteristic pattern of C–C and C=C bond lengths in the six-membered rings in benzoxadiazoles (I)–(III), as a result of the bond-order fusing effects of the oxadiazole nucleus. The usual structure written for benzoxadiazoles shows the six-membered ring as a 1,3-cyclohexadiene, whereas the form is that of a cyclohexene in the benzo-bis-oxadiazoles. While the C=C–C=C pattern is quite clear in (II) and (III), the corresponding C=C in (I) is stretched to 1.405 (6) Å by a resonance interaction between the C(8)–amino and C(7)–nitro groups. The associated distances of 1.309 (6) Å for C–NH<sub>2</sub> and 1.417 (5) Å for C–NO<sub>2</sub> are indicative of substantial amounts of C=N character in these linkages. For comparison, the H<sub>2</sub>N–C–C–C–C–NO<sub>2</sub> distances in *p*-nitroaniline (Colapietro, Domenicano, Marcianti & Portalone, 1981) are 1.356, 1.407, 1.367, 1.391, 1.437 Å, and in *trans*-(CH<sub>3</sub>)<sub>2</sub>N–CH=CH–NO<sub>2</sub> the values are 1.334, 1.345, 1.394 Å (Hazell & Mukhopadhyay, 1980).

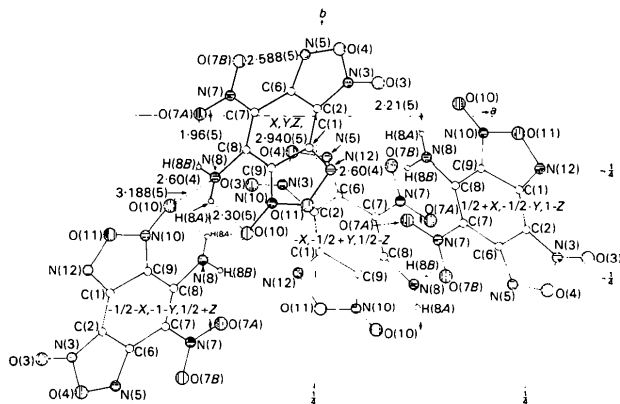
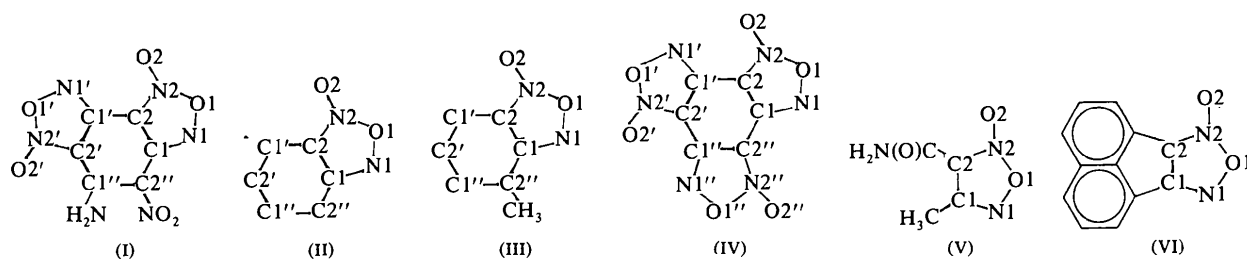


Fig. 2. Packing view down *c*; some contact distances (Å) and *e.s.d.*'s (in parentheses) are given.

Table 3. Bond lengths (Å), some angles (°) and their averages in the ring systems of six oxadiazole N-oxide structures



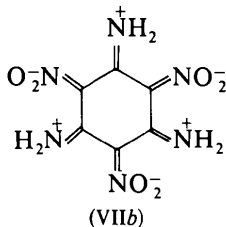
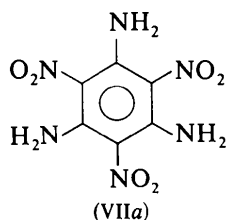
Bond/angle	(I)	(II) <sup>a</sup>	(III) <sup>b</sup>	(IV) <sup>c</sup>	(V) <sup>d</sup>	(VI) <sup>e</sup>
C(1)–C(2)	1.422 (5)	1.418 (4)	1.407 (5)	1.439 (7)	1.421 (3)	1.415 (6)
C(2)–C(1')	1.433 (6)	1.426 (4)	1.413 (5)	1.435 (7)		
C(1')–C(2')	1.400 (6)	1.337 (5)	1.365 (5)	1.416 (8)		
C(2')–C(1'')	1.457 (5)	1.441 (6)	1.455 (5)	1.454 (7)		
C(1'')–C(2'')	1.405 (6)	1.347 (5)	1.354 (5)	1.419 (7)		
C(2'')–C(1)	1.440 (6)	1.416 (5)	1.430 (5)	1.444 (8)		
C(1)–N(1)	1.315 (5) 1.321 (5)			1.300 (7) 1.305 (7) 1.322 (6)		
	<1.318 (5)>	1.319 (4)	1.330 (5)	<1.309 (7)>	1.305 (3)	1.334 (5)
C(2)–N(2)	1.312 (5) 1.332 (5)			1.330 (7) 1.336 (7) 1.318 (7)		
	<1.322 (5)>	1.312 (4)	1.327 (5)	<1.328 (7)>	1.324 (3)	1.311 (5)
N(1)–O(1)	1.412 (5) 1.387 (4)			1.377 (5) 1.398 (5) 1.380 (7)		
	<1.400 (5)>	1.374 (4)	1.385 (4)	<1.385 (7)>	1.379 (3)	1.371 (5)
N(2)–O(1)	1.443 (5) 1.469 (4)			1.475 (6) 1.466 (7) 1.462 (6)		
	<1.456 (5)>	1.454 (4)	1.443 (4)	<1.468 (7)>	1.436 (3)	1.501 (5)
N(2)–O(2)	1.224 (5) 1.226 (4)			1.201 (6) 1.207 (5) 1.211 (7)		
	<1.225 (5)>	1.231 (5)	1.235 (4)	<1.206 (7)>	1.236 (3)	1.179 (5)
C(2)–N(2)–O(2)	136.4 (3) 136.0 (4)			135.2 (6) 135.0 (6) 136.2 (6)		
	<136.2 (4)>	136.7 (3)	135.8 (3)	<135.5 (6)>	135.0 (2)	137.5 (4)
O(1)–N(2)–O(2)	117.4 (3) 118.3 (3)			118.8 (6) 119.3 (6) 118.4 (6)		
	<117.9 (3)>	116.6 (3)	117.9 (3)	<118.8 (6)>	117.2 (2)	116.6 (3)
C(2)–N(2)–O(1)	108.9 (3) 106.2 (3)			105.9 (6) 105.7 (6) 105.4 (6)		
	<107.6 (3)>	106.7 (3)	106.5 (3)	<105.7 (6)>	107.9 (1)	105.9 (3)

References: (a) Britton & Olson (1979); (b) Britton & Noland (1972); (c) Cady, Larson & Cromer (1966); (d) Calleri, Chiari, Villa, Manfredotti, Guastini & Viterbo (1975); (e) Calleri, Chiari & Verbo (1973).

In the case of the tris-oxadiazoles, (IV), the usual structure suggests that the six-membered ring should have a substantial degree of single-bond character. The C–C averages of 1.435 (5) and 1.426 (6) Å for (IV) and (I) are in line with this expectation, and with the

1.442 Å average observed in 1,3,5-triamino-2,4,6-trinitrobenzene (VII) (Cady & Larson, 1965). The expanded carbon–carbon distances in (VII) reflect the importance of a mesomeric structure like (VIIb) to the resonance hybrid and it is expected that the  $\pi$ -bond

orders in the six-membered ring of (VIIb) should be qualitatively similar to those in (IV).



A molecular packing diagram projected down *c* is shown in Fig. 2. The NH<sub>2</sub> group forms intramolecular hydrogen bonds H(8B)···O(7A), 1.96 (5) Å, and H(8A)···O(10), 2.30 (5) Å, with the flanking *N*-oxide and nitro groups. H(8A) forms a 2.21 (5) Å intermolecular contact with O(3) at  $-\frac{1}{2} + x, -\frac{1}{2} - y, 1 + z$ , whereas the shortest intramolecular contacts for H(8B) are 2.60 (4) Å with O(1) at  $-\frac{1}{2} - x, -1 - y, \frac{1}{2} + z$  and N(12) at  $-\frac{1}{2} + x, -\frac{1}{2} - y, 1 - z$ . Fig. 2 illustrates that the molecules are approximately perpendicular to *c*, and that there are no hydrogen bonds in this direction. The 2<sub>1</sub> axis at 0,0, $\frac{1}{2}$ , for example, orients two molecules so that there is partial overlap of the O(4) oxadiazole ring in one molecule with the O(11) ring of another. With the exception of C(7)···O(4), the contacts are greater than the appropriate sums of the van der Waals radii of aromatic C, O and N. The 2.940 (5) Å C(7)···O(4) distance, compared to a 3.1 Å value from the van der Waals radii of C and O (1.7 + 1.4 Å), may reflect a net attraction between these atoms. O(4) is electron rich while C(7) can be assumed to have a deficiency of electron density because of its NO<sub>2</sub> substituent.

Dr J. R. Holden has calculated the volumes of nearly fifty poly-nitro-group-containing organic compounds with the use of the atomic radii of Kitaigorodsky (1973). The packing coefficients (PC = total molecular volume/unit cell volume) have ranged from a low of 0.70 in [FC(NO<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>C(=NH)]<sub>2</sub> to a high of 0.81 in 1,3,5-triamino-2,4,6-trinitrobenzene. The PC of (I) at 0.78 is near the top of the list indicating that one component in the high crystal density observed for the compound is an efficient intermolecular packing ar-

angement which limits the amount of unoccupied space in the unit cell.

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### References

- AMMON, H. L. & BHATTACHARJEE, S. K. (1982). *Acta Cryst.* **B38**, 2083–2086.
- BHATTACHARJEE, S. K. & AMMON, H. L. (1981). *Acta Cryst.* **B37**, 2082–2085.
- BRITTON, D. & NOLAND, W. E. (1972). *Acta Cryst.* **B28**, 1116–1121.
- BRITTON, D. & OLSON, J. M. (1979). *Acta Cryst.* **B35**, 3076–3078.
- CADY, H. H. & LARSON, A. C. (1965). *Acta Cryst.* **18**, 485–496.
- CADY, H. H., LARSON, A. C. & CROMER, D. T. (1966). *Acta Cryst.* **20**, 336–341.
- CALLERI, M., CHIARI, G., VILLA, A. C., MANFREDOTTI, A. G., GUASTINI, C. & VITERBO, D. (1975). *Acta Cryst.* **B31**, 2384–2389.
- CALLERI, M., CHIARI, G. & VITERBO, D. (1973). *Cryst. Struct. Commun.* **2**, 335–338.
- COLAPIETRO, M., DOMENICANO, A., MARCIANTE, C. & PORTALONE, G. (1981). *Acta Cryst.* **A37**, C199.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- HAZELL, A. & MUKHOPADHYAY, A. (1980). *Acta Cryst.* **B36**, 747–748.
- JOHNSON, C. K. (1971). *ORTEP II*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- KITAIGORODSKY, A. I. (1973). *Molecular Crystals and Molecules*. New York: Academic Press.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN-80*. Univ. of York, England.
- STEWART, J. M., MACHIN, P. A., DICKINSON, C., AMMON, H. L., HECK, H. S. & FLACK, H. (1976). The XRAY system—version of 1976. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.