

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)
$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i\cdot\mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
P	0.0576 (1)	0.2056 (1)	0.4991 (1)	2.90 (1)
O(1)	0.0423 (1)	0.1593 (1)	0.4066 (1)	3.76 (3)
O(2)	0.0276 (1)	0.3217 (1)	0.5044 (1)	3.01 (3)
O(3)	0.1833 (1)	0.1946 (1)	0.5471 (1)	3.79 (3)
O(4)	-0.0136 (1)	0.1584 (1)	0.5768 (1)	3.55 (3)
C(1)	0.0325 (1)	0.3898 (1)	0.4283 (1)	2.76 (4)
C(2)	-0.0729 (1)	0.4317 (1)	0.3947 (1)	3.31 (4)
C(3)	-0.0762 (1)	0.5011 (1)	0.3243 (1)	3.58 (5)
C(4)	0.0249 (1)	0.5299 (1)	0.2844 (1)	3.20 (5)
C(5)	0.0216 (2)	0.6007 (1)	0.2089 (1)	4.14 (6)
C(6)	0.1177 (2)	0.6251 (2)	0.1677 (1)	4.56 (6)
C(7)	0.2228 (2)	0.5818 (2)	0.2002 (1)	4.36 (6)
C(8)	0.2302 (1)	0.5147 (1)	0.2733 (1)	3.53 (5)
C(9)	0.1320 (1)	0.4859 (1)	0.3186 (1)	2.87 (4)
C(10)	0.1346 (1)	0.4136 (1)	0.3947 (1)	2.71 (4)
C(11)	0.2443 (1)	0.3596 (1)	0.4307 (1)	3.01 (4)
C(12)	0.2743 (1)	0.3574 (1)	0.5367 (1)	2.87 (4)
C(13)	0.3368 (1)	0.4385 (1)	0.5845 (1)	2.90 (4)
C(14)	0.3626 (1)	0.5307 (1)	0.5386 (1)	3.46 (5)
C(15)	0.4199 (2)	0.6073 (1)	0.5878 (1)	4.16 (5)
C(16)	0.4560 (2)	0.5961 (2)	0.6834 (2)	4.39 (5)
C(17)	0.4338 (2)	0.5094 (2)	0.7291 (1)	3.96 (5)
C(18)	0.3721 (1)	0.4290 (1)	0.6821 (1)	3.18 (5)
C(19)	0.3432 (2)	0.3397 (1)	0.7310 (1)	3.67 (4)
C(20)	0.2799 (2)	0.2656 (1)	0.6860 (1)	3.62 (5)
C(21)	0.2463 (1)	0.2762 (1)	0.5897 (1)	3.12 (4)
C(22)	-0.1336 (1)	0.1422 (1)	0.5580 (1)	3.24 (4)
C(23)	-0.1720 (2)	0.0680 (1)	0.4953 (1)	3.96 (5)
C(24)	-0.2886 (2)	0.0512 (2)	0.4816 (2)	4.72 (5)
C(25)	-0.3639 (2)	0.1068 (2)	0.5306 (2)	4.79 (6)
C(26)	-0.3243 (2)	0.1804 (1)	0.5946 (1)	4.18 (6)
C(27)	-0.2063 (2)	0.1984 (1)	0.6074 (1)	3.84 (5)
C(28)	-0.4054 (2)	0.2383 (2)	0.6501 (2)	6.05 (8)

Table 2. Selected geometric parameters (Å, °)

P—O(1)	1.451 (1)	P—O(2)	1.571 (1)
P—O(3)	1.575 (1)	P—O(4)	1.574 (1)
O(2)—C(1)	1.413 (2)	O(3)—C(21)	1.410 (2)
O(4)—C(22)	1.424 (2)	C(11)—C(12)	1.523 (2)
C(1)—C(10)	1.365 (2)	C(12)—C(21)	1.366 (2)
C(10)—C(11)	1.518 (2)		
O(3)—P—O(4)	101.0 (1)	O(2)—P—O(4)	102.4 (1)
O(2)—P—O(3)	105.9 (1)	O(1)—P—O(4)	116.4 (1)
O(1)—P—O(3)	113.1 (1)	O(1)—P—O(2)	116.2 (1)
P—O(2)—C(1)	123.7 (1)	P—O(3)—C(21)	123.5 (1)
P—O(4)—C(22)	120.2 (1)	O(2)—C(1)—C(10)	120.8 (1)
O(2)—C(1)—C(2)	114.8 (1)	C(2)—C(1)—C(10)	124.4 (1)
C(1)—C(10)—C(9)	117.1 (1)	C(9)—C(10)—C(11)	121.6 (1)
C(1)—C(10)—C(11)	121.1 (1)	C(10)—C(11)—C(12)	117.3 (1)
C(11)—C(12)—C(21)	121.3 (1)	C(11)—C(12)—C(13)	121.5 (1)
O(3)—C(21)—C(20)	116.0 (1)	O(3)—C(21)—C(12)	119.9 (1)
O(4)—C(22)—C(27)	118.1 (1)	O(4)—C(22)—C(23)	119.4 (1)
O(2)—P—O(3)—C(21)	3.6 (1)		
O(3)—P—O(2)—C(1)	100.1 (1)		
P—O(2)—C(1)—C(10)	-66.5 (2)		
P—O(3)—C(21)—C(12)	-63.2 (2)		
O(2)—C(1)—C(10)—C(11)	6.6 (2)		
C(1)—C(10)—C(11)—C(12)	-52.9 (2)		
C(10)—C(11)—C(12)—C(21)	95.8 (2)		
C(11)—C(12)—C(21)—O(3)	-3.3 (2)		

The preliminary cell parameters and the space group were obtained from oscillation and Weissenberg photographs. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and refined by a full-matrix least-squares method using *SHELX76* (Sheldrick, 1976). The H-atom positions were located from a difference Fourier map. The positional parameters of the non-H atoms were refined anisotrop-

ically. The U_{iso} values of the H atoms were obtained from those of their attached C atoms and the positional and U_{iso} parameters were not refined. Other geometrical calculations were carried out using *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including least-squares-planes data, torsion angles and intermolecular contact distances less than 3.75 Å, have been deposited with the IUCr (Reference: HA1100). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 1054–1056

2,4-Pentanedione Bis(2,4-dinitrophenyl-hydrazone)

S. MANI NAIDU,^a M. KRISHNAIAH,^{a*} K. SIVAKUMAR,^b AND R. P. SHARMA^c

^aDepartment of Physics, College of Engineering, S. V. University, Tirupati 517 502, India, ^bDepartment of Physics, Anna University, Madras 600 025, India, and ^cDepartment of Chemistry, Punjab University, Chandigarh, India

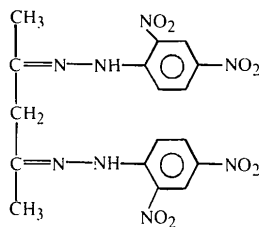
(Received 20 January 1994; accepted 9 October 1995)

Abstract

The title molecule, C₁₇H₁₆N₈O₈, has approximate non-crystallographic twofold symmetry. The bond lengths in the chain of atoms connecting the two dinitrophenyl groups are consistent with some delocalization of the double bonds in the chain. The molecule adopts an *E,E* conformation, which favours intramolecular N—H···O hydrogen bonding. The dihedral angle between the planes of the phenyl rings is 69.6 (1)°.

Comment

The bond lengths and angles in the two chemically equivalent halves of the title molecule, (I), are similar,



(I)

with the largest differences in torsion angles being *ca* 8°, *e.g.* C(6)—N(3)—N(4)—C(7) 168.5 (4) and C(12)—N(6)—N(5)—C(10) 175.8 (4)°. The endocyclic bond angles in the phenyl rings are normal, except for C(1)—C(6)—C(5) of 115.4 (4) and C(13)—C(12)—C(17) of 116.8 (4)°, adjacent to the *o*-nitro groups at atoms C(1) and C(17). Such reductions in endocyclic bond angles are a common feature of the *ortho*-substituted phenylhydrazones (Willey & Drew, 1985; Drew, Vickery & Willey, 1984). The C_{sp^2} —N bond distances are different: C(1)—N(1), C(3)—N(2) and C(15)—N(7), which are all in the range 1.45–1.46 Å, show single-bond character, while C(6)—N(3) of 1.343 (6) and C(12)—N(6) of 1.357 (6) Å must have partial double-bond character. Likewise, the bonds N(3)—N(4) of 1.384 (6) and N(5)—N(6) of 1.370 (6) Å are shorter than single bonds (Pauling, 1960), but agree well with those in related structures (Willey & Drew, 1983, 1985; Edmonds & Hamilton, 1972).

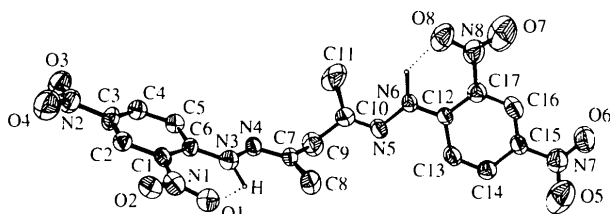


Fig. 1. A view of the molecule of the title compound drawn using ORTEPII (Johnson, 1976) and showing 50% probability ellipsoids. H atoms have been omitted for clarity.

Experimental

Crystals of the title compound were prepared by refluxing acetyl acetone with 2,4-dinitrophenylhydrazine, washing the product with ether and recrystallizing it from methanol by slow evaporation. The density D_m was obtained by flotation in KI solution.

Crystal data

$C_{17}H_{16}N_8O_8$
 $M_r = 460.362$
 Triclinic
 $P\bar{1}$
 $a = 4.987 (5) \text{ \AA}$
 $b = 9.998 (3) \text{ \AA}$
 $c = 20.567 (1) \text{ \AA}$

Mo $K\alpha$ radiation
 $\lambda = 0.71069 \text{ \AA}$
 Cell parameters from 20 reflections
 $\theta = 10\text{--}15^\circ$
 $\mu = 0.12 \text{ mm}^{-1}$
 $T = 293 \text{ K}$

$\alpha = 101.81 (2)^\circ$
 $\beta = 94.21 (2)^\circ$
 $\gamma = 94.95 (2)^\circ$
 $V = 995.6 (9) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.5436 \text{ Mg m}^{-3}$
 $D_m = 1.54 \text{ Mg m}^{-3}$

Needle
 $0.4 \times 0.4 \times 0.3 \text{ mm}$
 Golden yellow

Data collection

Syntex $P2_1$ diffractometer
 θ - 2θ scans
 Absorption correction: none
 2677 measured reflections
 2596 independent reflections
 1971 observed reflections
 $[F > 4\sigma(F)]$
 $R_{int} = 0.0145$

$\theta_{max} = 25^\circ$
 $h = -5 \rightarrow 5$
 $k = -10 \rightarrow 10$
 $l = 0 \rightarrow 22$
 3 standard reflections monitored every 100 reflections
 intensity decay: <3%

Refinement

Refinement on F
 $R = 0.060$
 $wR = 0.066$
 $S = 1.36$
 1971 reflections
 297 parameters
 H-atom parameters not refined

$w = 1.427/[\sigma^2(F_o) + 0.000825F_o^2]$
 $(\Delta/\sigma)_{max} = 0.005$
 $\Delta\rho_{max} = 0.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.30 \text{ e \AA}^{-3}$
 Atomic scattering factors from SHELX76 (Sheldrick, 1976)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{eq} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
O(1)	1.1648 (9)	-0.5023 (4)	0.7722 (2)	7.0 (1)
O(2)	1.4768 (11)	-0.5819 (5)	0.8242 (2)	10.0 (2)
O(3)	1.8637 (8)	-0.3699 (4)	1.0406 (2)	5.8 (1)
O(4)	1.7557 (10)	-0.1703 (5)	1.0863 (2)	8.5 (2)
O(5)	-0.4411 (8)	0.1461 (4)	0.4390 (2)	5.4 (1)
O(6)	-0.1558 (9)	0.3050 (5)	0.4208 (2)	7.4 (2)
O(7)	0.6012 (6)	0.4879 (4)	0.5751 (2)	5.6 (1)
O(8)	0.6894 (7)	0.4146 (3)	0.6634 (2)	5.2 (1)
N(1)	1.3213 (10)	-0.4968 (4)	0.8209 (2)	5.1 (2)
N(2)	1.7268 (9)	-0.2743 (5)	1.0415 (2)	4.8 (2)
N(3)	0.9680 (8)	-0.2767 (4)	0.8269 (2)	3.6 (1)
N(4)	0.7964 (7)	-0.1740 (4)	0.8339 (2)	3.5 (1)
N(5)	0.3691 (7)	0.0788 (3)	0.7098 (2)	3.1 (1)
N(6)	0.4243 (8)	0.1892 (4)	0.6812 (2)	3.2 (1)
N(7)	-0.2327 (9)	0.2244 (5)	0.4538 (2)	4.4 (1)
N(8)	0.5473 (8)	0.4099 (4)	0.6112 (2)	3.6 (1)
C(1)	1.3221 (9)	-0.3849 (4)	0.8788 (2)	3.5 (1)
C(2)	1.5093 (10)	-0.3822 (5)	0.9320 (2)	3.6 (2)
C(3)	1.5216 (9)	-0.2780 (5)	0.9868 (2)	3.6 (1)
C(4)	1.3523 (10)	-0.1749 (5)	0.9902 (3)	4.0 (2)
C(5)	1.1707 (10)	-0.1757 (5)	0.9376 (2)	3.7 (1)
C(6)	1.1448 (8)	-0.2807 (4)	0.8788 (2)	3.0 (1)
C(7)	0.6648 (9)	-0.1597 (4)	0.7802 (2)	3.4 (1)
C(8)	0.6856 (13)	-0.2381 (5)	0.7115 (3)	6.0 (2)
C(9)	0.4682 (12)	-0.0524 (6)	0.7895 (2)	4.4 (2)
C(10)	0.5391 (8)	0.0658 (4)	0.7580 (2)	3.1 (1)
C(11)	0.7931 (9)	0.1562 (5)	0.7843 (2)	3.9 (1)
C(12)	0.2613 (8)	0.2025 (4)	0.6276 (2)	2.7 (1)
C(13)	0.0291 (9)	0.1101 (4)	0.6045 (2)	3.1 (1)
C(14)	-0.1304 (10)	0.1195 (5)	0.5495 (2)	3.4 (1)
C(15)	-0.0655 (9)	0.2204 (4)	0.5146 (2)	3.3 (1)
C(16)	0.1552 (9)	0.3138 (5)	0.5351 (2)	3.3 (1)
C(17)	0.3162 (9)	0.3076 (4)	0.5918 (2)	3.0 (1)

Table 2. Selected geometric parameters (Å, °)

O(1)—N(1)	1.213 (6)	O(2)—N(1)	1.208 (7)
O(3)—N(2)	1.220 (7)	O(4)—N(2)	1.230 (6)
O(5)—N(7)	1.225 (6)	O(6)—N(7)	1.211 (7)
O(7)—N(8)	1.209 (6)	O(8)—N(8)	1.232 (6)
N(1)—C(1)	1.457 (5)	N(2)—C(3)	1.457 (6)
N(3)—N(4)	1.384 (6)	N(3)—C(6)	1.343 (6)
N(4)—C(7)	1.283 (6)	N(5)—N(6)	1.370 (6)
N(5)—C(10)	1.291 (6)	N(6)—C(12)	1.357 (6)
N(7)—C(15)	1.461 (6)	N(8)—C(17)	1.448 (6)
C(7)—C(8)	1.485 (6)	C(7)—C(9)	1.508 (8)
C(9)—C(10)	1.489 (8)	C(10)—C(11)	1.489 (6)
N(3)···O(1)	2.612 (6)	N(6)···O(8)	2.618 (5)
O(1)—N(1)—O(2)	122.8 (5)	O(2)—N(1)—C(1)	117.7 (4)
O(1)—N(1)—C(1)	119.5 (5)	O(3)—N(2)—O(4)	123.3 (5)
O(4)—N(2)—C(3)	116.6 (5)	O(3)—N(2)—C(3)	120.1 (5)
N(4)—N(3)—C(6)	118.5 (4)	N(3)—N(4)—C(7)	116.2 (4)
N(6)—N(5)—C(10)	116.7 (4)	N(5)—N(6)—C(12)	118.9 (4)
O(5)—N(7)—O(6)	123.9 (5)	O(6)—N(7)—C(15)	117.7 (5)
O(5)—N(7)—C(15)	118.4 (5)	O(7)—N(8)—O(8)	120.9 (5)
O(8)—N(8)—C(17)	119.8 (4)	O(7)—N(8)—C(17)	119.3 (4)
N(1)—C(1)—C(6)	121.1 (4)	N(1)—C(1)—C(2)	116.9 (4)
N(2)—C(3)—C(2)	118.2 (5)	N(6)—C(12)—C(13)	120.7 (4)
N(2)—C(3)—C(4)	120.1 (4)	N(7)—C(15)—C(14)	119.3 (4)
N(3)—C(6)—C(5)	120.8 (4)	N(3)—C(6)—C(1)	123.7 (4)
N(4)—C(7)—C(9)	115.3 (4)	N(4)—C(7)—C(8)	126.8 (5)
C(8)—C(7)—C(9)	118.0 (4)	C(7)—C(9)—C(10)	113.9 (4)
N(5)—C(10)—C(9)	115.3 (4)	C(9)—C(10)—C(11)	118.1 (4)
N(5)—C(10)—C(11)	126.6 (4)	N(6)—C(12)—C(17)	122.5 (4)
N(7)—C(15)—C(16)	119.6 (4)	C(13)—C(12)—C(17)	116.8 (4)
N(8)—C(17)—C(12)	122.0 (4)		

Acta Cryst. (1996). C52, 1056–1058

1-(2,4-Dinitrophenyl)-3,5-dimethylpyrazole

S. MANI NAIDU,^a M. KRISHNAIAH,^{a*} K. SIVAKUMAR,^b
AND R. P. SHARMA^c^aDepartment of Physics, College of Engineering, S. V. University, Tirupati 517 502, India, ^bDepartment of Physics, Anna University, Madras 600 025, India, and ^cDepartment of Chemistry, Punjab University, Chandigarh, India

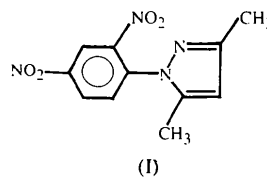
(Received 20 January 1994; accepted 9 October 1995)

Abstract

The asymmetric unit of the title compound consists of two independent molecules of C₁₁H₁₀N₄O₄. The pyrazole and dinitrophenyl rings are individually planar and are twisted about the N—C_{sp²} bond joining them by 64°. The two nitro groups attached to the phenyl ring show different degrees of rotation away from the plane of the phenyl ring, with those groups at the *para* positions rotated by 8.2 (3) and 14.8 (2)°, and those at the *ortho* positions rotated by 33.5 (1) and 33.2 (1)°. The crystal structure is stabilized by van der Waals interactions.

Comment

Pyrazole and several *N*-substituted pyrazoles are used as inhibitors and deactivators of liver alcohol dehydrogenase and many pyrazole derivatives constitute a family of interesting pharmaceutical agents such as analgesics, antipyretics and anti-inflammatories (Potts, 1986). In view of the wide biological applications of the pyrazole compounds, the crystal structure of 3,5-dimethyl-1-(2,4-dinitrophenyl)pyrazole, (I), has been determined.



The bond lengths and angles of the two molecules in the asymmetric unit are in agreement. The C_{sp²}—N bonds associated with the nitro groups are clearly single bonds, while the C(6)—N(3) [1.408 (4) Å] and C(6')—N(3') [1.416 (3) Å] bonds show partial double-bond character. The nitro O(4) and O(4') atoms have very high atomic displacement parameters (Table 1, Fig. 1). This accounts for the apparent shortening of the N(2)—O(4) [1.182 (6) Å] and N(2')—O(4') [1.185 (5) Å] bonds.

The structure of the title compound was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and refined by full-matrix least squares using *SHELX76* (Sheldrick, 1976). The positional parameters of the non-H atoms were refined anisotropically. The positional parameters of the H atoms were located from a difference Fourier map and their isotropic displacement parameters were set equivalent to those of the corresponding C atoms and included in the final cycles of refinement but not refined. All other geometrical calculations were carried out using *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including least-squares-planes data, torsion angles and intermolecular contact distances less than 3.5 Å, have been deposited with the IUCr (Reference: HA1101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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