Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(2,4-Dinitrophenyl)-2-(3-phenylallylidene)hydrazine pyridine hemisolvate

Zhi-Gang Yin,* Heng-Yu Qian, Yu-Zhen Chen and Yu-Li Feng

Key Laboratory of Surface and Interface Science of Henan, School of Materials and Chemical Engineering, Zhengzhou University of Light Industry, Zhengzhou 450002, People's Republic of China
Correspondence e-mail: yinzhigang3141@yahoo.com.cn

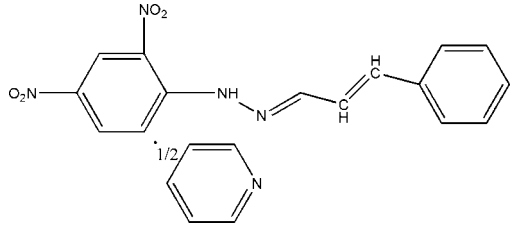
Received 20 October 2007; accepted 2 November 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.064; wR factor = 0.143; data-to-parameter ratio = 14.1.

The principal molecule of the title compound, $C_{15}H_{12}N_4O_4\cdot 0.5C_5H_5N$, is nearly planar, the largest deviation from the mean plane being 0.094 (2) Å. The pyridine solvent molecule lies on a twofold axis and is connected to the 1-(2,4-dinitrophenyl)-2-(3-phenylallylidene)hydrazine through weak C—H···O hydrogen bonding. An intramolecular N—H···O hydrogen bond helps to stabilize the molecular structure.

Related literature

For related literature, see: Okabe *et al.* (1993).



Experimental

Crystal data

$C_{15}H_{12}N_4O_4\cdot 0.5C_5H_5N$
M_r = 351.89
Monoclinic, $C2/c$
a = 17.970 (4) Å
b = 7.1370 (14) Å
c = 26.305 (5) Å
β = 104.73 (3)°
V = 3262.8 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.27 × 0.23 × 0.23 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.966, T_{max} = 0.972
7279 measured reflections
3328 independent reflections
2372 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.143
S = 1.13
3328 reflections
236 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C18—H18···O2	0.93	2.68	3.252 (3)	120
N3—H3···O2	0.86	1.98	2.609 (3)	129

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the Startup Fund for PhDs (grant No. 2005001) and the Startup Fund for Masters (grant No. 000455) of Natural Scientific Research of Zhengzhou University of Light Industry.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2254).

References

Bruker (1998). *SMART* (Version 5.628), *SAINT* (Version 6.02) and *SADABS* (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Okabe, N., Nakamura, T. & Fukuda, H. (1993). *Acta Cryst.* C**49**, 1678–1680.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

Acta Cryst. (2007). E**63**, o4625 [**doi:10.1107/S1600536807055444**]

1-(2,4-Dinitrophenyl)-2-(3-phenylallylidene)hydrazine pyridine hemisolvate

Z.-G. Yin, H.-Y. Qian, Y.-Z. Chen and Y.-L. Feng

Comment

2,4-Dinitrophenylhydrazine has applications in organic synthesis and some of its derivatives have been shown to be potentially DNA-damaging and mutagenic agents (Okabe *et al.*,1993). Some phenylhydrazone derivatives have been synthesized in our laboratory. As part of our work, we report the synthesis and crystal structure of the title compound(I).

The title molecule is nearly planar with the largest deviation from the mean plane containing the two benzene rings and the C3—N2 chain,being 0.094 (2) Å at C3 (Fig. 1). The two nitro groups, O1/N1/O2 and O3/N2/O4 are slightly twisted with respect to this plane by 11.2 (2)° and 14.4 (2)° respectively. The pyridine ring is roughly perpendicular to this plane making a dihedral angle of 73.85 (9)° with it.

The Pyridine is linked to the title compound through weak intermolecular C—H···O hydrogen bonds (Table 1). An intramolecular N—H···O hydrogen bond is also observed and helps to stablize the conformation of the molecule (Table 1, Fig.1).

Experimental

2,4-Dinitrophenylhydrazine (1 mmol, 0.198 g) was dissolved in anhydrous methanol, H_2SO_4 (98% 0.5 ml) was added to this, the mixture was stirred for several minitutes at 351 K, cinnamaldehyde (1 mmol 0.132 g) in methanol (8 ml) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized in pyridine, brown single crystals of (I) was obtained after two weeks.

Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) and N—H = 0.86 Å with U_{iso}(H) = 1.2U_{eq}(C or N).

Figures

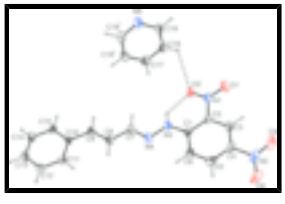


Fig. 1. Molecular view of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

1-(2,4-Dinitrophenyl)-2-(3-phenylallylidene)hydrazine pyridine solvate

Crystal data

$C_{15}H_{12}N_4O_4 \cdot 0.5C_5H_5N$	$F_{000} = 1464$
$M_r = 351.78$	$D_x = 1.432$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 1160 reflections
$a = 17.970$ (4) Å	$\theta = 2.1$–25.5º
$b = 7.1370$ (14) Å	$\mu = 0.11$ mm^{-1}
$c = 26.305$ (5) Å	$T = 298$ (2) K
$\beta = 104.73$ (3)º	Block, red
$V = 3262.8$ (11) Å^3	0.27 × 0.23 × 0.23 mm
$Z = 8$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3328 independent reflections
Radiation source: sealed tube	2372 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 298$(2) K	$\theta_{max} = 26.5$º
φ and ω scans	$\theta_{min} = 1.6$º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$h = -22 \rightarrow 22$
$T_{min} = 0.966$, $T_{max} = 0.972$	$k = -8 \rightarrow 8$
7279 measured reflections	$l = -30 \rightarrow 32$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.064$	H-atom parameters constrained
$wR(F^2) = 0.143$	$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 1.2883P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.13$	$(\Delta/\sigma)_{max} < 0.001$
3328 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
236 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.74691 (9)	0.6036 (3)	0.22779 (7)	0.0714 (6)
O2	0.63086 (9)	0.6562 (3)	0.22975 (6)	0.0565 (5)
O3	0.84516 (12)	0.7909 (4)	0.08715 (10)	0.0985 (8)
O4	0.77336 (12)	0.9361 (3)	0.02160 (8)	0.0815 (7)
N1	0.68219 (10)	0.6592 (3)	0.20666 (7)	0.0457 (5)
N2	0.78311 (14)	0.8491 (3)	0.06274 (10)	0.0637 (6)
N3	0.52874 (9)	0.7524 (3)	0.14416 (7)	0.0447 (5)
H3	0.5347	0.7200	0.1764	0.054*
N4	0.45643 (10)	0.7859 (3)	0.11322 (7)	0.0478 (5)
N5	0.5000	−0.0289 (4)	0.2500	0.0600 (8)
C1	0.59055 (12)	0.7698 (3)	0.12459 (9)	0.0391 (5)
C2	0.66606 (12)	0.7276 (3)	0.15397 (8)	0.0386 (5)
C3	0.72816 (12)	0.7493 (3)	0.13259 (9)	0.0441 (6)
H3A	0.7775	0.7186	0.1521	0.053*
C4	0.71660 (13)	0.8154 (3)	0.08307 (9)	0.0455 (6)
C5	0.64336 (14)	0.8555 (3)	0.05233 (9)	0.0482 (6)
H5	0.6365	0.8991	0.0181	0.058*
C6	0.58213 (13)	0.8303 (3)	0.07269 (9)	0.0441 (6)
H6	0.5330	0.8539	0.0517	0.053*
C7	0.40318 (12)	0.7550 (3)	0.13646 (9)	0.0455 (6)
H7	0.4160	0.7122	0.1710	0.055*
C8	0.32407 (12)	0.7854 (3)	0.11005 (9)	0.0453 (6)
H8	0.3116	0.8288	0.0756	0.054*
C9	0.26760 (12)	0.7532 (3)	0.13355 (9)	0.0408 (5)
H9	0.2826	0.7125	0.1682	0.049*
C10	0.18547 (11)	0.7746 (3)	0.11100 (8)	0.0360 (5)
C11	0.15505 (12)	0.8241 (3)	0.05934 (9)	0.0443 (6)
H11	0.1878	0.8455	0.0377	0.053*
C12	0.07709 (13)	0.8425 (3)	0.03930 (10)	0.0509 (6)
H12	0.0574	0.8765	0.0043	0.061*
C13	0.02810 (13)	0.8108 (4)	0.07090 (10)	0.0538 (7)
H13	−0.0248	0.8228	0.0573	0.065*
C14	0.05713 (13)	0.7617 (4)	0.12213 (10)	0.0511 (6)

H14	0.0241	0.7405	0.1436	0.061*
C15	0.13524 (12)	0.7434 (3)	0.14219 (9)	0.0434 (6)
H15	0.1546	0.7095	0.1772	0.052*
C17	0.5000	0.3582 (5)	0.2500	0.0517 (9)
H17	0.5000	0.4885	0.2500	0.062*
C18	0.56730 (13)	0.2605 (4)	0.26336 (9)	0.0539 (7)
H18	0.6142	0.3230	0.2727	0.065*
C19	0.56465 (13)	0.0702 (4)	0.26284 (10)	0.0557 (7)
H19	0.6110	0.0053	0.2720	0.067*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0396 (10)	0.1113 (17)	0.0603 (11)	0.0149 (10)	0.0073 (9)	0.0132 (11)
O2	0.0419 (9)	0.0822 (14)	0.0499 (10)	0.0000 (9)	0.0198 (8)	0.0096 (9)
O3	0.0558 (13)	0.130 (2)	0.127 (2)	0.0015 (13)	0.0540 (14)	0.0153 (16)
O4	0.1057 (16)	0.0824 (15)	0.0755 (14)	−0.0282 (12)	0.0583 (13)	−0.0061 (12)
N1	0.0336 (10)	0.0547 (13)	0.0491 (12)	−0.0027 (9)	0.0109 (9)	−0.0006 (10)
N2	0.0654 (16)	0.0642 (16)	0.0760 (17)	−0.0164 (13)	0.0448 (14)	−0.0116 (13)
N3	0.0313 (10)	0.0592 (13)	0.0442 (11)	−0.0005 (9)	0.0108 (8)	0.0028 (9)
N4	0.0327 (10)	0.0595 (14)	0.0498 (12)	−0.0014 (9)	0.0082 (9)	−0.0032 (10)
N5	0.0417 (17)	0.060 (2)	0.078 (2)	0.000	0.0158 (15)	0.000
C1	0.0364 (12)	0.0363 (13)	0.0463 (13)	−0.0034 (10)	0.0137 (10)	−0.0058 (10)
C2	0.0359 (12)	0.0400 (13)	0.0416 (12)	−0.0038 (10)	0.0129 (10)	−0.0017 (10)
C3	0.0346 (12)	0.0441 (14)	0.0559 (15)	−0.0021 (10)	0.0156 (11)	−0.0084 (11)
C4	0.0481 (14)	0.0398 (14)	0.0565 (15)	−0.0089 (11)	0.0275 (12)	−0.0078 (11)
C5	0.0606 (16)	0.0451 (15)	0.0426 (13)	−0.0076 (12)	0.0198 (12)	−0.0032 (11)
C6	0.0422 (13)	0.0454 (14)	0.0442 (13)	−0.0032 (11)	0.0103 (10)	−0.0028 (11)
C7	0.0357 (12)	0.0506 (15)	0.0498 (14)	−0.0024 (11)	0.0101 (11)	−0.0034 (12)
C8	0.0355 (12)	0.0523 (16)	0.0464 (13)	−0.0040 (11)	0.0073 (10)	−0.0019 (11)
C9	0.0379 (12)	0.0444 (14)	0.0386 (12)	−0.0004 (10)	0.0067 (10)	−0.0016 (10)
C10	0.0334 (11)	0.0334 (12)	0.0405 (12)	−0.0022 (9)	0.0085 (9)	−0.0021 (10)
C11	0.0389 (12)	0.0445 (14)	0.0504 (14)	−0.0009 (10)	0.0133 (10)	0.0059 (11)
C12	0.0431 (13)	0.0526 (16)	0.0515 (14)	0.0029 (12)	0.0022 (11)	0.0111 (12)
C13	0.0320 (12)	0.0588 (17)	0.0671 (17)	0.0021 (11)	0.0063 (12)	−0.0009 (13)
C14	0.0408 (13)	0.0570 (17)	0.0606 (16)	−0.0016 (12)	0.0222 (12)	−0.0064 (13)
C15	0.0389 (12)	0.0505 (15)	0.0422 (13)	−0.0017 (11)	0.0127 (10)	−0.0022 (11)
C17	0.053 (2)	0.057 (2)	0.046 (2)	0.000	0.0150 (16)	0.000
C18	0.0394 (13)	0.0649 (19)	0.0561 (16)	−0.0073 (13)	0.0099 (12)	0.0081 (13)
C19	0.0351 (13)	0.069 (2)	0.0623 (17)	0.0045 (13)	0.0117 (12)	0.0127 (14)

Geometric parameters (Å, °)

O1—N1	1.221 (2)	C7—H7	0.9300
O2—N1	1.227 (2)	C8—C9	1.336 (3)
O3—N2	1.210 (3)	C8—H8	0.9300
O4—N2	1.221 (3)	C9—C10	1.452 (3)
N1—C2	1.428 (3)	C9—H9	0.9300
N2—C4	1.448 (3)	C10—C11	1.376 (3)

N3—C1	1.343 (3)	C10—C15	1.383 (3)
N3—N4	1.368 (2)	C11—C12	1.372 (3)
N3—H3	0.8600	C11—H11	0.9300
N4—C7	1.279 (3)	C12—C13	1.375 (3)
N5—C19	1.328 (3)	C12—H12	0.9300
N5—C19^{i}	1.328 (3)	C13—C14	1.361 (3)
C1—C6	1.403 (3)	C13—H13	0.9300
C1—C2	1.413 (3)	C14—C15	1.374 (3)
C2—C3	1.380 (3)	C14—H14	0.9300
C3—C4	1.351 (3)	C15—H15	0.9300
C3—H3A	0.9300	C17—C18^{i}	1.362 (3)
C4—C5	1.388 (3)	C17—C18	1.362 (3)
C5—C6	1.353 (3)	C17—H17	0.9300
C5—H5	0.9300	C18—C19	1.359 (4)
C6—H6	0.9300	C18—H18	0.9300
C7—C8	1.431 (3)	C19—H19	0.9300
O1—N1—O2	121.2 (2)	C9—C8—H8	119.1
O1—N1—C2	119.20 (18)	C7—C8—H8	119.1
O2—N1—C2	119.59 (18)	C8—C9—C10	127.3 (2)
O3—N2—O4	123.4 (2)	C8—C9—H9	116.4
O3—N2—C4	118.9 (3)	C10—C9—H9	116.4
O4—N2—C4	117.8 (3)	C11—C10—C15	118.2 (2)
C1—N3—N4	120.60 (19)	C11—C10—C9	122.5 (2)
C1—N3—H3	119.7	C15—C10—C9	119.3 (2)
N4—N3—H3	119.7	C12—C11—C10	121.0 (2)
C7—N4—N3	113.50 (19)	C12—C11—H11	119.5
C19—N5—C19^{i}	115.7 (3)	C10—C11—H11	119.5
N3—C1—C6	120.4 (2)	C11—C12—C13	120.0 (2)
N3—C1—C2	122.9 (2)	C11—C12—H12	120.0
C6—C1—C2	116.67 (19)	C13—C12—H12	120.0
C3—C2—C1	121.1 (2)	C14—C13—C12	119.9 (2)
C3—C2—N1	116.63 (19)	C14—C13—H13	120.1
C1—C2—N1	122.24 (18)	C12—C13—H13	120.1
C4—C3—C2	119.3 (2)	C13—C14—C15	120.1 (2)
C4—C3—H3A	120.3	C13—C14—H14	120.0
C2—C3—H3A	120.3	C15—C14—H14	120.0
C3—C4—C5	121.6 (2)	C14—C15—C10	120.9 (2)
C3—C4—N2	118.4 (2)	C14—C15—H15	119.5
C5—C4—N2	120.0 (2)	C10—C15—H15	119.5
C6—C5—C4	119.2 (2)	C18^{i}—C17—C18	118.4 (4)
C6—C5—H5	120.4	C18^{i}—C17—H17	120.8
C4—C5—H5	120.4	C18—C17—H17	120.8
C5—C6—C1	121.9 (2)	C19—C18—C17	118.8 (3)
C5—C6—H6	119.0	C19—C18—H18	120.6
C1—C6—H6	119.0	C17—C18—H18	120.6
N4—C7—C8	120.9 (2)	N5—C19—C18	124.1 (3)
N4—C7—H7	119.6	N5—C19—H19	117.9
C8—C7—H7	119.6	C18—C19—H19	117.9

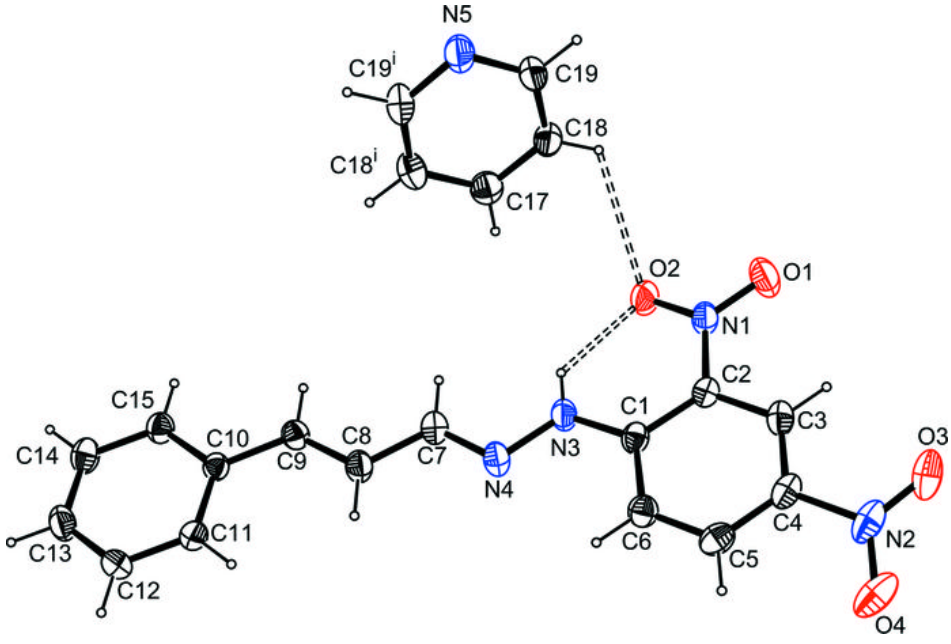
C9—C8—C7	121.8 (2)

Symmetry codes: (i) −*x*+1, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H18···O2	0.93	2.68	3.252 (3)	120
N3—H3···O2	0.86	1.98	2.609 (3)	129

Fig. 1


N5
C19i
C19
C18
C18i
C17
O2
O1
N1
C2
C15
C1
C3
O3
C14
C10
C9
C8
C7
N3
N4
C4
N2
C13
C11
C6
C5
C12
O4