1-(4-Nitrophenyl)-3,5-diphenyl-4,5-dihydro-1H-pyrazole acetone hemisolvate

Wen-Zhong Ge

College of Life Science and Technology, Heilongjiang August First Land Reclamation University, Daqing 163319, People's Republic of China

Correspondence e-mail: wenzhongge@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
Disorder in solvent or counterion
R factor = 0.053
wR factor = 0.180
Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The asymmetric unit of the title compound, $C_{21}H_{17}N_3O_2\cdot0.5C_3H_6O$, is composed of two independent but similar molecules. The pyrazole ring in each molecule is almost planar and the N—N bond lengths in the pyrazole rings are 1.384 (2) and 1.385 (2) Å.

Comment

Pyrazole derivatives are widely used as biologically active compounds (Ono *et al.*, 1997), and the structures of these compounds have been studied extensively (Akama & Tong,1996; Fraghaly *et al.*, 1989). One of our aims is to study the chemistry of pyrazole derivatives.

(I)
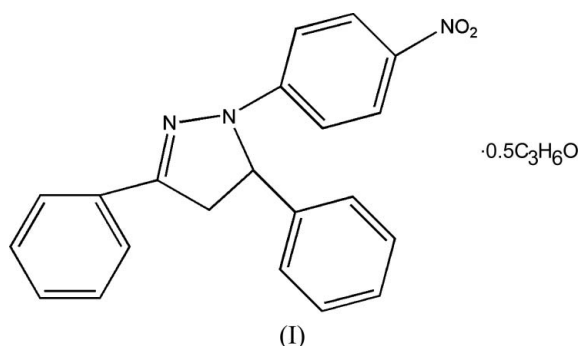
The structure of the asymmetric unit is shown in Fig. 1 and selected bond lengths and angles are given in Table 1. The pyrazole rings in the two molecules are almost planar; the corresponding bond lengths and angles in the two pyrazole rings are nearly identical, while the dihedral angles between the pyrazole ring and the nitrophenyl ring are different in the two unique molecules. The dihedral angle between the pyrazole ring N2/N3/C7/C8/C9 and nitro group N1/O1/O2 is 4.44 (12)°, and the corresponding angle between the pyrazole ring N5/N6/C28/C35/C36 and the nitro group N4/O3/O4 is 11.85 (12)°. The N2—N3 and N5—N6 bond lengths in the pyrazole rings approximate the length of a single bond (1.41 Å; Burke-Laing & Laing, 1976).

Experimental

4-Nitrophenylhydrazine (1 mmol, 0.153 g) was dissolved in anhydrous ethanol, H_2SO_4 (98% 0.5 ml) was added, the mixture was stirred for several minutes at 351 K, benzylideneacetophenone (1 mmol 0.208 g) in ethanol (6 ml) was added dropwise and the mixture was stirred at refluxing temperature for 1 h. The product was separated and recrystallized from acetone; brown single crystals of (I) were obtained after 3 d.

Crystal data

$C_{21}H_{17}N_3O_2 \cdot 0.5C_3H_6O$
$M_r = 372.42$
Triclinic, $P\bar{1}$
$a = 11.605$ (2) Å
$b = 12.150$ (2) Å
$c = 14.846$ (3) Å
$\alpha = 75.68$ (3)°
$\beta = 68.86$ (3)°
$\gamma = 81.52$ (3)°

$V = 1887.8$ (7) Å^3
$Z = 4$
$D_x = 1.310$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 298$ (2) K
Block, brown
$0.38 \times 0.26 \times 0.13$ mm

Data collection

Bruker SMART CCD area-detector
 diffractometer
ω scans
Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 2002)
 $T_{min} = 0.968$, $T_{max} = 0.989$

15422 measured reflections
7750 independent reflections
4059 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 26.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.180$
$S = 0.92$
7750 reflections
502 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.088P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N2—N3	1.384 (2)	C7—C8	1.533 (3)
N5—N6	1.385 (2)	C8—C9	1.499 (3)
N5—C36	1.481 (3)	C28—C35	1.500 (3)
N6—C28	1.291 (3)	C35—C36	1.528 (3)
N3—N2—C7	113.08 (18)	N6—N5—C36	112.80 (18)
C9—N3—N2	108.51 (18)	C28—N6—N5	108.71 (18)

H atoms were placed in calculated positions and refined as riding with the following constraints: methyl C—H = 0.96 Å, $U_{iso}(H) = 1.5U_{eq}(C)$; methylene C—H = 0.97 Å, $U_{iso}(H) = 1.2U_{eq}(C)$; methine C—H = 0.98 Å, $U_{iso}(H) = 1.2U_{eq}(C)$; aromatic C—H = 0.93 Å, $U_{iso}(H) = 1.2U_{eq}(C)$.
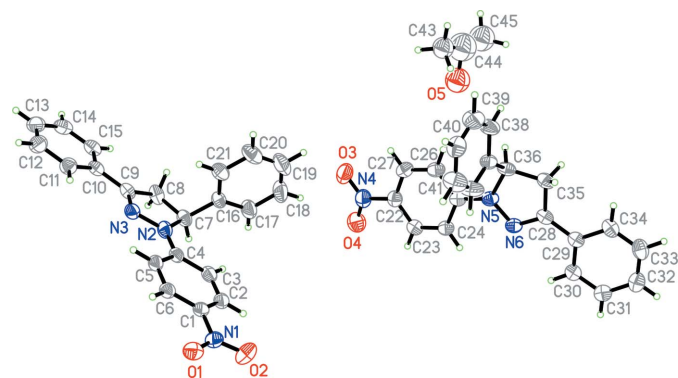

Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

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: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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