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Acetone 2-nitrophenylhydrazone

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.153
Data-to-parameter ratio = 16.3

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

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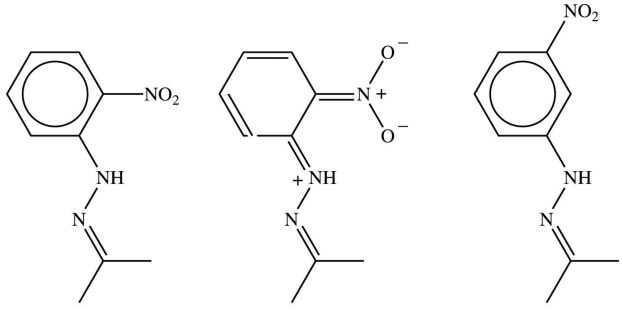
There are no direction-specific interactions between the almost-planar molecules of the title compound, $C_9H_{11}N_3O_2$.

Comment

We report here the structure of acetone 2-nitrophenylhydrazone, (I) (Fig. 1), whose behaviour differs significantly from that of the isomeric compound acetone 3-nitrophenylhydrazone, (II) (Wardell *et al.*, 2006).



(I) (Ia) (II)

The non-H atoms in the molecule of (I) are virtually coplanar, as shown by the key torsion angles (Table 1). There is a short intermolecular N—H···O hydrogen bond (Table 2), which may assist in controlling the planar conformation. The bond distances (Table 1) show evidence for a significant contribution from the quinonoid form (I*a*). In particular, the bonds C3—C4 and C5—C6 are shorter than the remaining bonds in the ring, while C2—N21 is very short for its type and the N—O bonds are long (Allen *et al.*, 1987). In contrast, the bond distances in (II) show no unusual values (Wardell *et al.*, 2006).

Whereas the molecules of (II) are linked into complex sheets by a combination of N—H···O, C—H···O and C—H···N hydrogen bonds, there are no direction-specific intermolecular interactions in the structure of (I). In particular, hydrogen bonds of all types and aromatic π–π stacking interactions are absent.

Experimental

2-Nitrophenylhydrazine (3 mmol) was dissolved in acetone (30 ml) and the solution was heated under reflux for 1 h. The solution was then cooled and excess solvent was removed under reduced pressure. The resulting solid product, (I), was crystallized from ethanol (m.p. 339–341 K).

Crystal data

$C_9H_{11}N_3O_2$
M_r = 193.21
Monoclinic, $P2_1/c$
a = 3.8451 (2) Å
b = 11.4926 (8) Å
c = 21.3214 (13) Å
β = 92.208 (4)°
V = 941.50 (10) Å^3
Z = 4
D_x = 1.363 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 120 (2) K
Needle, orange
0.40 × 0.04 × 0.04 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.971, T_{max} = 0.996
10829 measured reflections
2104 independent reflections
1738 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 27.8°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.153
S = 1.06
2104 reflections
129 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0854P)^2 + 0.2667P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.417 (2)	C1—N1	1.3637 (17)
C2—C3	1.4027 (19)	N1—N2	1.3799 (17)
C3—C4	1.370 (2)	N2—C7	1.2877 (18)
C4—C5	1.398 (2)	C2—N21	1.4387 (18)
C5—C6	1.3772 (19)	N21—O21	1.2513 (16)
C6—C1	1.4100 (19)	N21—O22	1.2267 (17)
C1—C2—N21—O21	−0.3 (2)	C1—N1—N2—C7	175.80 (12)
C1—C2—N21—O22	178.88 (13)	N1—N2—C7—C8	179.80 (12)
C2—C1—N1—N2	−175.52 (12)	N1—N2—C7—C9	0.2 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O21	0.88	1.97	2.6010 (17)	128

All H atoms were located in a difference map and then treated as riding, with C—H distances of 0.95 or 0.98 Å and N—H distances of 0.88 Å, and with U_{iso}(H) = kU_{eq}(C,N) where k = 1.5 for methyl groups and 1.2 for all other H.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *FLIPPER* (Oszlányi & Sütő, 2004, 2005; Spek, 2003); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

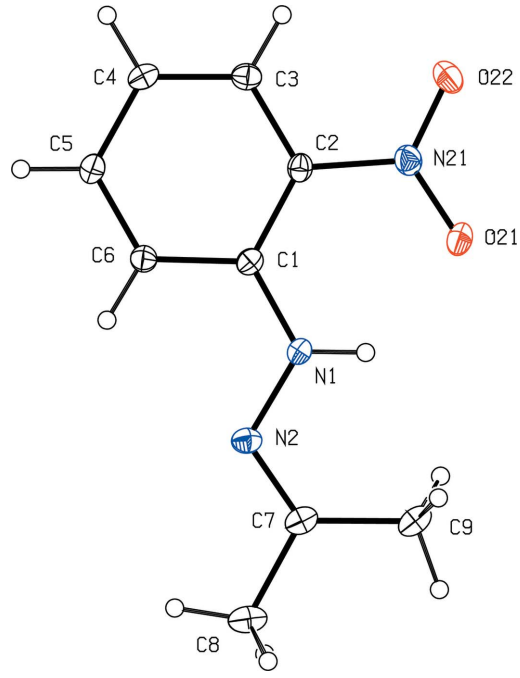


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

The X-ray data were collected at the EPSRC X-Ray Crystallographic Service, University of Southampton, UK; the authors thank the staff of the Service for all their help and advice. JLW thanks CNPq and FAPERJ for financial support.

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$C_9H_{11}N_3O_2$
$M_r = 193.21$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 3.8451$ (2) Å
$b = 11.4926$ (8) Å
$c = 21.3214$ (13) Å
$\beta = 92.208$ (4)°
$V = 941.50$ (10) Å^3
$Z = 4$
$F(000) = 408$
$D_x = 1.363$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2040 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Plate, orange
0.40 × 0.04 × 0.04 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.971$, $T_{max} = 0.996$
10829 measured reflections
2104 independent reflections
1738 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 27.8°$, $\theta_{min} = 3.4°$
$h = -3\rightarrow4$
$k = -14\rightarrow14$
$l = -27\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.153$
$S = 1.06$
2104 reflections
129 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0854P)^2 + 0.2667P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3793 (4)	0.68934 (13)	0.08125 (6)	0.0202 (3)
N1	0.5144 (3)	0.63071 (11)	0.13218 (5)	0.0230 (3)
N2	0.5246 (3)	0.51069 (11)	0.13215 (6)	0.0239 (3)

C7	0.6772 (4)	0.46464 (14)	0.18091 (7)	0.0250 (4)
C8	0.6941 (4)	0.33459 (14)	0.18324 (8)	0.0328 (4)
C9	0.8357 (4)	0.53242 (15)	0.23532 (7)	0.0304 (4)
C2	0.3832 (4)	0.81231 (13)	0.07662 (6)	0.0211 (3)
N21	0.5444 (3)	0.88451 (12)	0.12460 (6)	0.0265 (3)
O21	0.6846 (3)	0.83762 (10)	0.17212 (5)	0.0328 (3)
O22	0.5455 (3)	0.99053 (10)	0.11794 (6)	0.0402 (4)
C3	0.2387 (4)	0.86983 (13)	0.02377 (7)	0.0235 (3)
C4	0.0882 (4)	0.80780 (13)	−0.02496 (7)	0.0242 (3)
C5	0.0830 (4)	0.68637 (13)	−0.02143 (7)	0.0236 (3)
C6	0.2246 (4)	0.62841 (13)	0.03000 (6)	0.0221 (3)
H1	0.5953	0.6695	0.1652	0.028*
H8A	0.5712	0.3022	0.1461	0.049*
H8B	0.5843	0.3068	0.2212	0.049*
H8C	0.9380	0.3096	0.1840	0.049*
H9A	0.9952	0.5912	0.2196	0.046*
H9B	0.9643	0.4793	0.2637	0.046*
H9C	0.6509	0.5708	0.2580	0.046*
H3	0.2451	0.9524	0.0218	0.028*
H4	−0.0111	0.8467	−0.0606	0.029*
H5	−0.0203	0.6430	−0.0552	0.028*
H6	0.2179	0.5458	0.0310	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0194 (8)	0.0240 (8)	0.0176 (7)	0.0006 (5)	0.0030 (5)	0.0011 (5)
N1	0.0290 (7)	0.0222 (7)	0.0175 (6)	−0.0002 (5)	−0.0029 (5)	0.0002 (5)
N2	0.0262 (7)	0.0227 (7)	0.0230 (6)	0.0020 (5)	0.0031 (5)	0.0040 (5)
C7	0.0206 (8)	0.0313 (9)	0.0234 (7)	0.0036 (6)	0.0037 (5)	0.0059 (6)
C8	0.0344 (9)	0.0314 (9)	0.0329 (9)	0.0080 (7)	0.0063 (6)	0.0104 (7)
C9	0.0279 (9)	0.0398 (10)	0.0234 (8)	0.0042 (6)	−0.0026 (6)	0.0066 (6)
C2	0.0210 (8)	0.0243 (8)	0.0181 (7)	−0.0019 (5)	0.0022 (5)	−0.0017 (5)
N21	0.0326 (8)	0.0252 (7)	0.0220 (6)	−0.0041 (5)	0.0030 (5)	−0.0017 (5)
O21	0.0411 (7)	0.0343 (7)	0.0222 (6)	−0.0054 (5)	−0.0076 (5)	−0.0015 (4)
O22	0.0629 (9)	0.0230 (6)	0.0342 (7)	−0.0086 (5)	−0.0040 (6)	−0.0025 (5)
C3	0.0238 (8)	0.0230 (8)	0.0240 (7)	0.0006 (5)	0.0042 (5)	0.0027 (5)
C4	0.0240 (8)	0.0278 (8)	0.0207 (7)	0.0028 (6)	0.0007 (5)	0.0058 (6)
C5	0.0234 (8)	0.0277 (8)	0.0196 (7)	−0.0020 (6)	−0.0012 (5)	−0.0011 (6)
C6	0.0235 (8)	0.0217 (7)	0.0213 (7)	−0.0014 (5)	0.0007 (5)	0.0004 (5)

Geometric parameters (Å, º)

C1—C2	1.417 (2)	C7—C9	1.507 (2)
C2—C3	1.4027 (19)	N1—H1	0.88
C3—C4	1.370 (2)	C8—H8A	0.98
C4—C5	1.398 (2)	C8—H8B	0.98
C5—C6	1.3772 (19)	C8—H8C	0.98

C6—C1	1.4100 (19)	C9—H9A	0.98
C1—N1	1.3637 (17)	C9—H9B	0.98
N1—N2	1.3799 (17)	C9—H9C	0.98
N2—C7	1.2877 (18)	C3—H3	0.95
C2—N21	1.4387 (18)	C4—H4	0.95
N21—O21	1.2513 (16)	C5—H5	0.95
N21—O22	1.2267 (17)	C6—H6	0.95
C7—C8	1.497 (2)		

N1—C1—C6	120.54 (13)	H9A—C9—H9C	109.5
N1—C1—C2	122.93 (12)	H9B—C9—H9C	109.5
C6—C1—C2	116.53 (12)	C3—C2—C1	121.41 (13)
C1—N1—N2	120.23 (11)	C3—C2—N21	116.47 (13)
C1—N1—H1	119.9	C1—C2—N21	122.09 (12)
N2—N1—H1	119.9	O22—N21—O21	121.21 (12)
C7—N2—N1	114.99 (12)	O22—N21—C2	119.59 (12)
N2—C7—C8	117.07 (14)	O21—N21—C2	119.19 (13)
N2—C7—C9	124.58 (14)	C4—C3—C2	120.45 (14)
C8—C7—C9	118.35 (13)	C4—C3—H3	119.8
C7—C8—H8A	109.5	C2—C3—H3	119.8
C7—C8—H8B	109.5	C3—C4—C5	119.03 (13)
H8A—C8—H8B	109.5	C3—C4—H4	120.5
C7—C8—H8C	109.5	C5—C4—H4	120.5
H8A—C8—H8C	109.5	C6—C5—C4	121.31 (13)
H8B—C8—H8C	109.5	C6—C5—H5	119.3
C7—C9—H9A	109.5	C4—C5—H5	119.3
C7—C9—H9B	109.5	C5—C6—C1	121.26 (14)
H9A—C9—H9B	109.5	C5—C6—H6	119.4
C7—C9—H9C	109.5	C1—C6—H6	119.4

C1—C2—N21—O21	−0.3 (2)	C6—C1—C2—N21	−177.67 (12)
C1—C2—N21—O22	178.88 (13)	C3—C2—N21—O22	0.8 (2)
C6—C1—N1—N2	4.88 (19)	C3—C2—N21—O21	−178.37 (13)
C2—C1—N1—N2	−175.52 (12)	C1—C2—C3—C4	0.2 (2)
C1—N1—N2—C7	175.80 (12)	N21—C2—C3—C4	178.23 (12)
N1—N2—C7—C8	179.80 (12)	C2—C3—C4—C5	−0.4 (2)
N1—N2—C7—C9	0.2 (2)	C3—C4—C5—C6	0.2 (2)
N1—C1—C2—C3	−179.34 (13)	C4—C5—C6—C1	0.2 (2)
C6—C1—C2—C3	0.28 (19)	N1—C1—C6—C5	179.15 (13)
N1—C1—C2—N21	2.7 (2)	C2—C1—C6—C5	−0.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O21	0.88	1.97	2.6010 (17)	128