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But-2-enal 2,4-dinitrophenylhydrazone

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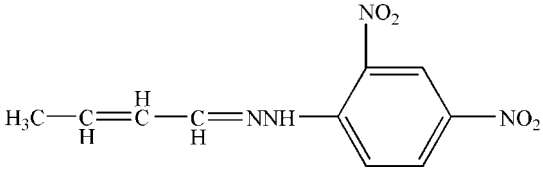
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.111; data-to-parameter ratio = 15.0.

In the title compound, $C_{10}H_{10}N_4O_4$, the but-2-enal chain is almost planar, the largest deviation from the mean plane being 0.013 (1) Å, and this plane makes a dihedral angle of 9.95 (24)° with the benzene ring,. Of the two nitro groups, one is twisted with respect to the benzene ring, making a dihedral angle of 5.7 (1)°, whereas the other is nearly in the plane of the benzene ring, with a twist angle of only 0.7 (1)°. This difference is related to the occurence of an intramolecular N—H···O hydrogen bond with the O atom of the less twisted nitro group. The NH group is also involved in a weak interaction with the same O atom of a symmetry-related molecule, thus forming a pseudo inversion dimer.

Related literature

For general background, see: Okabe *et al.* (1993). For related structures, see: Bolte & Dill (1998); Ohba (1996).



Experimental

Crystal data

$C_{10}H_{10}N_4O_4$
M_r = 250.22
Monoclinic, $P2_1/c$
a = 4.6699 (8) Å
b = 13.188 (2) Å
c = 18.880 (3) Å
β = 92.565 (3)°
V = 1161.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (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} = 0.972, T_{max} = 0.976
9152 measured reflections
2458 independent reflections
1326 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.111
S = 0.95
2458 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	2.02	2.6314 (18)	127
N2—H2···O1^{i}	0.86	2.52	3.331 (2)	159

Symmetry code: (i) $-x+1, -y+1, -z$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2390).

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But-2-enal 2,4-dinitrophenylhydrazone

Zhi-Gang Yin, Heng-Yu Qian, Yu-Zhen Chen and Jie Hu

S1. 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).

In the title compound, the but-2-enal chain is planar with the largest deviation from the mean plane being 0.013 (1)Å at C1. This plane makes a dihedral angle of 9.95 (24)° with the benzene ring, so the whole molecule is roughly planar (Fig. 1). Of the the two nitro groups, one is twisted with respect to the benzene ring making a dihedral angle of 5.7 (1)° whereas the other is nearly in the plane of the benzene ring with a twist angle of only 0.7 (1)°. This difference is related to the occurence of an intramolecular hydrogen N-H···bond with the O atom of the less twisted nitro group (Table 1). The NH is also in weak intermolecular interaction with the same O atom of a symmetry related molecule building a pseudo dimer (Table 1, Fig. 2).

Bond lengths and bond angles are consistent with those of other dinitrophenylhydrazone derivatives(Ohba,1996; Bolte & Dill, 1998)

S2. 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, but-2-enal (1 mmol 0.070 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 methanol, red single crystals of (I) was obtained after two weeks.

S3. Refinement

H atoms were placed in calculated position and treated as riding with C—H= 0.93Å (aromatic), 0.96Å(methyl) and N-H= 0.86\%A with U_{iso}(H)=1.2U_{eq}(C,N) or U_{iso}(H)=1.5U_{eq}(methyl).

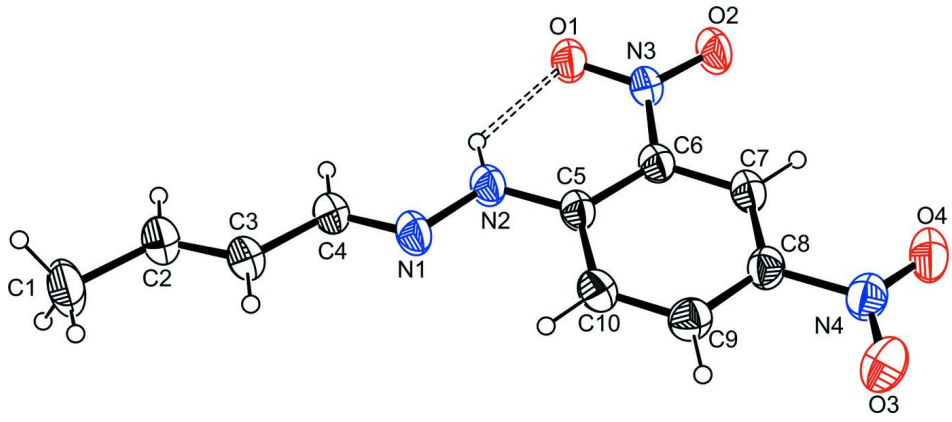

Figure 1

Molecular view of (I) with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radii. Intramolecular H bond is shown as dashed line.

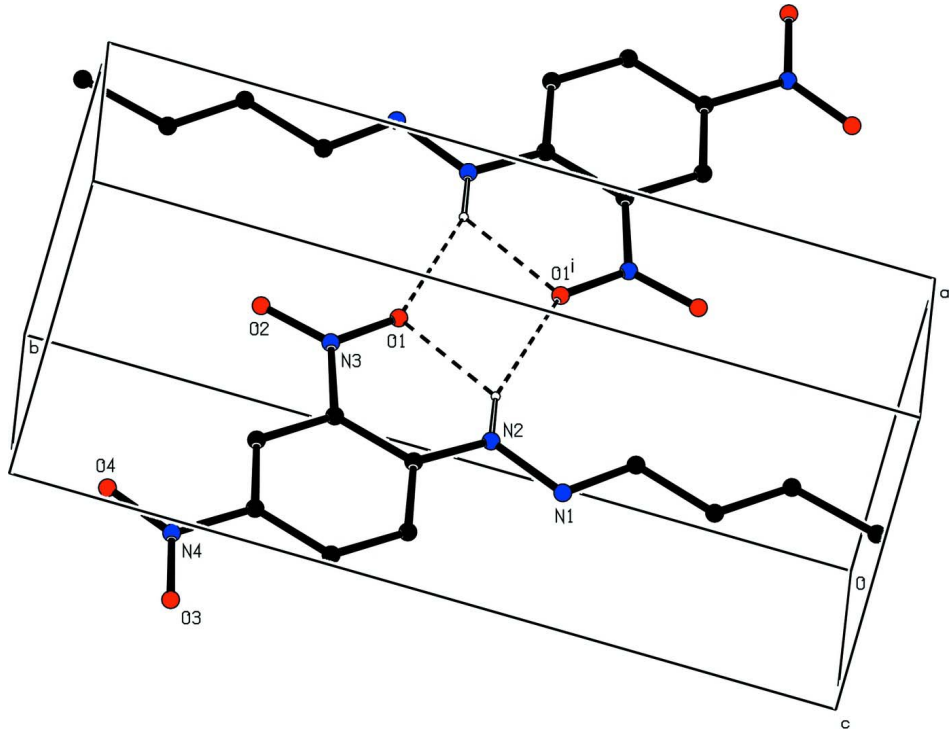

Figure 2

Partial packing view showing the formation of pseudo dimer through weak intermolecular N-H···O hydrogen bonds which are shown as dashed lines. H atoms not involved in hydrogen bondings have been omitted for clarity.[Symmetry code: (i) -x+1, -y+1, -z]

But-2-enal 2,4-dinitrophenylhydrazone

Crystal data

$C_{10}H_{10}N_4O_4$
M_r = 250.22
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 4.6699 (8) Å
b = 13.188 (2) Å

c = 18.880 (3) Å
β = 92.565 (3)°
V = 1161.6 (3) Å^3
Z = 4
F(000) = 520
D_x = 1.432 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 1673 reflections
θ = 2.1–25.5°
μ = 0.11 mm^{-1}
T = 293 K
Block, red
0.27 × 0.23 × 0.23 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.972, T_{max} = 0.976

9152 measured reflections
2458 independent reflections
1326 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.0°, θ_{min} = 2.7°
h = −5→5
k = −16→16
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.111
S = 0.95
2458 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > \sigma(F^2)$ 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}
N1	0.0380 (3)	0.35052 (10)	0.11439 (8)	0.0607 (4)
N2	0.1292 (3)	0.44685 (9)	0.09740 (8)	0.0563 (4)
H2	0.2613	0.4552	0.0676	0.068*
N3	0.2785 (3)	0.65667 (11)	0.05938 (8)	0.0554 (4)
N4	−0.3915 (4)	0.77803 (13)	0.22849 (9)	0.0673 (5)
O1	0.4042 (3)	0.58766 (9)	0.02881 (7)	0.0693 (4)
O2	0.3266 (3)	0.74610 (9)	0.04720 (8)	0.0761 (4)
O3	−0.5851 (3)	0.75891 (12)	0.26884 (9)	0.0872 (5)
O4	−0.3125 (3)	0.86398 (12)	0.21507 (9)	0.0909 (5)
C1	0.1822 (6)	−0.01443 (14)	0.09371 (14)	0.1054 (9)

H1A	0.0157	−0.0191	0.1216	0.158*
H1B	0.3428	−0.0449	0.1191	0.158*
H1C	0.1469	−0.0492	0.0495	0.158*
C2	0.2468 (5)	0.09525 (15)	0.07954 (12)	0.0770 (6)
H22	0.4003	0.1089	0.0513	0.092*
C3	0.1060 (5)	0.17380 (13)	0.10341 (10)	0.0651 (5)
H3	−0.0512	0.1615	0.1307	0.078*
C4	0.1811 (4)	0.27691 (12)	0.08969 (10)	0.0585 (5)
H4	0.3370	0.2906	0.0623	0.070*
C5	0.0079 (4)	0.52752 (12)	0.12821 (9)	0.0497 (4)
C6	0.0733 (4)	0.63020 (12)	0.11150 (9)	0.0506 (4)
C7	−0.0580 (4)	0.71065 (13)	0.14479 (10)	0.0539 (5)
H7	−0.0131	0.7771	0.1330	0.065*
C8	−0.2534 (4)	0.69186 (13)	0.19495 (10)	0.0556 (5)
C9	−0.3232 (4)	0.59241 (15)	0.21334 (10)	0.0603 (5)
H9	−0.4562	0.5806	0.2476	0.072*
C10	−0.1960 (4)	0.51299 (13)	0.18104 (10)	0.0582 (5)
H10	−0.2439	0.4473	0.1939	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0727 (10)	0.0384 (8)	0.0716 (10)	−0.0054 (7)	0.0084 (8)	0.0014 (7)
N2	0.0651 (9)	0.0405 (8)	0.0639 (9)	−0.0042 (7)	0.0116 (7)	0.0008 (7)
N3	0.0681 (10)	0.0380 (8)	0.0602 (9)	0.0009 (7)	0.0032 (8)	0.0008 (7)
N4	0.0677 (11)	0.0668 (12)	0.0670 (11)	0.0122 (9)	−0.0015 (9)	−0.0095 (9)
O1	0.0902 (10)	0.0448 (7)	0.0747 (9)	0.0027 (7)	0.0259 (7)	−0.0002 (6)
O2	0.1022 (11)	0.0386 (7)	0.0894 (10)	−0.0035 (7)	0.0259 (8)	0.0066 (6)
O3	0.0782 (10)	0.0913 (11)	0.0937 (11)	0.0151 (8)	0.0195 (9)	−0.0164 (8)
O4	0.1150 (13)	0.0545 (9)	0.1047 (12)	0.0163 (9)	0.0220 (10)	−0.0058 (8)
C1	0.162 (2)	0.0418 (11)	0.110 (2)	0.0019 (14)	−0.0186 (17)	0.0004 (12)
C2	0.1028 (17)	0.0469 (11)	0.0809 (14)	0.0004 (11)	0.0013 (12)	−0.0004 (10)
C3	0.0838 (14)	0.0428 (10)	0.0687 (12)	−0.0059 (9)	0.0057 (10)	0.0026 (9)
C4	0.0724 (12)	0.0427 (10)	0.0606 (11)	−0.0040 (9)	0.0060 (9)	0.0011 (8)
C5	0.0527 (10)	0.0394 (9)	0.0564 (11)	−0.0010 (8)	−0.0037 (8)	−0.0010 (7)
C6	0.0528 (10)	0.0445 (9)	0.0543 (10)	−0.0006 (8)	−0.0001 (9)	0.0012 (8)
C7	0.0572 (11)	0.0406 (9)	0.0632 (11)	0.0014 (8)	−0.0062 (9)	−0.0002 (8)
C8	0.0548 (11)	0.0519 (11)	0.0596 (11)	0.0098 (9)	−0.0014 (9)	−0.0042 (9)
C9	0.0545 (11)	0.0661 (12)	0.0605 (12)	0.0025 (9)	0.0051 (9)	−0.0002 (9)
C10	0.0622 (11)	0.0474 (10)	0.0649 (12)	−0.0042 (9)	0.0039 (9)	0.0056 (9)

Geometric parameters (Å, º)

N1—C4	1.278 (2)	C2—C3	1.317 (3)
N1—N2	1.3821 (17)	C2—H22	0.9300
N2—C5	1.349 (2)	C3—C4	1.431 (2)
N2—H2	0.8600	C3—H3	0.9300
N3—O2	1.2245 (17)	C4—H4	0.9300

N3—O1	1.2396 (17)	C5—C10	1.422 (3)
N3—C6	1.446 (2)	C5—C6	1.427 (2)
N4—O4	1.2221 (19)	C6—C7	1.390 (2)
N4—O3	1.234 (2)	C7—C8	1.367 (3)
N4—C8	1.465 (2)	C7—H7	0.9300
C1—C2	1.504 (3)	C8—C9	1.399 (3)
C1—H1A	0.9600	C9—C10	1.362 (2)
C1—H1B	0.9600	C9—H9	0.9300
C1—H1C	0.9600	C10—H10	0.9300
C4—N1—N2	116.23 (15)	N1—C4—C3	121.30 (18)
C5—N2—N1	119.02 (14)	N1—C4—H4	119.4
C5—N2—H2	120.5	C3—C4—H4	119.4
N1—N2—H2	120.5	N2—C5—C10	120.21 (15)
O2—N3—O1	121.65 (15)	N2—C5—C6	123.70 (16)
O2—N3—C6	119.54 (15)	C10—C5—C6	116.08 (16)
O1—N3—C6	118.80 (14)	C7—C6—C5	121.43 (16)
O4—N4—O3	123.62 (17)	C7—C6—N3	116.26 (15)
O4—N4—C8	119.16 (19)	C5—C6—N3	122.30 (15)
O3—N4—C8	117.21 (17)	C8—C7—C6	119.78 (16)
C2—C1—H1A	109.5	C8—C7—H7	120.1
C2—C1—H1B	109.5	C6—C7—H7	120.1
H1A—C1—H1B	109.5	C7—C8—C9	120.80 (17)
C2—C1—H1C	109.5	C7—C8—N4	118.64 (17)
H1A—C1—H1C	109.5	C9—C8—N4	120.55 (18)
H1B—C1—H1C	109.5	C10—C9—C8	119.93 (18)
C3—C2—C1	126.1 (2)	C10—C9—H9	120.0
C3—C2—H22	117.0	C8—C9—H9	120.0
C1—C2—H22	117.0	C9—C10—C5	121.97 (16)
C2—C3—C4	123.7 (2)	C9—C10—H10	119.0
C2—C3—H3	118.1	C5—C10—H10	119.0
C4—C3—H3	118.1		
C5—N2—N1—C4	−172.68 (16)	N1—C4—C3—C2	−179.7 (2)
N2—N1—C4—C3	−179.67 (15)	C4—C3—C2—C1	178.41 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.86	2.02	2.6314 (18)	127
N2—H2···O1^{i}	0.86	2.52	3.331 (2)	159

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.