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## Key indicators

Single-crystal X-ray study

T = 298 K

Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$ 

R factor = 0.067

wR factor = 0.159

Data-to-parameter ratio = 17.3

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

## Isobutyraldehyde 2,4-dinitrophenylhydrazone

Crystals of the title compound,  $\text{C}_{10}\text{H}_{12}\text{N}_4\text{O}_4$ , were obtained from a condensation reaction of isobutyraldehyde and 2,4-dinitrophenylhydrazine. The molecule exists in an *E* configuration. In the dinitrophenyl moiety, the average distance of 1.419 (3) Å for the aromatic C—C bonds adjacent to the imino group is appreciably longer than the average distance of 1.375 (3) Å for the other aromatic C—C bonds in the same ring. The overlapped arrangement and separations of 3.387 (15) and 3.369 (15) Å between parallel rings suggest the existence of  $\pi$ – $\pi$ -stacking interactions between neighboring molecules.

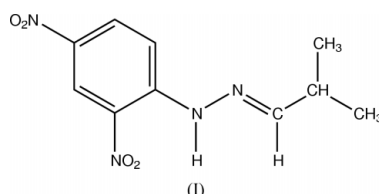
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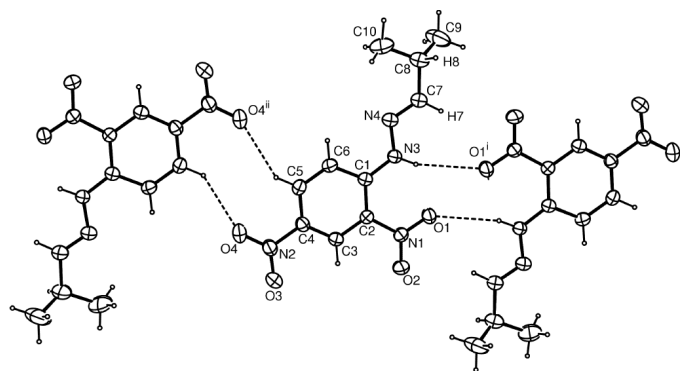
## Comment

Some phenylhydrazones have been shown to be potentially DNA-damaging and mutagenic agents (Okabe *et al.*, 1993). In order to investigate the relationship of the molecular structure and biological activity, a series of phenylhydrazones has been synthesized and their structures determined in this laboratory (Shan *et al.*, 2003).

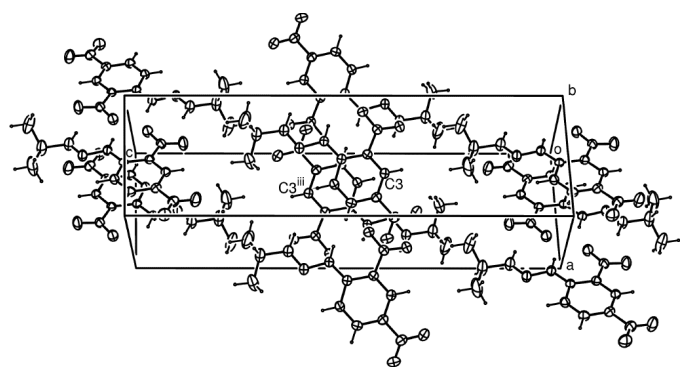


The molecular structure of the title compound, (I), is shown in Fig. 1. The compound crystallizes in an *E* configuration, with the isopropyl and dinitrophenyl groups on opposite sides of the  $\text{N}4=\text{C}7$  double bond. Distances of 1.420 (3) and 1.418 (3) Å for the  $\text{C}1-\text{C}2$  and  $\text{C}1-\text{C}6$  bonds, both adjacent to the imino group, are appreciably longer than the average distance of 1.375 (3) Å for the other aromatic C—C bonds in the same ring, which range from 1.356 (3) to 1.397 (3) Å. This agrees with the situation found in 2,4-dinitrophenylhydrazones reported previously (Bolte & Dill, 1998; Ohba, 1996; Borwick *et al.*, 1997; Naidu *et al.*, 1996; Shan *et al.*, 2002, 2003). The molecule is essentially planar, except for the  $\text{C}9$ -methyl group,  $\text{H}8$  attached to  $\text{C}8$ , and the  $\text{H}$  atoms attached to  $\text{C}10$ . The  $\text{C}10-\text{C}8-\text{C}7-\text{H}7$  torsion angle of  $179^\circ$  and the deviation of 0.049 (3) Å for the  $\text{C}10$  atom from the phenylhydrazone mean plane confirms the coplanarity of atom  $\text{C}10$  with the phenylhydrazone moiety. This configuration minimizes repulsion between atom  $\text{H}7$  and the adjacent isopropyl group.

Hydrogen bonding occurs in the crystal structure, as shown in Fig. 1. Both intramolecular and intermolecular hydrogen



**Figure 1**  
The molecular structure of (I), with 30% probability displacement ellipsoids. Dashed lines indicate the intermolecular hydrogen bonding. [Symmetry codes: (i)  $-x, 2 - y, 1 - z$ ; (ii)  $2 - x, 1 - y, 1 - z$ .]



**Figure 2**  
A molecular packing diagram, showing  $\pi$ - $\pi$  stacking between neighboring rings. [Symmetry code: (iii)  $1 - x, 1 - y, 1 - z$ .]

bonding exists between the imino and nitro groups; the latter very weak interaction results in a short  $O1 \cdots O1(-x, 2 - y, 1 - z)$  distance of 2.758 (3) Å. A weak intermolecular  $C-H \cdots O$  hydrogen bond occurs between the nitro and aromatic groups, as shown in Fig. 1 and Table 2.

The overlapped arrangement of aromatic rings from neighboring molecules (Fig. 2) and the separations of 3.387 (15) Å (symmetry code:  $1 - x, 1 - y, 1 - z$ ) and 3.369 (15) Å (symmetry code:  $1 - x, 2 - y, 1 - z$ ) between parallel rings suggest the existence of  $\pi$ - $\pi$ -stacking interactions between neighboring molecules.

## Experimental

2,4-Dinitrophenylhydrazine (0.4 g, 2 mmol) was dissolved in ethanol (10 ml), and  $H_2SO_4$  solution (98%, 0.5 ml) was slowly added to the ethanol solution with stirring. The solution was heated at about 333 K for several minutes until the solution cleared. Isobutyraldehyde (0.14 g, 2 mmol) was added dropwise to the solution with continuous stirring, and the mixture was refluxed for 30 min. When the solution had cooled to room temperature yellow powdery crystals appeared. The powdery crystals were separated and washed with water three times. Recrystallization from absolute ethanol yielded well shaped single crystals.

## Crystal data

$C_{10}H_{12}N_4O_4$   
 $M_r = 252.24$   
Monoclinic,  $P2_1/c$   
 $a = 7.0514$  (10) Å  
 $b = 7.3862$  (12) Å  
 $c = 23.6459$  (16) Å  
 $\beta = 92.372$  (13)°  
 $V = 1230.5$  (3) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.362$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 6730 reflections  
 $\theta = 1.8$ – $27.4$ °  
 $\mu = 0.11$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
Plate, yellow  
 $0.22 \times 0.20 \times 0.11$  mm

## Data collection

Rigaku R-Axis RAPID diffractometer  
 $\omega$  scans  
Absorption correction: none  
11248 measured reflections  
2820 independent reflections

1899 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.037$   
 $\theta_{max} = 27.5$ °  
 $h = -8 \rightarrow 9$   
 $k = -9 \rightarrow 9$   
 $l = -30 \rightarrow 30$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.067$   
 $wR(F^2) = 0.159$   
 $S = 1.07$   
2820 reflections  
163 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.539P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.36$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.24$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å).

N3—C1	1.346 (3)	C3—C4	1.363 (3)
N3—N4	1.387 (2)	C4—C5	1.397 (3)
N4—C7	1.259 (3)	C5—C6	1.356 (3)
C1—C6	1.418 (3)	C7—C8	1.501 (3)
C1—C2	1.420 (3)	C8—C9	1.485 (5)
C2—C3	1.384 (3)	C8—C10	1.490 (5)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N3—H3 $\cdots$ O1	0.86	2.02	2.624 (2)	127
N3—H3 $\cdots$ O1 <sup>i</sup>	0.86	2.56	3.353 (3)	154
C5—H5 $\cdots$ O4 <sup>ii</sup>	0.93	2.48	3.283 (3)	145

Symmetry codes: (i)  $-x, 2 - y, 1 - z$ ; (ii)  $2 - x, 1 - y, 1 - z$ .

The H atoms were placed in calculated positions with  $C-H = 0.93$ – $0.98$  Å and  $N-H = 0.86$  Å; they were included in the final cycles of refinement in the riding-model approximation, with  $U_{iso}(H) = 1.2U_{eq}$  or  $1.5U_{eq}$  of the carrier atoms.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK & Rigaku, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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