

## Propanone 2-chloro-4,6-dinitrophenylhydrazone

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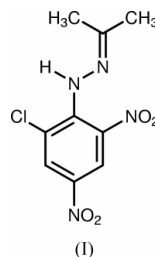
## Key indicators

Single-crystal X-ray study  
 $T = 93$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.030  
 $wR$  factor = 0.103  
Data-to-parameter ratio = 12.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The molecule of the title compound,  $\text{C}_9\text{H}_9\text{ClN}_4\text{O}_4$ , is nearly planar except for the *ortho*-nitro group, with the non-H atoms exhibiting an average deviation of 0.099 Å from the mean plane. The *ortho*-nitro group is nearly perpendicular to the phenyl group, with a dihedral angle of 88.46 (3)°. The N–N and N=C bond lengths of the isopropylidene-hydrazine moiety are 1.3928 (14) and 1.2865 (15) Å, respectively.

## Comment

The structures of a wide range of 2,4-dinitrophenylhydrazones have been studied extensively, particularly with respect to hydrogen bonding (Fronczek, 1994; Kaberia *et al.*, 1980; Vickery *et al.*, 1981). In most of these compounds, the *ortho*-nitro group forms a strong intramolecular hydrogen bond to the imino-group and nearly all the atoms are coplanar (Fronczek, 1994; Vickery *et al.*, 1981).



However, the introduction of a chlorine atom in the other *ortho*-position drastically changes the molecular structure. When an *ortho*-chlorine atom is present, there are no intra- and inter-molecular hydrogen bonds. The *ortho*-nitro group of the title compound, (I), is nearly perpendicular to the phenyl group, with an O1–N3–C5–C6 torsion angle of 88.7 (2)°.

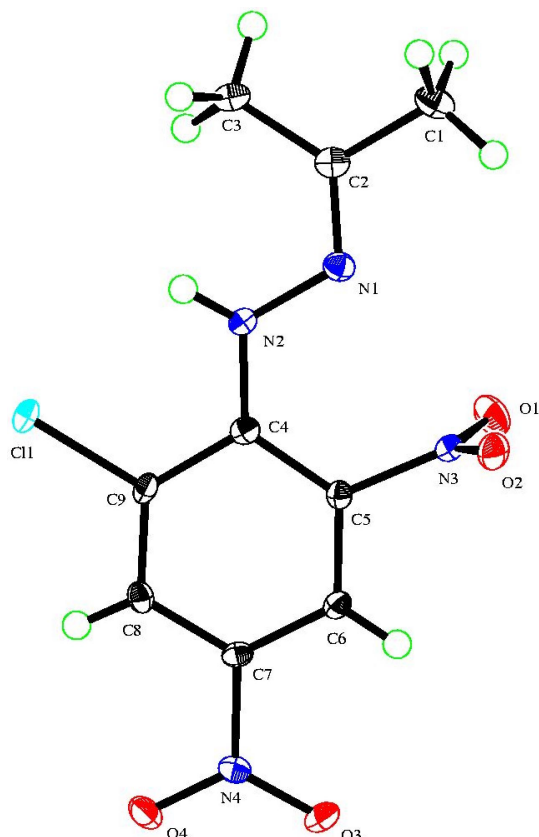
## Experimental

The title compound was synthesized according to the method of Kawai *et al.* (2002). Spectroscopic analysis for (I),  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz,  $\delta$ , p.p.m.): 2.02(s, 3H), 2.06(s, 3H), 8.36(d, 1H,  $J = 2.54$  Hz), 8.46(s, 1H), 8.50(d, 1H,  $J = 2.54$  Hz). Crystals of (I), suitable for X-ray diffraction, were obtained by slow evaporation of a dichloromethane solution at room temperature.

## Crystal data

$\text{C}_9\text{H}_9\text{ClN}_4\text{O}_4$   
 $M_r = 272.65$   
Monoclinic,  $P2_1/a$   
 $a = 8.6057$  (11) Å  
 $b = 16.110$  (1) Å  
 $c = 8.9967$  (11) Å  
 $\beta = 115.557$  (5)°  
 $V = 1125.3$  (2) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.609$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 3753  
reflections  
 $\theta = 1.5$ –13.7°  
 $\mu = 0.35$  mm<sup>-1</sup>  
 $T = 93.1$  K  
Prism, yellow  
0.47 × 0.38 × 0.22 mm



**Figure 1**  
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

#### Data collection

Rigaku Mercury CCD  
diffractometer  
 $\omega$  scans  
Absorption correction: none  
8602 measured reflections  
2545 independent reflections

2376 reflections with  $F^2 > 2\sigma(F^2)$   
 $R_{\text{int}} = 0.020$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -11 \rightarrow 9$   
 $k = -20 \rightarrow 20$   
 $l = -11 \rightarrow 11$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.103$   
 $S = 1.03$   
2545 reflections  
199 parameters

All H-atom parameters refined  
 $w = (4F_o^2)/[0.0012F_o^2 + \sigma^2(F_o)]$   
 $(\Delta/\sigma)_{\text{max}} = 0.004$   
 $\Delta\rho_{\text{max}} = 0.38 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.32 \text{ e } \text{\AA}^{-3}$

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C1—C9	1.7303 (12)	N2—C4	1.3552 (15)
O1—N3	1.2177 (14)	C1—C2	1.4961 (16)
O2—N3	1.2291 (14)	C2—C3	1.5039 (16)
O3—N4	1.2310 (13)	C4—C5	1.4088 (15)
O4—N4	1.2316 (13)	C4—C9	1.4249 (15)
N1—N2	1.3928 (14)	C8—C9	1.3697 (16)
N1—C2	1.2865 (15)		
N2—N1—C2	115.6 (1)	N2—C4—C9	119.5 (1)
N1—N2—C4	119.5 (1)	N3—C5—C4	122.1 (1)
O1—N3—O2	125.0 (1)	N3—C5—C6	114.5 (1)
O3—N4—O4	124.0 (1)	N4—C7—C6	118.6 (1)
N1—C2—C1	116.15 (11)	N4—C7—C8	119.5 (1)
N1—C2—C3	125.95 (11)	C11—C9—C4	117.49 (9)
C1—C2—C3	117.9 (1)	C11—C9—C8	119.09 (9)
N2—C4—C5	125.7 (1)		
C2—N1—N2—C4	172.4 (2)	O1—N3—C5—C6	88.7 (2)
N1—N2—C4—C5	−7.9 (2)	O4—N4—C7—C6	179.8 (1)

H atoms were found in difference Fourier maps and refined isotropically, with C—H distances in the range 0.87 (1)–0.88 (2)  $\text{\AA}$  for aromatic-H and 0.96 (2)–1.01 (2)  $\text{\AA}$  for alkyl-H, and an N—H distance of 0.85 (2)  $\text{\AA}$ .

Data collection: *CrystalClear* (Molecular Structure Corporation/Rigaku Corporation, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Molecular Structure Corporation/Rigaku Corporation, 2001); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1999); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *CrystalStructure*.

#### References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Fronczek, F. R. (1994). *Acta Cryst.* **C50**, 122–124.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Kaberia, F., Vickery, B., Willey, G. R. & Drew, M. G. B. (1980). *J. Chem. Soc. Perkin Trans. 2*, pp. 1622–1626.
- Kawai, Y., Hayashi, M. & Tokitoh, N. (2002). *Tetrahedron Lett.* **43**, 465–467.
- Molecular Structure Corporation/Rigaku Corporation. (2001). *CrystalClear* (Version 1.3) and *CrystalStructure* (Version 3.00). MSC, 9009 New Trails Drive, The Woodlands, TX, 77381-5209, USA, and Rigaku, 3-9-12 Akishima, Tokyo 196-8666, Japan.
- Vickery, B., Willey, G. R. & Drew, M. G. B. (1981). *J. Chem. Soc. Perkin Trans. 2*, pp. 155–160.
- Watkin, D. J., Prout, C. K., Carruthers, J. R. & Betteridge, P. W. (1999). *CRYSTALS*. Issue 10, Chemical Crystallography Laboratory, Oxford, UK.