

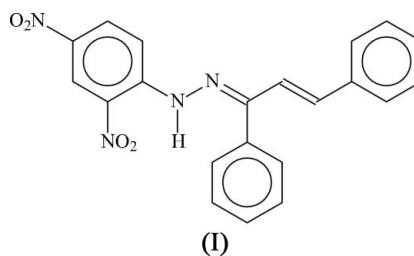
Benzylideneacetophenone 2,4-dinitrophenyl-  
hydrazoneHeng-Yu Qian,<sup>a</sup> Cheng Liu,<sup>b</sup>  
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## Key indicators

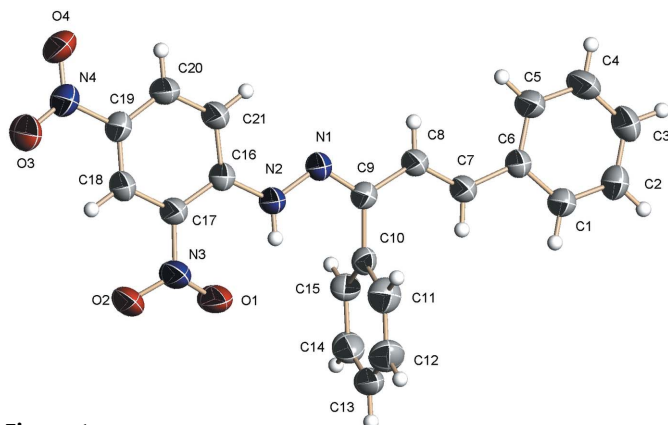
Single-crystal X-ray study  
 $T = 291$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
Disorder in main residue  
 $R$  factor = 0.038  
 $wR$  factor = 0.120  
Data-to-parameter ratio = 11.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.The title Schiff base compound,  $\text{C}_{21}\text{H}_{16}\text{N}_4\text{O}_4$ , features a  
crystallographically imposed planar  $-\text{NH}-\text{N}=\text{C}$  linkage of an  
*E* configuration.Received 31 March 2006  
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## Comment

2,4-Dinitrophenylhydrazine is a reagent that is used for the  
formation of crystalline derivatives of aldehydes and ketones,  
as it usually affords sharp-melting solids (Furniss *et al.*, 1989),  
and the crystal structures of a large number of these Schiff  
base hydrazones have been determined, as noted from the  
Cambridge Structural Database (Version 5.27; Allen, 2002).  
The majority of the aryl-aryl and aryl-alkyl ketone derivatives  
whose structures have been reported have substituents on the  
phenyl ring. There are few examples of phenyl-alkyl ketone  
derivatives, and these include only the derivatives of acetophenone,  
*viz.* the derivatives of acetophenone itself (Shan *et al.*, 2002*a*),  
phenyl ethyl ketone (Shan *et al.*, 2002*b*) and phenyl  
phenylhydroxymethyl ketone (de Lima *et al.*, 2001).In the title compound, (I), the aromatic ring of the  
dinitrophenylhydrazine unit and benzylidene portion of the  
benzylideneacetophenone unit lie on a mirror plane (Fig. 1).  
The structure is disordered, as the two nitro groups of the  
phenylhydrazine portion as well as the phenyl ring that is next  
to the  $-\text{NH}-\text{N}=\text{C}$  unit, lie off the mirror plane; the phenyl  
ring is perpendicular to the mirror plane [dihedral angle  
 $90.0(4)^\circ$ ].

## Experimental

2,4-Dinitrophenylhydrazine (0.20 g, 1 mmol) was dissolved in anhy-  
drous ethanol (10 ml) and to the solution was added concentrated  
sulfuric acid (0.5 ml). The solution was heated to the boiling point of  
ethanol and then benzylideneacetophenone (0.21 g, 1 mmol)  
dissolved in ethanol (6 ml) was added. The mixture was then refluxed  
for several hours. After removing the solvent by evaporation, the  
product was collected and recrystallized from dichloromethane to  
yield orange crystals of (I). The preparation was first reported in 1937  
(Dippy *et al.*, 1937).



**Figure 1**  
A plot of (I). Displacement ellipsoids are drawn at the 50% probability level. Only one disorder component is shown.

#### Crystal data

$C_{21}H_{16}N_4O_4$	$Z = 4$
$M_r = 388.38$	$D_x = 1.369 \text{ Mg m}^{-3}$
Orthorhombic, $Pnma$	Mo $K\alpha$ radiation
$a = 18.570 (1) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$b = 6.9335 (4) \text{ \AA}$	$T = 291 (2) \text{ K}$
$c = 14.6389 (9) \text{ \AA}$	Plate, brown
$V = 1884.8 (2) \text{ \AA}^3$	$0.24 \times 0.21 \times 0.05 \text{ mm}$

#### Data collection

Bruker APEX area-detector diffractometer	2328 independent reflections
$\varphi$ and $\omega$ scans	1654 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.025$
16257 measured reflections	$\theta_{\text{max}} = 27.5^\circ$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.176P]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.120$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
2328 reflections	$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$
209 parameters	Extinction correction: <i>SHELXL97</i> (Sheldrick, 1997)
H-atom parameters constrained	Extinction coefficient: 0.0030 (8)

The two nitro groups are disordered across a mirror plane, as is the phenyl ring that is near to the  $-\text{NH}-\text{N}=\text{}$  unit. These were allowed

to refine across the symmetry element. The ring was restrained to be nearly flat and the 1,2-related distance was restrained to  $1.39 (1) \text{ \AA}$ , whereas the 1,4-related distance was restrained to  $2.78 (1) \text{ \AA}$ . The two nitro groups were allowed to refine off the mirror plane but with distance restraints of  $\text{N}-\text{O} = 1.22 (1) \text{ \AA}$  and  $\text{O}\cdots\text{O} = 2.11 (1) \text{ \AA}$ . Additionally, the four-atom unit  $\text{C}(\text{NO}_2)$  was restrained to be approximately flat. As the refinement led to a  $y$ -coordinate of  $0.251 (2)$  for N4, the N atom of this nitro group is ordered. The refinement proceeded with this atom lying on the mirror plane. H atoms were placed in calculated positions ( $\text{N}-\text{H} = 0.86 \text{ \AA}$ ) and  $\text{C}-\text{H} = 0.93 \text{ \AA}$ ), and were included in the refinement in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

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