

Ji-Cheng Chu,* Wei Li and
Dong-Zhi LiuSchool of Chemical Engineering and
Technology, Tianjin University, Tianjin 300072,
People's Republic of China

Correspondence e-mail: cheryycao@eyou.com

Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.038
 wR factor = 0.106
Data-to-parameter ratio = 15.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.***N*-Methyl-2-nitro-*N*-(4-nitrobenzoyloxy)-1-phenyl-
propan-1-amine**Two compounds were obtained *via* the addition of *N*-methylhydroxylamine to 1-phenyl-2-nitropropene. The structure of the major product, the title compound, (I), $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_6$, is reported here. Both asymmetric C atoms (C1 and C2) have the same chirality, either *R,R* or *S,S*.

Received 28 June 2005

Accepted 15 July 2005

Online 23 July 2005

Comment

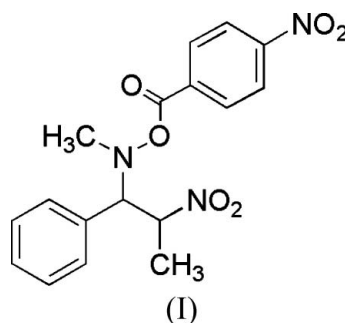
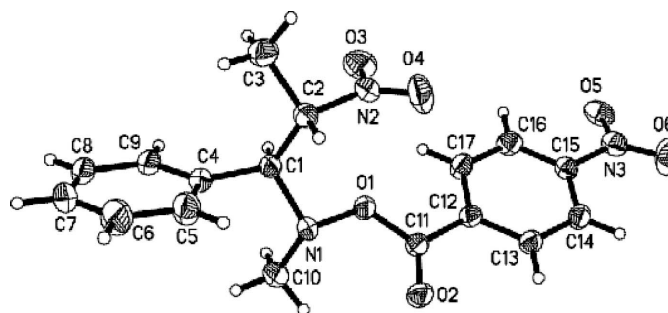
The Michael addition reaction of *N*-methylhydroxylamine and 1-phenyl-2-nitropropene was expected to be stereoselective. However, the absolute configuration of the product was difficult to determine because it did not crystallize. When the main product of this reaction was further reacted with *p*-nitrobenzoyl chloride (Pan *et al.*, 1999), it produced the title compound, (I) (Fig. 1), which crystallized readily. However, since the space group is centrosymmetric, only the relative configuration of the two asymmetric C atoms could be determined. The X-ray study showed that both asymmetric C atoms (C1 and C2) have the same hand, either *R,R* or *S,S*.The C10/N1/C1–C3 chain is extended and planar to within ± 0.097 (2) Å. The aromatic ring (C4–C9) is approximately perpendicular to this plane, with a dihedral angle between the two planes of 85.96 (7)°. The benzyloxy group is also planar

Figure 1
The molecular structure of (I), drawn with 30% probability displacement ellipsoids.

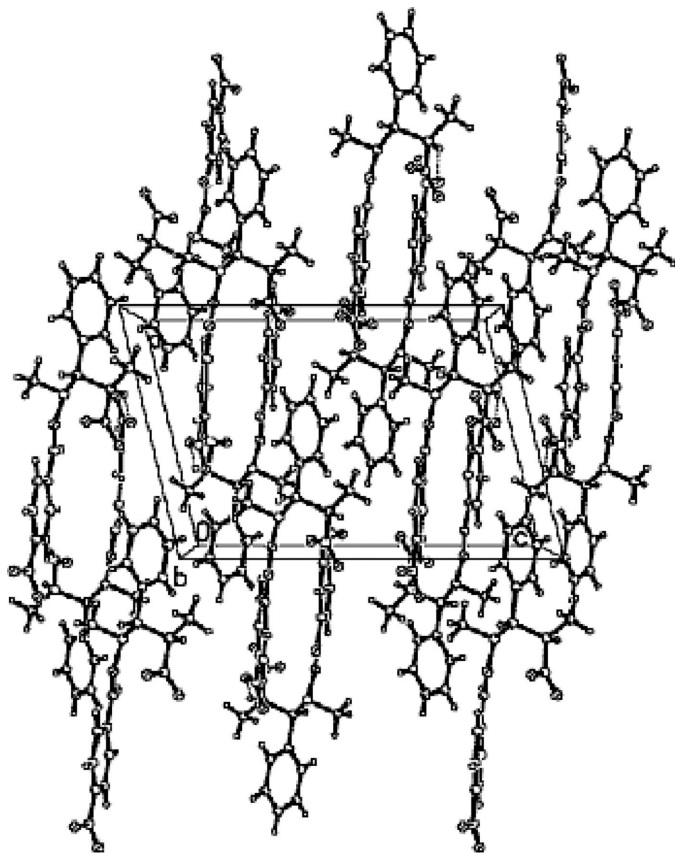


Figure 2
The crystal packing for (I), viewed along the *b* axis.

to within ± 0.112 (1) Å, with a dihedral angle of 71.73 (6) $^\circ$ to the C10/N1/C1–C3 chain. The crystal packing of (I) is illustrated in Fig. 2.

Experimental

To a solution of 1-phenyl-2-nitropropene (1 mmol) in methanol (5 ml), *N*-methylhydroxyamine hydrochloride (1.5 mmol) and triethylamine (1.3 mmol) were added (Baldwin & Aube, 1987). The resulting solution was stirred at room temperature for 35 min, and then water (30 ml) was added. The mixture was extracted with dichloromethane, and the organic phase was washed with water and dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure and the residue was purified using column chromatography, to give two products in a ratio of 2.36:1. Triethylamine (1.4 mmol) was added dropwise at 273 K to a solution of the main product (1 mmol) and *p*-nitrobenzoyl chloride (1.3 mmol) in dichloromethane (15 ml). The resulting solution was stirred at room temperature for 20 min and water was added (20 ml). The separated organic phase was washed with water again, and then dried with

anhydrous sodium sulfate. The solvent was removed under reduced pressure and the residue was purified using column chromatography to give the title compound. Good quality single crystals of (I) were obtained by the slow evaporation of a dichloromethane–ethanol solution (1:4 *v/v*) over 2 d.

Crystal data

$C_{17}H_{17}N_3O_6$
 $M_r = 359.34$
Monoclinic, $P2_1/n$
 $a = 10.4009$ (15) Å
 $b = 11.3173$ (18) Å
 $c = 15.353$ (3) Å
 $\beta = 103.427$ (5) $^\circ$
 $V = 1757.8$ (5) Å³
 $Z = 4$

$D_x = 1.358$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 1010 reflections
 $\theta = 2.8$ – 22.2 $^\circ$
 $\mu = 0.11$ mm⁻¹
 $T = 294$ (2) K
Block, colourless
 $0.42 \times 0.38 \times 0.36$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{min} = 0.927$, $T_{max} = 0.963$
9626 measured reflections

3584 independent reflections
2249 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.029$
 $\theta_{max} = 26.5$ $^\circ$
 $h = -12 \rightarrow 11$
 $k = -12 \rightarrow 14$
 $l = -19 \rightarrow 14$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.106$
 $S = 1.01$
3584 reflections
238 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 0.3383P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.15$ e Å⁻³
 $\Delta\rho_{min} = -0.14$ e Å⁻³

The methyl H atoms were constrained, with C–H distances of 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$, but each group was allowed to rotate freely about its C–C bond. All other H atoms were positioned geometrically and constrained to ride on their parent atoms, with C–H distances in the range 0.93–0.98 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); 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, 1997); software used to prepare material for publication: SHELXTL.

References

- Bruker (1997). SADABS (Version ?), SMART (Version ?), SAINT (Version ?) and SHELXTL (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Baldwin, S. W. & Aube, J. (1987). *Tetrahedron Lett.* **28**, 179–182.
Pan, S., Wang, J. & Zhao, K. (1999). *J. Org. Chem.* **64**, 4–5.
Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.