Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2′-Dinitrodibenzyl

Hemmige S. Yathirajan,[a] Basavegowda Nagaraj,[a] Padmarajaiah Nagaraja[a] and Daniel E. Lynch[b]*

[a]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and [b]School of Science and the Environment, Coventry University, Coventry CV1 5FB, England

Correspondence e-mail: apx106@coventry.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.061
wR factor = 0.165
Data-to-parameter ratio = 11.9

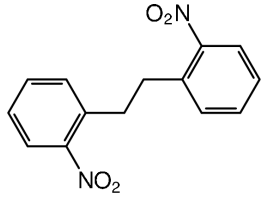
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{14}H_{12}N_2O_4$, there is an inversion centre at the mid-point of the ethylene bridge. The nitro group is inclined at an angle of 33 (2)° to the plane of the phenyl ring. The benzene rings in each molecule are coplanar, but the dihedral angle between the benzene rings in neighbouring molecules is 55.2 (1)°.

Received 18 November 2004
Accepted 22 November 2004
Online 27 November 2004

Comment

The title compound, (I), is an intermediate in the syntheses of the anticonvulsant drugs carbamazepine and oxcarbazepine, and also the antidepressant drugs imipramine and desipramine. The Cambridge Structural Database (Version of Aptil 2004; Allen, 2002) reveals that there are currently eight known crystal structures of 2,2′-disubstituted dibenzyls, including derivatives with substituents such as bromo, methyl and methoxy groups, but not nitro. In the title compound (Fig. 1), there is an inversion centre at the mid-point of the ethylene bridge. Thus, the molecule adopts a stepped *trans* conformation with respect to the benzene rings and nitro groups, respectively. The nitro group is inclined at an angle of 33 (2)° to the plane of the benzene ring. The benzene rings in each molecule are coplanar, but the dihedral angle between the benzene rings in neighbouring molecules is 55.2 (1)°.



(I)

Experimental

The title compound was obtained from Max India Ltd and crystals were grown from ethanol.

Crystal data

$C_{14}H_{12}N_2O_4$
M_r = 272.26
Monoclinic, $P2_1/c$
a = 7.5678 (8) Å
b = 14.4964 (16) Å
c = 5.9874 (5) Å
β = 108.607 (6)°
V = 622.52 (11) Å^3
Z = 2
D_x = 1.452 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1390 reflections
θ = 2.9–27.5°
μ = 0.11 mm^{-1}
T = 120 (2) K
Plate, colourless
0.36 × 0.10 × 0.04 mm

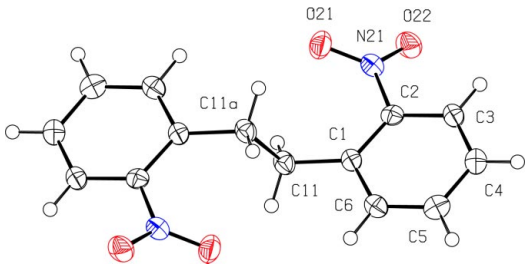


Figure 1
Molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms as spheres of arbitrary radius. [Symmetry code: (*a*) $-x, 1-y, 1-z$.]

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan *SADABS* (Sheldrick, 2003)
$T_{min} = 0.962$, $T_{max} = 0.996$
5201 measured reflections
1092 independent reflections
730 reflections with $I > 2\sigma(I)$
$R_{int} = 0.128$
$\theta_{max} = 25.0°$
$h = -8 \rightarrow 8$
$k = -17 \rightarrow 17$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.165$
$S = 1.03$
1092 reflections
92 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0865P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.033 (12)

All H atoms were included in the refinement at calculated positions, with C—H distances of 0.95 (aromatic H atoms) and 0.99 Å (CH_2 H atoms), and refined as riding, with $U_{iso}(H) = 1.25U_{eq}$(carrier atom). The high R_{int} is the result of weak high-angle data.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO, SCALEPACK* (Otwinowski & Minor, 1997) and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*97 (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

The authors thank the EPSRC National Crystallography Service (Southampton, England) and acknowledge the use of the EPSRC's Chemical Database Service at Daresbury.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.