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2,2′-Dinitrodibenzyl

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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)°.

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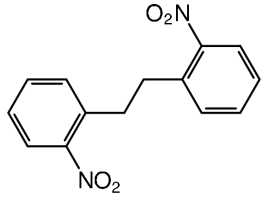
Comment

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

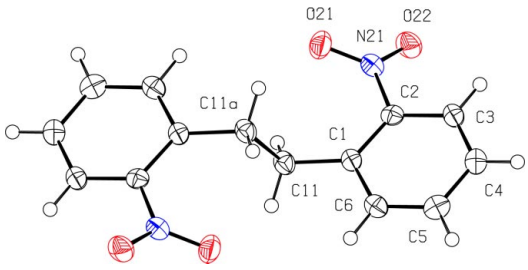


Figure 1
Molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms as spheres of arbitrary radius. [Symmetry code: (*a*) $-x, 1-y, 1-z$.]

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.

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supporting information

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Hall symbol: -P 2ybc
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$b = 14.4964$ (16) Å
$c = 5.9874$ (5) Å
$\beta = 108.607$ (6)°
$V = 622.52$ (11) Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.452$ Mg m^{-3}
Melting point: 404 K K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1390 reflections
$\theta = 2.9$–27.5°
$\mu = 0.11$ mm^{-1}
$T = 120$ K
Plate, colourless
0.36 × 0.10 × 0.04 mm

Data collection

Bruker Nonius 95 mm CCD camera on κ-goniostat diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
10 cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
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 $I > 2\sigma(I)$
$R_{int} = 0.128$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.2°$
$h = -8 \rightarrow 8$
$k = -17 \rightarrow 17$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.165$
$S = 1.03$
1092 reflections
92 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.033 (12)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.1601 (5)	0.40796 (18)	−0.4314 (5)	0.0213 (8)
C11	−0.0148 (5)	0.44779 (18)	−0.5294 (5)	0.0261 (8)
H11	−0.0556	0.4392	−0.7024	0.033*
H12	0.1046	0.4146	−0.4610	0.033*
C2	−0.1235 (5)	0.36536 (18)	−0.2106 (5)	0.0221 (8)
N21	0.0685 (4)	0.35474 (16)	−0.0517 (4)	0.0257 (7)
O21	0.1838 (3)	0.41516 (16)	−0.0476 (4)	0.0381 (7)
O22	0.1050 (3)	0.28615 (13)	0.0754 (3)	0.0347 (7)
C3	−0.2630 (5)	0.32949 (18)	−0.1317 (5)	0.0243 (8)
H3	−0.2323	0.3009	0.0187	0.030*
C4	−0.4460 (5)	0.3356 (2)	−0.2727 (5)	0.0273 (8)
H4	−0.5432	0.3125	−0.2196	0.034*
C5	−0.4868 (5)	0.37598 (19)	−0.4939 (5)	0.0290 (8)
H5	−0.6126	0.3796	−0.5937	0.036*
C6	−0.3460 (5)	0.41086 (19)	−0.5695 (5)	0.0254 (8)
H6	−0.3776	0.4379	−0.7218	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.029 (2)	0.0130 (14)	0.0240 (14)	0.0009 (13)	0.0122 (15)	−0.0019 (10)
C11	0.036 (2)	0.0198 (16)	0.0261 (15)	−0.0012 (14)	0.0143 (15)	−0.0002 (11)
C2	0.027 (2)	0.0165 (14)	0.0222 (14)	0.0010 (13)	0.0076 (15)	−0.0042 (10)
N21	0.0283 (19)	0.0263 (14)	0.0233 (13)	0.0035 (13)	0.0094 (12)	0.0022 (10)
O21	0.0335 (17)	0.0363 (14)	0.0411 (13)	−0.0104 (11)	0.0071 (11)	0.0096 (10)
O22	0.0413 (18)	0.0302 (13)	0.0305 (12)	0.0026 (11)	0.0084 (11)	0.0090 (9)
C3	0.033 (2)	0.0182 (15)	0.0249 (14)	−0.0005 (14)	0.0141 (16)	−0.0010 (11)
C4	0.029 (2)	0.0233 (16)	0.0327 (16)	0.0004 (14)	0.0144 (16)	−0.0004 (12)
C5	0.028 (2)	0.0237 (16)	0.0334 (16)	0.0041 (14)	0.0077 (15)	−0.0033 (12)
C6	0.034 (2)	0.0190 (15)	0.0244 (14)	0.0039 (14)	0.0105 (16)	0.0015 (11)

Geometric parameters (Å, º)

C1—C6	1.386 (4)	N21—O22	1.229 (3)
C1—C2	1.405 (4)	C3—C4	1.376 (5)
C1—C11	1.516 (4)	C3—H3	0.95
C11—C11^{i}	1.554 (5)	C4—C5	1.390 (4)
C11—H11	0.99	C4—H4	0.95
C11—H12	0.99	C5—C6	1.380 (4)
C2—C3	1.388 (4)	C5—H5	0.95
C2—N21	1.469 (4)	C6—H6	0.95
N21—O21	1.231 (3)		
C6—C1—C2	115.6 (2)	O22—N21—C2	117.8 (3)
C6—C1—C11	118.8 (2)	C4—C3—C2	119.5 (3)

C2—C1—C11	125.6 (3)	C4—C3—H3	120.2
C1—C11—C11^{i}	110.7 (3)	C2—C3—H3	120.2
C1—C11—H11	109.5	C3—C4—C5	119.1 (3)
C11^{i}—C11—H11	109.5	C3—C4—H4	120.5
C1—C11—H12	109.5	C5—C4—H4	120.5
C11^{i}—C11—H12	109.5	C6—C5—C4	120.5 (3)
H11—C11—H12	108.1	C6—C5—H5	119.8
C3—C2—C1	122.9 (3)	C4—C5—H5	119.8
C3—C2—N21	116.3 (2)	C5—C6—C1	122.4 (3)
C1—C2—N21	120.8 (2)	C5—C6—H6	118.8
O21—N21—O22	123.1 (3)	C1—C6—H6	118.8
O21—N21—C2	119.1 (2)		

C6—C1—C11—C11^{i}	88.7 (4)	C1—C2—N21—O22	−147.1 (2)
C2—C1—C11—C11^{i}	−92.9 (4)	C1—C2—C3—C4	−0.3 (4)
C6—C1—C2—C3	−1.0 (4)	N21—C2—C3—C4	−179.0 (2)
C11—C1—C2—C3	−179.5 (2)	C2—C3—C4—C5	1.3 (4)
C6—C1—C2—N21	177.6 (2)	C3—C4—C5—C6	−1.0 (4)
C11—C1—C2—N21	−0.9 (4)	C4—C5—C6—C1	−0.3 (4)
C3—C2—N21—O21	−146.8 (2)	C2—C1—C6—C5	1.3 (4)
C1—C2—N21—O21	34.5 (3)	C11—C1—C6—C5	179.9 (2)
C3—C2—N21—O22	31.6 (3)		

Symmetry code: (i) −*x*, −*y*+1, −*z*−1.