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1,2-Bis(2,4,6-trinitrophenyl)ethane

Wen-Yan Wang, Ying Diao, Zhi-Hua Wei and Jian-Long Wang*

School of Chemical Engineering and Environment, North University of China, Taiyuan, People's Republic of China
Correspondence e-mail: wangjianlong@nuc.edu.cn

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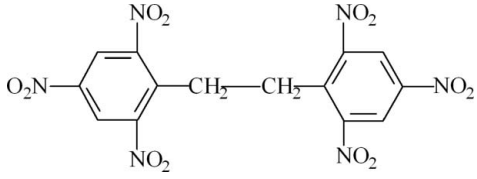
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.034; wR factor = 0.099; data-to-parameter ratio = 10.8.

The title compound, $C_{14}H_8N_6O_{12}$, is centrosymmetric, the midpoint of the central C—C bond being located on an inversion centre. Two of the three independent nitro groups are disordered over two sites, with a site-occupancy ratio of 0.513 (3):0.487 (3). Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure.

Related literature

For the synthesis of the title compound, see: Shipp (1964); Gilbert & Morristown (1980).



Experimental

Crystal data

$C_{14}H_8N_6O_{12}$
M_r = 452.26
Monoclinic, $P2_1/c$
a = 5.8468 (5) Å
b = 8.1253 (11) Å
c = 17.977 (2) Å
β = 97.154 (8)°
V = 847.38 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 113 K
0.22 × 0.20 × 0.16 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2000)
T_{min} = 0.966, T_{max} = 0.975
7531 measured reflections
2013 independent reflections
1503 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.099
S = 1.04
2013 reflections
186 parameters
70 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O4^{i}	0.99	2.43	3.3669 (15)	158
C1—H1B···O5^{ii}	0.99	2.37	3.147 (2)	134

Symmetry codes: (i) $x, y+1, z$; (ii) $-x, -y+1, -z$.

Data collection: *CrystalClear* (Rigaku/MSC, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5348).

References

Gilbert, E. E. & Morristown, N. J. (1980). US Patent 4221745.
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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shipp, K. G. (1964). *J. Org. Chem.* **29**, 2620–2623.

supporting information

Acta Cryst. (2011). E**67**, o3044 [doi:10.1107/S1600536811042802]

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S1. Comment

2,2′,4,4′,6,6′-Hexanitrostilbene is one of the most important heat resistant explosives. It can be prepared by treating the solution of TNT in tetrahydrofuran–methanol mixture with 5% sodium hypochlorite (Shipp, 1964). Later on its synthesis method was improved by Gilbert & Morristown (1980). As an intermediate, 2,2′,4,4′,6,6′-hexanitrobibenzyl was synthesized by the oxidation of TNT. Here we report the crystal structure of the title compound.

In the crystal structure, there is an inversion center in the molecule. Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure.

S2. Experimental

The title compound was prepared according to literature method (Gilbert & Morristown, 1980). Single crystals were obtained by evaporation of a solution of the title compound in acetone at room temperature.

S3. Refinement

N1-Nitro and N3-nitro groups are disordered over two sites, occupancy ratio was refined to 0.513 (3):0.487 (3). For the disordered components, thermal parameters of the primed atoms were set to those of the unprimed ones, and all anisotropic thermal parameters were restrained to be nearly isotropic. The N—O distances were restrained to within 0.01 Å in the N1-nitro and N3-nitro groups. H atoms were positioned geometrically with C—H = 0.95 Å for benzene ring H and 0.99 Å for methylene H atoms, refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C).

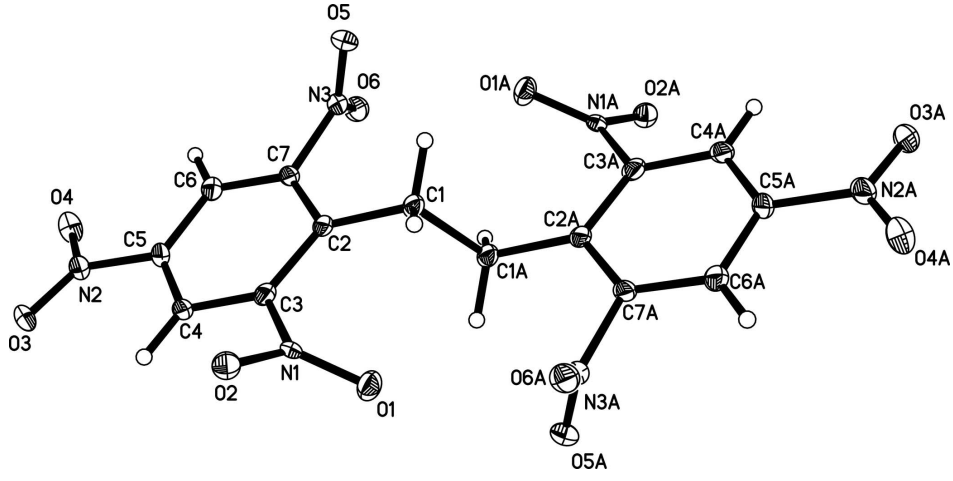

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

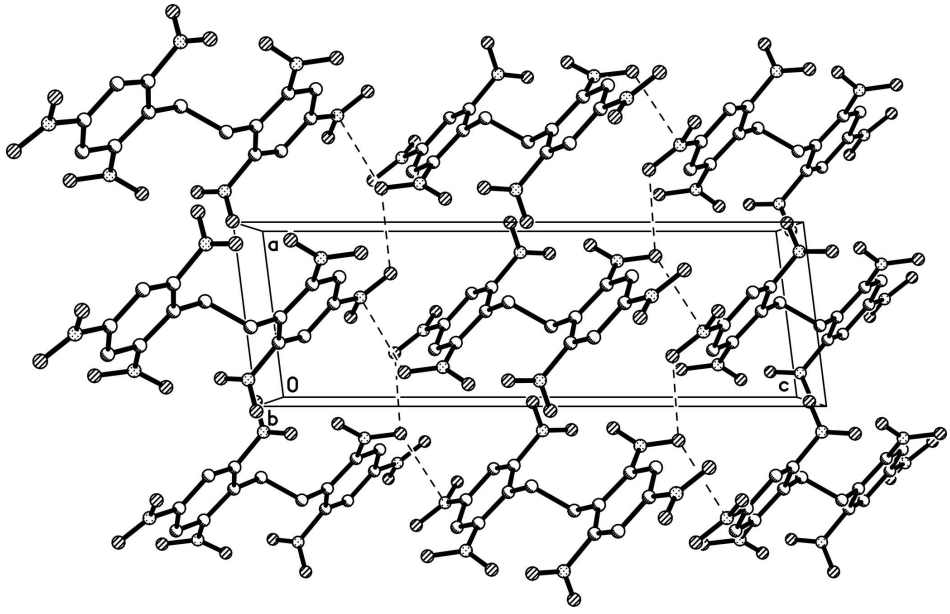

Figure 2

The crystal packing of the title compound.

1,2-Bis(2,4,6-trinitrophenyl)ethane

Crystal data

$C_{14}H_8N_6O_{12}$
$M_r = 452.26$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.8468\ (5)$ Å
$b = 8.1253\ (11)$ Å
$c = 17.977\ (2)$ Å
$\beta = 97.154\ (8)°$
$V = 847.38\ (17)$ Å^3
$Z = 2$
$F(000) = 460$
$D_x = 1.773$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3228 reflections
$\theta = 2.3–27.9°$
$\mu = 0.16$ mm^{-1}
$T = 113$ K
Prism, colourless
0.22 × 0.20 × 0.16 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.22 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2000)
$T_{min} = 0.966$, $T_{max} = 0.975$
7531 measured reflections
2013 independent reflections
1503 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.3°$
$h = -7\rightarrow7$
$k = -8\rightarrow10$
$l = -23\rightarrow23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.099$
$S = 1.04$
2013 reflections
186 parameters
70 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
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.0176P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.025 (5)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.788 (2)	0.4687 (13)	0.1645 (5)	0.0296 (16)	0.513 (3)
O1	0.8771 (5)	0.5628 (3)	0.12216 (13)	0.0302 (6)	0.513 (3)
O2	0.815 (3)	0.489 (2)	0.2328 (6)	0.0260 (15)	0.513 (3)
N1′	0.824 (2)	0.4472 (14)	0.1631 (5)	0.0296 (16)	0.487 (3)
O1′	0.9430 (5)	0.4872 (4)	0.11433 (14)	0.0355 (7)	0.487 (3)
O2′	0.822 (3)	0.503 (2)	0.2263 (7)	0.035 (3)	0.487 (3)
O3	0.72410 (17)	−0.11091 (14)	0.26187 (5)	0.0387 (3)	
O4	0.48405 (19)	−0.23368 (11)	0.17726 (5)	0.0344 (3)	
N3	0.15225 (18)	0.19328 (12)	0.00293 (5)	0.0229 (3)	
O5	−0.0105 (3)	0.2748 (3)	0.01446 (10)	0.0295 (6)	0.513 (3)
O6	0.1555 (3)	0.0949 (3)	−0.04988 (9)	0.0301 (7)	0.513 (3)
O5′	−0.0438 (3)	0.1733 (4)	0.01768 (11)	0.0328 (6)	0.487 (3)
O6′	0.2022 (4)	0.2111 (3)	−0.06168 (10)	0.0345 (7)	0.487 (3)
N2	0.59258 (19)	−0.11271 (15)	0.20272 (6)	0.0299 (3)	
C1	0.4345 (2)	0.49440 (15)	0.03484 (6)	0.0222 (3)	
H1A	0.4797	0.5885	0.0685	0.027*	
H1B	0.2668	0.5029	0.0185	0.027*	
C2	0.4834 (2)	0.33521 (15)	0.07792 (6)	0.0226 (3)	
C3	0.6575 (2)	0.31668 (17)	0.13844 (6)	0.0267 (3)	
C4	0.6978 (2)	0.17462 (17)	0.18056 (6)	0.0271 (3)	
H4	0.8168	0.1692	0.2216	0.033*	
C5	0.5584 (2)	0.04194 (16)	0.16040 (7)	0.0263 (3)	
C6	0.3810 (2)	0.04812 (17)	0.10198 (7)	0.0268 (3)	
H6	0.2850	−0.0445	0.0892	0.032*	
C7	0.3490 (2)	0.19428 (16)	0.06306 (6)	0.0229 (3)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.011 (3)	0.047 (2)	0.0282 (8)	−0.005 (2)	−0.0062 (11)	0.0187 (11)

O1	0.0339 (14)	0.0303 (14)	0.0260 (10)	−0.0121 (10)	0.0020 (9)	0.0048 (10)
O2	0.027 (3)	0.024 (3)	0.026 (2)	−0.0047 (19)	0.001 (2)	−0.002 (3)
N1′	0.011 (3)	0.047 (2)	0.0282 (8)	−0.005 (2)	−0.0062 (11)	0.0187 (11)
O1′	0.0315 (16)	0.0473 (18)	0.0260 (12)	−0.0210 (12)	−0.0031 (10)	0.0114 (12)
O2′	0.028 (3)	0.031 (4)	0.048 (5)	−0.004 (2)	0.010 (3)	0.008 (2)
O3	0.0289 (6)	0.0549 (7)	0.0298 (5)	−0.0031 (5)	−0.0069 (4)	0.0208 (5)
O4	0.0452 (7)	0.0309 (5)	0.0262 (5)	0.0059 (4)	0.0002 (4)	0.0024 (4)
N3	0.0192 (6)	0.0277 (6)	0.0213 (5)	−0.0009 (4)	0.0011 (4)	0.0056 (4)
O5	0.0182 (10)	0.0352 (14)	0.0345 (10)	0.0056 (9)	0.0009 (8)	0.0059 (9)
O6	0.0282 (11)	0.0410 (15)	0.0203 (9)	−0.0031 (9)	0.0001 (7)	−0.0006 (8)
O5′	0.0170 (11)	0.0421 (16)	0.0390 (12)	0.0004 (10)	0.0016 (9)	0.0030 (11)
O6′	0.0379 (13)	0.0474 (16)	0.0169 (9)	−0.0111 (10)	−0.0022 (8)	0.0054 (9)
N2	0.0254 (6)	0.0418 (7)	0.0225 (5)	0.0051 (5)	0.0030 (4)	0.0104 (5)
C1	0.0214 (7)	0.0296 (6)	0.0155 (5)	−0.0056 (5)	0.0013 (5)	0.0023 (5)
C2	0.0172 (6)	0.0361 (7)	0.0151 (5)	−0.0018 (5)	0.0039 (4)	0.0053 (5)
C3	0.0194 (7)	0.0426 (8)	0.0182 (6)	−0.0075 (5)	0.0026 (5)	0.0063 (5)
C4	0.0170 (7)	0.0473 (8)	0.0166 (6)	−0.0013 (5)	0.0003 (5)	0.0089 (5)
C5	0.0235 (7)	0.0370 (7)	0.0186 (6)	0.0023 (6)	0.0033 (5)	0.0108 (5)
C6	0.0235 (7)	0.0347 (7)	0.0220 (6)	−0.0040 (5)	0.0016 (5)	0.0066 (5)
C7	0.0162 (6)	0.0371 (7)	0.0149 (5)	−0.0006 (5)	0.0004 (4)	0.0052 (5)

Geometric parameters (Å, º)

N1—O2	1.231 (8)	N2—C5	1.4697 (16)
N1—O1	1.238 (7)	C1—C2	1.5164 (16)
N1—C3	1.496 (7)	C1—C1^{i}	1.550 (2)
N1′—O2′	1.226 (8)	C1—H1A	0.9900
N1′—O1′	1.228 (8)	C1—H1B	0.9900
N1′—C3	1.471 (7)	C2—C7	1.3957 (17)
O3—N2	1.2323 (13)	C2—C3	1.4024 (16)
O4—N2	1.2271 (15)	C3—C4	1.3846 (18)
N3—O5	1.199 (2)	C4—C5	1.3728 (19)
N3—O5′	1.219 (2)	C4—H4	0.9500
N3—O6′	1.241 (2)	C5—C6	1.3820 (17)
N3—O6	1.243 (2)	C6—C7	1.3792 (18)
N3—C7	1.4772 (14)	C6—H6	0.9500
O2—N1—O1	121.2 (11)	C1^{i}—C1—H1B	109.1
O2—N1—C3	114.9 (9)	H1A—C1—H1B	107.8
O1—N1—C3	123.7 (6)	C7—C2—C3	113.41 (11)
O2′—N1′—O1′	129.5 (12)	C7—C2—C1	122.43 (10)
O2′—N1′—C3	117.6 (10)	C3—C2—C1	124.07 (11)
O1′—N1′—C3	112.9 (6)	C4—C3—C2	124.86 (12)
O5—N3—O5′	41.21 (12)	C4—C3—N1′	112.0 (6)
O5—N3—O6′	112.46 (16)	C2—C3—N1′	123.1 (6)
O5′—N3—O6′	123.81 (15)	C4—C3—N1	118.2 (5)
O5—N3—O6	125.15 (16)	C2—C3—N1	116.5 (5)
O5′—N3—O6	100.65 (16)	N1′—C3—N1	10.7 (11)

O6′—N3—O6	48.11 (12)	C5—C4—C3	117.07 (11)
O5—N3—C7	115.66 (13)	C5—C4—H4	121.5
O5′—N3—C7	120.60 (13)	C3—C4—H4	121.5
O6′—N3—C7	115.58 (13)	C4—C5—C6	122.48 (11)
O6—N3—C7	118.54 (12)	C4—C5—N2	119.78 (11)
O4—N2—O3	124.71 (11)	C6—C5—N2	117.73 (12)
O4—N2—C5	117.46 (10)	C7—C6—C5	117.28 (12)
O3—N2—C5	117.81 (11)	C7—C6—H6	121.4
C2—C1—C1^{i}	112.47 (13)	C5—C6—H6	121.4
C2—C1—H1A	109.1	C6—C7—C2	124.88 (11)
C1^{i}—C1—H1A	109.1	C6—C7—N3	114.24 (11)
C2—C1—H1B	109.1	C2—C7—N3	120.87 (10)
C1^{i}—C1—C2—C7	−90.86 (16)	C3—C4—C5—C6	−1.5 (2)
C1^{i}—C1—C2—C3	92.64 (16)	C3—C4—C5—N2	179.85 (11)
C7—C2—C3—C4	0.13 (18)	O4—N2—C5—C4	−171.13 (12)
C1—C2—C3—C4	176.91 (12)	O3—N2—C5—C4	10.35 (18)
C7—C2—C3—N1′	178.3 (5)	O4—N2—C5—C6	10.15 (18)
C1—C2—C3—N1′	−4.9 (5)	O3—N2—C5—C6	−168.36 (12)
C7—C2—C3—N1	−172.0 (4)	C4—C5—C6—C7	0.7 (2)
C1—C2—C3—N1	4.8 (5)	N2—C5—C6—C7	179.42 (11)
O2′—N1′—C3—C4	−64.8 (15)	C5—C6—C7—C2	0.6 (2)
O1′—N1′—C3—C4	117.4 (9)	C5—C6—C7—N3	−178.30 (11)
O2′—N1′—C3—C2	116.8 (13)	C3—C2—C7—C6	−0.98 (18)
O1′—N1′—C3—C2	−61.0 (13)	C1—C2—C7—C6	−177.82 (12)
O2′—N1′—C3—N1	62 (4)	C3—C2—C7—N3	177.83 (10)
O1′—N1′—C3—N1	−116 (5)	C1—C2—C7—N3	0.99 (18)
O2—N1—C3—C4	−38.7 (14)	O5—N3—C7—C6	109.24 (19)
O1—N1—C3—C4	136.6 (10)	O5′—N3—C7—C6	62.5 (2)
O2—N1—C3—C2	134.0 (11)	O6′—N3—C7—C6	−116.35 (17)
O1—N1—C3—C2	−50.7 (13)	O6—N3—C7—C6	−61.96 (18)
O2—N1—C3—N1′	−96 (4)	O5—N3—C7—C2	−69.7 (2)
O1—N1—C3—N1′	79 (3)	O5′—N3—C7—C2	−116.4 (2)
C2—C3—C4—C5	1.1 (2)	O6′—N3—C7—C2	64.72 (19)
N1′—C3—C4—C5	−177.3 (5)	O6—N3—C7—C2	119.11 (17)
N1—C3—C4—C5	173.0 (5)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O4^{ii}	0.99	2.43	3.3669 (15)	158
C1—H1*B*···O5^{iii}	0.99	2.37	3.147 (2)	134

Symmetry codes: (ii) *x*, *y*+1, *z*; (iii) −*x*, −*y*+1, −*z*.