

**10,10'-Dinitro-10,10'-(butane-1,4-diyl)-
dianthracen-9(10*H*)-one****Mustafa Arslan,^{a*} Erol Asker,^b
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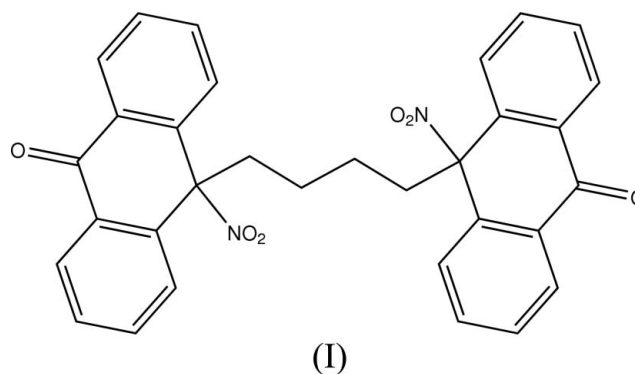
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Key indicatorsSingle-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.042
 wR factor = 0.110
Data-to-parameter ratio = 12.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{32}\text{H}_{24}\text{N}_2\text{O}_6$, was obtained as the decomposition product of (*E,E*)-1,4-bis[9,10-dihydro-9-nitro-10-(trinitromethyl)-9-anthryl]butane, which was synthesized via a photochemical reaction of 1,4-bis(9-anthryl)butane with tetranitromethane. The asymmetric unit contains one half-molecule; the complete molecule is generated by a center of inversion. The crystal packing is determined mainly by intermolecular C—H...O interactions.

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Photonitration of aromatic compounds through the use of tetranitromethane (TNM) offers an alternative route to the conventional nitration processes which require the use of concentrated nitric and sulfuric acids. (Kochi, 1991; Butts *et al.*, 1996; Cox, 1998; Lehnig & Schürmann, 1998). In general, 9-alkyl substituted anthracene compounds lead to addition of a nitro group at the C-atom bearing an alkyl group and trinitromethylation takes place at the sterically less hindered unsubstituted C10 center. Photolysis of the 1, 4-bis(9-anthryl)butane/TNM charge transfer complex led to unstable (*E,E*)-1,4-bis[9,10-dihydro-9-nitro-10-(trinitromethyl)-9-anthryl]butane. When this was passed through a column of basic alumina or silica gel, trinitromethyl groups were eliminated to give the corresponding anthrone derivative. In this paper we report the crystal structure of the title compound, (I), as the decomposition product of this process.



The asymmetric unit contains one half-molecule; the complete molecule is generated by a center of inversion. Bond lengths and angles in the anthracene system (Table 1) are in agreement with those of related compounds (Brinkmann *et al.*, 1970; Rabideau, 1978; Dalling *et al.*, 1981; Arslan *et al.*, 2005).

The fourteen atoms of the anthracene skeleton in (I) (Fig. 1) have a total puckering amplitude $Q = 0.167$ (2) Å (Cremer & Pople, 1975). The methylene chain connecting the two rings

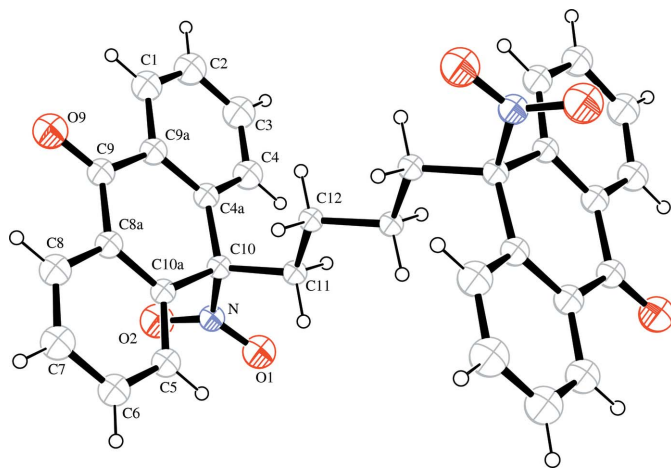


Figure 1
ORTEP drawing of (I) with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 35% probability level. Unlabelled atoms are related to labelled atoms by $(-x, -y, 1 - z)$.

exhibits an *anti-anti-anti* conformation. The O1–N–C10–C11 and O2–N–C10–C11 torsion angles (Table 1) indicate that the O atoms of the nitro groups lie in the same plane as the methylene chain.

Examination of the packing diagram (Fig. 2) reveals that the molecular packing is mainly determined by intermolecular C–H \cdots O interactions (Table 2).

Experimental

The title compound was obtained as the decomposition product of (*E,E*)-1,4-bis[9,10-dihydro-9-nitro-10(trinitromethyl)-9-anthryl]butane, which was synthesized by irradiation of a solution containing 21 mg (0.049 mmol) of 1,4-bis(9-anthryl)butane, 325 mg (1.67 mmol) of TNM, 45 ml pentane, and 5 ml CCl₄ under the conditions described by Arslan *et al.*, (2005). (*E,E*)-1,4-bis[9,10-dihydro-9-nitro-10(trinitromethyl)-9-anthryl]butane was obtained as yellow needles after removal of the solvents under reduced pressure and washing with acetone to remove the unreacted starting compound. The remaining yellow solid was column chromatographed using alumina (80–200 mesh, activity III) as the carrier and dichloromethane/hexane (1:4 *v/v*) as the eluent to yield the title compound (16.1% yield, m.p. 464–465 K, dichloromethane). Pale-yellow single crystals suitable for the X-ray diffraction study were grown from a concentrated solution of (I) in dichloromethane through slow evaporation under ambient conditions. ¹H-NMR (300 MHz, CDCl₃, p.p.m.): δ 8.33 [4 H, *d*, 2 \times (H1, H8)], 7.63 [8 H, *m*, 2 \times (H2, H3, H6, H7)], 7.34 [4 H, *d*, 2 \times (H4, H5)], 2.55 [4 H, *t*, 2 \times (H11a, H11b)], 0.38 [4 H, *m*, 2 \times (H12a, H12b)]

Crystal data

C₃₂H₂₄N₂O₆
 $M_r = 532.53$
 Monoclinic, $P2_1/c$
 $a = 11.316$ (1) Å
 $b = 8.330$ (1) Å
 $c = 13.880$ (2) Å
 $\beta = 97.728$ (9)°
 $V = 1296.6$ (2) Å³

$Z = 2$
 $D_x = 1.364$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 295$ (2) K
 Cube, pale yellow
 $0.34 \times 0.34 \times 0.34$ mm

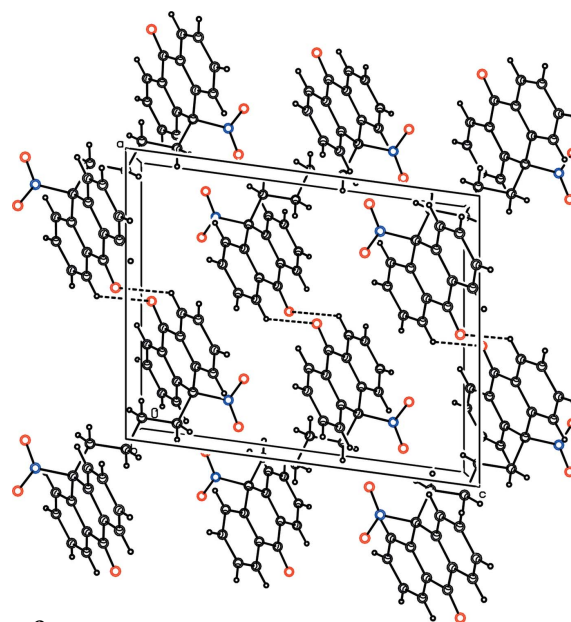


Figure 2
The crystal packing of (I), viewed down the *b* axis. Dashed lines indicate C–H \cdots O interactions.

Data collection

Enraf-Nonius CAD-4
 diffractometer
 ω scans
 Absorption correction: none
 2296 measured reflections
 2296 independent reflections

1638 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 25.0^\circ$
 3 standard reflections
 frequency: 120 min
 intensity decay: 0.5%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.110$
 $S = 1.01$
 2296 reflections
 181 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.4238P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.006$
 $\Delta\rho_{\max} = 0.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.14$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1–N	1.199 (2)	O9–C9	1.224 (2)
O2–N	1.207 (2)	N–C10	1.562 (2)
O1–N–O2	123.27 (17)	C4A–C10–C10A	115.12 (15)
O1–N–C10	119.15 (16)	C4A–C10–N	105.39 (14)
O2–N–C10	117.58 (15)	C10A–C10–N	105.02 (14)
O1–N–C10–C11	1.4 (2)	O2–N–C10–C11	–178.02 (18)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H \cdots <i>A</i>	<i>D</i> –H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> –H \cdots <i>A</i>
C1–H1 \cdots O9 ⁱ	0.93	2.64	3.118 (3)	112

Symmetry code: (i) $-x + 1, -y, -z + 1$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of 0.93 and 0.97 Å for aromatic and methylene H atoms, respectively, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *CAD-4-PC* Software (Enraf-Nonius, 1993); cell refinement: *CAD-4-PC* Software; data reduction: *DATRD2* in *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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