10,10'-Dinitro-10,10'-(butane-1,4-diyl)dianthracen-9(10H)-one

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The title compound, C$_{32}$H$_{24}$N$_2$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 tetrinitromethane. 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.

Comment

Photonitration of aromatic compounds through the use of tetrinitromethane (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.
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···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) 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.): 8.33 [4 H, d, 2 (H1, H8)], 7.63 [8 H, m, 2 (H2, H3, H6, H7)], 7.34 [4 H, d, 2 (H4, H5)], 2.55 [4 H, t, 2 x (H11a, H11b)], 0.38 [4 H, m, 2 x (H12a, H12b)]

**Crystal data**

C₃₂H₂₄N₂O₆

Mᵣ = 532.53

Monoclinic, P2₁/c

a = 11.316 (1) Å

b = 8.330 (1) Å

c = 13.880 (2) Å

β = 97.728 (9)°

V = 1296.6 (2) Å³

Z = 2

Dᵣ = 1.364 Mg m⁻³

Mo Kα radiation

µ = 0.10 mm⁻¹

T = 298 (2) K

Cubic, pale yellow

0.34 × 0.34 × 0.34 mm

Data collection

Enraf-Nonius CAD-4 diffractometer

ω scans

Absorption correction: none

2296 measured reflections

2296 independent reflections

1638 reflections with I > 2σ(I)

θmax = 25.0°

3 standard reflections

frequency: 120 min

intensity decay: 0.5%

Refinement

Refinement on F²

R[F² > 2σ(F²)] = 0.042

wR(F²) = 0.110

S = 1.01

2296 reflections

181 parameters

H-atom parameters constrained

w = 1/[σ²(Fo)² + (0.0433P)² + 0.4238P]

where P = (Fo² + 2Fc²)/3

(Δ/σ)max = 0.006

Δρmax = 0.19 e Å⁻³

Δρmin = −0.14 e Å⁻³

Table 1

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Table 2

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

**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).

**Figure 2**

The crystal packing of (I), viewed down the b axis. Dashed lines indicate C—H···O interactions.
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 $U_{iso}(H) = 1.2U_{eq}(C)$.

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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References


