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10,10'-Dinitro-10,10'-(propane-1,3-diyl)di-10H-anthracen-9-one

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Key indicators

Single-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{C}-\text{C}) = 0.008$ Å
 R factor = 0.076
 wR factor = 0.214
Data-to-parameter ratio = 12.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

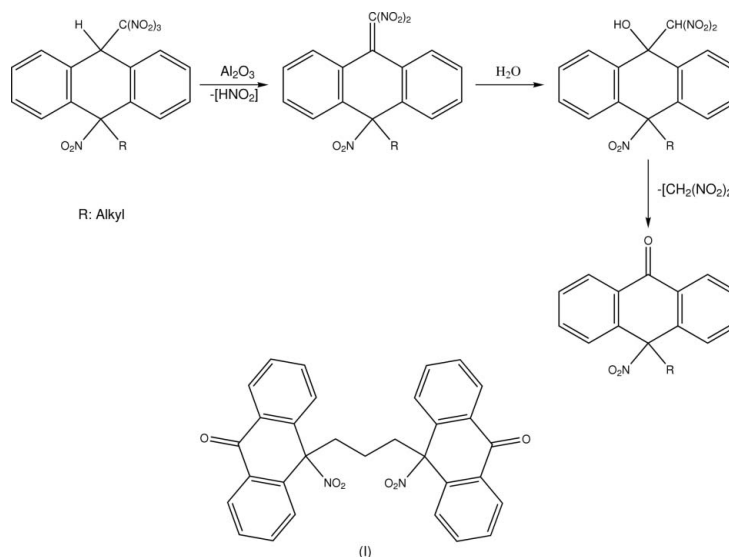
The title compound, $\text{C}_{31}\text{H}_{22}\text{N}_2\text{O}_6$, was obtained as the decomposition product of (*E,E*)-1,3-bis[9,10-dihydro-9-nitro-10-(trinitromethyl)-9-anthryl]propane, which was synthesized via a photochemical reaction of 1,3-di-9-anthrylpropane with tetranitromethane. Intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions are the most prominent features of the crystal packing; no indications of any intermolecular $\pi-\pi$ stacking were found.

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Comment

Photonitration of various aromatic compounds using tetranitromethane (TNM) has attracted some attention as an alternative 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, addition of TNM to 9-alkyl-substituted anthracenes proceeds at the 9- and 10-positions, with the nitro group bonding to atom C9, bearing the alkyl group, and the trinitromethyl group being attached to the sterically less hindered unsubstituted C10 center. We have already reported the structure of the product of such a process, (*E*)-9,10-dihydro-9-methyl-9-nitro-10-(trinitromethyl)anthracene (Arslan *et al.*, 2005). One interesting feature of these trinitromethyl-substituted anthracene derivatives is that they contain the highly labile $\text{C}-\text{C}(\text{NO}_2)_3$ bond and, therefore, when passed through silica gel or an alumina column, easily decompose to form the corresponding anthrone derivatives. The decomposition process is believed to involve retro-aldol reaction.



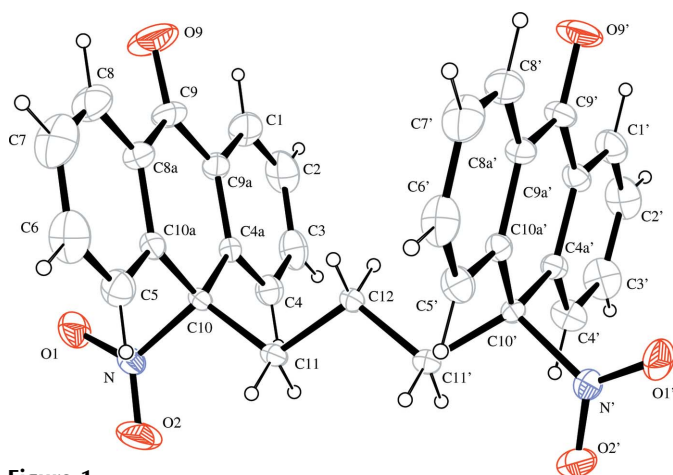


Figure 1
ORTEP (Farrugia, 1997) drawing of (I), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 20% probability level. The H atoms are shown as a small circles of arbitrary radii.

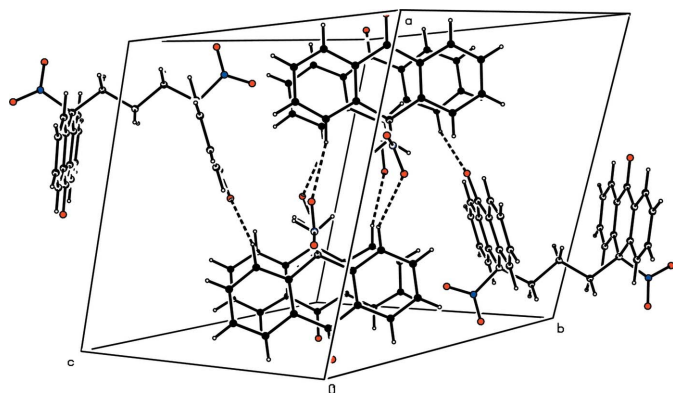


Figure 2
The crystal packing of (I), viewed along the diagonal of the *bc* plane. Dashed lines indicate the C—H...O interactions.

In this paper we report the crystal structure of the title compound, (I), the decomposition product of (*E,E*)-1,3-bis[9,10-dihydro-9-nitro-10-(trinitromethyl)-9-anthryl]propane. The bond lengths and angles in the two anthracene ring systems (Table 1) are in agreement with each other, as well as with those of related compounds (Brinkmann *et al.*, 1970; Rabideau, 1978; Dalling *et al.*, 1981; Arslan *et al.*, 2005).

The 14 atoms of each anthracene system in (I) (Fig. 1) are coplanar to within 0.062 and 0.035 Å for the unprimed and primed ring systems, respectively. The trimethylene chain exhibits an *anti-anti* conformation. The dihedral angle formed by the anthracene planes is 22.51 (9)°.

Examination of the packing diagram (Fig. 2) reveals that the crystal packing is mainly determined by intermolecular C—H...O interactions; there are no indications of intermolecular π - π stacking in the crystal structure of (I).

Experimental

The title compound was obtained as the decomposition product of (*E,E*)-1,3-bis[9,10-dihydro-9-nitro-10-(trinitromethyl)-9-anthryl]-

propane, which was synthesized by irradiation with visual light of a solution containing 20 mg (0.050 mmol) of 1,3-bis(9-anthryl)propane, 325 mg (1.67 mmol) of TNM, 45 ml of pentane, and 5 ml of CCl₄ as described by Arslan *et al.* (2005). A 450 W Hanovia medium-pressure mercury lamp with a 500 nm sharp cut-off filter was used as a light source. The (*E,E*)-1,3-bis[9,10-dihydro-9-nitro-10-(trinitromethyl)-9-anthryl]propane was column chromatographed using alumina (80–200 mesh, activity III) as the carrier and dichloromethane–hexane as eluant to give (I) (38.9% yield, m.p. 464–465 K). Single crystals suitable for X-ray diffraction study were grown from a concentrated solution of (I) in dichloromethane through slow evaporation under ambient conditions.

Crystal data

C₃₁H₂₂N₂O₆
M_r = 518.51
 Monoclinic, *P*₂₁/*n*
a = 13.438 (2) Å
b = 14.490 (3) Å
c = 13.974 (3) Å
 β = 109.505 (5)°
V = 2564.8 (9) Å³

Z = 4
D_x = 1.343 Mg m⁻³
 Mo *K* α radiation
 μ = 0.09 mm⁻¹
T = 295 (2) K
 Block, yellow
 0.32 × 0.29 × 0.13 mm

Data collection

Enraf–Nonius CAD-4
 diffractometer
 ω scans
 Absorption correction: none
 4554 measured reflections
 4554 independent reflections

1912 reflections with *I* > 2 σ (*I*)
 θ_{\max} = 25.1°
 3 standard reflections
 every 120 min
 intensity decay: 1.1%

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.076
wR (*F*²) = 0.214
S = 0.99
 4554 reflections
 352 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0908P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1—N	1.187 (5)	O2'—N'	1.210 (5)
O2—N	1.175 (5)	O9'—C9'	1.221 (5)
O9—C9	1.228 (5)	N—C10	1.554 (5)
O1'—N'	1.185 (4)	N'—C10'	1.558 (5)
O1—N—O2	122.4 (4)	C4A—C10—N	105.5 (3)
O1—N—C10	117.6 (4)	C10A—C10—N	105.0 (3)
O2—N—C10	120.0 (4)	C11—C10—N	107.6 (3)
O1'—N'—O2'	123.4 (4)	C11'—C10'—C10A'	111.6 (4)
O1'—N'—C10'	118.7 (4)	C11'—C10'—C4A'	111.9 (3)
O2'—N'—C10'	117.6 (4)	C10A'—C10'—C4A'	115.5 (3)
C4A—C10—C10A	115.1 (3)	C11'—C10'—N'	107.9 (3)
C4A—C10—C11	111.2 (4)	C10A'—C10'—N'	105.2 (3)
C10A—C10—C11	111.7 (4)	C4A'—C10'—N'	104.0 (3)
O2—N—C10—C10A	−116.4 (6)	O1'—N'—C10'—C11'	−164.3 (4)
O1—N—C10—C11	−175.4 (4)	O2'—N'—C10'—C11'	21.6 (5)
N—C10—C11—C12	178.1 (4)	C11—C12—C11'—C10'	178.4 (4)
C10—C11—C12—C11'	179.1 (4)	N'—C10'—C11'—C12	178.5 (4)

Table 2
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C2'-H2'\cdots O2^i$	0.93	2.67	3.586 (10)	168
$C4-H4\cdots O9^{ii}$	0.93	2.60	3.417 (6)	147
$C5-H5\cdots O2^{iii}$	0.93	2.69	3.363 (7)	130
$C5'-H5'\cdots O2^{iii}$	0.93	2.53	3.343 (7)	146
$C7'-H7'\cdots O2^{iv}$	0.93	2.57	3.473 (7)	165

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$; (ii) $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$; (iii) $-x + 1, -y + 1, -z + 3$; (iv) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$

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

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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