5-15-2006

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 |C–C| = 0.008 Å
R factor = 0.076
wR factor = 0.214
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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; Lehning & 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 C—C(NO2)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.

The title compound, C31H22N2O6, 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 tetrinitromethane. Intermolecular C—H · · · O interactions are the most prominent features of the crystal packing; no indications of any intermolecular π–π stacking were found.

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propane, which was synthesized by irradiation with visible 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

\[
\begin{align*}
\text{C}_{31}H_{22}N_{2}O_{6} & \quad Z = 4 \\
M_r & = 518.51 \\
\text{Monoclinic, } P2_1/\text{n} & \\
\alpha & = 13.438 (2) \text{ Å} \\
b & = 14.490 (3) \text{ Å} \\
c & = 13.974 (3) \text{ Å} \\
\beta & = 109.505 (5)^\circ \\
V & = 5268.4 (9) \text{ Å}^3
\end{align*}
\]

Data collection

Enraf–Nonius CAD-4 diffractometer

\(\omega\) scans

Absorption correction: none

4554 measured reflections

4554 independent reflections

Refinement

Reinson on \(F^2\)

\(R[F^2 > 2\sigma(F^2)] = 0.076\)

\(wR(F^2) = 0.214\)

\(S = 0.99\)

4554 reflections

352 parameters

H-atom parameters constrained

\(w = 1/[\sigma^2(F^2) + (0.0908P)^2]\)

where \(P = (F^2 + 2F_c^2)/3\)

\((\Delta/\sigma)_{\text{max}} < 0.001\)

\(\Delta p_{\text{max}} = 0.20 e \text{ Å}^{-3}\)

\(\Delta p_{\text{min}} = -0.20 e \text{ Å}^{-3}\)

Table 1

<table>
<thead>
<tr>
<th>O1—N</th>
<th>1.187 (5)</th>
<th>O2—N’</th>
<th>1.210 (5)</th>
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<tbody>
<tr>
<td>O2—N</td>
<td>1.175 (5)</td>
<td>O9—C9</td>
<td>1.228 (5)</td>
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<tr>
<td>O2—N’</td>
<td>1.185 (4)</td>
<td>N—C10</td>
<td>1.554 (5)</td>
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<tr>
<td>O2—N’</td>
<td>1.185 (4)</td>
<td>N’—C10′</td>
<td>1.558 (5)</td>
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<tr>
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<td>122.4 (4)</td>
<td>C4A—C10—N</td>
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<tr>
<td>O1—N—C10</td>
<td>117.6 (4)</td>
<td>C10A—C10—N</td>
<td>105.0 (3)</td>
</tr>
<tr>
<td>O1—N’—C10’</td>
<td>115.3 (3)</td>
<td>C11—C10′—C10A’</td>
<td>107.6 (3)</td>
</tr>
<tr>
<td>O1’—N’—O2’</td>
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<td>C4A′—C10—N’</td>
<td>111.4 (4)</td>
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<tr>
<td>O2’—N—C10’</td>
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<td>C11′—C10′—C4A′</td>
<td>111.9 (3)</td>
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<tr>
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<td>C10A’—C10′—C4A′</td>
<td>115.3 (3)</td>
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<tr>
<td>O2’—N—C10A</td>
<td>115.3 (3)</td>
<td>C11—C10—C10A</td>
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<td>115.3 (3)</td>
<td>C10A—C10′—C11′</td>
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<td>O2—N—C10—C10A</td>
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<td>−164.3 (4)</td>
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<tr>
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<td>−175.4 (4)</td>
<td>O2’—N—C10—C11’</td>
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<td>C10—C11—C12—C11’</td>
<td>179.3 (4)</td>
<td>N’—C10′—C11—C12</td>
<td>178.5 (4)</td>
</tr>
</tbody>
</table>

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

<table>
<thead>
<tr>
<th>D—H—A</th>
<th>D—H</th>
<th>H—A</th>
<th>D—A</th>
<th>D—H—A</th>
</tr>
</thead>
<tbody>
<tr>
<td>C2′—H2′⋯O2′</td>
<td>0.93</td>
<td>2.67</td>
<td>3.586(10)</td>
<td>168</td>
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<tr>
<td>C4—H4⋯O9ii</td>
<td>0.93</td>
<td>2.60</td>
<td>3.417(6)</td>
<td>147</td>
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<td>C5—H5⋯O2iii</td>
<td>0.93</td>
<td>2.69</td>
<td>3.363(7)</td>
<td>130</td>
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<tr>
<td>C7—H7⋯O2iv</td>
<td>0.93</td>
<td>2.57</td>
<td>3.473(7)</td>
<td>165</td>
</tr>
</tbody>
</table>

Symmetry codes: (i) x + 1/2, y + 1/2, z — 1; (ii) x — 1/4, y + 1/2, z — 1/4; (iii) —x + 1, —y + 1, —z + 3; (iv) x + 1/4, —y + 1/2, z + 1/4.

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 Uiso(H) = 1.2 Ueq(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).

The authors thank the Turkish Ministry of Education and the CSU College of Graduate Studies for their support of this work.

References