*Acta Cryst.* (1987). C**43**, 1840–1841

# Structure of 5-Nitro-13*H*-dibenz[*a*,*de*]anthracen-13-one

BY SHOJI FUJISAWA, ISAO OONISHI AND JUNJI AOKI

*Department of Chemistry, Faculty of Science, Toho University, Funabashi, Chiba* 274, *Japan*

AND YUJI OHASHI

*Department of Chemistry, Faculty of Science, Ochanomizu University, Otsuka* 2–1–1, *Bunkyo-ku, Tokyo* 112, *Japan*

(*Received* 28 *March* 1987; *accepted* 29 *April* 1987)

**Abstract.** $C_{21}H_{11}NO_3$, $M_r = 325{\cdot}3$, monoclinic, $P2_1/c$, $a = 8{\cdot}727\ (2)$, $b = 22{\cdot}441\ (6)$, $c = 7{\cdot}295\ (2)$ Å, $\beta = 91{\cdot}98\ (2)°$, $V = 1427{\cdot}7\ (6)$ Å$^3$, $Z = 4$, $D_m = 1{\cdot}50$, $D_x = 1{\cdot}513$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}10$ mm$^{-1}$, $F(000) = 672$, $T = 298$ K. Final $R = 0{\cdot}059$ for 1831 independent observed reflections. The title compound was synthesized by nitration of 13*H*-dibenz[*a*,*de*]anthracen-13-one at 288 K. It is confirmed that the $NO_2$ group was introduced to the 5 position of the above ketone. The mother skeleton is planar within −0·122 (3)–0·083 (3) Å. The angle of the $NO_2$ group to the mean plane of the mother skeleton is 92·5 (1)°.

**Experimental.** The details of the synthesis will be reported elsewhere.

Reddish brown needle-like crystals from chlorobenzene solution; $D_m$ by flotation in $ZnCl_2$ solution; systematic absences: $h0l$, $l = 2n+1$, $0k0$, $k = 2n+1$; crystal dimensions 0·40 × 0·40 × 0·20 mm; Rigaku AFC-6 diffractometer; graphite monochromator; cell parameters refined by least-squares method on the basis of 25 independent $2\theta$ values, $18 < 2\theta < 25°$; intensity measurement performed up to $2\theta = 55°$; range of $hkl$ −11 to 11, 0 to 29 and 0 to 9; $\omega$–$2\theta$ scan, scan speed 4° min$^{-1}$ ($2\theta$), scan width $(1{\cdot}21 + 0{\cdot}5\tan\theta)°$; background 5 s before and after each scan; three standard reflections monitored every 100 reflections, no significant variation in intensities; 3415 reflections measured,

Table 1. *Final atomic coordinates* (× 10$^4$) *and equivalent isotropic thermal parameters* (Å$^2$ × 10)

$B_{eq} = \frac{4}{3}\sum_i\sum_j B_{ij}\mathbf{a}_i.\mathbf{a}_j$. The $B_{ij}$'s are defined by: $\exp[-(h^2B_{11} + k^2B_{22} + l^2B_{33} + 2klB_{23} + 2hlB_{13} + 2hkB_{12})]$.

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| C(1) | 4355 (3) | 3233 (1) | 8223 (4) | 33 (1) |
| C(2) | 3124 (3) | 2866 (1) | 8659 (4) | 36 (1) |
| C(3) | 3248 (3) | 2270 (1) | 8527 (4) | 34 (1) |
| C(4) | 4822 (3) | 1402 (1) | 7786 (4) | 32 (1) |
| C(5) | 6155 (3) | 1113 (1) | 7285 (4) | 33 (1) |
| C(6) | 7460 (3) | 1479 (1) | 6959 (3) | 29 (1) |
| C(7) | 8617 (3) | 2509 (1) | 6636 (4) | 36 (1) |
| C(8) | 9606 (3) | 3523 (1) | 6255 (4) | 39 (1) |
| C(9) | 9442 (4) | 4128 (1) | 6234 (4) | 46 (1) |
| C(10) | 8061 (4) | 4379 (1) | 6668 (5) | 47 (1) |
| C(11) | 6848 (3) | 4022 (1) | 7141 (4) | 41 (1) |
| C(12) | 6990 (3) | 3402 (1) | 7175 (3) | 29 (1) |
| C(13) | 5732 (3) | 3009 (1) | 7667 (3) | 27 (1) |
| C(14) | 5931 (3) | 2374 (1) | 7564 (3) | 26 (1) |
| C(15) | 4643 (3) | 2007 (1) | 7979 (3) | 29 (1) |
| C(16) | 7326 (3) | 2108 (1) | 7069 (3) | 27 (1) |
| C(17) | 8386 (3) | 3155 (1) | 6711 (3) | 30 (1) |
| C(18) | 6245 (4) | 483 (1) | 7132 (4) | 44 (1) |
| C(19) | 7573 (4) | 221 (1) | 6690 (5) | 50 (1) |
| C(20) | 8872 (4) | 572 (1) | 6407 (4) | 47 (1) |
| C(21) | 8824 (3) | 1169 (1) | 6518 (4) | 37 (1) |
| N | 3479 (3) | 1025 (1) | 8115 (4) | 43 (1) |
| O(1) | 9853 (3) | 2320 (1) | 6158 (5) | 77 (1) |
| O(2) | 2669 (3) | 893 (1) | 6772 (3) | 63 (1) |
| O(3) | 3245 (3) | 866 (1) | 9664 (3) | 67 (1) |

Table 2. *Bond lengths* (Å) *and angles* (°) *with their e.s.d.'s*

| | | | |
|---|---|---|---|
| C(1)–C(2) | 1·399 (4) | C(8)–C(17) | 1·397 (4) |
| C(1)–C(13) | 1·378 (4) | C(9)–C(10) | 1·377 (5) |
| C(2)–C(3) | 1·346 (4) | C(10)–C(11) | 1·380 (5) |
| C(3)–C(15) | 1·423 (4) | C(11)–C(12) | 1·396 (4) |
| C(4)–C(5) | 1·391 (4) | C(12)–C(13) | 1·463 (4) |
| C(4)–C(15) | 1·375 (4) | C(12)–C(17) | 1·391 (4) |
| C(4)–N | 1·471 (4) | C(13)–C(14) | 1·438 (4) |
| C(5)–C(6) | 1·431 (4) | C(14)–C(15) | 1·434 (4) |
| C(5)–C(18) | 1·420 (4) | C(18)–C(19) | 1·349 (5) |
| C(6)–C(16) | 1·418 (4) | C(19)–C(20) | 1·401 (5) |
| C(7)–C(16) | 1·484 (4) | C(20)–C(21) | 1·344 (4) |
| C(7)–C(17) | 1·464 (4) | N–O(2) | 1·224 (4) |
| C(7)–O(1) | 1·221 (4) | N–O(3) | 1·209 (4) |
| C(8)–C(9) | 1·365 (4) | | |

| | | | |
|---|---|---|---|
| C(2)–C(1)–C(13) | 122·4 (3) | C(1)–C(13)–C(12) | 121·4 (3) |
| C(1)–C(2)–C(3) | 120·3 (3) | C(1)–C(13)–C(14) | 119·1 (3) |
| C(2)–C(3)–C(15) | 120·3 (3) | C(12)–C(13)–C(14) | 119·5 (3) |
| C(5)–C(4)–C(15) | 125·9 (3) | C(13)–C(14)–C(15) | 117·5 (3) |
| C(5)–C(4)–N | 117·0 (3) | C(13)–C(14)–C(16) | 122·5 (3) |
| C(15)–C(4)–N | 117·1 (3) | C(15)–C(14)–C(16) | 120·0 (3) |
| C(4)–C(5)–C(6) | 117·0 (3) | C(3)–C(15)–C(4) | 122·7 (3) |
| C(4)–C(5)–C(18) | 122·2 (3) | C(3)–C(15)–C(14) | 120·4 (3) |
| C(6)–C(5)–C(18) | 120·8 (3) | C(4)–C(15)–C(14) | 116·9 (3) |
| C(5)–C(6)–C(16) | 119·7 (3) | C(6)–C(16)–C(7) | 121·8 (3) |
| C(5)–C(6)–C(21) | 115·6 (3) | C(6)–C(16)–C(14) | 120·5 (3) |
| C(16)–C(6)–C(21) | 124·7 (3) | C(7)–C(16)–C(14) | 117·7 (3) |
| C(16)–C(7)–C(17) | 119·0 (3) | C(7)–C(17)–C(8) | 118·0 (3) |
| C(16)–C(7)–O(1) | 122·3 (3) | C(7)–C(17)–C(12) | 121·8 (3) |
| C(17)–C(7)–O(1) | 118·6 (3) | C(8)–C(17)–C(12) | 120·2 (3) |
| C(9)–C(8)–C(17) | 120·7 (3) | C(5)–C(18)–C(19) | 120·2 (3) |
| C(8)–C(9)–C(10) | 119·7 (3) | C(18)–C(19)–C(20) | 119·7 (3) |
| C(9)–C(10)–C(11) | 120·4 (3) | C(19)–C(20)–C(21) | 121·7 (3) |
| C(10)–C(11)–C(12) | 120·9 (3) | C(6)–C(21)–C(20) | 121·9 (3) |
| C(11)–C(12)–C(13) | 122·6 (3) | C(4)–N–O(2) | 116·7 (3) |
| C(11)–C(12)–C(17) | 118·1 (3) | C(4)–N–O(3) | 118·9 (3) |
| C(13)–C(12)–C(17) | 119·3 (3) | O(2)–N–O(3) | 124·3 (3) |

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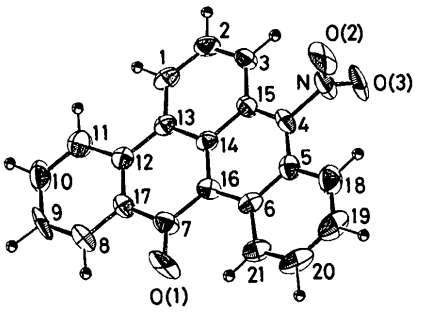


Fig. 1. Perspective drawing of the molecule. 50% probability ellipsoids are shown.

1831 with $|F_o| > 3\sigma(|F_o|)$ considered observed and used for structure determination; corrections for Lorentz and polarization, absorption ignored; direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and subsequent difference calculation; block-diagonal least squares (*HBLS*; Ohashi, 1975) with anisotropic thermal parameters for all non-H atoms; H atoms derived geometrically (C–H 1·08 Å) and refined isotropically; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + (0{\cdot}03F)^2]^{-1}$; max. $(\Delta/\sigma)$ 0·15; final $R = 0{\cdot}059$ and $wR = 0{\cdot}066$; $S = 5{\cdot}6$; $\Delta\rho$ excursions in final difference map 0·3 e Å$^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); calculations carried out on the HITAC M-680 computer at the Computer Center of the University of Tokyo. The final atomic parameters for non-H atoms are in Table 1.* A perspective drawing of the molecule with the numbering scheme is shown in Fig. 1. The bond lengths and angles are listed in Table 2.

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, positional and thermal parameters for H atoms and equations of mean planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44014 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1987). C**43**, 1841–1843

# Structure of Methyl 3,4-Epoxy-5,5-ethylenedioxy-*exo*-7,*endo*-11-dihydroxytricyclo[7.2.1.0$^{4,10}$]dodecane-8-carboxylate

BY A. L. SPEK* AND B. P. VAN EIJCK

*Vakgroep Algemene Chemie, afdeling Kristal- en Structuurchemie, University of Utrecht, Padualaan* 8, 3584 *CH Utrecht, The Netherlands*

(*Received* 4 *March* 1987; *accepted* 10 *April* 1987)

**Abstract.** $C_{16}H_{22}O_7$, $M_r = 326{\cdot}35$, monoclinic, $P2_1/n$, $a = 6{\cdot}520$ (1), $b = 15{\cdot}119$ (4), $c = 15{\cdot}584$ (5) Å, $\beta = 96{\cdot}82$ (2)°, $U = 1525{\cdot}3$ (7) Å$^3$, $Z = 4$, $D_x = 1{\cdot}421$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}0$ cm$^{-1}$, $F(000) = 696$, $T = 295$ K, $R = 0{\cdot}044$ for 2064 reflections with $I \geq 2{\cdot}5\sigma(I)$. The structure contains one intramolecular and one intermolecular H bond. Hydrogen bonding links the molecules into infinite chains that run in the **b** direction.

**Experimental.** X-ray data for a plate-shaped transparent colourless crystal (0·075 × 0·50 × 0·50 mm), glued on top of a glass fibre, were collected on an Enraf–Nonius CAD-4*F* diffractometer using Zr-filtered Mo $K\alpha$ radiation. Lattice parameters and their estimated standard deviations were derived from the setting angles of 20 reflections ($10 < \theta < 12°$). The space group was determined from the observed systematic absences. A total of 3745 unique reflections [$\theta < 27{\cdot}5°$; $\omega/2\theta$ scan; $\Delta\omega = 0{\cdot}70 + 0{\cdot}35\tan(\theta)°$; $-8 \leq h \leq 8$, $0 \leq k \leq 19$, $0 \leq l \leq 20$] were scanned. Two reference reflections ($\bar{2}08$, $1\bar{6}\bar{2}$) showed no decay during the 67 h of X-ray exposure time. The intensities were corrected for Lp but not for absorption. Variance $\sigma^2(I)$ calculated based on counting statistics plus a term $(PI)^2$, where $P$ (=0·012) is the instability constant as

* Author to whom correspondence should be addressed.

0108-2701/87/091841-03$01.50