

Fig. 2. Stereoviews of the molecule. Note the possible positions (a) for atoms C(16) and C(17), and (b) for C(16') and C(17').

The cyclooctanone ring appears to be rather flexible. The C atoms C(16) and C(17) are found in two possible positions with occupation factors for C(16)

and C(17) 0.56, and for C(16') and C(17') 0.44. It should be pointed out that both positions of the mentioned atoms define a twist-*sofa* conformation. This conformational freedom has a low-energy transformation and does not lead to any significant differences in chemical properties of the compound.

All the bond lengths and angles are in good agreement with expected ones. The interaction between neighbouring molecules is of the van der Waals type.

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### Structure of 6,8-Dinitro-13*H*-dibenz[*a,de*]anthracen-13-one

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**Abstract.**  $C_{21}H_{10}N_2O_5$ ,  $M_r = 370.3$ , monoclinic,  $P2_1/n$ ,  $a = 22.511(3)$ ,  $b = 7.316(2)$ ,  $c = 9.688(2)$  Å,  $\beta = 99.39(2)^\circ$ ,  $V = 1574.1(6)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.56$ ,  $D_x = 1.562$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54184$  Å,  $\mu = 9.90$  cm<sup>-1</sup>,  $F(000) = 760$ ,  $T = 298$  K. Final  $R = 0.060$  for 1362 independent reflections. The molecule is slightly distorted from a planar structure. The angles of the two NO<sub>2</sub> groups to the mean molecular plane are  $6.1(3)$  and  $73.8(2)^\circ$ . The molecules are stacked face-to-face to make a column structure along the  $b$  axis. The distance between the mean molecular planes is  $3.396(4)$  Å.

**Introduction.** It is of great interest to study the nitration of ketones of condensed polycyclic aromatic com-

pounds, because much uncertainty exists regarding the positions of the substitution. Treatment of 13*H*-dibenz[*a,de*]anthracen-13-one (5,6-BzBT) under a variety of experimental conditions yields mixtures of three nitro compounds: mono-, di- and tri-substituted derivatives. Fortunately, we succeeded in isolating a dinitro derivative. Since it was difficult to assign the positions of the nitro groups by chemical procedures, the crystal structure analysis of the title compound was carried out. The analysed structure was compared with that of the original compound (5,6-BzBT) (Oonishi, Fujisawa, Aoki, Ohashi & Sasada, 1984).

**Experimental.** Orange plate-like crystals from chlorobenzene solution;  $D_m$  by flotation in ZnCl<sub>2</sub> solution;

Table 1. Final atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters,  $B_{eq}(\text{\AA}^2) (\times 10)$ 

$$B_{eq} = \frac{1}{3} \sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$B_{eq}$
C(1)	5318 (2)	3620 (5)	7637 (4)	47 (1)
C(2)	5932 (2)	3592 (6)	8151 (4)	47 (1)
C(3)	6354 (2)	3013 (5)	7432 (4)	45 (1)
C(4)	6565 (2)	1821 (6)	5204 (4)	45 (1)
C(5)	6417 (2)	1253 (5)	3809 (4)	49 (1)
C(6)	5790 (2)	1287 (5)	3212 (4)	46 (1)
C(7)	4705 (2)	1939 (6)	3463 (4)	48 (1)
C(8)	3661 (2)	2608 (6)	3806 (4)	52 (1)
C(9)	3246 (2)	3174 (6)	4603 (4)	56 (1)
C(10)	3443 (2)	3713 (6)	5972 (4)	58 (1)
C(11)	4038 (2)	3673 (6)	6522 (4)	51 (1)
C(12)	4471 (2)	3098 (5)	5735 (4)	43 (1)
C(13)	5111 (2)	3046 (5)	6292 (4)	42 (1)
C(14)	5535 (2)	2439 (5)	5436 (4)	41 (1)
C(15)	6163 (2)	2387 (5)	6041 (4)	43 (1)
C(16)	5354 (2)	1871 (5)	4023 (4)	42 (1)
C(17)	4273 (2)	2561 (5)	4345 (4)	45 (1)
C(18)	6859 (2)	657 (6)	3042 (4)	56 (1)
C(19)	6694 (2)	98 (7)	1693 (4)	65 (2)
C(20)	6084 (2)	107 (7)	1076 (4)	64 (2)
C(21)	5646 (2)	667 (6)	1794 (4)	56 (1)
N(1)	6134 (2)	4332 (5)	9596 (3)	62 (1)
N(2)	7201 (1)	1768 (5)	5840 (3)	52 (1)
O(1)	4504 (1)	1477 (5)	2264 (3)	84 (1)
O(2)	5746 (2)	4980 (6)	10171 (3)	95 (1)
O(3)	6658 (1)	4221 (5)	10094 (3)	78 (1)
O(4)	7366 (1)	578 (4)	6701 (3)	68 (1)
O(5)	7538 (1)	2935 (4)	5501 (3)	74 (1)

systematic absences:  $h0l$ ,  $h+l = 2n+1$ ,  $0k0$ ,  $k = 2n+1$ ; crystal dimensions  $0.40 \times 0.35 \times 0.05$  mm; Rigaku AFC-6 diffractometer; graphite monochromator; cell parameters refined by least-squares method on the basis of 19 independent  $2\theta$  values,  $36 < 2\theta < 54^\circ$ ; intensity measurement performed up to  $2\theta = 125^\circ$ ; range of  $hkl$   $-25$  to  $25$ ,  $0$  to  $8$  and  $0$  to  $11$ ;  $\omega-2\theta$  scan, scan speed  $8^\circ \text{ min}^{-1}$  ( $2\theta$ ), scan width  $(1.2 + 0.15 \tan \theta)^\circ$ ; background  $5$  s before and after each scan; three standard reflections monitored every 100 reflections, no significant variation in intensities; 2431 unique reflections measured, 1362 with  $|F_o| > 3\sigma(|F_o|)$  considered observed and used for structure determination; corrected for Lorentz-polarization, absorption ignored; direct methods (MULTAN78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and subsequent difference Fourier calculation; block-diagonal least-squares (HBL5; Ohashi, 1975) with anisotropic thermal parameters for all non-H atoms; H atoms derived geometrically (C-H  $1.08 \text{ \AA}$ ) and refined;  $\sum w(|F_o| - |F_c|)^2$  minimized with  $w = [\sigma^2(F_o) + (0.03F)^2]^{-1}$ ; max.  $(\Delta/\sigma)$   $0.35$ ; final  $wR = 0.060$ ;  $\Delta\rho$  excursions in the final difference map  $0.3 \text{ e \AA}^{-3}$ ; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); calculations carried out on HITAC M-280 computer at the Computer Center of the University of Tokyo.

**Discussion.** The final atomic parameters for non-H atoms are in Table 1.\* A projection of the structure along the  $b$  axis is shown in Fig. 1 and the perspective drawing of the molecule with the numbering scheme is presented in Fig. 2. The two  $\text{NO}_2$  groups are bonded to C(2) and C(4). Bond distances and angles are listed in Table 2. The bond lengths and angles of the mother

\* 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 43241 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

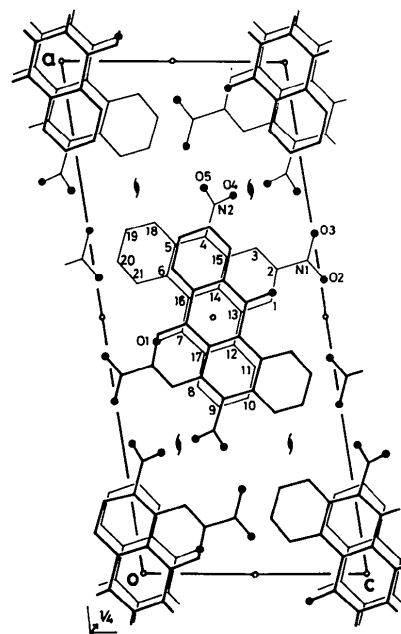


Fig. 1. A projection of the crystal structure along the  $b$  axis and the numbering scheme.

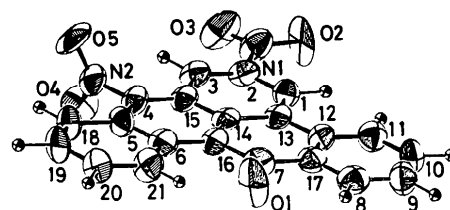


Fig. 2. Perspective drawing of the molecule.

Table 2. Bond lengths (Å) and angles (°) with their estimated standard deviations

C(1)—C(2)	1.389 (6)	C(8)—C(17)	1.393 (6)
C(1)—C(13)	1.376 (5)	C(9)—C(10)	1.385 (6)
C(2)—C(3)	1.335 (6)	C(10)—C(11)	1.360 (6)
C(2)—N(1)	1.501 (6)	C(11)—C(12)	1.397 (6)
C(3)—C(15)	1.422 (6)	C(12)—C(13)	1.454 (5)
C(4)—C(5)	1.401 (6)	C(12)—C(17)	1.404 (5)
C(4)—C(15)	1.373 (6)	C(13)—C(14)	1.434 (5)
C(4)—N(2)	1.464 (5)	C(14)—C(15)	1.441 (5)
C(5)—C(6)	1.436 (6)	C(14)—C(16)	1.425 (5)
C(5)—C(18)	1.406 (6)	C(18)—C(19)	1.362 (7)
C(6)—C(16)	1.418 (6)	C(19)—C(20)	1.405 (7)
C(6)—C(21)	1.433 (6)	C(20)—C(21)	1.361 (7)
C(7)—C(16)	1.474 (6)	N(1)—O(2)	1.207 (6)
C(7)—C(17)	1.468 (6)	N(1)—O(3)	1.204 (5)
C(7)—O(1)	1.224 (6)	N(2)—O(4)	1.221 (5)
C(8)—C(9)	1.369 (6)	N(2)—O(5)	1.222 (5)
C(2)—C(1)—C(13)	119.9 (4)	C(1)—C(13)—C(14)	119.2 (4)
C(1)—C(2)—C(3)	124.6 (4)	C(12)—C(13)—C(14)	120.4 (4)
C(1)—C(2)—N(1)	117.4 (4)	C(13)—C(14)—C(15)	118.4 (4)
C(3)—C(2)—N(1)	117.9 (4)	C(13)—C(14)—C(16)	122.2 (4)
C(2)—C(3)—C(15)	117.9 (4)	C(15)—C(14)—C(16)	119.4 (4)
C(5)—C(4)—C(15)	125.7 (4)	C(3)—C(15)—C(4)	122.1 (4)
C(5)—C(4)—N(2)	117.4 (4)	C(3)—C(15)—C(14)	120.1 (4)
C(15)—C(4)—N(2)	116.9 (4)	C(4)—C(15)—C(14)	117.7 (4)
C(4)—C(5)—C(6)	116.5 (4)	C(6)—C(16)—C(7)	122.7 (4)
C(4)—C(5)—C(18)	121.6 (4)	C(6)—C(16)—C(14)	120.3 (4)
C(6)—C(5)—C(18)	121.9 (4)	C(7)—C(16)—C(14)	117.0 (4)
C(5)—C(6)—C(16)	120.4 (4)	C(7)—C(17)—C(8)	120.0 (4)
C(5)—C(6)—C(21)	115.8 (4)	C(7)—C(17)—C(12)	120.5 (4)
C(16)—C(6)—C(21)	123.9 (4)	C(8)—C(17)—C(12)	119.5 (4)
C(16)—C(7)—C(17)	120.5 (4)	C(5)—C(18)—C(19)	119.6 (5)
C(16)—C(7)—O(1)	122.1 (4)	C(18)—C(19)—C(20)	120.2 (5)
C(17)—C(7)—O(1)	117.4 (4)	C(19)—C(20)—C(21)	121.6 (5)
C(9)—C(8)—C(17)	121.4 (4)	C(6)—C(21)—C(20)	121.1 (5)
C(8)—C(9)—C(10)	119.0 (5)	C(2)—N(1)—O(2)	116.2 (4)
C(9)—C(10)—C(11)	120.7 (5)	C(2)—N(1)—O(3)	118.5 (4)
C(10)—C(11)—C(12)	121.6 (4)	O(2)—N(1)—O(3)	125.3 (5)
C(11)—C(12)—C(13)	122.9 (4)	C(4)—N(2)—O(4)	118.4 (4)
C(11)—C(12)—C(17)	117.8 (4)	C(4)—N(2)—O(5)	118.4 (4)
C(13)—C(12)—C(17)	119.3 (4)	O(4)—N(2)—O(5)	123.2 (4)
C(1)—C(13)—C(12)	120.4 (4)		

skeleton are almost the same as those of the original 5,6-BzBT. The molecular structure is slightly distorted from a planar conformation. Such a distortion, which is also found in the structure of the 5,6-BzBT molecule, should be caused by the steric repulsion between the carbonyl group and the terminal benzene ring. The enlargement of the C(6)—C(16)—C(7) and the twisting angle around the pseudo bond C(6)···C(7), C(21)—C(6)···C(7)—O(1), can be regarded as a measure of the strength of the repulsion. The C(6)—C(16)—C(7) angle and the twisting angle are 122.7 (4) and 6.1 (5)°, which are comparable with the corresponding angles of 5,6-BzBT, 121 (1) and 8 (1)°, respectively. The angles of the two NO<sub>2</sub> groups [O(2)—N(1)—O(3) and O(4)—N(2)—O(5)] to the mean plane of the molecule are 6.1 (3) and 73.8 (2)°, respectively. The molecules are stacked face-to-face to make a column structure along the *b* axis. The distance between the mean molecular planes is 3.396 (4) Å. There is no unusual short contact between the columns.

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