de la molécule ($\frac{1}{2}+x$, $\frac{1}{2}-y$, $1-z$): $d(\mathrm{N}\cdots\mathrm{O}) =$ 2,784 (4) Å. Cette quasi absence de liaisons hydrogène est en accord avec la faible densité observée.

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2-Phenyl-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyrid-10-one

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Abstract. $C_{17}H_{17}NO$, $M_r = 251{\cdot}32$, monoclinic, $P2_1/n$ (non-standard group), $a = 10{\cdot}692$ (2), $b = 8{\cdot}408$ (1), $c = 15{\cdot}677$ (2) Å, $\beta = 95{\cdot}07$ (1)°, $V = 1403{\cdot}8$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}189$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 5{\cdot}01$ cm^{-1}, $F(000) = 536$, room temperature, $R = 0{\cdot}038$ for 1222 reflexions with $I > 3\sigma(I)$. The pyridine ring is planar, and the angle between the ring and the carbonyl bond is 62·4°. The cyclooctanone ring is in a twist-sofa conformation with two atoms [C(16) and C(17)] found in two possible positions. All bond lengths and angles are in agreement with expected values.

Introduction. This work was undertaken to establish the relation between the number of C atoms in the cycloalkanone ring of cycloalkanone–phenylpyridine derivatives and the conjugated double bond system (that of carbonyl and pyridine). We were also interested in studying the influence of these changes on the conformation of the cycloalkanone ring.

The results presented are a continuation of previous crystal structure investigations of cycloalkanone–phenylpyridine derivatives (Cygler, Dobrynin & Stępień, 1981*a*,*b*). A discussion and comparison of the results for all derivatives studied will be given in a separate paper.

Experimental. Colourless crystals obtained from ethanol. Space group and approximate lattice parameters obtained from oscillation and Weissenberg photographs, cell parameters and intensity data measured on an Enraf–Nonius CAD-4 diffractometer with a needle crystal, 0·2 × 0·2 × 0·5 mm. Orientation matrix and unit cell from 25 carefully centred reflections with $\theta < 16°$, graphite-monochromatized Cu $K\alpha$, range of *hkl*: −12 to 11, 0 to 10, 0 to 18, respectively. Standard reflection 212 monitored every 120 reflections showed no significant intensity change. Total of 2049 independent reflections measured to $\sin\theta/\lambda = 0{\cdot}63$ Å^{-1}, data not corrected for absorption, 1222 reflections with $I > 3\sigma(I)$ used in refinement calculations. Structure solution by direct methods using *SHELX*76 (Sheldrick, 1976), C(16) and C(17) found using Fourier synthesis,

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refinement by full-matrix least-squares procedure (on F). After initial anisotropic refinement, a difference synthesis at $R = 0{\cdot}094$ revealed positions of H atoms except those bound to atoms C(16) and C(17), which were placed in calculated positions with C—H = 1·08 Å. In subsequent cycles of refinement H atoms allowed to refine isotropically, $R = 0{\cdot}038$, $w = 1$, max. and min. values in final difference Fourier map = 0·14, −0·10 e Å^{-3}, ratio of max. shift/e.s.d. = 0·266. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The final positional parameters are listed in Table 1,* and selected bond lengths, bond angles and torsion angles are given in Table 2. Fig. 1 shows the numbering scheme and Fig. 2 shows stereoviews of the conformation of the molecule.

Table 1. *Final fractional coordinates* (× 10^4), *occupation factors and isotropic temperature factors* (× 10^4) *with e.s.d.'s in parentheses*

For non-H atoms $U_{\rm iso} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	Occ.	$U_{\rm iso}$(Å^2)
C(1)	5913 (3)	4577 (4)	6372 (2)	1·0	702 (21)
C(2)	6640 (4)	4512 (4)	5694 (2)	1·0	804 (25)
C(3)	7808 (4)	5207 (5)	5749 (3)	1·0	887 (29)
C(4)	8245 (4)	5992 (4)	6495 (3)	1·0	864 (28)
C(5)	7516 (3)	6066 (4)	7170 (3)	1·0	762 (24)
C(6)	6336 (3)	5354 (3)	7130 (2)	1·0	607 (19)
C(7)	5533 (3)	5450 (3)	7847 (2)	1·0	583 (18)
C(8)	5580 (3)	6731 (4)	8412 (2)	1·0	729 (23)
C(9)	4764 (4)	6781 (5)	9033 (2)	1·0	785 (25)
C(10)	3898 (3)	5581 (4)	9125 (2)	1·0	664 (19)
C(11)	3946 (3)	4321 (3)	8551 (2)	1·0	589 (18)
C(12)	2945 (4)	5613 (6)	9776 (2)	1·0	863 (30)
C(13)	1695 (4)	6367 (6)	9436 (3)	1·0	1047 (36)
C(14)	3121 (3)	2878 (4)	8604 (2)	1·0	691 (23)
C(15)	1818 (4)	2928 (6)	8147 (3)	1·0	880 (32)
C(16)	864 (8)	3851 (19)	8578 (11)	0·560 (25)	905 (88)
C(16′)	1155 (12)	4745 (24)	8139 (11)	0·440 (25)	801 (95)
C(17)	1055 (17)	5614 (20)	8600 (16)	0·560 (25)	1155 (130)
C(17′)	790 (23)	5011 (45)	8986 (19)	0·440 (25)	1552 (235)
N(1)	4713 (2)	4247 (3)	7923 (1)	1·0	588 (14)
O(1)	3528 (2)	1703 (3)	8979 (2)	1·0	966 (20)
H(1)	5065 (24)	4077 (31)	6349 (15)	1·0	722 (86)
H(2)	6282 (28)	3951 (38)	5131 (20)	1·0	1102 (120)
H(3)	8328 (29)	5154 (39)	5275 (20)	1·0	1038 (127)
H(4)	9122 (30)	6509 (38)	6533 (19)	1·0	1114 (121)
H(5)	7751 (26)	6662 (35)	7715 (18)	1·0	844 (104)
H(8)	6211 (28)	7617 (38)	8334 (19)	1·0	1000 (114)
H(9)	4750 (25)	7618 (35)	9393 (18)	1·0	798 (101)
H(121)	3350 (32)	6211 (43)	10292 (23)	1·0	1149 (136)
H(122)	2614 (33)	4539 (43)	9964 (21)	1·0	1036 (146)
H(131)	1091 (49)	6519 (70)	9947 (34)	1·0	2118 (267)
H(132)	1801 (65)	7847 (95)	9196 (45)	1·0	3297 (414)
H(151)	1446 (38)	1927 (49)	8269 (26)	1·0	1468 (179)
H(152)	1935 (35)	3120 (52)	7546 (25)	1·0	1184 (180)
H(161)	−63 (8)	3696 (19)	8256 (11)	0·560 (25)	1151 (258)
H(162)	911 (8)	3276 (19)	9196 (11)	0·560 (25)	1539 (597)
H(161′)	320 (12)	4471 (24)	7727 (11)	0·440 (25)	1099 (293)
H(162′)	1822 (12)	5349 (24)	7774 (11)	0·440 (25)	687 (363)
H(171)	116 (17)	6089 (20)	8486 (16)	0·560 (25)	780 (265)
H(172)	1725 (17)	6489 (20)	8423 (16)	0·560 (25)	4559 (561)
H(171′)	1132 (23)	4632 (45)	9623 (19)	0·440 (25)	2517 (549)
H(172′)	−49 (23)	5715 (45)	9018 (19)	0·440 (25)	611 (213)

The pyridine ring is planar within the limits of error with a maximum deviation from the best plane of less than 0·015 Å. The angle between the carbonyl bond and the pyridine ring plane is 62·4°.

The cyclooctanone ring adopts a twist-sofa conformation; the torsion angles are given in Table 2.

The benzene ring is planar within the limits of error with a maximum deviation from the best plane of less than 0·004 Å. The ring is twisted about the C(6)—C(7) bond, the angle being 152·2°.

Table 2. *Selected bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

C(1)—C(2)	1·372 (5)	C(2)—C(3)	1·375 (6)
C(3)—C(4)	1·387 (6)	C(4)—C(5)	1·370 (6)
C(5)—C(6)	1·393 (4)	C(6)—C(1)	1·397 (4)
C(6)—C(7)	1·475 (4)	C(7)—C(8)	1·392 (5)
C(8)—C(9)	1·365 (5)	C(9)—C(10)	1·386 (5)
C(10)—C(11)	1·393 (4)	C(11)—N(1)	1·337 (4)
N(1)—C(7)	1·350 (4)	C(10)—C(12)	1·505 (5)
C(12)—C(13)	1·531 (6)	C(13)—C(17)	1·559 (2)
C(13)—C(17′)	1·617 (3)	C(17)—C(16)	1·496 (2)
C(17′)—C(16′)	1·434 (35)	C(16)—C(15)	1·490 (15)
C(16′)—C(15)	1·683 (20)	C(15)—C(14)	1·509 (5)
C(14)—C(11)	1·507 (5)	C(14)—O(1)	1·211 (4)
C(1)—C(2)—C(3)	120·5 (3)	C(2)—C(3)—C(4)	119·4 (4)
C(3)—C(4)—C(5)	120·1 (4)	C(4)—C(5)—C(6)	121·4 (3)
C(5)—C(6)—C(1)	117·6 (3)	C(6)—C(1)—C(2)	121·0 (3)
C(1)—C(6)—C(7)	120·5 (3)	C(5)—C(6)—C(7)	121·9 (3)
C(8)—C(7)—C(6)	122·4 (3)	N(1)—C(7)—C(6)	116·9 (2)
C(7)—C(8)—C(9)	119·2 (3)	C(8)—C(9)—C(10)	121·8 (3)
C(9)—C(10)—C(11)	115·1 (3)	C(10)—C(11)—N(1)	124·8 (3)
C(11)—N(1)—C(7)	118·4 (2)	N(1)—C(7)—C(8)	120·7 (3)
C(9)—C(10)—C(12)	123·8 (3)	C(12)—C(10)—C(11)	121·1 (3)
N(1)—C(11)—C(14)	113·8 (3)	C(10)—C(11)—C(14)	121·4 (3)
C(11)—C(14)—O(1)	119·7 (3)	C(15)—C(14)—O(1)	122·0 (4)
C(11)—C(14)—C(15)	118·2 (3)	C(14)—C(15)—C(16)	116·1 (6)
C(14)—C(15)—C(16′)	113·5 (6)	C(15)—C(16)—C(17)	115·4 (12)
C(15)—C(16′)—C(17′)	106·5 (18)	C(16)—C(17)—C(13)	118·1 (16)
C(16′)—C(17′)—C(13)	108·2 (20)	C(17)—C(13)—C(12)	115·4 (7)
C(17′)—C(13)—C(12)	109·3 (12)	C(13)—C(12)—C(10)	113·2 (3)
C(1)—C(6)—C(7)—C(8)	−148·8 (3)	C(10)—C(12)—C(13)—C(17)	55 (1)
C(1)—C(6)—C(7)—N(1)	29·5 (4)	C(10)—C(12)—C(13)—C(17′)	88 (1)
C(5)—C(6)—C(7)—C(8)	29·2 (4)	C(12)—C(13)—C(17)—C(16)	52 (2)
C(5)—C(6)—C(7)—N(1)	−152·4 (3)	C(12)—C(13)—C(17′)—C(16′)	−81 (2)
C(7)—C(8)—C(9)—C(10)	0·8 (5)	C(13)—C(17)—C(16)—C(15)	−99 (2)
C(8)—C(9)—C(10)—C(11)	1·3 (5)	C(13)—C(17′)—C(16′)—C(15)	110 (2)
C(9)—C(10)—C(11)—N(1)	−2·8 (5)	C(17)—C(16)—C(15)—C(14)	69 (2)
C(10)—C(11)—N(1)—C(7)	2·1 (4)	C(17′)—C(16′)—C(15)—C(14)	−75 (1)
C(11)—N(1)—C(7)—C(8)	0·3 (4)	C(16)—C(15)—C(14)—O(1)	105 (1)
N(1)—C(7)—C(8)—C(9)	−1·7 (5)	C(16′)—C(15)—C(14)—O(1)	149 (1)
C(8)—C(9)—C(10)—C(12)	−177·4 (3)	C(16)—C(15)—C(14)—C(11)	−77 (1)
C(9)—C(10)—C(12)—C(13)	91·0 (4)	C(16′)—C(15)—C(14)—C(11)	−34 (1)
C(7)—N(1)—C(11)—C(14)	−176·9 (2)	C(15)—C(14)—C(11)—C(10)	86·5 (4)
N(1)—C(11)—C(14)—O(1)	82·9 (4)	C(14)—C(11)—C(10)—C(12)	−5·1 (5)
N(1)—C(11)—C(14)—C(15)	−94·4 (4)	C(11)—C(10)—C(12)—C(13)	−87·6 (5)

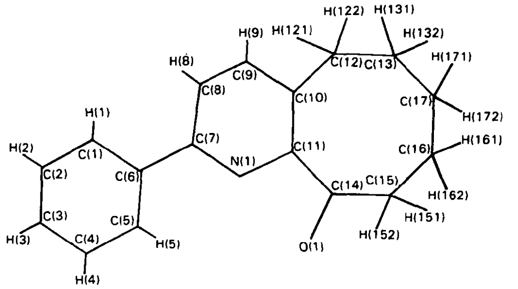

Fig. 1. The atom-numbering scheme.

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* Lists of structure factors, anisotropic thermal parameters, bond lengths, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43220 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

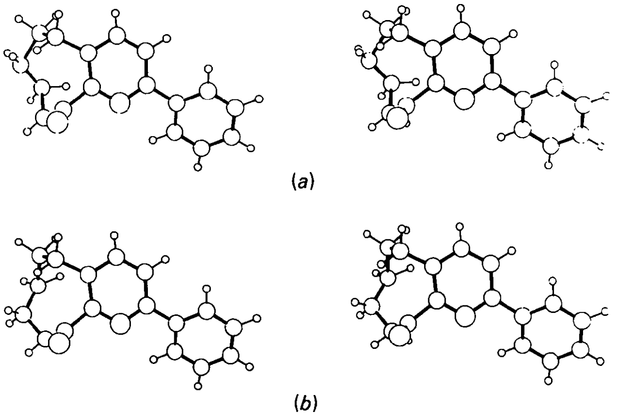

Fig. 2. Stereoviews of the molecule. Note the possible positions (*a*) for atoms C(16) and C(17), and (*b*) for C(16′) and C(17′).

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, $\beta = 99·39\ (2)°$, $V = 1574·1\ (6)$ Å^3, $Z = 4$, $D_m = 1·56$, $D_x = 1·562$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1·54184$ Å, $\mu = 9·90$ cm^{-1}, $F(000) = 760$, $T = 298$ K. Final $R = 0·060$ for 1362 independent reflections. The molecule is slightly distorted from a planar structure. The angles of the two NO_2 groups to the mean molecular plane are 6·1 (3) and 73·8 (2)°. 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 compounds, 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 $ZnCl_2$ solution;