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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1215). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hindered Cavity-Shaped *N*-Oxides

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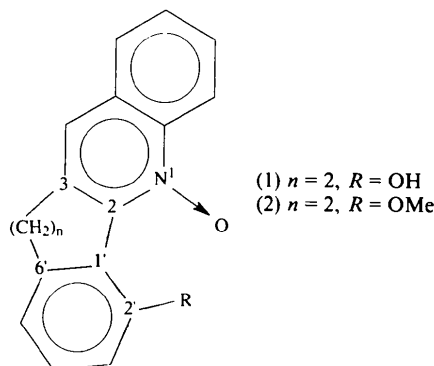
Abstract

The crystal structures of 1-hydroxy-5,6-dihydro-12-azabenz[*a*]anthracene *N*-oxide (1), C₁₇H₁₃NO₂, and 1-methoxy-5,6-dihydro-12-azabenz[*a*]anthracene *N*-oxide (2), C₁₈H₁₅NO₂, have been determined. In both (1) and (2), the 2-phenylquinoline *N*-oxide skeleton has been strengthened by a —CH₂—CH₂— linkage through the 3- and 6'-positions. Moreover, in compound (1) a strong intramolecular O—H···O hydrogen bond is formed which accounts for the fact that a more pla-

nar conformation of molecule (1) is observed compared with molecule (2). Modes of steric strain compensation in both (1) and (2) are discussed.

Comment

Annellation of 2-phenylquinoline *N*-oxide at the 3- and 6'-positions gives a series of cavity-shaped molecules whose shapes depend on the number and hybridization of C atoms in the 3,6'-bridge. The introduction of a substituent at position 2' causes steric strain in the bay region of the molecule which can affect its reactivity. In particular, these compounds have been synthesized as model compounds in order to study the mechanism of the observed facile deoxidation of orellanine (3,3',4,4'-tetrahydroxy-2,2'-bipyridine 1,1'-dioxide) (Antkowiak & Gessner, 1979, 1984; Antkowiak, Antkowiak & Czerwiński, 1990). We present here the crystal structures of 1-hydroxy-5,6-dihydro-12-azabenz[*a*]anthracene *N*-oxide and 1-methoxy-5,6-dihydro-12-azabenz[*a*]anthracene *N*-oxide [hereinafter referred to as (1) and (2), respectively].



The asymmetric unit of compound (2) contains two molecules, denoted as *A* and *B*, which do not differ significantly. The differences in bond lengths and angles are less than $2\sigma_+$ [$\sigma_+ = (\sigma_1^2 + \sigma_2^2)^{1/2}$]. The geometries of molecules (1) and (2) are affected by two elements, namely, steric hindrance and/or intramolecular hydrogen bonding. We have selected some structural parameters of (1) and (2) in order to analyse steric hindrance (Table 5); these include: the sum of the deviations, $\Sigma|d|$, of the atoms from the least-squares planes of the aromatic systems, the dihedral angle between the aromatic ring planes (denoted α), the distance between atoms N12 and C1 (denoted $d_{12,1}$) and the O12—N12—C11a and O1—C1—C12b bond angles. All the special features of the molecular structure result from the tendency towards steric hindrance minimization. Examination of Table 5 shows that the modes of steric strain compensation in compounds (1) and (2) differ. In (1), a short intramolecular O1—H1···O12 hydrogen bond [O1···O12 2.450 (3), H1···O12 1.47 (4) Å, O1—H1···O12 159 (3)°] is formed, which restrains the con-

formational versatility to a greater measure than in compound (2). Nevertheless, a smaller value of the sum of distortions, $\Sigma|d|$, in compound (1) is observed. On the other hand, the dihedral angle between the two aromatic systems is much smaller in (1) than in (2). The same tendency has been found in other cavity-shaped *N*-oxides, namely, 5-hydroxy-9,10-dihydro-4-azaphenanthrene 4-oxide (Kubicki, Borowiak, Chruścicki & Antkowiak, 1993) and its 5-methoxy derivative (Kubicki, Borowiak, Antkowiak, Antkowiak & Chruścicki, 1994). In neither (1) nor (2) is pyramidalization at C1 or N12 observed, the sum of bond angles around these atoms being equal

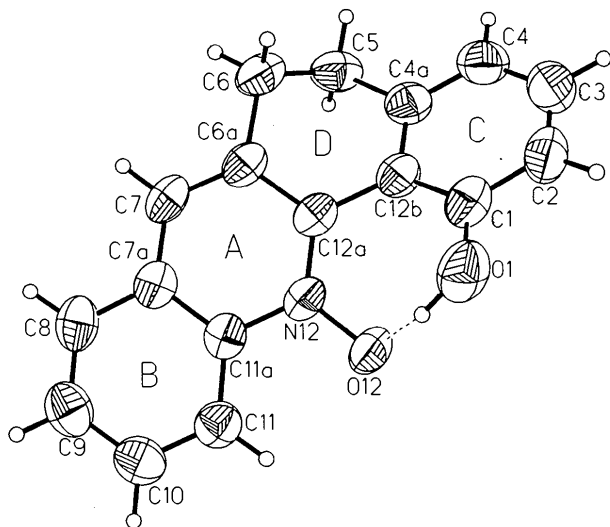


Fig. 1. A displacement ellipsoid representation of compound (1) with the labelling scheme. The ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii. The hydrogen bond is depicted by a dashed line.

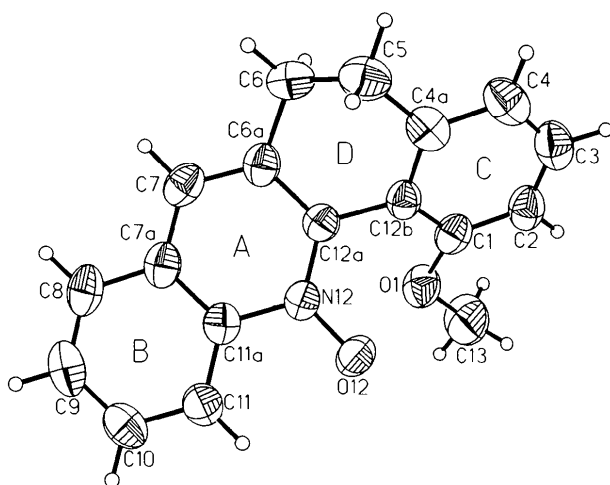


Fig. 2. A displacement ellipsoid representation of molecule (2A) with the labelling scheme. The ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

to 360° . However, examination of the χ^2 test, calculated for the least-squares planes of the aromatic rings, shows that both atoms of the pivot bond, C12a and C12b, are pushed out of the ring planes. In both compounds (1) and (2), the substituents deviate in opposite directions from their aromatic ring planes.

The N—O bond lengths of (1) and (2) are different, that of compound (1) being longer due to the hydrogen bond in which it is involved. In (2), the N—O bond lengths are very close to the length of the 'free' N—O bond found in pyridine *N*-oxide [1.2806 (4) Å; Sørensen, Mahler & Rastrup-Andersen, 1974; Eichhorn, 1987]. Also, the C—O bond length in (1) is shortened compared with the typical value of 1.362 Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The non-aromatic rings of compounds (1) and (2) exhibit a 1,3-diplanar conformation with a twofold axis passing through the midpoints of the pivot bond C12a—C12b. In compound (1), this conformation is an ideal one, the ΔC_2 asymmetry parameter (Duax & Norton, 1975) being equal to 0.3° , whereas in (2) these values are 5.4 and 3.1° in molecules A and B, respectively. Molecules of (1) and (2) can occur as two enantiomeric isomers due to the restrained rotation around the pivot bond.

No significant short contacts are observed in the crystal packing.

Experimental

Compound (1)

Crystal data

$C_{17}H_{13}NO_2$
 $M_r = 263.28$
 Monoclinic
 $P2_1/a$
 $a = 14.257 (2) \text{ \AA}$
 $b = 7.2001 (8) \text{ \AA}$
 $c = 12.871 (1) \text{ \AA}$
 $\beta = 108.88 (1)^\circ$
 $V = 1250.1 (2) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.399 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation
 $\lambda = 1.54178 \text{ \AA}$
 Cell parameters from 15 reflections
 $\theta = 21.60\text{--}44.15^\circ$
 $\mu = 0.742 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Prism
 $0.35 \times 0.20 \times 0.20 \text{ mm}$
 Colorless
 Crystal source: benzene-hexane solution

Data collection

Syntex $P2_1$ diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 1653 measured reflections
 1653 independent reflections
 1258 observed reflections
 $[I > 2\sigma(I)]$

$\theta_{\max} = 57.35^\circ$
 $h = -15 \rightarrow 14$
 $k = 0 \rightarrow 7$
 $l = 0 \rightarrow 14$
 2 standard reflections monitored every 100 reflections
 intensity decay: 1.5%

Refinement

Refinement on F^2
 $R(F) = 0.0421$
 $wR(F^2) = 0.1244$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.132 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.150 \text{ e \AA}^{-3}$

$S = 1.109$	Extinction correction:	C6	0.1782 (2)	0.5933 (3)	0.7048 (2)	0.0613 (7)
1653 reflections	<i>SHELXL93</i> (Sheldrick,	C6a	0.2529 (2)	0.5186 (3)	0.8073 (2)	0.0502 (6)
186 parameters	1993)	C7	0.2940 (2)	0.6235 (3)	0.8987 (2)	0.0545 (6)
H-atom parameters not	Extinction coefficient:	C7a	0.3607 (2)	0.5451 (3)	0.9946 (2)	0.0514 (6)
refined except for H1	0.0136 (14)	C8	0.4084 (2)	0.6470 (3)	1.0902 (2)	0.0628 (7)
for which all parameters	Atomic scattering factors	C9	0.4717 (2)	0.5623 (4)	1.1808 (2)	0.0678 (7)
refined	from <i>International Tables</i>	C10	0.4908 (2)	0.3719 (4)	1.1798 (2)	0.0635 (7)
$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2$	for <i>Crystallography</i> (1992,	C11	0.4465 (2)	0.2675 (3)	1.0882 (2)	0.0571 (6)
+ 0.3321P]	Vol. C, Tables 4.2.6.8 and	C11a	0.38195 (15)	0.3546 (3)	0.9957 (2)	0.0484 (6)
where $P = (F_o^2 + 2F_c^2)/3$	6.1.1.4)	N12	0.33687 (13)	0.2498 (2)	0.90075 (14)	0.0497 (5)
		O12	0.35461 (12)	0.0677 (2)	0.90806 (14)	0.0652 (5)
		C12a	0.27791 (15)	0.3271 (3)	0.8065 (2)	0.0479 (6)
		C12b	0.2362 (2)	0.2177 (3)	0.7052 (2)	0.0499 (6)

Compound (2)*Crystal data*C₁₈H₁₅NO₂ $M_r = 277.31$

Monoclinic

Pn $a = 16.092 (2) \text{ \AA}$ $b = 7.9060 (10) \text{ \AA}$ $c = 11.8890 (10) \text{ \AA}$ $\beta = 115.200 (10)^\circ$ $V = 1368.6 (3) \text{ \AA}^3$ $Z = 4$ $D_x = 1.346 \text{ Mg m}^{-3}$ Cu $K\alpha$ radiation $\lambda = 1.54178 \text{ \AA}$

Cell parameters from 15 reflections

 $\theta = 8.25\text{--}43.35^\circ$ $\mu = 0.704 \text{ mm}^{-1}$ $T = 293 (2) \text{ K}$

Prism

 $0.3 \times 0.2 \times 0.2 \text{ mm}$

Colorless

Crystal source: CH₂Cl₂–toluene solution*Data collection*Syntex *P2*₁ diffractometer $\omega/2\theta$ scans

Absorption correction:

none

1935 measured reflections

1865 independent reflections

1768 observed reflections

 $[I > 2\sigma(I)]$ $R_{\text{int}} = 0.0352$ $\theta_{\text{max}} = 57.32^\circ$ $h = -17 \rightarrow 15$ $k = -8 \rightarrow 8$ $l = 0 \rightarrow 12$

2 standard reflections

monitored every 100

reflections

intensity decay: 2.2%

*Refinement*Refinement on F^2 $R(F) = 0.0553$ $wR(F^2) = 0.1450$ $S = 1.072$

1865 reflections

382 parameters

H-atom parameters not

refined

 $w = 1/[\sigma^2(F_o^2) + (0.1141P)^2$

+ 0.4126P]

where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} < 0.001$ $\Delta\rho_{\text{max}} = 0.377 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.225 \text{ e \AA}^{-3}$ Extinction correction:
SHELXL93 (Sheldrick,
1993)

Extinction coefficient:

0.0035 (8)

Atomic scattering factors
from *International Tables*
for *Crystallography* (1992,Vol. C, Tables 4.2.6.8 and
6.1.1.4)Table 2. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C1A	0.6580 (3)	0.5956 (6)	0.3154 (4)	0.0468 (11)
O1A	0.5701 (2)	0.5368 (4)	0.2543 (3)	0.0550 (9)
C13A	0.4985 (4)	0.6395 (9)	0.2558 (7)	0.084 (2)
C2A	0.6822 (4)	0.7402 (6)	0.3886 (5)	0.0543 (12)
C3A	0.7726 (4)	0.7772 (7)	0.4592 (5)	0.0620 (14)
C4A	0.8421 (4)	0.6702 (7)	0.4640 (5)	0.0616 (14)
C4aA	0.8198 (4)	0.5230 (6)	0.3897 (5)	0.0517 (12)
C5A	0.8889 (3)	0.3933 (7)	0.3998 (5)	0.0599 (13)
C6A	0.8494 (3)	0.2194 (7)	0.4046 (5)	0.0560 (12)
C6aA	0.7651 (3)	0.1909 (6)	0.2852 (4)	0.0466 (11)
C7A	0.7446 (4)	0.0411 (6)	0.2233 (5)	0.0507 (12)
C7aA	0.6687 (4)	0.0303 (6)	0.1048 (5)	0.0479 (11)
C8A	0.6398 (4)	−0.1225 (7)	0.0380 (5)	0.0555 (12)
C9A	0.5720 (4)	−0.1233 (7)	−0.0792 (6)	0.0620 (14)
C10A	0.5284 (4)	0.0267 (7)	−0.1356 (5)	0.0589 (13)
C11A	0.5522 (4)	0.1779 (6)	−0.0727 (4)	0.0513 (12)
C11aA	0.6211 (3)	0.1786 (5)	0.0491 (5)	0.0439 (10)
N12A	0.6447 (3)	0.3334 (5)	0.1155 (4)	0.0441 (9)
O12A	0.6051 (2)	0.4721 (4)	0.0580 (3)	0.0519 (8)
C12aA	0.7092 (3)	0.3355 (6)	0.2351 (4)	0.0428 (10)
C12bA	0.7266 (3)	0.4920 (5)	0.3083 (4)	0.0422 (10)
C1B	0.0953 (3)	−0.0975 (6)	0.0013 (5)	0.0483 (11)
O1B	0.0213 (3)	−0.0370 (4)	−0.0983 (3)	0.0556 (9)
C13B	−0.0130 (5)	−0.1365 (9)	−0.2081 (6)	0.080 (2)
C2B	0.1425 (4)	−0.2431 (6)	−0.0004 (6)	0.0585 (13)
C3B	0.2236 (4)	−0.2834 (7)	0.0999 (6)	0.0651 (15)
C4B	0.2620 (4)	−0.1758 (7)	0.2001 (6)	0.0636 (15)
C4aB	0.2152 (3)	−0.0288 (7)	0.2065 (5)	0.0544 (12)
C5B	0.2574 (4)	0.1002 (8)	0.3072 (5)	0.0612 (13)
C6B	0.2384 (3)	0.2748 (7)	0.2463 (5)	0.0573 (13)
C6aB	0.1364 (3)	0.3025 (6)	0.1814 (4)	0.0446 (10)
C7B	0.0956 (4)	0.4540 (6)	0.1841 (5)	0.0504 (12)
C7aB	−0.0014 (4)	0.4647 (6)	0.1292 (5)	0.0461 (11)
C8B	−0.0482 (4)	0.6187 (7)	0.1221 (5)	0.0600 (13)
C9B	−0.1408 (4)	0.6225 (8)	0.0771 (5)	0.0636 (14)
C10B	−0.1923 (4)	0.4731 (7)	0.0354 (5)	0.0617 (15)
C11B	−0.1497 (3)	0.3231 (7)	0.0399 (5)	0.0527 (12)
C11aB	−0.0543 (3)	0.3191 (6)	0.0838 (4)	0.0438 (11)
N12B	−0.0098 (3)	0.1638 (5)	0.0851 (3)	0.0414 (8)
O12B	−0.0587 (2)	0.0258 (4)	0.0532 (3)	0.0515 (8)
C12aB	0.0826 (3)	0.1610 (5)	0.1212 (4)	0.0419 (10)
C12bB	0.1286 (3)	0.0052 (6)	0.1089 (5)	0.0455 (11)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (1)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C1	0.2861 (2)	0.0725 (3)	0.6711 (2)	0.0583 (6)
O1	0.37884 (13)	0.0199 (2)	0.7300 (2)	0.0772 (6)
C2	0.2399 (2)	−0.0158 (3)	0.5709 (2)	0.0683 (7)
C3	0.1475 (2)	0.0349 (4)	0.5068 (2)	0.0768 (8)
C4	0.0989 (2)	0.1800 (4)	0.5369 (2)	0.0692 (7)
C4a	0.1428 (2)	0.2751 (3)	0.6335 (2)	0.0558 (6)
C5	0.0971 (2)	0.4481 (3)	0.6615 (2)	0.0654 (7)

Table 3. Selected geometric parameters (\AA , $^\circ$) for (1)

C1—O1	1.349 (3)	C6a—C12a	1.425 (3)
C1—C2	1.397 (3)	C7—C7a	1.409 (3)
C1—C12b	1.411 (3)	C7a—C11a	1.404 (3)
O1—H1	1.02 (4)	C7a—C8	1.405 (3)
C2—C3	1.358 (4)	C8—C9	1.366 (3)
C3—C4	1.377 (4)	C9—C10	1.399 (4)
C4—C4a	1.380 (3)	C10—C11	1.369 (3)
C4a—C12b	1.414 (3)	C11—C11a	1.396 (3)
C4a—C5	1.502 (3)	C11a—N12	1.402 (3)
C5—C6	1.524 (3)	N12—O12	1.333 (2)
C6—C6a	1.501 (3)	N12—C12a	1.352 (3)
C6a—C7	1.361 (3)	C12a—C12b	1.474 (3)

O1—C1—C2	118.3 (2)	C8—C7a—C7	123.8 (2)
O1—C1—C12b	122.3 (2)	C9—C8—C7a	120.9 (2)
C2—C1—C12b	119.3 (2)	C8—C9—C10	120.5 (2)
C3—C2—C1	120.8 (2)	C11—C10—C9	120.6 (2)
C2—C3—C4	120.7 (3)	C10—C11—C11a	118.8 (2)
C3—C4—C4a	120.5 (2)	C11—C11a—N12	119.4 (2)
C4—C4a—C12b	120.0 (2)	C11—C11a—C7a	121.9 (2)
C4—C4a—C5	121.5 (2)	N12—C11a—C7a	118.7 (2)
C12b—C4a—C5	118.4 (2)	O12—N12—C12a	121.1 (2)
C4a—C5—C6	108.7 (2)	O12—N12—C11a	116.4 (2)
C6a—C6—C5	108.3 (2)	C12a—N12—C11a	122.5 (2)
C7—C6a—C12a	120.3 (2)	N12—C12a—C6a	118.4 (2)
C7—C6a—C6	123.0 (2)	N12—C12a—C12b	122.0 (2)
C12a—C6a—C6	116.6 (2)	C6a—C12a—C12b	119.5 (2)
C6a—C7—C7a	121.0 (2)	C1—C12b—C4a	118.5 (2)
C11a—C7a—C8	117.4 (2)	C1—C12b—C12a	124.8 (2)
C11a—C7a—C7	118.8 (2)	C4a—C12b—C12a	116.5 (2)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (2)

	Molecule A	Molecule B
C1—O1	1.368 (6)	1.359 (6)
C1—C2	1.388 (7)	1.384 (7)
C1—C12b	1.405 (7)	1.414 (7)
O1—C13	1.415 (7)	1.419 (7)
C2—C3	1.366 (8)	1.379 (8)
C3—C4	1.384 (8)	1.377 (9)
C4—C4a	1.412 (7)	1.404 (7)
C4a—C12b	1.418 (7)	1.409 (7)
C4a—C5	1.480 (8)	1.497 (8)
C5—C6	1.526 (8)	1.528 (8)
C6—C6a	1.505 (7)	1.503 (7)
C6a—C7	1.359 (7)	1.373 (7)
C6a—C12a	1.420 (6)	1.409 (6)
C7—C7a	1.420 (8)	1.414 (7)
C7a—C11a	1.403 (6)	1.397 (7)
C7a—C8	1.412 (7)	1.416 (7)
C8—C9	1.356 (8)	1.353 (8)
C9—C10	1.395 (8)	1.406 (9)
C10—C11	1.375 (7)	1.359 (8)
C11—C11a	1.399 (7)	1.395 (7)
C11a—N12	1.418 (6)	1.419 (6)
N12—O12	1.306 (5)	1.304 (5)
N12—C12a	1.357 (6)	1.361 (6)
C12a—C12b	1.470 (6)	1.475 (6)
O1—C1—C2	124.2 (4)	124.3 (5)
O1—C1—C12b	115.6 (4)	116.1 (4)
C2—C1—C12b	120.0 (4)	119.5 (5)
C1—O1—C13	117.5 (4)	117.4 (4)
C3—C2—C1	120.1 (5)	120.2 (5)
C2—C3—C4	121.7 (5)	121.0 (5)
C3—C4—C4a	119.5 (5)	120.3 (5)
C4—C4a—C12b	118.8 (5)	118.6 (5)
C4—C4a—C5	122.6 (5)	122.4 (5)
C12b—C4a—C5	118.4 (4)	118.8 (4)
C4a—C5—C6	108.5 (4)	107.8 (4)
C6a—C6—C5	108.8 (4)	109.2 (4)
C7—C6a—C12a	120.6 (4)	120.5 (4)
C7—C6a—C6	123.7 (4)	123.0 (4)
C12a—C6a—C6	115.5 (4)	116.5 (4)
C6a—C7—C7a	119.9 (4)	119.1 (4)
C11a—C7a—C8	117.8 (5)	120.0 (4)
C11a—C7a—C7	119.0 (4)	117.7 (5)
C8—C7a—C7	123.2 (5)	122.3 (5)
C9—C8—C7a	120.7 (5)	120.8 (5)
C8—C9—C10	120.6 (5)	120.3 (5)
C11—C10—C9	120.7 (5)	120.5 (5)
C10—C11—C11a	118.7 (4)	119.3 (5)
C11—C11a—C7a	121.2 (4)	121.3 (4)
C11—C11a—N12	119.1 (4)	119.6 (4)
C7a—C11a—N12	119.7 (4)	119.1 (4)
O12—N12—C12a	121.6 (4)	121.4 (4)
O12—N12—C11a	118.5 (3)	118.8 (3)
C12a—N12—C11a	119.9 (4)	119.8 (4)
N12—C12a—C6a	119.7 (4)	120.0 (4)
N12—C12a—C12b	119.9 (4)	120.6 (4)
C6a—C12a—C12b	120.2 (4)	119.2 (4)

C1—C12b—C4a	119.2 (4)	119.7 (4)
C1—C12b—C12a	124.8 (4)	116.1 (4)
C4a—C12b—C12a	115.4 (4)	123.8 (4)

Table 5. Selected structural parameters (\AA , $^\circ$) for (1) and (2)

$\sum|d|$ is the sum of the deviations of the atoms from the least-squares plane of the aromatic system, α is the dihedral angle between the aromatic ring planes, and $d_{12,1}$ is the distance between atoms N12 and C1.

	(1)	(2A)	(2B)
$\sum d $ Ring A	0.101	0.241	0.253
$\sum d $ Ring B	0.094	0.161	0.173
α	33.0 (1)	39.3 (2)	40.5 (2)
$d_{12,1}$	3.083 (3)	3.091 (6)	3.091 (6)
O12—N12—C11a	116.4 (2)	118.5 (3)	118.8 (3)
O1—C1—C12b	122.3 (2)	115.6 (4)	116.1 (4)

For both compounds, data collection: *P2*₁ software; cell refinement: *P2*₁ software; data reduction: *PRADIR* (Jaskólski, 1990); program(s) used to solve structures: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989); software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including bond distances and angles involving H atoms, and torsion angles have been deposited with the IUCr (Reference: KA1078). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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