Data collection

Rigaku AFC-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North *et al.*, 1968)
$T_{\min} = 0.575$, $T_{\max} = 0.693$
2115 measured reflections
2115 independent reflections
1806 reflections with $I > 3\sigma(I)$
$\theta_{\max} = 80°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 36$
$l = 0 \rightarrow 8$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.047$
$wR = 0.055$
$S = 2.86$
1806 reflections
189 parameters
H atoms not refined
Weighting scheme based on measured s.u.'s; $w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.04$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.35$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 3. *Selected geometric parameters (Å, °) for Leu.-p-TS.H_2O*

S—O3	1.447 (3)	C4—C5	1.512 (8)
S—O4	1.445 (3)	C4—C6	1.506 (7)
S—O5	1.442 (3)	C7—C8	1.370 (6)
S—C7	1.763 (4)	C7—C12	1.355 (6)
O1—C2	1.200 (4)	C8—C9	1.379 (6)
O2—C2	1.285 (5)	C9—C10	1.350 (7)
N—C1	1.472 (4)	C10—C11	1.367 (7)
C1—C2	1.515 (5)	C10—C13	1.516 (6)
C1—C3	1.529 (5)	C11—C12	1.386 (6)
C3—C4	1.504 (6)		
O1—C2—C1—N	−9.2 (6)	N—C1—C3—C4	−168.4 (3)
O1—C2—C1—C3	112.0 (5)	C1—C3—C4—C5	−176.0 (5)
O2—C2—C1—N	168.3 (4)	C1—C3—C4—C6	61.3 (6)
O2—C2—C1—C3	−70.5 (5)	C2—C1—C3—C4	72.6 (5)

Because of the poor ratio of the number of reflections to the number of parameters, the H-atom parameters were not included in the refinement.

For all compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *MSC/AFC Diffractometer Control Software*; program(s) used to solve structures: *MITHRIL* (Gilmore, 1984); program(s) used to refine structures: *ALLS* (Lapp & Jacobson, 1979); software used to prepare material for publication: *PLUTO* (Motherwell & Clegg, 1983) and *ORTEP*II (Johnson, 1976).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1024). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 641–643

10-Chloro-6,7-dihydro-5*H*-benzo[6,7]cyclohepta[1,2-*b*]quinoline and 10-Methyl-6,7-dihydro-5*H*-benzo[6,7]cyclohepta[1,2-*b*]quinoline

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Abstract

The quinoline ring systems in the title molecules, $C_{18}H_{14}ClN$ and $C_{19}H_{17}N$, are planar. The cycloheptane rings are in half-chair conformations and the crystal structures are stabilized by van der Waals interactions.

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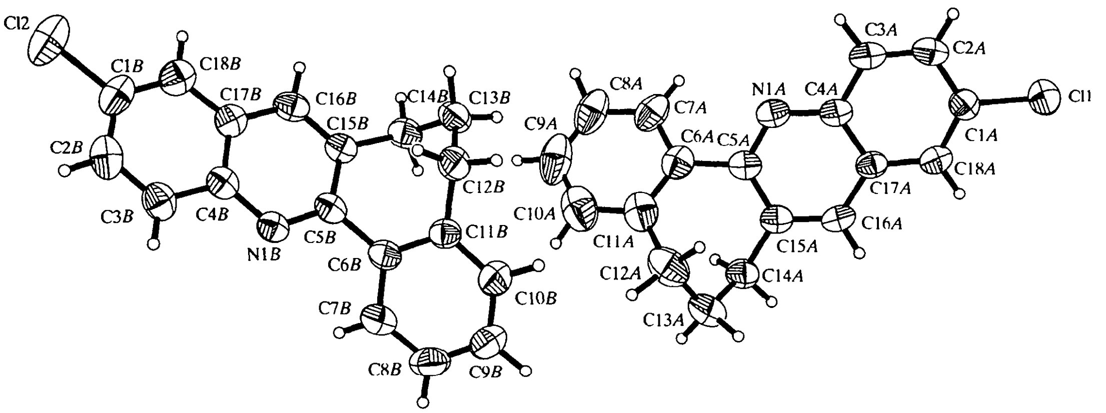
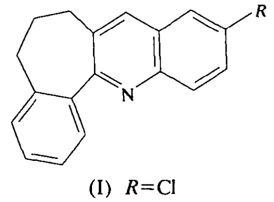


Fig. 1. View of the two molecules in the asymmetric unit of (I) showing the numbering schemes; displacement ellipsoids are drawn at the 50% probability level.

Comment

The structures of two benzosuberinoquinoline derivatives, namely, 10-chloro-6,7-dihydro-5*H*-benzo[6,7]cyclohepta[1,2-*b*]quinoline, (I), and 10-methyl-6,7-dihydro-5*H*-benzo[6,7]cyclohepta[1,2-*b*]quinoline, (II), have been determined as part of studies on the synthesis, characterization and bioactivity of polycyclic aromatic hydrocarbon derivatives. Both compounds are intermediates for bioactive B-homoazasteroids and are also interesting hosts for the recognition of molecules.



(I) R=Cl
(II) R=CH_3

A displacement ellipsoid plot of the two independent molecules of (I) is shown in Fig. 1. Bond lengths and angles in (I) are normal, and values for the two independent molecules agree with each other. The C2—C3, C1—C18 and C15—C16 bonds show localized double-bond character, whereas N1—C5 is a partial double bond (Allen *et al.*, 1987). The quinoline ring system is planar to within ±0.050 (2) in (I*A*) and to within −0.041 (2) Å in (I*B*). In both molecules, the cycloheptane ring adopts a half-chair conformation with a twofold axis passing through C13 and bisecting the C5—C6 bond. The dihedral angle between the quinoline and benzene ring planes is 136.42 (7) in (I*A*) and 130.28 (7)° in (I*B*). The dihedral angle between the two independent quinoline rings in the asymmetric unit is 133.23 (4)°. The crystal structure is stabilized by van der Waals interactions. Structural details for (II) are similar to those for (I).

Experimental

On treatment with Vilsmeier–Haack reagent (phosphoryl chloride/dimethylformamide), benzosuberone produced the chloroaldehyde as an oil which, on treatment with arylamines in ethanol, afforded the corresponding anil derivatives as red solids. Heating the anil derivative at 523–543 K for 5 min furnished the title compounds (Ray *et al.*, 1980). Single crystals were grown by slow evaporation from chloroform solutions.

Compound (I)

Crystal data

$C_{18}H_{14}ClN$
M_r = 279.75
Monoclinic
$P2_1/n$
a = 8.873 (1) Å
b = 12.921 (1) Å
c = 24.812 (3) Å
β = 100.16 (1)°
V = 2800.0 (5) Å^3
Z = 8
D_x = 1.327 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 47 reflections
θ = 5.0–12.5°
μ = 0.261 mm^{-1}
T = 293 (2) K
Slab
0.76 × 0.36 × 0.32 mm
Colourless

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
8340 measured reflections
6438 independent reflections
2972 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

$\theta_{\max}$ = 27.5°
$h = -1 \rightarrow 11$
$k = -1 \rightarrow 16$
$l = -32 \rightarrow 32$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.103
S = 0.798

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.13 e Å^{-3}
$\Delta\rho_{\min}$ = −0.27 e Å^{-3}
Extinction correction: none

6438 reflections
473 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from
International Tables for Crystallography (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

	(I*A*)	(I*B*)
N1—C4	1.364 (3)	1.364 (3)
N1—C5	1.328 (3)	1.325 (3)
C1—C2	1.403 (3)	1.390 (4)
C1—C18	1.360 (3)	1.363 (4)
C2—C3	1.353 (3)	1.363 (3)
C3—C4	1.411 (3)	1.413 (3)
C4—C17	1.416 (3)	1.417 (3)
C5—C6	1.483 (3)	1.482 (3)
C5—C15	1.427 (3)	1.429 (3)
C6—C7	1.391 (3)	1.391 (3)
C6—C11	1.405 (4)	1.405 (3)
C7—C8	1.383 (4)	1.382 (3)
C8—C9	1.369 (5)	1.375 (4)
C9—C10	1.373 (5)	1.373 (4)
C10—C11	1.390 (4)	1.375 (3)
C11—C12	1.504 (4)	1.507 (3)
C12—C13	1.532 (4)	1.532 (3)
C13—C14	1.530 (4)	1.525 (3)
C14—C15	1.502 (3)	1.504 (3)
C15—C16	1.358 (3)	1.358 (3)
C16—C17	1.413 (3)	1.403 (3)
C17—C18	1.409 (3)	1.413 (3)
C4—N1—C5	118.3 (2)	117.6 (2)
N1—C5—C15	123.3 (2)	123.6 (2)
N1—C5—C6	116.6 (2)	117.3 (2)
C6—C5—C15	119.9 (2)	119.2 (2)
C5—C6—C11	121.0 (2)	120.0 (2)
C5—C6—C7	119.2 (2)	120.9 (2)
C7—C6—C11	119.7 (2)	119.1 (2)
C6—C11—C10	117.8 (2)	118.7 (2)
C10—C11—C12	122.3 (2)	121.1 (2)
C6—C11—C12	119.9 (2)	120.2 (2)
C11—C12—C13	113.7 (2)	112.7 (2)
C12 C13 C14	112.7 (2)	112.6 (2)
C13—C14—C15	113.8 (2)	112.9 (2)
C5—C15—C14	120.6 (2)	119.6 (2)
C14—C15—C16	121.6 (2)	122.5 (2)
C5—C15—C16	117.7 (2)	117.9 (2)
C6—C5—C15—C14	−1.6 (3)	1.6 (3)
C15—C5—C6—C11	44.8 (3)	48.5 (3)
C5—C6—C11—C12	9.0 (4)	2.1 (3)
C6—C11—C12—C13	−75.2 (3)	−73.6 (3)
C11—C12—C13—C14	38.0 (3)	42.2 (3)
C12—C13—C14—C15	47.0 (3)	44.2 (3)
C13—C14—C15—C5	−70.7 (3)	−74.0 (3)

Compound (II)

Crystal data

$C_{19}H_{17}N$
$M_r = 259.34$
Monoclinic
$P2_1/c$
$a = 8.972$ (5) Å
$b = 12.973$ (4) Å
$c = 24.699$ (9) Å
$\beta = 98.41$ (3)°
$V = 2844$ (2) Å^3
$Z = 8$
$D_x = 1.211$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 12 reflections
$\theta = 5.15$–$8.01°$
$\mu = 0.070$ mm^{-1}
$T = 293$ (2) K
Slab
0.64 × 0.40 × 0.14 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5030 measured reflections
3711 independent reflections
1450 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.065$

$\theta_{\rm max} = 22.50°$
$h = -1 \rightarrow 9$
$k = -1 \rightarrow 13$
$l = -26 \rightarrow 26$
3 standard reflections
every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.133$
$S = 0.810$
3710 reflections
361 parameters
H atoms as riding
$w = 1/[\sigma^2(F_o^2) + (0.041P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Extinction correction: none
Scattering factors from
International Tables for Crystallography (Vol. C)

The data collection for (II) was restricted to a 2θ maximum of 45° as there were hardly any observed reflections beyond that range owing to the poor quality of the crystal. The structures were solved by direct methods and refined by full-matrix least-squares techniques. All H atoms in (I) were located from a difference Fourier map and refined isotropically, whereas in (II), they were fixed geometrically and allowed to ride on the atoms to which they are attached.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Geometrical calculations: *PARST* (Nardelli, 1983).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1348). Services for accessing these data are described at the back of the journal.

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