Monoclinic
$P2_1/c$
$a = 8.814\ (1)$ Å
$b = 8.718\ (1)$ Å
$c = 22.125\ (2)$ Å
$\beta = 90.80\ (1)^\circ$
$V = 1699.9\ (3)$ Å^3
$Z = 4$
$D_x = 1.445$ Mg m^{-3}
D_m not measured

Cell parameters from 32 reflections
$\theta = 8$–25°
$\mu = 0.359$ mm^{-1}
$T = 293\ (2)$ K
Needle
0.58 × 0.38 × 0.08 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5267 measured reflections
3908 independent reflections
2924 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$

$\theta_{\rm max} = 27.5^\circ$
$h = -1 \rightarrow 11$
$k = -1 \rightarrow 11$
$l = -28 \rightarrow 28$
3 standard reflections every 97 reflections intensity decay: <4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.111$
$S = 0.962$
3908 reflections
300 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.30$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.30$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

	(I)	(II)
Cl—C20	1.749 (5)	1.741 (2)
S—C12	1.747 (4)	1.733 (2)
S—C1	1.752 (5)	1.740 (2)
C6—C7	1.413 (7)	1.434 (2)
C7—C12	1.379 (6)	1.383 (2)
C7—C8	1.422 (6)	1.420 (2)
C8—N13	1.335 (6)	1.326 (2)
C8—N9	1.377 (6)	1.374 (2)
N9—N15	1.376 (5)	1.374 (2)
N9—C10	1.385 (6)	1.382 (2)
C10—N11	1.303 (6)	1.306 (2)
C10—N16	1.349 (6)	1.358 (2)
N11—C12	1.363 (6)	1.365 (2)
N13—C14	1.352 (7)	1.372 (2)
C14—N15	1.323 (7)	1.317 (2)
C14—C23		1.488 (2)
N16—C17	1.413 (6)	1.412 (2)
C12—S—C1	91.1 (2)	91.20 (8)
C10—N16—C17	129.7 (4)	128.27 (14)

Disorder in the cyclohexene rings was detected during refinement from the large displacement ellipsoids of C3 in (I) and of C3 and C4 in (II). The positional and occupancy parameters of the alternative sites were refined. The displacement parameters of the alternative sites were set equal during the final stages of the refinement. The populations of the major conformers are 94 (1) and 92 (1)% for (I) and (II), respectively. The bond lengths associated with the minor component atoms [C3′ in (I), and C3′ and C4′ in (II)] were made equal to those for the major component atoms using *DFIX* (Sheldrick, 1993).

For both compounds, data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXTL/PC* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993) and *PARST* (Nardelli, 1983); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1295). Services for accessing these data are described at the back of the journal.

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2,12-Dibromo-5,6-dihydrodibenz[*c*,*h*]acridine

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Abstract

The butterfly-like title molecule, $C_{21}H_{13}Br_2N$, is almost planar except for the cyclohexadiene ring which is in a half-chair conformation.

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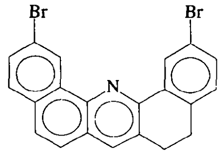
Comment

Macrocyclic polyazaarenes are important host molecules for guest–host complexation (Ray, Roy & Kar, 1996). The dibromo derivative is unique in connecting the two halves to form a macrocyclic network. We have prepared the title compound, (I), from bromonaphthylamine and bromotetralone (Ray & Haldar, 1997), and performed an X-ray study in order to establish the structure and conformation of the molecule.



(I)

The cyclohexadiene ring (C9–C12, C17, C18) adopts a half-chair conformation, with atoms C10 and C11 deviating by 0.298 (5) and −0.357 (6) Å, respectively, from the mean plane formed by the remaining atoms in the ring; the asymmetry parameter ΔC_2(C10—C11) is 0.011 (2)° (Nardelli, 1983*a*). The planarity of the molecule is disturbed by the cyclohexane ring and the two planar segments on either side of it make a dihedral angle of 158.0 (1)°. The aromatic C—C distances vary from 1.339 (8) to 1.474 (5) Å, with an average value of 1.397 (6) Å. The shortest intermolecular methylene C···N contacts are C10···N1($\frac{3}{2}-x$, $y+\frac{1}{2}$, $\frac{1}{2}-z$) 3.638 (6) and C11···N1($\frac{3}{2}-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$) 3.624 (7) Å.

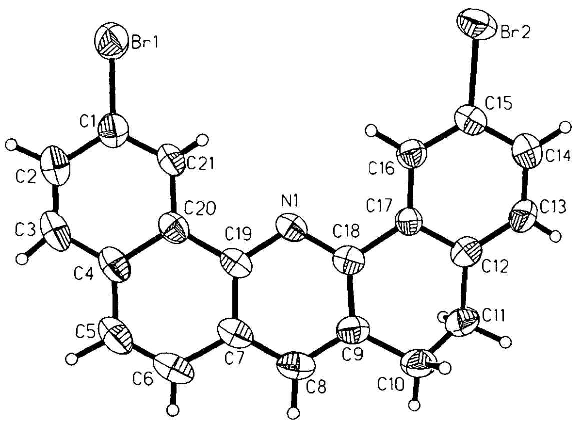


Fig. 1. The structure of title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

Single crystals of the title compound were obtained by slow cooling of a chloroform and petroleum ether solvent system.

Crystal data

$C_{21}H_{13}Br_2N$
M_r = 439.14
Monoclinic
*C*2/*c*
a = 18.262 (2) Å
b = 7.0960 (10) Å
c = 26.315 (2) Å
β = 105.090 (10)°
V = 3292.5 (6) Å^3
Z = 8
D_x = 1.772 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 28 reflections
θ = 5.44–12.47°
μ = 4.925 mm^{-1}
T = 293 (2) K
Slab
0.72 × 0.36 × 0.32 mm
Light yellow

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical ψ scans (*XSCANS*; Siemens, 1994)
$T_{\min}$ = 0.671, $T_{\max}$ = 0.909
4737 measured reflections
3732 independent reflections
1897 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.037
$\theta_{\max}$ = 27.49°
$h = -1 \rightarrow 23$
$k = -1 \rightarrow 9$
$l = -34 \rightarrow 33$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 0.865
3730 reflections
269 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.678 e Å^{-3}
$\Delta\rho_{\min}$ = −0.533 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Br1—C1	1.896 (4)	C8—C9	1.357 (6)
Br2—C15	1.907 (4)	C9—C18	1.407 (5)
N1—C18	1.336 (5)	C9—C10	1.507 (6)
N1—C19	1.354 (4)	C10—C11	1.494 (7)
C1—C21	1.371 (6)	C11—C12	1.497 (6)
C1—C2	1.397 (6)	C12—C13	1.393 (6)
C2—C3	1.347 (7)	C12—C17	1.415 (5)
C3—C4	1.411 (6)	C13—C14	1.359 (6)
C4—C20	1.415 (5)	C14—C15	1.386 (6)
C4—C5	1.427 (6)	C15—C16	1.367 (5)
C5—C6	1.339 (7)	C16—C17	1.392 (5)
C6—C7	1.428 (6)	C17—C18	1.474 (5)
C7—C8	1.400 (6)	C19—C20	1.459 (5)
C7—C19	1.403 (5)	C20—C21	1.394 (6)
C8—C7—C6	123.1 (4)	N1—C18—C9	123.2 (4)
C8—C9—C10	122.2 (4)	N1—C18—C17	118.0 (3)
C11—C10—C9	111.6 (4)	N1—C19—C7	122.7 (4)
C13—C12—C11	122.5 (4)		

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from difference Fourier maps and refined isotropically.

Programs used: data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994); structure solution and molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); structure refinement: *SHELXL*93 (Sheldrick, 1993); geometrical calculations: *PARST* (Nardelli, 1983*b*).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1331). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1619–1620

N-[9-(2-Ethoxycarbonylphenyl)-6-(ethylamino)-2,7-dimethyl-3-xanthenylidene]-ethylammonium Iodide Monohydrate

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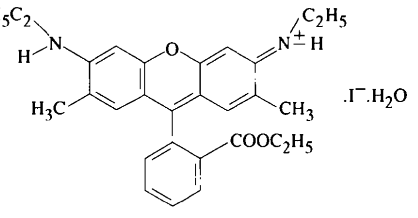
Abstract

The xanthene moiety in the title compound, $C_{28}H_{31}N_2O_3^+.I^-.H_2O$, is planar, with the attached phenyl ring twisted by −78.8 (6)° from the molecular plane. Of the two ethylamino groups, one is coplanar and the other makes a dihedral angle of 152.1 (5)° with the xanthene ring system. The positive charge of the cation is delocalized and both N—C_{sp^2} bonds show double-bond character.

Comment

Rhodamine 6G, with its excellent photophysical properties, has found extensive applications in tunable lasers (Hung & Meyer, 1992; Wittmann, Penzkofer & Baeumler, 1992), fluorescence depolarization diagnostic devices (Herz, 1974), photographic technology (Norland, Ames & Taylor, 1970) and electroluminescent devices (Johnson & McGrane, 1993). Furthermore, it can be used in concentrators of solar diagnostic devices (Batchelder, Zewail & Cole, 1979; Bhownik, Huri & Rohatgi-Mukherjee, 1987). The structure of the dye influences the molecular aggregation and also affects the absorption and emission properties (Ojeda, Katime, Ochoa & Arbeloa, 1988; Arbeloa, Aguirresacona & Arbeloa, 1989). In order to ascertain the structures of rhodamine 6G and its analogous complexes, we obtained crystals of the title compound, (I), from rhodamine 6G and $ZnI_2.6H_2O$ by ion-exchange reaction. To our knowledge, this is the first crystal structure report on a rhodamine derivative.



(I)

A displacement ellipsoid plot of (I) with the atom-numbering scheme is shown in Fig. 1. The xanthene moiety is planar, with the C2 atom showing the largest out-of-plane displacement of 0.075 (5) Å; the phenyl ring is twisted from the xanthene moiety by −78.8 (6)°. While one ethylamino group is almost coplanar with the xanthene moiety, the other makes a dihedral angle of 152.1 (5)° with it. The mean

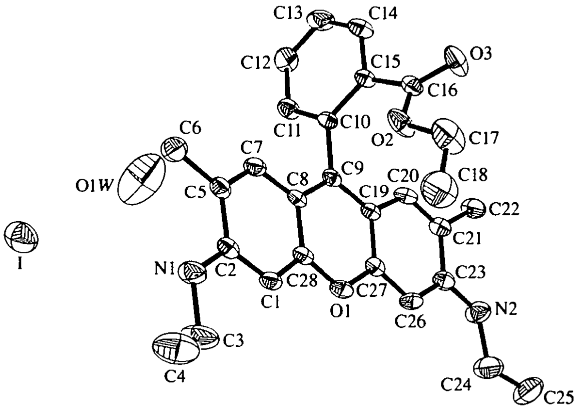


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are not shown.

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