

N2—N1—C1—S2	1.3 (3)
N1—C1—S2—C11	179.9 (2)
S1—C1—S2—C11	-0.1 (2)
C1—S2—C11—C12	-178.0 (2)
S2—C11—C12—C13	82.4 (3)
S2—C11—C12—C17	-97.1 (3)
N2A—N1A—C1A—S2A	-9.4 (4)
N1A—C1A—S2A—C11A	-173.4 (3)
S1A—C1A—S2A—C11A	-3.5 (4)
C1A—S2A—C11A—C12A	-121.6 (7)
S2A—C11A—C12A—C13A	64.9 (8)
S2A—C11A—C12A—C17A	-119.6 (8)
N2A—N1A—C1A—S2B	12.1 (4)
N1A—C1A—S2B—C11B	172.8 (4)
S1A—C1A—S2B—C11B	5.1 (4)
C1A—S2B—C11B—C12B	-176.8 (6)
S2B—C11B—C12B—C13B	97.1 (7)
S2B—C11B—C12B—C17B	-86.0 (8)

Table 3. Dihedral angles ( $^{\circ}$ ) between the planes of various molecular groups

	Molecule 1	Molecule 1A	Molecule 2B
Phenyl/amino	9.8 (2)	10.1 (3)	
Phenyl/carbazate	6.9 (1)	5.1 (1)	8.9 (1)
Phenyl/S-benzyl	82.2 (1)	101.6 (3)	100.4 (3)
Carbazate/S-benzyl	83.5 (1)	98.8 (3)	96.7 (2)

The structure of molecule 1 was solved by direct methods. For the second molecule, the complete structure was not solved by direct methods, due to the disorder, but was completed by locating the missing atoms from successive Fourier maps. Initially, the disorder in molecule 2 led to non-convergence of refinement and unreliable bond lengths and angles. Subsequently, the *S*-benzyl group was split into two sets of atoms and their occupancies were refined at the beginning and fixed at 0.5 (the refined value), resulting in satisfactory convergence and geometric parameters. During refinement, the phenyl rings of both the disordered components of the *S*-benzyl group were restrained as regular hexagons with C—C distances of 1.39 Å. All H atoms were fixed geometrically and not refined, but were allowed to ride on those atoms to which they are attached with a displacement parameter 1.2 times that of the parent atom.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990a). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976), SHELXTL/PC (Sheldrick, 1990b). Software used to prepare material for publication: SHELXL93. Geometric calculations: PARST (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1178). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 14-Methyl-7,14-dihydrodibenzo[*a,j*]acridine

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## Abstract

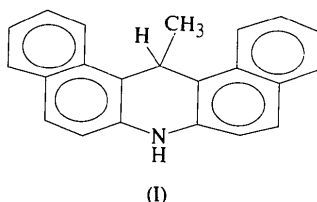
14-Methyl-7,14-dihydrodibenzo[*a,j*]acridine, C<sub>22</sub>H<sub>17</sub>N, has butterfly-like mirror symmetry. The central ring of the acridine moiety adopts a flattened-boat conformation. The crystal structure is stabilized by van der Waals forces.

## Comment

In recent years, cavity-shaped azaarenes have invaded the field of molecular recognition because of their unique structural features that help to hold the planar

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hosts in the cavity by weak van der Waals forces or hydrogen bonding (Bell & Santora, 1992). In an attempt to synthesize monoalkylated polyazaarenes (PAA's) having an alkyl group in the bay region (Kumar, 1985), we developed a direct alkylation technique using alkyl-lithiums (Ray, Roy & Kar, 1994). In order to understand the mechanism of conversion to alkyl azaarenes, we presumed an intermediate acridine derivative. We were able to isolate this intermediate, (I), and carry out an X-ray study in order to establish the structure and conformation of the molecule.



In this symmetric molecule, the mirror plane passes through atoms N1, C12, C23 and H12. The two parts of the molecule are planar and make a dihedral angle of 24.87(4)° with respect to each other. The methyl C atom (C23) and the H atom at C12 occupy axial positions with respect to the plane of the molecule. The flattened-boat conformation of the central ring (N1, C2, C11, C12, C13, C22) of the acridine moiety is due to the geometric constraints of the neighbouring benzene rings; the asymmetry parameter  $\Delta C_s(N1)$  is 0.012(1) (Nardelli, 1983a). The aromatic C—C distances vary from 1.350(3) to 1.433(2) Å, with an average value of 1.396(3) Å, the longer distances being observed at the benzene ring junctions, *i.e.* around atoms C5, C10, C14 and C19. The symmetrically related bond lengths and angles in the molecule are equivalent within agreeable deviations. In general, the bond lengths and angles in this structure show normal values. The shortest intermolecular C...C and N...C contacts are C14...C21(1 - x,  $\frac{1}{2}$  + y,  $\frac{1}{2}$  - z) 3.468(3) and N1...C8(x, y - 1, z) 3.493(3) Å.

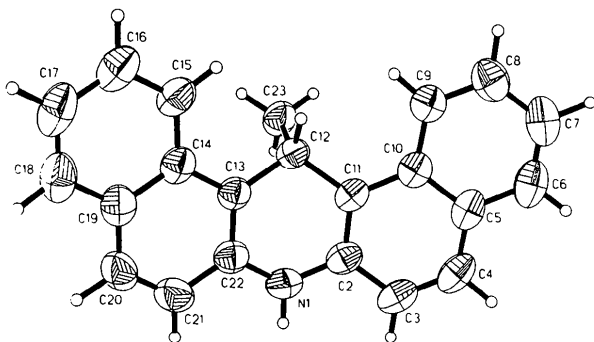


Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

## Experimental

Single crystals of the title compound were obtained by slow evaporation of a mixture of chloroform and petroleum ether (333–353 K).

### Crystal data

C<sub>22</sub>H<sub>17</sub>N  
*M<sub>r</sub>* = 295.37  
 Orthorhombic  
*P*2<sub>1</sub>2<sub>1</sub>  
*a* = 7.815(1) Å  
*b* = 8.116(1) Å  
*c* = 24.972(2) Å  
*V* = 1583.9(3) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.239 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 8–25°  
 $\mu$  = 0.072 mm<sup>-1</sup>  
*T* = 293(2) K  
 Transparent block  
 0.52 × 0.44 × 0.42 mm  
 Light yellow

### Data collection

Siemens *P4* diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction: none  
 2796 measured reflections  
 2098 independent reflections  
 1628 observed reflections  
 $|I| > 2\sigma(I)$   
*R<sub>int</sub>* = 0.0208

$\theta_{\max}$  = 27.49°  
*h* = -1 → 10  
*k* = -1 → 10  
*l* = -1 → 32  
 3 standard reflections monitored every 100 reflections  
 intensity decay: < 3%

### Refinement

Refinement on *F*<sup>2</sup>  
*R*(*F*) = 0.0332  
*wR*(*F*<sup>2</sup>) = 0.1447  
*S* = 1.044  
 2094 reflections  
 277 parameters  
 All H-atom parameters refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 0.0068P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.131 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.098 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL93* (Sheldrick, 1993)  
 Extinction coefficient: 0.0272(32)  
 Atomic scattering factors from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
N1	0.4785 (2)	-0.0465 (2)	0.14513 (7)	0.0609 (4)
C2	0.5331 (2)	0.0808 (2)	0.11219 (7)	0.0521 (5)
C3	0.6715 (3)	0.0536 (3)	0.07629 (9)	0.0631 (5)
C4	0.7280 (3)	0.1755 (3)	0.04408 (8)	0.0652 (6)
C5	0.6525 (2)	0.3338 (3)	0.04532 (7)	0.0567 (5)
C6	0.7105 (4)	0.4639 (4)	0.01228 (9)	0.0746 (7)
C7	0.6354 (4)	0.6145 (3)	0.01356 (10)	0.0789 (7)
C8	0.4991 (3)	0.6427 (3)	0.04863 (9)	0.0696 (6)
C9	0.4394 (3)	0.5225 (3)	0.08147 (8)	0.0562 (5)
C10	0.5135 (2)	0.3619 (2)	0.08141 (7)	0.0490 (4)
C11	0.4540 (2)	0.2327 (2)	0.11478 (7)	0.0464 (4)
C12	0.3013 (2)	0.2558 (2)	0.15152 (7)	0.0478 (4)
C13	0.3082 (2)	0.1363 (2)	0.19807 (7)	0.0478 (4)
C14	0.2264 (2)	0.1732 (3)	0.24800 (7)	0.0519 (4)
C15	0.1453 (2)	0.3262 (3)	0.25851 (8)	0.0607 (5)
C16	0.0719 (3)	0.3574 (4)	0.30698 (9)	0.0735 (6)
C17	0.0742 (3)	0.2396 (4)	0.34776 (9)	0.0790 (7)

C18	0.1506 (3)	0.0918 (4)	0.33955 (8)	0.0724 (7)
C19	0.2270 (3)	0.0534 (3)	0.28979 (7)	0.0595 (5)
C20	0.3036 (3)	-0.1011 (3)	0.27981 (9)	0.0686 (6)
C21	0.3825 (3)	-0.1337 (3)	0.23287 (10)	0.0677 (6)
C22	0.3885 (2)	-0.0132 (2)	0.19209 (8)	0.0539 (5)
C23	0.1331 (3)	0.2341 (3)	0.12036 (9)	0.0615 (5)

Table 2. Selected geometric parameters (Å, °)

N1—C2	1.388 (3)	C12—C13	1.515 (2)
N1—C22	1.394 (3)	C12—C23	1.538 (3)
C2—C11	1.380 (3)	C13—C22	1.374 (3)
C2—C3	1.422 (3)	C13—C14	1.433 (2)
C3—C4	1.350 (3)	C14—C15	1.418 (3)
C4—C5	1.414 (3)	C14—C19	1.426 (3)
C5—C6	1.414 (3)	C15—C16	1.364 (3)
C5—C10	1.430 (3)	C16—C17	1.397 (4)
C6—C7	1.356 (4)	C17—C18	1.355 (4)
C7—C8	1.398 (3)	C18—C19	1.413 (3)
C8—C9	1.357 (3)	C19—C20	1.412 (3)
C9—C10	1.426 (3)	C20—C21	1.350 (3)
C10—C11	1.418 (3)	C21—C22	1.412 (3)
C11—C12	1.517 (2)		
C2—N1—C22	120.6 (2)	C13—C12—C23	110.2 (2)
C11—C2—N1	120.0 (2)	C11—C12—C23	110.6 (1)
C2—C11—C12	119.4 (2)	C22—C13—C12	119.9 (2)
C13—C12—C11	110.9 (2)	C13—C22—N1	119.5 (2)

The H atoms were located from difference Fourier maps and refined isotropically.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990a). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976), SHELXTL/PC (Sheldrick, 1990b). Software used to prepare material for publication: SHELXL93. Geometric calculations: PARST (Nardelli, 1983b).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1170). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Hydrazinium L-Tartrate

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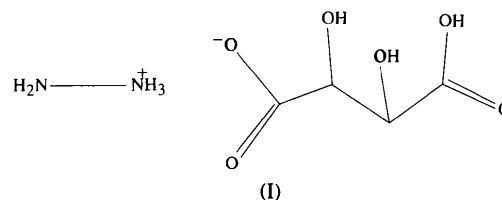
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## Abstract

The crystal structure of  $N_2H_5^+ \cdot C_4H_5O_6^-$  consists of infinite chains of tartrate anions linked head-to-tail by O—H...O hydrogen bonds. Two such chains are cross-connected by O—H...O hydrogen bonds to form dimeric chains. The hydrazinium cations sit at the centre of four such tartrate dimers and bridge them by two-centre and three-centre N—H...O hydrogen bonds. As a whole, the structure is stabilized by numerous hydrogen bonds.

## Comment

The salts of tartaric acid are becoming the latest interest among scientists of various disciplines owing to their applications in diversified fields such as chiral synthesis, crystal engineering, nonlinear optics, solid-state physics and wine-making technology (Aakeroy & Hitchcock, 1994; Zyss, Pecaut, Levy & Masse, 1993; Marshall & Luke, 1992; Aakeroy, Hitchcock & Seddon, 1992). Rare earth tartrates are used as precursors in the synthesis of high  $T_c$  superconductors (Garcia-Jaca *et al.*, 1994). Chiral tartaric acid is a good resolving agent; basic enantiomers with N atoms can easily be turned into salts and resolved *via* recrystallization. Single crystals of the title compound, (I), were obtained when we attempted to resolve the hydrazone from a spirodiacetone derivative.



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