lengths for both axial and equatorial cyclohexyl *p*-nitrobenzoates at low temperature for comparison purposes. Thus *cis* (1) and *trans* (2) 4-*tert*-butylcyclohexyl *p*-nitrobenzoate were prepared by treatment of a *ca* 1:1 mixture of *cis* and *trans* 4-*tert*-butylcyclohexanol (3) (as supplied by Aldrich) with *p*-nitrobenzoyl chloride in the presence of pyridine. The resulting mixture of isomers was separated by chromatography on silica gel. Crystals of (1) and (2) suitable for X-ray analysis were grown from pentane.

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Structure of 4′-Dimethylamino-4-methyl-4-azastilbenium *p*-Toluenesulfonate Hydrate

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Abstract. 4′-Dimethylamino-*N*-methyl-4-stilbazolium tosylate hydrate, $C_{16}H_{19}N_2^+.C_7H_7O_3S^-.H_2O$, M_r = 428.5, triclinic, $P\bar{1}$, a = 8.006 (2), b = 9.548 (2), c = 14.647 (12) Å, α = 80.34 (2), β = 80.30 (2), γ = 77.98 (2)°, V = 1069.2 Å^3, Z = 2, D_x = 1.330 g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ = 1.75 cm^{-1}, $F(000)$ = 456, T = 213 K, R = 0.0649 for 3754 unique reflections with $I > 2\sigma(I)$. The 4-methyl-4′-dimethylamino-4-azastilbenium molecules pack head to tail within a sheet and are aligned in the opposite direction in the neighboring sheets. The phenyl rings in the *p*-toluenesulfonate groups lie at an angle of 72° relative to the aromatic rings in the azastilbenium groups. The axes for both molecules lie along the *a* axis. The rings for the azastilbenium group lie in the *ab* plane and the phenyl ring of the *p*-tolunesulfonate group lies in the *ac* plane.

Experimental. Compound obtained by reaction of one equivalent of γ-picoline and one equivalent of methyl *p*-toluenesulfonate heated at reflux for 1 h in 1 l of ethanol. Treatment with 1.1 equivalents of *N*,*N*-dimethylaminobenzaldehyde and 0.2 equivalents of piperidine yielded dark green crystals of the desired product. Large platelets of the reported crystals were grown by slow evaporation at 300 K from a saturated solution of 4-methyl-4′-dimethylamino-4-azastilbenium *p*-toluenesulfonate and 95% methanol/water solution. The crystal was sealed in a glass capillary for low-temperature data collection. Siemens *R*3*m*/*V* upgrade of Nicolet *P*3*F* automated diffractometer, 2θ–θ scan with variable scan speeds. Structure solved by direct methods and refined on F using the *SHELXTL-Plus* (MicroVAX II) program package (Sheldrick, 1988). H atoms were placed in idealized positions and constrained to have C—H = 0.96 Å and isotropic thermal parameters, U = 0.08 Å^2. All non-H atoms treated as anisotropic. No absorption correction was applied. Details of the data collection are in Table 1.* Scattering factors from *International Tables for X-ray Crystallography*

Table 1. *Experimental details*

Crystal habit and size (mm)	Plate 0.40 × 0.24 × 0.04
Number of reflections, 2θ range (°) for lattice parameters	29, 13.3–36.6
Range of h,k,l	−1 to 10, −12 to 12, −18 to 19
Max. $\sin\theta/\lambda$ (Å^{-1})	0.650
Check reflections	$1\bar{1}\bar{3}$, 104, $31\bar{3}$
% variation	5, 5, 3
Reflections collected	4932
Unique observed reflections	3754
R_{int}	0.014
Observed criterion	$I > 2\sigma(I)$
Number of parameters	272
R	0.065
wR	0.070
S	1.34
Secondary-extinction parameter (χ) $F^* = F[1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$	0.0007 (4)
Weighting factor (g), $w^{-1} = \sigma^2(F) + gF^2$	0.0011
Fourier difference peaks, max., min. (e Å^{-3})	0.47, −0.43
Max. Δ/σ	0.002

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55456 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0417]

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Table 2. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($Å^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	−1823 (3)	3238 (3)	8589 (2)	21 (1)
C(2)	−3553 (4)	3354 (4)	9207 (2)	31 (1)
C(3)	−1732 (4)	3398 (3)	7621 (2)	23 (1)
C(4)	−154 (3)	3282 (3)	7052 (2)	21 (1)
C(5)	1370 (3)	2989 (3)	7452 (2)	17 (1)
C(6)	1291 (3)	2849 (3)	8410 (2)	19 (1)
C(7)	−301 (4)	2972 (3)	8972 (2)	22 (1)
S(1)	3381 (1)	2784 (1)	6716 (1)	19 (1)
O(1)	3281 (3)	1797 (2)	6082 (2)	29 (1)
O(2)	3519 (3)	4236 (2)	6222 (2)	27 (1)
O(3)	4683 (2)	2231 (2)	7332 (2)	27 (1)
O(4)	6834 (3)	5026 (2)	5680 (2)	28 (1)
C(8)	3694 (3)	−2213 (3)	7744 (2)	20 (1)
C(9)	2745 (3)	−953 (3)	8098 (2)	20 (1)
C(10)	1050 (3)	−862 (3)	8504 (2)	21 (1)
C(11)	198 (3)	−2038 (3)	8579 (2)	18 (1)
N(1)	−1507 (3)	−1938 (3)	8949 (2)	23 (1)
C(12)	−2284 (4)	−3217 (3)	9141 (2)	26 (1)
C(13)	−2505 (4)	−597 (3)	9237 (2)	29 (1)
C(14)	1156 (3)	−3325 (3)	8260 (2)	21 (1)
C(15)	2854 (3)	−3389 (3)	7850 (2)	22 (1)
C(16)	5436 (3)	−2311 (3)	7249 (2)	21 (1)
C(17)	6343 (3)	−1236 (3)	7047 (2)	20 (1)
C(18)	8102 (3)	−1371 (3)	6571 (2)	19 (1)
C(19)	9015 (4)	−258 (3)	6539 (2)	21 (1)
C(20)	10701 (4)	−370 (3)	6129 (2)	21 (1)
N(2)	11498 (3)	−1545 (3)	5728 (2)	21 (1)
C(21)	13318 (3)	−1665 (4)	5284 (2)	30 (1)
C(22)	10650 (4)	−2636 (3)	5723 (2)	25 (1)
C(23)	8978 (4)	−2578 (3)	6130 (2)	23 (1)

Table 3. *Selected torsion angles* (°)

C(14)—C(11)—N(1)—C(12)	8.8 (4)	C(16)—C(17)—C(18)—C(23)	10.5 (5)
C(15)—C(8)—C(16)—C(17)	175.5 (3)	C(6)—C(5)—S(1)—O(3)	9.6 (3)
C(8)—C(16)—C(17)—C(18)	178.4 (3)		

(1974, Vol. IV). Atomic coordinates are contained in Table 2. The C—C distances for the phenyl groups range between 1.378 and 1.412 Å. The cation molecule is planar with a mean deviation of 0.081 Å from the 18-atom least-squares plane. Selected torsion angles are in Table 3. Fig. 1 illustrates the molecule with the atomic numbering scheme employed. Fig. 2 illustrates the projected packing of the molecules viewed down the *a* axis.

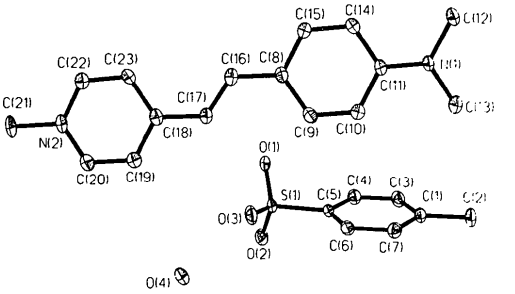

Fig. 1. Thermal-ellipsoid (50% probability) plot showing the atomic numbering scheme.

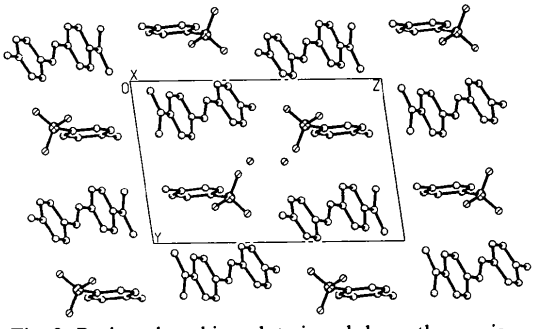

Fig. 2. Projected packing plot viewed down the *a* axis.

Related literature. For additional information on related structures and chemistry, see Williams (1983) and Marder, Perry & Schaefer (1989).

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Structure of 17-(3-Oxazolin-4-yl)androsta-4,16-dien-3-one

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Abstract. 17-(2,5-Dihydro-3-oxazol-4-yl)androsta-4,16-dien-3-one, $C_{22}H_{29}NO_2$, $M_r = 339.48$, orthorhombic, $P2_12_12_1$, $a = 7.715$ (5), $b = 12.033$ (1), $c = 19.199$ (1) Å, $V = 1782.3$ (1) Å^3, $Z = 4$, $D_x = 1.265$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0.71073 Å, $\mu = 0.7$ cm^{-1}, $F(000) = 736$, $T = 130$ K, $R(F) = 0.044$ for 1984

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