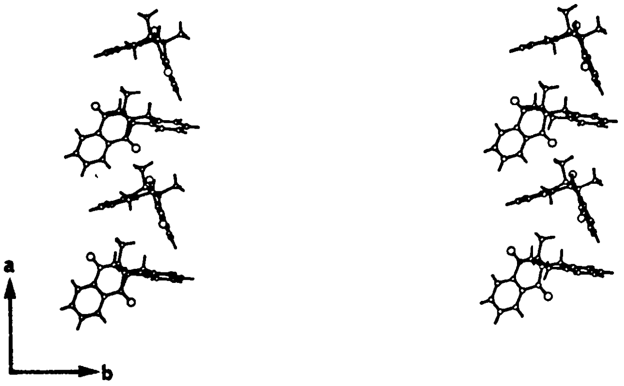

Fig. 2. Stereodiagram of molecular stack consisting of alternating *A* and *B* molecules.

consisting of alternating *A* and *B* molecules (Fig. 2). A notable feature is that a methyl group of a lower molecule projects directly into the space between the two aromatic rings of an upper molecule. Cyclopropanol formation requires that the two aromatic rings move considerably closer together, and this is prevented by the presence of the methyl group. The γ-H-atom abstraction by an enone C atom [C(2) or C(3)] observed for compound (II) is not possible for compound (I) since C(2) and C(3) are part of an aromatic ring system.

Intermolecular distances correspond to van der Waals contacts. The independent molecules are related by approximate $P2_1/b11$ symmetry, with a pseudo screw axis parallel to **a** at $y\ \frac{1}{4}$, z 0, and pseudo b-glide plane perpendicular to **a** at x 0·275.

We thank Professor J. R. Scheffer and Mr S. Askari for their collaborative studies on the solid-state photochemistry, the Natural Sciences and Engineering Research Council of Canada for financial support, and the University of British Columbia Computing Centre for assistance.

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Acta Cryst. (1986). C**42**, 1807–1809

Structure of 2-(*p*-Toluenesulfonyl)-1,2,3,4-tetrahydroisoquinoline

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(*Received* 28 *March* 1986; *accepted* 30 *June* 1986)

Abstract. $C_{16}H_{17}NO_2S$, $M_r = 287{\cdot}4$, monoclinic, $P2_1$, $a = 8{\cdot}234\ (2)$, $b = 6{\cdot}135\ (1)$, $c = 14{\cdot}479\ (2)$ Å, $\beta = 94{\cdot}56\ (1)°$, $V = 729{\cdot}1\ (2)$ Å^3, $Z = 2$, $D_m = 1{\cdot}24$, $D_x = 1{\cdot}31$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo } K\alpha) = 2{\cdot}11$ mm^{-1}, $F(000) = 304$, room temperature, $R = 0{\cdot}065$ for 1209 observed reflexions. The heterocyclic fragment of the molecule exhibits a half-chair conformation. The N atom shows considerable pyramidization [sum of valency angles = 343·2 (9)°]. Bond lengths and angles in the aromatic rings do not deviate significantly from standard values.

Introduction. This work is a continuation of our studies of the structures of Reissert compounds (Reissert, 1905) and their analogs. The present compound is a product of reductive decyanation of the corresponding Reissert analog in alkaline solution.

Experimental. Crystals obtained from methanol, colorless prisms. Space group from Weissenberg photographs. Crystal 0·3 × 0·4 × 0·6 mm. D_m by flotation. Syntex $P2_1$ diffractometer, θ–2θ scan. Cell parameters from least-squares treatment of setting angles of 15 reflexions with $15 \le 2\theta \le 20°$. No absorption or extinction correction. Profile analysis

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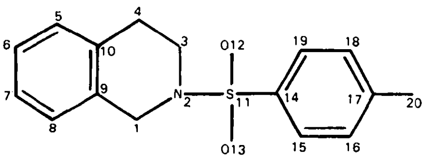
according to Lehmann & Larsen (1974). Max. $\theta = 48°$, 1294 reflexions measured in range h $-9\rightarrow+9$, k $0\rightarrow+6$, l $0\rightarrow+16$. No significant intensity variation for two standard reflexions recorded every 1·5 h, 1209 observed reflexions with $I \geq 2\sigma(I)$. Structure solved by *DIRDIF* (Beurskens *et al.*, 1982). Full-matrix least-squares refinement on F, $w^{-1} = \sigma^2(F)$, H atoms from molecular geometry included with fixed isotropic thermal parameters in F_c, anisotropic thermal parameters for non-H atoms. $R = 0{\cdot}065$, $wR = 0{\cdot}058$, $S = 4{\cdot}3$, $(\Delta/\sigma)_{\max} = 0{\cdot}2$, $(\Delta\rho)_{\max} = 0{\cdot}30$, $(\Delta\rho)_{\min} = -0{\cdot}30$ e Å^{-3}. Computer programs: *SHELX76* (Sheldrick, 1976), *DIRDIF* (Beurskens *et al.*, 1982) and local programs (Jaskólski, 1982). Molecular illustrations drawn using *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates and bond lengths and angles are given in Tables 1 and 2, respectively.[*]

The labeling sequence is shown in the formula below, and a stereoscopic view of the molecule is presented in Fig. 1. The molecule consists of an aromatic ring condensed with the heterocyclic hydrogenated ring and linked through the N atom to the toluenesulfonyl group. Bond lengths and angles in the aromatic rings do not deviate significantly from standard values, and the rings are almost perpendicular to each other [dihedral angle 89·0 (6)°].



The C(9)—C(1)—N(2) angle is tetrahedral [109·7 (5)°] in contrast to the value of 114·4 (2)° found in 2-benzoyl-1,2,3,4-tetrahydro-1-isoquinolinecarbonitrile (Pływaczyk, Tykarska, Jaskólski & Kosturkiewicz, 1984). The sum of valency angles around the N atom is 343·9 (9)° showing its considerable pyramidization. The average of the absolute values of the torsion angles around the heterocyclic ring is 37·5°.

The C(1)—C(9)—C(10)—C(4) angle is equal to −3·7 (8)° and the remaining torsion angles in the heterocyclic ring are consistent with a half-chair conformation. The corresponding asymmetry parameter (Duax & Norton, 1975) is $\Delta C_2^{2,3} = 9{\cdot}24°$. The present conformation is similar to that found in the ethoxy derivative of the Reissert analog (Gzella, Jaskólski, Rychlewska & Kosturkiewicz, 1984) but is different from the sofa conformation of the benzoyl derivative (Pływaczyk *et al.*, 1984). The sulfonyl S—O bond lengths [1·445 (6) and 1·437 (6) Å] are comparable with those [1·432 (6) and 1·410 (6) Å] in the tosyl-L-arginine methyl ester (Barrans & Cotrait, 1976), but shorter than those in *p*-toluenesulfonic acid [1·450–1·457 (2) Å] reported by Arora & Sundarlingam

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{\rm eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	$U_{\rm eq}$
C(1)	0·3295 (8)	−0·001 (2)	0·1124 (4)	0·054 (3)
N(2)	0·2136 (7)	0·108 (1)	0·1681 (4)	0·047 (3)
C(3)	0·212 (1)	0·345 (2)	0·1530 (5)	0·058 (4)
C(4)	0·1351 (9)	0·389 (2)	0·0555 (5)	0·062 (4)
C(5)	0·170 (1)	0·285 (2)	−0·1108 (6)	0·068 (4)
C(6)	0·221 (1)	0·147 (2)	−0·1778 (7)	0·073 (4)
C(7)	0·307 (1)	−0·040 (2)	−0·1507 (6)	0·073 (5)
C(8)	0·3421 (8)	−0·083 (2)	−0·0569 (5)	0·068 (4)
C(9)	0·2894 (8)	0·053 (2)	0·0098 (5)	0·048 (3)
C(10)	0·2024 (9)	0·240 (2)	−0·0159 (6)	0·053 (3)
S(11)	0·2163 (3)	0·0257	0·2779 (1)	0·0596 (9)
O(12)	0·0807 (6)	0·136 (1)	0·3147 (3)	0·070 (2)
O(13)	0·2251 (7)	−0·208 (1)	0·2774 (4)	0·062 (3)
C(14)	0·3979 (9)	0·132 (2)	0·3355 (5)	0·048 (3)
C(15)	0·5410 (9)	0·018 (2)	0·3346 (4)	0·052 (3)
C(16)	0·684 (1)	0·103 (2)	0·3740 (5)	0·062 (4)
C(17)	0·687 (1)	0·311 (2)	0·4155 (5)	0·059 (4)
C(18)	0·541 (1)	0·423 (2)	0·4149 (5)	0·057 (3)
C(19)	0·3938 (9)	0·337 (1)	0·3749 (5)	0·046 (3)
C(20)	0·8454 (9)	0·410 (3)	0·4598 (6)	0·084 (4)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—N(2)	1·459 (10)	N(2)—S(11)	1·666 (6)
N(2)—C(3)	1·469 (12)	S(11)—O(12)	1·443 (6)
C(3)—C(4)	1·524 (11)	S(11)—O(13)	1·437 (7)
C(4)—C(10)	1·517 (13)	S(11)—C(14)	1·778 (8)
C(10)—C(9)	1·388 (14)	C(14)—C(15)	1·371 (12)
C(5)—C(6)	1·378 (15)	C(15)—C(16)	1·369 (12)
C(9)—C(1)	1·533 (10)	C(16)—C(17)	1·410 (16)
C(5)—C(10)	1·407 (12)	C(17)—C(18)	1·383 (13)
C(6)—C(7)	1·389 (19)	C(18)—C(19)	1·404 (11)
C(7)—C(8)	1·393 (11)	C(19)—C(14)	1·382 (13)
C(8)—C(9)	1·373 (13)	C(17)—C(20)	1·531 (13)
C(9)—C(1)—N(2)	109·7 (5)	N(2)—S(11)—O(13)	107·1 (4)
C(1)—N(2)—C(3)	111·7 (7)	O(12)—S(11)—O(13)	120·8 (4)
C(1)—N(2)—S(11)	115·4 (4)	O(13)—S(11)—C(14)	109·1 (4)
C(3)—N(2)—S(11)	116·1 (5)	O(12)—S(11)—C(14)	107·5 (3)
N(2)—C(3)—C(4)	108·2 (7)	N(2)—S(11)—C(14)	106·5 (3)
C(3)—C(4)—C(10)	112·1 (6)	S(11)—C(14)—C(19)	118·7 (6)
C(4)—C(10)—C(9)	121·6 (7)	S(11)—C(14)—C(15)	120·0 (7)
C(10)—C(9)—C(1)	120·5 (7)	C(14)—C(15)—C(16)	120·6 (7)
C(10)—C(9)—C(8)	119·9 (8)	C(15)—C(16)—C(17)	120·7 (8)
C(5)—C(10)—C(9)	118·8 (7)	C(16)—C(17)—C(18)	117·2 (8)
C(6)—C(5)—C(10)	121·4 (8)	C(17)—C(18)—C(19)	122·5 (7)
C(5)—C(6)—C(7)	119·1 (9)	C(18)—C(19)—C(14)	117·7 (4)
C(6)—C(7)—C(8)	119·7 (9)	C(19)—C(14)—C(15)	121·3 (7)
C(7)—C(8)—C(9)	121·1 (8)	C(20)—C(17)—C(16)	121·6 (8)
N(2)—S(11)—O(12)	105·0 (3)	C(20)—C(17)—C(18)	121·1 (8)

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[*] Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43206 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

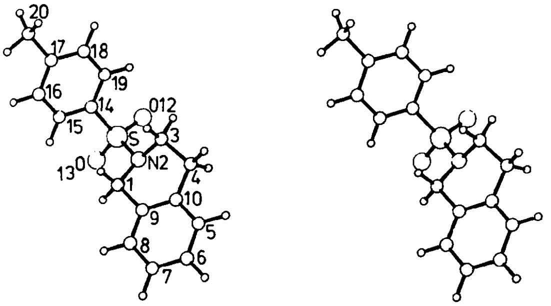

Fig. 1. Stereodrawing of the molecule (*PLUTO*; Motherwell & Clegg, 1978).

(1971). The C—S distance [1·778 (8) Å] is longer than the 1·753 (2) Å in *p*-toluenesulfonic acid and the 1·755 (3) Å found by Singh, Tiwari & Singh (1985), but similar to the 1·766 (7) Å found by Barrans & Cotrait (1976).

The N—S bond length [1·666 (6) Å] is comparable with the values in the above cited structures. The O—S—O angle in the present structure is 120·8 (4)°. In *p*-toluenesulfonic acid and in a number of sulfonate derivatives (Arora & Sundarlingam, 1971) it ranges from 111 to 114°. In N^1-(2,6-dimethyl-4-pyrimidinyl)-sulfanilamide (Singh *et al.*, 1985) it equals 115·0 (4)° while in tosyl-L-arginine methyl ester (Barrans & Cotrait, 1976) it is widened to 121·2 (4)°.

There are no unusual intermolecular contacts in the structure.

The authors are indebted to Dr D. Brózda for providing a sample of the compound. The work was supported by Project CPBP.01.12.10.3.(PAN).

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Structures of Five *trans*-2-Hydroxy and *trans*-2-Methoxy-2-methyl-3,4-dihydro-4-aryl-2*H*,5*H*-pyrano[3,2-*c*]benzopyran-5-ones

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(*Received* 15 *February* 1986; *accepted* 4 *July* 1986)

Abstract. Derivatives of 2-methyl-3,4-dihydro-2*H*,5*H*-pyrano[3,2-*c*][1]benzopyran-5-one. (1) Racemic *trans*-2-methoxy-4-phenyl, $M_r = 322{\cdot}4$, monoclinic, $P2_1/n$, $a = 5{\cdot}858\ (1)$, $b = 16{\cdot}732\ (9)$, $c = 16{\cdot}383\ (9)$ Å, $\beta = 94{\cdot}82\ (3)°$, $V = 1600{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}338$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}866$ cm^{-1}, $F(000) =$

0108-2701/86/121809-05$01.50