

Bertolasi (1989). For a related compound, 1,2,7a-trihydroxy-2-methylperhydro-1-phosphaindene 1-oxide, see Bartczak & Yagbasan (1991).

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3-Chloro-6-methoxy-5-tosylmethylpyridazine

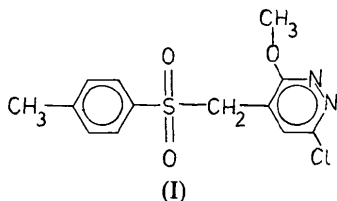
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Abstract. C₁₃H₁₃ClN₂O₃S, *M_r* = 312.77, monoclinic, *P*2₁/*c*, *a* = 12.365 (2), *b* = 5.524 (1), *c* = 21.646 (4) Å, β = 106.98 (1)°, *V* = 1414.0 (4) Å³, *Z* = 4, *D_x* = 1.47 (1) g cm⁻³, λ(Cu *Kα*) = 1.54178 Å, μ = 38.67 cm⁻¹, *F*(000) = 648, *T* = 293 K, *R* = 0.049, *wR* = 0.049 for 1470 unique observed reflections. The conformation of the molecule as a whole can be described as *cis*-staggered. The tosylmethyl substituent is at C(5) in the pyridazine ring. The C(phenyl)—S—CH₂—C(pyridazine) torsion angle is 62.5 (2)°.

Experimental. Crystals of the title compound (I) were obtained from ethanol solution as small colourless elongated prisms. The crystal selected for data collec-



tion had dimensions 0.37 × 0.18 × 0.05 mm. Measurements were carried out on a Syntex P2₁ diffractometer using graphite-monochromated Cu *Kα* radiation. The unit-cell parameters were determined by least-squares fit to ten automatically centred reflections (2θ range 12.90–27.74°). Two control reflections were monitored after every 100 intensity measurements and showed no systematic variation in intensity throughout the data collection. The θ–2θ scan method with a variable scan speed (1.9–29.3° min⁻¹) was used. 2336 reflections were col-

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lected up to 2θ = 115.0°, with indices *h* 0 → 14, *k* 0 → 7, *l* -24 → 24. The background and integrated intensity for each reflection were calculated according to the profile-analysis method of Lehmann & Larsen (1974). Of 1874 unique reflections, 1471 reflections had *I* ≥ 1.96σ(*I*) and were used in the structure refinement. Only *L_p* corrections were applied. The structure was solved using *SHELXS86* (MS DOS version) (Sheldrick, 1986). After the refinement of the non-H atoms with anisotropic temperature factors (*R* = 0.073) eight H atoms (of the two methyl groups and the methylene) were calculated from the geometry of the molecule (C—H = 1.08 Å) and assigned isotropic temperature factors of 0.06 Å² — their coordinates were recalculated after each cycle of refinement following the shifts of their carriers. The other H atoms were located from a Δ*F* map. They were included in the refinement with isotropic temperature factors. The function minimized was Σ*w*(|*F_o*| - |*F_c*|)² where *w* = [σ²(*F_o*)]⁻¹. The final cycles of the full-matrix least-squares refinement gave *R* = 0.049, *wR* = 0.049 and *S* = 1.7, for 1470 reflections — one reflection with a large Δ*F*/σ value was excluded from the last cycles. For all the refined parameters the final Δ/σ was smaller than 0.1; the maximum peak on the final Δ*F* map was 0.31, the minimum -0.32 e Å⁻³. The final atomic parameters are listed in Table 1.* Atomic scattering factors incorporated in *SHELX76* (Sheldrick, 1976) were

* Lists of structure factors, anisotropic thermal parameters and H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55021 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (\AA^2) of the non-H atoms
$$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$$

	x	y	z	U_{eq}
O(1)	0.8139 (3)	0.5072 (6)	0.1567 (1)	0.047
C(1)	0.9045 (5)	0.5844 (12)	0.2120 (3)	0.062
C(6)	0.8350 (4)	0.3078 (9)	0.1248 (2)	0.040
N(1)	0.9369 (3)	0.2168 (8)	0.1428 (2)	0.048
N(2)	0.9595 (3)	0.0231 (8)	0.1091 (2)	0.054
C(3)	0.8776 (4)	-0.0562 (9)	0.0608 (2)	0.049
Cl(1)	0.9133 (1)	-0.2982 (3)	0.0188 (1)	0.077
C(4)	0.7680 (4)	0.0294 (9)	0.0412 (2)	0.041
C(5)	0.7435 (4)	0.2241 (9)	0.0742 (2)	0.038
C(2)	0.6279 (3)	0.3324 (8)	0.0582 (2)	0.038
S(1)	0.5377 (1)	0.1712 (2)	0.0953 (1)	0.041
O(2)	0.5405 (3)	-0.0830 (6)	0.0792 (1)	0.050
O(3)	0.4311 (2)	0.2934 (7)	0.0780 (2)	0.052
C(7)	0.6030 (4)	0.2028 (8)	0.1792 (2)	0.038
C(8)	0.6760 (4)	0.0255 (10)	0.2114 (2)	0.047
C(9)	0.7284 (5)	0.0504 (11)	0.2779 (2)	0.054
C(10)	0.7059 (4)	0.2487 (10)	0.3106 (2)	0.050
C(11)	0.6324 (5)	0.4236 (11)	0.2770 (3)	0.058
C(12)	0.5795 (4)	0.4035 (10)	0.2106 (2)	0.050
C(13)	0.7601 (6)	0.2752 (16)	0.3831 (2)	0.074

Table 2. Bond lengths (\AA), valency angles ($^\circ$) and selected torsion angles ($^\circ$)

O(1)—C(1)	1.444 (6)	O(1)—C(6)	1.365 (6)
C(6)—N(1)	1.306 (6)	C(6)—C(5)	1.403 (6)
N(1)—N(2)	1.369 (6)	N(2)—C(3)	1.301 (5)
C(3)—Cl(1)	1.744 (5)	C(3)—C(4)	1.380 (7)
C(4)—C(5)	1.373 (7)	C(4)—H(4)	0.916 (35)
C(5)—C(2)	1.494 (6)	C(2)—S(1)	1.790 (5)
S(1)—O(2)	1.450 (4)	S(1)—O(3)	1.430 (3)
S(1)—C(7)	1.768 (4)	C(7)—C(8)	1.376 (7)
C(7)—C(12)	1.375 (7)	C(8)—C(9)	1.402 (6)
C(8)—H(8)	1.022 (5)	C(9)—C(10)	1.376 (8)
C(9)—H(9)	1.002 (5)	C(10)—C(13)	1.523 (6)
C(10)—C(11)	1.379 (8)	C(11)—C(12)	1.399 (7)
C(11)—H(11)	0.918 (5)	C(12)—H(12)	1.049 (54)
C(1)—O(1)—C(6)	116.2 (4)	O(1)—C(6)—C(5)	115.5 (4)
O(1)—C(6)—N(1)	118.0 (4)	N(1)—C(6)—C(5)	126.4 (5)
C(6)—N(1)—N(2)	118.1 (4)	N(1)—N(2)—C(3)	117.3 (4)
N(2)—C(3)—C(4)	126.6 (4)	N(2)—C(3)—Cl(1)	114.9 (4)
Cl(1)—C(3)—C(4)	118.5 (4)	C(3)—C(4)—C(5)	117.0 (4)
C(6)—C(5)—C(4)	114.5 (4)	C(4)—C(5)—C(2)	122.3 (4)
C(6)—C(5)—C(2)	123.2 (4)	C(5)—C(2)—S(1)	112.1 (3)
C(2)—S(1)—C(7)	104.7 (2)	C(2)—S(1)—O(3)	107.2 (2)
C(2)—S(1)—O(2)	108.2 (2)	O(3)—S(1)—C(7)	109.4 (2)
O(2)—S(1)—C(7)	107.9 (2)	O(2)—S(1)—O(3)	118.6 (2)
S(1)—C(7)—C(12)	119.2 (3)	S(1)—C(7)—C(8)	119.1 (3)
C(8)—C(7)—C(12)	121.7 (4)	C(7)—C(8)—C(9)	119.2 (5)
C(8)—C(9)—C(10)	120.3 (4)	C(9)—C(10)—C(11)	119.2 (5)
C(9)—C(10)—C(13)	120.8 (5)	C(13)—C(10)—C(11)	120.0 (5)
C(10)—C(11)—C(12)	121.6 (5)	C(7)—C(12)—C(11)	118.0 (5)
C(1)—O(1)—C(6)—N(1)	-5.9 (6)	C(5)—C(2)—S(1)—O(3)	178.7 (3)
C(1)—O(1)—C(6)—C(5)	175.4 (4)	C(5)—C(2)—S(1)—C(7)	62.5 (4)
O(1)—C(6)—C(5)—C(2)	-4.0 (7)	C(2)—S(1)—C(7)—C(8)	-93.8 (4)
N(1)—C(6)—C(5)—C(4)	-1.0 (7)	C(2)—S(1)—C(7)—C(12)	86.3 (4)
C(5)—C(6)—N(1)—N(2)	1.2 (7)	O(3)—S(1)—C(7)—C(8)	151.7 (4)
C(6)—N(1)—N(2)—C(3)	0.6 (7)	O(2)—S(1)—C(7)—C(8)	21.3 (5)
C(4)—C(5)—C(2)—S(1)	82.9 (5)	O(3)—S(1)—C(7)—C(12)	-28.3 (5)
C(6)—C(5)—C(2)—S(1)	-95.5 (5)	O(2)—S(1)—C(7)—C(12)	-158.6 (4)
C(5)—C(2)—S(1)—O(3)	78.7 (3)		

applied. Most of the calculations were performed with *SHELX76* on an IBM PC-XT TURBO computer.

Related literature. The interest in pyridazine derivatives in pharmacological chemistry dates from the early 1950's when their biological properties were discovered (Druey, Meier & Eichenberger, 1954).

Pyridazine derivatives have also been found in antibiotics produced by *Streptomyces Jamaicensis* (Hassall, Morton, Ogihara & Phillips, 1971). The compound whose structure is presented here was synthesized and investigated spectroscopically by Ostrowicz (1986). The molecular conformation and labelling of the atoms are presented in Fig. 1. The bond distances and valency angles are listed in Table 2. They are in good agreement with the expected values for this molecule. Selected torsion angles are also given in Table 2. The conformation of the molecule can be conveniently described by three torsion angles measured along the bonds bridging the phenyl and pyridazine rings, and is *cis*-staggered with respect to the positions of the rings. It is difficult to specify any reasons why this flexible molecule assumes this shape in the crystal structure. Presumably the main role played here is by the intramolecular interactions with the O atoms at S(1) and the H atoms at C(2): short intramolecular contacts O(3)⋯H(12) of 2.62, O(2)⋯H(8) of 2.58, O(2)⋯H(4) of 3.00 and H(21)⋯O(1) of 2.40 Å are observed. There are no short intramolecular contacts between the atoms of the benzene and pyridazine rings. The benzene and pyridazine rings are planar within experimental error. The arrangement of the molecules in the crystal lattice is shown in Fig. 2. No intermolecular distances shorter than van der Waals contacts are present in this structure.

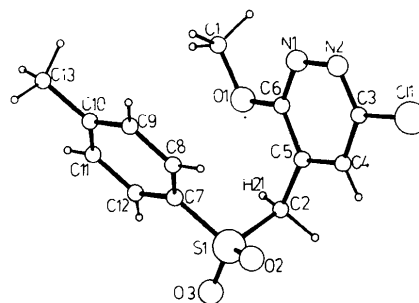


Fig. 1. A perspective drawing of the molecule of (I) (Motherwell & Clegg, 1978).

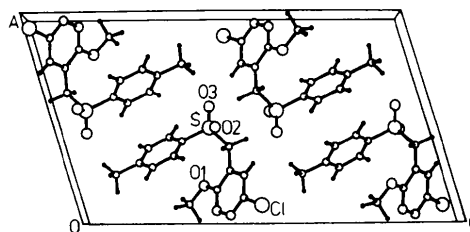


Fig. 2. The arrangement of the molecules in the crystal lattice (Motherwell & Clegg, 1978).

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Molecular Recognition in Amides. Structure of *N,N'*-Di(triphenylmethyl)urea–*N*-Acetylmethionine Ethyl Ester (1/1)

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Abstract. C₃₉H₃₂N₂O.C₉H₁₇NO₃S, *M_r* = 763.99, orthorhombic, *P*2₁2₁2₁, *a* = 17.108 (4), *b* = 19.415 (5), *c* = 24.066 (5) Å, *V* = 7993.6 Å³, *Z* = 8, *D_x* = 1.270 g cm⁻³, Mo *Kα* radiation, λ = 0.7107 Å, μ = 1.23 cm⁻¹, *F*(000) = 3248, *T* = 128 K, final *R* = 0.054 and *wR* = 0.059 for 5563 observed unique reflections. The results illustrate the excellent functional complementarity between coplanar amide groups of host and guest for an effective interaction and favourable guest inclusion.

Experimental. The title compound was prepared by H. Hart and co-workers (Michigan State University) by dissolving the host and guest components in hot ethyl acetate, and subsequent crystallization by gradual cooling. Crystal size 0.4 × 0.3 × 0.2 mm; cell dimensions determined from setting angles of 15 reflections in the range 10 < θ < 14°; space group derived by systematic absences. Data measured at low temperature on an upgraded Picker diffractometer with graphite-monochromated Mo *Kα* radiation, θ_{max} = 27° (sinθ/λ < 0.64 Å⁻¹), ω–2θ scans, constant scan speed 3° min⁻¹, *h* = 0 to 21, *k* = 0 to 24, *l* = 0 to 30. Three reflections monitored periodically during data collection indicated no crystal deterioration. 8111 unique reflections were measured of which 5563 with *I* > 3σ(*I*) were used in refinement. No corrections for absorption or secondary extinction were applied.

The structure was solved by direct methods (*MULTAN80*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), and refined

by block-diagonal least squares [using an extensively modified version of *ORFLS* (Busing, Martin & Levy, 1962)] on *F*, including positional and anisotropic thermal parameters of all the non-H atoms. H atoms involved in hydrogen bonds were located in difference Fourier maps. The remaining H atoms were introduced in calculated positions, the methyls being treated as rigid groups. Minimization of *w*(Δ*F*)², with *w* = 1/[σ²(*F*) + 0.002*F*²], converged at *R* = 0.056, *wR* = 0.059, *S* = 1.56. Final Δ/σ < 0.36, residual densities in Fourier maps ranging from –0.29 to 0.26 e Å⁻³. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Atomic parameters are listed in Table 1; bond distances and bond angles (Nardelli, 1983) are presented in Table 2. The molecular structure and atom-labelling scheme are shown in Fig. 1 (Johnson, 1976).*

Related literature. Structural features of the title compound have already been described elsewhere (Goldberg, 1988, 1991). The hydrogen-bond-directed molecular-recognition features of *N*-tritylurea and *N,N*-ditritylurea (Goldberg, Lin & Hart, 1985; Hart, Lin & Goldberg, 1986; Hart, Lin, Ng, Ward,

* Lists of anisotropic thermal parameters, H-atom coordinates and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55019 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.