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Structure of a Cytochalasin Model Compound

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Abstract. 7a-Acetoxy-3-benzyl-7-methoxy-4-methyl-*N*-tosylperhydroisoindol-1,5-dione (3-benzyl-7-methoxy-4-methyl-1,5-dioxo-*N*-tosyl-7a-isoindolidinyl acetate), $C_{26}H_{29}NO_7S$, $M_r = 499{\cdot}58$, monoclinic, $P2_1/a$, $a = 12{\cdot}903\,(1)$, $b = 12{\cdot}530\,(1)$, $c = 15{\cdot}818\,(2)$ Å, $\beta = 99{\cdot}65\,(1)°$, $V = 2521{\cdot}2\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}316$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0{\cdot}70930$ Å, $\mu = 1{\cdot}65$ cm^{-1}, $F(000) = 1056$, $T = 295$ K, $R = 0{\cdot}036$ for 3188 reflections. The molecule contains a five-membered N-containing ring, slightly distorted from planarity (half-chair conformation), *cis* fused to a six-membered carbocyclic ring which has a slightly distorted boat conformation; the geometry at the N atom is planar. Molecular dimensions are normal; in the tosylate group, S—O = 1·424 (2), S—N = 1·677 (2), S—C = 1·750 (2) Å, O—S—O = 119·6 (1)°, other angles at S = 103·6–109·9 (1)°.

Table 1. *Positional* (*fractional* × 10^5) *and equivalent isotropic thermal parameters* ($U \times 10^3$ Å^2)

	x	y	z	U_{eq}*
S	68255 (5)	62533 (5)	11866 (3)	45
O(1)	59868 (14)	57812 (14)	6115 (10)	63
O(2)	78755 (14)	58881 (13)	11977 (11)	59
O(3)	76842 (11)	72254 (12)	29161 (9)	46
O(4)	69683 (11)	61989 (12)	43863 (8)	42
O(5)	79057 (13)	49888 (14)	37982 (11)	60
O(6)	52408 (11)	77866 (11)	29260 (9)	45
O(7)	37923 (16)	59282 (17)	46531 (12)	82
N	64977 (13)	60616 (13)	21575 (10)	36
C(1)	69553 (16)	66184 (16)	28698 (12)	35
C(2)	63112 (15)	63963 (15)	35720 (12)	35
C(3)	57534 (18)	74312 (17)	37401 (14)	42
C(4)	49981 (22)	72299 (21)	43631 (17)	53
C(5)	43407 (18)	62521 (19)	41574 (15)	50
C(6)	43945 (16)	57147 (18)	33099 (14)	41
C(7)	55572 (15)	54731 (16)	32495 (13)	35
C(8)	57352 (16)	52297 (15)	23197 (13)	35
C(9)	67875 (17)	76417 (18)	10667 (12)	42
C(10)	58377 (20)	81643 (21)	10081 (17)	57
C(11)	58157 (22)	92668 (21)	9278 (17)	60
C(12)	67168 (20)	98444 (18)	8936 (14)	50
C(13)	76458 (21)	92947 (21)	9191 (16)	57
C(14)	76937 (20)	81995 (20)	10212 (16)	51
C(15)	66951 (39)	110405 (25)	8553 (29)	73
C(16)	77459 (17)	54784 (20)	44123 (15)	47
C(17)	83790 (25)	53966 (37)	52896 (19)	69
C(18)	50274 (24)	89081 (19)	29039 (20)	57
C(19)	36915 (21)	47366 (23)	31637 (21)	58
C(20)	61925 (19)	41160 (17)	21927 (18)	46
C(21)	54284 (17)	32159 (16)	21996 (14)	41
C(22)	47312 (20)	29563 (20)	14638 (17)	52
C(23)	40392 (21)	21106 (22)	14595 (21)	65
C(24)	40385 (22)	15158 (21)	21943 (22)	66
C(25)	47171 (24)	17745 (22)	29231 (23)	70
C(26)	54122 (21)	26145 (20)	29259 (18)	57

* $U_{eq} = \frac{1}{3} \times$ trace of the diagonalized **U** tensor.

Experimental. {100}, {110}, {001}, ±(011) developed, 0·4 × 0·5 × 0·5 mm, Enraf–Nonius CAD-4F diffractometer, lattice parameters from 25 reflections with $\theta = 20$–$24°$. Intensities for $\theta \leq 27{\cdot}5°$, *hkl*: 0 to 16, 0 to 16, −20 to 20, ω–2θ scan, ω-scan width (0·85 +

Table 2. *Bond lengths* (Å), *bond angles* (°) *and intra-annular torsion angles* (°), *with e.s.d.'s in parentheses*

S—O(1)	1·421 (2)	C(6)—C(7)	1·549 (3)
S—O(2)	1·427 (2)	C(6)—C(19)	1·519 (3)
S—N	1·677 (2)	C(7)—C(8)	1·556 (3)
S—C(9)	1·750 (2)	C(8)—C(20)	1·541 (3)
O(3)—C(1)	1·202 (2)	C(9)—C(10)	1·379 (3)
O(4)—C(2)	1·440 (2)	C(9)—C(14)	1·375 (3)
O(4)—C(16)	1·345 (3)	C(10)—C(11)	1·387 (3)
O(5)—C(16)	1·196 (3)	C(11)—C(12)	1·378 (3)
O(6)—C(3)	1·417 (3)	C(12)—C(13)	1·377 (3)
O(6)—C(18)	1·431 (3)	C(12)—C(15)	1·500 (4)
O(7)—C(5)	1·211 (3)	C(13)—C(14)	1·382 (3)
N—C(1)	1·372 (3)	C(16)—C(17)	1·491 (3)
N—C(8)	1·485 (2)	C(20)—C(21)	1·499 (3)
C(1)—C(2)	1·520 (3)	C(21)—C(22)	1·385 (3)
C(2)—C(3)	1·528 (3)	C(21)—C(26)	1·377 (3)
C(2)—C(7)	1·542 (3)	C(22)—C(23)	1·385 (4)
C(3)—C(4)	1·519 (3)	C(23)—C(24)	1·381 (4)
C(4)—C(5)	1·495 (3)	C(24)—C(25)	1·365 (4)
C(5)—C(6)	1·512 (3)	C(25)—C(26)	1·382 (4)

O(1)—S—O(2)	119·60 (11)	C(7)—C(6)—C(19)	113·1 (2)
O(1)—S—N	104·16 (9)	C(2)—C(7)—C(6)	113·4 (2)
O(1)—S—C(9)	109·88 (10)	C(2)—C(7)—C(8)	105·95 (15)
O(2)—S—N	109·11 (9)	C(6)—C(7)—C(8)	113·2 (2)
O(2)—S—C(9)	109·23 (10)	N—C(8)—C(7)	103·45 (15)
N—S—C(9)	103·58 (9)	N—C(8)—C(20)	109·5 (2)
C(2)—O(4)—C(16)	117·8 (2)	C(7)—C(8)—C(20)	114·9 (2)
C(3)—O(6)—C(18)	112·9 (2)	S—C(9)—C(10)	119·2 (2)
S—N—C(1)	122·64 (13)	S—C(9)—C(14)	120·2 (2)
S—N—C(8)	122·33 (13)	C(10)—C(9)—C(14)	120·6 (2)
C(1)—N—C(8)	114·93 (15)	C(9)—C(10)—C(11)	118·9 (2)
O(3)—C(1)—N	126·6 (2)	C(10)—C(11)—C(12)	121·5 (3)
O(3)—C(1)—C(2)	126·1 (2)	C(11)—C(12)—C(13)	118·2 (2)
N—C(1)—C(2)	107·1 (2)	C(11)—C(12)—C(15)	121·1 (3)
O(4)—C(2)—C(1)	111·90 (15)	C(13)—C(12)—C(15)	120·7 (3)
O(4)—C(2)—C(3)	102·37 (15)	C(12)—C(13)—C(14)	121·4 (2)
O(4)—C(2)—C(7)	114·29 (15)	C(9)—C(14)—C(13)	119·3 (2)
C(1)—C(2)—C(3)	107·63 (15)	O(4)—C(16)—O(5)	123·3 (2)
C(1)—C(2)—C(7)	106·70 (15)	O(4)—C(16)—C(17)	111·4 (3)
C(3)—C(2)—C(7)	113·9 (2)	O(5)—C(16)—C(17)	125·4 (3)
O(6)—C(3)—C(2)	105·7 (2)	C(8)—C(20)—C(21)	114·4 (2)
O(6)—C(3)—C(4)	112·9 (2)	C(20)—C(21)—C(22)	120·4 (2)
C(2)—C(3)—C(4)	110·1 (2)	C(20)—C(21)—C(26)	121·3 (2)
C(3)—C(4)—C(5)	113·4 (2)	C(22)—C(21)—C(26)	118·3 (2)
O(7)—C(5)—C(4)	120·4 (2)	C(21)—C(22)—C(23)	120·7 (3)
O(7)—C(5)—C(6)	122·9 (2)	C(22)—C(23)—C(24)	120·0 (3)
C(4)—C(5)—C(6)	116·7 (2)	C(23)—C(24)—C(25)	119·5 (3)
C(5)—C(6)—C(7)	109·3 (2)	C(24)—C(25)—C(26)	120·5 (3)
C(5)—C(6)—C(19)	112·3 (2)	C(21)—C(26)—C(25)	121·0 (3)

C(7)—C(2)—C(3)—C(4)	55·4 (2)	C(8)—N—C(1)—C(2)	−14·8 (2)
C(2)—C(3)—C(4)—C(5)	−45·5 (3)	N—C(1)—C(2)—C(7)	11·0 (2)
C(3)—C(4)—C(5)—C(6)	−9·0 (3)	C(1)—C(2)—C(7)—C(8)	−3·9 (2)
C(4)—C(5)—C(6)—C(7)	54·0 (3)	C(2)—C(7)—C(8)—N	−4·0 (2)
C(5)—C(6)—C(7)—C(2)	−42·5 (2)	C(1)—N—C(8)—C(7)	12·0 (2)
C(3)—C(2)—C(7)—C(6)	−10·0 (2)		

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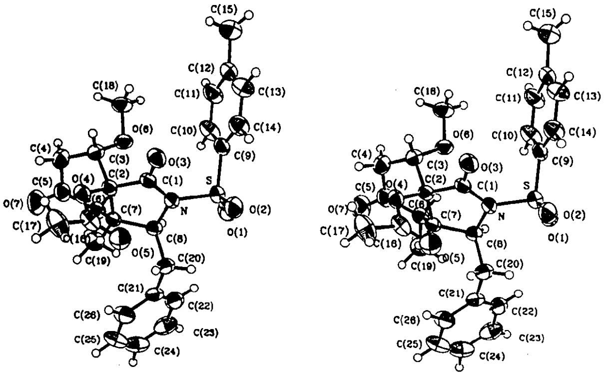
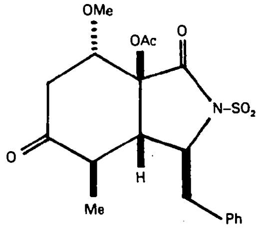


Fig. 1. Stereoview of the molecule (one enantiomorph; the crystals are racemic) with crystallographic numbering scheme.

0·35tanθ)° at 1·3–10° min^{-1}, extended 25% on each side for background measurement, three standard reflections showed negligible variations in intensity, Lp but no absorption corrections, 5772 independent reflections measured, 3188 with $I \geq 3\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04I)^2$, S = scan, B_1 and B_2 = background counts. Structure by direct methods, refined by full-matrix least squares on F, H atoms from a difference map, $w = 1/\sigma^2(F)$, scattering factors from *International Tables for X-ray Crystallography* (1974), locally written, or locally modified versions of standard computer programs, final $R = 0{\cdot}036$, $wR = 0{\cdot}047$ for 3188 reflections, $S = 1{\cdot}84$, 316 parameters (non-H atoms, plus 116 H-atom parameters), $R = 0{\cdot}091$ for all 5772 reflections, $\Delta/\sigma = 0{\cdot}06$ (mean), 0·93 [maximum, for x of H(17b)], maximum final difference density −0·3 to 0·2 e Å^{-3}. Atomic parameters are in Table 1, bond lengths and angles in Table 2, and a view of the molecule in Fig. 1.*

Related literature. Bates & Ramaswamy (1983) describe the use of the title compound (I) in a possible synthetic route to cytochalasin mould metabolites.



(I)

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* Lists of anisotropic thermal parameters, hydrogen positions, bond lengths and angles involving H, torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51050 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of *tert*-Butyldiphenylsilyl *tert*-Butyldiphenylsilanecarboxylate

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Abstract. (^{t}Bu)Ph$_2$SiCOOSiPh$_2$(^{t}Bu), $C_{33}H_{38}O_2Si_2$, M_r = 522·84, monoclinic, $C2/c$, a = 19·277 (3), b = 10·220 (1), c = 31·312 (4) Å, β = 103·30 (1)°, V = 6003·4 (13) Å^3, Z = 8, D_x = 1·157 g cm^{-3}, Mo $K\alpha_1$, λ = 0·70930 Å, μ = 1·4 cm^{-1}, $F(000)$ = 2240, T = 295 K, R = 0·038 for 3484 observed reflections. The molecule contains a central Si—C(O)—O—Si grouping, torsion angle 159·6 (1)°, Si—C = 1·924 (3), Si—O = 1·702 (2) Å, with planar geometry at the carboxyl C atom, and tetrahedral geometries at the Si atoms; molecular dimensions are within normal ranges.

Experimental. {100}, {111}, {001}, ±($\bar{1}$13) developed, 0·43 × 0·34 × 0·38 mm, Enraf–Nonius CAD-4F diffractometer, lattice parameters from 25 reflections with θ = 14–17°. Intensities for $\theta \leq 27{\cdot}5°$, hkl: −25 to 25, 0 to 13, 0 to 40, ω–2θ scan, ω-scan width (0·57 + 0·35 tanθ)° at 1·0–10° min^{-1}, extended 25% on each side for background measurement, standard reflections showed negligible variations in intensity, Lp but no absorption corrections, 6868 independent reflections measured, 3484 with $I \geq 3\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04I)^2$, S = scan, B_1 and

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