

Table 3. Distances and angles involving hydrogen atoms

The e.s.d.'s are 0.05 Å for the distances and 3° for the angles.

H(3)—C(3)	0.91 Å	C(2)—C(3)—H(3)	118°
H(4)—C(4)	1.01	C(3)—C(4)—H(4)	125
H(5)—C(5)	1.05	C(4)—C(5)—H(5)	117
H(7)—C(7)	0.96	C(2)—C(7)—H(7)	122
H(151)—C(15)	1.10	C(14)—C(15)—H(151)	111
H(152)—C(15)	1.05	C(14)—C(15)—H(152)	113
H(141)—C(14)	1.34	C(13)—C(14)—H(141)	109
H(142)—C(14)	0.98	C(13)—C(14)—H(142)	96
H(131)—C(13)	1.23	C(12)—C(13)—H(131)	118
H(132)—C(13)	1.11	C(12)—C(13)—H(132)	101
H(121)—C(12)	0.95	N(11)—C(12)—H(121)	103
H(122)—C(12)	1.09	N(11)—C(12)—H(122)	99
H(161)—C(16)	0.98	C(8)—C(16)—H(161)	108
H(162)—C(16)	0.99	C(8)—C(16)—H(162)	109
H(171)—C(17)	0.92	C(16)—C(17)—H(171)	107
H(172)—C(17)	1.04	C(16)—C(17)—H(172)	110
H(191)—C(19)	1.06	N(18)—C(19)—H(191)	110
H(192)—C(19)	1.07	N(18)—C(19)—H(192)	118
H(201)—C(20)	0.95	C(19)—C(20)—H(201)	103
H(202)—C(20)	1.10	C(19)—C(20)—H(202)	109
H(211)—C(21)	0.97	N(18)—C(21)—H(211)	112
H(212)—C(21)	1.10	N(18)—C(21)—H(212)	107
H(221)—C(22)	1.10	C(21)—C(22)—H(221)	102
H(222)—C(22)	1.11	C(21)—C(22)—H(222)	109
H(231)—C(23)	1.04	C(22)—C(23)—H(231)	112
H(232)—C(23)	1.02	C(22)—C(23)—H(232)	113
H(27)—C(27)	1.12	C(26)—C(27)—H(27)	122
H(28)—C(28)	1.14	C(27)—C(28)—H(28)	115
H(30)—C(30)	1.06	C(29)—C(30)—H(30)	119
H(31)—C(31)	1.06	C(30)—C(31)—H(31)	118

pected energy minima (Koch, 1974). Energy calculations using the PCILO method [Perturbative Configuration Interaction using Localized Orbitals (Diner, Malrieu, Jordan & Gilbert, 1969)] show that for the set of torsion angles ($\psi 1, \psi 2$) only combinations close

Table 4. Torsion angles

$\psi 1$	O(10)—C(9)—C(8)—C(6)	107°
$\psi 2$	C(5)—C(6)—C(8)—C(9)	134
$\psi 3$	O(10)—C(9)—N(11)—C(12)	-2
$\psi 4$	C(6)—C(8)—C(20)—C(19)	-175
$\psi 5$	C(6)—C(8)—C(16)—C(17)	175
$\psi 6$	C(8)—C(20)—C(19)—N(18)	63
$\psi 7$	C(8)—C(16)—C(17)—N(18)	-54
$\psi 8$	C(21)—N(18)—C(17)—C(16)	178
$\psi 9$	C(21)—N(18)—C(19)—C(20)	179
$\psi 10$	C(17)—N(18)—C(21)—C(22)	169
$\psi 11$	N(18)—C(21)—C(22)—C(23)	-179
$\psi 12$	C(21)—C(22)—C(23)—C(24)	-76
$\psi 13$	C(22)—C(23)—C(24)—C(26)	-175
$\psi 14$	O(25)—C(24)—C(26)—C(27)	1

to (120, 140), (-120, -140), (120, -40) and (-120, 40°) are allowed, in agreement with the experimental values. Hence the angle between the mean planes of the piperidine ring and the phenyl group is always close to 45°, at variance with the situation found in other neuroleptics.

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5-Methoxy-5-phenyl-10,11-dihydro-5H-dibenzo[b, f]silepin

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Abstract. C₂₁H₂₀O_{Si}, *M* = 316.48; monoclinic, *P*2₁/*c*; *a* = 10.833 (2), *b* = 8.330 (3), *c* = 20.665 (7) Å, β = 110.58 (3)°; *Z* = 4, *D_m* = 1.18 (2), *D_c* = 1.20 g cm⁻³; *U* = 1745.8 Å³. 5-Methoxy-5-phenyl-10,11-dihydro-5H-dibenzo-

[*b, f*]silepin adopts a folded boat conformation with a dihedral angle between benzo-group planes of 138.3°. The phenyl substituent occupies the pseudo-axial position.

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Introduction. Crystals were prepared by J. Corey (Corey, Dueber & Bichlmeir, 1971). A crystal 0.22 × 0.24 × 0.30 mm was mounted on a Syntex *P*2₁ diffrac-

tometer and data were collected to $2\theta = 45^\circ$ with Mo $K\alpha$ ($\lambda = 0.71069 \text{ \AA}$) radiation (graphite monochromator) and the θ - 2θ scan technique at 2° min^{-1} . Backgrounds were measured at each end of the scan (2θ scan range: $2\theta \text{ Mo } K\alpha_1 - 1.0^\circ$ to $2\theta \text{ Mo } K\alpha_2 + 1.0^\circ$) for a total time equal to one-half the scan time. Systematic absences were $h0l, l \text{ odd}$; $0k0, k \text{ odd}$, uniquely determining the space group $P2_1/c$. Fifteen reflections with 2θ between 16 and 23° were centered with a programmed centering routine; cell parameters were obtained by least-squares refinement of these angles. During data collection, the intensities of three standard reflections were measured every 97 reflections with no significant variation in intensity observed. The data were reduced to F^2 and $\sigma(F^2)$ by procedures similar to those described previously (Schmonsees, 1974). Standard deviations were assigned as follows: $\sigma(I) = [\sigma_{\text{counter}}(I)^2 + (0.04 \times I)^2]^{1/2}$, where $\sigma_{\text{counter}} = (I + K^2 B)^{1/2}$, $I = \text{net intensity}$, $B = \text{total background count}$ and $K = \text{ratio of scan time to background time}$. No corrections were made for absorption [$\mu(\text{Mo } K\alpha) = 1.47 \text{ cm}^{-1}$]. The 1065 data with $F^2 > 2\sigma(F^2)$ from 3560 data scanned were used in the structure solution and refinement.

The structure was solved by an iterative application of the Σ_2 relationship (Long, 1965; Sayre, 1952) using 138 normalized structure factors of magnitude 1.5 or greater. An E map based on the set of phases for the solution with the largest consistency index (0.86) contained the positions of the 23 nonhydrogen atoms. Least-squares refinement (Busing, Martin & Levy, 1962) with isotropic thermal parameters gave a discrepancy value $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.133$. Refinement was continued with anisotropic thermal parameters for the 17 nonhydrogen atoms of the tricyclic framework and methoxy group, and isotropic thermal

parameters for the carbon atoms of the phenyl group. The hydrogen atoms were included in the calculations at ideal locations (Zalkin, 1974) with fixed hydrogen distances of 1.0 \AA and isotropic thermal parameters 10% larger than the equivalent isotropic B of the atom to which they are bonded. Tables 1 and 2 give final positional and thermal parameters with associated estimated standard deviations. Final discrepancy values were $R_1 = 0.073$ and $R_2 = [\sum w(|F_o| - |F_c|)^2 / \sum w F_o^2]^{1/2} = 0.064$. The largest parameter shift in the final cycle of full-matrix refinement was 4% of its standard deviation; the error of fit was 1.99. Atomic scattering factors were taken from *International Tables for X-ray Crystal-*

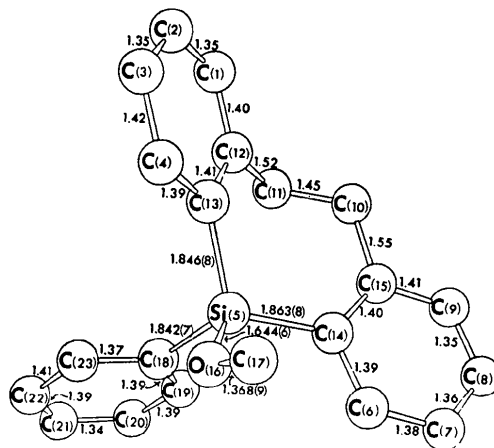


Fig. 1. Interatomic bond distances: the mean estimated standard deviation of C-C bond distances is 0.011 \AA (range: 0.009 – 0.014 \AA).

Table 1. Final positional ($\times 10^4$) and anisotropic thermal ($\times 10^4$) parameters with estimated standard deviations in parentheses

Anisotropic thermal parameters are in the form $\exp [-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$.

	<i>x</i>	<i>y</i>	<i>z</i>	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	5873 (13)	2279 (14)	1692 (6)	204 (18)	400 (30)	39 (5)	28 (19)	39 (8)	12 (10)
C(2)	4994 (12)	3476 (21)	1444 (6)	176 (19)	535 (44)	40 (5)	57 (24)	40 (8)	53 (12)
C(3)	5035 (12)	4807 (17)	1820 (8)	161 (17)	412 (34)	67 (7)	117 (22)	53 (10)	72 (12)
C(4)	6037 (12)	4963 (12)	2483 (7)	165 (15)	290 (24)	64 (5)	68 (18)	51 (8)	47 (10)
Si(5)	8230 (2)	3869 (3)	3622 (1)	151 (3)	154 (4)	42 (1)	11 (4)	41 (1)	-7 (2)
C(6)	10956 (11)	3912 (11)	4139 (5)	149 (14)	239 (20)	48 (4)	-8 (16)	26 (6)	-17 (8)
C(7)	12244 (11)	3600 (13)	4198 (5)	158 (16)	286 (25)	63 (5)	-29 (18)	33 (7)	-13 (9)
C(8)	12463 (9)	2543 (14)	3751 (6)	106 (14)	344 (27)	52 (5)	25 (16)	35 (7)	38 (9)
C(9)	11462 (11)	1798 (11)	3260 (5)	152 (15)	262 (23)	39 (4)	49 (15)	32 (6)	16 (7)
C(10)	9087 (11)	1272 (12)	2568 (5)	152 (13)	316 (23)	74 (5)	4 (17)	50 (7)	-43 (10)
C(11)	7798 (11)	971 (12)	2614 (5)	213 (17)	241 (22)	50 (4)	-17 (18)	30 (7)	-28 (8)
C(12)	6865 (9)	2373 (14)	2343 (5)	133 (13)	327 (27)	24 (4)	6 (16)	17 (6)	15 (9)
C(13)	6960 (8)	3755 (14)	2749 (4)	152 (14)	224 (19)	33 (3)	20 (15)	43 (6)	19 (8)
C(14)	9881 (8)	3204 (9)	3634 (5)	126 (12)	159 (16)	35 (3)	4 (12)	33 (6)	6 (6)
C(15)	10145 (9)	2125 (10)	3182 (4)	134 (13)	199 (18)	37 (4)	24 (13)	34 (6)	-4 (7)
O(16)	8321 (6)	5712 (7)	3919 (3)	236 (10)	195 (13)	70 (3)	19 (10)	72 (5)	-11 (5)
C(17)	8729 (12)	7070 (12)	3680 (6)	361 (23)	212 (23)	98 (6)	-13 (18)	101 (10)	41 (10)
	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{iso}} (\text{\AA}^2)$	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{iso}} (\text{\AA}^2)$	
C(18)	7720 (7)	2762 (9)	4257 (4)	4.7 (2)	C(21)	6881 (9)	1157 (11)	5228 (4)	6.9 (2)
C(19)	6600 (8)	3232 (9)	4393 (4)	6.1 (2)	C(22)	7988 (9)	610 (11)	5099 (5)	7.6 (2)
C(20)	6223 (8)	2449 (11)	4888 (4)	7.1 (2)	C(23)	8384 (8)	1452 (10)	4611 (4)	6.4 (2)

lography (1974). The highest residual electron density in the final difference map was $0.28 \text{ e } \text{Å}^{-3}$.*

Discussion. 5-Methoxy-5-phenyl-10,11-dihydro-5H-dibenzo[*b,f*]silepin (Fig. 1) is one of a group of silepins which are presently under investigation in an attempt to correlate the spatial characteristics of the tricyclic framework with heteroatom substitution in the central ring (Corey, Corey & Glick, 1975, 1976, and references therein). The title compound adopts a folded boat conformation with the phenyl substituent in the pseudo-axial position.

The molecule, excluding hydrogen atoms, is shown in Figs. 1 and 2 with the values of interatomic bond

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31760 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

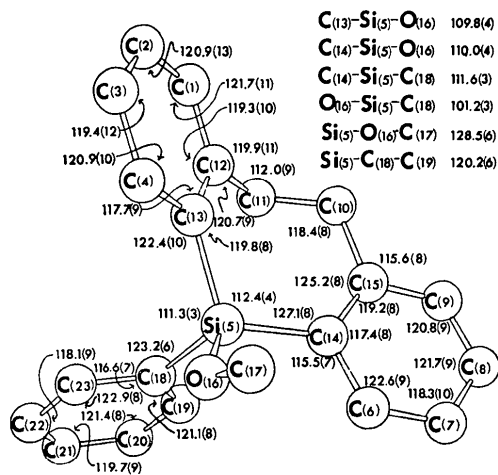


Fig. 2. Interatomic bond angles.

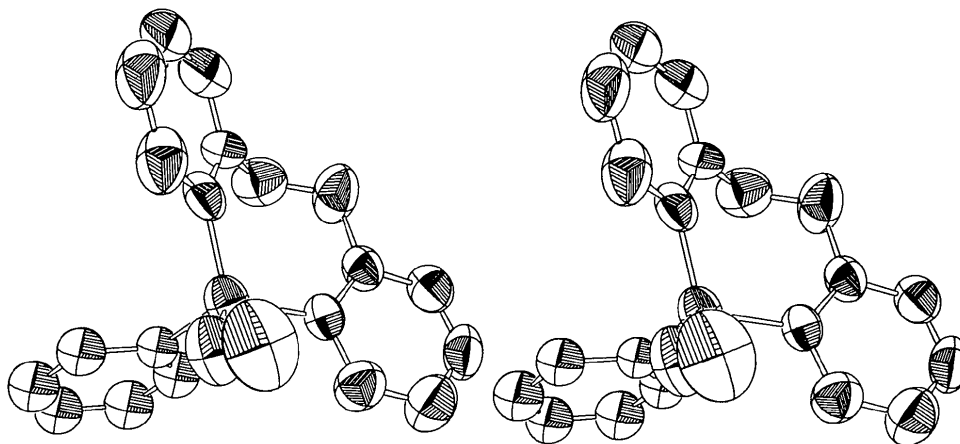


Fig. 3. A stereoscopic view of 5-methoxy-5-phenyl-10,11-dihydro-5H-dibenzo[*b,f*]silepin. The atoms are represented by 50% probability thermal ellipsoids.

Table 2. Final hydrogen-atom positional ($\times 10^3$) and thermal parameters

Isotropic thermal parameters have been assigned fixed values 10% greater than the equivalent B of the atom bonded to the hydrogen atom.

	x	y	z	B (Å^2)
H(C1)	581	130	140	8.7
H(C2)	430	338	97	8.7
H(C3)	437	568	164	8.6
H(C4)	607	595	276	8.0
H(C6)	1079	468	447	7.4
H(C7)	1300	413	456	8.6
H(C8)	1339	231	378	7.0
H(C9)	1166	100	295	6.8
H'(C10)	928	174	217	8.6
H''(C10)	965	30	274	8.6
H'(C11)	765	42	301	8.5
H''(C11)	734	36	218	8.5
H'(C17)	835	751	320	10.6
H''(C17)	971	719	385	10.6
H'''(C17)	868	801	397	10.6
H(C19)	610	421	414	6.8
H(C20)	540	284	497	7.8
H(C21)	660	58	557	7.6
H(C22)	850	-33	535	8.4
H(C23)	920	104	452	7.0

distances and angles included (Busing, Martin & Levy, 1964). Least-squares planes for the benzo groups are given in Table 3. Torsion angles about the central ring are presented in Table 4. A stereoscopic view of the nonhydrogen atoms in the asymmetric unit is shown in Fig. 3.* Table 5 contains values of the four structural parameters which have been used to describe the tricyclic framework.

The bend angle, ring center distance and skew distance are not markedly different in the title compound from the values reported for other dibenzo[*b,f*]silepins (Corey, Corey & Glick, 1975, 1976). The twist angle is

* A packing diagram of the title compound is included as Fig. 4 in a paper on interactive graphics for structural chemistry (Glick, Anderson, Butler, Corey & Srodawa, 1976).

Table 3. Equations of least-squares planes of the benzo groups and deviations (Å) of atoms from these planes

X, Y, Z are orthogonal unit vectors defined by the equations $X=a, Y=b, Z=a \times b$. Atoms not included in least-squares plane calculation are indicated by asterisks.

Plane 1: $0.804X + 0.425Y - 0.415Z = 3.575$

C(1)	0.00	C(11)*	-0.01
C(2)	0.01	C(12)	-0.01
C(3)	-0.01	C(13)	0.01
C(4)	0.00	C(14)*	1.13
Si(5)*	-0.01	C(15)*	1.61
C(10)*	1.23		

Plane 2: $0.206X + 0.749Y - 0.630Z = 7.776$

Si(5)*	0.07	C(11)*	-0.46
C(6)	-0.01	C(12)*	0.58
C(7)	0.00	C(13)*	0.91
C(8)	0.01	C(14)	0.00
C(9)	-0.01	C(15)	0.01
C(10)*	0.08		

Table 4. Torsion angles (°) about the seven-membered ring

Si(5)–C(14)	26.8 (8)	C(11)–C(12)	78.1 (11)
C(14)–C(15)	-0.5 (12)	C(12)–C(13)	0.1 (11)
C(15)–C(10)	28.1 (14)	C(13)–Si(5)	-46.2 (7)
C(10)–C(11)	-88.3 (11)		

Table 5. Values for dibenzotricyclic ring system structural parameters (Corey, Corey & Glick, 1975)

Bend angle (°)	138.3
Distance between benzo centers (Å)	5.56
Skew distance (Å)	0.29
Twist angle (°)	16.9

outside the range of 21–23° displayed by other dibenzo[*b,f*]silepins. The short C(10)–C(11) bond length (1.45 Å) may be the result of the large thermal motions of these atoms and the relatively smaller twist angle in this dihydrodibenzosilepin. Similar alternation of bond lengths for the ethano bridge in one of the two independent molecules of imipramine hydrochloride has been reported (Post, Kennard & Horn, 1975).

The Si(5)–O(16)–C(17) angle of 128.5 (6)° is similar to the analogous angle in other methoxy-substituted silanes: methyl silyl ether, 120.6 (9)° (Glidewell, Rankin, Robiette & Sheldrick, 1970); methoxytrifluorosilane, 131 (3)° (Airey, Glidewell, Robiette & Sheldrick, 1971). Torsion angles about the seven-membered ring are given in Table 4.

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