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Chlorotricyclohexylsilane, $C_{18}H_{33}ClSi$

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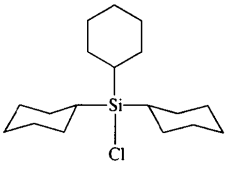
Abstract

The $(C_6H_{11})_3SiCl$ molecule in the title compound lies on a crystallographic mirror plane. Bond lengths include Si—Cl 2.087 (1), and Si—C 1.871 (3) and 1.875 (2) Å.

Comment

We are investigating the connection between the size of the alkyl group *R* and the efficiency of photolysis of molecules of the type $(CpCH_2CH_2OSiR_3)_2Mo_2(CO)_6$ (Cp = cyclopentadienyl), which are prepared from $(CpCH_2CH_2OH)_2Mo_2(CO)_6$ by successive reaction with *n*-BuLi and R_3SiCl (Male *et al.*, 1997). The structure determination of the title reagent, $(c\text{-Hx})_3SiCl$ (*c*-Hx = cyclohexyl), (I), was undertaken as part of its characterization.



(I)

The Cl and Si atoms lie on a crystallographic mirror plane which bisects one of the three chair-conformation *c*-Hx rings. The Si atom is sited equatorially with respect to each ring. The crystal is isomorphous (cell dimensions, space group and molecular packing) with $(c\text{-Hx})_3CCl$ (Gillies *et al.*, 1996) and $(c\text{-Hx})_3SnX$ (*X* = Br, I; Calogero *et al.*, 1981), and also with $(c\text{-Hx})_3GeSH$ (Brisse *et al.*, 1983). As in these compounds [and also $(c\text{-Hx})_3SnX$ (*X* = F, Cl; Calogero *et al.*, 1979, 1981)], the packing density is low (here, 22.9 Å^3/non-H atom). The shortest intermolecular distance for non-H atoms is from C5 to C8 [3.831 (3) Å], while the nearest non-H atom to Cl is C10 at a distance of 4.043 (5) Å, and the nearest Si atom to Cl is at a distance of 5.523 (1) Å [contrast the non-isomorphous $(c\text{-Hx})_3SnCl$ (Calogero *et al.*, 1979), with a 3.30 Å Sn···Cl contact]. The reduction in the Cl—Si—C bond angles compared with the C—Si—C angles is greater than in Ph_3SiCl (Lobkovskii *et al.*, 1981), but is paralleled in the other $(c\text{-Hx})_3MX$ molecules.

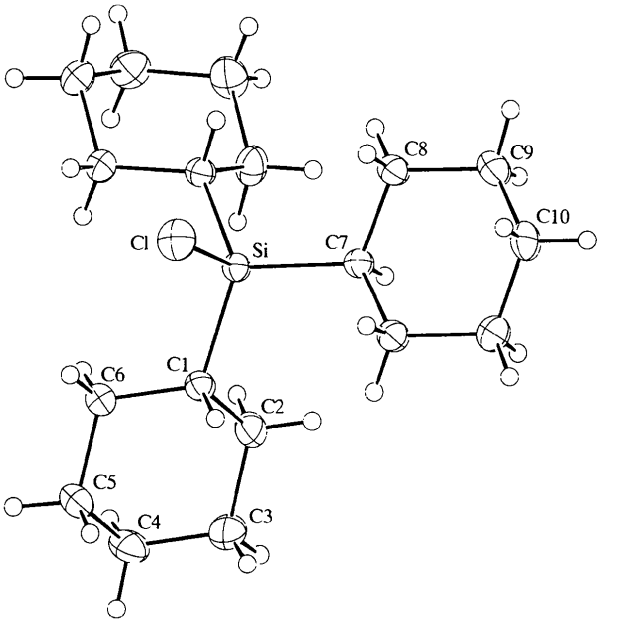


Fig. 1. The molecular structure of $(C_6H_{11})_3SiCl$ with 30% probability ellipsoids.

Experimental

Hexanes (75 ml) and cyclohexyl chloride (35 ml) were added to 6.14 g Li (from 25 g Li dispersion) under argon over a 5 h period with reflux. $SiCl_4$ (3.5 ml) in 10 ml hexane was then added over 2 h at 273 K. The mixture was kept at 296 K for 36 h, filtered, and treated dropwise at 273 K with 6 *M* HCl (60 ml), and the organic layer was dried over $CaCl_2$. The solvent was removed under vacuum, and the crude product was recrystallized from hexane. A colorless block cleaved from a larger crystal was glued to a glass fiber.

Crystal data

$C_{18}H_{33}ClSi$
M_r = 313.00
Orthorhombic
Pnma
a = 10.855 (1) Å
b = 16.045 (2) Å
c = 10.516 (2) Å
V = 1831.5 (8) Å^3
Z = 4
D_x = 1.135 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 14.0–16.0°
μ = 0.26 mm^{-1}
T = 295 K
Block
0.36 × 0.34 × 0.31 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.89, T_{max} = 0.92
1665 measured reflections
1665 independent reflections

1275 reflections with $I<\sigma(I)$
θ_{max} = 25.0°
h = 0 → 12
k = 0 → 19
l = 0 → 12
3 standard reflections
every 60 reflections
intensity decay: 3.2%

Refinement

Refinement on F^2	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$R(F)$ = 0.043	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
$wR(F^2)$ = 0.073	Extinction correction:
S = 1.47	$I_o(\text{corr}) = I_o[1 + 2gI_c]$
1665 reflections	(Stout & Jensen, 1968)
166 parameters	Extinction coefficient:
All H atoms refined	g = 9.8 (13) × 10^{-7}
$w = 4F_o^2/[\sigma^2(I) + (0.02I)^2]$	Scattering factors from
$(\Delta/\sigma)_{max}$ = 0.001	Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\sum_i\sum_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cl	0.66388 (7)	3/4	0.55486 (8)	0.0569 (3)
Si	0.47333 (7)	3/4	0.5282	0.0335 (2)
C1	0.4193 (2)	0.6496 (1)	0.6002 (2)	0.0369 (5)
C2	0.2867 (2)	0.6262 (2)	0.5669 (2)	0.0497 (6)
C3	0.2552 (3)	0.5382 (2)	0.6117 (2)	0.0594 (8)
C4	0.2783 (3)	0.5278 (2)	0.7525 (3)	0.0597 (7)
C5	0.4077 (3)	0.5523 (1)	0.7886 (2)	0.0539 (7)
C6	0.4380 (2)	0.6403 (1)	0.7435 (2)	0.0434 (6)
C7	0.4462 (3)	3/4	0.3525 (3)	0.0351 (7)
C8	0.4939 (3)	0.8281 (1)	0.2851 (2)	0.0478 (7)
C9	0.4643 (3)	0.8277 (2)	0.1437 (2)	0.0571 (7)
C10	0.5127 (4)	3/4	0.0800 (3)	0.060 (1)

Table 2. *Selected geometric parameters (Å, °)*

Cl—Si	2.087 (1)	C3—C4	1.512 (3)
Si—C1	1.875 (2)	C4—C5	1.508 (4)
Si—C7	1.871 (3)	C5—C6	1.525 (3)
C1—C2	1.528 (3)	C7—C8	1.530 (2)
C1—C6	1.528 (3)	C8—C9	1.521 (3)
C2—C3	1.528 (3)	C9—C10	1.509 (3)
Cl—Si—C1	104.82 (7)	C3—C4—C5	111.9 (2)
Cl—Si—C7	106.78 (9)	C4—C5—C6	111.3 (2)
C1—Si—C1^i	118.5 (1)	C1—C6—C5	111.6 (2)
C1—Si—C7	110.47 (7)	Si—C7—C8	113.8 (1)
Si—C1—C2	114.4 (1)	C8—C7—C8^i	110.0 (2)
Si—C1—C6	116.2 (1)	C7—C8—C9	112.2 (2)
C2—C1—C6	109.1 (2)	C8—C9—C10	111.3 (2)
C1—C2—C3	111.6 (2)	C9—C10—C9^i	111.4 (3)
C2—C3—C4	111.5 (2)		

Symmetry code: (i) $x, \frac{3}{2} - y, z$.

Data were corrected for both decay and absorption. All non-H atoms were apparent in an E map. Refinement was on F^2; all reflections were used, with net negative intensities set at zero. All H atoms were located and refined isotropically. Refined C—H bond lengths were in the range 0.92 (2)–1.02 (2) Å; values of U_{iso}(H) were 0.035 (7)–0.074 (11) Å^2.

Data collection: *CAD-4/PC* (Enraf–Nonius, 1993). Cell refinement: *CAD-4/PC*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1098). Services for accessing these data are described at the back of the journal.

References

Brisse, F., Bélanger-Gariépy, F., Zacharie, B., Gareau, Y. & Steliou, K. (1983). *Nouv. J. Chim.* **7**, 391–394.

Calogero, S., Ganis, P., Peruzzo, V. & Tagliavini, G. (1979). *J. Organomet. Chem.* **179**, 145–152.

Calogero, S., Ganis, P., Peruzzo, V., Tagliavini, G. & Valle, G. (1981). *J. Organomet. Chem.* **220**, 11–20.

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 71, 148. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Enraf–Nonius (1993). *CAD-4/PC Diffractometer Software*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.

Gillies, D. G., Luff, S., Smith, G. W. & Sutcliffe, L. H. (1996). *J. Chem. Cryst.* **26**, 539–542.

Lobkovskii, E. B., Fokin, V. N. & Semenenko, K. N. (1981). *Zh. Strukt. Khim.* **22**, 152–155.

Male, J. L., Lindfors, B. E., Covert, K. J. & Tyler, D. R. (1997). *Macromolecules*, **30**, 6404–6406.

Molecular Structure Corporation (1989). *TEXSAN. TEXRAY Structure Analysis Software*. Version 5.0. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

Stout, G. H. & Jensen, L. H. (1968). *X-ray Structure Determination: A Practical Guide*, p. 411. London: Macmillan.

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(*E*)-1-(3-Methoxy-4-nitrophenyl)-2-(3,4,5-trimethoxyphenyl)ethene

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Abstract

The design and synthesis of new photoaffinity labeling (PAL) reagents is important in order to obtain detailed binding-site and structural information about tubulin. (*E*)-1-(3-Methoxy-4-nitrophenyl)-2-(3,4,5-trimethoxyphenyl)ethene, $C_{18}H_{19}NO_6$, crystallizes in the centrosymmetric space group $P2_1/n$ (No. 14), with four molecules in the unit cell. The *trans*-stilbene base component is nearly planar (mean deviation 0.05 Å), with a dihedral angle between the two phenyl rings of 6.1°. Important bond distances include C=C 1.326 (3) Å and mean N—O 1.187 (8) Å.

Comment

The design and synthesis of new photoaffinity labeling (PAL) reagents for tubulin is important in order to obtain detailed binding-site and structural information about this protein (Nare *et al.*, 1996; Sawada *et al.*, 1993; Rao *et al.*, 1992; Olszewski *et al.*, 1994). We have