Fig. 1. The molecular structure of the title compound with numbering.

on Tektronix plotter with the *SHELXTL* system of programs.

Atomic coordinates are in Table 1.* A perspective molecular drawing and the atom labelling are displayed in Fig. 1. Bond distances and angles are listed in Table 2.

We thank Mrs Cynthia Lesh de Soriano for technical assistance.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53108 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). All computations performed on a Nova 4S computer and plots drawn

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(±)-*tert*-Butyl [(Methyl)(1-naphthyl)(phenyl)silyl]acetate

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Abstract. $C_{23}H_{26}O_2Si$, $M_r = 362·6$, triclinic, $P\bar{1}$, $a = 11·275$ (1), $b = 11·585$ (1), $c = 9·682$ (1) Å, $\alpha = 101·91$ (1), $\beta = 99·81$ (1), $\gamma = 119·09$ (1)°, $V = 1025·7$ (6) Å^3, $Z = 2$, $D_x = 1·174$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1·54184$ Å, $\mu = 10·89$ cm^{-1}, $F(000) = 388$, $T = 301$ K, $R = 0·038$ for 3157 observations (of 3884 unique data). Si—C distances are 1·859 (1) (methyl), 1·871 (1) (phneyl), 1·879 (1) (naphthyl) and 1·896 (1) Å (*tert*-butyl acetate). No evidence for an interaction between the Si atom and the carbonyl O atom was found.

Experimental. Colorless crystal of (1), dimensions 0·48 × 0·48 × 0·64 mm, mounted in capillary, space group from successful refinement of centrosymmetric model, cell dimensions from setting angles of 25

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(1)
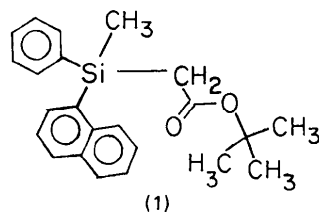
reflections having $32 > \theta > 30°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, θ–2θ scans designed for $I = 50\sigma(I)$, subject to maximum scan time 120 s, scan rates varied 0·53–5·0° min^{-1}. Data with $2 < \theta < 70°$, $0 \le h \le 13$, $-14 \le k \le 14$, $-11 \le l \le 11$ measured, corrected for background, Lorentz, polarization, absorption by ψ scans, minimum relative transmission coefficient 97·46%.

Standard reflections 500, 030, 003, ±2·4% maximum variation; no decay correction. 3884 unique

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Si	0.26190 (4)	0.12430 (4)	0.86726 (4)	3.524 (9)
O1	0.1421 (1)	0.3375 (1)	0.8142 (1)	5.93 (3)
O2	0.1470 (1)	0.23152 (9)	0.5938 (1)	4.05 (3)
C1	0.0936 (2)	0.1044 (1)	0.7557 (2)	3.96 (4)
C2	0.1281 (2)	0.2372 (1)	0.7272 (2)	3.99 (4)
C3	0.3170 (2)	0.2444 (2)	1.0585 (2)	5.42 (5)
C4	0.4073 (2)	0.1978 (1)	0.7803 (2)	3.70 (3)
C5	0.3936 (1)	0.1339 (1)	0.6300 (2)	3.59 (3)
C6	0.2700 (2)	0.0053 (2)	0.5334 (2)	4.25 (4)
C7	0.2595 (2)	−0.0521 (2)	0.3896 (2)	5.19 (5)
C8	0.3739 (2)	0.0157 (2)	0.3353 (2)	5.79 (5)
C9	0.4940 (2)	0.1376 (2)	0.4244 (2)	5.55 (5)
C10	0.5092 (2)	0.2002 (1)	0.5735 (2)	4.31 (4)
C11	0.6361 (2)	0.3261 (2)	0.6688 (2)	5.35 (5)
C12	0.6485 (2)	0.3857 (2)	0.8100 (2)	5.37 (5)
C13	0.5336 (2)	0.3216 (2)	0.8650 (2)	4.61 (4)
C14	0.2188 (2)	−0.0498 (1)	0.8808 (2)	3.66 (3)
C15	0.1050 (2)	−0.1283 (2)	0.9291 (2)	4.70 (4)
C16	0.0759 (2)	−0.2540 (2)	0.9474 (2)	6.02 (6)
C17	0.1582 (2)	−0.3042 (2)	0.9148 (2)	6.67 (6)
C18	0.2716 (2)	−0.2295 (2)	0.8684 (2)	7.17 (6)
C19	0.3021 (2)	−0.1036 (2)	0.8510 (2)	5.42 (4)
C20	0.2015 (2)	0.3554 (1)	0.5443 (2)	4.27 (4)
C21	0.3441 (2)	0.4751 (2)	0.6547 (2)	5.68 (5)
C22	0.2215 (2)	0.3019 (2)	0.3994 (2)	5.92 (6)
C23	0.0895 (2)	0.3919 (2)	0.5192 (2)	6.00 (5)
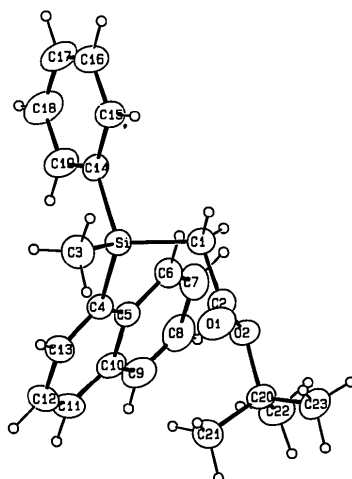
Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level.

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

Si—C1	1.896 (1)	C8—C9	1.346 (2)
Si—C3	1.859 (1)	C9—C10	1.415 (2)
Si—C4	1.879 (1)	C10—C11	1.412 (2)
Si—C14	1.871 (1)	C11—C12	1.348 (2)
O1—C2	1.207 (1)	C12—C13	1.406 (2)
O2—C2	1.339 (1)	C14—C15	1.392 (2)
O2—C20	1.481 (1)	C14—C19	1.392 (2)
C1—C2	1.491 (2)	C15—C16	1.386 (2)
C4—C5	1.434 (2)	C16—C17	1.359 (3)
C4—C13	1.374 (2)	C17—C18	1.365 (3)
C5—C6	1.414 (2)	C18—C19	1.380 (2)
C5—C10	1.425 (2)	C20—C21	1.512 (2)
C6—C7	1.372 (2)	C20—C22	1.512 (2)
C7—C8	1.404 (2)	C20—C23	1.514 (2)
		C—H range	0.75 (2)–1.01 (2)
C1—Si—C3	107.09 (8)	C5—C10—C9	119.22 (13)
C1—Si—C4	110.54 (5)	C5—C10—C11	118.85 (12)
C1—Si—C14	109.11 (5)	C9—C10—C11	121.93 (13)
C3—Si—C4	110.69 (7)	C10—C11—C12	121.35 (13)
C3—Si—C14	107.63 (7)	C11—C12—C13	119.8 (2)
C4—Si—C14	111.62 (5)	C12—C13—C4	122.4 (2)
C2—O2—C20	122.18 (9)	Si—C14—C15	120.74 (10)
Si—C1—C2	110.26 (9)	Si—C14—C19	122.21 (9)
O1—C2—O2	124.70 (11)	C15—C14—C19	116.97 (12)
O1—C2—C1	124.90 (11)	C14—C15—C16	121.7 (2)
O2—C2—C1	110.35 (10)	C15—C16—C17	119.6 (2)
Si—C4—C5	123.99 (9)	C16—C17—C18	120.4 (2)
Si—C4—C13	117.99 (10)	C17—C18—C19	120.4 (2)
C5—C4—C13	118.01 (11)	C14—C19—C18	121.0 (2)
C4—C5—C6	123.03 (11)	O2—C20—C21	110.11 (10)
C4—C5—C10	119.54 (11)	O2—C20—C22	102.07 (10)
C6—C5—C10	117.42 (11)	O2—C20—C23	109.22 (11)
C5—C6—C7	121.56 (13)	C21—C20—C22	110.0 (2)
C6—C7—C8	120.2 (2)	C21—C20—C23	113.4 (2)
C7—C8—C9	120.0 (2)	C22—C20—C23	111.4 (2)
C8—C9—C10	121.6 (2)		
C1—Si—C14—C15	−48.71 (14)	Si—C1—C2—O2	100.11 (14)
C1—Si—C4—C5	−48.21 (14)	C1—C2—O2—C20	−170.63 (15)
C14—Si—C1—C2	−173.71 (10)	C2—O2—C20—C22	172.2 (2)

Fig. 2. Stereoview of the unit cell.

data, 3157 observed with $I > 3\sigma(I)$. Structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refined by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$, using Enraf-Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H anisotropic, H atoms located by ΔF and refined isotropically. Final $R = 0.038$ (0.053 for all data), $wR = 0.060$, $S = 2.319$ for 340 variables. Maximum $\Delta/\sigma = 0.06$ in final cycle, maximum residual density 0.20, minimum -0.17 e Å^{-3}, extinction $g = 6.5$ (6) × 10^{-6}, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c.
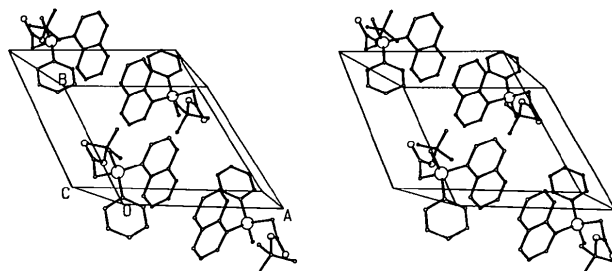
Coordinates* are given in Table 1; bond distances and angles are given in Table 2; the molecule is illustrated in Fig. 1; the molecular packing is shown in Fig. 2.

Related literature. Reaction of α-(methyldiphenyl-silyl) esters with Grignard reagents: Larson, Hernandez, Montes de Lopez-Cepero & Torres (1985); crystal structures containing the α-[(methyl)-

(naphthyl)(phenyl)silyl] group: Okaya & Ashida (1966), Nyburg, Brook, Pascoe & Szymanski (1972), Hitchcock (1976), Larson, Sandoval, Cartledge & Fronczek (1983); structure of naphthalene: Cruickshank (1957).

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Structure of the Coumarin Angustifolin

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Abstract. Angustifolin, $C_{14}H_{14}O_3$, $M_r = 230\cdot3$, monoclinic, $P2_1/c$, $a = 7\cdot5555$ (9), $b = 14\cdot1532$ (11), $c = 11\cdot307$ (3) Å, $\beta = 99\cdot07$ (2)°, $V = 1193\cdot9$ (6) Å^3, $Z = 4$, $D_x = 1\cdot281$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot54184$ Å, $\mu = 6\cdot92$ cm^{-1}, $F(000) = 488$, $T = 294$ K, $R = 0\cdot040$ for 2205 observations with $I > 3\sigma(I)$ (of 2455 unique data). Crystals were prepared *via* sigmatropic rearrangements from 7-hydroxycoumarin, which was isolated from *Ruta angustifolia* Pers., collected in San Agustin de Gaudalix, Madrid province, Spain. The aromatic ring exhibits a maximum deviation of 0·008 (1) Å from planarity, and the ten atoms of the ring system have a maximum deviation of 0·027 (1) Å from a common plane. The double bond of the prenyl group has a C=C distance of 1·286 (1) Å, and it forms a torsion angle having a magnitude of 31·2 (2)° with one of the C—C(methyl) bonds. Molecules related by the screw axis form linear hydrogen bonds involving the hydroxy group and the carbonyl O atom, having an O⋯O distance of 2·7064 (7) Å and an angle at H of 174·6 (11)°.

Experimental. Angustifolin was obtained as colorless needles. The sample was a fragment with dimensions

$0\cdot43 \times 0\cdot62 \times 0\cdot65$ mm. Space group from absences *h0l* with *l* odd and 0*k*0 with *k* odd. Enraf–Nonius CAD-4 diffractometer with graphite monochromator, cell dimensions from setting angles of 25 reflections having $30 > \theta > 25°$. Data collection by ω–2θ scans designed for $I = 25\sigma(I)$, subject to maximum scan time = 90 s. Scan rates varied 0·82–3·30° min^{-1}. Reflections having $4 < 2\theta < 150°$, $0 \le h \le 9$, $0 \le k \le 17$, $-14 \le l \le 14$ measured, corrected for background, Lorentz–polarization, and absorption by ψ scans, minimum relative transmission 0·8861. Redundant 0*kl* and 0*kl* data average ($R_{int} = 0\cdot020$), yielding 2455 unique data. Standard reflections 300, 080, 002, random variation, no decay correction. Structure solved using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0\cdot02F_o^2)^2]^{-1}$ with 2205 data for which $I > 3\sigma(I)$ (250 unobserved reflections), using Enraf–Nonius *SDP* (Frenz & Okaya, 1980). Non-H positions refined with anisotropic thermal parameters; H-atom positions located from difference maps and refined with isotropic thermal parameters. Atomic scattering