Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Benzyl 2-{(*tert*-butyldimethylsilyloxy)-[1-(4-nitrophenyl)-2,5-dioxotetrahydro-1*H*-furo[3,2-*d*]oxazol-6a-yl]methyl}-acrylate

S. N. Patil,[a] D. C. Craig[b] and F. Liu[a]*

[a]Department of Chemistry and Biomolecular Sciences, Macquarie University, Sydney, NSW 2019, Australia, and [b]School of Chemistry, University of New South Wales, Sydney, NSW 2052, Australia
Correspondence e-mail: fei.liu@mq.edu.au

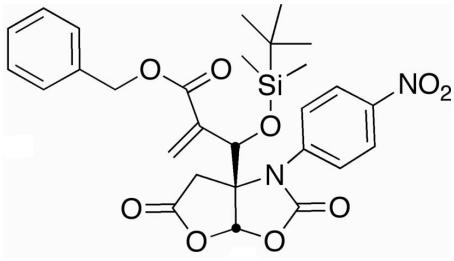
Received 27 April 2007; accepted 14 May 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.049; wR factor = 0.056; data-to-parameter ratio = 9.3.

The title compound, $C_{28}H_{32}N_2O_9Si$, contains a rigid [3,3]-bicyclic oxazoledione core. The substituent on the quaternary bridge C atom contains two long-chain hydrophobic groups that are in extended conformations.

Related literature

For background literature, see Patil & Liu (2007).



Experimental

Crystal data

$C_{28}H_{32}N_2O_9Si$
M_r = 568.7
Triclinic, $P\overline{1}$
a = 9.680 (3) Å
b = 11.674 (5) Å
c = 13.565 (6) Å
α = 88.75 (3)°
β = 79.74 (2)°
γ = 77.13 (3)°
V = 1470.2 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 294 K
0.25 × 0.20 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
5435 measured reflections
5155 independent reflections
3251 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
1 standard reflection
frequency: 30 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.056
S = 1.63
3251 reflections
349 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: local program; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *RAELS* (Rae, 1996); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: local programs.

This work was supported by an Australian Research Council Discovery Project Grant to FL (ARC-DP055068). SP is supported by a predoctoral research fellowship from Macquarie University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2397).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Patil, S. N. & Liu, F. (2007). *Org. Lett.* **9**, 195–198.
Rae, A. D. (1996). *RAELS*. University of New South Wales, Australia.

 doi:10.1107/S1600536807023549

supplementary materials

Acta Cryst. (2007). E**63**, o3021 [**doi:10.1107/S1600536807023549**]

Benzyl 2-{(*tert*-butyldimethylsilyloxy)[1-(4-nitrophenyl)-2,5-dioxotetrahydro-1*H*-furo[3,2-*d*]oxazol-6a-yl]methyl}acrylate

S. N. Patil, D. C. Craig and F. Liu

Comment

We have reported a facile synthesis of alpha-substituted gamma- hydroxybutenolides (Patil & Liu, 2007). In an effort to trap the epimerizable gamma-hydroxy group of butenolide, (I), using an isocyanate, (II), we report the synthesis and structure of the title compound, (III). This molecule contains a substituted dihydro-1*H*-furo[3,2-*d*]oxazole-2,5-dione ring, which is a new bicyclic oxazole-dione ring.

Experimental

To a stirred solution of TBS-protected butenolide, (I) (48.7 mg, 0.12 mmol), in ethyl acetate (0.1 ml) was added 4-nitrophenyl isocyanate (23.9 mg, 0.13 mmol) under nitrogen. The reaction mixture was refluxed for 4 h, cooled to room temperature, stirred for 5 min and filtered. The yellow solid obtained was recrystallized from analytical grade methanol, resulting in colourless blocks of (III) after two weeks at room temperature.

Refinement

The hydrogen atoms were included in positions calculated each cycle (C—H = 1.00 Å) with U_{iso}(H) = U_{eq}(carrier).

Figures

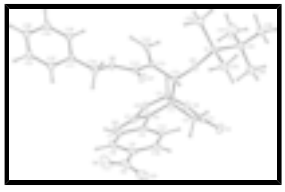


Fig. 1. View of the molecular structure of (III) with ellipsoids at the 10% probability level. H atoms are drawn as spheres of arbitrary radius.

Fig. 2. Reaction scheme for the synthesis of the title compound.

Benzyl 2-{(*tert*-butyldimethylsilyloxy)[1-(4-nitrophenyl)-2,5-dioxo- tetrahydro-1*H*-furo[3,2-*d*]oxazol-6a-yl]methyl}acrylate

Crystal data

$C_{28}H_{32}N_2O_9Si$	$Z = 2$
$M_r = 568.7$	$F_{000} = 600.0$
Triclinic, $P\bar{1}$	$D_x = 1.28$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation

	λ = 0.71073 Å
a = 9.680 (3) Å	Cell parameters from 11 reflections
b = 11.674 (5) Å	θ = 10–11°
c = 13.565 (6) Å	μ = 0.13 mm^{-1}
α = 88.75 (3)°	*T* = 294 K
β = 79.74 (2)°	Block, colourless
γ = 77.13 (3)°	0.25 × 0.20 × 0.18 mm
V = 1470.2 (9) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 25°
ω–2θ scans	*h* = −11→11
Absorption correction: none	*k* = 0→13
5435 measured reflections	*l* = −16→16
5155 independent reflections	1 standard reflections
3251 reflections with $I > 2\sigma(I)$	every 30 min
R_{int} = 0.014	intensity decay: none

Refinement

Refinement on *F*	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.049	$w = 1/[\sigma^2(F) + 0.0004F^2]$
$wR(F^2)$ = 0.056	$(\Delta/\sigma)_{max}$ = 0.004
S = 1.63	$\Delta\rho_{max}$ = 0.35 e Å^{-3}
3251 reflections	$\Delta\rho_{min}$ = −0.31 e Å^{-3}
349 parameters	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Si	−0.08195 (9)	0.80484 (8)	0.28619 (7)	0.0587 (2)
O1	0.0289 (3)	0.6174 (2)	0.6306 (2)	0.0848 (6)
O2	0.1842 (2)	0.7201 (2)	0.5547 (2)	0.0633 (5)
O3	0.4176 (2)	0.6957 (2)	0.4719 (2)	0.0610 (5)
O4	0.6132 (2)	0.5552 (2)	0.4210 (2)	0.0676 (5)
O5	0.4899 (4)	0.0689 (3)	0.1494 (3)	0.1317 (9)
O6	0.6812 (4)	0.1245 (3)	0.0786 (3)	0.175 (1)
O7	0.3549 (2)	0.5878 (2)	0.1139 (2)	0.0739 (5)
O8	0.5189 (2)	0.6936 (2)	0.1211 (1)	0.0619 (5)
O9	0.0631 (2)	0.7679 (2)	0.3410 (1)	0.0492 (4)
N1	0.3987 (2)	0.5594 (2)	0.3654 (2)	0.0450 (5)
N2	0.5680 (5)	0.1378 (3)	0.1371 (3)	0.1077 (9)
C1	0.2516 (3)	0.6296 (2)	0.3915 (2)	0.0407 (5)
C2	0.1478 (3)	0.5681 (3)	0.4599 (2)	0.0456 (6)
C3	0.1107 (3)	0.6329 (3)	0.5571 (2)	0.0568 (7)
C4	0.2720 (3)	0.7247 (3)	0.4602 (2)	0.0518 (6)

C5	0.4890 (3)	0.5969 (3)	0.4187 (2)	0.0531 (6)
C6	0.4399 (3)	0.4533 (2)	0.3081 (2)	0.0459 (6)
C7	0.5839 (3)	0.4126 (3)	0.2643 (3)	0.0688 (8)
C8	0.6237 (4)	0.3104 (3)	0.2080 (3)	0.0861 (9)
C9	0.5229 (4)	0.2480 (3)	0.1969 (3)	0.0721 (8)
C10	0.3809 (4)	0.2869 (3)	0.2384 (2)	0.0617 (7)
C11	0.3395 (3)	0.3907 (3)	0.2929 (2)	0.0522 (6)
C12	0.1906 (3)	0.6864 (2)	0.2996 (2)	0.0445 (6)
C13	0.2981 (3)	0.7435 (3)	0.2322 (2)	0.0464 (6)
C14	0.3029 (3)	0.8543 (3)	0.2396 (2)	0.0651 (7)
C15	0.3908 (3)	0.6662 (3)	0.1503 (2)	0.0511 (6)
C16	0.6105 (4)	0.6225 (3)	0.0376 (3)	0.0742 (8)
C17	0.7291 (3)	0.6814 (3)	−0.0059 (2)	0.0540 (7)
C18	0.7153 (3)	0.8004 (3)	−0.0022 (2)	0.0613 (7)
C19	0.8243 (4)	0.8509 (3)	−0.0514 (3)	0.0816 (9)
C20	0.9458 (4)	0.7817 (4)	−0.1050 (3)	0.103 (1)
C21	0.9597 (4)	0.6624 (4)	−0.1092 (3)	0.099 (1)
C22	0.8527 (4)	0.6119 (3)	−0.0592 (3)	0.0712 (8)
C23	−0.1238 (4)	0.6702 (3)	0.2413 (3)	0.0854 (9)
C24	−0.0455 (5)	0.9020 (4)	0.1812 (4)	0.121 (1)
C25	−0.2271 (4)	0.8814 (3)	0.3883 (3)	0.079 (1)
C26	−0.2458 (4)	0.8042 (4)	0.4791 (3)	0.108 (2)
C27	−0.1963 (6)	0.9921 (5)	0.4198 (5)	0.170 (2)
C28	−0.3707 (4)	0.9073 (4)	0.3484 (4)	0.126 (2)
H1C2	0.1954	0.4843	0.4690	0.046*
H2C2	0.0592	0.5714	0.4307	0.046*
HC4	0.2476	0.8042	0.4306	0.052*
HC7	0.6575	0.4574	0.2738	0.069*
HC8	0.7263	0.2814	0.1751	0.086*
HC10	0.3085	0.2406	0.2294	0.062*
HC11	0.2355	0.4214	0.3218	0.052*
HC12	0.1656	0.6248	0.2604	0.045*
H1C14	0.2330	0.9066	0.2920	0.065*
H2C14	0.3765	0.8865	0.1927	0.065*
H1C16	0.5524	0.6140	−0.0147	0.074*
H2C16	0.6524	0.5430	0.0619	0.074*
HC18	0.6258	0.8514	0.0363	0.061*
HC19	0.8141	0.9379	−0.0476	0.082*
HC20	1.0244	0.8179	−0.1412	0.103*
HC21	1.0484	0.6118	−0.1489	0.099*
HC22	0.8645	0.5246	−0.0614	0.071*
H1C23	−0.2104	0.6920	0.2084	0.085*
H2C23	−0.0398	0.6274	0.1918	0.085*
H3C23	−0.1436	0.6183	0.2993	0.085*
H1C24	−0.1320	0.9243	0.1482	0.121*
H2C24	−0.0235	0.9745	0.2068	0.121*
H3C24	0.0388	0.8602	0.1315	0.121*
H1C26	−0.3241	0.8478	0.5321	0.132*
H2C26	−0.2721	0.7309	0.4593	0.126*

H3C26	−0.1537	0.7831	0.5057	0.158*
H1C27	−0.2755	1.0311	0.4743	0.220*
H2C27	−0.1030	0.9746	0.4450	0.209*
H3C27	−0.1896	1.0455	0.3613	0.219*
H1C28	−0.4501	0.9484	0.4019	0.158*
H2C28	−0.3628	0.9585	0.2885	0.183*
H3C28	−0.3920	0.8318	0.3290	0.142*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si	0.0498 (5)	0.0596 (5)	0.0659 (5)	−0.0102 (4)	−0.0114 (4)	0.0070 (4)
O1	0.083 (1)	0.118 (2)	0.055 (1)	−0.046 (1)	0.017 (1)	−0.011 (1)
O2	0.065 (1)	0.078 (1)	0.049 (1)	−0.027 (1)	−0.0001 (9)	−0.0165 (9)
O3	0.055 (1)	0.071 (1)	0.064 (1)	−0.0302 (9)	−0.0105 (9)	−0.012 (1)
O4	0.041 (1)	0.087 (1)	0.081 (1)	−0.025 (1)	−0.0166 (9)	0.003 (1)
O5	0.176 (2)	0.064 (2)	0.161 (2)	−0.003 (2)	−0.072 (2)	−0.020 (2)
O6	0.132 (3)	0.144 (3)	0.215 (3)	0.026 (2)	0.001 (2)	−0.104 (2)
O7	0.082 (1)	0.078 (1)	0.064 (1)	−0.039 (1)	0.012 (1)	−0.023 (1)
O8	0.057 (1)	0.074 (1)	0.052 (1)	−0.0241 (9)	0.0128 (9)	−0.0151 (9)
O9	0.0393 (9)	0.053 (1)	0.052 (1)	−0.0062 (8)	−0.0028 (8)	−0.0068 (8)
N1	0.034 (1)	0.051 (1)	0.052 (1)	−0.0138 (9)	−0.0080 (9)	−0.002 (1)
N2	0.112 (2)	0.069 (2)	0.128 (3)	0.025 (2)	−0.041 (2)	−0.028 (2)
C1	0.034 (1)	0.046 (1)	0.043 (1)	−0.012 (1)	−0.003 (1)	−0.003 (1)
C2	0.041 (1)	0.054 (2)	0.042 (1)	−0.016 (1)	−0.002 (1)	−0.002 (1)
C3	0.051 (2)	0.068 (2)	0.054 (2)	−0.021 (1)	−0.005 (1)	−0.003 (1)
C4	0.049 (2)	0.063 (2)	0.047 (2)	−0.021 (1)	−0.004 (1)	−0.006 (1)
C5	0.048 (2)	0.063 (2)	0.053 (2)	−0.026 (1)	−0.006 (1)	0.004 (1)
C6	0.044 (1)	0.045 (2)	0.048 (2)	−0.009 (1)	−0.008 (1)	0.005 (1)
C7	0.043 (2)	0.072 (2)	0.086 (2)	−0.005 (1)	−0.007 (2)	−0.011 (2)
C8	0.056 (2)	0.079 (2)	0.111 (3)	0.016 (2)	−0.014 (2)	−0.030 (2)
C9	0.083 (2)	0.049 (2)	0.077 (2)	0.012 (2)	−0.027 (2)	−0.013 (2)
C10	0.075 (2)	0.050 (2)	0.063 (2)	−0.015 (1)	−0.016 (2)	0.001 (1)
C11	0.054 (2)	0.050 (2)	0.052 (2)	−0.015 (1)	−0.005 (1)	−0.002 (1)
C12	0.040 (1)	0.046 (2)	0.046 (1)	−0.013 (1)	−0.001 (1)	−0.005 (1)
C13	0.049 (2)	0.046 (2)	0.044 (2)	−0.015 (1)	−0.002 (1)	−0.001 (1)
C14	0.068 (2)	0.056 (2)	0.068 (2)	−0.023 (1)	0.008 (1)	−0.005 (1)
C15	0.058 (2)	0.055 (2)	0.039 (1)	−0.017 (1)	−0.001 (1)	0.002 (1)
C16	0.077 (2)	0.076 (2)	0.059 (2)	−0.024 (2)	0.024 (1)	−0.018 (2)
C17	0.054 (2)	0.067 (2)	0.039 (1)	−0.016 (1)	0.003 (1)	−0.005 (1)
C18	0.059 (2)	0.061 (2)	0.057 (2)	−0.013 (1)	0.007 (1)	−0.006 (1)
C19	0.077 (2)	0.072 (2)	0.092 (2)	−0.023 (2)	0.005 (2)	−0.001 (2)
C20	0.074 (2)	0.091 (3)	0.128 (3)	−0.030 (2)	0.035 (2)	−0.006 (2)
C21	0.062 (2)	0.090 (3)	0.125 (3)	−0.011 (2)	0.031 (2)	−0.016 (2)
C22	0.060 (2)	0.071 (2)	0.073 (2)	−0.012 (2)	0.011 (2)	−0.008 (2)
C23	0.077 (2)	0.090 (2)	0.096 (2)	−0.014 (2)	−0.034 (2)	−0.018 (2)
C24	0.098 (3)	0.150 (3)	0.123 (3)	−0.037 (3)	−0.040 (2)	0.070 (2)
C25	0.058 (1)	0.059 (2)	0.113 (2)	−0.003 (1)	−0.008 (1)	−0.013 (1)

C26	0.069 (2)	0.140 (3)	0.102 (2)	−0.007 (3)	−0.008 (2)	0.011 (2)
C27	0.158 (4)	0.107 (3)	0.226 (4)	−0.057 (3)	0.065 (4)	−0.097 (3)
C28	0.051 (2)	0.159 (4)	0.139 (3)	0.031 (2)	−0.010 (1)	0.019 (3)

Geometric parameters (Å, °)

Si—O9	1.673 (2)	C12—HC12	1.000
Si—C23	1.849 (4)	C13—C14	1.312 (4)
Si—C24	1.835 (4)	C13—C15	1.477 (4)
Si—C25	1.869 (4)	C14—H1C14	1.000
O1—C3	1.195 (3)	C14—H2C14	1.000
O2—C3	1.363 (3)	C16—C17	1.494 (4)
O2—C4	1.413 (3)	C16—H1C16	1.000
O3—C4	1.411 (3)	C16—H2C16	1.000
O3—C5	1.353 (4)	C17—C18	1.367 (4)
O4—C5	1.198 (3)	C17—C22	1.378 (4)
O5—N2	1.211 (4)	C18—C19	1.384 (4)
O6—N2	1.214 (5)	C18—HC18	1.000
O7—C15	1.195 (3)	C19—C20	1.365 (5)
O8—C15	1.339 (3)	C19—HC19	1.000
O8—C16	1.454 (3)	C20—C21	1.370 (5)
O9—C12	1.413 (3)	C20—HC20	1.000
N1—C1	1.462 (3)	C21—C22	1.373 (5)
N1—C5	1.374 (3)	C21—HC21	1.000
N1—C6	1.417 (3)	C22—HC22	1.000
N2—C9	1.474 (4)	C23—H1C23	1.000
C1—C2	1.532 (4)	C23—H2C23	1.000
C1—C4	1.533 (4)	C23—H3C23	1.000
C1—C12	1.547 (4)	C24—H1C24	1.000
C2—C3	1.480 (4)	C24—H2C24	1.000
C2—H1C2	1.000	C24—H3C24	1.000
C2—H2C2	1.000	C25—C26	1.519 (5)
C4—HC4	1.000	C25—C27	1.477 (5)
C6—C7	1.394 (4)	C25—C28	1.546 (5)
C6—C11	1.385 (4)	C26—H1C26	1.000
C7—C8	1.373 (5)	C26—H2C26	1.000
C7—HC7	1.000	C26—H3C26	1.000
C8—C9	1.370 (5)	C27—H1C27	1.000
C8—HC8	1.000	C27—H2C27	1.000
C9—C10	1.367 (5)	C27—H3C27	1.000
C10—C11	1.376 (4)	C28—H1C28	1.000
C10—HC10	1.000	C28—H2C28	1.000
C11—HC11	1.000	C28—H3C28	1.000
C12—C13	1.518 (4)		
O9—Si—C23	109.3 (1)	C13—C14—H1C14	120.0
O9—Si—C24	109.2 (2)	C13—C14—H2C14	120.0
O9—Si—C25	103.9 (1)	H1C14—C14—H2C14	120.0
C23—Si—C24	110.5 (2)	O7—C15—O8	123.6 (3)
C23—Si—C25	111.7 (2)	O7—C15—C13	123.4 (3)

C24—Si—C25	112.1 (2)	O8—C15—C13	113.0 (3)
C3—O2—C4	111.5 (2)	O8—C16—C17	109.0 (3)
C4—O3—C5	110.7 (2)	O8—C16—H1C16	109.6
C15—O8—C16	114.4 (2)	O8—C16—H2C16	109.6
Si—O9—C12	124.0 (2)	C17—C16—H1C16	109.6
C1—N1—C5	111.1 (2)	C17—C16—H2C16	109.6
C1—N1—C6	125.2 (2)	H1C16—C16—H2C16	109.5
C5—N1—C6	122.8 (2)	C16—C17—C18	123.1 (3)
O5—N2—O6	125.5 (4)	C16—C17—C22	117.3 (3)
O5—N2—C9	118.0 (4)	C18—C17—C22	119.4 (3)
O6—N2—C9	116.5 (4)	C17—C18—C19	120.4 (3)
N1—C1—C2	114.5 (2)	C17—C18—HC18	119.8
N1—C1—C4	101.4 (2)	C19—C18—HC18	119.8
N1—C1—C12	113.1 (2)	C18—C19—C20	119.9 (4)
C2—C1—C4	103.2 (2)	C18—C19—HC19	120.1
C2—C1—C12	113.2 (2)	C20—C19—HC19	120.1
C4—C1—C12	110.2 (2)	C19—C20—C21	119.8 (3)
C1—C2—C3	106.2 (2)	C19—C20—HC20	120.1
C1—C2—H1C2	110.3	C21—C20—HC20	120.1
C1—C2—H2C2	110.3	C20—C21—C22	120.5 (3)
C3—C2—H1C2	110.3	C20—C21—HC21	119.8
C3—C2—H2C2	110.3	C22—C21—HC21	119.8
H1C2—C2—H2C2	109.5	C17—C22—C21	120.0 (3)
O1—C3—O2	120.8 (3)	C17—C22—HC22	120.0
O1—C3—C2	128.7 (3)	C21—C22—HC22	120.0
O2—C3—C2	110.5 (2)	Si—C23—H1C23	109.5
O2—C4—O3	108.8 (2)	Si—C23—H2C23	109.5
O2—C4—C1	108.6 (2)	Si—C23—H3C23	109.5
O2—C4—HC4	110.9	H1C23—C23—H2C23	109.5
O3—C4—C1	106.7 (2)	H1C23—C23—H3C23	109.5
O3—C4—HC4	110.9	H2C23—C23—H3C23	109.5
C1—C4—HC4	110.9	Si—C24—H1C24	109.5
O3—C5—O4	121.3 (3)	Si—C24—H2C24	109.5
O3—C5—N1	109.7 (3)	Si—C24—H3C24	109.5
O4—C5—N1	128.9 (3)	H1C24—C24—H2C24	109.5
N1—C6—C7	119.4 (3)	H1C24—C24—H3C24	109.5
N1—C6—C11	121.3 (3)	H2C24—C24—H3C24	109.5
C7—C6—C11	119.3 (3)	Si—C25—C26	111.6 (3)
C6—C7—C8	119.4 (3)	Si—C25—C27	111.2 (3)
C6—C7—HC7	120.3	Si—C25—C28	108.0 (3)
C8—C7—HC7	120.3	C26—C25—C27	108.9 (4)
C7—C8—C9	120.1 (3)	C26—C25—C28	107.0 (3)
C7—C8—HC8	120.0	C27—C25—C28	110.0 (4)
C9—C8—HC8	120.0	C25—C26—H1C26	109.5
N2—C9—C8	119.3 (4)	C25—C26—H2C26	109.5
N2—C9—C10	119.2 (4)	C25—C26—H3C26	109.5
C8—C9—C10	121.5 (3)	H1C26—C26—H2C26	109.5
C9—C10—C11	118.8 (3)	H1C26—C26—H3C26	109.5
C9—C10—HC10	120.6	H2C26—C26—H3C26	109.5

C11—C10—HC10	120.6	C25—C27—H1C27	109.5
C6—C11—C10	120.8 (3)	C25—C27—H2C27	109.5
C6—C11—HC11	119.6	C25—C27—H3C27	109.5
C10—C11—HC11	119.6	H1C27—C27—H2C27	109.5
O9—C12—C1	104.6 (2)	H1C27—C27—H3C27	109.5
O9—C12—C13	112.8 (2)	H2C27—C27—H3C27	109.5
O9—C12—HC12	109.2	C25—C28—H1C28	109.5
C1—C12—C13	111.7 (2)	C25—C28—H2C28	109.5
C1—C12—HC12	109.2	C25—C28—H3C28	109.5
C13—C12—HC12	109.2	H1C28—C28—H2C28	109.5
C12—C13—C14	124.0 (3)	H1C28—C28—H3C28	109.5
C12—C13—C15	114.3 (2)	H2C28—C28—H3C28	109.5
C14—C13—C15	121.5 (3)		
C23—Si—O9—C12	46.3 (2)	H2C2—C2—C3—O1	57.5
C24—Si—O9—C12	−74.7 (3)	H2C2—C2—C3—O2	−122.0
C25—Si—O9—C12	165.6 (2)	N1—C6—C7—C8	−179.4 (3)
O9—Si—C23—H1C23	180.0	N1—C6—C7—HC7	0.6
O9—Si—C23—H2C23	−60.0	C11—C6—C7—C8	−0.9 (5)
O9—Si—C23—H3C23	60.0	C11—C6—C7—HC7	179.1
C24—Si—C23—H1C23	−59.8	N1—C6—C11—C10	−179.0 (3)
C24—Si—C23—H2C23	60.2	N1—C6—C11—HC11	1.0
C24—Si—C23—H3C23	−179.8	C7—C6—C11—C10	2.5 (4)
C25—Si—C23—H1C23	65.6	C7—C6—C11—HC11	−177.5
C25—Si—C23—H2C23	−174.4	C6—C7—C8—C9	−1.3 (6)
C25—Si—C23—H3C23	−54.4	C6—C7—C8—HC8	178.7
O9—Si—C24—H1C24	−180.0	HC7—C7—C8—C9	178.7
O9—Si—C24—H2C24	−60.0	HC7—C7—C8—HC8	−1.3
O9—Si—C24—H3C24	60.0	C7—C8—C9—N2	−179.3 (3)
C23—Si—C24—H1C24	59.8	C7—C8—C9—C10i	2.0 (6)
C23—Si—C24—H2C24	179.8	HC8i—C8i—C9i—N2i	0.7
C23—Si—C24—H3C24	−60.2	HC8i—C8i—C9i—C10i	−178.0
C25—Si—C24—H1C24	−65.4	N2i—C9i—C10i—C11i	−179.0 (3)
C25—Si—C24—H2C24	54.6	N2i—C9i—C10i—HC10i	1.0
C25—Si—C24—H3C24	174.6	C8i—C9i—C10i—C11i	−0.4 (5)
O9—Si—C25—C26	−56.4 (3)	C8i—C9i—C10i—HC10i	179.6
O9—Si—C25—C27	65.4 (4)	C9i—C10i—C11i—C6i	−1.9 (5)
O9—Si—C25—C28	−173.8 (3)	C9i—C10i—C11i—HC11i	178.1
C23—Si—C25—C26	61.3 (3)	HC10i—C10i—C11i—C6i	178.1
C23—Si—C25—C27	−176.9 (4)	HC10i—C10i—C11i—HC11i	−1.9
C23—Si—C25—C28	−56.1 (3)	O9i—C12i—C13i—C14i	24.8 (4)
C24—Si—C25—C26	−174.2 (3)	O9i—C12i—C13i—C15i	−150.0 (2)
C24—Si—C25—C27	−52.4 (4)	C1i—C12i—C13i—C14i	−92.6 (4)
C24—Si—C25—C28	68.4 (3)	C1i—C12i—C13i—C15i	92.5 (3)
C4—O2—C3—O1	−178.6 (3)	HC12i—C12i—C13i—C14i	146.5
C4—O2—C3—C2	1.0 (3)	HC12i—C12i—C13i—C15i	−28.4
C3—O2—C4—O3	−114.7 (3)	C12i—C13i—C14i—H1C14i	0.0
C3—O2—C4—C1	1.0 (3)	C12i—C13i—C14i—H2C14i	−180.0
C3—O2—C4—HC4	123.1	C15i—C13i—C14i—H1C14i	174.5
C5—O3—C4—O2	117.7 (2)	C15i—C13i—C14i—H2C14i	−5.5

C5—O3—C4—C1	0.8 (3)	C12i—C13i—C15i—O7i	28.0 (4)
C5—O3—C4—HC4	−120.0	C12i—C13i—C15i—O8i	−152.2 (2)
C4—O3—C5—O4	−177.6 (3)	C14i—C13i—C15i—O7i	−147.0 (3)
C4—O3—C5—N1	3.2 (3)	C14i—C13i—C15i—O8i	32.8 (4)
C16—O8—C15—O7	2.3 (4)	O8i—C16i—C17i—C18i	−28.4 (4)
C16—O8—C15—C13	−177.5 (2)	O8i—C16i—C17i—C22i	157.1 (3)
C15—O8—C16—C17	164.3 (3)	H1C16i—C16i—C17i—C18i	91.5
C15—O8—C16—H1C16	44.3	H1C16i—C16i—C17i—C22i	−83.0
C15—O8—C16—H2C16	−75.8	H2C16i—C16i—C17i—C18i	−148.4
Si—O9—C12—C1	−149.1 (2)	H2C16i—C16i—C17i—C22i	37.2
Si—O9—C12—C13	89.4 (2)	C16i—C17i—C18i—C19i	−174.3 (3)
Si—O9—C12—HC12	−32.3	C16i—C17i—C18i—HC18i	5.7
C5—N1—C1—C2	−104.1 (3)	C22i—C17i—C18i—C19i	0.0 (5)
C5—N1—C1—C4	6.2 (3)	C22i—C17i—C18i—HC18i	180.0
C5—N1—C1—C12	124.2 (2)	C16i—C17i—C22i—C21i	173.6 (4)
C6—N1—C1—C2	64.8 (3)	C16i—C17i—C22i—HC22i	−6.4
C6—N1—C1—C4	175.1 (2)	C18i—C17i—C22i—C21i	−1.0 (5)
C6—N1—C1—C12	−67.0 (3)	C18i—C17i—C22i—HC22i	179.0
C1—N1—C5—O3	−6.2 (3)	C17i—C18i—C19i—C20i	0.8 (6)
C1—N1—C5—O4	174.7 (3)	C17i—C18i—C19i—HC19i	−179.2
C6—N1—C5—O3	−175.4 (2)	HC18i—C18i—C19i—C20i	−179.2
C6—N1—C5—O4	5.5 (5)	HC18i—C18i—C19i—HC19i	0.8
C1—N1—C6—C7	161.7 (3)	C18i—C19i—C20i—C21i	−0.6 (7)
C1—N1—C6—C11	−16.8 (4)	C18i—C19i—C20i—HC20i	179.4
C5—N1—C6—C7	−30.7 (4)	HC19i—C19i—C20i—C21i	179.4
C5—N1—C6—C11	150.8 (3)	HC19i—C19i—C20i—HC20i	−0.6
O5—N2—C9—C8	160.6 (4)	C19i—C20i—C21i—C22i	−0.5 (8)
O5—N2—C9—C10	−20.7 (5)	C19i—C20i—C21i—HC21i	179.5
O6—N2—C9—C8	−19.9 (6)	HC20i—C20i—C21i—C22i	179.5
O6—N2—C9—C10	158.8 (4)	HC20i—C20i—C21i—HC21i	−0.5
N1—C1—C2—C3	112.2 (3)	C20i—C21i—C22i—C17i	1.3 (7)
N1—C1—C2—H1C2	−7.3	C20i—C21i—C22i—HC22i	−178.7
N1—C1—C2—H2C2	−128.3	HC21i—C21i—C22i—C17i	−178.7
C4—C1—C2—C3	2.9 (3)	HC21i—C21i—C22i—HC22i	1.3
C4—C1—C2—H1C2	−116.6	Sii—C25i—C26i—H1C26i	−180.0
C4—C1—C2—H2C2	122.4	Sii—C25i—C26i—H2C26i	−60.0
C12—C1—C2—C3	−116.1 (2)	Sii—C25i—C26i—H3C26i	60.0
C12—C1—C2—H1C2	124.4	C27i—C25i—C26i—H1C26i	56.9
C12—C1—C2—H2C2	3.3	C27i—C25i—C26i—H2C26i	176.9
N1—C1—C4—O2	−121.2 (2)	C27i—C25i—C26i—H3C26i	−63.1
N1—C1—C4—O3	−4.2 (3)	C28i—C25i—C26i—H1C26i	−62.0
N1—C1—C4—HC4	116.7	C28i—C25i—C26i—H2C26i	58.0
C2—C1—C4—O2	−2.5 (3)	C28i—C25i—C26i—H3C26i	178.0
C2—C1—C4—O3	114.6 (2)	Sii—C25i—C27i—H1C27i	−180.0
C2—C1—C4—HC4	−124.5	Sii—C25i—C27i—H2C27i	−60.0
C12—C1—C4—O2	118.7 (2)	Sii—C25i—C27i—H3C27i	60.0
C12—C1—C4—O3	−124.2 (2)	C26i—C25i—C27i—H1C27i	−56.7
C12—C1—C4—HC4	−3.4	C26i—C25i—C27i—H2C27i	63.3
N1—C1—C12—O9	−168.4 (2)	C26i—C25i—C27i—H3C27i	−176.7

N1—C1—C12—C13	−46.1 (3)	C28i—C25i—C27i—H1C27i	60.4
N1—C1—C12—HC12	74.8	C28i—C25i—C27i—H2C27i	−179.6
C2—C1—C12—O9	59.2 (3)	C28i—C25i—C27i—H3C27i	−59.6
C2—C1—C12—C13	−178.5 (2)	Sii—C25i—C28i—H1C28i	180.0
C2—C1—C12—HC12	−57.6	Sii—C25i—C28i—H2C28i	−60.0
C4—C1—C12—O9	−55.7 (3)	Sii—C25i—C28i—H3C28i	60.0
C4—C1—C12—C13	66.6 (3)	C26i—C25i—C28i—H1C28i	59.7
C4—C1—C12—HC12	−172.5	C26i—C25i—C28i—H2C28i	179.7
C1—C2—C3—O1	177.0 (3)	C26i—C25i—C28i—H3C28i	−60.3
C1—C2—C3—O2	−2.5 (3)	C27i—C25i—C28i—H1C28i	−58.5
H1C2—C2—C3—O1	−63.6	C27i—C25i—C28i—H2C28i	61.5
H1C2—C2—C3—O2	116.9	C27i—C25i—C28i—H3C28i	−178.5

Fig. 1

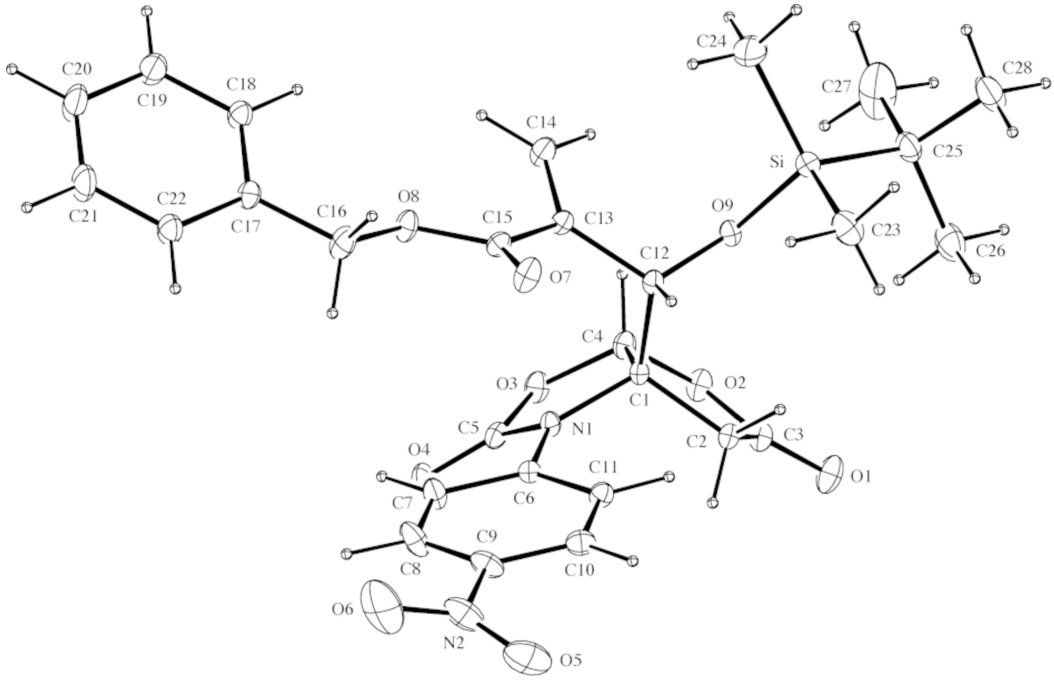

C24
C28
C27
C25
Si
C14
C20
C19
C18
C23
C26
O8
C15
C13
O9
C22
C21
C17
C16
O7
C12
C4
O3
O2
C1
C5
N1
C2
C3
O4
C11
O1
C7
C6
C9
C8
C10
O6
N2
O5

Fig. 2