Supporting Information for the Communication

Entitled

Sulfur-Substituted Tetrahedranes

Tatsumi Ochiai, Masaaki Nakamoto, Yusuke Inagaki, and Akira Sekiguchi*

Department of Chemistry, Graduate School of Pure and Applied Sciences,

University of Tsukuba, Tsukuba, Ibaraki 305-8571, Japan

Experimental Section

General Procedure. All experiments were performed using high-vacuum line techniques or in an argon atmosphere using an MBRAUN MB 150B-G glove box. All solvents were dried and degassed over potassium mirror in vacuum prior to use. NMR spectra were recorded on a Bruker AV-400FT NMR spectrometer (^{1}H NMR at 400 MHz; ^{13}C NMR at 100.6 MHz; ^{29}Si NMR at 79.5 MHz). High-resolution mass spectra were performed on Bruker Daltonics micrOTOF mass spectrometer with APCI (atmospheric pressure chemical ionization method). UV-Vis spectra were recorded on Shimadzu UV-3150 UV-Vis spectrophotometer. HPLC (JAIGEL-ODS column) and GPC (Gel Permeation Chromatography, JAIGEL-H column) separations were performed using recycling preparative JAI LC-918 and JAI LC-908W instruments, respectively. Tetrakis(trimethylsilyl)tetrahedrane [1] and tris(trimethylsilyl)tetrahedranyllithium **1** [2] were prepared according to the published procedures.

Experimental Procedure and Spectral Data for Phenyl Tris(trimethylsilyl)tetrahedranyl Sulfide (2).

Tris(trimethylsilyl)tetrahedranyllithium **1** was prepared by the reaction of tetrakis(trimethylsilyl)tetrahedrane (172 mg, 0.506 mmol) and MeLi (112 mg, 5.06 mmol) in THF (2 mL) as described previously [2]. After removal of the solvent, dry hexane (5 mL) was added to precipitate the remaining MeLi in hexane. Tetrahedranyllithium **1** is soluble in hexane and thus it was separated from the excess MeLi. The hexane was removed in vacuo and then diphenyl disulfide (165 mg, 0.757 mmol) in toluene (4 mL) was added to **1**. After 9 h stirring, the solvent was removed under vacuum and the residue was subjected to flash chromatography (SiO_2, eluent: THF) to remove the inorganic salts. Purification of the product was performed by HPLC

(eluent ^{t}BuOMe:MeOH = 1:1), giving **2** as a colorless oil (104 mg, 55%). ^{1}H NMR (C_6D_6, δ) 0.18 (s, 27 H, $SiMe_3$), 6.89 (t, J = 7.3 Hz, 1 H, ArH), 7.04 (t, J = 7.3 Hz, 2 H, ArH), 7.25 (d, J = 7.3 Hz, 2 H, ArH); ^{13}C NMR (C_6D_6, δ) –15.0 (*C*–$SiMe_3$), –0.1 (Si*Me*$_3$), 3.9 (*C*–S), 125.4, 127.2, 128.8, 138.9; ^{29}Si NMR (C_6D_6, δ) –2.2; UV-Vis (hexane) λ_{max}/nm (ε) 273 nm (7300); HRMS (APCI) *m/z*: calcd for $C_{19}H_{32}SSi_3$ (M^+) 376.1527, found: 376.1544.

Experimental Procedure and Spectral Data for 4-Nitrophenyl Tris(trimethylsilyl)tetrahedranyl Sulfide (3) and Crystallographic Data of 3.

In a similar manner to that for **2**, tris(trimethylsilyl)tetrahedranyllithium **1**, prepared by the reaction of tetrakis(trimethylsilyl)tetrahedrane (97 mg, 0.285 mmol) and MeLi (75 mg, 3.41 mmol) in THF, was reacted with bis(4-nitrophenyl) disulfide (132 mg, 0.428 mmol) in toluene (5 mL). The product was purified by HPLC (eluent ^{t}BuOMe:MeOH = 1:1) to afford **3** as yellow crystals (63 mg, 52%). Mp 51.5–54.0; ^{1}H NMR (C_6D_6, δ) 0.13 (s, 27 H, $SiMe_3$), 6.86 (d, J = 9.0 Hz, 2 H, ArH), 7.84 (d, J = 9.0 Hz, 2 H, ArH); ^{13}C NMR (C_6D_6, δ) –15.1 (*C*–$SiMe_3$), –0.2 (Si*Me*$_3$), 2.2 (*C*–S), 123.8, 125.5, 145.6, 148.6; ^{29}Si NMR (C_6D_6, δ) –2.3; UV-Vis (hexane) λ_{max}/nm (ε) 324 nm (11800); HRMS (APCI) *m/z*: calcd for $C_{19}H_{32}NO_2SSi_3$ ($[M + H]^+$) 422.1456, found: 422.14773.

The single crystals of **3** for X-ray diffraction analysis were grown from an acetonitrile solution. Diffraction data were collected at 150 K on a Bruker APEXII CCD area detector with a rotating anode (50 kV, 30 mA) employing graphite-monochromatized Mo-*K*α radiation (λ = 0.71073 Å). The structure was solved by the direct method, using SIR-92 program, and refined by the full-matrix least-squares method by SHELXL-97 program [3]. Crystal data for **3** at 150 K: MF = $C_{19}H_{31}NO_2SSi_3$, MW = 421.78, triclinic, space group P–1, a = 9.3927(5), b = 17.0136(9), c = 17.3220(9) Å, α = 111.9630(10), β = 95.4370(10), γ = 99.0200(10)º, V = 2499.7(2) Å^3, Z = 4,

D_{calcd} = 1.121 g/cm^3. The final R factor was 0.0322 (R_w = 0.0866 for all data) for 9204 reflections with $I > 2\sigma(I)$, GOF = 1.021.

Experimental Procedure and Spectral Data for 2,4-Dinitrophenyl Tris(trimethylsilyl)tetrahedranyl Sulfide (4) and Crystallographic Data of 4.

In a similar manner to that for **2**, tris(trimethylsilyl)tetrahedranyllithium **1**, prepared by the reaction of tetrakis(trimethylsilyl)tetrahedrane (100 mg, 0.294 mmol) and MeLi (65 mg, 2.95 mmol) in THF, was reacted with bis(2,4-dinitrophenyl) disulfide (142 mg, 0.356 mmol) in THF (5 mL). The product was purified by HPLC (eluent ^{t}BuOMe:MeOH = 1:1) to afford **4** as orange crystals (41 mg, 30%). Mp 91.5–93.5; ^{1}H NMR (C_6D_6, δ) 0.12 (s, 27 H, $SiMe_3$), 7.10 (d, J = 8.9 Hz, 1 H, ArH), 7.95 (dd, J = 8.9 Hz, J = 2.4 Hz, 1 H, ArH), 8.51 (d, J = 2.4 Hz, 1 H, ArH); ^{13}C NMR (C_6D_6, δ) –14.5 (*C*–$SiMe_3$), –0.3 (Si*Me*$_3$), 2.2 (*C*–S), 121.4, 126.1, 128.8, 143.7, 144.5, 148.6; ^{29}Si NMR (C_6D_6, δ) –2.4; UV-Vis (hexane) λ_{max}/nm (ε) 362 nm (7900); (APCI) *m/z*: calcd for $C_{19}H_{30}N_2O_4SSi_3$ (M^+) 466.1229, found: 466.1182.

The single crystals of **4** for X-ray diffraction analysis were grown from an acetonitrile solution. Diffraction data were collected at 120 K on a Bruker APEXII CCD area detector with a rotating anode (50 kV, 30 mA) employing graphite-monochromatized Mo-*K*α radiation (λ = 0.71073 Å). Crystal data for **4** at 120 K: MF = $C_{19}H_{30}N_2O_4SSi_3$, MW = 466.78, monoclinic, space group $P2_1/c$, a = 20.2544(19), b = 13.9007(13), c = 20.932(2) Å, β = 117.6900(10)º, V = 5218.5(9) Å^3, Z = 8, D_{calcd} = 1.188 g/cm^3. The final R factor was 0.0560 (R_w = 0.1613 for all data) for 9937 reflections with $I > 2\sigma(I)$, GOF = 1.112.

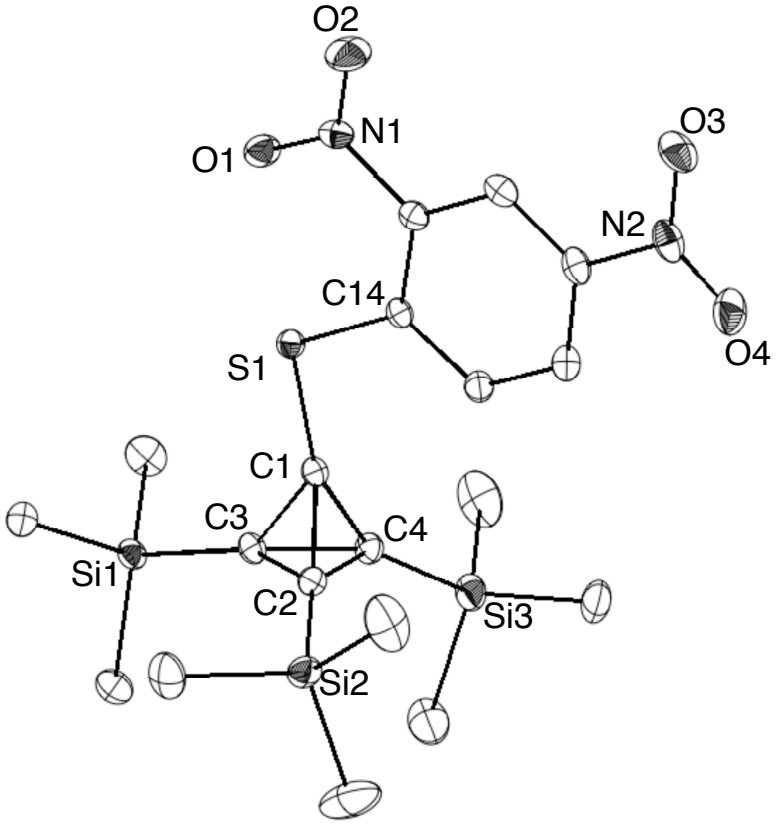


Figure S1. ORTEP drawing of **4** (30% thermal ellipsoids). Hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (deg): C1−C2 = 1.489(4), C1−C3 = 1.481(4), C1−C4 = 1.476(4), C2−C3 = 1.501(4), C3−C4 = 1.540(4), C4−C2 = 1.511(4), S1−C1 = 1.727(3), S1−C14 = 1.757(3), C2−Si1 = 1.836(3), C3−Si2 = 1.838(3), C4−Si3 = 1.836(3). C2−C1−C3 = 60.7(2), C3−C1−C4 = 62.8(2), C4−C1−C2 = 61.3(2), C1−C2−C3 = 59.4(2), C1−C2−C4 = 58.9(2), C1−C3−C4 = 58.5(2), C1−C3−C2 = 59.9(2), C1−C4−C2 = 59.80(19), C1−C4−C3 = 58.8(2), C2−C3−C4 = 59.6(2), C3−C4−C2 = 58.9(2), C4−C2−C3 = 61.5(2), C1−S1−C14 = 101.72(15).

Experimental Procedure and Spectral Data for Phenyl Tris(trimethylsilyl)tetrahedranyl Sulfone (5) and Crystallographic Data of 5.

Phenyl tris(trimethylsilyl)tetrahedranyl sulfide **2** (49 mg, 0.13 mmol) was reacted with *m*-chloroperbenzoic acid (43 mg, 0.25 mmol) in CH_2Cl_2 (0.5 mL) for 2 h. The product was purified by gel permeation chromatography (eluent: toluene) to give **5** as colorless crystals (53 mg, 90%). Mp 87.5–90.0; ^{1}H NMR (C_6D_6, δ) 0.11 (s, 27 H, $SiMe_3$), 6.92 (m, 3 H, ArH), 7.95 (d, J = 7.4 Hz, 2 H, ArH); ^{13}C NMR (C_6D_6, δ) –10.7 (*C*–$SiMe_3$), –0.6 (Si*Me*$_3$), 16.4 (*C*–S), 127.5, 128.8, 132.7, 143.6; ^{29}Si NMR (C_6D_6, δ) –2.3.; HRMS (APCI) *m/z*: calcd for $C_{19}H_{33}O_2SSi_3$ ([M + H]$^+$) 409.1504, found: 409.1507.

The single crystals of **5** for X-ray diffraction analysis were grown from a hexane solution. Diffraction data were collected at 120 K on a Bruker APEXII CCD area detector with a rotating anode (50 kV, 30 mA) employing graphite-monochromatized Mo-*K*α radiation (λ = 0.71073 Å). Crystal data for **5** at 120 K: MF = $C_{19}H_{32}O_2SSi_3$, MW = 408.78, monoclinic, space group $P2_1/c$, a = 17.366(3), b = 9.1430(16), c = 17.699(3) Å, β = 119.157(2)º, V = 2454.1(7) Å^3, Z = 4, D_{calcd} = 1.106 g/cm^3. The final R factor was 0.0321 (R_w = 0.0865 for all data) for 4590 reflections with $I > 2\sigma(I)$, GOF = 1.008.

Experimental Procedure for the Thermal Reaction of Phenyl Tris(trimethylsilyl)tetrahedranyl Sulfide (2).

A benzene solution of **2** (103 mg, 0.27 mmol) was sealed in an NMR tube and heated at 120 °C for 2 h. NMR analysis of the reaction mixture showed the complete absence of **2** and the formation of bis(trimethylsilyl)acetylene **6** (92%) and phenyl trimethylsilylethynyl sulfide **7** (92%). The spectral data of the products were compared with those of authentic samples [4].

Experimental Procedure for the Thermal Reaction of Phenyl Tris(trimethylsilyl)tetrahedranyl Sulfone (5).

A benzene solution of **5** (43 mg, 0.11 mmol) was sealed in an NMR tube and heated at 80 °C for 2 h. NMR analysis of the reaction mixture showed the isomerization of **5** to (phenylsulfonyl)tris(trimethylsilyl)cyclobutadiene **8**, which was obtained as a red powder (40 mg, 93%). ^{1}H NMR (C_6D_6, δ) 0.07 (s, 9 H, $SiMe_3$), 0.17 (s, 18 H, $SiMe_3$), 6.88–6.91 (m, 3 H, ArH), 7.98 (d, J = 6.4 Hz, 2 H, ArH); ^{13}C NMR (C_6D_6, δ) –0.3, –0.2, 127.2, 129.1, 132.7, 141.6, 154.6, 166.5, 169.2; ^{29}Si NMR (C_6D_6, δ) –12.9, –12.2; UV-Vis (hexane) λ_{max}/nm (ε) 346 nm (2000), 459 nm (200); HRMS (APCI) *m/z*: calcd for $C_{19}H_{33}O_2SSi_3$ ($[M + H]^+$), 409.1504, found 409.1528.

References

[1] Maier, G.; Nuedert, J.; Wolf, O.; Peppusch, D.; Sekiguchi, A. Tanaka, M.; Matsuo, T. *J. Am. Chem. Soc.* **2002**, *124*, 13819.

[2] Sekiguchi, A.; Tanaka, M. *J. Am. Chem. Soc*. **2003**, *125*, 12684.

[3] Sheldrick, G. M. *Acta Cryst.* **2008**, *A64*, 112..

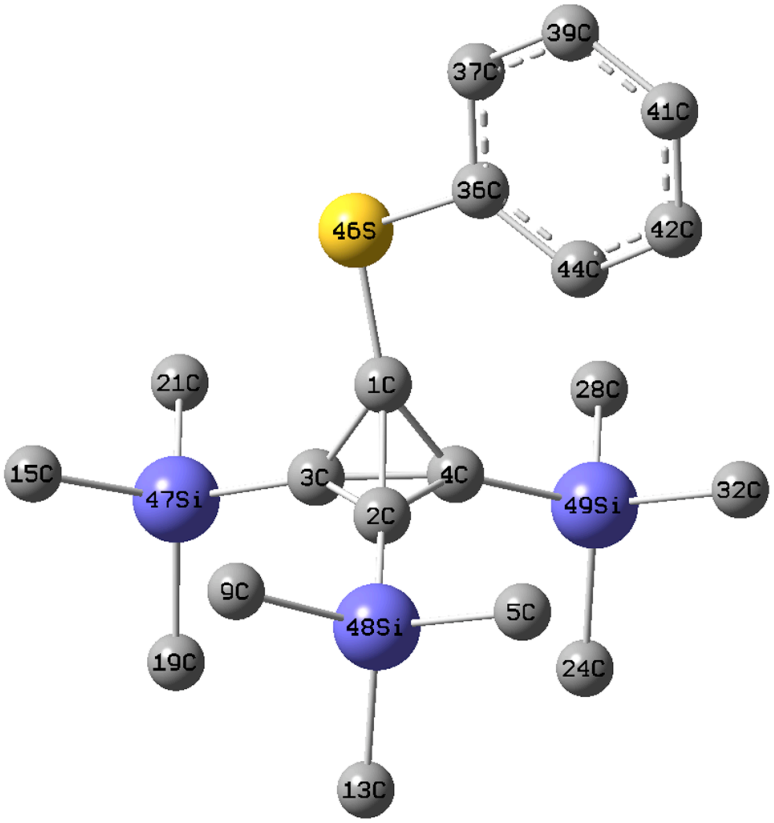
[4] Herunsalee, A.; Isobe, M.; Fukuda, Y.; Goto, T. *Synlett* **1990**, *11*, 701.

Table 1. Energy and atomic coordinates of **2**

Energy = -2009.95761289 A.U. (B3LYP/6-31G(d))

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	6	0	-0.115065	-0.112466	-0.686143
2	6	0	0.516611	-0.668009	0.530686
3	6	0	1.367053	-0.145790	-0.593468
4	6	0	0.568334	0.831253	0.225624
5	6	0	-1.063257	-1.646061	2.969386
6	1	0	-1.017863	-0.670789	3.468819
7	1	0	-1.131698	-2.413843	3.750197
8	1	0	-1.989848	-1.681181	2.385503
9	6	0	0.355039	-3.653714	1.090571
10	1	0	-0.539992	-3.748068	0.464985
11	1	0	0.311692	-4.437816	1.856712
12	1	0	1.225894	-3.855900	0.456205
13	6	0	2.024474	-1.794099	2.929936
14	1	0	2.122123	-0.795498	3.371804
15	6	0	2.886881	-2.151591	-2.314418
16	1	0	2.876660	-2.911860	-1.524633
17	1	0	3.774695	-2.329027	-2.934173
18	1	0	2.003845	-2.313933	-2.943179
19	6	0	4.418423	-0.179078	-0.479209
20	1	0	4.454459	0.829131	-0.050419
21	6	0	2.943561	0.867569	-2.997395
22	1	0	2.071019	0.761388	-3.652270
23	1	0	2.946315	1.894134	-2.612691
24	6	0	2.193052	2.675752	2.033735
25	1	0	2.202562	1.936629	2.843536
26	1	0	2.255994	3.670294	2.492761
27	1	0	3.101338	2.527319	1.438397
28	6	0	0.667383	3.802668	-0.421569
29	1	0	1.538117	3.661597	-1.072424
30	1	0	0.713329	4.822694	-0.020120
31	1	0	-0.229721	3.730060	-1.047078
32	6	0	-0.891157	2.816272	2.059937
33	1	0	-1.820582	2.734387	1.485397
34	1	0	-0.861313	3.820948	2.500032
35	1	0	-0.941051	2.096157	2.885517
36	6	0	-2.860255	-0.192446	-1.210994
37	6	0	-3.974758	-0.433342	-2.027736
38	1	0	-3.834685	-0.706809	-3.070763
39	6	0	-5.261077	-0.323841	-1.503516
40	1	0	-6.117374	-0.512744	-2.145686
41	6	0	-5.452607	0.021388	-0.163120
42	6	0	-4.342436	0.257754	0.647584
43	1	0	-4.477630	0.527151	1.692006

44	6	0	-3.048202	0.154647	0.131401
45	1	0	-2.193253	0.342960	0.769717
46	16	0	-1.251733	-0.340696	-1.984491
47	14	0	2.906189	-0.402060	-1.594643
48	14	0	0.455980	-1.937465	1.879247
49	14	0	0.631544	2.525057	0.973408
50	1	0	2.011179	-2.520385	3.752173
51	1	0	2.925204	-1.983693	2.334538
52	1	0	4.412971	-0.893568	0.352185
53	1	0	5.346480	-0.333018	-1.043643
54	1	0	3.841575	0.741880	-3.615003
55	1	0	-6.456682	0.103908	0.243533



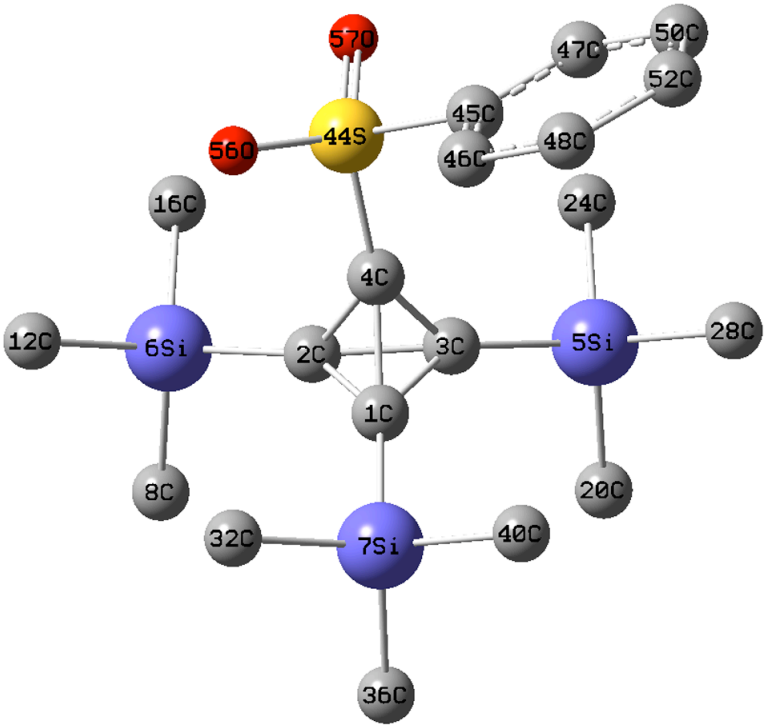
Selected Bond Lengths (Å): C1-C2 = 1.47930, C2-C3 = 1.50323, C3-C4 = 1.50449, C1-C3 = 1.48539, C2-C4 = 1.53086, C1-S46 = 1.74063, S46-C36 = 1.79098, C2-Si48 = 1.85305, C4-49Si = 1.85261, C3-Si47 = 1.85391.

Table 2. Energy and atomic coordinates of **5**

Energy = -2160.34422963 A.U. (B3LYP/6-31G(d))

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	6	0	-0.653604	0.774103	0.605602
2	6	0	-1.523508	-0.010999	-0.333765
3	6	0	-0.648710	-0.747887	0.638450
4	6	0	-0.044354	-0.008556	-0.486018
5	14	0	-0.509169	-2.264205	1.701518
6	14	0	-3.033887	-0.036772	-1.423505
7	14	0	-0.532852	2.331455	1.610049
8	6	0	-4.581700	-0.017295	-0.334646
9	1	0	-4.622304	0.881723	0.291661
10	1	0	-5.489434	-0.032545	-0.950579
11	1	0	-4.618112	-0.889010	0.329368
12	6	0	-2.968658	1.497526	-2.522715
13	1	0	-2.004917	1.561102	-3.040366
14	1	0	-3.762546	1.476638	-3.279426
15	1	0	-3.092010	2.415882	-1.936451
16	6	0	-2.963301	-1.613506	-2.460508
17	1	0	-3.754059	-1.622496	-3.220719
18	1	0	-1.997413	-1.696627	-2.971304
19	1	0	-3.088748	-2.508357	-1.839450
20	6	0	-1.937415	-2.266848	2.942299
21	1	0	-2.908270	-2.266733	2.433197
22	1	0	-1.897842	-3.157891	3.581026
23	1	0	-1.904601	-1.388195	3.597142
24	6	0	-0.606334	-3.779936	0.577388
25	1	0	-0.410380	-4.703054	1.136643
26	1	0	-1.598960	-3.871029	0.121306
27	1	0	0.122589	-3.715108	-0.238497
28	6	0	1.145371	-2.221122	2.617053
29	1	0	1.270888	-3.116007	3.239206
30	1	0	1.989992	-2.176776	1.920145
31	1	0	1.214024	-1.347041	3.275277
32	6	0	-0.635188	3.804743	0.431086
33	1	0	-1.621833	3.864666	-0.042793
34	1	0	-0.462224	4.749601	0.960918
35	1	0	0.107226	3.724417	-0.371125
36	6	0	-1.969783	2.368680	2.839935
37	1	0	-1.941032	3.282441	3.446317
38	1	0	-2.936852	2.343151	2.324317
39	1	0	-1.935650	1.514435	3.526233
40	6	0	1.115849	2.336896	2.537112
41	1	0	1.235045	3.260639	3.116866
42	1	0	1.182668	1.495242	3.236530
43	1	0	1.964929	2.262023	1.848263

44	16	0	1.060722	-0.039331	-1.824136
45	6	0	2.694268	-0.015967	-1.067691
46	6	0	3.310464	1.210882	-0.811998
47	6	0	3.327278	-1.225542	-0.774243
48	6	0	4.577519	1.222197	-0.227483
49	1	0	2.810479	2.132612	-1.090554
50	6	0	4.594518	-1.201176	-0.190335
51	1	0	2.839573	-2.162109	-1.023388
52	6	0	5.215139	0.019416	0.086736
53	1	0	5.070454	2.169353	-0.027490
54	1	0	5.100564	-2.134764	0.038607
55	1	0	6.203000	0.033304	0.538919
56	8	0	0.893081	1.220845	-2.568812
57	8	0	0.899394	-1.337158	-2.502946



Selected Bond Lengths (Å): C1-C2 = 1.50184, C2-C3 = 1.50116, C3-C4 = 1.47522, C1-C3 = 1.52235, C2-C4 = 1.48697, C1-Si7 = 1.85711, C2-Si6 = 1.86264, C3-Si5 = 1.85710, C4-S44 = 1.73571, S44-C45 = 1.80034.

Table 3. Crystal data and structure refinement for **3**.

Identification code	tdsc6h4no2_0m
Empirical formula	C19 H31 N O2 S Si3
Formula weight	421.78
Temperature	150 K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 9.3927(5) Å α = 111.9630(10)°
	b = 17.0136(9) Å β = 95.4370(10)°
	c = 17.3220(9) Å γ = 99.0200(10)°
Volume	2499.7(2) Å^3
Z	4
Density (calculated)	1.121 Mg/m^3
Absorption coefficient	0.286 mm^-1
F(000)	904
Crystal size	0.39 x 0.31 x 0.19 mm^3
Theta range for data collection	1.29 to 27.45° .
Index ranges	-12<=h<=12, -21<=k<=21, -22<=l<=22
Reflections collected	27789
Independent reflections	11075 [R(int) = 0.0203]
Completeness to theta = 27.45°	96.8 %
Absorption correction	Empirical
Max. and min. transmission	0.9477 and 0.8968
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	11075 / 0 / 487
Goodness-of-fit on F^2	1.021
Final R indices [I>2sigma(I)]	R1 = 0.0322, wR2 = 0.0805
R indices (all data)	R1 = 0.0418, wR2 = 0.0866
Largest diff. peak and hole	0.294 and -0.307 e.Å^-3

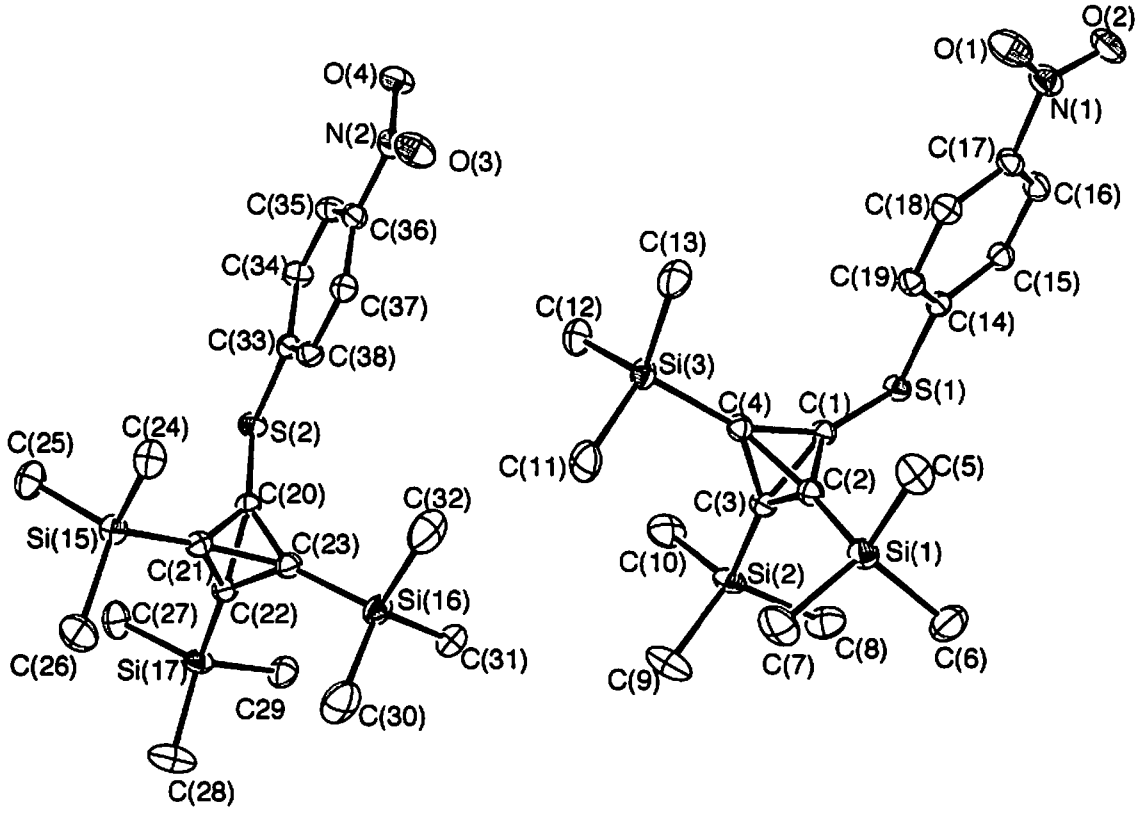


Figure S-2. ORTEP drawing of **3** (30% thermal ellipsoids). Hydrogen atoms are omitted for clarity.

Table 4. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2x 10^3) for **3**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	9161(1)	7947(1)	2706(1)	25(1)
C(2)	9961(2)	7455(1)	2055(1)	28(1)
C(3)	8451(2)	7049(1)	2109(1)	28(1)
C(4)	9690(2)	7212(1)	2809(1)	28(1)
C(5)	12934(2)	8256(1)	1878(1)	50(1)
C(6)	10231(2)	7906(1)	570(1)	58(1)
C(7)	11470(2)	6401(1)	684(1)	58(1)
C(8)	5908(2)	7046(1)	969(1)	54(1)
C(9)	7029(2)	5375(1)	691(1)	64(1)
C(10)	5463(2)	6134(1)	2175(1)	56(1)
C(11)	10892(2)	5660(1)	2551(1)	53(1)
C(12)	9129(2)	6297(1)	3981(1)	46(1)
C(13)	12167(2)	7329(1)	4120(1)	54(1)
C(14)	10133(2)	9629(1)	3791(1)	26(1)
C(15)	9983(2)	10480(1)	4236(1)	32(1)
C(16)	11156(2)	11089(1)	4783(1)	35(1)
C(17)	12468(2)	10842(1)	4894(1)	32(1)
C(18)	12647(2)	10006(1)	4465(1)	32(1)
C(19)	11471(2)	9399(1)	3903(1)	29(1)
C(20)	4275(2)	2421(1)	3099(1)	28(1)
C(21)	5167(2)	1794(1)	2666(1)	31(1)
C(22)	3638(2)	1726(1)	2259(1)	30(1)
C(23)	4824(2)	2483(1)	2347(1)	32(1)
C(24)	8283(2)	1920(1)	3338(1)	45(1)
C(25)	5777(2)	626(1)	3510(1)	48(1)
C(26)	6628(2)	329(1)	1782(1)	59(1)
C(27)	1170(2)	411(1)	2251(1)	52(1)
C(28)	2274(2)	264(2)	608(1)	70(1)
C(29)	642(2)	1700(1)	1474(1)	49(1)
C(30)	6031(3)	2258(2)	776(1)	69(1)
C(31)	4023(2)	3537(1)	1382(1)	50(1)
C(32)	7105(3)	3945(2)	2350(2)	80(1)
C(33)	5142(2)	3683(1)	4684(1)	27(1)
C(34)	4948(2)	4178(1)	5502(1)	34(1)
C(35)	6054(2)	4844(1)	6044(1)	36(1)

C(36)	7360(2)	5011(1)	5765(1)	31(1)
C(37)	7586(2)	4521(1)	4965(1)	31(1)
C(38)	6472(2)	3850(1)	4424(1)	29(1)
N(1)	13690(2)	11478(1)	5512(1)	42(1)
N(2)	8533(2)	5725(1)	6336(1)	38(1)
O(1)	14761(1)	11217(1)	5712(1)	58(1)
O(2)	13578(2)	12239(1)	5812(1)	56(1)
O(3)	9670(1)	5876(1)	6075(1)	55(1)
O(4)	8335(1)	6136(1)	7052(1)	49(1)
S(1)	8557(1)	8896(1)	3128(1)	29(1)
S(2)	3629(1)	2883(1)	4026(1)	32(1)
Si(1)	11168(1)	7507(1)	1299(1)	35(1)
Si(2)	6685(1)	6390(1)	1484(1)	35(1)
Si(3)	10484(1)	6627(1)	3385(1)	32(1)
Si(4)	1910(1)	1014(1)	1632(1)	33(1)
Si(5)	6492(1)	1161(1)	2821(1)	34(1)
Si(6)	5506(1)	3063(1)	1711(1)	41(1)

Table 5. Bond lengths [Å] and angles [deg] for 3.

C(1)-C(2)	1.4792(19)
C(1)-C(4)	1.4794(18)
C(1)-C(3)	1.4876(18)
C(1)-S(1)	1.7167(13)
C(2)-C(3)	1.5041(19)
C(2)-C(4)	1.5410(19)
C(2)-Si(1)	1.8293(15)
C(3)-C(4)	1.5103(19)
C(3)-Si(2)	1.8301(14)
C(4)-Si(3)	1.8362(14)
C(5)-Si(1)	1.8573(18)
C(5)-H(5A)	0.9800
C(5)-H(5B)	0.9800
C(5)-H(5C)	0.9800
C(6)-Si(1)	1.8576(19)
C(6)-H(6A)	0.9800
C(6)-H(6B)	0.9800
C(6)-H(6C)	0.9800
C(7)-Si(1)	1.8647(18)
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(7)-H(7C)	0.9800
C(8)-Si(2)	1.8578(19)
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-Si(2)	1.8599(18)
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-Si(2)	1.8537(19)
C(10)-H(10A)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
C(11)-Si(3)	1.8618(19)
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800

C(12)-Si(3)	1.8605(17)
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-Si(3)	1.8542(17)
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-C(19)	1.3910(19)
C(14)-C(15)	1.3998(19)
C(14)-S(1)	1.7591(14)
C(15)-C(16)	1.377(2)
C(15)-H(15)	0.9500
C(16)-C(17)	1.382(2)
C(16)-H(16)	0.9500
C(17)-C(18)	1.382(2)
C(17)-N(1)	1.4649(19)
C(18)-C(19)	1.386(2)
C(18)-H(18)	0.9500
C(19)-H(19)	0.9500
C(20)-C(23)	1.4785(19)
C(20)-C(22)	1.4832(19)
C(20)-C(21)	1.485(2)
C(20)-S(2)	1.7189(14)
C(21)-C(22)	1.505(2)
C(21)-C(23)	1.534(2)
C(21)-Si(5)	1.8314(15)
C(22)-C(23)	1.5145(19)
C(22)-Si(4)	1.8294(15)
C(23)-Si(6)	1.8297(15)
C(24)-Si(5)	1.8616(17)
C(24)-H(24A)	0.9800
C(24)-H(24B)	0.9800
C(24)-H(24C)	0.9800
C(25)-Si(5)	1.8645(17)
C(25)-H(25A)	0.9800
C(25)-H(25B)	0.9800
C(25)-H(25C)	0.9800
C(26)-Si(5)	1.8593(19)
C(26)-H(26A)	0.9800
C(26)-H(26B)	0.9800

C(26)-H(26C)	0.9800
C(27)-Si(4)	1.8525(18)
C(27)-H(27A)	0.9800
C(27)-H(27B)	0.9800
C(27)-H(27C)	0.9800
C(28)-Si(4)	1.8577(19)
C(28)-H(28A)	0.9800
C(28)-H(28B)	0.9800
C(28)-H(28C)	0.9800
C(29)-Si(4)	1.8614(18)
C(29)-H(29A)	0.9800
C(29)-H(29B)	0.9800
C(29)-H(29C)	0.9800
C(30)-Si(6)	1.856(2)
C(30)-H(30A)	0.9800
C(30)-H(30B)	0.9800
C(30)-H(30C)	0.9800
C(31)-Si(6)	1.8606(19)
C(31)-H(31A)	0.9800
C(31)-H(31B)	0.9800
C(31)-H(31C)	0.9800
C(32)-Si(6)	1.862(2)
C(32)-H(32A)	0.9800
C(32)-H(32B)	0.9800
C(32)-H(32C)	0.9800
C(33)-C(38)	1.392(2)
C(33)-C(34)	1.398(2)
C(33)-S(2)	1.7591(14)
C(34)-C(35)	1.376(2)
C(34)-H(34)	0.9500
C(35)-C(36)	1.387(2)
C(35)-H(35)	0.9500
C(36)-C(37)	1.381(2)
C(36)-N(2)	1.4642(19)
C(37)-C(38)	1.384(2)
C(37)-H(37)	0.9500
C(38)-H(38)	0.9500
N(1)-O(2)	1.2263(18)
N(1)-O(1)	1.2325(19)
N(2)-O(4)	1.2226(17)
N(2)-O(3)	1.2269(18)

C(2)-C(1)-C(4)	62.78(9)
C(2)-C(1)-C(3)	60.93(9)
C(4)-C(1)-C(3)	61.20(9)
C(2)-C(1)-S(1)	144.61(11)
C(4)-C(1)-S(1)	149.98(11)
C(3)-C(1)-S(1)	134.11(10)
C(1)-C(2)-C(3)	59.81(9)
C(1)-C(2)-C(4)	58.61(9)
C(3)-C(2)-C(4)	59.45(9)
C(1)-C(2)-Si(1)	145.67(11)
C(3)-C(2)-Si(1)	142.29(11)
C(4)-C(2)-Si(1)	147.45(10)
C(1)-C(3)-C(2)	59.26(9)
C(1)-C(3)-C(4)	59.13(9)
C(2)-C(3)-C(4)	61.49(9)
C(1)-C(3)-Si(2)	141.82(10)
C(2)-C(3)-Si(2)	143.91(11)
C(4)-C(3)-Si(2)	148.06(11)
C(1)-C(4)-C(3)	59.67(9)
C(1)-C(4)-C(2)	58.60(9)
C(3)-C(4)-C(2)	59.06(9)
C(1)-C(4)-Si(3)	156.40(11)
C(3)-C(4)-Si(3)	139.33(10)
C(2)-C(4)-Si(3)	138.02(10)
Si(1)-C(5)-H(5A)	109.5
Si(1)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
Si(1)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
Si(1)-C(6)-H(6A)	109.5
Si(1)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
Si(1)-C(6)-H(6C)	109.5
H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5
Si(1)-C(7)-H(7A)	109.5
Si(1)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
Si(1)-C(7)-H(7C)	109.5

H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
Si(2)-C(8)-H(8A)	109.5
Si(2)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
Si(2)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
Si(2)-C(9)-H(9A)	109.5
Si(2)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
Si(2)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
Si(2)-C(10)-H(10A)	109.5
Si(2)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
Si(2)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
Si(3)-C(11)-H(11A)	109.5
Si(3)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
Si(3)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
Si(3)-C(12)-H(12A)	109.5
Si(3)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
Si(3)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
Si(3)-C(13)-H(13A)	109.5
Si(3)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
Si(3)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(19)-C(14)-C(15)	119.90(13)
C(19)-C(14)-S(1)	123.60(11)
C(15)-C(14)-S(1)	116.48(11)

C(16)-C(15)-C(14)	120.14(14)
C(16)-C(15)-H(15)	119.9
C(14)-C(15)-H(15)	119.9
C(15)-C(16)-C(17)	118.92(13)
C(15)-C(16)-H(16)	120.5
C(17)-C(16)-H(16)	120.5
C(16)-C(17)-C(18)	122.19(14)
C(16)-C(17)-N(1)	118.79(14)
C(18)-C(17)-N(1)	118.98(14)
C(17)-C(18)-C(19)	118.70(14)
C(17)-C(18)-H(18)	120.7
C(19)-C(18)-H(18)	120.7
C(18)-C(19)-C(14)	120.14(13)
C(18)-C(19)-H(19)	119.9
C(14)-C(19)-H(19)	119.9
C(23)-C(20)-C(22)	61.51(9)
C(23)-C(20)-C(21)	62.35(10)
C(22)-C(20)-C(21)	60.95(10)
C(23)-C(20)-S(2)	150.31(11)
C(22)-C(20)-S(2)	134.84(11)
C(21)-C(20)-S(2)	144.07(11)
C(20)-C(21)-C(22)	59.48(9)
C(20)-C(21)-C(23)	58.63(9)
C(22)-C(21)-C(23)	59.77(9)
C(20)-C(21)-Si(5)	144.43(11)
C(22)-C(21)-Si(5)	143.78(11)
C(23)-C(21)-Si(5)	147.23(11)
C(20)-C(22)-C(21)	59.57(9)
C(20)-C(22)-C(23)	59.09(9)
C(21)-C(22)-C(23)	61.05(10)
C(20)-C(22)-Si(4)	142.21(11)
C(21)-C(22)-Si(4)	147.11(11)
C(23)-C(22)-Si(4)	144.73(11)
C(20)-C(23)-C(22)	59.40(9)
C(20)-C(23)-C(21)	59.02(9)
C(22)-C(23)-C(21)	59.17(9)
C(20)-C(23)-Si(6)	154.27(12)
C(22)-C(23)-Si(6)	139.15(11)
C(21)-C(23)-Si(6)	140.89(11)
Si(5)-C(24)-H(24A)	109.5
Si(5)-C(24)-H(24B)	109.5

H(24A)-C(24)-H(24B)	109.5
Si(5)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
Si(5)-C(25)-H(25A)	109.5
Si(5)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
Si(5)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
Si(5)-C(26)-H(26A)	109.5
Si(5)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
Si(5)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
Si(4)-C(27)-H(27A)	109.5
Si(4)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	109.5
Si(4)-C(27)-H(27C)	109.5
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
Si(4)-C(28)-H(28A)	109.5
Si(4)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
Si(4)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
Si(4)-C(29)-H(29A)	109.5
Si(4)-C(29)-H(29B)	109.5
H(29A)-C(29)-H(29B)	109.5
Si(4)-C(29)-H(29C)	109.5
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
Si(6)-C(30)-H(30A)	109.5
Si(6)-C(30)-H(30B)	109.5
H(30A)-C(30)-H(30B)	109.5
Si(6)-C(30)-H(30C)	109.5
H(30A)-C(30)-H(30C)	109.5
H(30B)-C(30)-H(30C)	109.5
Si(6)-C(31)-H(31A)	109.5

Si(6)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
Si(6)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
Si(6)-C(32)-H(32A)	109.5
Si(6)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
Si(6)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(38)-C(33)-C(34)	119.83(13)
C(38)-C(33)-S(2)	123.52(11)
C(34)-C(33)-S(2)	116.63(11)
C(35)-C(34)-C(33)	120.26(14)
C(35)-C(34)-H(34)	119.9
C(33)-C(34)-H(34)	119.9
C(34)-C(35)-C(36)	118.94(14)
C(34)-C(35)-H(35)	120.5
C(36)-C(35)-H(35)	120.5
C(37)-C(36)-C(35)	121.86(13)
C(37)-C(36)-N(2)	119.11(13)
C(35)-C(36)-N(2)	119.04(13)
C(36)-C(37)-C(38)	119.00(13)
C(36)-C(37)-H(37)	120.5
C(38)-C(37)-H(37)	120.5
C(37)-C(38)-C(33)	120.09(13)
C(37)-C(38)-H(38)	120.0
C(33)-C(38)-H(38)	120.0
O(2)-N(1)-O(1)	123.69(14)
O(2)-N(1)-C(17)	118.18(15)
O(1)-N(1)-C(17)	118.12(14)
O(4)-N(2)-O(3)	123.31(14)
O(4)-N(2)-C(36)	118.42(14)
O(3)-N(2)-C(36)	118.27(13)
C(1)-S(1)-C(14)	102.91(7)
C(20)-S(2)-C(33)	103.18(7)
C(2)-Si(1)-C(5)	109.25(8)
C(2)-Si(1)-C(6)	107.26(8)
C(5)-Si(1)-C(6)	110.89(9)
C(2)-Si(1)-C(7)	109.62(8)

C(5)-Si(1)-C(7)	110.46(9)
C(6)-Si(1)-C(7)	109.29(10)
C(3)-Si(2)-C(10)	110.30(7)
C(3)-Si(2)-C(8)	106.22(8)
C(10)-Si(2)-C(8)	110.88(9)
C(3)-Si(2)-C(9)	107.74(8)
C(10)-Si(2)-C(9)	110.15(10)
C(8)-Si(2)-C(9)	111.45(10)
C(4)-Si(3)-C(13)	109.88(7)
C(4)-Si(3)-C(12)	109.64(7)
C(13)-Si(3)-C(12)	110.04(9)
C(4)-Si(3)-C(11)	104.88(7)
C(13)-Si(3)-C(11)	111.48(10)
C(12)-Si(3)-C(11)	110.80(9)
C(22)-Si(4)-C(27)	106.69(8)
C(22)-Si(4)-C(28)	108.87(8)
C(27)-Si(4)-C(28)	111.01(10)
C(22)-Si(4)-C(29)	108.42(7)
C(27)-Si(4)-C(29)	110.30(9)
C(28)-Si(4)-C(29)	111.39(10)
C(21)-Si(5)-C(26)	109.23(8)
C(21)-Si(5)-C(24)	108.10(7)
C(26)-Si(5)-C(24)	111.32(9)
C(21)-Si(5)-C(25)	106.64(7)
C(26)-Si(5)-C(25)	110.00(10)
C(24)-Si(5)-C(25)	111.39(8)
C(23)-Si(6)-C(30)	107.03(9)
C(23)-Si(6)-C(31)	108.80(8)
C(30)-Si(6)-C(31)	110.73(10)
C(23)-Si(6)-C(32)	109.50(8)
C(30)-Si(6)-C(32)	111.13(12)
C(31)-Si(6)-C(32)	109.57(11)

Symmetry transformations used to generate equivalent atoms:

Table 6. Anisotropic displacement parameters (Å^2x 10^3) for 3. The anisotropic displacement factor exponent takes the form:
-2pi^2[h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	25(1)	24(1)	25(1)	9(1)	2(1)	5(1)
C(2)	29(1)	27(1)	29(1)	9(1)	4(1)	9(1)
C(3)	28(1)	26(1)	27(1)	7(1)	2(1)	6(1)
C(4)	25(1)	26(1)	30(1)	11(1)	1(1)	3(1)
C(5)	39(1)	47(1)	60(1)	16(1)	18(1)	9(1)
C(6)	69(1)	68(1)	46(1)	32(1)	12(1)	15(1)
C(7)	65(1)	44(1)	60(1)	7(1)	31(1)	17(1)
C(8)	42(1)	72(1)	47(1)	22(1)	-3(1)	20(1)
C(9)	49(1)	46(1)	61(1)	-13(1)	-4(1)	9(1)
C(10)	40(1)	58(1)	55(1)	16(1)	3(1)	-8(1)
C(11)	61(1)	49(1)	62(1)	30(1)	17(1)	29(1)
C(12)	47(1)	43(1)	55(1)	28(1)	12(1)	5(1)
C(13)	39(1)	60(1)	65(1)	40(1)	-16(1)	-4(1)
C(14)	30(1)	25(1)	24(1)	10(1)	6(1)	4(1)
C(15)	37(1)	29(1)	32(1)	12(1)	8(1)	10(1)
C(16)	46(1)	25(1)	32(1)	9(1)	11(1)	7(1)
C(17)	34(1)	29(1)	29(1)	8(1)	7(1)	-3(1)
C(18)	27(1)	34(1)	34(1)	12(1)	10(1)	4(1)
C(19)	30(1)	26(1)	29(1)	8(1)	9(1)	6(1)
C(20)	28(1)	29(1)	26(1)	11(1)	1(1)	4(1)
C(21)	30(1)	33(1)	29(1)	13(1)	5(1)	5(1)
C(22)	31(1)	30(1)	26(1)	11(1)	3(1)	3(1)
C(23)	32(1)	33(1)	29(1)	14(1)	1(1)	2(1)
C(24)	32(1)	50(1)	61(1)	29(1)	10(1)	11(1)
C(25)	38(1)	55(1)	62(1)	37(1)	9(1)	8(1)
C(26)	62(1)	54(1)	57(1)	12(1)	20(1)	22(1)
C(27)	48(1)	43(1)	72(1)	31(1)	19(1)	7(1)
C(28)	58(1)	75(1)	42(1)	-8(1)	10(1)	-5(1)
C(29)	41(1)	53(1)	52(1)	27(1)	-8(1)	0(1)
C(30)	81(2)	90(2)	65(1)	48(1)	41(1)	36(1)
C(31)	63(1)	53(1)	43(1)	27(1)	7(1)	12(1)
C(32)	64(1)	89(2)	88(2)	62(2)	-18(1)	-34(1)
C(33)	27(1)	27(1)	28(1)	11(1)	2(1)	7(1)
C(34)	31(1)	39(1)	32(1)	11(1)	9(1)	8(1)

C(35)	38(1)	36(1)	28(1)	7(1)	6(1)	10(1)
C(36)	31(1)	26(1)	32(1)	10(1)	-1(1)	7(1)
C(37)	28(1)	33(1)	35(1)	15(1)	5(1)	8(1)
C(38)	30(1)	32(1)	27(1)	10(1)	5(1)	10(1)
N(1)	41(1)	37(1)	37(1)	7(1)	9(1)	-6(1)
N(2)	39(1)	31(1)	39(1)	10(1)	-3(1)	7(1)
O(1)	35(1)	55(1)	63(1)	6(1)	-1(1)	-4(1)
O(2)	65(1)	31(1)	52(1)	3(1)	4(1)	-7(1)
O(3)	37(1)	51(1)	59(1)	8(1)	4(1)	-5(1)
O(4)	56(1)	39(1)	36(1)	3(1)	-2(1)	4(1)
S(1)	27(1)	28(1)	29(1)	8(1)	3(1)	9(1)
S(2)	27(1)	34(1)	30(1)	9(1)	4(1)	4(1)
Si(1)	39(1)	33(1)	35(1)	11(1)	14(1)	10(1)
Si(2)	27(1)	34(1)	32(1)	2(1)	-2(1)	6(1)
Si(3)	28(1)	32(1)	40(1)	20(1)	0(1)	4(1)
Si(4)	31(1)	32(1)	28(1)	7(1)	3(1)	-1(1)
Si(5)	31(1)	36(1)	39(1)	17(1)	10(1)	10(1)
Si(6)	39(1)	46(1)	39(1)	25(1)	4(1)	-2(1)

Table 7. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2x 10^3 for **3**.

	x	y	z	U(eq)
H(5A)	12752	8824	2226	75
H(5B)	13556	8320	1472	75
H(5C)	13427	8021	2241	75
H(6A)	9362	7470	225	87
H(6B)	10899	8014	202	87
H(6C)	9938	8446	899	87
H(7A)	12017	6203	1061	87
H(7B)	12028	6420	238	87
H(7C)	10524	6001	428	87
H(8A)	5768	7584	1402	81
H(8B)	4965	6715	614	81
H(8C)	6582	7186	618	81
H(9A)	7540	5509	273	95
H(9B)	6096	4971	407	95
H(9C)	7635	5111	975	95
H(10A)	5903	5799	2449	84
H(10B)	4514	5794	1835	84
H(10C)	5326	6674	2607	84
H(11A)	10072	5407	2079	79
H(11B)	11038	5230	2785	79
H(11C)	11782	5833	2350	79
H(12A)	8879	6814	4390	69
H(12B)	9550	5978	4281	69
H(12C)	8245	5926	3587	69
H(13A)	12867	7507	3802	81
H(13B)	12601	7008	4413	81
H(13C)	11921	7844	4535	81
H(15)	9069	10637	4159	38
H(16)	11065	11670	5080	42
H(18)	13558	9850	4555	38
H(19)	11579	8824	3593	35
H(24A)	8540	2271	3014	68
H(24B)	9039	1588	3359	68
H(24C)	8212	2301	3914	68
H(25A)	5825	1065	4078	72

H(25B)	6370	209	3538	72
H(25C)	4759	322	3274	72
H(26A)	5672	-60	1525	88
H(26B)	7346	-5	1861	88
H(26C)	6937	615	1410	88
H(27A)	1863	68	2345	77
H(27B)	234	24	1940	77
H(27C)	1022	821	2796	77
H(28A)	2633	596	284	105
H(28B)	1369	-152	287	105
H(28C)	3013	-49	712	105
H(29A)	561	2133	2023	74
H(29B)	-325	1334	1194	74
H(29C)	1022	1993	1122	74
H(30A)	6842	2035	961	104
H(30B)	6337	2536	398	104
H(30C)	5192	1778	477	104
H(31A)	3218	3070	1004	76
H(31B)	4404	3896	1088	76
H(31C)	3665	3896	1883	76
H(32A)	6810	4342	2855	120
H(32B)	7457	4262	2013	120
H(32C)	7887	3693	2520	120
H(34)	4049	4055	5685	41
H(35)	5925	5185	6599	43
H(37)	8493	4643	4789	37
H(38)	6615	3503	3874	35

Table 8. Crystal data and structure refinement for **4**.

Identification code	dinitro2_0m
Empirical formula	C19 H30 N2 O4 S Si3
Formula weight	466.78
Temperature	120 K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 20.2544(19) Å α= 90°
	b = 13.9007(13) Å β=117.6900(10)°
	c = 20.932(2) Å γ = 90°
Volume	5218.5(9) Å^3
Z	8
Density (calculated)	1.188 Mg/m^3
Absorption coefficient	0.286 mm^-1
F(000)	1984
Crystal size	0.35 x 0.35 x 0.20 mm^3
Theta range for data collection	1.14 to 27.49 deg.
Index ranges	-18<=h<=26, -18<=k<=15, -27<=l<=17
Reflections collected	28354
Independent reflections	11598 [R(int) = 0.0240]
Completeness to theta = 27.49-	96.9 %
Absorption correction	Empirical
Max. and min. transmission	0.9449 and 0.9064
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	11598 / 0 / 541
Goodness-of-fit on F^2	1.112
Final R indices [I>2sigma(I)]	R1 = 0.0560, wR2 = 0.1573
R indices (all data)	R1 = 0.0642, wR2 = 0.1613
Largest diff. peak and hole	0.588 and -0.427 e.Å^-3

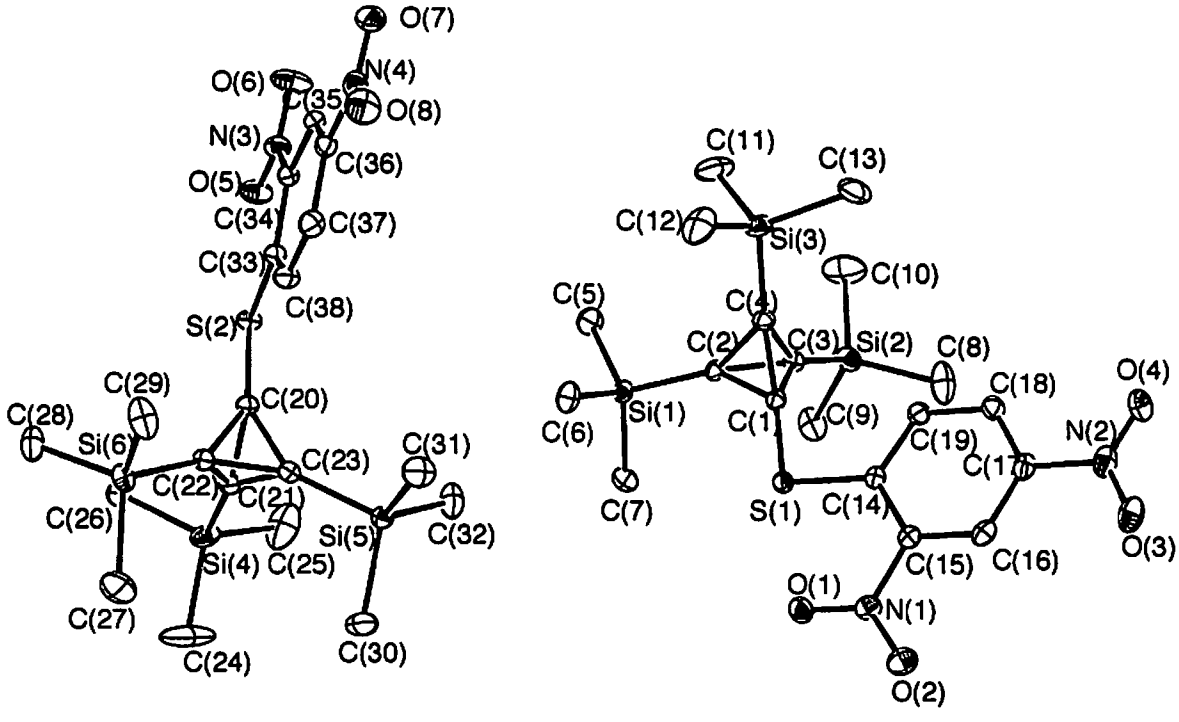


Figure S-3. ORTEP drawing of 4 (30% thermal ellipsoids). Hydrogen atoms are omitted for clarity

Table 9. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2x 10^3) for **4**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	777(2)	2855(2)	4014(2)	22(1)
C(2)	1275(2)	2439(2)	3740(2)	24(1)
C(3)	824(2)	1797(2)	3965(2)	25(1)
C(4)	1477(2)	2388(2)	4531(2)	26(1)
C(5)	2285(2)	1772(3)	3124(2)	45(1)
C(6)	2081(2)	3916(3)	3359(2)	41(1)
C(7)	805(2)	2784(3)	2175(2)	35(1)
C(8)	-402(3)	644(4)	4044(3)	66(2)
C(9)	-136(3)	551(3)	2730(2)	50(1)
C(10)	1002(3)	-335(3)	4163(3)	76(2)
C(11)	2905(3)	1330(4)	5257(2)	65(2)
C(12)	2818(3)	3431(4)	5648(3)	64(1)
C(13)	2056(3)	1864(4)	6099(2)	53(1)
C(14)	-212(2)	3802(2)	4313(2)	22(1)
C(15)	-748(2)	4470(2)	4282(2)	24(1)
C(16)	-1065(2)	4438(2)	4742(2)	28(1)
C(17)	-848(2)	3705(3)	5245(2)	29(1)
C(18)	-337(2)	3020(3)	5294(2)	30(1)
C(19)	-18(2)	3071(2)	4835(2)	27(1)
C(20)	4201(2)	7136(2)	3227(2)	24(1)
C(21)	3671(2)	7445(2)	2479(2)	25(1)
C(22)	4123(2)	8175(2)	3044(2)	25(1)
C(23)	3498(2)	7623(2)	3095(2)	25(1)
C(24)	2694(4)	8078(5)	961(3)	102(3)
C(25)	2787(3)	5943(5)	1383(3)	76(2)
C(26)	4107(2)	6960(3)	1337(2)	42(1)
C(27)	3826(3)	10217(3)	2550(3)	62(1)
C(28)	5281(2)	9223(3)	2835(2)	43(1)
C(29)	4973(3)	9724(3)	4105(2)	49(1)
C(30)	2035(2)	8601(4)	2576(2)	53(1)
C(31)	2945(2)	8120(3)	4178(2)	44(1)
C(32)	2242(2)	6496(3)	3076(2)	48(1)
C(33)	5198(2)	6307(2)	4469(2)	25(1)
C(34)	5691(2)	5623(2)	4960(2)	24(1)
C(35)	5989(2)	5723(2)	5702(2)	27(1)

C(36)	5815(2)	6534(3)	5969(2)	29(1)
C(37)	5347(2)	7240(3)	5514(2)	31(1)
C(38)	5044(2)	7118(2)	4776(2)	30(1)
N(1)	-994(2)	5254(2)	3758(2)	31(1)
N(2)	-1193(2)	3652(2)	5726(2)	37(1)
N(3)	5902(2)	4757(2)	4703(2)	32(1)
N(4)	6132(2)	6643(3)	6755(2)	37(1)
O(1)	-677(1)	5349(2)	3384(1)	36(1)
O(2)	-1498(2)	5779(2)	3715(2)	52(1)
O(3)	-1611(2)	4303(2)	5697(2)	45(1)
O(4)	-1038(2)	2947(2)	6126(2)	48(1)
O(5)	5645(2)	4644(2)	4051(1)	39(1)
O(6)	6316(2)	4179(2)	5149(2)	57(1)
O(7)	6512(2)	5986(2)	7136(1)	45(1)
O(8)	5994(2)	7389(2)	6982(2)	50(1)
S(1)	212(1)	3860(1)	3747(1)	24(1)
S(2)	4777(1)	6146(1)	3532(1)	26(1)
Si(1)	1621(1)	2720(1)	3092(1)	26(1)
Si(2)	322(1)	645(1)	3727(1)	33(1)
Si(3)	2320(1)	2259(1)	5401(1)	37(1)
Si(4)	3308(1)	7103(1)	1531(1)	37(1)
Si(5)	4559(1)	9357(1)	3138(1)	30(1)
Si(6)	2671(1)	7713(1)	3241(1)	28(1)

Table 10. Bond lengths [Å] and angles [deg] for **4**.

C(1)-C(3)	1.481(4)
C(1)-C(4)	1.476(4)
C(1)-C(2)	1.489(4)
C(1)-S(1)	1.727(3)
C(2)-C(3)	1.501(4)
C(2)-C(4)	1.511(4)
C(2)-Si(1)	1.836(3)
C(3)-C(4)	1.540(4)
C(3)-Si(2)	1.838(3)
C(4)-Si(3)	1.836(3)
C(5)-Si(1)	1.861(4)
C(5)-H(5A)	0.9800
C(5)-H(5B)	0.9800
C(5)-H(5C)	0.9800
C(6)-Si(1)	1.859(4)
C(6)-H(6A)	0.9800
C(6)-H(6B)	0.9800
C(6)-H(6C)	0.9800
C(7)-Si(1)	1.865(4)
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(7)-H(7C)	0.9800
C(8)-Si(2)	1.870(5)
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-Si(2)	1.853(4)
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-Si(2)	1.848(5)
C(10)-H(10A)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
C(11)-Si(3)	1.869(5)
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(12)-Si(3)	1.859(5)

C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-Si(3)	1.858(4)
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-C(15)	1.407(4)
C(14)-C(19)	1.409(4)
C(14)-S(1)	1.757(3)
C(15)-C(16)	1.383(4)
C(15)-N(1)	1.460(4)
C(16)-C(17)	1.381(5)
C(16)-H(16)	0.9500
C(17)-C(18)	1.374(5)
C(17)-N(2)	1.470(4)
C(18)-C(19)	1.386(4)
C(18)-H(18)	0.9500
C(19)-H(19)	0.9500
C(20)-C(22)	1.484(4)
C(20)-C(23)	1.482(4)
C(20)-C(21)	1.492(4)
C(20)-S(2)	1.723(3)
C(21)-C(22)	1.504(4)
C(21)-C(23)	1.505(4)
C(21)-Si(4)	1.830(3)
C(22)-C(23)	1.526(4)
C(22)-Si(5)	1.831(3)
C(23)-Si(6)	1.841(3)
C(24)-Si(4)	1.851(5)
C(24)-H(24A)	0.9800
C(24)-H(24B)	0.9800
C(24)-H(24C)	0.9800
C(25)-Si(4)	1.873(6)
C(25)-H(25A)	0.9800
C(25)-H(25B)	0.9800
C(25)-H(25C)	0.9800
C(26)-Si(4)	1.853(4)
C(26)-H(26A)	0.9800
C(26)-H(26B)	0.9800
C(26)-H(26C)	0.9800

C(27)-Si(5)	1.858(5)
C(27)-H(27A)	0.9800
C(27)-H(27B)	0.9800
C(27)-H(27C)	0.9800
C(28)-Si(5)	1.855(4)
C(28)-H(28A)	0.9800
C(28)-H(28B)	0.9800
C(28)-H(28C)	0.9800
C(29)-Si(5)	1.865(4)
C(29)-H(29A)	0.9800
C(29)-H(29B)	0.9800
C(29)-H(29C)	0.9800
C(30)-Si(6)	1.859(4)
C(30)-H(30A)	0.9800
C(30)-H(30B)	0.9800
C(30)-H(30C)	0.9800
C(31)-Si(6)	1.862(4)
C(31)-H(31A)	0.9800
C(31)-H(31B)	0.9800
C(31)-H(31C)	0.9800
C(32)-Si(6)	1.859(4)
C(32)-H(32A)	0.9800
C(32)-H(32B)	0.9800
C(32)-H(32C)	0.9800
C(33)-C(38)	1.402(5)
C(33)-C(34)	1.415(4)
C(33)-S(2)	1.752(3)
C(34)-C(35)	1.386(4)
C(34)-N(3)	1.461(4)
C(35)-C(36)	1.375(5)
C(35)-H(35)	0.9500
C(36)-C(37)	1.388(5)
C(36)-N(4)	1.470(4)
C(37)-C(38)	1.384(5)
C(37)-H(37)	0.9500
C(38)-H(38)	0.9500
N(1)-O(2)	1.225(4)
N(1)-O(1)	1.228(4)
N(2)-O(3)	1.222(4)
N(2)-O(4)	1.232(4)
N(3)-O(6)	1.223(4)

N(3)-O(5)	1.223(4)
N(4)-O(8)	1.226(4)
N(4)-O(7)	1.221(4)

C(3)-C(1)-C(4)	62.8(2)
C(3)-C(1)-C(2)	60.7(2)
C(4)-C(1)-C(2)	61.3(2)
C(3)-C(1)-S(1)	146.7(2)
C(4)-C(1)-S(1)	149.3(2)
C(2)-C(1)-S(1)	130.9(2)
C(1)-C(2)-C(3)	59.4(2)
C(1)-C(2)-C(4)	58.9(2)
C(3)-C(2)-C(4)	61.5(2)
C(1)-C(2)-Si(1)	139.7(2)
C(3)-C(2)-Si(1)	148.8(2)
C(4)-C(2)-Si(1)	144.8(2)
C(1)-C(3)-C(2)	59.9(2)
C(1)-C(3)-C(4)	58.5(2)
C(2)-C(3)-C(4)	59.6(2)
C(1)-C(3)-Si(2)	147.0(2)
C(2)-C(3)-Si(2)	140.8(2)
C(4)-C(3)-Si(2)	147.4(2)
C(1)-C(4)-C(2)	59.80(19)
C(1)-C(4)-C(3)	58.8(2)
C(2)-C(4)-C(3)	58.9(2)
C(1)-C(4)-Si(3)	154.7(2)
C(2)-C(4)-Si(3)	138.2(2)
C(3)-C(4)-Si(3)	141.3(2)
Si(1)-C(5)-H(5A)	109.5
Si(1)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
Si(1)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
Si(1)-C(6)-H(6A)	109.5
Si(1)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
Si(1)-C(6)-H(6C)	109.5
H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5
Si(1)-C(7)-H(7A)	109.5

Si(1)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
Si(1)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
Si(2)-C(8)-H(8A)	109.5
Si(2)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
Si(2)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
Si(2)-C(9)-H(9A)	109.5
Si(2)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
Si(2)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
Si(2)-C(10)-H(10A)	109.5
Si(2)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
Si(2)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
Si(3)-C(11)-H(11A)	109.5
Si(3)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
Si(3)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
Si(3)-C(12)-H(12A)	109.5
Si(3)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
Si(3)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
Si(3)-C(13)-H(13A)	109.5
Si(3)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
Si(3)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5

C(15)-C(14)-C(19)	116.3(3)
C(15)-C(14)-S(1)	123.2(2)
C(19)-C(14)-S(1)	120.5(2)
C(16)-C(15)-C(14)	123.0(3)
C(16)-C(15)-N(1)	116.2(3)
C(14)-C(15)-N(1)	120.9(3)
C(15)-C(16)-C(17)	117.9(3)
C(15)-C(16)-H(16)	121.1
C(17)-C(16)-H(16)	121.1
C(18)-C(17)-C(16)	122.0(3)
C(18)-C(17)-N(2)	119.7(3)
C(16)-C(17)-N(2)	118.3(3)
C(17)-C(18)-C(19)	119.4(3)
C(17)-C(18)-H(18)	120.3
C(19)-C(18)-H(18)	120.3
C(18)-C(19)-C(14)	121.5(3)
C(18)-C(19)-H(19)	119.3
C(14)-C(19)-H(19)	119.3
C(22)-C(20)-C(23)	61.9(2)
C(22)-C(20)-C(21)	60.7(2)
C(23)-C(20)-C(21)	60.8(2)
C(22)-C(20)-S(2)	147.7(3)
C(23)-C(20)-S(2)	149.3(2)
C(21)-C(20)-S(2)	130.7(2)
C(20)-C(21)-C(22)	59.4(2)
C(20)-C(21)-C(23)	59.3(2)
C(22)-C(21)-C(23)	60.9(2)
C(20)-C(21)-Si(4)	142.5(2)
C(22)-C(21)-Si(4)	145.0(2)
C(23)-C(21)-Si(4)	146.7(2)
C(20)-C(22)-C(21)	59.9(2)
C(20)-C(22)-C(23)	59.0(2)
C(21)-C(22)-C(23)	59.6(2)
C(20)-C(22)-Si(5)	149.0(2)
C(21)-C(22)-Si(5)	141.0(2)
C(23)-C(22)-Si(5)	144.8(2)
C(20)-C(23)-C(21)	59.9(2)
C(20)-C(23)-C(22)	59.1(2)
C(21)-C(23)-C(22)	59.5(2)
C(20)-C(23)-Si(6)	150.4(2)
C(21)-C(23)-Si(6)	138.1(2)

C(22)-C(23)-Si(6)	145.6(2)
Si(4)-C(24)-H(24A)	109.5
Si(4)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
Si(4)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
Si(4)-C(25)-H(25A)	109.5
Si(4)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
Si(4)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
Si(4)-C(26)-H(26A)	109.5
Si(4)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
Si(4)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
Si(5)-C(27)-H(27A)	109.5
Si(5)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	109.5
Si(5)-C(27)-H(27C)	109.5
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
Si(5)-C(28)-H(28A)	109.5
Si(5)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
Si(5)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
Si(5)-C(29)-H(29A)	109.5
Si(5)-C(29)-H(29B)	109.5
H(29A)-C(29)-H(29B)	109.5
Si(5)-C(29)-H(29C)	109.5
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
Si(6)-C(30)-H(30A)	109.5
Si(6)-C(30)-H(30B)	109.5
H(30A)-C(30)-H(30B)	109.5
Si(6)-C(30)-H(30C)	109.5

H(30A)-C(30)-H(30C)	109.5
H(30B)-C(30)-H(30C)	109.5
Si(6)-C(31)-H(31A)	109.5
Si(6)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
Si(6)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
Si(6)-C(32)-H(32A)	109.5
Si(6)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
Si(6)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(38)-C(33)-C(34)	116.1(3)
C(38)-C(33)-S(2)	121.2(2)
C(34)-C(33)-S(2)	122.6(2)
C(35)-C(34)-C(33)	122.6(3)
C(35)-C(34)-N(3)	116.5(3)
C(33)-C(34)-N(3)	121.0(3)
C(36)-C(35)-C(34)	118.5(3)
C(36)-C(35)-H(35)	120.8
C(34)-C(35)-H(35)	120.8
C(35)-C(36)-C(37)	121.5(3)
C(35)-C(36)-N(4)	118.5(3)
C(37)-C(36)-N(4)	119.9(3)
C(38)-C(37)-C(36)	119.1(3)
C(38)-C(37)-H(37)	120.4
C(36)-C(37)-H(37)	120.4
C(37)-C(38)-C(33)	122.1(3)
C(37)-C(38)-H(38)	118.9
C(33)-C(38)-H(38)	118.9
O(2)-N(1)-O(1)	123.6(3)
O(2)-N(1)-C(15)	118.8(3)
O(1)-N(1)-C(15)	117.6(3)
O(3)-N(2)-O(4)	125.0(3)
O(3)-N(2)-C(17)	118.0(3)
O(4)-N(2)-C(17)	117.0(3)
O(6)-N(3)-O(5)	123.6(3)
O(6)-N(3)-C(34)	118.3(3)
O(5)-N(3)-C(34)	118.1(3)

O(8)-N(4)-O(7)	124.6(3)
O(8)-N(4)-C(36)	117.4(3)
O(7)-N(4)-C(36)	118.0(3)
C(1)-S(1)-C(14)	101.72(15)
C(20)-S(2)-C(33)	101.94(15)
C(2)-Si(1)-C(5)	109.89(17)
C(2)-Si(1)-C(6)	106.14(16)
C(5)-Si(1)-C(6)	111.7(2)
C(2)-Si(1)-C(7)	108.06(16)
C(5)-Si(1)-C(7)	110.53(18)
C(6)-Si(1)-C(7)	110.33(18)
C(3)-Si(2)-C(9)	106.89(17)
C(3)-Si(2)-C(10)	108.5(2)
C(9)-Si(2)-C(10)	111.9(3)
C(3)-Si(2)-C(8)	109.32(19)
C(9)-Si(2)-C(8)	109.6(2)
C(10)-Si(2)-C(8)	110.6(3)
C(4)-Si(3)-C(12)	108.9(2)
C(4)-Si(3)-C(13)	109.51(19)
C(12)-Si(3)-C(13)	111.0(2)
C(4)-Si(3)-C(11)	105.92(18)
C(12)-Si(3)-C(11)	110.3(3)
C(13)-Si(3)-C(11)	111.0(2)
C(21)-Si(4)-C(26)	108.31(17)
C(21)-Si(4)-C(24)	108.6(2)
C(26)-Si(4)-C(24)	109.3(2)
C(21)-Si(4)-C(25)	108.3(2)
C(26)-Si(4)-C(25)	110.8(2)
C(24)-Si(4)-C(25)	111.4(3)
C(22)-Si(5)-C(28)	106.88(17)
C(22)-Si(5)-C(27)	108.03(19)
C(28)-Si(5)-C(27)	110.6(2)
C(22)-Si(5)-C(29)	108.26(16)
C(28)-Si(5)-C(29)	111.7(2)
C(27)-Si(5)-C(29)	111.2(2)
C(23)-Si(6)-C(32)	106.82(17)
C(23)-Si(6)-C(31)	110.26(17)
C(32)-Si(6)-C(31)	110.9(2)
C(23)-Si(6)-C(30)	106.79(17)
C(32)-Si(6)-C(30)	111.3(2)
C(31)-Si(6)-C(30)	110.5(2)

Symmetry transformations used to generate equivalent atoms:

Table 11. Anisotropic displacement parameters (Å^2x 10^3) for **4**. The anisotropic displacement factor exponent takes the form: -2pi^2[h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	20(1)	25(2)	22(1)	-2(1)	10(1)	-1(1)
C(2)	21(1)	28(2)	23(1)	-4(1)	11(1)	0(1)
C(3)	25(2)	25(2)	24(2)	0(1)	10(1)	1(1)
C(4)	23(2)	29(2)	22(1)	-1(1)	9(1)	0(1)
C(5)	48(2)	51(2)	47(2)	2(2)	32(2)	15(2)
C(6)	38(2)	40(2)	51(2)	-7(2)	25(2)	-12(2)
C(7)	42(2)	34(2)	28(2)	2(1)	16(2)	1(2)
C(8)	79(4)	70(3)	71(3)	-18(3)	53(3)	-37(3)
C(9)	60(3)	48(2)	38(2)	-14(2)	20(2)	-22(2)
C(10)	73(4)	29(2)	91(4)	7(2)	9(3)	4(2)
C(11)	46(3)	99(4)	42(2)	2(2)	12(2)	38(3)
C(12)	40(2)	90(4)	49(3)	-26(3)	12(2)	-21(2)
C(13)	63(3)	67(3)	29(2)	15(2)	22(2)	25(2)
C(14)	21(1)	24(1)	21(1)	-4(1)	10(1)	-4(1)
C(15)	22(2)	24(2)	25(2)	-5(1)	10(1)	-2(1)
C(16)	21(2)	30(2)	30(2)	-11(1)	10(1)	-5(1)
C(17)	24(2)	39(2)	23(2)	-10(1)	11(1)	-8(1)
C(18)	30(2)	37(2)	24(2)	0(1)	12(1)	-2(1)
C(19)	26(2)	31(2)	24(2)	0(1)	12(1)	2(1)
C(20)	22(2)	26(2)	21(1)	2(1)	8(1)	1(1)
C(21)	22(2)	28(2)	23(2)	3(1)	8(1)	3(1)
C(22)	24(2)	28(2)	24(2)	4(1)	11(1)	2(1)
C(23)	25(2)	24(2)	24(2)	4(1)	10(1)	1(1)
C(24)	99(5)	165(7)	33(2)	30(3)	24(3)	101(5)
C(25)	61(3)	105(5)	65(3)	-49(3)	31(3)	-38(3)
C(26)	45(2)	50(2)	37(2)	5(2)	25(2)	13(2)
C(27)	66(3)	34(2)	83(4)	20(2)	31(3)	8(2)
C(28)	43(2)	56(2)	37(2)	-7(2)	25(2)	-18(2)
C(29)	75(3)	42(2)	43(2)	-16(2)	38(2)	-22(2)
C(30)	41(2)	70(3)	49(2)	24(2)	21(2)	26(2)
C(31)	51(2)	51(2)	35(2)	-3(2)	23(2)	10(2)
C(32)	48(2)	55(3)	50(2)	-3(2)	30(2)	-16(2)
C(33)	23(2)	28(2)	22(1)	3(1)	8(1)	0(1)
C(34)	22(2)	24(2)	25(2)	2(1)	10(1)	-1(1)
C(35)	20(1)	34(2)	25(2)	8(1)	7(1)	-2(1)

C(36)	24(2)	42(2)	20(2)	0(1)	10(1)	-7(1)
C(37)	32(2)	35(2)	29(2)	-3(1)	16(1)	1(1)
C(38)	30(2)	30(2)	28(2)	3(1)	12(1)	4(1)
N(1)	32(2)	25(1)	35(2)	-3(1)	15(1)	1(1)
N(2)	32(2)	51(2)	30(2)	-13(1)	17(1)	-11(1)
N(3)	30(2)	26(1)	33(2)	4(1)	9(1)	4(1)
N(4)	31(2)	56(2)	25(1)	-1(1)	14(1)	-8(1)
O(1)	41(1)	29(1)	43(1)	7(1)	24(1)	4(1)
O(2)	52(2)	47(2)	69(2)	15(2)	38(2)	26(1)
O(3)	46(2)	53(2)	51(2)	-13(1)	34(1)	-2(1)
O(4)	54(2)	63(2)	40(2)	7(1)	32(1)	2(2)
O(5)	46(2)	36(1)	30(1)	0(1)	13(1)	14(1)
O(6)	70(2)	42(2)	41(2)	11(1)	11(2)	29(2)
O(7)	36(1)	71(2)	24(1)	7(1)	10(1)	4(1)
O(8)	56(2)	58(2)	33(1)	-13(1)	20(1)	-6(2)
S(1)	25(1)	23(1)	25(1)	1(1)	13(1)	0(1)
S(2)	27(1)	25(1)	21(1)	1(1)	8(1)	5(1)
Si(1)	25(1)	29(1)	26(1)	-2(1)	15(1)	0(1)
Si(2)	39(1)	25(1)	34(1)	0(1)	15(1)	-5(1)
Si(3)	26(1)	55(1)	22(1)	-2(1)	5(1)	10(1)
Si(4)	28(1)	57(1)	21(1)	-1(1)	7(1)	11(1)
Si(5)	38(1)	26(1)	32(1)	1(1)	20(1)	-4(1)
Si(6)	26(1)	33(1)	27(1)	7(1)	14(1)	5(1)

Table 12. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2x 10^3 for **4**.

	x	y	z	U(eq)
H(5A)	2727	1779	3598	67
H(5B)	2435	1896	2749	67
H(5C)	2043	1141	3042	67
H(6A)	1718	4394	3343	62
H(6B)	2272	4105	3024	62
H(6C)	2495	3879	3850	62
H(7A)	601	2137	2020	53
H(7B)	964	3048	1834	53
H(7C)	422	3201	2189	53
H(8A)	-161	737	4568	99
H(8B)	-667	28	3923	99
H(8C)	-757	1168	3808	99
H(9A)	-413	1144	2518	75
H(9B)	-480	4	2576	75
H(9C)	243	456	2570	75
H(10A)	1360	-347	3970	114
H(10B)	739	-952	4064	114
H(10C)	1268	-226	4685	114
H(11A)	2600	762	5027	98
H(11B)	3320	1148	5723	98
H(11C)	3102	1594	4945	98
H(12A)	2855	3692	5231	95
H(12B)	3320	3337	6048	95
H(12C)	2542	3883	5794	95
H(13A)	1697	2320	6119	80
H(13B)	2502	1841	6570	80
H(13C)	1830	1223	5977	80
H(16)	-1420	4905	4713	33
H(18)	-204	2517	5639	36
H(19)	340	2601	4874	32
H(24A)	2257	8131	1043	153
H(24B)	2532	7931	452	153
H(24C)	2968	8688	1087	153
H(25A)	3090	5485	1762	114
H(25B)	2680	5679	911	114

H(25C)	2317	6060	1399	114
H(26A)	4314	7594	1327	62
H(26B)	3938	6646	868	62
H(26C)	4492	6565	1714	62
H(27A)	3579	9971	2055	93
H(27B)	4054	10842	2559	93
H(27C)	3457	10293	2727	93
H(28A)	5608	8682	3089	64
H(28B)	5577	9815	2942	64
H(28C)	5040	9102	2314	64
H(29A)	4585	9723	4260	73
H(29B)	5184	10372	4161	73
H(29C)	5368	9270	4401	73
H(30A)	2280	9230	2668	80
H(30B)	1576	8651	2621	80
H(30C)	1913	8389	2087	80
H(31A)	3296	7658	4522	67
H(31B)	2501	8163	4249	67
H(31C)	3182	8754	4257	67
H(32A)	2116	6290	2584	71
H(32B)	1789	6517	3133	71
H(32C)	2597	6040	3423	71
H(35)	6307	5243	6018	33
H(37)	5236	7798	5708	37
H(38)	4722	7600	4466	36

Table 13. Crystal data and structure refinement for **5**.

Identification code	tdsoph_0m	
Empirical formula	C19 H32 O2 S Si3	
Formula weight	408.78	
Temperature	120 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 17.366(3) Å	α = 90°
	b = 9.1430(16) Å	β = 119.157(2)°
	c = 17.699(3) Å	γ = 90°
Volume	2454.1(7) Å^3	
Z	4	
Density (calculated)	1.106 Mg/m^3	
Absorption coefficient	0.288 mm^-1	
F(000)	880	
Crystal size	0.40 x 0.38 x 0.08 mm^3	
Theta range for data collection	1.34 to 27.51 deg.	
Index ranges	-22<=h<=22, -11<=k<=11, -22<=l<=22	
Reflections collected	26544	
Independent reflections	5570 [R(int) = 0.0375]	
Completeness to theta = 27.51-	98.9 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9773 and 0.8936	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5570 / 0 / 235	
Goodness-of-fit on F^2	1.008	
Final R indices [I>2sigma(I)]	R1 = 0.0321, wR2 = 0.0800	
R indices (all data)	R1 = 0.0431, wR2 = 0.0865	
Largest diff. peak and hole	0.311 and -0.278 e.Å^-3	

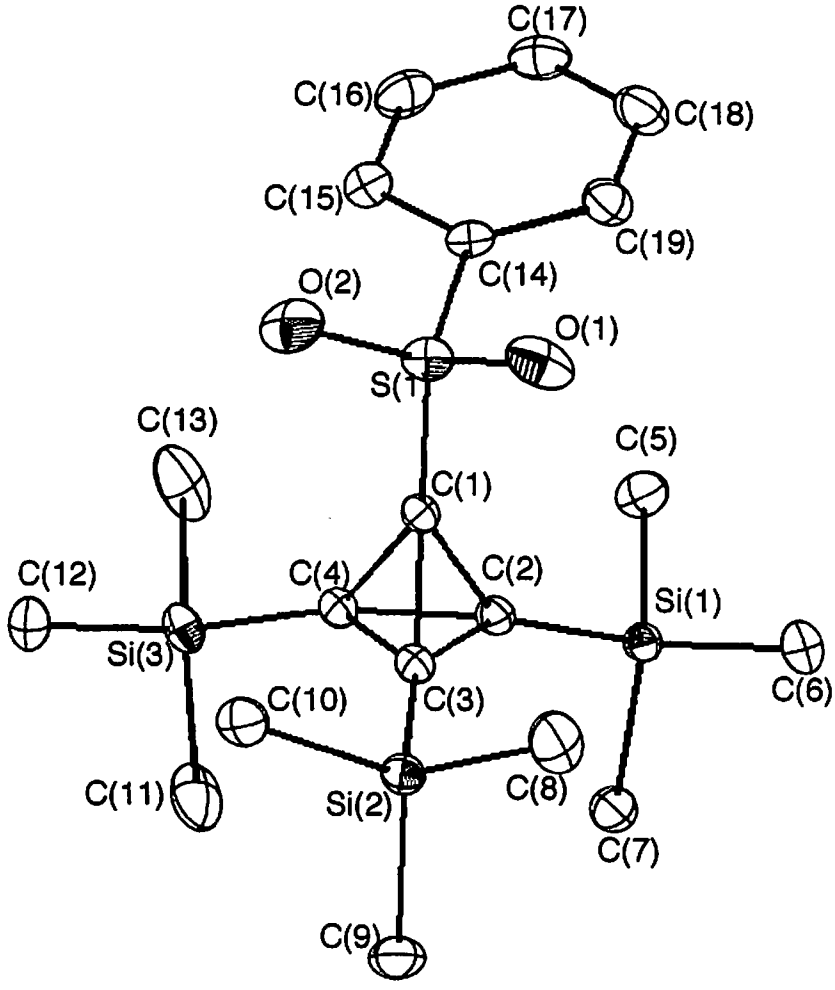


Figure S-4. ORTEP drawing of **5** (30% thermal ellipsoids). Hydrogen atoms are omitted for clarity.

Table 14. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2x 10^3)for **5**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	2684(1)	2069(2)	2407(1)	23(1)
C(2)	2418(1)	1744(2)	3066(1)	25(1)
C(3)	1736(1)	1782(2)	2125(1)	26(1)
C(4)	2407(1)	562(2)	2451(1)	25(1)
C(5)	3892(1)	1648(2)	4880(1)	38(1)
C(6)	2389(1)	3904(2)	4319(1)	39(1)
C(7)	2025(1)	642(2)	4426(1)	38(1)
C(8)	526(1)	4356(2)	1366(1)	54(1)
C(9)	-197(1)	1325(2)	1431(1)	49(1)
C(10)	585(1)	1921(2)	223(1)	39(1)
C(11)	1917(2)	-2437(2)	2739(1)	64(1)
C(12)	2198(1)	-1810(2)	1201(1)	37(1)
C(13)	3778(1)	-1796(2)	3050(1)	63(1)
C(14)	4370(1)	3010(2)	2990(1)	28(1)
C(15)	4891(1)	1851(2)	3015(1)	38(1)
C(16)	5737(1)	1724(2)	3716(1)	47(1)
C(17)	6049(1)	2756(2)	4366(1)	45(1)
C(18)	5532(1)	3911(2)	4333(1)	46(1)
C(19)	4683(1)	4048(2)	3643(1)	38(1)
O(1)	3002(1)	4658(1)	2098(1)	50(1)
O(2)	3262(1)	2574(2)	1359(1)	52(1)
S(1)	3285(1)	3173(1)	2120(1)	31(1)
Si(1)	2690(1)	1997(1)	4204(1)	25(1)
Si(2)	638(1)	2351(1)	1275(1)	29(1)
Si(3)	2581(1)	-1402(1)	2357(1)	31(1)

Table 15. Bond lengths [Å] and angles [deg] for **5**.

C(1)-C(4)	1.4739(19)
C(1)-C(2)	1.4792(19)
C(1)-C(3)	1.4932(19)
C(1)-S(1)	1.6973(14)
C(2)-C(3)	1.5022(18)
C(2)-C(4)	1.5276(19)
C(2)-Si(1)	1.8462(15)
C(3)-C(4)	1.5111(19)
C(3)-Si(2)	1.8361(14)
C(4)-Si(3)	1.8409(15)
C(5)-Si(1)	1.8589(16)
C(5)-H(5A)	0.9800
C(5)-H(5B)	0.9800
C(5)-H(5C)	0.9800
C(6)-Si(1)	1.8593(16)
C(6)-H(6A)	0.9800
C(6)-H(6B)	0.9800
C(6)-H(6C)	0.9800
C(7)-Si(1)	1.8606(16)
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(7)-H(7C)	0.9800
C(8)-Si(2)	1.8586(19)
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-Si(2)	1.8589(18)
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-Si(2)	1.8602(17)
C(10)-H(10A)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
C(11)-Si(3)	1.854(2)
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(12)-Si(3)	1.8562(17)

C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-Si(3)	1.8632(19)
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-C(15)	1.380(2)
C(14)-C(19)	1.386(2)
C(14)-S(1)	1.7650(15)
C(15)-C(16)	1.391(2)
C(15)-H(15)	0.9500
C(16)-C(17)	1.379(3)
C(16)-H(16)	0.9500
C(17)-C(18)	1.369(3)
C(17)-H(17)	0.9500
C(18)-C(19)	1.388(2)
C(18)-H(18)	0.9500
C(19)-H(19)	0.9500
O(1)-S(1)	1.4377(13)
O(2)-S(1)	1.4368(13)

C(4)-C(1)-C(2)	62.30(9)
C(4)-C(1)-C(3)	61.23(9)
C(2)-C(1)-C(3)	60.71(9)
C(4)-C(1)-S(1)	146.73(11)
C(2)-C(1)-S(1)	145.95(11)
C(3)-C(1)-S(1)	137.98(10)
C(1)-C(2)-C(3)	60.11(9)
C(1)-C(2)-C(4)	58.68(9)
C(3)-C(2)-C(4)	59.83(9)
C(1)-C(2)-Si(1)	145.42(10)
C(3)-C(2)-Si(1)	148.37(11)
C(4)-C(2)-Si(1)	140.70(10)
C(1)-C(3)-C(2)	59.18(9)
C(1)-C(3)-C(4)	58.75(9)
C(2)-C(3)-C(4)	60.92(9)
C(1)-C(3)-Si(2)	139.44(11)
C(2)-C(3)-Si(2)	149.11(11)
C(4)-C(3)-Si(2)	145.29(10)
C(1)-C(4)-C(3)	60.02(9)

C(1)-C(4)-C(2)	59.02(9)
C(3)-C(4)-C(2)	59.25(9)
C(1)-C(4)-Si(3)	146.60(11)
C(3)-C(4)-Si(3)	144.63(10)
C(2)-C(4)-Si(3)	143.95(10)
Si(1)-C(5)-H(5A)	109.5
Si(1)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
Si(1)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
Si(1)-C(6)-H(6A)	109.5
Si(1)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
Si(1)-C(6)-H(6C)	109.5
H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5
Si(1)-C(7)-H(7A)	109.5
Si(1)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
Si(1)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
Si(2)-C(8)-H(8A)	109.5
Si(2)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
Si(2)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
Si(2)-C(9)-H(9A)	109.5
Si(2)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
Si(2)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
Si(2)-C(10)-H(10A)	109.5
Si(2)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
Si(2)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5

Si(3)-C(11)-H(11A)	109.5
Si(3)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
Si(3)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
Si(3)-C(12)-H(12A)	109.5
Si(3)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
Si(3)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
Si(3)-C(13)-H(13A)	109.5
Si(3)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
Si(3)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(15)-C(14)-C(19)	121.01(14)
C(15)-C(14)-S(1)	119.83(12)
C(19)-C(14)-S(1)	119.15(12)
C(14)-C(15)-C(16)	118.91(16)
C(14)-C(15)-H(15)	120.5
C(16)-C(15)-H(15)	120.5
C(17)-C(16)-C(15)	120.14(16)
C(17)-C(16)-H(16)	119.9
C(15)-C(16)-H(16)	119.9
C(18)-C(17)-C(16)	120.65(16)
C(18)-C(17)-H(17)	119.7
C(16)-C(17)-H(17)	119.7
C(17)-C(18)-C(19)	120.05(17)
C(17)-C(18)-H(18)	120.0
C(19)-C(18)-H(18)	120.0
C(14)-C(19)-C(18)	119.23(16)
C(14)-C(19)-H(19)	120.4
C(18)-C(19)-H(19)	120.4
O(1)-S(1)-O(2)	118.73(8)
O(1)-S(1)-C(1)	108.62(7)
O(2)-S(1)-C(1)	108.22(7)
O(1)-S(1)-C(14)	108.01(7)
O(2)-S(1)-C(14)	108.37(8)

C(1)-S(1)-C(14)	103.91(7)
C(2)-Si(1)-C(5)	106.91(7)
C(2)-Si(1)-C(7)	106.93(7)
C(5)-Si(1)-C(7)	111.57(8)
C(2)-Si(1)-C(6)	107.47(7)
C(5)-Si(1)-C(6)	112.23(8)
C(7)-Si(1)-C(6)	111.40(8)
C(3)-Si(2)-C(10)	106.61(7)
C(3)-Si(2)-C(9)	107.99(8)
C(10)-Si(2)-C(9)	112.37(8)
C(3)-Si(2)-C(8)	108.62(7)
C(10)-Si(2)-C(8)	109.87(9)
C(9)-Si(2)-C(8)	111.19(10)
C(4)-Si(3)-C(11)	107.89(9)
C(4)-Si(3)-C(12)	108.00(7)
C(11)-Si(3)-C(12)	110.28(9)
C(4)-Si(3)-C(13)	107.97(7)
C(11)-Si(3)-C(13)	110.72(12)
C(12)-Si(3)-C(13)	111.83(9)

Symmetry transformations used to generate equivalent atoms:

Table 16. Anisotropic displacement parameters (Å^2x 10^3) for **5**. The anisotropic displacement factor exponent takes the form:
-2pi^2[h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	22(1)	24(1)	21(1)	0(1)	9(1)	0(1)
C(2)	23(1)	28(1)	22(1)	-2(1)	10(1)	-2(1)
C(3)	24(1)	26(1)	24(1)	-2(1)	10(1)	-2(1)
C(4)	26(1)	24(1)	24(1)	0(1)	11(1)	-1(1)
C(5)	29(1)	56(1)	27(1)	4(1)	12(1)	3(1)
C(6)	42(1)	37(1)	34(1)	-7(1)	15(1)	1(1)
C(7)	43(1)	44(1)	34(1)	-6(1)	24(1)	-11(1)
C(8)	41(1)	43(1)	62(1)	-6(1)	12(1)	13(1)
C(9)	32(1)	73(1)	43(1)	-10(1)	20(1)	-12(1)
C(10)	35(1)	48(1)	27(1)	0(1)	10(1)	-5(1)
C(11)	110(2)	37(1)	66(1)	-12(1)	60(1)	-27(1)
C(12)	40(1)	35(1)	34(1)	-4(1)	16(1)	5(1)
C(13)	52(1)	36(1)	60(1)	-5(1)	-4(1)	14(1)
C(14)	25(1)	30(1)	30(1)	5(1)	15(1)	-3(1)
C(15)	36(1)	39(1)	43(1)	-3(1)	23(1)	0(1)
C(16)	33(1)	51(1)	59(1)	7(1)	25(1)	11(1)
C(17)	26(1)	60(1)	43(1)	8(1)	13(1)	-2(1)
C(18)	36(1)	51(1)	44(1)	-8(1)	13(1)	-11(1)
C(19)	34(1)	32(1)	44(1)	-2(1)	17(1)	-2(1)
O(1)	38(1)	30(1)	67(1)	20(1)	12(1)	2(1)
O(2)	49(1)	85(1)	26(1)	6(1)	21(1)	-6(1)
S(1)	29(1)	34(1)	28(1)	10(1)	12(1)	-2(1)
Si(1)	23(1)	31(1)	22(1)	-2(1)	10(1)	-2(1)
Si(2)	21(1)	34(1)	28(1)	-3(1)	8(1)	0(1)
Si(3)	37(1)	22(1)	28(1)	-1(1)	11(1)	-2(1)

Table 17. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2x 10^3) for **5**.

	x	y	z	U(eq)
H(5A)	4021	633	4801	57
H(5B)	4069	1815	5490	57
H(5C)	4220	2311	4705	57
H(6A)	2683	4587	4116	59
H(6B)	2576	4102	4928	59
H(6C)	1749	4027	3973	59
H(7A)	1397	813	4030	58
H(7B)	2146	749	5024	58
H(7C)	2183	-349	4338	58
H(8A)	565	4577	1926	81
H(8B)	-47	4682	898	81
H(8C)	999	4865	1323	81
H(9A)	-106	272	1406	73
H(9B)	-789	1590	974	73
H(9C)	-137	1571	1997	73
H(10A)	1071	2413	192	58
H(10B)	22	2262	-252	58
H(10C)	635	862	174	58
H(11A)	2129	-2220	3351	95
H(11B)	1975	-3488	2668	95
H(11C)	1296	-2151	2399	95
H(12A)	1567	-1592	858	56
H(12B)	2298	-2846	1137	56
H(12C)	2526	-1207	997	56
H(13A)	4120	-1183	2866	94
H(13B)	3890	-2830	2992	94
H(13C)	3953	-1584	3655	94
H(15)	4675	1151	2560	45
H(16)	6101	924	3747	56
H(17)	6629	2665	4841	54
H(18)	5755	4619	4783	56
H(19)	4320	4844	3619	46