Submitted to *Org. Lett.*
Version 1 of October 13, 2003

Supporting Information

Molecular Tectonics. Dendritic Construction of Porous Hydrogen-Bonded Networks

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Contents

Selected experimental procedures

Dipentaerythrityl hexatosylate (8). A mixture of dipentaerythritol (4.00 g, 15.7 mmol) and tosyl chloride (21.0 g, 110 mmol) in dry pyridine (50 mL) was stirred at 25 °C for 48 h. Aqueous HCl (6 N, 300 mL) was then added, and the mixture was extracted twice with ether. The organic layers were combined, washed with brine, and dried over Na_2SO_4. Removal of volatiles by evaporation under reduced pressure left a residue that was purified by crystallization from benzene/methanol to give dipentaerythrityl hexatosylate (**8**; 15.7 g, 13.3 mmol, 85%) as a colorless solid: mp 54 °C; IR (KBr) 3091, 3036, 2958, 2924, 1597, 1364, 1192, 1178, 1096, 981, 968, 832, 812, 786, 666, 553 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, ^3J = 8.2 Hz, 12H), 7.37 (d, ^3J = 8.2 Hz, 12H), 3.82 (s, 12H), 3.18 (s, 4H), 2.47 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.7, 132.0, 130.4, 128.2, 68.1, 66.8, 44.0, 21.9; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 1179 (M+1). Anal. Calcd for $C_{52}H_{58}O_{19}S_6$: C, 52.96; H, 4.96; S, 16.31. Found: C, 52.82; H, 4.88; S, 16.15.

1,1'-Oxybis[3-(4-bromophenoxy)-2,2-bis[(4-bromophenoxy)methyl]]propane (9) (General Procedure A). A mixture of dipentaerythrityl hexatosylate (**8**; 2.00 g; 1.70 mmol), 4-bromophenol (2.05 g, 11.8 mmol), and K_2CO_3 (0.820 g, 5.93 mmol) in DMF (15 mL) was heated at reflux for 16 h. The mixture was then cooled, water (100 mL) was added, and the mixture was extracted twice with ether. The organic layers were combined, washed twice with water and then with brine, and dried over Na_2SO_4. Removal of volatiles by evaporation under reduced pressure left a residue that was purified by flash chromatography (silica, CH_2Cl_2 (30%)/hexane (70%), R_f 0.57) to give 1,1'-oxybis[3-(4-bromophenoxy)-2,2-bis[(4-bromophenoxy)methyl]]propane (**9**; 1.59 g, 1.34 mmol, 79%) as a colorless solid: mp 127 °C; IR (KBr) 2933, 2886, 1590, 1488, 1467, 1234, 1172, 1045, 814 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, ^3J = 8.7 Hz, 12H), 6.65 (d, ^3J = 8.7 Hz, 12H), 4.04

(s, 12H), 3.76 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 132.5, 116.4, 113.6, 69.9, 67.0, 45.1; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 1178. Anal. Calcd for $C_{46}H_{40}Br_6O_7$: C, 46.65; H, 3.40. Found: C, 46.74; H, 3.60.

1,1'-Oxybis[3-(3-bromophenoxy)-2,2-bis[(3-bromophenoxy)methyl]]propane (10). By General Procedure A, dipentaerythrityl hexatosylate (**8**; 2.00 g; 1.70 mmol) and 3-bromophenol (2.05 g, 11.8 mmol) were converted into crude 1,1'-oxybis[3-(3-bromophenoxy)-2,2-bis[(3-bromophenoxy)methyl]]propane (**10**), which was purified by flash chromatography (R_f 0.61) to give the product as a colorless solid (1.45 g, 1.22 mmol, 72%): mp 116 °C; IR (KBr) 3069, 2933, 2877, 1592, 1575, 1480, 1459, 1246, 1225, 1034, 866, 763, 675 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11-7.05 (m, 12H), 7.00 (m, 6H), 6.72 (dt, ^3J = 7.0 Hz, ^4J = 2.4 Hz, 6H), 4.08 (s, 12H), 3.78 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 130.8, 124.5, 123.1, 118.2, 113.5, 70.0, 66.9, 45.2; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 1178. Anal. Calcd for $C_{46}H_{40}Br_6O_7$: C, 46.65; H, 3.40. Found: C, 46.63; H, 3.39.

1,1'-Oxybis[3-(2-bromophenoxy)-2,2-bis[(2-bromophenoxy)methyl]]propane (11). By General Procedure A, dipentaerythrityl hexatosylate (**8**; 2.00 g; 1.70 mmol) and 2-bromophenol (2.05 g, 11.8 mmol) were converted into crude 1,1'-oxybis[3-(2-bromophenoxy)-2,2-bis[(2-bromophenoxy)methyl]]propane (**11**), which was purified by flash chromatography (R_f 0.59) to give the product as a colorless solid (1.37 g, 1.16 mmol, 68%): mp 57 °C; IR (KBr) 3062, 2934, 2883, 1585, 1481, 1461, 1442, 1276, 1245, 1124, 1053, 1030, 743 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (dd, ^3J = 7.8 Hz, ^4J = 1.6 Hz, 6H), 7.17 (td, ^3J = 7.8 Hz, ^4J = 1.6 Hz, 6H), 6.82-6.77 (m, 12H), 4.31 (s, 12H), 4.07 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.1, 133.3, 128.7, 122.1, 113.3, 112.5, 70.3, 68.0, 45.9; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 1178. Anal. Calcd for $C_{46}H_{40}Br_6O_7$: C, 46.65; H, 3.40. Found: C, 46.60; H, 3.34.

1,1'-Oxybis[3-(4-nitrophenoxy)-2,2-bis[(4-nitrophenoxy)methyl]]propane (12) General Procedure B. A mixture of dipentaerythrityl hexatosylate (**8**; 2.00 g; 1.70 mmol), 4-nitrophenol (1.65 g . 11.9 mmol), and K_2CO_3 (0.820 g, 5.93 mmol) in DMF (15 mL) was heated at reflux for 16 h. The mixture was then cooled to 25 °C, water (200 mL) and methanol (50 mL) were added, and the resulting precipitate was separated by filtration and washed with water. The resulting solid was crystallized from DMF/ethanol to afford 1,1'-oxybis[3-(4-nitrophenoxy)-2,2-bis[(4-nitrophenoxy)methyl]]propane (**12**; 1.41 g, 1.44 mmol, 85%) as a beige microcrystalline solid: mp 207 °C; IR (KBr) 3114, 3084, 2934, 2886, 1592, 1509, 1344, 1255, 1111, 1020, 843, 751, 689 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 8.04 (d, ^3J = 8.8 Hz, 12H), 7.03 (d, ^3J = 8.8 Hz, 12H), 4.26 (s, 12H), 3.74 (s, 4H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 163.5, 140.9, 125.6, 114.9, 68.3, 66.8, 44.5; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 981 (M+1); HRMS (FAB, 3-nitrobenzyl alcohol) calcd for $C_{46}H_{41}N_6O_{19}$ *m/e* 981.24268, found 981.24630 (M+1). Anal. Calcd for $C_{46}H_{40}N_6O_{19}$: C, 56.33; H, 4.11; N, 8.57. Found: C, 56.09; H, 4.05; N, 8.35.

1,1'-Oxybis[3-(4-cyanophenoxy)-2,2-bis[(4-cyanophenoxy)methyl]]propane (13). By General Procedure B, dipentaerythrityl hexatosylate (**8**; 4.00 g; 3.39 mmol) and 4-cyanophenol (2.83 g, 23.8 mmol) were converted into crude 1,1'-oxybis[3-(4-cyanophenoxy)-2,2-bis[(4-cyanophenoxy)methyl]]propane (**13**), which was purified by flash chromatography (silica, CH_2Cl_2, R_f 0.36) to give the product as a colorless solid (2.51 g, 2.92 mmol, 86%): mp 212 °C; IR (KBr) 3101, 3076, 2940, 2886, 2223, 1605, 1508, 1304, 1254, 1171, 1021, 827, 544 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, ^3J = 8.7 Hz, 12H), 6.85 (d, ^3J = 8.7 Hz, 12H), 4.18 (s, 12H), 3.85 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.6, 134.1, 118.9, 115.2, 104.4, 69.6, 66.6, 44.9; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 861 (M+1). Anal. Calcd for $C_{52}H_{40}N_6O_7$: C, 72.55; H, 4.68; N, 9.76. Found: C, 72.22; H, 4.64; N, 9.67.

1,1'-Oxybis[3-(3-cyanophenoxy)-2,2-bis[(3-cyanophenoxy)methyl]]propane (14). By General Procedure B, dipentaerythrityl hexatosylate (**8**; 4.00 g; 3.39 mmol) and 3-cyanophenol (2.83 g, 23.8 mmol) were converted into crude 1,1'-oxybis[3-(3-cyanophenoxy)-2,2-bis[(3-cyanophenoxy)methyl]]propane (**14**), which was purified by flash chromatography (silica, CH_2Cl_2 , R_f 0.52) to give the product as a colorless solid (2.21 g, 2.57 mmol, 76%): mp 79 °C; IR (KBr) 3075, 2939, 2883, 2230, 1596, 1578, 1482, 1431, 1289, 1261, 1144, 1028, 785, 680 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.37-7.30 (m, 6H), 7.23 (d, ^3J = 7.6 Hz, 6H), 7.07-7.02 (m, 12H), 4.13 (s, 12H), 3.84 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 130.7, 125.4, 119.6, 118.5, 117.4, 113.5, 69.5, 66.6, 45.1; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 861 (M+1). Anal. Calcd for $C_{52}H_{40}N_6O_7$: C, 72.55; H, 4.68; N, 9.76. Found: C, 72.43; H, 4.76; N, 9.73.

1,1'-Oxybis[3-(4-formylphenoxy)-2,2-bis[(4-formylphenoxy)methyl]]propane (15). By General Procedure B, dipentaerythrityl hexatosylate (**8**; 4.00 g; 3.39 mmol) and 4-hydroxybenzaldehyde (2.90 g, 23.7 mmol) were converted under N_2 into crude 1,1'-oxybis[3-(4-formylphenoxy)-2,2-bis[(4-formylphenoxy)methyl]]propane (**15**), which was purified by flash chromatography (silica, $CH_3COOC_2H_5$ (10%)/CH_2Cl_2 (90%)) to give the product as a colorless solid (2.23 g, 2.54 mmol, 75%): mp 69 °C; IR (KBr) 3073, 2939, 2881, 2826, 2737, 1690, 1600, 1577, 1508, 1249, 1158, 1026, 830 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 6H), 7.75 (d, ^3J = 8.6 Hz, 12H), 6.90 (d, ^3J = 8.6 Hz, 12H), 4.23 (s, 12H), 3.89 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.6, 163.3, 132.0, 130.4, 114.7, 69.6, 66.7, 45.0; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 879 (M+1). Anal. Calcd for $C_{52}H_{46}O_{13} \bullet 0.5\ H_2O$: C, 70.28; H, 5.34. Found: C, 70.00; H, 5.39.

1,1'-Oxybis[3-(4-aminophenoxy)-2,2-bis[(4-aminophenoxy)methyl]]propane (16). A mixture of 1,1'-oxybis[3-(4-nitrophenoxy)-2,2-bis[(4-nitrophenoxy)methyl]]propane (**12**; 13.3 g; 13.6 mmol)

and 10% Pd/C (1.35 g) in THF (300 mL) was stirred at 25 °C for 36 h under an atmosphere of H_2 (180 psi) in a Parr reactor. The resulting mixture was filtered through Celite, and volatiles were removed from the filtrate by evaporation under reduced pressure. This yielded a residue of 1,1'-oxybis[3-(4-aminophenoxy)-2,2-bis[(4-aminophenoxy)methyl]]propane (**16**; 10.8 g, 13.5 mmol, 99%) as a colorless solid. Crystals of analytical purity could be grown from CH_3OH/H_2O: mp 164 °C; IR (KBr) 3418, 3346, 3213, 2927, 2875, 1623, 1511, 1466, 1229, 1120, 1029, 824, 516 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 6.58 (d, ^3J = 8.8 Hz, 12H), 6.46 (d, ^3J = 8.8 Hz, 12H), 4.58 (s, 12H), 3.90 (s, 12H), 3.63 (s, 4H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 150.2, 142.6, 115.7, 114.9, 69.6, 67.5, 44.8; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 800. Anal. Calcd for $C_{46}H_{52}N_6O_7 \bullet 0.5\ H_2O$: C, 68.21; H, 6.60; N, 10.38. Found: C, 68.42; H, 6.81; N, 10.21.

1,1'-Oxybis[3-[4-(hydroxymethyl)phenoxy]-2,2-bis[[4-(hydroxymethyl)phenoxy]methyl]]propane (17). A solution of 1,1'-oxybis[3-(4-formylphenoxy)-2,2-bis[(4-formylphenoxy)methyl]]propane (**15**; 1.00 g, 1.14 mmol) in CH_3OH (50 mL) was stirred at -10 ^oC and treated with $NaBH_4$ (0.775 g, 20.5 mmol), added in small portions. The resulting mixture was stirred at 0 ^oC for 30 min and then at 25 ^oC for 7 h. The mixture was poured into cold water (200 mL), and the resulting mixture was extracted twice with $CH_3COOC_2H_5$. The organic layers were combined and dried over Na_2SO_4, and volatiles were removed by evaporation under reduced pressure. The residue was purified by flash chromatography (silica, $CH_3COOC_2H_5$ (95%)/C_2H_5OH (5%), R_f 0.43) to give 1,1'-oxybis[3-[4-(hydroxymethyl)phenoxy)-2,2-bis[[4-(hydroxymethyl)phenoxy]methyl]]propane (**17**; 0.833 g, 0.935 mmol, 82%) as a colorless solid: mp 51 °C; IR (KBr) 3400, 2932, 2876, 1611, 1512, 1465, 1239, 1173, 1034, 827 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 7.15 (d, ^3J = 8.7 Hz, 12H), 6.80 (d, ^3J = 8.7 Hz, 12H), 5.05 (t, ^3J = 5.6 Hz, 6H), 4.38 (d, ^3J = 5.6 Hz, 12H), 4.07 (s, 12H), 3.71 (s, 4H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 157.6,

134.9, 127.9, 114.3, 69.5, 66.6, 62.6, 44.8; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 890. Anal. Calcd for $C_{52}H_{58}O_{13}$: C, 70.10; H, 6.56. Found: C, 69.72; H, 6.74.

Hexaboronic Acid 18. A solution of 1,1'-oxybis[3-(4-bromophenoxy)-2,2-bis[(4-bromophenoxy)methyl]]propane (**9**; 0.406 g, 0.343 mmol) in dry THF (50 mL) was stirred vigorously at -78 ^{o}C under dry N_2 and treated dropwise with a solution of butyllithium (1.10 mL, 2.50 M in hexane, 2.75 mmol). The resulting mixture was kept at -78 ^{o}C for 45 min, and then B(O-*i*Pr)$_3$ (0.63 mL, 2.7 mmol) was added dropwise. The mixture was stirred at -78 ^{o}C for 20 min and warmed to 25 °C over 2 h. After addition of water (20 mL), the mixture was concentrated by partial evaporation of volatiles under reduced pressure. The concentrate was dissolved in aqueous NaOH (1 N, 50 mL) and the solution was washed with ether. Aqueous HCl (1 N) was then added until the pH was reduced to 2. The resulting precipitate was separated by filtration and dried to afford hexaboronic acid **18** (0.291 mg, 0.299 mmol, 87%) as a colorless solid: mp > 300°C; IR (KBr) 3600-3000 (bs), 2928, 1603, 1410, 1364, 1282, 1250, 1179, 1113, 1026, 832, 737, 644, 628 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 7.83 (s, 12H), 7.68 (d, ^{3}J = 8.5 Hz, 12H), 6.83 (d, ^{3}J = 8.5 Hz, 12H), 4.12 (s, 12H), 3.74 (s, 4H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 160.4, 136.0, 126,0 113.7, 69.8, 66.3, 44.8. Anal. Calcd for $C_{46}H_{52}B_6O_{19} \bullet 2\ H_2O$: C, 54.71; H, 5.59. Found: C, 54.68; H, 5.76.

1,1'-Oxybis[3-[4-(4,6-diamino-1,3,5-triazin-2-yl)phenoxy]-2,2-bis[[4-(4,6-diamino-1,3,5-triazin-2-yl)phenoxy]methyl]]propane (7). A mixture of 1,1'-oxybis[3-(4-cyanophenoxy)-2,2-bis[(4-cyanophenoxy)methyl]]propane (**13**; 0.717 g, 0.833 mmol), dicyandiamide (0.840 g, 9.99 mmol), and powdered KOH (0.280 g, 4.99 mmol) in 2-methoxyethanol (15 mL) was heated at reflux for 16 h. The resulting mixture was cooled, and a mixture of 80:20 (v:v) water/CH_3OH (200 mL) was added. The resulting precipitate was separated by filtration and washed thoroughly with water and CH_3OH. The crude solid was dissolved in DMF and reprecipitated by adding CH_3OH,

and the reprecipitated solid was further purified by trituration with CH_3OH to give tecton **7** (0.901 g, 0.660 mmol, 79%) as a colorless solid: mp 218-220 °C; IR (KBr) 3500-2800 (bs), 2930, 2880, 1606, 1542, 1435, 1396, 1241, 1159, 816 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 8.17 (d, ^3J = 8.7 Hz, 12H), 6.96 (d, ^3J = 8.7 Hz, 12H), 6.65 (s, 24H), 4.23 (s, 12H), 3.79 (s, 4H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 169.7, 167.4, 161.1, 129.7, 129.5, 114.1, 69.9, 66.8, 44.6; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 1365 (M+1). Anal. Calcd for $C_{64}H_{64}N_{30}O_7 \bullet 5\ H_2O$: C, 52.81; H, 5.12; N, 28.87. Found: C, 52.74; H, 5.37; N, 28.61.

Université de Montréal

CRYSTAL AND MOLECULAR STRUCTURE OF

C54 H66 O21 S6 COMPOUND (JIW578)

Equipe WUEST

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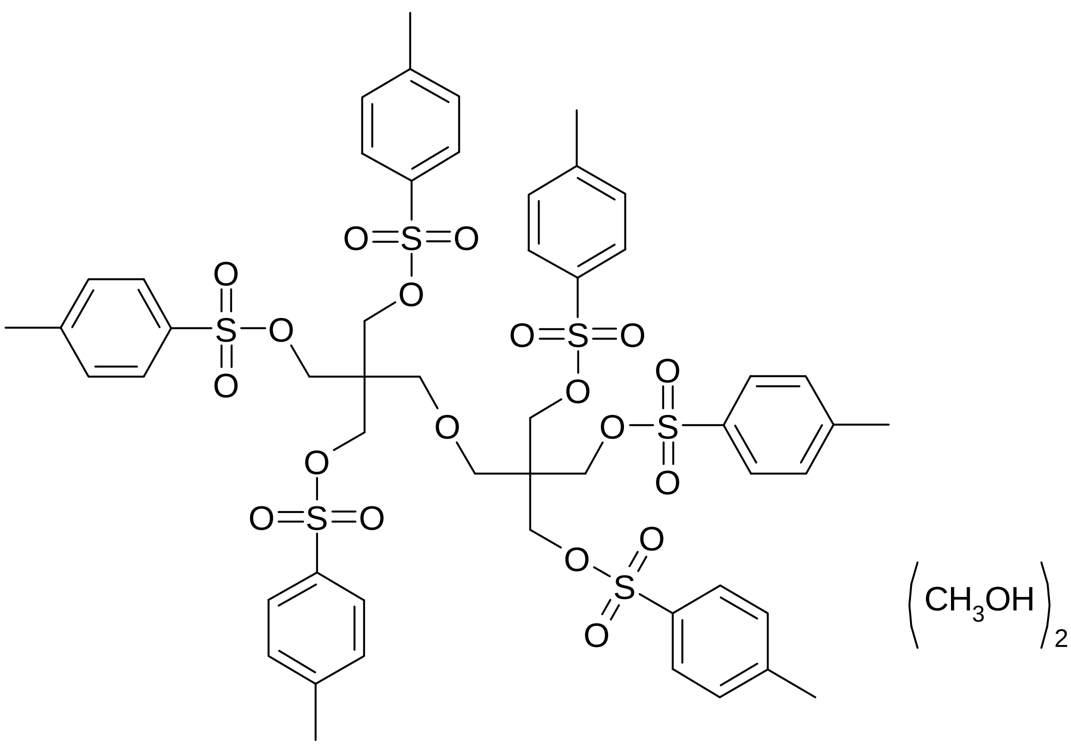


Structure solved and refined in the Laboratory of X-Ray Diffraction, Université de Montréal by Dr. Thierry Maris.

Table 1. Crystal data and structure refinement for C54 H66 O21 S6.

Identification code	JIW578
Empirical formula	C54 H66 O21 S6
Formula weight	1243.43
Temperature	293(2)K
Wavelength	1.54178 Å
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 14.1959(19) Å α = 67.503(3)° b = 14.724(2) Å β = 67.112(4)° c = 17.312(2) Å γ = 88.948(4)°
Volume	3043.6(7)Å^3
Z	2
Density (calculated)	1.357 g/cm^3
Absorption coefficient	2.701 mm^{-1}
F(000)	1308
Crystal size	0.80 x 0.50 x 0.20 mm
Theta range for data collection	3.03 to 69.86°
Index ranges	$-16 \le h \le 17$, $-17 \le k \le 17$, $-21 \le \ell \le 21$
Reflections collected	36160
Independent reflections	11115 [R_{int} = 0.032]
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.6900
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	11115 / 3 / 711
Goodness-of-fit on F^2	1.038
Final R indices [I>2sigma(I)]	R_1 = 0.0561, wR_2 = 0.1391
R indices (all data)	R_1 = 0.0929, wR_2 = 0.1519
Largest diff. peak and hole	0.450 and -0.323 e/Å^3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for C54 H66 O21 S6.

U_{eq} is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U_{eq}
S(1)	2431(1)	-2775(1)	10387(1)	75(1)
S(2)	3359(1)	341(1)	11164(1)	85(1)
S(3)	941(1)	64(1)	8040(1)	102(1)
S(4)	6203(1)	3204(1)	5360(1)	76(1)
S(5)	6223(1)	5843(1)	6587(1)	85(1)
S(6)	1466(1)	3473(1)	9162(1)	76(1)
O(1)	3460(2)	1842(1)	8136(2)	76(1)
O(10)	1563(2)	-3334(2)	11214(2)	96(1)
O(11)	3451(2)	-2876(2)	10345(2)	91(1)
O(12)	2255(1)	-1657(1)	10178(1)	68(1)
O(20)	2788(2)	1102(2)	11308(2)	101(1)
O(21)	4381(2)	369(2)	11104(2)	116(1)
O(22)	3466(2)	327(2)	10226(2)	78(1)
O(30)	-37(2)	-294(2)	8831(2)	126(1)
O(31)	1231(3)	-359(2)	7385(3)	153(1)
O(32)	1848(2)	-95(2)	8372(2)	90(1)
O(40)	7261(2)	3191(2)	5217(2)	99(1)
O(41)	5917(2)	3880(2)	4678(2)	90(1)
O(42)	5670(2)	3390(2)	6266(1)	74(1)
O(50)	6161(2)	6746(2)	5925(2)	116(1)
O(51)	7013(2)	5287(2)	6344(2)	109(1)
O(52)	5118(2)	5197(2)	6973(2)	81(1)
O(60)	1194(2)	2479(2)	9323(2)	97(1)
O(61)	1040(2)	4225(2)	8665(2)	100(1)
O(62)	2690(2)	3754(2)	8650(2)	79(1)
C(1)	4212(2)	3545(2)	7491(2)	58(1)
C(2)	4163(2)	2491(2)	8152(2)	67(1)
C(3)	3643(2)	841(2)	8370(2)	72(1)
C(4)	2743(2)	112(2)	9242(2)	60(1)
C(10)	2354(2)	-2945(2)	9461(2)	70(1)
C(11)	3132(3)	-3353(2)	8968(3)	86(1)
C(12)	3056(3)	-3479(3)	8233(3)	91(1)
C(13)	2268(3)	-3198(2)	7977(3)	84(1)
C(14)	1500(3)	-2795(3)	8489(3)	86(1)
C(15)	1536(2)	-2677(2)	9221(3)	76(1)
C(16)	2228(3)	-3306(3)	7150(3)	119(1)
C(17)	3079(2)	-901(2)	9386(2)	64(1)
C(20)	2631(3)	-802(3)	11987(3)	92(1)
C(21)	1679(5)	-848(4)	12638(4)	137(2)
C(22)	1126(5)	-1809(7)	13328(4)	171(2)
C(23)	1551(7)	-2655(6)	13281(6)	166(3)
C(24)	2465(7)	-2568(5)	12635(6)	157(3)
C(25)	3011(4)	-1670(4)	11977(4)	123(2)
C(26)	892(6)	-3643(5)	14059(6)	274(5)
C(27)	2535(2)	361(2)	10063(2)	66(1)
C(30)	1090(3)	1359(2)	7481(2)	77(1)
C(31)	1878(3)	1856(4)	6635(3)	114(1)
C(32)	2006(4)	2866(4)	6216(3)	120(2)

C(33)	1367(4)	3410(3)	6625(3)	95(1)
C(34)	595(3)	2909(3)	7463(3)	95(1)
C(35)	454(3)	1890(3)	7902(2)	83(1)
C(36)	1540(4)	4529(3)	6156(3)	153(2)
C(37)	1730(2)	171(2)	9122(2)	71(1)
C(40)	5624(3)	1998(2)	5691(2)	76(1)
C(41)	4872(3)	1844(3)	5443(3)	88(1)
C(42)	4406(3)	852(4)	5743(3)	107(1)
C(43)	4696(4)	75(3)	6291(3)	111(2)
C(44)	5481(5)	257(3)	6518(3)	125(2)
C(45)	5935(3)	1194(3)	6236(3)	103(1)
C(46)	4131(4)	-961(3)	6621(4)	168(2)
C(47)	4633(2)	3626(2)	6501(2)	63(1)
C(50)	6202(2)	6025(2)	7529(2)	69(1)
C(51)	5899(3)	6882(3)	7641(3)	87(1)
C(52)	5853(3)	7004(3)	8389(3)	96(1)
C(53)	6096(3)	6313(3)	9045(2)	81(1)
C(54)	6424(3)	5481(3)	8915(3)	93(1)
C(55)	6483(3)	5329(2)	8173(3)	84(1)
C(56)	6000(3)	6456(4)	9891(3)	123(2)
C(57)	4962(3)	4196(2)	7597(2)	74(1)
C(60)	1272(2)	3540(2)	10200(2)	71(1)
C(61)	1275(3)	2712(3)	10914(3)	92(1)
C(62)	1096(3)	2760(3)	11735(3)	104(1)
C(63)	900(3)	3633(3)	11863(3)	96(1)
C(64)	892(3)	4455(3)	11128(3)	92(1)
C(65)	1080(3)	4424(2)	10303(3)	83(1)
C(66)	706(4)	3698(4)	12761(3)	145(2)
C(67)	3140(2)	3845(2)	7706(2)	69(1)
O(101)	1440(20)	-496(18)	5307(17)	696(13)
C(102)	410(20)	-380(30)	5410(30)	830(30)
O(103)	1650(20)	1160(20)	14710(20)	799(19)
C(104)	1810(30)	910(20)	13950(20)	730(20)

Table 3. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2\ x\ 10^3$) for C54 H66 O21 S6.

	x	y	z	U_{eq}
H(2A)	4844	2293	7974	80
H(2B)	3940	2460	8768	80
H(3A)	4274	782	8467	86
H(3B)	3741	661	7863	86
H(11)	3686	-3536	9123	103
H(12)	3562	-3765	7909	110
H(14)	953	-2603	8326	103
H(15)	1011	-2417	9559	91
H(16A)	1619	-3077	7076	179
H(16B)	2210	-3993	7243	179
H(16C)	2831	-2917	6609	179
H(17A)	3222	-1028	8844	77
H(17B)	3706	-919	9488	77
H(21)	1395	-272	12635	165
H(22)	491	-1865	13800	205
H(24)	2749	-3140	12627	188
H(25)	3645	-1639	11516	148
H(26A)	243	-3514	14427	412
H(26B)	1254	-3956	14435	412
H(26C)	769	-4076	13800	412
H(27A)	2339	1019	9943	79
H(27B)	1971	-115	10601	79
H(31)	2326	1503	6345	137
H(32)	2543	3195	5639	144
H(34)	147	3263	7750	114
H(35)	-75	1564	8484	100
H(36A)	2133	4746	5578	229
H(36B)	1657	4805	6538	229
H(36C)	941	4748	6054	229
H(37A)	1171	-282	9682	85
H(37B)	1563	840	8992	85
H(41)	4661	2379	5080	105
H(42)	3899	737	5562	128
H(44)	5703	-276	6872	149
H(45)	6454	1301	6406	124
H(46A)	3610	-914	6388	253
H(46B)	3810	-1260	7280	253
H(46C)	4616	-1363	6402	253
H(47A)	4200	3164	6455	76
H(47B)	4637	4294	6086	76
H(51)	5728	7370	7205	105
H(52)	5647	7581	8458	115
H(54)	6611	5008	9348	112
H(55)	6712	4759	8099	101
H(56A)	6219	5905	10267	184
H(56B)	5293	6492	10232	184
H(56C)	6429	7062	9710	184
H(57A)	4676	4153	8220	89
H(57B)	5620	3950	7480	89
H(61)	1398	2119	10845	110
H(62)	1106	2195	12217	125
H(64)	755	5045	11198	110

H(65)	1079	4989	9816	99
H(66A)	757	3069	13187	218
H(66B)	26	3861	13010	218
H(66C)	1212	4204	12658	218
H(67A)	3187	4526	7290	82
H(67B)	2705	3418	7627	82
H(101)	1707	-744	4936	1044
H(10A)	-27	-529	6043	1245
H(10B)	159	-833	5227	1245
H(10C)	387	288	5035	1245
H(103)	2088	952	14908	1199
H(10D)	1882	224	14115	1102
H(10E)	2432	1315	13437	1102
H(10F)	1233	1042	13790	1102

Table 4. Anisotropic parameters ($Å^2$ x 10^3) for C54 H66 O21 S6.

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2 [h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
S(1)	81(1)	45(1)	87(1)	-21(1)	-26(1)	6(1)
S(2)	79(1)	81(1)	122(1)	-52(1)	-59(1)	24(1)
S(3)	124(1)	71(1)	143(1)	-42(1)	-89(1)	19(1)
S(4)	81(1)	70(1)	69(1)	-28(1)	-21(1)	6(1)
S(5)	92(1)	75(1)	74(1)	-24(1)	-25(1)	-21(1)
S(6)	63(1)	66(1)	91(1)	-32(1)	-25(1)	9(1)
O(1)	68(1)	44(1)	109(2)	-19(1)	-42(1)	3(1)
O(10)	109(2)	61(1)	85(2)	-23(1)	-13(1)	-12(1)
O(11)	93(2)	64(1)	124(2)	-33(1)	-58(2)	27(1)
O(12)	64(1)	47(1)	80(1)	-23(1)	-22(1)	6(1)
O(20)	114(2)	84(2)	141(2)	-63(2)	-71(2)	37(1)
O(21)	89(2)	135(2)	169(3)	-79(2)	-83(2)	27(2)
O(22)	59(1)	90(2)	98(2)	-46(1)	-39(1)	17(1)
O(30)	97(2)	91(2)	158(3)	-3(2)	-67(2)	-25(2)
O(31)	246(4)	111(2)	213(3)	-102(2)	-171(3)	71(2)
O(32)	105(2)	76(2)	122(2)	-55(2)	-65(2)	34(1)
O(40)	66(2)	111(2)	109(2)	-49(2)	-21(1)	10(1)
O(41)	111(2)	73(1)	70(2)	-19(1)	-33(1)	10(1)
O(42)	71(1)	78(1)	76(1)	-38(1)	-29(1)	12(1)
O(50)	158(2)	83(2)	80(2)	4(1)	-58(2)	-31(2)
O(51)	103(2)	97(2)	114(2)	-50(2)	-23(2)	-7(2)
O(52)	97(2)	60(1)	84(2)	-23(1)	-42(1)	0(1)
O(60)	97(2)	72(2)	108(2)	-40(1)	-26(1)	-6(1)
O(61)	91(2)	97(2)	110(2)	-38(2)	-46(2)	32(1)
O(62)	64(1)	92(2)	78(2)	-42(1)	-21(1)	5(1)
C(1)	60(2)	49(2)	62(2)	-20(1)	-24(1)	1(1)
C(2)	73(2)	56(2)	74(2)	-23(2)	-36(2)	7(2)
C(3)	67(2)	45(2)	83(2)	-18(2)	-18(2)	6(1)
C(4)	61(2)	41(1)	77(2)	-22(2)	-30(2)	8(1)
C(10)	66(2)	44(2)	83(2)	-25(2)	-16(2)	2(1)
C(11)	73(2)	63(2)	99(3)	-28(2)	-18(2)	17(2)
C(12)	86(3)	73(2)	93(3)	-36(2)	-13(2)	16(2)
C(13)	91(3)	62(2)	83(3)	-31(2)	-18(2)	-2(2)
C(14)	78(2)	72(2)	107(3)	-40(2)	-35(2)	3(2)
C(15)	58(2)	67(2)	101(3)	-46(2)	-19(2)	8(2)
C(16)	136(4)	113(3)	99(3)	-49(3)	-32(3)	-7(3)
C(17)	58(2)	44(2)	79(2)	-20(2)	-21(2)	5(1)
C(20)	92(3)	93(3)	114(3)	-38(2)	-69(3)	27(2)
C(21)	125(4)	121(4)	132(4)	-18(3)	-52(3)	19(3)
C(22)	123(5)	185(7)	145(5)	-11(5)	-51(4)	-7(5)
C(23)	175(7)	125(5)	193(7)	9(5)	-143(6)	-4(6)
C(24)	181(6)	90(4)	239(8)	-36(5)	-157(6)	30(4)
C(25)	139(4)	88(3)	178(5)	-47(3)	-107(4)	38(3)
C(26)	243(8)	169(6)	308(9)	95(6)	-196(7)	-72(6)
C(27)	56(2)	56(2)	88(2)	-28(2)	-34(2)	13(1)
C(30)	73(2)	76(2)	78(2)	-27(2)	-32(2)	14(2)
C(31)	108(3)	119(4)	88(3)	-44(3)	-10(2)	30(3)
C(32)	111(3)	111(4)	72(3)	-8(3)	-1(2)	-10(3)
C(33)	112(3)	76(3)	85(3)	-10(2)	-50(3)	-1(2)

C(34)	102(3)	81(3)	91(3)	-31(2)	-34(2)	26(2)
C(35)	68(2)	81(2)	73(2)	-17(2)	-16(2)	10(2)
C(36)	218(6)	75(3)	150(4)	-2(3)	-103(4)	-5(3)
C(37)	67(2)	59(2)	90(2)	-30(2)	-37(2)	16(1)
C(40)	80(2)	71(2)	71(2)	-30(2)	-25(2)	12(2)
C(41)	101(3)	77(2)	92(3)	-41(2)	-41(2)	18(2)
C(42)	106(3)	103(3)	117(3)	-62(3)	-34(3)	6(3)
C(43)	130(4)	66(3)	95(3)	-37(2)	-2(3)	2(3)
C(44)	157(5)	69(3)	118(4)	-24(3)	-42(3)	25(3)
C(45)	130(3)	76(3)	104(3)	-32(2)	-53(3)	28(2)
C(46)	195(5)	85(3)	155(5)	-53(3)	2(4)	-27(3)
C(47)	66(2)	56(2)	69(2)	-25(2)	-31(2)	8(1)
C(50)	67(2)	62(2)	68(2)	-17(2)	-27(2)	-10(2)
C(51)	111(3)	72(2)	99(3)	-34(2)	-63(2)	23(2)
C(52)	101(3)	96(3)	120(3)	-63(3)	-58(3)	23(2)
C(53)	73(2)	92(3)	75(2)	-26(2)	-34(2)	-13(2)
C(54)	115(3)	73(2)	96(3)	-18(2)	-64(2)	0(2)
C(55)	98(3)	62(2)	108(3)	-33(2)	-60(2)	11(2)
C(56)	117(3)	161(4)	96(3)	-60(3)	-40(3)	-28(3)
C(57)	91(2)	61(2)	66(2)	-24(2)	-30(2)	-6(2)
C(60)	54(2)	62(2)	87(2)	-29(2)	-19(2)	9(1)
C(61)	99(3)	73(2)	92(3)	-33(2)	-31(2)	31(2)
C(62)	106(3)	96(3)	95(3)	-32(3)	-35(2)	33(2)
C(63)	77(3)	106(3)	101(3)	-51(3)	-25(2)	10(2)
C(64)	80(2)	79(3)	111(3)	-50(3)	-23(2)	6(2)
C(65)	78(2)	56(2)	100(3)	-29(2)	-26(2)	7(2)
C(66)	141(4)	196(5)	117(4)	-94(4)	-41(3)	26(4)
C(67)	72(2)	59(2)	70(2)	-23(2)	-28(2)	10(2)

Table 5. Bond lengths [Å] and angles [°] for C54 H66 O21 S6

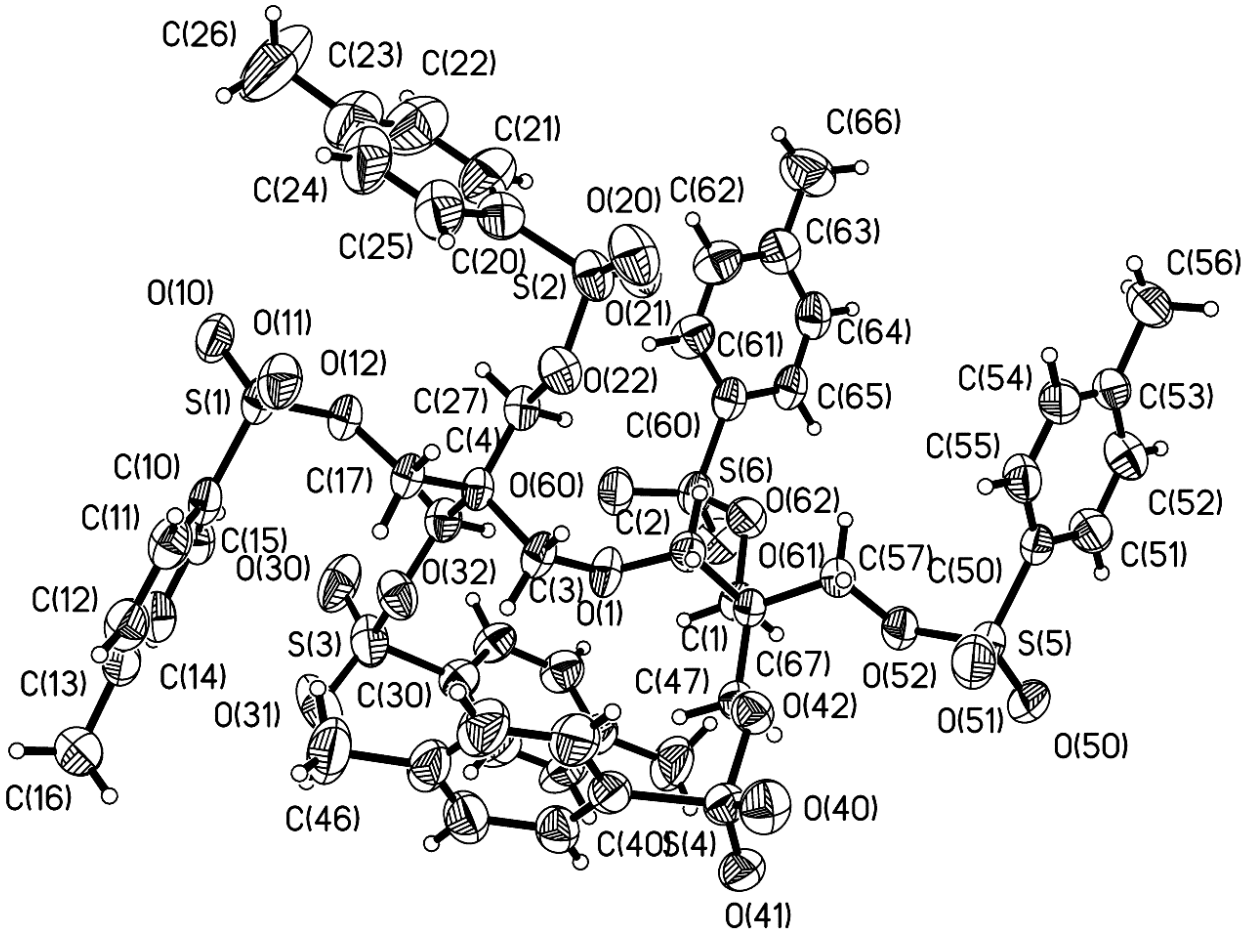
S(1)-O(10)	1.417(2)	C(32)-C(33)	1.368(6)
S(1)-O(11)	1.430(2)	C(33)-C(34)	1.350(5)
S(1)-O(12)	1.5824(18)	C(33)-C(36)	1.506(5)
S(1)-C(10)	1.757(3)	C(34)-C(35)	1.374(5)
S(2)-O(21)	1.413(2)	C(40)-C(41)	1.347(4)
S(2)-O(20)	1.414(2)	C(40)-C(45)	1.391(5)
S(2)-O(22)	1.579(2)	C(41)-C(42)	1.425(5)
S(2)-C(20)	1.737(4)	C(42)-C(43)	1.354(6)
S(3)-O(31)	1.416(3)	C(43)-C(44)	1.378(6)
S(3)-O(30)	1.441(3)	C(43)-C(46)	1.524(5)
S(3)-O(32)	1.583(2)	C(44)-C(45)	1.353(6)
S(3)-C(30)	1.750(3)	C(50)-C(55)	1.382(4)
S(4)-O(41)	1.417(2)	C(50)-C(51)	1.385(4)
S(4)-O(40)	1.425(2)	C(51)-C(52)	1.351(5)
S(4)-O(42)	1.584(2)	C(52)-C(53)	1.361(5)
S(4)-C(40)	1.751(3)	C(53)-C(54)	1.369(5)
S(5)-O(51)	1.403(3)	C(53)-C(56)	1.512(5)
S(5)-O(50)	1.412(2)	C(54)-C(55)	1.359(5)
S(5)-O(52)	1.602(2)	C(60)-C(61)	1.366(4)
S(5)-C(50)	1.739(3)	C(60)-C(65)	1.389(4)
S(6)-O(61)	1.409(2)	C(61)-C(62)	1.373(5)
S(6)-O(60)	1.412(2)	C(62)-C(63)	1.392(5)
S(6)-O(62)	1.589(2)	C(63)-C(64)	1.384(5)
S(6)-C(60)	1.750(3)	C(63)-C(66)	1.511(5)
O(1)-C(2)	1.409(3)	C(64)-C(65)	1.365(5)
O(1)-C(3)	1.420(3)	O(101)-C(102)	1.427(5)
O(12)-C(17)	1.456(3)	O(103)-C(104)	1.422(5)
O(22)-C(27)	1.450(3)		
O(32)-C(37)	1.443(4)	O(10)-S(1)-O(11)	119.61(15)
O(42)-C(47)	1.441(3)	O(10)-S(1)-O(12)	104.19(12)
O(52)-C(57)	1.413(3)	O(11)-S(1)-O(12)	109.52(12)
O(62)-C(67)	1.455(3)	O(10)-S(1)-C(10)	110.53(15)
C(1)-C(2)	1.518(4)	O(11)-S(1)-C(10)	108.83(15)
C(1)-C(67)	1.520(4)	O(12)-S(1)-C(10)	102.77(13)
C(1)-C(47)	1.535(4)	O(21)-S(2)-O(20)	119.80(16)
C(1)-C(57)	1.552(4)	O(21)-S(2)-O(22)	104.55(15)
C(3)-C(4)	1.540(4)	O(20)-S(2)-O(22)	108.80(14)
C(4)-C(17)	1.515(3)	O(21)-S(2)-C(20)	109.69(18)
C(4)-C(27)	1.520(4)	O(20)-S(2)-C(20)	108.71(19)
C(4)-C(37)	1.527(4)	O(22)-S(2)-C(20)	104.12(16)
C(10)-C(15)	1.382(4)	O(31)-S(3)-O(30)	121.0(2)
C(10)-C(11)	1.394(4)	O(31)-S(3)-O(32)	103.02(17)
C(11)-C(12)	1.397(5)	O(30)-S(3)-O(32)	109.07(16)
C(12)-C(13)	1.359(5)	O(31)-S(3)-C(30)	109.71(19)
C(13)-C(14)	1.398(5)	O(30)-S(3)-C(30)	109.58(18)
C(13)-C(16)	1.521(5)	O(32)-S(3)-C(30)	102.81(14)
C(14)-C(15)	1.364(5)	O(41)-S(4)-O(40)	121.21(15)
C(20)-C(21)	1.365(6)	O(41)-S(4)-O(42)	108.56(13)
C(20)-C(25)	1.381(5)	O(40)-S(4)-O(42)	104.59(13)
C(21)-C(22)	1.436(8)	O(41)-S(4)-C(40)	108.96(16)
C(22)-C(23)	1.392(9)	O(40)-S(4)-C(40)	109.01(16)
C(23)-C(24)	1.311(9)	O(42)-S(4)-C(40)	102.94(13)
C(23)-C(26)	1.542(8)	O(51)-S(5)-O(50)	120.92(18)
C(24)-C(25)	1.358(8)	O(51)-S(5)-O(52)	109.88(14)
C(30)-C(35)	1.363(5)	O(50)-S(5)-O(52)	102.69(15)
C(30)-C(31)	1.366(5)	O(51)-S(5)-C(50)	108.38(17)
C(31)-C(32)	1.362(6)	O(50)-S(5)-C(50)	110.00(17)
		O(52)-S(5)-C(50)	103.53(13)

O(61)-S(6)-O(60)	118.64(16)	C(24)-C(25)-C(20)	120.5(5)
O(61)-S(6)-O(62)	107.77(14)	O(22)-C(27)-C(4)	108.9(2)
O(60)-S(6)-O(62)	108.54(13)	C(35)-C(30)-C(31)	119.1(3)
O(61)-S(6)-C(60)	110.86(15)	C(35)-C(30)-S(3)	120.5(3)
O(60)-S(6)-C(60)	109.58(15)	C(31)-C(30)-S(3)	120.4(3)
O(62)-S(6)-C(60)	99.73(13)	C(32)-C(31)-C(30)	120.0(4)
C(2)-O(1)-C(3)	115.8(2)	C(31)-C(32)-C(33)	121.7(4)
C(17)-O(12)-S(1)	116.22(16)	C(34)-C(33)-C(32)	117.6(4)
C(27)-O(22)-S(2)	117.21(19)	C(34)-C(33)-C(36)	121.7(5)
C(37)-O(32)-S(3)	116.90(19)	C(32)-C(33)-C(36)	120.6(4)
C(47)-O(42)-S(4)	117.93(17)	C(33)-C(34)-C(35)	121.8(4)
C(57)-O(52)-S(5)	117.11(19)	C(30)-C(35)-C(34)	119.8(3)
C(67)-O(62)-S(6)	115.47(18)	O(32)-C(37)-C(4)	108.7(2)
C(2)-C(1)-C(67)	111.0(2)	C(41)-C(40)-C(45)	120.1(3)
C(2)-C(1)-C(47)	110.6(2)	C(41)-C(40)-S(4)	120.9(3)
C(67)-C(1)-C(47)	106.6(2)	C(45)-C(40)-S(4)	119.0(3)
C(2)-C(1)-C(57)	104.8(2)	C(40)-C(41)-C(42)	119.2(4)
C(67)-C(1)-C(57)	113.3(2)	C(43)-C(42)-C(41)	120.3(4)
C(47)-C(1)-C(57)	110.6(2)	C(42)-C(43)-C(44)	119.0(4)
O(1)-C(2)-C(1)	109.3(2)	C(42)-C(43)-C(46)	117.5(6)
O(1)-C(3)-C(4)	112.1(2)	C(44)-C(43)-C(46)	123.5(5)
C(17)-C(4)-C(27)	112.0(2)	C(45)-C(44)-C(43)	121.4(4)
C(17)-C(4)-C(37)	112.2(2)	C(44)-C(45)-C(40)	119.9(4)
C(27)-C(4)-C(37)	105.1(2)	O(42)-C(47)-C(1)	107.6(2)
C(17)-C(4)-C(3)	104.5(2)	C(55)-C(50)-C(51)	118.9(3)
C(27)-C(4)-C(3)	112.0(2)	C(55)-C(50)-S(5)	121.3(3)
C(37)-C(4)-C(3)	111.2(2)	C(51)-C(50)-S(5)	119.7(3)
C(15)-C(10)-C(11)	120.3(3)	C(52)-C(51)-C(50)	119.4(3)
C(15)-C(10)-S(1)	120.6(2)	C(51)-C(52)-C(53)	122.5(4)
C(11)-C(10)-S(1)	119.2(3)	C(52)-C(53)-C(54)	117.7(4)
C(10)-C(11)-C(12)	118.1(3)	C(52)-C(53)-C(56)	121.2(4)
C(13)-C(12)-C(11)	122.3(3)	C(54)-C(53)-C(56)	121.2(4)
C(12)-C(13)-C(14)	118.0(4)	C(55)-C(54)-C(53)	121.8(3)
C(12)-C(13)-C(16)	121.1(4)	C(54)-C(55)-C(50)	119.6(3)
C(14)-C(13)-C(16)	120.8(4)	O(52)-C(57)-C(1)	110.2(2)
C(15)-C(14)-C(13)	121.5(3)	C(61)-C(60)-C(65)	120.4(3)
C(14)-C(15)-C(10)	119.8(3)	C(61)-C(60)-S(6)	119.7(3)
O(12)-C(17)-C(4)	109.0(2)	C(65)-C(60)-S(6)	119.9(3)
C(21)-C(20)-C(25)	119.8(4)	C(60)-C(61)-C(62)	119.6(3)
C(21)-C(20)-S(2)	120.3(4)	C(61)-C(62)-C(63)	121.5(4)
C(25)-C(20)-S(2)	119.9(4)	C(64)-C(63)-C(62)	117.3(4)
C(20)-C(21)-C(22)	118.2(5)	C(64)-C(63)-C(66)	120.6(4)
C(23)-C(22)-C(21)	119.2(6)	C(62)-C(63)-C(66)	122.1(4)
C(24)-C(23)-C(22)	120.0(7)	C(65)-C(64)-C(63)	122.0(3)
C(24)-C(23)-C(26)	125.3(9)	C(64)-C(65)-C(60)	119.2(3)
C(22)-C(23)-C(26)	114.6(9)	O(62)-C(67)-C(1)	108.9(2)
C(23)-C(24)-C(25)	122.2(7)		

Table 6. Torsion angles [°] for C54 H66 O21 S6.

O(10)-S(1)-O(12)-C(17)	176.0(2)	C(26)-C(23)-C(24)-C(25)	-178.9(5)
O(11)-S(1)-O(12)-C(17)	46.9(2)	C(23)-C(24)-C(25)-C(20)	2.5(9)
C(10)-S(1)-O(12)-C(17)	-68.7(2)	C(21)-C(20)-C(25)-C(24)	-3.1(7)
O(21)-S(2)-O(22)-C(27)	-176.2(2)	S(2)-C(20)-C(25)-C(24)	177.8(4)
O(20)-S(2)-O(22)-C(27)	-47.1(2)	S(2)-O(22)-C(27)-C(4)	-167.63(17)
C(20)-S(2)-O(22)-C(27)	68.7(2)	C(17)-C(4)-C(27)-O(22)	58.1(3)
O(31)-S(3)-O(32)-C(37)	-173.9(2)	C(37)-C(4)-C(27)-O(22)	-179.8(2)
O(30)-S(3)-O(32)-C(37)	-44.2(3)	C(3)-C(4)-C(27)-O(22)	-58.9(3)
C(30)-S(3)-O(32)-C(37)	72.1(3)	O(31)-S(3)-C(30)-C(35)	148.4(3)
O(41)-S(4)-O(42)-C(47)	38.0(2)	O(30)-S(3)-C(30)-C(35)	13.4(4)
O(40)-S(4)-O(42)-C(47)	168.7(2)	O(32)-S(3)-C(30)-C(35)	-102.5(3)
C(40)-S(4)-O(42)-C(47)	-77.4(2)	O(31)-S(3)-C(30)-C(31)	-34.7(4)
O(51)-S(5)-O(52)-C(57)	44.0(3)	O(30)-S(3)-C(30)-C(31)	-169.7(3)
O(50)-S(5)-O(52)-C(57)	173.9(2)	O(32)-S(3)-C(30)-C(31)	74.4(4)
C(50)-S(5)-O(52)-C(57)	-71.6(2)	C(35)-C(30)-C(31)-C(32)	-1.2(7)
O(61)-S(6)-O(62)-C(67)	-60.0(2)	S(3)-C(30)-C(31)-C(32)	-178.1(4)
O(60)-S(6)-O(62)-C(67)	69.7(2)	C(30)-C(31)-C(32)-C(33)	0.4(8)
C(60)-S(6)-O(62)-C(67)	-175.7(2)	C(31)-C(32)-C(33)-C(34)	-0.1(7)
C(3)-O(1)-C(2)-C(1)	153.3(2)	C(31)-C(32)-C(33)-C(36)	178.6(4)
C(67)-C(1)-C(2)-O(1)	54.8(3)	C(32)-C(33)-C(34)-C(35)	0.6(6)
C(47)-C(1)-C(2)-O(1)	-63.2(3)	C(36)-C(33)-C(34)-C(35)	-178.2(4)
C(57)-C(1)-C(2)-O(1)	177.5(2)	C(31)-C(30)-C(35)-C(34)	1.6(6)
C(2)-O(1)-C(3)-C(4)	117.4(3)	S(3)-C(30)-C(35)-C(34)	178.6(3)
O(1)-C(3)-C(4)-C(17)	-179.4(2)	C(33)-C(34)-C(35)-C(30)	-1.4(6)
O(1)-C(3)-C(4)-C(27)	-58.0(3)	S(3)-O(32)-C(37)-C(4)	-171.63(19)
O(1)-C(3)-C(4)-C(37)	59.3(3)	C(17)-C(4)-C(37)-O(32)	-55.9(3)
O(10)-S(1)-C(10)-C(15)	60.9(3)	C(27)-C(4)-C(37)-O(32)	-177.8(2)
O(11)-S(1)-C(10)-C(15)	-165.9(2)	C(3)-C(4)-C(37)-O(32)	60.8(3)
O(12)-S(1)-C(10)-C(15)	-49.8(3)	O(41)-S(4)-C(40)-C(41)	-12.4(3)
O(10)-S(1)-C(10)-C(11)	-119.2(3)	O(40)-S(4)-C(40)-C(41)	-146.7(3)
O(11)-S(1)-C(10)-C(11)	14.0(3)	O(42)-S(4)-C(40)-C(41)	102.7(3)
O(12)-S(1)-C(10)-C(11)	130.1(2)	O(41)-S(4)-C(40)-C(45)	169.1(3)
C(15)-C(10)-C(11)-C(12)	0.0(5)	O(40)-S(4)-C(40)-C(45)	34.8(3)
S(1)-C(10)-C(11)-C(12)	-179.9(2)	O(42)-S(4)-C(40)-C(45)	-75.8(3)
C(10)-C(11)-C(12)-C(13)	1.5(5)	C(45)-C(40)-C(41)-C(42)	-0.2(6)
C(11)-C(12)-C(13)-C(14)	-1.7(5)	S(4)-C(40)-C(41)-C(42)	-178.7(3)
C(11)-C(12)-C(13)-C(16)	177.5(3)	C(40)-C(41)-C(42)-C(43)	1.7(6)
C(12)-C(13)-C(14)-C(15)	0.4(5)	C(41)-C(42)-C(43)-C(44)	-3.0(6)
C(16)-C(13)-C(14)-C(15)	-178.8(3)	C(41)-C(42)-C(43)-C(46)	177.4(4)
C(13)-C(14)-C(15)-C(10)	1.0(5)	C(42)-C(43)-C(44)-C(45)	2.8(7)
C(11)-C(10)-C(15)-C(14)	-1.2(5)	C(46)-C(43)-C(44)-C(45)	-177.6(4)
S(1)-C(10)-C(15)-C(14)	178.7(2)	C(43)-C(44)-C(45)-C(40)	-1.2(7)
S(1)-O(12)-C(17)-C(4)	175.17(19)	C(41)-C(40)-C(45)-C(44)	-0.1(6)
C(27)-C(4)-C(17)-O(12)	61.0(3)	S(4)-C(40)-C(45)-C(44)	178.4(3)
C(37)-C(4)-C(17)-O(12)	-56.9(3)	S(4)-O(42)-C(47)-C(1)	167.87(17)
C(3)-C(4)-C(17)-O(12)	-177.6(2)	C(2)-C(1)-C(47)-O(42)	-62.0(3)
O(21)-S(2)-C(20)-C(21)	131.8(4)	C(67)-C(1)-C(47)-O(42)	177.3(2)
O(20)-S(2)-C(20)-C(21)	-0.9(4)	C(57)-C(1)-C(47)-O(42)	53.7(3)
O(22)-S(2)-C(20)-C(21)	-116.8(4)	O(51)-S(5)-C(50)-C(55)	-31.1(3)
O(21)-S(2)-C(20)-C(25)	-49.1(4)	O(50)-S(5)-C(50)-C(55)	-165.3(3)
O(20)-S(2)-C(20)-C(25)	178.2(3)	O(52)-S(5)-C(50)-C(55)	85.6(3)
O(22)-S(2)-C(20)-C(25)	62.3(3)	O(51)-S(5)-C(50)-C(51)	148.9(3)
C(25)-C(20)-C(21)-C(22)	3.5(7)	O(50)-S(5)-C(50)-C(51)	14.7(3)
S(2)-C(20)-C(21)-C(22)	-177.4(4)	O(52)-S(5)-C(50)-C(51)	-94.5(3)
C(20)-C(21)-C(22)-C(23)	-3.4(9)	C(55)-C(50)-C(51)-C(52)	-2.1(5)
C(21)-C(22)-C(23)-C(24)	2.8(1)	S(5)-C(50)-C(51)-C(52)	177.9(3)
C(21)-C(22)-C(23)-C(26)	179.7(5)	C(50)-C(51)-C(52)-C(53)	0.1(6)
C(22)-C(23)-C(24)-C(25)	-2.30(11)	C(51)-C(52)-C(53)-C(54)	1.7(6)

C(51)-C(52)-C(53)-C(56)	-177.8(4)	O(62)-S(6)-C(60)-C(65)	92.0(3)
C(52)-C(53)-C(54)-C(55)	-1.6(6)	C(65)-C(60)-C(61)-C(62)	-0.6(5)
C(56)-C(53)-C(54)-C(55)	177.9(3)	S(6)-C(60)-C(61)-C(62)	-178.3(3)
C(53)-C(54)-C(55)-C(50)	-0.4(6)	C(60)-C(61)-C(62)-C(63)	0.6(6)
C(51)-C(50)-C(55)-C(54)	2.2(5)	C(61)-C(62)-C(63)-C(64)	0.0(6)
S(5)-C(50)-C(55)-C(54)	-177.8(3)	C(61)-C(62)-C(63)-C(66)	-179.7(4)
S(5)-O(52)-C(57)-C(1)	-148.5(2)	C(62)-C(63)-C(64)-C(65)	-0.8(6)
C(2)-C(1)-C(57)-O(52)	176.8(2)	C(66)-C(63)-C(64)-C(65)	179.0(4)
C(67)-C(1)-C(57)-O(52)	-62.0(3)	C(63)-C(64)-C(65)-C(60)	0.8(6)
C(47)-C(1)-C(57)-O(52)	57.5(3)	C(61)-C(60)-C(65)-C(64)	-0.1(5)
O(61)-S(6)-C(60)-C(61)	156.4(3)	S(6)-C(60)-C(65)-C(64)	177.6(3)
O(60)-S(6)-C(60)-C(61)	23.5(3)	S(6)-O(62)-C(67)-C(1)	-148.91(19)
O(62)-S(6)-C(60)-C(61)	-90.2(3)	C(2)-C(1)-C(67)-O(62)	57.8(3)
O(61)-S(6)-C(60)-C(65)	-21.4(3)	C(47)-C(1)-C(67)-O(62)	178.4(2)
O(60)-S(6)-C(60)-C(65)	-154.2(2)	C(57)-C(1)-C(67)-O(62)	-59.8(3)



ORTEP view of the C54 H66 O21 S6 compound with the numbering scheme adopted. Ellipsoids drawn at 30% probability level. Hydrogens represented by sphere of arbitrary size.

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Université de Montréal

CRYSTAL AND MOLECULAR STRUCTURE OF

C136 H216 N30 O43 S4 COMPOUND (JIW669)

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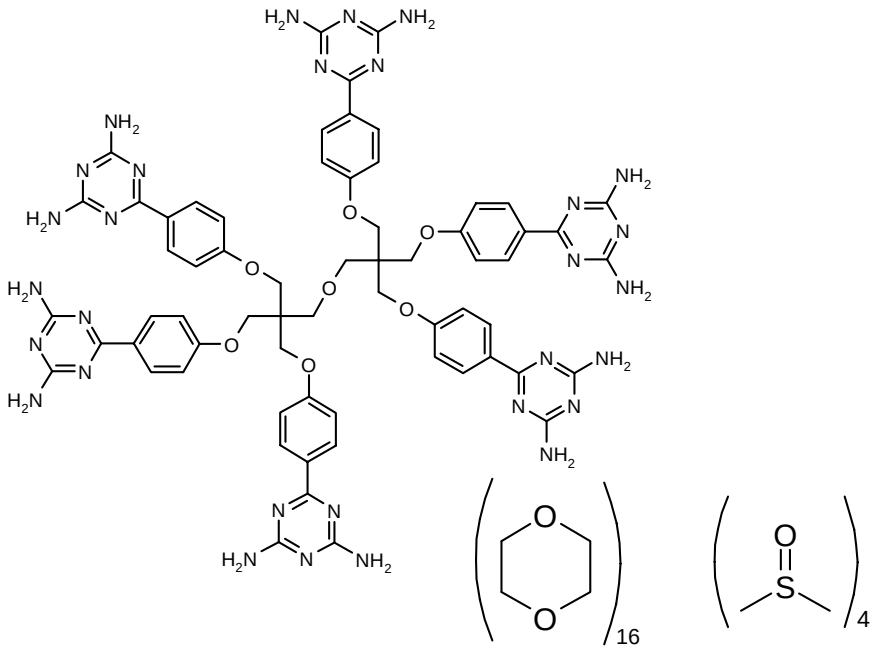


Structure solved and refined in the Laboratory of X-Ray Diffraction, Université de Montréal by Dr. Thierry Maris.

Table 1. Crystal data and structure refinement for C136 H216 N30 O43 S4.

Identification code	JIW669
Empirical formula	C136 H216 N30 O43 S4
Formula weight	3087.63
Temperature	223(2)K
Wavelength	1.54178 Å
Crystal system	Monoclinic
Space group	P2/c
Unit cell dimensions	a = 19.7580(12) Å α = 90° b = 10.8646(7) Å β = 90.759(3)° c = 36.649(2) Å γ = 90°
Volume	7866.5(8)Å^3
Z	2
Density (calculated)	1.304 g/cm^3
Absorption coefficient	1.284 mm^{-1}
F(000)	3300
Crystal size	0.22 x 0.12 x 0.10 mm
Theta range for data collection	2.24 to 62.36°
Index ranges	$-22 \le h \le 21$, $-10 \le k \le 11$, $-40 \le \ell \le 41$
Reflections collected	63526
Independent reflections	11176 [R_{int} = 0.052]
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.6600
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	11176 / 0 / 581
Goodness-of-fit on F^2	1.041
Final R indices [I>2sigma(I)]	R_1 = 0.0970, wR_2 = 0.2195
R indices (all data)	R_1 = 0.1337, wR_2 = 0.2326
Largest diff. peak and hole	0.681 and -0.566 e/Å^3

Table 2. Atomic coordinates ($x\ 10^4$) and equivalent isotropic displacement parameters ($Å^2\ x\ 10^3$) for C136 H216 N30 O43 S4.

U_{eq} is defined as one third of the trace of the orthogonalized Uij tensor.

	Occ.	x	y	z	U_{eq}
C(1)	1	3855(2)	-2123(3)	2322(1)	37(1)
O(1)	1	3605(1)	-531(2)	1881(1)	49(1)
O(2)	1	3723(1)	-1614(2)	2957(1)	48(1)
O(3)	1	3168(1)	-3706(2)	2050(1)	53(1)
O(4)	1	5000	-2378(3)	2500	52(1)
C(10)	1	4038(2)	-1572(3)	1953(1)	42(1)
N(10)	1	4122(2)	1848(2)	312(1)	50(1)
C(11)	1	3723(2)	130(3)	1570(1)	43(1)
N(11)	1	3893(2)	3603(2)	678(1)	56(1)
C(12)	1	4163(2)	-228(3)	1298(1)	43(1)
N(12)	1	4169(2)	3859(2)	51(1)	58(1)
C(13)	1	4230(2)	509(3)	991(1)	44(1)
N(13)	1	3968(2)	5513(3)	419(1)	82(1)
C(14)	1	3885(2)	1610(3)	950(1)	43(1)
N(14)	1	4318(2)	2126(2)	-299(1)	61(1)
C(15)	1	3448(2)	1961(3)	1234(1)	56(1)
C(16)	1	3371(2)	1231(3)	1535(1)	60(1)
C(17)	1	3970(2)	2400(3)	627(1)	48(1)
C(18)	1	4201(2)	2634(3)	30(1)	51(1)
C(19)	1	4010(2)	4298(3)	381(1)	57(1)
C(20)	1	3851(2)	-1080(3)	2604(1)	42(1)
N(20)	1	4173(2)	2440(2)	4178(1)	48(1)
C(21)	1	3815(2)	-882(3)	3254(1)	41(1)
N(21)	1	4039(2)	661(2)	4551(1)	46(1)
C(22)	1	3606(2)	-1336(3)	3583(1)	49(1)
N(22)	1	4143(2)	2655(2)	4825(1)	52(1)
C(23)	1	3685(2)	-672(3)	3898(1)	47(1)
N(23)	1	4011(2)	912(3)	5171(1)	70(1)
C(24)	1	3991(2)	489(3)	3896(1)	42(1)
N(24)	1	4335(2)	4314(3)	4458(1)	64(1)
C(25)	1	4200(2)	944(3)	3562(1)	47(1)
C(26)	1	4117(2)	270(3)	3244(1)	50(1)
C(27)	1	4074(2)	1247(3)	4229(1)	44(1)
C(28)	1	4071(2)	1425(3)	4839(1)	51(1)
C(29)	1	4218(2)	3107(3)	4488(1)	51(1)
C(30)	1	3163(2)	-2730(3)	2312(1)	47(1)
N(30)	1	287(2)	-6230(4)	2012(1)	97(1)
C(31)	1	2558(2)	-4269(3)	1988(1)	56(1)
N(31)	1	-483(3)	-7219(5)	1597(2)	140(2)
C(32)	1	1981(2)	-4045(4)	2177(1)	69(1)
N(32)	1	613(2)	-6565(5)	1399(1)	121(2)
C(33)	1	1390(2)	-4664(4)	2097(1)	80(1)
N(33)	1	-745(3)	-6920(6)	2201(2)	143(2)
C(34)	1	1366(2)	-5514(4)	1817(1)	79(1)
N(34)	1	-133(4)	-7558(7)	1014(2)	212(4)
C(35)	1	1944(3)	-5746(5)	1630(1)	87(2)
C(36)	1	2546(2)	-5151(4)	1709(1)	80(1)
C(37)	1	708(3)	-6142(5)	1746(2)	93(2)
C(38)	1	-311(3)	-6785(6)	1935(2)	115(2)

C(39)	1	0(4)	-7105(7)	1347(2)	138(2)
C(40)	1	4409(2)	-3056(3)	2426(1)	43(1)
O(50)	1	-2077(2)	-8119(5)	2120(2)	174(2)
C(51)	1	-2569(4)	-7554(14)	2375(2)	222(6)
C(52)	1	-2779(5)	-6292(11)	2235(3)	204(5)
O(53)	1	-3077(4)	-6578(7)	1912(2)	205(3)
C(54)	1	-2376(5)	-8198(10)	1787(3)	190(4)
C(55)	1	-2632(6)	-7019(11)	1668(3)	211(5)
S(90)	0.548(4)	4764(1)	-3699(2)	3615(1)	70(1)
O(90)	0.548(4)	4582(3)	-3584(5)	3999(2)	83(2)
C(91)	0.548(4)	5468(2)	-4561(4)	3601(1)	26(1)
C(92)	0.548(4)	4126(3)	-4646(5)	3459(2)	37(2)
S(93)	0.452(4)	4310(3)	-4017(5)	3830(2)	161(2)
O(94)	0.452(4)	5153(3)	-5026(5)	3268(2)	72(2)
C(95)	0.452(4)	4501(5)	-5448(8)	3357(2)	66(3)
C(96)	0.452(4)	5101(6)	-3514(8)	3729(3)	61(2)
S(100)	0.606(11)	2757(3)	7360(7)	950(2)	168(2)
O(100)	0.606(11)	3568(3)	7516(5)	861(2)	72(2)
C(101)	0.606(11)	2539(6)	8888(10)	786(3)	138(4)
C(102)	0.606(11)	2477(8)	7192(14)	488(4)	205(7)
S(103)	0.394(11)	2890(3)	8053(7)	848(2)	150(4)
O(104)	0.394(11)	3429(5)	7144(9)	995(3)	88(3)
C(105)	0.394(11)	2407(9)	6250(17)	771(5)	144(7)
C(106)	0.394(11)	2819(6)	7656(11)	1096(3)	57(3)

Table 3. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for C136 H216 N30 O43 S4.

	Occ.	x	y	z	U_{eq}
H(10A)	1	4512	-1310	1956	50
H(10B)	1	3978	-2191	1761	50
H(12)	1	4413	-961	1321	52
H(13)	1	4519	251	805	53
H(13A)	1	4043	5991	233	99
H(13B)	1	3866	5829	629	99
H(14A)	1	4370	2594	-489	74
H(14B)	1	4341	1330	-321	74
H(15)	1	3206	2705	1215	67
H(16)	1	3077	1479	1721	72
H(20A)	1	4288	-654	2607	50
H(20B)	1	3497	-481	2541	50
H(22)	1	3404	-2119	3592	59
H(23)	1	3531	-1001	4120	57
H(23A)	1	3955	121	5191	84
H(23B)	1	4029	1372	5365	84
H(24A)	1	4360	4771	4652	77
H(24B)	1	4386	4643	4243	77
H(25)	1	4403	1726	3551	56
H(26)	1	4264	593	3020	60
H(30A)	1	3057	-3059	2554	57
H(30B)	1	2816	-2123	2246	57
H(32)	1	1987	-3458	2366	83
H(33)	1	999	-4507	2233	96
H(33A)	1	-644	-6656	2419	171
H(33B)	1	-1133	-7273	2159	171
H(34A)	1	-521	-7908	968	254
H(34B)	1	168	-7502	844	254
H(35)	1	1933	-6331	1441	104
H(36)	1	2940	-5333	1578	96
H(40A)	1	4277	-3528	2641	51
H(40B)	1	4484	-3630	2224	51
H(51A)	1	-2362	-7475	2618	266
H(51B)	1	-2969	-8083	2395	266
H(52A)	1	-3098	-5893	2399	245
H(52B)	1	-2386	-5756	2202	245
H(54A)	1	-2750	-8789	1796	228
H(54B)	1	-2048	-8502	1610	228
H(55A)	1	-2255	-6439	1643	253
H(55B)	1	-2855	-7105	1429	253
H(91A)	1	5852	-4077	3686	38
H(91B)	1	5544	-4829	3353	38
H(91C)	1	5416	-5274	3758	38
H(92A)	1	3700	-4202	3460	56
H(92B)	1	4094	-5360	3617	56
H(92C)	1	4223	-4911	3213	56
H(10C)	1	2617	9482	980	207
H(10D)	1	2066	8905	712	207
H(10E)	1	2819	9092	580	207
H(10F)	1	2507	6334	417	307
H(10G)	1	2761	7685	331	307
H(10H)	1	2012	7467	465	307

Table 4. Anisotropic parameters ($Å^2$ x 10^3) for C136 H216 N30 O43 S4.

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2 [h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
C(1)	51(2)	39(2)	22(2)	1(1)	7(1)	-7(2)
O(1)	69(2)	52(1)	27(1)	12(1)	7(1)	10(1)
O(2)	83(2)	40(1)	21(1)	-1(1)	6(1)	-7(1)
O(3)	56(2)	61(2)	43(1)	-21(1)	11(1)	-16(1)
O(4)	65(2)	32(2)	59(2)	0	-12(2)	0
C(10)	63(2)	37(2)	26(2)	6(1)	2(2)	3(2)
N(10)	92(2)	32(2)	26(1)	0(1)	2(1)	5(2)
C(11)	68(3)	42(2)	20(2)	7(1)	0(2)	-2(2)
N(11)	109(3)	32(2)	29(2)	2(1)	7(2)	3(2)
C(12)	64(2)	37(2)	29(2)	3(1)	-1(2)	5(2)
N(12)	115(3)	30(2)	30(2)	6(1)	6(2)	3(2)
C(13)	64(2)	46(2)	23(2)	0(1)	7(2)	-3(2)
N(13)	176(4)	30(2)	41(2)	5(1)	18(2)	8(2)
C(14)	70(3)	31(2)	28(2)	1(1)	8(2)	-4(2)
N(14)	115(3)	39(2)	30(2)	2(1)	11(2)	-4(2)
C(15)	94(3)	40(2)	34(2)	5(2)	5(2)	20(2)
C(16)	91(3)	59(3)	31(2)	3(2)	15(2)	17(2)
C(17)	78(3)	36(2)	30(2)	-1(2)	-1(2)	3(2)
C(18)	90(3)	36(2)	25(2)	2(2)	0(2)	-2(2)
C(19)	100(3)	39(2)	32(2)	4(2)	2(2)	5(2)
C(20)	66(2)	39(2)	20(2)	3(1)	4(2)	2(2)
N(20)	90(2)	29(2)	27(1)	-2(1)	10(1)	2(2)
C(21)	59(2)	35(2)	27(2)	-5(1)	-3(2)	4(2)
N(21)	83(2)	34(2)	22(1)	-4(1)	6(1)	0(1)
C(22)	82(3)	34(2)	30(2)	4(2)	2(2)	-8(2)
N(22)	101(3)	27(2)	29(2)	-3(1)	8(2)	-2(2)
C(23)	75(3)	44(2)	23(2)	1(2)	5(2)	-3(2)
N(23)	146(3)	39(2)	24(2)	0(1)	6(2)	-11(2)
C(24)	67(2)	33(2)	25(2)	-1(1)	3(2)	4(2)
N(24)	134(3)	29(2)	30(2)	1(1)	11(2)	-5(2)
C(25)	74(3)	34(2)	32(2)	0(2)	9(2)	-5(2)
C(26)	79(3)	48(2)	23(2)	-4(2)	8(2)	-1(2)
C(27)	62(2)	37(2)	32(2)	0(2)	7(2)	7(2)
C(28)	91(3)	40(2)	23(2)	1(2)	2(2)	-3(2)
C(29)	84(3)	43(2)	27(2)	1(2)	5(2)	2(2)
C(30)	59(3)	53(2)	30(2)	-2(2)	10(2)	-7(2)
N(30)	47(3)	106(3)	139(4)	-6(3)	11(3)	-18(2)
C(31)	57(3)	63(2)	49(2)	-2(2)	7(2)	-14(2)
N(31)	71(4)	160(5)	187(6)	-26(4)	-21(4)	-40(3)
C(32)	63(3)	68(3)	77(3)	-20(2)	15(2)	-12(2)
N(32)	94(4)	144(4)	123(4)	-16(3)	-16(3)	-47(3)
C(33)	56(3)	89(3)	96(3)	-23(3)	14(2)	-7(3)
N(33)	57(3)	193(5)	179(5)	-23(4)	9(3)	-36(3)
C(34)	64(3)	81(3)	91(3)	-11(3)	-8(3)	-17(3)
N(34)	174(6)	294(9)	167(6)	-69(6)	-51(5)	-106(6)
C(35)	74(3)	108(4)	78(3)	-39(3)	17(3)	-36(3)
C(36)	72(3)	99(3)	69(3)	-36(3)	13(2)	-30(3)
C(37)	61(4)	97(4)	120(4)	-19(3)	-2(3)	-23(3)
C(38)	56(4)	119(5)	170(7)	-17(4)	-1(4)	-18(3)

C(39)	80(5)	174(6)	162(7)	-33(5)	-11(5)	-42(5)
C(40)	68(3)	29(2)	30(2)	3(1)	1(2)	-9(2)
O(50)	89(4)	182(5)	250(7)	39(5)	-8(4)	-26(3)
C(51)	86(6)	434(19)	146(7)	41(10)	32(5)	22(9)
C(52)	147(8)	251(12)	215(11)	-126(9)	-27(7)	-43(8)
O(53)	207(7)	196(6)	213(7)	-15(5)	1(6)	66(5)
C(54)	116(7)	225(11)	230(10)	-103(9)	-30(7)	-10(6)
C(55)	246(13)	232(11)	156(8)	23(8)	33(8)	96(10)

Table 5. Bond lengths [Å] and angles [°] for C136 H216 N30 O43 S4

C(1)-C(30)	1.518(5)	O(50)-C(51)	1.491(10)
C(1)-C(10)	1.527(4)	C(51)-C(52)	1.520(13)
C(1)-C(20)	1.533(4)	C(52)-O(53)	1.349(9)
C(1)-C(40)	1.536(4)	O(53)-C(55)	1.35(1)
O(1)-C(11)	1.370(3)	C(54)-C(55)	1.442(12)
O(1)-C(10)	1.440(4)	S(90)-O(90)	1.465(7)
O(2)-C(21)	1.359(3)	S(90)-C(91)	1.679(5)
O(2)-C(20)	1.445(3)	S(90)-C(92)	1.719(6)
O(3)-C(31)	1.366(4)	S(93)-C(96)	1.700(13)
O(3)-C(30)	1.432(4)	O(94)-C(95)	1.409(11)
O(4)-C(40)	1.404(3)	S(100)-O(100)	1.649(9)
O(4)-C(40)#1	1.404(3)	S(100)-C(102)	1.784(15)
N(10)-C(17)	1.337(4)	S(100)-C(101)	1.815(14)
N(10)-C(18)	1.352(4)	S(103)-C(106)	1.019(13)
C(11)-C(12)	1.386(5)	S(103)-O(104)	1.544(12)
C(11)-C(16)	1.388(5)	S(103)-C(105)	2.20(2)
N(11)-C(17)	1.329(4)	O(104)-C(106)	1.382(14)
N(11)-C(19)	1.346(4)		
C(12)-C(13)	1.388(4)	C(30)-C(1)-C(10)	111.9(2)
N(12)-C(18)	1.334(4)	C(30)-C(1)-C(20)	108.9(3)
N(12)-C(19)	1.341(4)	C(10)-C(1)-C(20)	108.1(2)
C(13)-C(14)	1.383(4)	C(30)-C(1)-C(40)	111.0(3)
N(13)-C(19)	1.330(4)	C(10)-C(1)-C(40)	107.5(3)
C(14)-C(15)	1.412(5)	C(20)-C(1)-C(40)	109.4(2)
C(14)-C(17)	1.475(4)	C(11)-O(1)-C(10)	117.2(2)
N(14)-C(18)	1.348(4)	C(21)-O(2)-C(20)	117.4(2)
C(15)-C(16)	1.371(5)	C(31)-O(3)-C(30)	115.4(3)
N(20)-C(27)	1.325(4)	C(40)-O(4)-C(40)#1	116.7(3)
N(20)-C(29)	1.349(4)	O(1)-C(10)-C(1)	108.9(3)
C(21)-C(22)	1.370(4)	C(17)-N(10)-C(18)	113.9(3)
C(21)-C(26)	1.387(5)	O(1)-C(11)-C(12)	124.5(3)
N(21)-C(28)	1.343(4)	O(1)-C(11)-C(16)	115.9(3)
N(21)-C(27)	1.344(4)	C(12)-C(11)-C(16)	119.6(3)
C(22)-C(23)	1.371(4)	C(17)-N(11)-C(19)	114.6(3)
N(22)-C(29)	1.341(4)	C(11)-C(12)-C(13)	119.2(3)
N(22)-C(28)	1.345(4)	C(18)-N(12)-C(19)	114.8(3)
C(23)-C(24)	1.398(4)	C(14)-C(13)-C(12)	122.4(3)
N(23)-C(28)	1.344(4)	C(13)-C(14)-C(15)	117.3(3)
C(24)-C(25)	1.390(4)	C(13)-C(14)-C(17)	122.0(3)
C(24)-C(27)	1.479(4)	C(15)-C(14)-C(17)	120.7(3)
N(24)-C(29)	1.337(4)	C(16)-C(15)-C(14)	120.8(3)
C(25)-C(26)	1.386(4)	C(15)-C(16)-C(11)	120.8(3)
N(30)-C(37)	1.293(6)	N(11)-C(17)-N(10)	126.2(3)
N(30)-C(38)	1.353(7)	N(11)-C(17)-C(14)	116.4(3)
C(31)-C(32)	1.365(5)	N(10)-C(17)-C(14)	117.4(3)
C(31)-C(36)	1.401(5)	N(12)-C(18)-N(14)	118.0(3)
N(31)-C(39)	1.337(8)	N(12)-C(18)-N(10)	125.5(3)
N(31)-C(38)	1.366(8)	N(14)-C(18)-N(10)	116.6(3)
C(32)-C(33)	1.376(6)	N(13)-C(19)-N(12)	117.6(3)
N(32)-C(39)	1.356(8)	N(13)-C(19)-N(11)	117.5(3)
N(32)-C(37)	1.364(7)	N(12)-C(19)-N(11)	125.0(3)
C(33)-C(34)	1.381(6)	O(2)-C(20)-C(1)	108.0(2)
N(33)-C(38)	1.315(8)	C(27)-N(20)-C(29)	114.6(3)
C(34)-C(35)	1.362(6)	O(2)-C(21)-C(22)	117.0(3)
C(34)-C(37)	1.488(6)	O(2)-C(21)-C(26)	123.9(3)
N(34)-C(39)	1.339(9)	C(22)-C(21)-C(26)	119.0(3)
C(35)-C(36)	1.381(6)	C(28)-N(21)-C(27)	113.2(3)
O(50)-C(54)	1.350(9)	C(21)-C(22)-C(23)	121.3(3)

C(29)-N(22)-C(28)	114.3(3)	C(35)-C(36)-C(31)	118.6(4)
C(22)-C(23)-C(24)	121.0(3)	N(30)-C(37)-N(32)	126.6(4)
C(25)-C(24)-C(23)	117.4(3)	N(30)-C(37)-C(34)	118.2(5)
C(25)-C(24)-C(27)	119.9(3)	N(32)-C(37)-C(34)	115.1(5)
C(23)-C(24)-C(27)	122.7(3)	N(33)-C(38)-N(30)	118.1(7)
C(26)-C(25)-C(24)	121.4(3)	N(33)-C(38)-N(31)	118.6(6)
C(25)-C(26)-C(21)	120.0(3)	N(30)-C(38)-N(31)	123.4(7)
N(20)-C(27)-N(21)	126.6(3)	N(31)-C(39)-N(34)	117.1(7)
N(20)-C(27)-C(24)	116.4(3)	N(31)-C(39)-N(32)	126.0(7)
N(21)-C(27)-C(24)	117.0(3)	N(34)-C(39)-N(32)	116.9(8)
N(21)-C(28)-N(23)	116.8(3)	O(4)-C(40)-C(1)	106.9(2)
N(21)-C(28)-N(22)	126.1(3)	C(54)-O(50)-C(51)	108.1(6)
N(23)-C(28)-N(22)	117.1(3)	O(50)-C(51)-C(52)	109.6(8)
N(24)-C(29)-N(22)	117.2(3)	O(53)-C(52)-C(51)	101.7(8)
N(24)-C(29)-N(20)	117.9(3)	C(52)-O(53)-C(55)	112.4(9)
N(22)-C(29)-N(20)	124.9(3)	O(50)-C(54)-C(55)	111.3(8)
O(3)-C(30)-C(1)	108.9(3)	O(53)-C(55)-C(54)	110.1(9)
C(37)-N(30)-C(38)	116.3(5)	O(90)-S(90)-C(91)	106.8(3)
C(32)-C(31)-O(3)	125.2(3)	O(90)-S(90)-C(92)	100.5(3)
C(32)-C(31)-C(36)	119.1(4)	C(91)-S(90)-C(92)	105.2(3)
O(3)-C(31)-C(36)	115.7(3)	O(100)-S(100)-C(102)	96.4(6)
C(39)-N(31)-C(38)	114.7(5)	O(100)-S(100)-C(101)	93.8(6)
C(31)-C(32)-C(33)	121.1(4)	C(102)-S(100)-C(101)	73.2(6)
C(39)-N(32)-C(37)	112.9(6)	C(106)-S(103)-O(104)	61.3(8)
C(32)-C(33)-C(34)	120.4(4)	C(106)-S(103)-C(105)	70.8(1)
C(35)-C(34)-C(33)	118.4(4)	O(104)-S(103)-C(105)	76.7(7)
C(35)-C(34)-C(37)	124.5(5)	C(106)-O(104)-S(103)	40.3(6)
C(33)-C(34)-C(37)	117.1(5)	S(103)-C(106)-O(104)	78.4(9)
C(34)-C(35)-C(36)	122.3(4)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1/2

Table 6. Torsion angles [°] for C136 H216 N30 O43 S4.

C(11)-O(1)-C(10)-C(1)	176.1(2)	C(25)-C(24)-C(27)-N(21)	-162.5(3)
C(30)-C(1)-C(10)-O(1)	68.9(3)	C(23)-C(24)-C(27)-N(21)	19.7(5)
C(20)-C(1)-C(10)-O(1)	-51.0(3)	C(27)-N(21)-C(28)-N(23)	177.4(3)
C(40)-C(1)-C(10)-O(1)	-169.0(2)	C(27)-N(21)-C(28)-N(22)	-1.2(5)
C(10)-O(1)-C(11)-C(12)	9.3(4)	C(29)-N(22)-C(28)-N(21)	-3.1(6)
C(10)-O(1)-C(11)-C(16)	-170.4(3)	C(29)-N(22)-C(28)-N(23)	178.4(3)
O(1)-C(11)-C(12)-C(13)	178.7(3)	C(28)-N(22)-C(29)-N(24)	-175.9(3)
C(16)-C(11)-C(12)-C(13)	-1.7(5)	C(28)-N(22)-C(29)-N(20)	5.2(6)
C(11)-C(12)-C(13)-C(14)	1.7(5)	C(27)-N(20)-C(29)-N(24)	178.3(3)
C(12)-C(13)-C(14)-C(15)	-0.8(5)	C(27)-N(20)-C(29)-N(22)	-2.8(5)
C(12)-C(13)-C(14)-C(17)	178.7(3)	C(31)-O(3)-C(30)-C(1)	-174.5(3)
C(13)-C(14)-C(15)-C(16)	-0.2(5)	C(10)-C(1)-C(30)-O(3)	60.7(3)
C(17)-C(14)-C(15)-C(16)	-179.6(3)	C(20)-C(1)-C(30)-O(3)	-179.9(2)
C(14)-C(15)-C(16)-C(11)	0.1(6)	C(40)-C(1)-C(30)-O(3)	-59.4(3)
O(1)-C(11)-C(16)-C(15)	-179.5(3)	C(30)-O(3)-C(31)-C(32)	-6.0(5)
C(12)-C(11)-C(16)-C(15)	0.8(5)	C(30)-O(3)-C(31)-C(36)	174.7(3)
C(19)-N(11)-C(17)-N(10)	-2.8(6)	O(3)-C(31)-C(32)-C(33)	-179.7(4)
C(19)-N(11)-C(17)-C(14)	176.6(3)	C(36)-C(31)-C(32)-C(33)	-0.4(6)
C(18)-N(10)-C(17)-N(11)	0.3(5)	C(31)-C(32)-C(33)-C(34)	-1.1(7)
C(18)-N(10)-C(17)-C(14)	-179.2(3)	C(32)-C(33)-C(34)-C(35)	1.7(7)
C(13)-C(14)-C(17)-N(11)	-149.8(3)	C(32)-C(33)-C(34)-C(37)	-179.8(4)
C(15)-C(14)-C(17)-N(11)	29.7(5)	C(33)-C(34)-C(35)-C(36)	-0.8(8)
C(13)-C(14)-C(17)-N(10)	29.7(5)	C(37)-C(34)-C(35)-C(36)	-179.1(5)
C(15)-C(14)-C(17)-N(10)	-150.8(3)	C(34)-C(35)-C(36)-C(31)	-0.7(8)
C(19)-N(12)-C(18)-N(14)	176.1(4)	C(32)-C(31)-C(36)-C(35)	1.2(6)
C(19)-N(12)-C(18)-N(10)	-3.6(6)	O(3)-C(31)-C(36)-C(35)	-179.4(4)
C(17)-N(10)-C(18)-N(12)	3.2(6)	C(38)-N(30)-C(37)-N(32)	-1.0(9)
C(17)-N(10)-C(18)-N(14)	-176.5(3)	C(38)-N(30)-C(37)-C(34)	178.7(5)
C(18)-N(12)-C(19)-N(13)	-179.7(4)	C(39)-N(32)-C(37)-N(30)	1.0(9)
C(18)-N(12)-C(19)-N(11)	0.6(6)	C(39)-N(32)-C(37)-C(34)	-178.7(5)
C(17)-N(11)-C(19)-N(13)	-177.3(4)	C(35)-C(34)-C(37)-N(30)	153.2(5)
C(17)-N(11)-C(19)-N(12)	2.3(6)	C(33)-C(34)-C(37)-N(30)	-25.1(7)
C(21)-O(2)-C(20)-C(1)	168.8(3)	C(35)-C(34)-C(37)-N(32)	-27.0(7)
C(30)-C(1)-C(20)-O(2)	62.1(3)	C(33)-C(34)-C(37)-N(32)	154.6(5)
C(10)-C(1)-C(20)-O(2)	-176.2(3)	C(37)-N(30)-C(38)-N(33)	178.3(6)
C(40)-C(1)-C(20)-O(2)	-59.4(3)	C(37)-N(30)-C(38)-N(31)	-1.1(9)
C(20)-O(2)-C(21)-C(22)	171.3(3)	C(39)-N(31)-C(38)-N(33)	-176.5(6)
C(20)-O(2)-C(21)-C(26)	-10.0(5)	C(39)-N(31)-C(38)-N(30)	2.9(1)
O(2)-C(21)-C(22)-C(23)	179.2(3)	C(38)-N(31)-C(39)-N(34)	177.6(7)
C(26)-C(21)-C(22)-C(23)	0.4(5)	C(38)-N(31)-C(39)-N(32)	-2.80(11)
C(21)-C(22)-C(23)-C(24)	-0.8(6)	C(37)-N(32)-C(39)-N(31)	1.0(1)
C(22)-C(23)-C(24)-C(25)	0.9(5)	C(37)-N(32)-C(39)-N(34)	-179.4(7)
C(22)-C(23)-C(24)-C(27)	178.8(3)	C(40)#1-O(4)-C(40)-C(1)	-175.3(3)
C(23)-C(24)-C(25)-C(26)	-0.7(5)	C(30)-C(1)-C(40)-O(4)	-168.3(2)
C(27)-C(24)-C(25)-C(26)	-178.6(3)	C(10)-C(1)-C(40)-O(4)	69.1(3)
C(24)-C(25)-C(26)-C(21)	0.3(5)	C(20)-C(1)-C(40)-O(4)	-48.1(3)
O(2)-C(21)-C(26)-C(25)	-178.9(3)	C(54)-O(50)-C(51)-C(52)	-58.6(1)
C(22)-C(21)-C(26)-C(25)	-0.2(5)	O(50)-C(51)-C(52)-O(53)	62.0(1)
C(29)-N(20)-C(27)-N(21)	-2.2(5)	C(51)-C(52)-O(53)-C(55)	-67.20(11)
C(29)-N(20)-C(27)-C(24)	177.3(3)	C(51)-O(50)-C(54)-C(55)	54.20(11)
C(28)-N(21)-C(27)-N(20)	4.1(5)	C(52)-O(53)-C(55)-C(54)	67.10(13)
C(28)-N(21)-C(27)-C(24)	-175.5(3)	O(50)-C(54)-C(55)-O(53)	-58.00(12)
C(25)-C(24)-C(27)-N(20)	17.9(5)	C(105)-S(103)-O(104)-C(10	-75.2(1)
C(23)-C(24)-C(27)-N(20)	-159.9(3)	C(105)-S(103)-C(106)-O(10	485.0(8)

Symmetry transformations used to generate equivalent atoms:

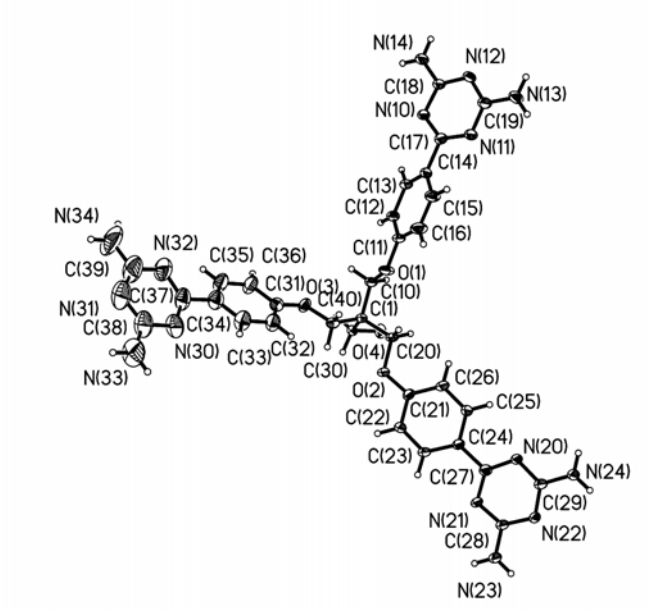
#1 -x+1,y,-z+1/2

Table 7. Bond lengths [Å] and angles [°] related to the hydrogen bonding for C136 H216 N30 O43 S4.

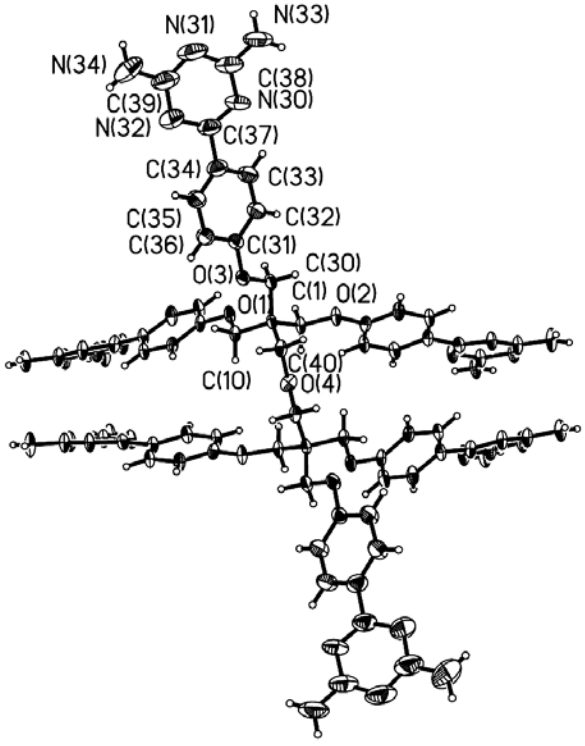
D-H	..A	d(D-H)	d(H..A)	d(D..A)	<DHA
N(13)-H(13A)	N(22)#2	0.87	2.11	2.972(4)	171.6
N(13)-H(13B)	O(104)	0.87	2.15	2.965(10)	156.3
N(14)-H(14A)	O(90)#3	0.87	2.21	3.072(7)	172.5
N(14)-H(14B)	N(21)#3	0.87	2.29	3.125(4)	160.7
N(23)-H(23A)	N(10)#4	0.87	2.21	3.051(4)	162.8
N(23)-H(23B)	O(100)#5	0.87	2.37	3.185(8)	155.1
N(24)-H(24A)	N(12)#5	0.87	2.12	2.966(4)	162.9
N(24)-H(24B)	O(90)#6	0.87	2.16	2.880(6)	139.7
N(33)-H(33A)	N(30)#7	0.87	2.24	3.101(8)	171.4
N(33)-H(33B)	O(50)	0.87	2.08	2.948(7)	173.8

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1/2 #2 x,-y+1,z-1/2 #3 x,-y,z-1/2
#4 x,-y,z+1/2 #5 x,-y+1,z+1/2 #6 x,y+1,z
#7 -x,y,-z+1/2

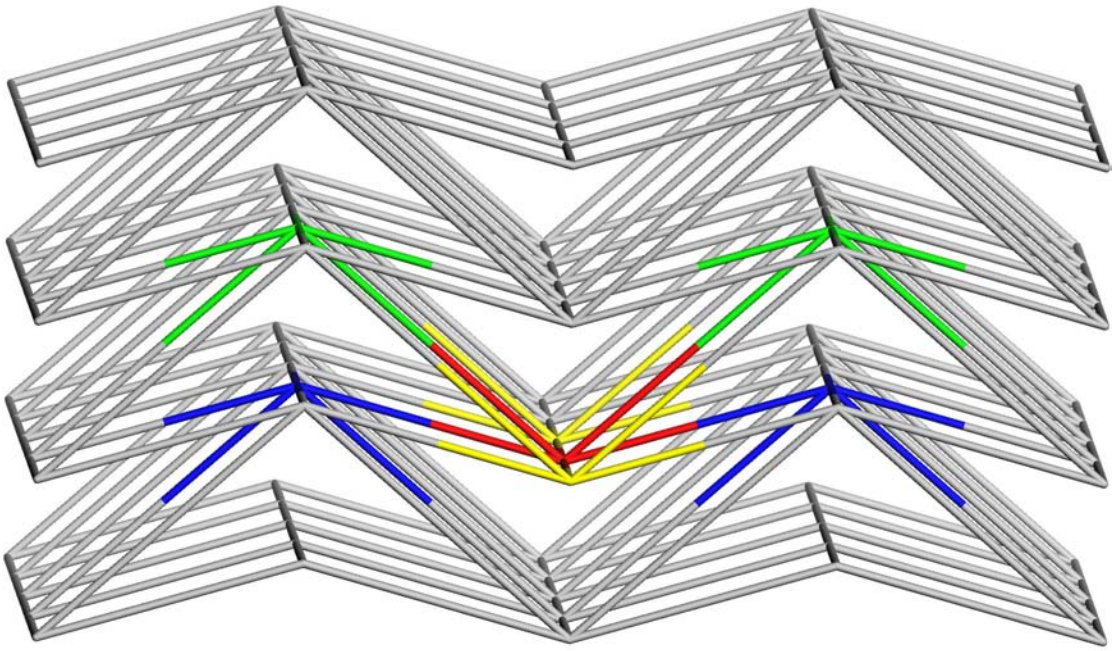


A



B

ORTEP view of the C136 H216 N30 O43 S4 compound with the numbering scheme adopted. **A**: View of the asymmetric unit. **B**: View of one complete molecule. The ellipsoids are drawn at 30% probability level. Hydrogen atoms are represented by sphere of arbitrary size.



Representation of the 6-connected network constructed from the C136 H216 N30 O43 S4 compound, defined by joining the central oxygen atom of each tecton (red) with the centers of the six neighboring tectons (blue, green, and yellow).

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Estimation of porosity

The percentage of volume accessible to guests was estimated using the PLATON program (Spek, A. L. *PLATON, A Multipurpose Crystallographic Tool*; Utrecht University: Utrecht, The Netherlands, 2001. van der Sluis, P.; Spek, A. L. *Acta Crystallogr.* **1990**, *A46*, 194). PLATON calculates the accessible volume by allowing a spherical probe of variable radius to roll over the internal van der Waals surface of the crystal structure. PLATON uses a default value of 1.20 Å for the radius of the probe, which is an appropriate model for small guests such as water. The van der Waals radii used to define surfaces for these calculations are as follows: C: 1.70 Å, H: 1.20 Å, N: 1.55 Å, O: 1.52 Å, and S: 1.80 Å. If V is the volume of the unit cell in $Å^3$ and Vs is the solvent-accessible volume as calculated by PLATON, then the porosity P in % is defined by 100Vs/V.