Supporting Information

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Designing Permeable Molecular Crystals that React with External Agents to Give Crystalline Products**

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Contents

- I. Estimation of porosity (S3)**
- II. Selected experimental procedures (S4-S7)**
- III. Crystal structure of tecton 2 • 6 dioxane (S8-S16)**
- IV. Crystal structure of thioether 5a, as prepared from crystals of tecton 2 by photochemical addition of CH₃SH in the solid state (S17-S25)**
- V. Crystal structure of the cross-linked product obtained from crystals of tecton 2 by photochemical addition of HSCH₂CH₂CH₂SH in the solid state (S26-S34)**

Estimation of porosity

The percentage of volume accessible to guests was estimated by the PLATON program (A. L. Spek, *PLATON, A Multipurpose Crystallographic Tool*; Utrecht University: Utrecht, The Netherlands, 2001. P. van der Sluis, A. L. Spek, *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 and V_g is the guest-accessible volume as calculated by PLATON, then the porosity P in % is given by $100V_g/V$.

Selected experimental procedures

Tetrakis[4-[(2,4-dichloro-1,3,5-triazin-6-yl)amino]phenyl]methane (4). Tetrakis(4-aminophenyl)methane (**3**) was prepared by a published procedure (F. A. Neugebauer, H. Fischer, R. Bernhardt, *Chem. Ber.* **1976**, *109*, 2389), modified by using THF instead of ethyl acetate in the reduction of tetrakis(4-nitrophenyl)methane. A solution of tetrakis(4-aminophenyl)methane (**3**; 0.761 g, 2.00 mmol) in acetone (16 mL) was added dropwise at 0 °C to a stirred solution of cyanuric chloride (1.51 g, 8.19 mmol) in acetone (24 mL). The resulting mixture was kept at 0 °C for 1 hour, treated with Na₂CO₃ (0.869 g, 8.20 mmol), and poured into water (160 mL). The precipitate was separated by filtration, washed, and dried to give tetrakis[4-[(2,4-dichloro-1,3,5-triazin-6-yl)amino]phenyl]methane (**4**; 1.48 g, 1.52 mmol, 76%) as a colorless solid: mp >310 °C; ¹H NMR (400 MHz, DMSO-*d*₆, 25 °C) δ 7.18 (d, 8H, ³*J* = 8.7 Hz), 7.55 (d, 8H, ³*J* = 8.7 Hz), 11.18 (s, 4H); ¹³C NMR (75.4 MHz, DMSO-*d*₆, 25 °C) δ 64.1, 121.7, 131.7, 135.8, 143.8, 164.6, 169.7, 170.7; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 969. Anal. Calcd for C₃₇H₂₀Cl₈N₁₆: C, 45.71; H, 2.07; N, 23.05. Found: C, 46.03; H, 2.09; N, 22.61.

Tetrakis[4-[[2,4-bis(allylamino)-1,3,5-triazin-6-yl]amino]phenyl]methane (2). A solution containing tetrakis[4-[(2,4-dichloro-1,3,5-triazin-6-yl)amino]phenyl]methane (**4**; 0.226 g, 0.232 mmol), allylamine (0.35 mL, 4.7 mmol), and *N,N*-diisopropylethylamine (0.40 mL, 2.3 mmol) in dioxane (6 mL) was sealed in a glass tube, which was then heated at 110 °C for 6 h. Addition of the contents to cold water caused the precipitation of a solid, which was separated by filtration and washed with water and ether to give tetrakis[4-[[2,4-bis(allylamino)-1,3,5-triazin-6-

yl]amino]phenyl]methane (**2**; 0.228 g, 0.200 mmol, 86%) as a light yellow solid. To effect crystallization, the compound (0.300 g, 0.264 mmol) was first dissolved in hot dioxane (3 mL). The hot solution was then divided into several portions, which were placed in closed 4 mL vials and allowed to cool slowly to room temperature in a pre-warmed insulated oil bath. Crystals were obtained within several days: mp 175 °C (dec); ¹H NMR (300 MHz, DMSO-*d*₆, 150 °C) δ 3.92 (s, 16H), 5.03 (d, 8H, ³*J* = 10.3 Hz), 5.16 (d, 8H, ³*J* = 17.2 Hz), 5.91 (m, 8H), 6.22 (s, 8H), 7.02 (d, 8H, ³*J* = 8.7 Hz), 7.63 (d, 8H, ³*J* = 8.7 Hz), 8.16 (4H); ¹³C NMR (75.4 MHz, DMSO-*d*₆, 150 °C) δ 43.0, 63.2, 115.1, 119.1, 130.8, 136.5, 138.5, 140.9, 164.6, 166.2; MS (FAB, 3-nitrobenzyl alcohol) *m/e* 1137. Anal. Calcd for C₆₁H₆₈N₂₄ + 1 H₂O: C, 63.41; H, 6.11; N, 29.10. Found: C, 63.34; H, 6.07; N, 28.92.

Tetrakis[4-[[2,4-bis[3-(methylthio)propanamino]-1,3,5-triazin-6-yl]amino]phenyl]methane (5a) (Synthesis in solution). A solution containing tetrakis[4-[[2,4-dichloro-1,3,5-triazin-6-yl]amino]phenyl]methane (**2**; 0.320 g, 0.329 mmol), 3-(methylthio)propanamine (0.312 g, 2.97 mmol), and *N,N*-diisopropylethylamine (0.52 mL, 3.0 mmol) in dioxane (5 mL) was sealed in a glass tube, which was then heated at 116 °C for 23 h. Addition of the contents to cold water caused the precipitation of a solid, which was separated by filtration and washed with water and ether to give tetrakis[4-[[2,4-bis[3-(methylthio)propanamino]-1,3,5-triazin-6-yl]amino]phenyl]methane (**5a**; 0.466 g, 0.306 mmol, 93%) as a colorless solid. Crystallization was effected by allowing methanol (1 mL) to diffuse into a solution of the crude product (0.010 mg, 0.0066 mmol) in THF (0.5 mL): ¹H NMR (300 MHz, DMSO-*d*₆, 150 °C) δ 1.81 (m, 16H), 2.02 (s, 24H), 2.50 (t, 16H, ³*J* = 7.1 Hz), 3.35 (t, 16H, ³*J* = 6.7 Hz), 6.10 (bs, 8H), 7.01 (d, 8H, ³*J* = 8.9 Hz), 7.62 (d, 8H, ³*J* = 8.9 Hz), 8.05 (bs, 4H); ¹³C NMR (75.4 MHz, DMSO-*d*₆, 150 °C) δ

14.1, 28.7, 30.7, 38.9, 62.0, 117.9, 129.7, 137.5, 139.8, 163.6, 165.4; MS (FAB, 3-nitrobenzyl alcohol) m/e 1521.

Exchange of dioxane in single crystals of tecton 2 • 6 dioxane. Crystals of tecton 2 • 6 dioxane with approximate dimensions 0.5 mm x 0.5 mm x 0.5 mm were grown in the normal way. The mother liquors were removed by pipette, and the crystals were immediately covered with toluene and kept at 25 °C for 24 h without stirring. The liquid phase was removed by pipette, then the crystals were washed with pentane and dried briefly in air. Analysis by ^1H NMR spectroscopy of dissolved samples showed complete replacement of dioxane by toluene.

Reaction of CH_3SH with single crystals of tecton 2. Dioxane in single crystals of tecton 2 • 6 dioxane (10 mg, 0.0059 mmol) was replaced with toluene in the normal way. The exchanged crystals, covered with toluene (5 mL), were placed in a jacketed quartz reactor, and He was bubbled through the stirred mixture at 25 °C. After 1 h, CH_3SH was added to the stream of He passing through the reactor, and after an additional 45 minutes, irradiation of the reactor was begun, using a medium-pressure 450-watt Hg lamp held at a distance of 10 cm. Irradiation was continued for 40 h with a constant current of $\text{CH}_3\text{SH}/\text{He}$. Analysis of the recovered crystals by mass spectrometry and by ^1H NMR spectroscopy of dissolved samples showed that an average of 70-85% of the allyl groups had reacted to give tetrakis[4-[[2,4-bis[3-(methylthio)propanamino]-1,3,5-triazin-6-yl]amino]phenyl]methane (**5a**), which was identified by comparison with authentic samples.

Cross-linking single crystals of tecton 2 with ethanedithiol. Single crystals of tecton **2** • 6 dioxane (10 mg, 0.0059 mmol) were placed in a jacketed quartz reactor and exposed briefly to neat ethanedithiol (5 mL), which was then replaced with a second portion (10 mL). He was bubbled through the stirred mixture at 25 °C for 1 h, and then the reactor was irradiated for 24 h using a medium-pressure 450-watt Hg lamp held at a distance of 10 cm. The recovered crystals were analyzed by optical microscopy, thermogravimetry, Raman spectroscopy, solid-state ¹³C NMR spectroscopy, and X-ray diffraction. Anal. Calcd for C₆₉H₁₀₀N₂₄S₈ + 6 HSCH₂CH₂SH: C, 51.50; H, 6.16; N, 19.75. Found: C, 50.83; H, 6.60; N, 19.17.

CRYSTAL AND MOLECULAR STRUCTURE OF
C85 H116 N24 O12 COMPOUND (JIW233)

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Table 1. Crystal data and structure refinement for C₈₅ H₁₁₆ N₂₄ O₁₂.

Identification code	JIW233
Empirical formula	C ₈₅ H ₁₁₆ N ₂₄ O ₁₂
Formula weight	1666.018
Temperature	173(2)K
Wavelength	0.71073 Å
Crystal system	Tetragonal
Space group	I4 ₁ /a
Unit cell dimensions	a = 23.0674(6) Å α = 90° b = 23.0674(6) Å β = 90° c = 16.1892(6) Å γ = 90°
Volume	8614.4(4) Å ³
Z	4
Density (calculated)	1.2846 Mg/m ³
Absorption coefficient	0.089 mm ⁻¹
F(000)	3560.0
Crystal size	0.15 x 0.20 x 0.30 mm
Theta range for data collection	1.54 to 20.85°
Index ranges	-23 ≤ h ≤ 23, -23 ≤ k ≤ 23, -16 ≤ l ≤ 16
Reflections collected	25463
Independent reflections	2269 [R _{int} = 0.0478]
Absorption correction	Multi-scan
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2269 / 0 / 273
Goodness-of-fit on F ²	1.071
Final R indices [I > 2σ(I)]	R ₁ = 0.0795, wR ₂ = 0.2308
R indices (all data)	R ₁ = 0.0974, wR ₂ = 0.2496
Largest diff. peak and hole	0.536 and -0.415 e/Å ³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C85 H116 N24 O12.

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

	Occ.	x	y	z	U_{eq}
O(1)	1	6015(3)	1955(2)	203(4)	98(2)
O(2)	1	5153(3)	1127(2)	585(3)	99(2)
O(3)	1	9534(2)	5344(2)	295(2)	54(1)
N(1)	1	6209(2)	4001(2)	1460(2)	32(1)
N(2)	1	6550(2)	3354(2)	471(2)	33(1)
N(3)	1	7241(2)	3713(2)	-506(2)	31(1)
N(4)	1	6835(2)	4350(2)	502(2)	32(1)
N(5)	1	6942(2)	2757(2)	-502(2)	40(1)
N(6)	1	7470(2)	4673(2)	-500(3)	39(1)
C(1)	1	5000	2500	3750	23(2)
C(2)	1	5333(2)	2920(2)	3169(3)	25(1)
C(3)	1	5677(2)	2683(2)	2550(3)	31(1)
C(4)	1	5964(2)	3019(2)	1973(3)	32(1)
C(5)	1	5914(2)	3617(2)	2002(3)	28(1)
C(6)	1	5578(2)	3861(2)	2614(3)	33(1)
C(7)	1	5287(2)	3517(2)	3188(3)	30(1)
C(8)	1	6541(2)	3882(2)	788(3)	28(1)
C(9)	1	6916(2)	3294(2)	-180(3)	31(1)
C(10)	1	7169(2)	4238(2)	-152(3)	31(1)
C(11)	1	7241(3)	2618(2)	-1257(4)	51(2)
C(12)	1	7227(4)	1999(4)	-1473(7)	115(3)
C(13)	1	7115(5)	1567(4)	-1054(7)	127(3)
C(14)	1	7437(3)	5272(2)	-249(4)	50(2)
C(15)	1	7945(4)	5597(3)	-448(6)	105(3)
C(16)	1	8108(4)	6003(3)	-839(7)	121(4)
C(20)	1	6106(4)	1485(4)	771(5)	95(3)
C(21)	1	5564(5)	1292(5)	1172(5)	123(4)
C(22)	1	5039(4)	1612(4)	91(7)	110(3)
C(23)	1	5549(3)	1802(4)	-365(5)	95(3)
C(30)	1	9508(3)	5064(3)	-477(4)	62(2)
C(31)	1	9896(3)	5024(3)	840(4)	64(2)

Table 3. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C85 H116 N24 O12.

	x	y	z	U _{eq}
H(1)	6172	4372	1577	38
H(5)	6766	2475	-236	48
H(6)	7704	4586	-911	47
H(3)	5717	2274	2523	38
H(4)	6194	2841	1559	38
H(6A)	5544	4271	2645	39
H(7)	5054	3696	3599	36
H(11A)	7650	2742	-1205	61
H(11B)	7065	2843	-1714	61
H(12)	7322	1918	-2032	137
H(13A)	7016	1608	-488	152
H(13B)	7127	1193	-1298	152
H(14A)	7099	5455	-521	60
H(14B)	7373	5290	355	60
H(15)	8267	5431	-170	126
H(16A)	7840	6222	-1158	145
H(16B)	8506	6108	-832	145
H(20A)	6384	1609	1202	114
H(20B)	6279	1154	471	114
H(21A)	5648	960	1541	148
H(21B)	5406	1611	1514	148
H(22A)	4901	1933	445	132
H(22B)	4727	1516	-305	132
H(23A)	5448	2142	-709	115
H(23B)	5680	1487	-737	115
H(30A)	9272	5300	-862	74
H(30B)	9316	4683	-412	74
H(31A)	9717	4641	952	76
H(31B)	9930	5233	1371	76

Table 4. Anisotropic parameters ($\text{\AA}^2 \times 10^3$) for C85 H116 N24 O12.

The anisotropic displacement factor exponent takes the form:

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

	U11	U22	U33	U23	U13	U12
O(1)	116(4)	62(3)	116(4)	3(3)	14(4)	-30(3)
O(2)	115(4)	83(4)	99(4)	-14(3)	22(3)	-35(3)
O(3)	57(3)	46(2)	59(3)	-6(2)	0(2)	4(2)
N(1)	41(2)	24(2)	31(2)	1(2)	11(2)	-1(2)
N(2)	40(2)	32(3)	27(2)	-2(2)	9(2)	-4(2)
N(3)	32(2)	33(2)	29(2)	3(2)	7(2)	-2(2)
N(4)	37(2)	30(2)	28(2)	0(2)	9(2)	-4(2)
N(5)	57(3)	29(3)	35(2)	-7(2)	19(2)	-5(2)
N(6)	47(3)	29(2)	42(3)	2(2)	19(2)	-3(2)
C(1)	24(3)	24(3)	21(5)	0	0	0
C(2)	24(2)	28(3)	23(3)	0(2)	-3(2)	0(2)
C(3)	40(3)	24(3)	30(3)	0(2)	4(2)	3(2)
C(4)	38(3)	30(3)	27(3)	0(2)	11(2)	1(2)
C(5)	27(3)	35(3)	23(3)	1(2)	3(2)	-2(2)
C(6)	43(3)	21(3)	35(3)	-1(2)	5(2)	0(2)
C(7)	32(3)	32(3)	26(3)	-1(2)	5(2)	1(2)
C(8)	32(3)	31(3)	22(3)	0(2)	2(2)	1(2)
C(9)	33(3)	37(3)	23(3)	2(2)	2(2)	-1(2)
C(10)	29(3)	33(3)	30(3)	2(2)	2(2)	1(2)
C(11)	56(4)	38(3)	57(4)	-13(3)	16(3)	-2(3)
C(12)	132(8)	70(6)	142(8)	16(6)	81(7)	-6(5)
C(13)	148(9)	81(6)	151(9)	-5(7)	-9(7)	14(6)
C(14)	57(4)	36(3)	56(4)	4(3)	25(3)	-8(3)
C(15)	107(6)	49(5)	158(8)	-24(5)	56(6)	-17(4)
C(16)	108(7)	46(5)	207(10)	13(6)	81(7)	-8(4)
C(20)	95(6)	98(6)	92(6)	5(5)	-20(5)	-32(5)
C(21)	151(9)	145(8)	74(6)	-31(5)	11(6)	-81(7)
C(22)	93(6)	69(5)	167(9)	25(6)	27(6)	-9(5)
C(23)	83(6)	87(6)	116(7)	29(5)	-13(5)	-8(4)
C(30)	55(4)	73(4)	58(4)	-5(3)	-9(3)	7(3)
C(31)	61(4)	83(5)	47(4)	1(3)	-4(3)	6(3)

Table 5. Bond lengths [Å] and angles [°] for C85 H116 N24 O12

O(1)-C(20)	1.438(10)	C(8)-N(2)-C(9)	113.8(4)
O(1)-C(23)	1.459(10)	C(9)-N(3)-C(10)	114.5(4)
O(2)-C(21)	1.396(10)	C(10)-N(4)-C(8)	114.0(4)
O(2)-C(22)	1.398(10)	C(9)-N(5)-C(11)	123.7(4)
O(3)-C(30)	1.408(7)	C(10)-N(6)-C(14)	124.9(4)
O(3)-C(31)	1.422(7)	C(2)#1-C(1)-C(2)	111.50(16)
N(1)-C(8)	1.358(6)	C(2)#1-C(1)-C(2)#2	105.5(3)
N(1)-C(5)	1.422(6)	C(2)-C(1)-C(2)#2	111.50(16)
N(2)-C(8)	1.323(6)	C(2)#1-C(1)-C(2)#3	111.50(16)
N(2)-C(9)	1.356(6)	C(2)-C(1)-C(2)#3	105.5(3)
N(3)-C(9)	1.333(6)	C(2)#2-C(1)-C(2)#3	111.49(16)
N(3)-C(10)	1.349(6)	C(7)-C(2)-C(3)	116.8(4)
N(4)-C(10)	1.335(6)	C(7)-C(2)-C(1)	124.8(4)
N(4)-C(8)	1.356(6)	C(3)-C(2)-C(1)	118.3(3)
N(5)-C(9)	1.345(6)	C(4)-C(3)-C(2)	122.7(4)
N(5)-C(11)	1.440(6)	C(3)-C(4)-C(5)	119.7(4)
N(6)-C(10)	1.342(6)	C(6)-C(5)-C(4)	118.6(4)
N(6)-C(14)	1.443(6)	C(6)-C(5)-N(1)	117.4(4)
C(1)-C(2)#1	1.554(4)	C(4)-C(5)-N(1)	124.0(4)
C(1)-C(2)	1.554(4)	C(5)-C(6)-C(7)	121.2(4)
C(1)-C(2)#2	1.554(4)	C(2)-C(7)-C(6)	121.0(4)
C(1)-C(2)#3	1.554(4)	N(2)-C(8)-N(4)	126.3(4)
C(2)-C(7)	1.382(6)	N(2)-C(8)-N(1)	120.4(4)
C(2)-C(3)	1.390(6)	N(4)-C(8)-N(1)	113.3(4)
C(3)-C(4)	1.382(6)	N(3)-C(9)-N(5)	119.3(4)
C(4)-C(5)	1.385(6)	N(3)-C(9)-N(2)	125.7(4)
C(5)-C(6)	1.377(6)	N(5)-C(9)-N(2)	115.0(4)
C(6)-C(7)	1.395(6)	N(4)-C(10)-N(6)	119.1(4)
C(11)-C(12)	1.470(10)	N(4)-C(10)-N(3)	125.5(4)
C(12)-C(13)	1.233(11)	N(6)-C(10)-N(3)	115.5(4)
C(14)-C(15)	1.427(10)	N(5)-C(11)-C(12)	114.1(5)
C(15)-C(16)	1.191(10)	C(13)-C(12)-C(11)	131.2(10)
C(20)-C(21)	1.477(11)	C(15)-C(14)-N(6)	113.4(5)
C(22)-C(23)	1.455(11)	C(16)-C(15)-C(14)	142.4(11)
C(30)-C(31)#4	1.508(8)	O(1)-C(20)-C(21)	112.7(8)
C(31)-C(30)#4	1.508(8)	O(2)-C(21)-C(20)	110.9(6)
		O(2)-C(22)-C(23)	112.3(7)
C(20)-O(1)-C(23)	109.1(5)	C(22)-C(23)-O(1)	110.4(7)
C(21)-O(2)-C(22)	107.4(7)	O(3)-C(30)-C(31)#4	111.6(5)
C(30)-O(3)-C(31)	109.8(4)	O(3)-C(31)-C(30)#4	111.3(5)
C(8)-N(1)-C(5)	129.8(4)		

Symmetry transformations used to generate equivalent atoms:

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

Table 6. Hydrogen bond lengths [$\text{\AA}$] and angles [$^\circ$] for C85 H116 N24 O12

D	H	A	D - H	H...A	D...A	<DHA
N1	H1	N3#1	0.8800	2.1573	3.0278	170.02
N5	H5	O1#2	0.8800	2.2228	3.0488	156.23
N6	H6	N4#3	0.8800	2.1204	2.9929	171.11

#1 $1/4+y, 1/4-x, 1/4+z$
#2 $1/2-x, 1/2-y, 1/2-z$
#3 $1/4-y, x-1/4, z-1/4$

ORTEP view of the C₈₅ H₁₁₆ N₂₄ O₁₂ compound (asymmetric unit) with the numbering scheme adopted. Ellipsoids drawn at 30% probability level. Hydrogens represented by sphere of arbitrary size.

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CRYSTAL AND MOLECULAR STRUCTURE OF
C64.2 H80.8 N24 S3.2 COMPOUND (JIW301)

Montag, Juli 28, 2003

Equipe WUEST

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Structure solved and refined by Dr. Gary Enright, Steacie Institute for Molecular Sciences, National Research Council, Ottawa, Ontario and by Dr. Thierry Maris, Laboratory of X-Ray Diffraction, Université de Montréal, Québec.

Table 1. Crystal data and structure refinement for C_{64.2} H_{80.8} N₂₄ S_{3.2}

Identification code	JIW301
Empirical formula	C _{64.2} H _{80.8} N ₂₄ S _{3.2}
Formula weight	1522.21
Temperature	173(2)K
Wavelength	0.71073 Å
Crystal system	Tetragonal
Space group	I4 ₁ /a
Unit cell dimensions	a = 20.988(2) Å $\alpha = 90^\circ$ b = 20.988(2) Å $\beta = 90^\circ$ c = 16.816(3) Å $\gamma = 90^\circ$
Volume	7407.6(16) Å ³
Z	4
Density (calculated)	1.365 Mg/m ³
Absorption coefficient	0.301 mm ⁻¹
F(000)	3240
Crystal size	0.20 x 0.15 x 0.08 mm
Theta range for data collection	1.55 to 20.99°
Index ranges	-21 ≤ h ≤ 21, -21 ≤ k ≤ 21, -16 ≤ l ≤ 16
Reflections collected	17939
Independent reflections	1976 [R _{int} = 0.2328]
Max. and min. transmission	0.9763 and 0.9422
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	1976 / 45 / 214
Goodness-of-fit on F ²	1.306
Final R indices [I > 2σ(I)]	R ₁ = 0.1210, wR ₂ = 0.3894
R indices (all data)	R ₁ = 0.2324, wR ₂ = 0.3894
Largest diff. peak and hole	0.620 and -0.570 e/Å ³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C64.2 H80.8 N24 S3.2.

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

	Occ.	x	y	z	U_{eq}
C(1)	1	0	7500	-1250	53(2)
C(2)	1	433(1)	7137(1)	-669(2)	48(1)
C(3)	1	180(1)	6732(2)	-119(2)	57(1)
C(4)	1	527(1)	6439(1)	456(2)	55(1)
C(5)	1	1180(1)	6555(1)	512(2)	54(1)
C(6)	1	1454(2)	6957(2)	-34(2)	67(1)
C(7)	1	1076(2)	7245(2)	-619(2)	62(1)
N(8)	1	1571(1)	6294(1)	1107(1)	60(1)
C(9)	1	1411(2)	6006(1)	1804(2)	54(1)
N(10)	1	808(1)	6006(1)	2049(1)	55(1)
C(11)	1	729(1)	5707(1)	2752(2)	56(1)
N(12)	1	1176(1)	5439(1)	3196(1)	57(1)
C(13)	1	1767(2)	5502(2)	2889(2)	61(1)
N(14)	1	1927(1)	5763(1)	2200(2)	61(1)
N(20)	1	135(1)	5688(1)	3015(2)	78(1)
C(21)	1	-65(2)	5438(2)	3760(2)	110(2)
C(22)	1	-223(3)	5927(2)	4369(3)	186(1)
C(23)	1	121(2)	6542(2)	4448(4)	240(1)
S(24)	0.3881(14)	803(1)	6317(1)	4989(1)	81(1)
C(25)	0.3881(14)	550(7)	6305(8)	5998(4)	215(1)
N(30)	1	2226(1)	5240(2)	3330(2)	86(1)
C(31)	1	2893(2)	5234(2)	3133(3)	131(2)
C(32)	1	3167(2)	4671(2)	3612(4)	186(1)
C(33)	1	3847(1)	4523(2)	3410(2)	240(1)
S(34)	0.3881(14)	4334(1)	5162(2)	3782(2)	119(1)
C(35)	0.3881(14)	3970(1)	5730(2)	4441(2)	215(1)
C(352)	0.893(7)	598(4)	6548(3)	5665(5)	191(3)
C(252)	0.893(7)	4127(4)	5222(4)	4763(6)	238(4)

Table 3. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C64.2 H80.8 N24 S3.2.

	Occ.	x	y	z	U _{eq}
H(3)	1	-265	6649	-141	68
H(4)	1	325	6157	819	66
H(6)	1	1899	7040	-15	81
H(7)	1	1271	7524	-991	75
H(8)	1	1982	6320	1014	72
H(20)	1	-162	5845	2702	94
H(21A)	1	277	5161	3971	132
H(21B)	1	-445	5167	3672	132
H(22A)	1	-678	6034	4293	223
H(22B)	0.3881(14)	-191	5712	4892	223
H(23C)	1	-136	6859	4743	288
H(23D)	1	236	6718	3921	288
H(25D)	0.3881(14)	905	6171	6338	322
H(25E)	0.3881(14)	195	6005	6057	322
H(25F)	0.3881(14)	410	6733	6154	322
H(30)	1	2111	5056	3777	103
H(31C)	1	3099	5639	3290	158
H(31D)	1	2956	5168	2555	158
H(32C)	1	3137	4771	4186	223
H(32D)	0.3881(14)	2903	4288	3511	223
H(33C)	1	3898	4485	2826	288
H(33D)	1	3976	4114	3657	288
H(35D)	0.3881(14)	4286	6050	4598	322
H(35E)	0.3881(14)	3811	5510	4916	322
H(35F)	0.3881(14)	3614	5940	4169	322

Table 4. Anisotropic parameters ($\text{\AA}^2 \times 10^3$) for C64.2 H80.8 N24 S3.2.

The anisotropic displacement factor exponent takes the form:

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

	U11	U22	U33	U23	U13	U12
C(1)	60(2)	60(2)	39(4)	0	0	0
C(2)	48(2)	60(2)	37(2)	-4(2)	-15(2)	1(2)
C(3)	51(2)	88(2)	31(2)	18(2)	-4(2)	-23(2)
C(4)	71(2)	67(2)	26(2)	4(2)	-7(2)	-24(2)
C(5)	53(2)	69(2)	42(2)	5(2)	3(2)	0(2)
C(6)	53(2)	92(2)	56(2)	20(2)	1(2)	-8(2)
C(7)	76(2)	75(2)	36(2)	16(2)	4(2)	8(2)
N(8)	47(2)	101(2)	33(1)	25(1)	-1(1)	1(2)
C(9)	77(2)	48(2)	38(2)	-4(2)	-2(2)	-4(2)
N(10)	56(2)	63(2)	47(2)	7(1)	1(1)	2(1)
C(11)	66(2)	54(2)	48(2)	3(2)	6(2)	3(2)
N(12)	68(2)	56(2)	48(2)	11(1)	-3(2)	1(1)
C(13)	68(2)	64(2)	50(2)	5(2)	-12(2)	2(2)
N(14)	59(2)	85(2)	39(2)	19(1)	2(1)	-4(2)
N(20)	83(2)	103(2)	49(2)	29(2)	21(2)	8(2)
C(21)	125(3)	133(4)	71(3)	10(3)	24(3)	24(3)
C(22)	184(2)	187(2)	187(2)	1(1)	12(1)	5(1)
C(23)	238(2)	241(2)	240(2)	-2(2)	1(2)	1(2)
S(24)	75(2)	116(2)	53(2)	4(2)	15(1)	2(2)
C(25)	215(2)	214(2)	215(2)	0(2)	-1(2)	-1(2)
N(30)	67(2)	123(2)	69(2)	27(2)	-15(2)	2(2)
C(31)	104(3)	175(4)	116(3)	75(3)	-19(3)	4(3)
C(32)	184(2)	187(2)	187(2)	1(1)	12(1)	5(1)
C(33)	238(2)	241(2)	240(2)	-2(2)	1(2)	1(2)
S(34)	101(2)	115(2)	140(3)	29(2)	-1(2)	-16(2)
C(35)	215(2)	214(2)	215(2)	0(2)	-1(2)	-1(2)

Table 5. Bond lengths [Å] and angles [°] for C64.2 H80.8 N24 S3.2

C(1)-C(2)#1	1.538(3)	C(2)-C(1)-C(2)#3	113.85(11)
C(1)-C(2)	1.538(3)	C(2)#2-C(1)-C(2)#3	113.85(11)
C(1)-C(2)#2	1.538(3)	C(3)-C(2)-C(7)	116.5(3)
C(1)-C(2)#3	1.538(3)	C(3)-C(2)-C(1)	120.6(2)
C(2)-C(3)	1.363(4)	C(7)-C(2)-C(1)	122.6(2)
C(2)-C(7)	1.371(4)	C(4)-C(3)-C(2)	123.7(3)
C(3)-C(4)	1.358(4)	C(3)-C(4)-C(5)	119.8(3)
C(4)-C(5)	1.396(4)	C(6)-C(5)-C(4)	118.2(3)
C(5)-C(6)	1.374(4)	C(6)-C(5)-N(8)	118.1(3)
C(5)-N(8)	1.405(4)	C(4)-C(5)-N(8)	123.6(3)
C(6)-C(7)	1.400(4)	C(5)-C(6)-C(7)	119.8(3)
N(8)-C(9)	1.360(4)	C(2)-C(7)-C(6)	121.9(3)
C(9)-N(10)	1.332(4)	C(9)-N(8)-C(5)	130.1(2)
C(9)-N(14)	1.369(4)	N(10)-C(9)-N(8)	120.0(3)
N(10)-C(11)	1.348(4)	N(10)-C(9)-N(14)	126.9(3)
C(11)-N(20)	1.324(4)	N(8)-C(9)-N(14)	113.0(3)
C(11)-N(12)	1.323(4)	C(9)-N(10)-C(11)	112.8(2)
N(12)-C(13)	1.350(4)	N(20)-C(11)-N(12)	117.7(3)
C(13)-N(14)	1.325(4)	N(20)-C(11)-N(10)	115.1(3)
C(13)-N(30)	1.334(4)	N(12)-C(11)-N(10)	127.2(3)
N(20)-C(21)	1.421(4)	C(11)-N(12)-C(13)	113.2(3)
C(21)-C(22)	1.487(6)	N(14)-C(13)-N(30)	118.2(3)
C(22)-C(23)	1.484(7)	N(14)-C(13)-N(12)	127.3(3)
C(23)-S(24)	1.761(6)	N(30)-C(13)-N(12)	114.3(3)
S(24)-C(25)	1.778(7)	C(13)-N(14)-C(9)	112.3(3)
N(30)-C(31)	1.439(4)	C(11)-N(20)-C(21)	125.8(3)
C(31)-C(32)	1.541(6)	N(20)-C(21)-C(22)	114.6(3)
C(32)-C(33)	1.502(5)	C(23)-C(22)-C(21)	123.7(5)
C(33)-S(34)	1.7980	C(22)-C(23)-S(24)	102.0(4)
S(34)-C(35)	1.7990	C(23)-S(24)-C(25)	104.7(5)
		C(13)-N(30)-C(31)	125.4(3)
C(2)#1-C(1)-C(2)	113.85(11)	N(30)-C(31)-C(32)	104.4(3)
C(2)#1-C(1)-C(2)#2	113.85(11)	C(33)-C(32)-C(31)	113.3(4)
C(2)-C(1)-C(2)#2	101.0(2)	C(32)-C(33)-S(34)	107.9(2)
C(2)#1-C(1)-C(2)#3	101.0(2)	C(33)-S(34)-C(35)	117.9

Symmetry transformations used to generate equivalent atoms:

#1 $y-3/4, -x+3/4, -z-1/4$ #2 $-x, -y+3/2, z$ #3 $-y+3/4, x+3/4, -z-1/4$

Table 6. Torsion angles [$^{\circ}$] for C64.2 H80.8 N24 S3.2

C(2)#1-C(1)-C(2)-C(3)	-64.62(18)	C(9)-N(10)-C(11)-N(20)	-179.2(3)
C(2)#2-C(1)-C(2)-C(3)	57.8(2)	C(9)-N(10)-C(11)-N(12)	1.1(4)
C(2)#3-C(1)-C(2)-C(3)	-179.7(3)	N(20)-C(11)-N(12)-C(13)	-177.9(3)
C(2)#1-C(1)-C(2)-C(7)	121.9(3)	N(10)-C(11)-N(12)-C(13)	1.8(4)
C(2)#2-C(1)-C(2)-C(7)	-115.6(3)	C(11)-N(12)-C(13)-N(14)	-3.8(5)
C(2)#3-C(1)-C(2)-C(7)	6.8(3)	C(11)-N(12)-C(13)-N(30)	179.5(3)
C(7)-C(2)-C(3)-C(4)	-0.1(5)	N(30)-C(13)-N(14)-C(9)	179.1(3)
C(1)-C(2)-C(3)-C(4)	-174.0(2)	N(12)-C(13)-N(14)-C(9)	2.6(4)
C(2)-C(3)-C(4)-C(5)	0.8(5)	N(10)-C(9)-N(14)-C(13)	0.9(4)
C(3)-C(4)-C(5)-C(6)	-1.2(4)	N(8)-C(9)-N(14)-C(13)	178.0(3)
C(3)-C(4)-C(5)-N(8)	177.4(3)	N(12)-C(11)-N(20)-C(21)	4.0(5)
C(4)-C(5)-C(6)-C(7)	0.9(5)	N(10)-C(11)-N(20)-C(21)	-175.8(3)
N(8)-C(5)-C(6)-C(7)	-177.7(3)	C(11)-N(20)-C(21)-C(22)	103.8(5)
C(3)-C(2)-C(7)-C(6)	-0.2(5)	N(20)-C(21)-C(22)-C(23)	-35.4(7)
C(1)-C(2)-C(7)-C(6)	173.6(3)	C(21)-C(22)-C(23)-S(24)	-81.1(6)
C(5)-C(6)-C(7)-C(2)	-0.3(5)	C(22)-C(23)-S(24)-C(25)	-82.4(7)
C(6)-C(5)-N(8)-C(9)	163.9(3)	N(14)-C(13)-N(30)-C(31)	2.3(5)
C(4)-C(5)-N(8)-C(9)	-14.7(5)	N(12)-C(13)-N(30)-C(31)	179.2(3)
C(5)-N(8)-C(9)-N(10)	-7.9(5)	C(13)-N(30)-C(31)-C(32)	-157.8(4)
C(5)-N(8)-C(9)-N(14)	174.8(3)	N(30)-C(31)-C(32)-C(33)	172.3(4)
N(8)-C(9)-N(10)-C(11)	-179.4(3)	C(31)-C(32)-C(33)-S(34)	70.0(4)
N(14)-C(9)-N(10)-C(11)	-2.6(4)	C(32)-C(33)-S(34)-C(35)	11.2(3)

Symmetry transformations used to generate equivalent atoms:

#1 $y-3/4, -x+3/4, -z-1/4$ #2 $-x, -y+3/2, z$ #3 $-y+3/4, x+3/4, -z-1/4$

Table 7. Bond lengths [$\text{\AA}$] and angles [$^{\circ}$] related to the hydrogen bonding for C64.2 H80.8 N24 S3.2

D-H	..A	d(D-H)	d(H..A)	d(D..A)	<DHA
N(8)-H(8)	N(12)#4	0.88	2.08	2.956(3)	174.2
N(20)-H(20)	S(24)#5	0.88	2.81	3.585(4)	147.8
N(30)-H(30)	N(14)#6	0.88	2.18	3.045(4)	166.5

Symmetry transformations used to generate equivalent atoms:

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

A

B

ORTEP view of the C_{64.2} H_{80.8} N₂₄ S_{3.2} compound with the numbering scheme adopted. Ellipsoids drawn at 30% probability level. Hydrogens represented by sphere of arbitrary size. For clarity, the remaining allyl functions are not represented. **A**: Asymmetric unit. **B**: View of one molecule.

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CRYSTAL AND MOLECULAR STRUCTURE OF
C₄₆ H₃₀ N₁₂ S COMPOUND (JIW278)

Montag, Juli 28, 2003

Equipe WUEST

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ED-255-B

Structure solved and refined by Dr. Gary Enright, Steacie Institute for Molecular Sciences, National Research Council, Ottawa, Ontario and by Dr. Thierry Maris, Laboratory of X-Ray Diffraction, Université de Montréal, Québec.

Table 1. Crystal data and structure refinement for C₄₆ H₃₀ N₁₂ S.

Identification code	JIW278
Empirical formula	C ₄₆ H ₃₀ N ₁₂ S
Formula weight	782.88
Temperature	173(2)K
Wavelength	0.71073 Å
Crystal system	Tetragonal
Space group	I4 ₁ /a
Unit cell dimensions	$a = 23.233(16) \text{ Å}$ $\alpha = 90^\circ$ $b = 23.233(16) \text{ Å}$ $\beta = 90^\circ$ $c = 16.274(17) \text{ Å}$ $\gamma = 90^\circ$
Volume	8785(13) Å ³
Z	8
Density (calculated)	1.184 Mg/m ³
Absorption coefficient	0.120 mm ⁻¹
F(000)	3248
Crystal size	0.30 x 0.30 x 0.15 mm
Theta range for data collection	1.53 to 13.77°
Index ranges	$-15 \leq h \leq 15$, $-15 \leq k \leq 15$, $-10 \leq l \leq 10$
Reflections collected	6088
Independent reflections	688 [$R_{\text{int}} = 0.1523$]
Max. and min. transmission	0.9823 and 0.9650
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	688 / 192 / 182
Goodness-of-fit on F^2	1.766
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.1888$, $wR_2 = 0.4611$
R indices (all data)	$R_1 = 0.2278$, $wR_2 = 0.4611$
Largest diff. peak and hole	0.342 and -0.372 e/Å ³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C46 H30 N12 S.

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

	Occ.	x	y	z	U_{eq}
C(1)	1	5000	7500	11250	54(4)
C(2)	1	4626(4)	7845(3)	10678(4)	51(3)
C(3)	1	3998(4)	7829(5)	10741(5)	60(3)
C(4)	1	3676(4)	8143(3)	10179(5)	66(3)
C(5)	1	3903(2)	8498(5)	9572(7)	51(3)
C(6)	1	4500(2)	8474(4)	9543(7)	66(3)
C(7)	1	4869(4)	8154(4)	10036(5)	60(3)
N(8)	1	3491(7)	8767(7)	9027(10)	57(3)
C(9)	1	3641(5)	9123(4)	8371(5)	54(4)
N(10)	1	3166(4)	9336(5)	8043(7)	55(2)
C(11)	1	3261(6)	9652(5)	7381(7)	62(4)
N(12)	1	3782(7)	9754(5)	7043(6)	55(2)
C(13)	1	4229(5)	9513(6)	7418(8)	57(4)
N(14)	1	4184(5)	9151(5)	8079(7)	57(3)
N(20)	1	2824(4)	9893(9)	6975(11)	114(3)
N(30)	1	4720(9)	9566(7)	7113(6)	114(3)
C(21)	1	2239(5)	9994(11)	7246(14)	226(15)
C(22)	1	1794(6)	10440(13)	6895(19)	226(15)
C(23)	1	1125(5)	10454(4)	6985(7)	400(40)
S(24)	0.470(15)	875(2)	9929(4)	6254(5)	310(20)
C(251)	0.230(15)	434(5)	9614(7)	5465(8)	168(3)
C(252)	0.230(15)	192(4)	10180(10)	5872(9)	168(3)
C(26)	0.94(3)	0	10000	5000	220(30)
C(31)	0.470(15)	4720(4)	9826(8)	6303(5)	260(30)
C(32)	0.470(15)	4347(6)	9630(5)	5563(5)	260(30)
C(33)	0.470(15)	4506(15)	9950(10)	4949(12)	210(40)
C(40)	0.530(15)	970(40)	9420(40)	6090(60)	250
C(41)	0.530(15)	590(40)	9910(40)	4890(60)	250
C(42)	0.530(15)	540(40)	10160(40)	5580(60)	250
C(43)	0.530(15)	5190(40)	9420(40)	5850(50)	250
C(44)	0.530(15)	5160(40)	9890(40)	6310(60)	250
C(45)	0.530(15)	4970(40)	9400(40)	4760(50)	250
C(46)	0.530(15)	1130(40)	9540(40)	5210(60)	250

Table 3. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C46 H30 N12 S.

	Occ.	x	y	z	U _{eq}
H(3)	1	3815	7609	11158	71
H(4)	1	3269	8113	10212	79
H(6)	1	4679	8705	9134	79
H(7)	1	5272	8148	9937	71
H(8)	1	3123	8701	9115	68
H(20)	1	2902	10006	6471	137
H(30)	1	5036	9456	7366	137
H(21A)	1	2269	10083	7839	271
H(21B)	1	2045	9616	7210	271
H(22A)	1	1926	10818	7102	271
H(22B)	1	1865	10445	6295	271
H(23A)	1	972	10841	6854	520
H(23B)	1	1007	10349	7550	520
H(25A)	0.230(15)	694	9441	5052	201
H(25B)	0.230(15)	214	9294	5717	201
H(25A)	0.230(15)	-108	10051	6261	201
H(25B)	0.230(15)	198	10606	5890	201
H(31A)	0.470(15)	5124	9811	6110	342
H(31B)	0.470(15)	4629	10237	6388	342
H(32)	0.470(15)	4062	9337	5560	342
H(33A)	0.470(15)	4798	10232	5023	270
H(33B)	0.470(15)	4330	9903	4426	270

Table 4. Anisotropic parameters ($\text{\AA}^2 \times 10^3$) for C46 H30 N12 S.

The anisotropic displacement factor exponent takes the form:

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

	U11	U22	U33	U23	U13	U12
C(1)	54(4)	54(4)	54(6)	0	0	0
C(2)	51(4)	52(4)	51(4)	0(3)	0(3)	-2(3)
C(3)	59(4)	60(4)	59(4)	-2(3)	0(3)	0(3)
C(4)	66(4)	66(4)	65(4)	1(3)	-1(3)	0(3)
C(5)	51(4)	52(4)	51(4)	0(3)	0(3)	-2(3)
C(6)	66(4)	66(4)	65(4)	1(3)	-1(3)	0(3)
C(7)	59(4)	60(4)	59(4)	-2(3)	0(3)	0(3)
N(8)	56(3)	57(4)	57(4)	1(3)	1(3)	0(3)
C(9)	54(5)	53(5)	55(6)	1(4)	0(4)	1(4)
N(10)	54(3)	56(3)	54(3)	0(2)	-1(2)	0(2)
C(11)	62(5)	61(5)	62(6)	1(4)	0(4)	-2(4)
N(12)	54(3)	56(3)	54(3)	0(2)	-1(2)	0(2)
C(13)	57(5)	57(5)	57(6)	2(4)	2(4)	1(4)
N(14)	56(3)	57(4)	57(4)	1(3)	1(3)	0(3)
N(20)	114(4)	115(4)	114(4)	2(3)	0(3)	0(3)
N(30)	114(4)	115(4)	114(4)	2(3)	0(3)	0(3)
C(21)	220(30)	330(40)	130(20)	50(20)	-20(20)	20(30)
C(22)	220(30)	330(40)	130(20)	50(20)	-20(20)	20(30)
C(23)	330(60)	390(60)	490(70)	0(50)	20(50)	80(50)
S(24)	290(40)	290(40)	350(40)	-70(30)	-70(30)	-30(30)
C(251)	168(4)	168(4)	168(4)	0(3)	0(3)	0(3)
C(252)	168(4)	168(4)	168(4)	0(3)	0(3)	0(3)
C(26)	110(40)	230(50)	310(60)	60(50)	-30(40)	60(40)
C(31)	270(50)	270(50)	240(50)	120(40)	-10(40)	100(40)
C(32)	270(50)	270(50)	240(50)	120(40)	-10(40)	100(40)
C(33)	210(60)	170(60)	240(60)	40(60)	30(60)	-10(50)

Table 5. Bond lengths [Å] and angles [°] for C46 H30 N12 S

C(1)-C(2)#1	1.505(8)	C(7)-C(2)-C(3)	118.1(7)
C(1)-C(2)#2	1.505(8)	C(7)-C(2)-C(1)	120.4(7)
C(1)-C(2)	1.505(7)	C(3)-C(2)-C(1)	121.2(4)
C(1)-C(2)#3	1.505(8)	C(4)-C(3)-C(2)	118.6(8)
C(2)-C(7)	1.389(5)	C(3)-C(4)-C(5)	125.1(9)
C(2)-C(3)	1.4625	C(4)-C(5)-C(6)	112.3(9)
C(3)-C(4)	1.389(5)	C(4)-C(5)-N(8)	116.2(8)
C(4)-C(5)	1.390(5)	C(6)-C(5)-N(8)	131.0(10)
C(5)-C(6)	1.390(5)	C(7)-C(6)-C(5)	128.2(9)
C(5)-N(8)	1.447(17)	C(2)-C(7)-C(6)	117.4(9)
C(6)-C(7)	1.389(5)	C(9)-N(8)-C(5)	124.1(13)
N(8)-C(9)	1.394(17)	N(10)-C(9)-N(14)	128.2
C(9)-N(10)	1.3210	N(10)-C(9)-N(8)	108.9(11)
C(9)-N(14)	1.3512	N(14)-C(9)-N(8)	122.2(10)
N(10)-C(11)	1.3220	C(9)-N(10)-C(11)	113.5
C(11)-N(20)	1.335(18)	N(10)-C(11)-N(20)	120.6(12)
C(11)-N(12)	1.3497	N(10)-C(11)-N(12)	125.4
N(12)-C(13)	1.3285	N(20)-C(11)-N(12)	114.0(12)
C(13)-N(30)	1.25(2)	C(13)-N(12)-C(11)	116.1
C(13)-N(14)	1.3694	N(30)-C(13)-N(12)	119.4(12)
N(20)-C(21)	1.448(10)	N(30)-C(13)-N(14)	116.2(12)
N(30)-C(31)	1.450(10)	N(12)-C(13)-N(14)	124.0
C(21)-C(22)	1.571(19)	C(9)-N(14)-C(13)	112.3
C(22)-C(23)	1.561(16)	C(11)-N(20)-C(21)	129.2(18)
C(23)-S(24)	1.800(6)	C(13)-N(30)-C(31)	113.6(15)
S(24)-C(251)	1.800(6)	N(20)-C(21)-C(22)	127.9(18)
S(24)-C(252)	1.800(6)	C(23)-C(22)-C(21)	129.4(18)
C(251)-C(26)	1.547(14)	C(22)-C(23)-S(24)	104.2(14)
C(252)-C(26)	1.545(16)	C(23)-S(24)-C(251)	158.8(8)
C(26)-C(252)#4	1.545(16)	C(23)-S(24)-C(252)	107.1(9)
C(26)-C(251)#4	1.547(14)	C(251)-S(24)-C(252)	52.0(9)
C(31)-C(32)	1.550(6)	C(26)-C(251)-S(24)	118.9(9)
C(32)-C(33)	1.300(6)	C(26)-C(252)-S(24)	119.0(10)
C(40)-C(46)	1.50(12)	C(252)-C(26)-C(252)#4	180.000(4)
C(41)-C(42)	1.27(12)	C(252)-C(26)-C(251)#4	118.7(8)
C(41)-C(46)	1.61(12)	C(252)#4-C(26)-C(251)#4	61.3(8)
C(43)-C(44)	1.32(11)	C(252)-C(26)-C(251)	61.3(8)
C(43)-C(45)	1.84(11)	C(252)#4-C(26)-C(251)	118.7(8)
		C(251)#4-C(26)-C(251)	180.000(5)
C(2)#1-C(1)-C(2)#2	112.5(3)	N(30)-C(31)-C(32)	125.8(12)
C(2)#1-C(1)-C(2)	112.5(3)	C(33)-C(32)-C(31)	105.7(16)
C(2)#2-C(1)-C(2)	103.6(6)	C(42)-C(41)-C(46)	91(8)
C(2)#1-C(1)-C(2)#3	103.6(6)	C(44)-C(43)-C(45)	123(8)
C(2)#2-C(1)-C(2)#3	112.5(3)	C(40)-C(46)-C(41)	102(8)
C(2)-C(1)-C(2)#3	112.5(3)		

Symmetry transformations used to generate equivalent atoms:

#1 $y-1/4, -x+5/4, -z+9/4$	#2 $-x+1, -y+3/2, z+0$
#3 $-y+5/4, x+1/4, -z+9/4$	#4 $-x, -y+2, -z+1$

Table 6. Bond lengths [Å] and angles [°] related to the hydrogen bonding for C46 H30 N12 S.

D-H	..A	d(D-H)	d(H..A)	d(D..A)	<DHA
N(8)-H(8)	N(12)#5	0.88	2.14	3.00(3)	165.6
N(20)-H(20)	N(10)#6	0.88	2.24	3.05(4)	152.8

Symmetry transformations used to generate equivalent atoms:

#1	$y-1/4, -x+5/4, -z+9/4$	#2	$-x+1, -y+3/2, z$
#3	$-y+5/4, x+1/4, -z+9/4$	#4	$-x, -y+2, -z+1$
#5	$y-3/4, -x+5/4, z+1/4$	#6	$-y+5/4, x+3/4, z-1/4$

A **B**

ORTEP view of the C₄₆ H₃₀ N₁₂ S compound with the numbering scheme adopted. Ellipsoids drawn at 30% probability level. Hydrogens represented by sphere of arbitrary size. **A**: Asymmetric unit. **B**: View of the polymeric system.

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