Supplementary Information II

Synthesis, characterization, and impact of carbon spacer on geometry and antibacterial activity of a new series of 1,n-bis(5-cycloalkylsulfenyl-1,3,4-thiadiazole-2-sulfenyl) alkanes (n = 4 and 6)

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Figure S1. The Oak Ridge thermal ellipsoid plot (ORTEP) diagrams of C5C6TD at a thermal value of 30 °C and a symmetry code of 1-x,2-y,-z.

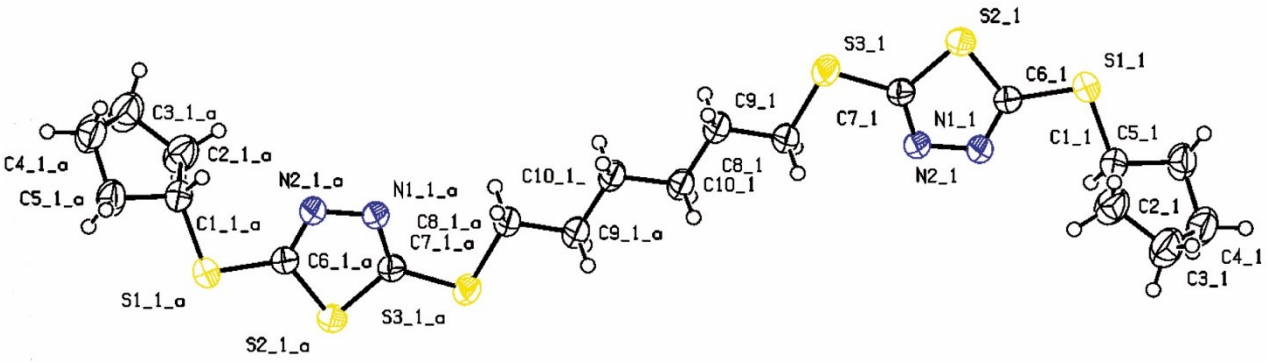

Figure S2a. The conformation and space direction of the carbon spacer, cyclopentyl, and TD rings regarding the face plane (010).

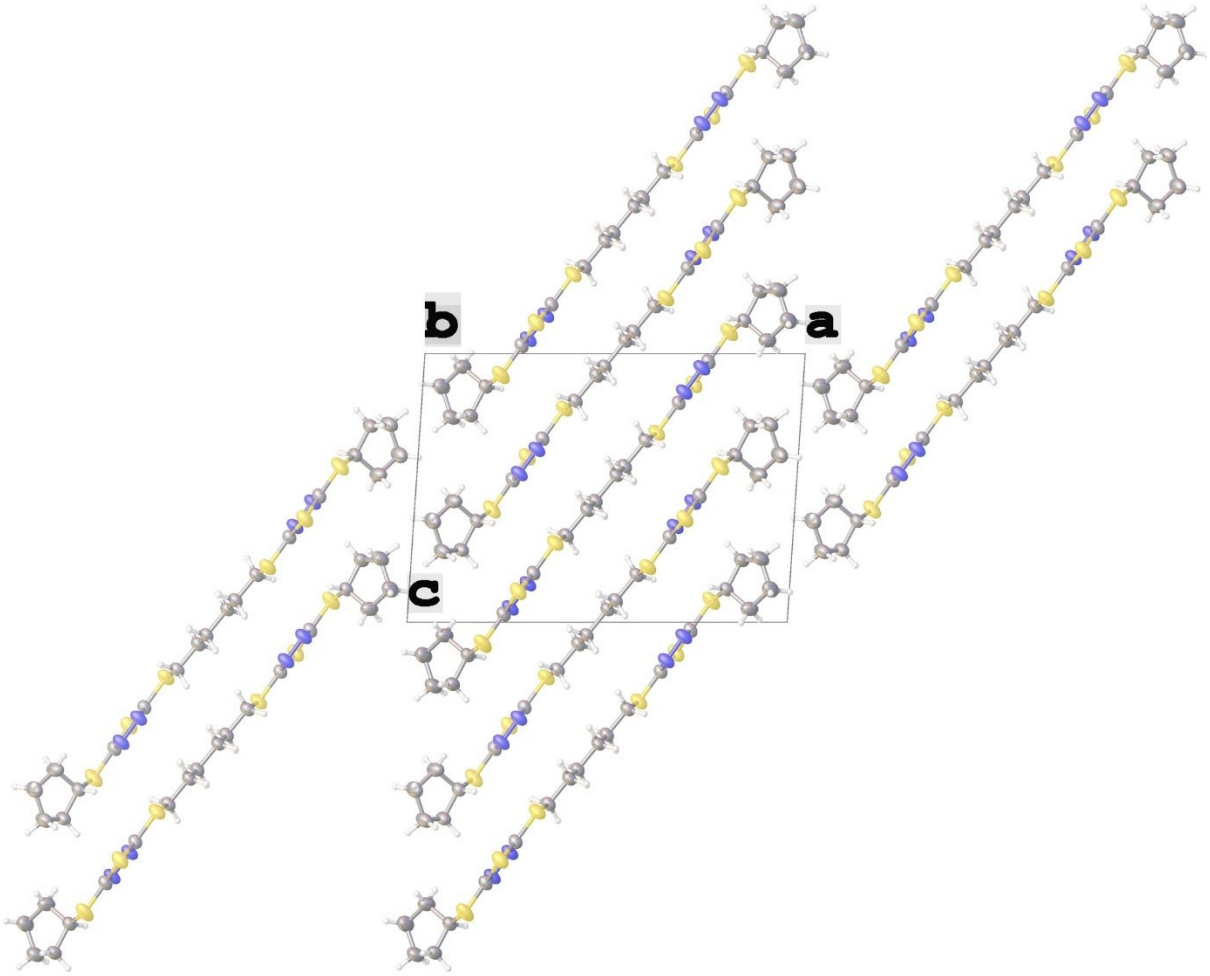

Figure S2b. The conformation and space direction of the carbon spacer and cyclopentyl, and TD rings regarding the diagonal plane of (011).

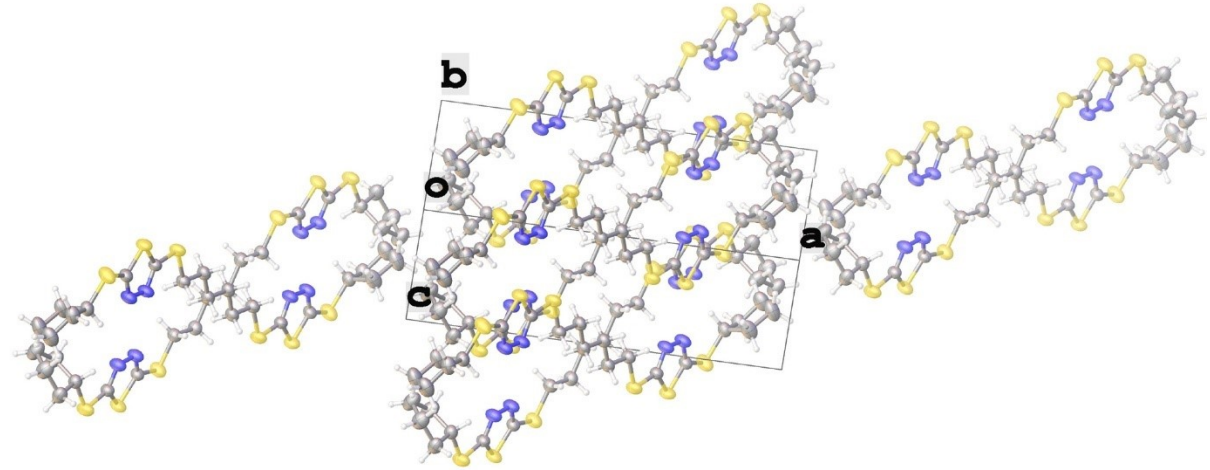

Table 1. Crystal data and structure refinement of 1,6-bis(2-cyclopentylsulfenyl-1,3,4-thiadiazole-5-sulfenyl) hexane.

Identification code	exp192_a
Empirical formula	$C_{10}H_{15}N_2S_3$
Formula weight	259.42
Temperature/K	303(2)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	17.644(6)
b/Å	5.6480(18)
c/Å	12.488(4)
α/°	90
β/°	93.834(12)
γ/°	90
Volume/Å^3	1241.7(7)
Z	4
ρ_{calc}g/cm^3	1.388
μ/mm^{-1}	0.567
F(000)	548.0
Crystal size/mm^3	0.52 × 0.39 × 0.05
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.54 to 55.158
Index ranges	$-22 \le h \le 22$, $-7 \le k \le 7$, $-16 \le l \le 14$
Reflections collected	35642
Independent reflections	2867 [R_{int} = 0.0570, R_{sigma} = 0.0265]
Data/restraints/parameters	2867/0/137
Goodness-of-fit on F^2	1.037
Final R indexes [I>=2σ (I)]	R_1 = 0.0347, wR_2 = 0.0850
Final R indexes [all data]	R_1 = 0.0431, wR_2 = 0.0913
Largest diff. peak/hole / e Å^{-3}	0.30/-0.32

Table 2. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) of 1,6-bis(2-cyclopentylsulfenyl-1,3,4-thiadiazole-5-sulfenyl) hexane. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
S1_1	7965.0(3)	6122.5(9)	4200.3(5)	63.86(18)
S2_1	7130.0(3)	7340.5(7)	6151.5(4)	54.03(16)
S3_1	6238.1(3)	5462.0(8)	7950.5(4)	53.71(15)
N1_1	6887.4(9)	2936(2)	6431.3(12)	49.2(4)
N2_1	7307.5(9)	3083(3)	5531.5(12)	51.0(4)
C1_1	8346.7(10)	3334(3)	3775.5(14)	47.0(4)
C2_1	8986.7(12)	2323(5)	4506.1(16)	67.3(6)
C3_1	9503.2(13)	1055(5)	3772.1(18)	75.6(7)
C4_1	9187.5(13)	1504(5)	2631.8(16)	69.8(6)
C5_1	8693.9(11)	3659(4)	2694.5(14)	57.1(5)
C6_1	7470.6(9)	5239(3)	5298.8(14)	44.0(4)
C7_1	6757.7(9)	4989(3)	6833.3(14)	41.6(4)
C8_1	5957.9(10)	2460(3)	8238.4(14)	45.0(4)
C9_1	5454.3(10)	2502(3)	9184.8(14)	47.2(4)
C10_1	5249.1(9)	21(3)	9525.0(13)	44.8(4)

Table 3. Anisotropic Displacement Parameters ($Å^2 \times 10^3$) of 1,6-bis(2-cyclopentylsulfenyl-1,3,4-thiadiazole-5-sulfenyl) hexane. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1_1	75.7(3)	46.1(3)	74.4(3)	15.6(2)	40.2(3)	7.7(2)
S2_1	66.1(3)	30.7(2)	68.3(3)	-4.81(18)	26.7(2)	-5.40(18)
S3_1	66.5(3)	41.2(2)	56.5(3)	-11.41(19)	27.4(2)	-4.5(2)
N1_1	66.2(9)	32.9(7)	51.5(8)	-1.5(6)	26.4(7)	0.6(6)
N2_1	67.3(9)	35.9(7)	53.2(8)	0.1(6)	28.3(7)	2.4(7)
C1_1	45.9(9)	50.3(9)	45.9(9)	2.8(7)	12.6(7)	3.1(8)
C2_1	72.5(13)	84.8(16)	44.8(10)	2.1(10)	5.4(9)	24.5(12)
C3_1	67.1(13)	99.6(19)	61.4(12)	11.0(12)	14.8(10)	31.5(13)
C4_1	72.4(13)	87.1(16)	52.1(11)	2.1(11)	20.1(10)	18.9(12)
C5_1	53.8(10)	75.6(13)	42.9(9)	8.4(9)	10.4(8)	8.0(9)
C6_1	46.2(9)	36.0(8)	51.3(9)	2.3(7)	14.1(7)	2.5(7)
C7_1	44.6(8)	34.2(8)	47.2(9)	-2.5(6)	11.1(7)	-1.0(6)
C8_1	50.1(9)	41.5(8)	45.2(9)	-1.9(7)	15.1(7)	-0.5(7)
C9_1	47.8(9)	48.5(9)	46.8(9)	-4.0(7)	15.3(7)	2.0(7)
C10_1	45.2(9)	48.3(9)	42.2(9)	-4.7(7)	12.9(7)	2.1(7)

Table 4. Geometric parameters of 1,6-bis(2-cyclopentylsulfenyl-1,3,4-thiadiazole-5-sulfenyl) hexane.

Bond length		
Atom	**Atom**	**Length/Å**
S1_1	C1_1	1.8066(19)
S1_1	C6_1	1.7470(17)
S2_1	C6_1	1.7289(17)
S2_1	C7_1	1.7307(17)
S3_1	C7_1	1.7406(17)
S3_1	C8_1	1.8091(18)
N1_1	N2_1	1.389(2)
N1_1	C7_1	1.290(2)
N2_1	C6_1	1.289(2)
C1_1	C2_1	1.515(3)
C1_1	C5_1	1.530(2)
C2_1	C3_1	1.515(3)
C3_1	C4_1	1.515(3)
C4_1	C5_1	1.502(3)
C8_1	C9_1	1.526(2)
C9_1	C10_1	1.515(2)
C10_1	C10_1[1]	1.523(3)

Bond Angles			
Atom	**Atom**	**Atom**	**Angle/°**
C6_1	S1_1	C1_1	101.37(8)
C6_1	S2_1	C7_1	86.28(8)
C7_1	S3_1	C8_1	100.43(8)
C7_1	N1_1	N2_1	112.27(13)
C6_1	N2_1	N1_1	112.23(14)
C2_1	C1_1	S1_1	115.41(14)
C2_1	C1_1	C5_1	104.49(15)
C5_1	C1_1	S1_1	109.47(13)
C3_1	C2_1	C1_1	105.55(16)
C2_1	C3_1	C4_1	106.94(17)
C5_1	C4_1	C3_1	105.34(18)
C4_1	C5_1	C1_1	102.49(16)
S2_1	C6_1	S1_1	120.00(10)
N2_1	C6_1	S1_1	125.28(13)
N2_1	C6_1	S2_1	114.68(13)
S2_1	C7_1	S3_1	120.94(9)
N1_1	C7_1	S2_1	114.54(12)
N1_1	C7_1	S3_1	124.51(12)
C9_1	C8_1	S3_1	108.68(12)
C10_1	C9_1	C8_1	111.42(14)
C9_1	C10_1	C10_1[1]	113.21(17)

Torsion Angles				
Atom	**Atom**	**Atom**	**Atom**	**Angle/°**
S1_1	C1_1	C2_1	C3_1	162.67(15)
S1_1	C1_1	C5_1	C4_1	-146.52(17)
S3_1	C8_1	C9_1	C10_1	-175.54(12)
N1_1	N2_1	C6_1	S1_1	-177.91(13)
N1_1	N2_1	C6_1	S2_1	0.0(2)
N2_1	N1_1	C7_1	S2_1	0.4(2)
N2_1	N1_1	C7_1	S3_1	179.08(13)
C1_1	S1_1	C6_1	S2_1	168.69(11)
C1_1	S1_1	C6_1	N2_1	-13.51(19)
C1_1	C2_1	C3_1	C4_1	-35.9(2)
C2_1	C1_1	C5_1	C4_1	-26.3(2)
C2_1	C3_1	C4_1	C5_1	20.1(3)
C3_1	C4_1	C5_1	C1_1	4.1(3)
C5_1	C1_1	C2_1	C3_1	38.5(2)
C6_1	S1_1	C1_1	C2_1	170.51(13)

Atom	Atom	Atom	Atom	Angle/°
C6_1	S1_1	C1_1	C5_1	-71.99(16)
C6_1	S2_1	C7_1	S3_1	-179.07(12)
C6_1	S2_1	C7_1	N1_1	-0.32(15)
C7_1	S2_1	C6_1	S1_1	178.20(12)
C7_1	S2_1	C6_1	N2_1	0.17(15)
C7_1	S3_1	C8_1	C9_1	-177.42(12)
C7_1	N1_1	N2_1	C6_1	-0.2(2)
C8_1	S3_1	C7_1	S2_1	176.30(11)
C8_1	S3_1	C7_1	N1_1	-2.32(19)
C8_1	C9_1	C10_1	C10_1[1]	179.55(18)

[1]1-X,-Y,2-Z

Table 5. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) of 1,6-bis(2-cyclopentylsulfenyl-1,3,4-thiadiazole-5-sulfenyl) hexane.

Atom	*x*	*y*	*z*	U(eq)
H1_1	7934.91	2169.65	3694.28	56
H2A_1	9259.08	3577.48	4897.55	81
H2B_1	8790.29	1226.03	5017.36	81
H3A_1	9511.8	-629.45	3923.81	91
H3B_1	10017.22	1662.02	3875.37	91
H4A_1	9595.2	1788.24	2163.89	84
H4B_1	8891.94	158.31	2360.08	84
H5A_1	8304.3	3697.18	2108.69	69
H5B_1	8991.99	5100.9	2683.6	69
H8A_1	5681.98	1778.53	7614.68	54
H8B_1	6404.5	1497.16	8412.81	54
H9A_1	5718.26	3312.19	9784.68	57
H9B_1	4993.23	3377.12	8985.67	57
H10A_1	5712.53	-851.48	9712.92	54
H10B_1	4984.85	-778.32	8922.28	54

Experimental

Single crystals of 1,6-bis(2-cyclopentylsulfenyl-1,3,4-thiadiazole-5-sulfenyl) hexane ($C_{20}H_{30}N_4S_6$) were isolated through recrystallization from ethyl acetate at room temperature. A suitable crystal was selected and mounted on the Mitegen Dual-Thickness Micromount loop. The experimental data were collected using a Bruker D8 Quest Photon III HPAD Diffractometer. The crystal was kept at 303(2) K during data collection. Using ShelXle Qt6 [1], the structure was solved using the direct method in SHELXS software [2] and refined with the SHELX refinement software [2] integrated in the graphical ShelXle software.

Crystal structure determination of 1,6-bis(2-cyclopentylsulfenyl-1,3,4-thiadiazole-5-sulfenyl) hexane

Crystal Data for $C_{10}H_{15}N_2S_3$ (M =259.42 g/mol): monoclinic, space group $P2_1/c$ (no. 14), a = 17.644(6) Å, b = 5.6480(18) Å, c = 12.488(4) Å, β = 93.834(12)°, V = 1241.7(7) Å^3, Z = 4, T = 303(2) K, μ(MoKα) = 0.567 mm^{-1}, *Dcalc* = 1.388 g/cm^3, 35642 reflections measured (6.54° ≤ 2Θ ≤ 55.158°), 2867 unique (R_{int} = 0.0570, R_{sigma} = 0.0265) which were used in all calculations. The final R_1 was 0.0347 (I > 2σ(I)), and wR_2 was 0.0913 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

[1.a] Ternary CH refined with riding coordinates:

C1(H1)

[1.b] Secondary CH2 refined with riding coordinates:

C2(H2A,H2B), C3(H3A,H3B), C4(H4A,H4B), C5(H5A,H5B), C8(H8A,H8B), C9(H9A,H9B),

C10(H10A,H10B)

This report has been created with Olex2, compiled on 2020.11.12 svn.r5f609507 for OlexSys. Please let us know if there are any errors or if you would like to have additional features.

Reference

[1] C.B. Hübschle, G.M. Sheldrick, B. Dittrich, ShelXle: a Qt graphical user interface for SHELXL, J. Appl. Cryst., 2011, 44, 1281-1284.

https://doi.org/10.1107/S0021889811043202

[2] G.M. Sheldrick, A short history of SHELX. Acta Crystallogr., Sect. A: Found. Crystallogr. 2008, 64(1), 112-122.

https://doi.org/10.1107/S0108767307043930