X-Ray Crystallographic Studies

Crystals of **(*R*)-18 and (*R*)-25** suitable for X-ray diffraction studies, were dissolved in a minimum amount of CH_2Cl_2 and recrystallized in Et_2O/hexanes (1/2). Crystallographic data were collected at 100 (2) K on a Bruker Appex-II CCD Diffractometer, using graphite-monocromated *Mo-Kα* radiation (λ = 0.71073 Å) from a fine focus sealed tube source. The data were processed with BRUKER APPEX2 software[1] and an empirical absorption correction was applied using SADABS.[2] The structure was solved by SIR-97 [3]and finally refined by full-matrix, least-squares based on F^2 by SHELXL.[4] All non-hydrogen atoms were anisotropically refined and the hydrogen atoms positions were included in the model by electronic density or were geometrically calculated and refined using a riding model.
Crystallographic data are summarized in Table S1 and S1b, Bond distances and angles are summarized in Table S2, while hydrogen bond parameters are listed in Table S3.

Table S1. Crystallographic data of **(*R*)-18** and **(*R*)-25**.

	(*R*)-18	**(*R*)-25**
Formula	$C_{14}H_{19}NO_5S$	$C_{14}H_{18}N_4O_5S$
Molecular weight	313.36	354.39
Space group	$P2_1$	$P2_12_12_1$
Crystal system	Monoclinic	Orthorhombic
Crystal size /mm	0.36 x 0.14 x 0.08	0.27 x 0.04 x 0.04
a/Å	10.9779(7)	5.7297(3)
b/Å	5.7869(5)	16.2240(9)
c/Å	12.1402(10)	17.5243(9)
α/º	90	90
β/º	92.349(4)	90
γ/º	90	90
Volume/Å^3	770.59(10)	1629.04(15)
Z	2	4
D/g cm^{-3}	1.351	1.445
T(K)	100(2)	100(2)
Interval θ/º	3.4 to 26.2	2.3 to 26.5
Measured reflexions	11862	8190
Independent reflexions [R_{int}]	3156[0.037]	3347[0.052]
μ /mm^{-1}	0.23	0.23
F(000)	332	744
Residues/e Å^{-3}	0.21 and -0.30	0.25 and -0.34
R_1 (I > 2s(I)) [R_1 (all data)]	0.0325 [0.0357]	0.0465 [0.0762]
$_wR^2$ (I > 2s(I)) [$_wR^2$ all data]	0.068 [0.0695]	0.0948 [0.1098]
GOF	1.014	0.854
Flack parameter	0.03(4)	0.10(8)

Table S1b. Bayesian Statistics analyzed by Platon[5] of (*R*)-18 and (*R*)-25**.**

Bayesian Statistics	**(*R*)-18**	**(*R*)-25**
Type	Gaussian	
Select Pairs	1411	1379
P2(true)	1.000	1.000
P3(true)	1.000	1.000
P3(rac-twin)	0.1E-38	0.1E-03
P3(false)	0.4E-160	0.1E-23
G	0.9713	0.7426
G (su)	0.0725	0.1643
Hooft y	0.01(4)	0.13(8)

Table S2. Selected bond distances (Å) and angles (º) of **(*R*)-18** and **(*R*)-25**.

Bond distances (Å)	**(*R*)-18**	**(*R*)-25**	**Bond Angles (º)**	**(*R*)-18**	**(*R*)-25**
C1-N1	1.465(3)	1.471(5)	**N1-C1-C2**	100.9(2)	100.9(3)
C1-C10	1.532(4)	1.538(6)	**N1-C1-C10**	110.3(2)	110.7(3)
C1-C2	1.529(3)	1.532(6)	**C2-C1-C10**	112.7(2)	111.4(3)
C1-C3	1.549(3)	1.542(5)	**N1-C1-C3**	112.2(2)	112.4(3)
C2-O3	1.464(3)	1.462(5)	**C2-C1-C3**	110.8(2)	111.5(4)
C3-C4	1.511(3)	1.509(6)	**C10-C1-C3**	109.8(2)	109.7(3)
C4-C5	1.388(4)	1.394(6)	**O3-C2-C1**	104.7(2)	104.2(3)
C4-C9	1.397(4)	1.391(6)	**C4-C3-C1**	114.9(2)	115.2(4)
C5-C6	1.391(4)	1.381(6)	**C5-C4-C9**	117.8(2)	117.6(4)
C6-C7	1.383(4)	1.392(6)	**C5-C4-C3**	122.2(2)	121.8(4)
C7-C8	1.385(4)	1.373(6)	**C9-C4-C3**	120.0(3)	120.6(4)
C8-C9	1.383(4)	1.391(6)	**C6-C5-C4**	121.4(3)	121.5(4)
C10-O4	1.206(3)	1.208(5)	**C7-C6-C5**	120.0(3)	119.7(4)
C10-O5	1.331(3)	1.328(5)	**C6-C7-C8**	119.3(2)	119.9(4)
C11-O5	1.494(3)	1.497(5)	**C9-C8-C7**	120.5(2)	119.9(4)
C11-C12	1.517(4)	1.519(6)	**C8-C9-C4**	121.0(3)	121.4(4)
C11-C13	1.516(4)	1.511(6)	**O4-C10-O5**	125.9(2)	126.8(4)
C11-C14	1.511(4)	1.521(6)	**O4-C10-C1**	123.2(2)	123.0(4)
N1-S1	1.643(2)	1.634(3)	**O5-C10-C1**	110.9(2)	110.1(4)
S1-O1	1.429(2)	1.429(3)	**O5-C11-C14**	102.4(2)	101.6(3)
S1-O2	1.424(2)	1.425(3)	**O5-C11-C12**	109.2(2)	109.6(3)
S1-O3	1.578(2)	1.574(3)	**C14-C11-C12**	111.0(3)	111.2(4)
C7-N2		1.446(6)	**O5-C11-C13**	109.7(2)	109.3(3)
N2-N3		1.223(5)	**C14-C11-C13**	111.6(3)	112.1(4)
N3-N4		1.136(6)	**C12-C11-C13**	112.4(3)	112.5(4)
			C1-N1-S1	111.0(1)	111.5(2)
			O2-S1-O1	116.7(1)	116.0 (2)
			O2-S1-O3	108.2(1)	108.8(2)
			O1-S1-O3	109.8(1)	109.6(2)
			O2-S1-N1	114.7(1)	114.5(2)
			O1-S1-N1	109.1(1)	109.7(2)
			O3-S1-N1	96.2(1)	96.5(2)
			C2-O3-S1	110.2(1)	109.3(2)
			C10-O5-C11	120.5(2)	121.5(3)
			N3-N2-C7		115.6(4)
			N4-N3-N2		172.7(5)
			C8-C7-N2		117.1(4)
			C6-C7-N2		123.0(4)

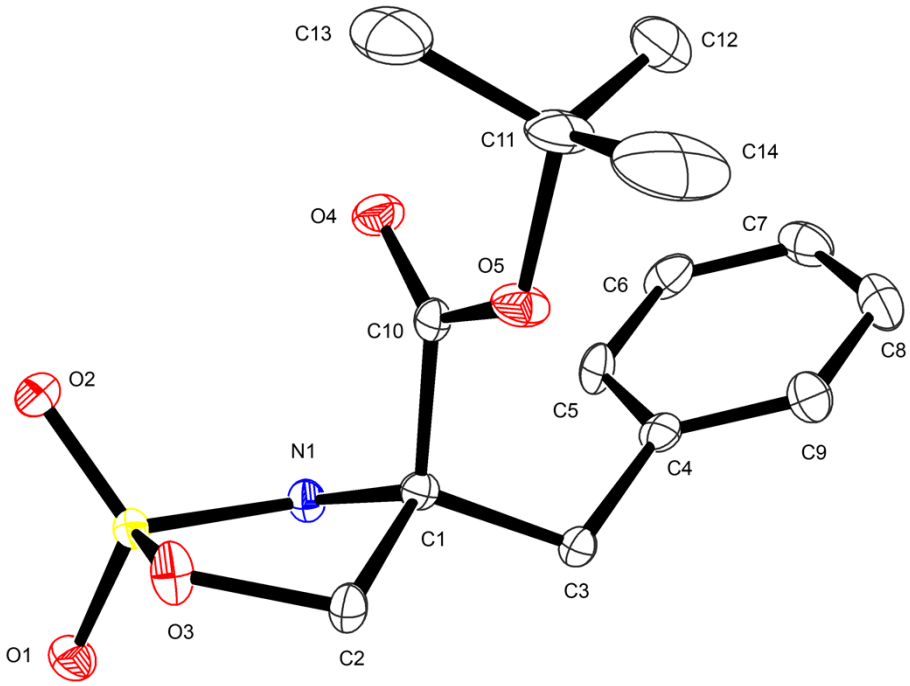

Figure S1.ORTEP diagram for (R)-18 with thermal ellipsoids drawn at the 50% probability level. Hydrogen atoms were omitted for the sake of clarity.

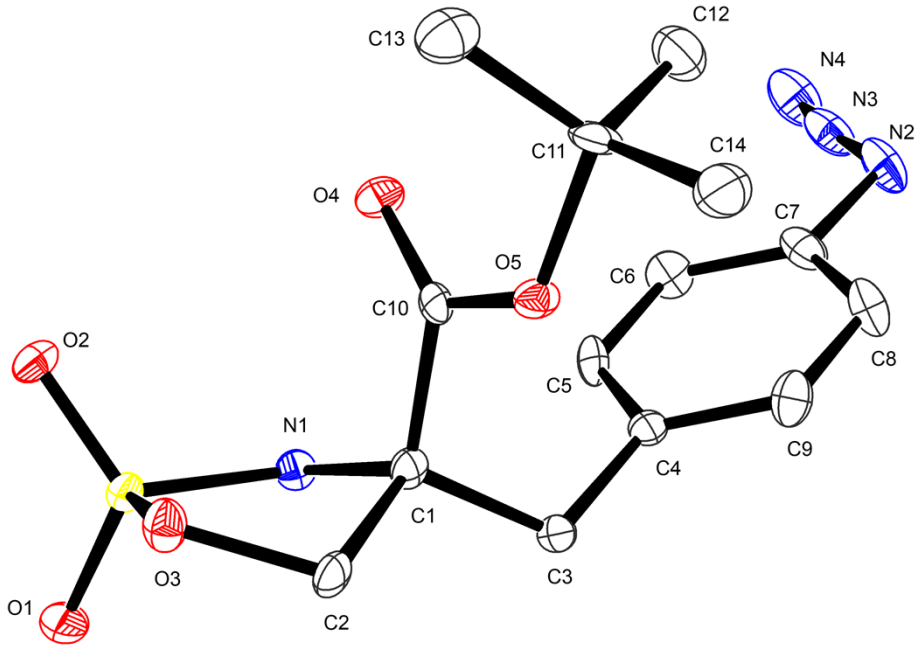

Figure S2.ORTEP diagram for (R)-25 with thermal ellipsoids drawn at the 50% probability level. Hydrogen atoms were omitted for the sake of clarity.

Table S3. Hydrogen bond parameters [Å] for **(*R*)-18** and **(*R*)-25**.

Sample	Parameter	D–H···A	D–H	H···A	∠DHA
(*R*)-18	N1—H1*N*···O1^{i}	2.947 (3)	0.82 (2)	2.16 (2)	162 (3)
(*R*)-25	N1—H1*N*···O1^{ii}	2.954 (4)	0.84 (2)	2.18 (3)	153 (4)

Symmetry transformations: (i) 1-x,0.5+ y, 2-z; (ii) 0.5+x,-y-0.5,-z

Compound **(*R*)-18** forms infinite 1D chains along the axis "b" by intermolecular hydrogen bonds between the N1 (NH) and the O1 (SO_2) (Figure S3a, S3b), while for **(*R*)-25** they form the same 1D chains along the axis "a" (Figure S4a, S4b).

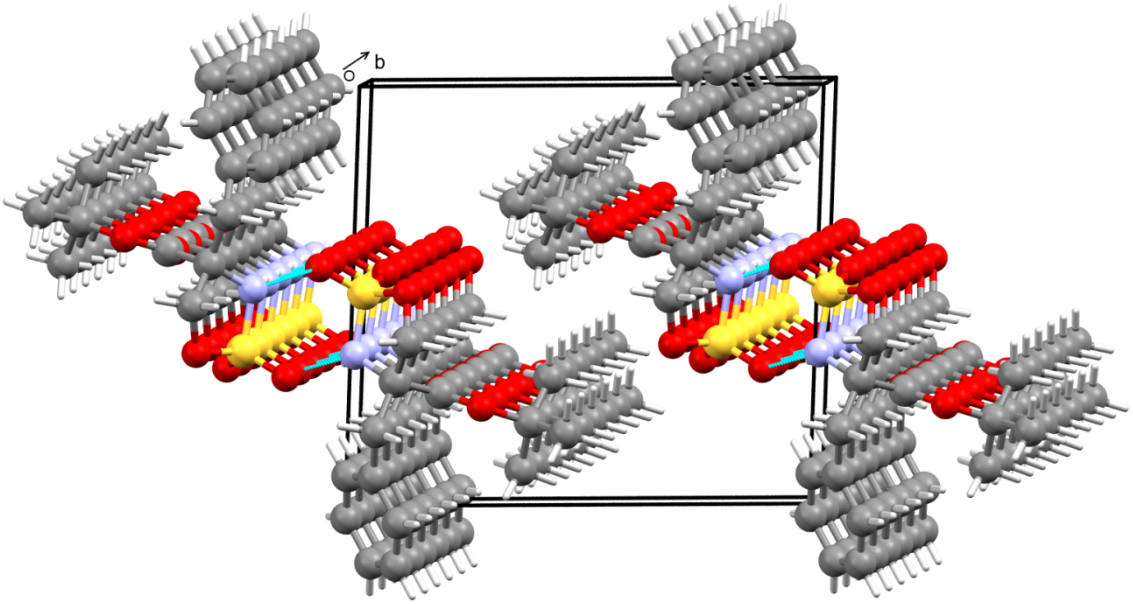

FIG.S3a: Packing ball and stick representation of infinite 1D chains along “b” axis for **(*R*)-18**.

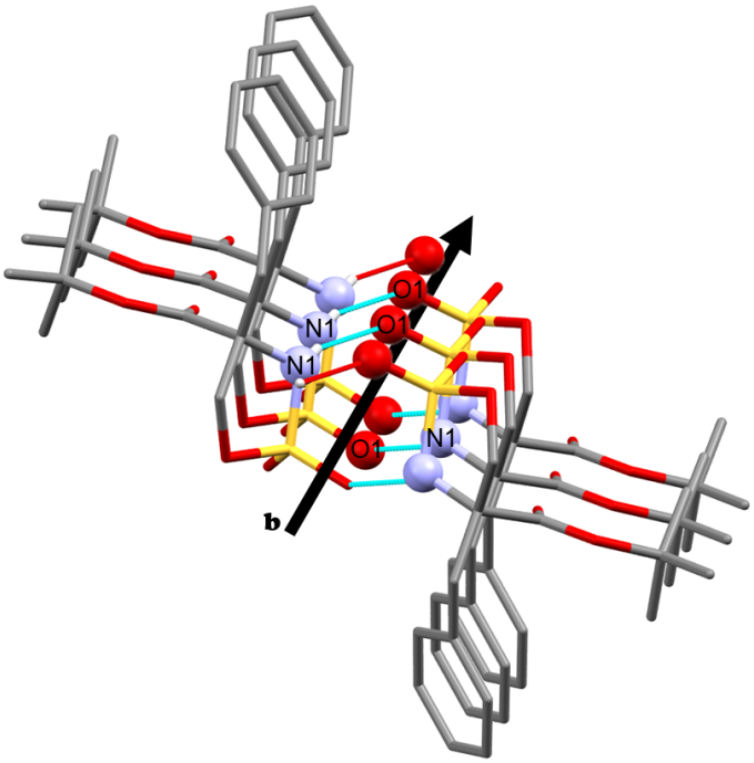

FIG.S3b: Detail of infinite hydrogen bonds 1D chain along "b" axis for **(*R*)-18**.

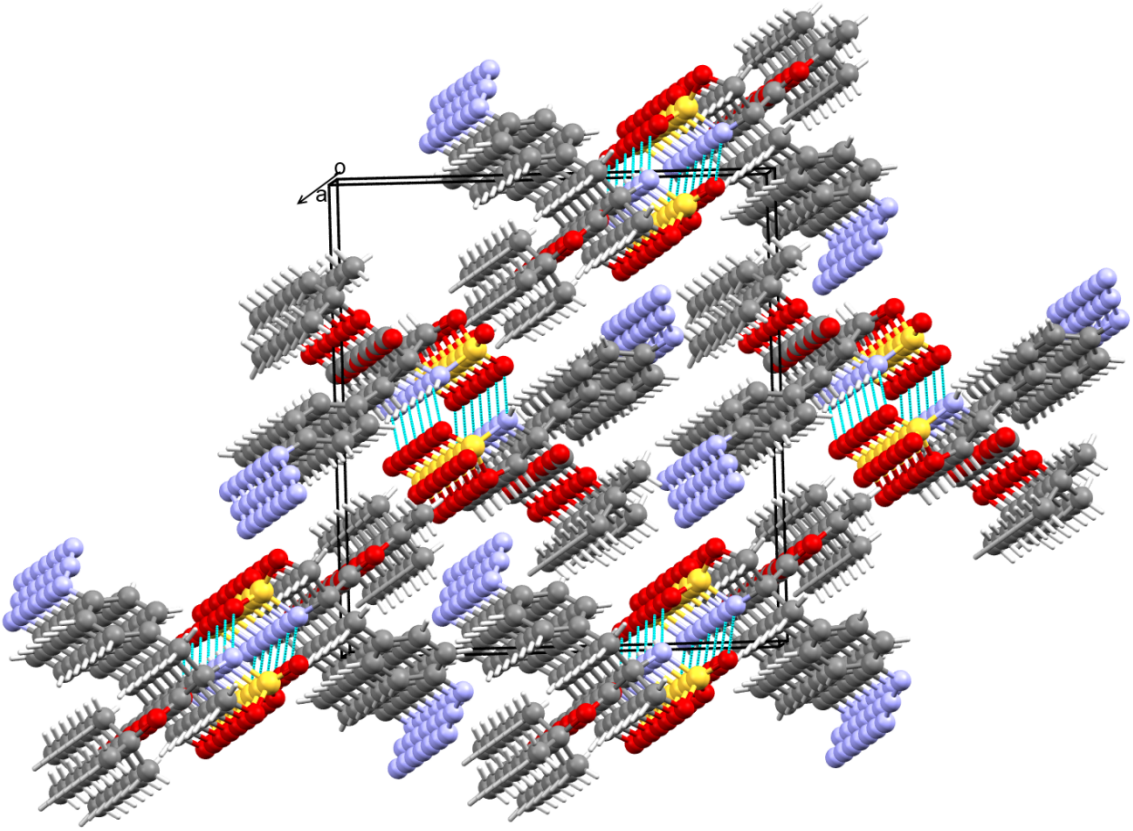

FIG.S4a: Packing ball and stick representation of 1D chains along "a" axis for **(*R*)-25**.

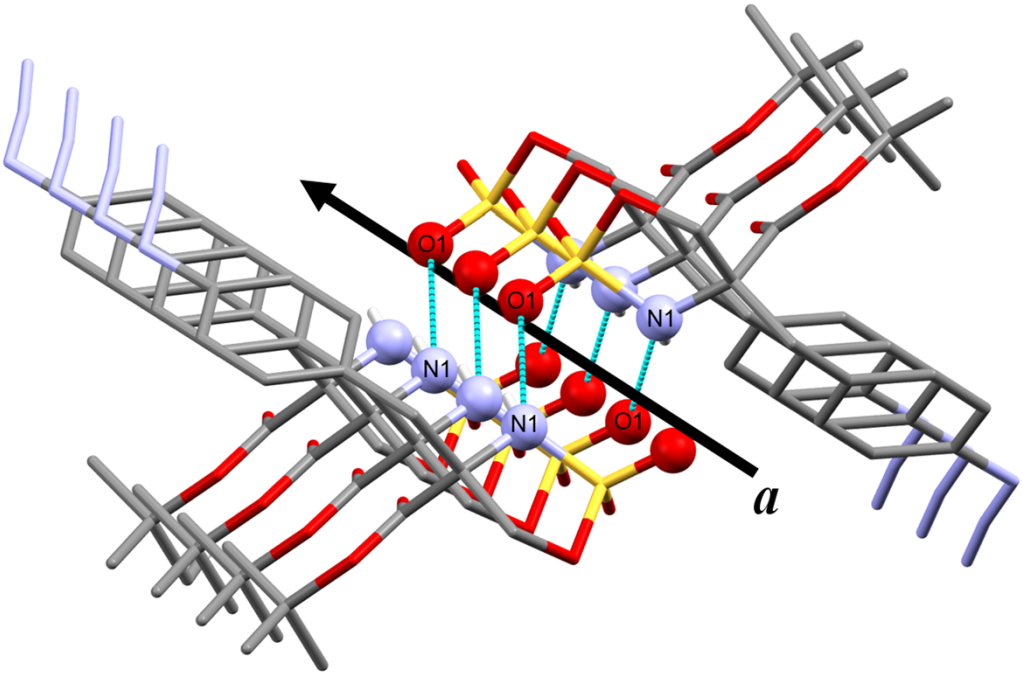

FIG.S4b: Detail of infinite hydrogen bonds 1D chain along "a" axis for **(*R*)-25**.

References

[1] Bruker, APPEX II, Bruker AXS Inc., Madison, WI, USA, 2004.(Version 2013.6).

[2] *G. M. Sheldrick, SADABS, Program for Scaling and Correction of Area Detector Data, University of Göttingen, Germany, 1996.*

[3] SIR97: A. Altomare, M. C. Burla, M. Camalli, G. L. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori, R. Spagna, *J. Appl. Cryst.,* 1999, 32, 115-119.

[4] SHELXL-2013: G.M. Sheldrick (2008) Acta Cryst., A64, 112-122. (version 2012-9).

[5] Spek, A.L. (2003). J. Appl. Cryst. 36, 7-13.