Two-Dimensional Molecular Layers: Interplay of H-Bonding and van der Waals Interactions in the Self-Assembly of N,N'-Dialkylsulfamides

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Supporting Information

Table 1. Crystal data and structure refinement for **1b.**

Empirical formula	C10 H24 N2 O2 S	
Formula weight	236.37	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P b c a	
Unit cell dimensions	a = 10.4797(6) Å	α= 90°.
	b = 9.9159(6) Å	β= 90°.
	c = 27.6361(14) Å	γ = 90°.
Volume	2871.8(3) Å3	
Z	8	
Density (calculated)	1.093 Mg/m3	
Absorption coefficient	0.213 mm-1	
F(000)	1040	
Crystal size	0.20 x 0.02 x 0.01 mm3	
Theta range for data collection	2.44 to 28.03°.	
Index ranges	-13<=h<=12, -12<=k<=13, -28<=l<=36	
Reflections collected	17191	
Independent reflections	3383 [R(int) = 0.1693]	
Completeness to theta = 28.03°	97.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000000 and 0.463896	
Refinement method	Full-matrix least-squares on F2	
Data / restraints / parameters	3383 / 0 / 146	
Goodness-of-fit on F2	1.208	
Final R indices [I>2sigma(I)]	R1 = 0.1491, wR2 = 0.2750	
R indices (all data)	R1 = 0.3480, wR2 = 0.3372	
Extinction coefficient	0.000(3)	
Largest diff. peak and hole	0.360 and -0.218 e.Å-3	

Table 2. Atomic coordinates (x 104) and equivalent isotropic displacement parameters (Å2x 103) for **1b**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S	7903(1)	1149(2)	2341(1)	108(1)
O(1)	8383(4)	2338(4)	2108(2)	156(2)
O(2)	6633(3)	1155(4)	2490(2)	147(2)
N(1)	7957(5)	-82(5)	1958(2)	118(2)
N(2)	8890(5)	914(6)	2769(2)	130(2)
C(11)	9239(9)	-428(12)	1753(5)	166(3)
C(12)	9185(11)	-1368(14)	1336(6)	251(6)
C(13)	8484(14)	-889(14)	908(5)	222(4)
C(21)	8679(11)	-332(11)	3053(4)	162(3)
C(22)	9216(18)	-36(18)	3561(5)	222(5)
C(23)	8939(17)	-979(17)	3908(4)	275(7)
C(131)	8410(20)	-1940(20)	517(6)	496(18)
C(132)	9118(16)	379(18)	713(6)	353(9)
C(231)	7975(17)	-1760(30)	3932(6)	455(17)
C(232)	9588(14)	-733(16)	4379(4)	338(8)

Table 3. Bond lengths [Å] and angles [°] for **1b**.

S-O(2)	1.393(3)
S-O(1)	1.434(4)
S-N(2)	1.588(6)
S-N(1)	1.617(5)
N(1)-C(11)	1.497(8)
N(2)-C(21)	1.480(8)
C(11)-C(12)	1.485(13)
C(12)-C(13)	1.470(14)
C(13)-C(131)	1.506(15)
C(13)-C(132)	1.521(15)
C(21)-C(22)	1.541(11)
C(22)-C(23)	1.371(13)
C(23)-C(231)	1.275(14)
C(23)-C(232)	1.488(13)
O(2)-S-O(1)	117.6(3)
O(2)-S-N(2)	113.8(3)
O(1)-S-N(2)	103.1(3)
O(2)-S-N(1)	103.3(3)
O(1)-S-N(1)	108.3(3)
N(2)-S-N(1)	110.7(3)
C(11)-N(1)-S	116.9(6)
C(21)-N(2)-S	114.8(5)
C(12)-C(11)-N(1)	113.8(8)
C(13)-C(12)-C(11)	116.1(12)
C(12)-C(13)-C(131)	112.3(16)
C(12)-C(13)-C(132)	109.5(13)
C(131)-C(13)-C(132)	109.9(14)
N(2)-C(21)-C(22)	105.6(9)
C(23)-C(22)-C(21)	115.5(12)
C(231)-C(23)-C(22)	128.3(15)
C(231)-C(23)-C(232)	114.5(15)
C(22)-C(23)-C(232)	113.8(13)

Table 4. Anisotropic displacement parameters (Å2x 103)for **1b**. The anisotropic displacement factor exponent takes the form: -2π2[h2a*2U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
S	81(1)	81(1)	164(2)	-2(1)	0(1)	2(1)
O(1)	109(3)	104(3)	254(5)	24(3)	-1(3)	4(2)
O(2)	70(2)	118(4)	253(5)	-20(3)	18(3)	5(2)
N(1)	96(4)	124(4)	135(4)	3(3)	-6(3)	-6(3)
N(2)	112(4)	135(5)	142(4)	-22(4)	1(3)	-10(3)
C(11)	142(7)	168(8)	188(8)	-62(7)	40(7)	-11(6)
C(12)	198(11)	279(17)	277(14)	16(14)	34(10)	24(9)
C(13)	239(12)	197(12)	231(12)	-19(10)	10(11)	26(10)
C(21)	171(9)	156(9)	161(8)	46(7)	-6(6)	-16(7)
C(22)	250(15)	220(17)	197(12)	65(11)	-27(9)	-66(10)
C(23)	319(18)	360(20)	144(9)	10(11)	-8(11)	-142(14)
C(131)	430(30)	620(30)	440(20)	-360(30)	190(20)	-160(20)
C(132)	360(20)	269(19)	430(20)	55(18)	26(14)	-3(15)
C(231)	340(20)	670(40)	350(20)	260(20)	-101(17)	-280(20)
C(232)	395(19)	430(20)	191(9)	61(12)	-77(11)	-166(14)

Table 5. Hydrogen coordinates (x 104) and isotropic displacement parameters (Å2x 103) for **1b**.

	x	y	z	U(eq)
H(1)	7282	-517	1875	420(90)
H(2)	9500	1468	2831	1600(1100)
H(11A)	9658	397	1651	340(70)
H(11B)	9755	-831	2006	390(100)
H(12A)	10053	-1575	1238	377
H(12B)	8797	-2204	1444	381
H(13)	7613	-660	1008	267
H(21A)	9122	-1087	2906	110(20)
H(21B)	7776	-542	3070	270(60)
H(22A)	10137	45	3536	360(110)
H(22B)	8889	829	3667	280(80)
H(23)	9540	-1663	3792	330
H(13A)	7824	-1651	271	744
H(13B)	8120	-2778	653	744
H(13C)	9240	-2066	376	744
H(13D)	9272	996	974	530
H(13E)	8569	796	479	530
H(13F)	9914	145	563	530
H(23A)	7684	-1969	3612	683
H(23B)	7304	-1324	4109	683
H(23C)	8213	-2578	4095	683
H(23D)	10316	-163	4328	507
H(23E)	9860	-1577	4514	507
H(23F)	9005	-301	4597	507

Table 6. Crystal data and structure refinement for **1c**.

Empirical formula	C14 H26 N2 O2 S	
Formula weight	286.43	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2	
Unit cell dimensions	a = 8.3975(8) Å	α = 90°.
	b = 6.4058(6) Å	β = 97.363(2)°.
	c = 29.530(3) Å	γ = 90°.
Volume	1575.4(3) Å3	
Z	4	
Density (calculated)	1.208 Mg/m3	
Absorption coefficient	0.207 mm-1	
F(000)	624	
Crystal size	0.40 x 0.20 x 0.10 mm3	
Theta range for data collection	0.70 to 27.96°.	
Index ranges	-9<=h<=11, -8<=k<=8, -35<=l<=38	
Reflections collected	5138	
Independent reflections	3373 [R(int) = 0.0799]	
Completeness to theta = 27.96°	97.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000000 and 0.549795	
Refinement method	Full-matrix least-squares on F2	
Data / restraints / parameters	3373 / 1 / 252	
Goodness-of-fit on F2	1.091	
Final R indices [I>2sigma(I)]	R1 = 0.1395, wR2 = 0.3041	
R indices (all data)	R1 = 0.1583, wR2 = 0.3296	
Absolute structure parameter	0.4(5)	
Extinction coefficient	0.0019(13)	
Largest diff. peak and hole	1.270 and -1.555 e.Å-3	

Table 7. Atomic coordinates (x 104) and equivalent isotropic displacement parameters (Å2x 103) for **1c**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S(1)	0	7431(11)	5000	84(2)
S(2)	0	-474(4)	0	0(1)
O(1)	1411(12)	8558(17)	5189(4)	72(3)
O(2)	-1377(13)	-1590(16)	-186(4)	69(3)
N(1)	585(14)	6015(19)	4610(4)	53(3)
N(2)	-552(15)	980(20)	402(4)	57(3)
C(11)	-303(15)	3955(17)	3909(4)	46(3)
C(12)	-1010(30)	5690(20)	3609(6)	74(5)
C(13)	-940(30)	5350(30)	3106(7)	86(6)
C(14)	-1710(30)	3320(30)	2942(6)	67(4)
C(15)	-1040(20)	1510(30)	3252(6)	71(4)
C(16)	-1140(20)	1910(30)	3740(7)	71(5)
C(17)	-501(19)	4192(17)	4402(5)	53(3)
C(21)	284(19)	3020(20)	1096(5)	53(3)
C(22)	960(20)	1240(20)	1393(5)	60(4)
C(23)	860(30)	1640(30)	1892(5)	80(6)
C(24)	1720(20)	3700(30)	2040(6)	70(4)
C(25)	1040(20)	5460(30)	1760(6)	73(4)
C(26)	1160(30)	5080(20)	1258(6)	74(5)
C(27)	520(20)	2550(30)	591(5)	61(4)

Table 8. Bond lengths [Å] and angles [°] for **1c**.

S(1)-O(1)	1.438(10)	O(2)#2-S(2)-N(2)#2	106.3(6)
S(1)-O(1)#1	1.438(10)	O(2)-S(2)-N(2)#2	107.6(6)
S(1)-N(1)#1	1.591(12)	N(2)-S(2)-N(2)#2	109.9(8)
S(1)-N(1)	1.591(12)	C(17)-N(1)-S(1)	119.6(9)
S(2)-O(2)#2	1.410(11)	C(27)-N(2)-S(2)	118.0(10)
S(2)-O(2)	1.410(11)	C(12)-C(11)-C(17)	114.6(12)
S(2)-N(2)	1.624(12)	C(12)-C(11)-C(16)	108.1(11)
S(2)-N(2)#2	1.624(12)	C(17)-C(11)-C(16)	107.4(12)
N(1)-C(17)	1.559(17)	C(11)-C(12)-C(13)	114.6(16)
N(2)-C(27)	1.416(17)	C(14)-C(13)-C(12)	111.8(17)
C(11)-C(12)	1.492(19)	C(13)-C(14)-C(15)	110.3(13)
C(11)-C(17)	1.495(18)	C(16)-C(15)-C(14)	113.0(15)
C(11)-C(16)	1.542(18)	C(15)-C(16)-C(11)	112.3(16)
C(12)-C(13)	1.51(3)	C(11)-C(17)-N(1)	109.2(10)
C(13)-C(14)	1.50(2)	C(22)-C(21)-C(26)	109.5(12)
C(14)-C(15)	1.54(2)	C(22)-C(21)-C(27)	109.1(13)
C(15)-C(16)	1.48(3)	C(26)-C(21)-C(27)	110.2(12)
C(21)-C(22)	1.51(2)	C(23)-C(22)-C(21)	112.1(13)
C(21)-C(26)	1.552(19)	C(22)-C(23)-C(24)	109.8(15)
C(21)-C(27)	1.556(19)	C(25)-C(24)-C(23)	111.5(12)
C(22)-C(23)	1.51(2)	C(24)-C(25)-C(26)	110.9(15)
C(23)-C(24)	1.54(2)	C(25)-C(26)-C(21)	110.8(14)
C(24)-C(25)	1.47(3)	N(2)-C(27)-C(21)	111.2(12)
C(25)-C(26)	1.52(2)		

O(1)-S(1)-O(1)#1	119.8(11)
O(1)-S(1)-N(1)#1	108.7(6)
O(1)#1-S(1)-N(1)#1	104.6(6)
O(1)-S(1)-N(1)	104.6(6)
O(1)#1-S(1)-N(1)	108.7(6)
N(1)#1-S(1)-N(1)	110.5(10)
O(2)#2-S(2)-O(2)	119.0(9)
O(2)#2-S(2)-N(2)	107.6(6)
O(2)-S(2)-N(2)	106.3(6)

Symmetry transformations used to generate equivalent atoms: #1 -x,y,-z+1 #2 -x,y,-z

Table 9. Anisotropic displacement parameters (Å2x 103) for **1c**. The anisotropic displacement factor exponent takes the form: -2_2[h2 a*2U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
S(1)	72(4)	79(4)	101(5)	0	15(3)	0
S(2)	0(1)	0(1)	0(1)	0	-1(1)	0
O(1)	65(6)	64(6)	86(7)	-21(5)	5(5)	-27(5)
O(2)	97(8)	45(5)	74(6)	-1(5)	43(6)	-15(6)
N(1)	45(6)	53(6)	63(6)	-10(5)	17(5)	-11(5)
N(2)	56(6)	58(7)	61(6)	-2(5)	24(5)	-15(6)
C(11)	48(6)	38(6)	49(6)	-6(4)	-2(5)	-7(4)
C(12)	89(12)	39(8)	90(11)	-2(8)	-3(10)	13(8)
C(13)	110(16)	67(10)	74(11)	8(8)	-20(11)	-38(11)
C(14)	90(12)	56(9)	53(8)	-1(7)	-3(8)	-12(9)
C(15)	84(11)	48(8)	74(10)	-14(7)	-11(8)	8(8)
C(16)	70(10)	43(8)	96(12)	-8(8)	-10(9)	-12(8)
C(17)	74(8)	21(8)	64(7)	-6(5)	10(6)	-7(5)
C(21)	75(9)	38(6)	47(6)	-2(5)	7(6)	-5(6)
C(22)	75(10)	41(7)	60(8)	-1(6)	-4(7)	3(7)
C(23)	145(18)	44(8)	47(8)	8(6)	0(9)	-8(10)
C(24)	69(9)	79(10)	58(8)	-8(7)	-13(8)	-9(8)
C(25)	79(11)	50(7)	86(11)	-21(8)	-2(9)	6(8)
C(26)	123(15)	43(9)	57(8)	-2(5)	15(9)	-19(8)
C(27)	62(8)	65(9)	57(7)	-18(7)	9(6)	-31(7)

Table 10. Hydrogen coordinates (x 104) and isotropic displacement parameters (Å2 x 10 3) for **1c**.

	x	y	z	U(eq)
H(1)	1490(190)	6300(300)	4520(50)	69
H(2)	-1470(190)	800(300)	500(50)	74
H(12A)	-400(200)	7000(400)	3700(70)	96
H(12B)	-2100(300)	5900(400)	3660(70)	96
H(13A)	-1500(300)	6500(500)	2930(80)	112
H(13B)	200(300)	5400(500)	3050(70)	112
H(14A)	-1500(200)	3100(400)	2630(60)	88
H(14B)	-2900(200)	3400(400)	2940(60)	88
H(15A)	-1600(200)	200(400)	3160(70)	92
H(15B)	100(300)	1300(400)	3210(60)	92
H(16A)	-600(200)	800(400)	3920(70)	93
H(16B)	-2300(300)	2000(400)	3790(60)	93
H(17A)	-200(190)	2900(300)	4560(50)	69
H(17B)	-1620(160)	4500(400)	4430(40)	69
H(22A)	380(180)	0(400)	1300(50)	78
H(22B)	2100(200)	1000(400)	1350(60)	78
H(23A)	1400(300)	500(400)	2070(70)	104
H(23B)	-300(300)	1700(500)	1940(70)	104
H(24A)	1600(200)	4000(400)	2360(60)	91
H(24B)	2900(200)	3600(400)	2010(60)	91
H(25A)	1600(300)	6700(400)	1860(70)	95
H(25B)	-100(300)	5700(400)	1800(60)	95
H(26A)	2300(200)	5000(400)	1210(60)	96
H(26B)	700(300)	6200(400)	1080(70)	96
H(27A)	340(180)	3800(300)	410(50)	80
H(27B)	1600(200)	2100(300)	580(50)	80

Table 11. Crystal data and structure refinement for **1d**.

Empirical formula	C10 H24 N2 O2 S	
Formula weight	236.37	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 8.7661(3) Å	α = 90°.
	b = 5.7893(2) Å	β = 98.059(2)°.
	c = 27.56600(10) Å	γ = 90°.
Volume	1385.15(7) Å3	
Z	4	
Density (calculated)	1.133 Mg/m3	
Absorption coefficient	0.221 mm-1	
F(000)	520	
Crystal size	0.30 x 0.20 x 0.02 mm3	
Theta range for data collection	2.99 to 27.95°.	
Index ranges	-10<=h<=11, -7<=k<=5, -36<=l<=35	
Reflections collected	4262	
Independent reflections	1591 [R(int) = 0.0412]	
Completeness to theta = 27.95°	96.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000000 and 0.433246	
Refinement method	Full-matrix least-squares on F2	
Data / restraints / parameters	1591 / 0 / 83	
Goodness-of-fit on F2	1.174	
Final R indices [I>2sigma(I)]	R1 = 0.0769, wR2 = 0.1725	
R indices (all data)	R1 = 0.0906, wR2 = 0.1812	
Extinction coefficient	0.0023(15)	
Largest diff. peak and hole	0.362 and -0.405 e.Å-3	

Table 12. Atomic coordinates (x 104) and equivalent isotropic displacement parameters (Å2x 103) for **1d**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S	5000	3681(2)	2500	46(1)
O	3841(2)	2441(4)	2708(1)	60(1)
N	5921(3)	5250(4)	2929(1)	49(1)
C(1)	5028(3)	6839(6)	3199(1)	59(1)
C(2)	6060(4)	8207(7)	3565(1)	65(1)
C(3)	5240(4)	9793(7)	3873(1)	67(1)
C(4)	6239(6)	11216(11)	4233(2)	114(2)
C(5)	5431(7)	12820(11)	4538(2)	118(2)

Table 13. Bond lengths [Å] and angles [°] for **1d**.

S-O	1.429(2)
S-O#1	1.4287(19)
S-N#1	1.614(2)
S-N	1.614(2)
N-C(1)	1.476(3)
C(1)-C(2)	1.486(5)
C(2)-C(3)	1.500(4)
C(3)-C(4)	1.479(5)
C(4)-C(5)	1.496(6)
O-S-O#1	119.67(19)
O-S-N#1	105.41(12)
O#1-S-N#1	107.46(12)
O-S-N	107.46(12)
O#1-S-N	105.41(12)
N#1-S-N	111.47(18)
C(1)-N-S	118.31(18)
N-C(1)-C(2)	111.1(2)
C(1)-C(2)-C(3)	114.6(3)
C(4)-C(3)-C(2)	115.8(3)
C(3)-C(4)-C(5)	116.1(4)

Symmetry transformations used to generate equivalent atoms:

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

Table 14. Anisotropic displacement parameters (Å2x 103)for **1d**. The anisotropic displacement factor exponent takes the form: -2_2[h2a*2U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
S	34(1)	36(1)	69(1)	0	12(1)	0
O	44(1)	48(1)	89(2)	8(1)	16(1)	-10(1)
N	33(1)	48(1)	68(2)	-5(1)	9(1)	1(1)
C(1)	46(2)	59(2)	73(2)	-15(2)	12(1)	3(1)
C(2)	50(2)	67(2)	79(2)	-17(2)	10(2)	-1(2)
C(3)	61(2)	67(2)	72(2)	-10(2)	10(2)	5(2)
C(4)	83(3)	127(4)	126(4)	-67(4)	-8(3)	21(3)
C(5)	122(4)	121(4)	106(3)	-49(4)	0(4)	36(4)

Table 15. Hydrogen coordinates (x 104) and isotropic displacement parameters (Å2x 103) for **1d**.

	x	y	z	U(eq)
H(0)	6907	5164	2997	280(50)
H(1A)	4314	5956	3365	116(16)
H(1B)	4432	7878	2970	82(12)
H(2A)	6744	9120	3394	128(18)
H(2B)	6692	7149	3780	107(15)
H(3A)	4588	10820	3656	110(15)
H(3B)	4572	8870	4048	134(19)
H(4A)	6914	12124	4058	260(50)
H(4B)	6884	10187	4451	180(30)
H(5A)	6179	13618	4766	160(20)
H(5B)	4756	11951	4715	160(30)
H(5C)	4842	13922	4329	160(30)

Table 16. Crystal data and structure refinement for **1e**.

Empirical formula	C12 H28 N2 O2 S	
Formula weight	264.42	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 8.878(3) Å	α= 90°.
	b = 5.768(2) Å	β= 97.676(6)°.
	c = 31.015(10) Å	γ = 90°.
Volume	1574.0(9) Å3	
Z	4	
Density (calculated)	1.116 Mg/m3	
Absorption coefficient	0.201 mm-1	
F(000)	584	
Crystal size	0.60 x 0.40 x 0.04 mm3	
Theta range for data collection	2.65 to 23.16°.	
Index ranges	-9<=h<=9, -5<=k<=6, -32<=l<=34	
Reflections collected	3310	
Independent reflections	1102 [R(int) = 0.0581]	
Completeness to theta = 23.16°	98.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00 and 0.34	
Refinement method	Full-matrix least-squares on F2	
Data / restraints / parameters	1102 / 0 / 93	
Goodness-of-fit on F2	1.125	
Final R indices [I>2sigma(I)]	R1 = 0.0731, wR2 = 0.1721	
R indices (all data)	R1 = 0.0768, wR2 = 0.1749	
Extinction coefficient	0.010(3)	
Largest diff. peak and hole	0.402 and -0.430 e.Å-3	

Table 17. Atomic coordinates (x 104) and equivalent isotropic displacement parameters (Å2x 103) for **1e**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S	5000	3102(2)	7500	37(1)
O	3684(3)	4348(5)	7312(1)	48(1)
N	4441(3)	1498(6)	7878(1)	42(1)
C(1)	5551(4)	-59(8)	8130(1)	46(1)
C(2)	4785(4)	-1588(8)	8425(1)	50(1)
C(3)	5879(4)	-3074(9)	8721(1)	54(1)
C(4)	5130(5)	-4705(9)	9010(2)	58(1)
C(5)	6188(6)	-6132(11)	9316(2)	82(2)
C(6)	5429(8)	-7795(13)	9596(2)	100(2)

Table 18. Bond lengths [Å] and angles [°] for **1e**.

S-O#1	1.429(3)
S-O	1.429(2)
S-N#1	1.623(3)
S-N	1.623(3)
N-C(1)	1.477(5)
C(1)-C(2)	1.498(6)
C(2)-C(3)	1.511(6)
C(3)-C(4)	1.513(6)
C(4)-C(5)	1.491(6)
C(5)-C(6)	1.513(7)
O#1-S-O	119.6(2)
O#1-S-N#1	105.66(15)
O-S-N#1	107.69(15)
O#1-S-N	107.69(15)
O-S-N	105.66(15)
N#1-S-N	110.5(2)
C(1)-N-S	118.8(2)
N-C(1)-C(2)	110.8(3)
C(1)-C(2)-C(3)	113.4(3)
C(2)-C(3)-C(4)	114.6(3)
C(5)-C(4)-C(3)	115.6(4)
C(4)-C(5)-C(6)	115.1(5)

Symmetry transformations used to generate equivalent atoms:

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

Table 19. Anisotropic displacement parameters (Å2x 103) for **1e**. The anisotropic displacement factor exponent takes the form: -2π2[h2 a*2U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
S	18(1)	42(1)	49(1)	0	-5(1)	0
O	30(1)	47(2)	66(2)	7(1)	-3(1)	11(1)
N	22(2)	53(2)	48(2)	1(2)	-1(1)	5(2)
C(1)	24(2)	58(2)	54(2)	5(2)	-6(2)	4(2)
C(2)	34(2)	63(3)	53(2)	8(2)	1(2)	3(2)
C(3)	37(2)	69(3)	55(2)	7(2)	1(2)	3(2)
C(4)	40(2)	70(3)	64(3)	13(3)	3(2)	1(2)
C(5)	67(3)	97(4)	78(3)	34(3)	-6(3)	0(3)
C(6)	110(5)	104(5)	84(4)	40(4)	0(4)	-13(5)

Table 20. Hydrogen coordinates (x 104) and isotropic displacement parameters (Å2x 10 3) for **1e**.

	x	y	z	U(eq)
H(0)	3515	1553	7931	150(30)
H(1A)	6328	859	8301	50(11)
H(1B)	6042	-1010	7931	56(12)
H(2A)	4069	-2587	8249	69(14)
H(2B)	4213	-626	8601	68(14)
H(3A)	6569	-2067	8903	66(13)
H(3B)	6480	-3982	8543	65(14)
H(4A)	4498	-3798	9178	95(18)
H(4B)	4467	-5743	8826	74(15)
H(5A)	6836	-5096	9505	130(30)
H(5B)	6837	-7015	9148	120(30)
H(6A)	6191	-8619	9785	110(20)
H(6B)	4819	-8878	9414	150(30)
H(6C)	4794	-6943	9767	150(30)

Table 21. Crystal data and structure refinement for **1f**.

Identification code	bg20a	
Empirical formula	C16 H36 N2 O2 S	
Formula weight	320.53	
Temperature	223(2) K	
Wavelength	0.71073 ≈	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 8.9629(2) ≈	α= 90°.
	b = 5.6446(2) ≈	β= 94.8950(10)°.
	c = 37.6934(8) ≈	γ = 90°.
Volume	1900.03(9) $≈^3$	
Z	4	
Density (calculated)	1.121 Mg/m^3	
Absorption coefficient	0.177 mm^{-1}	
F(000)	712	
Crystal size	0.48 x 0.40 x 0.17 mm^3	
Theta range for data collection	4.78 to 27.08∞.	
Index ranges	-11<=h<=11, -7<=k<=7, -47<=l<=47	
Reflections collected	7095	
Independent reflections	1951 [R(int) = 0.0766]	
Completeness to theta = 27.08∞	93.2 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1951 / 0 / 169	
Goodness-of-fit on F^2	1.167	
Final R indices [I>2sigma(I)]	R1 = 0.0397, wR2 = 0.1032	
R indices (all data)	R1 = 0.0444, wR2 = 0.1060	
Extinction coefficient	0.0011(10)	
Largest diff. peak and hole	0.238 and -0.308 $e.≈^{-3}$	

Table 22. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($\approx^2$x 10^3) for **1f**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S	0	755(1)	2500	25(1)
O	-1151(1)	-513(2)	2663(1)	34(1)
N(1)	872(1)	2364(2)	2809(1)	29(1)
C(2)	-13(2)	3951(3)	3020(1)	32(1)
C(3)	1021(2)	5603(3)	3243(1)	33(1)
C(4)	203(2)	7119(3)	3498(1)	34(1)
C(5)	1244(2)	8823(3)	3713(1)	36(1)
C(6)	468(2)	10297(3)	3983(1)	38(1)
C(7)	1508(2)	11993(3)	4198(1)	40(1)
C(8)	750(3)	13419(4)	4473(1)	54(1)
C(9)	1776(3)	15177(5)	4676(1)	64(1)

Table 3. Bond lengths [≈] and angles [∞] for **23**.

S-O	1.4351(10)
S-O#1	1.4351(10)
S-N(1)	1.6235(12)
S-N(1)#1	1.6235(12)
N(1)-C(2)	1.4746(18)
N(1)-H	0.819(19)
C(2)-C(3)	1.516(2)
C(2)-H(2A)	0.97(2)
C(2)-H(2B)	0.95(2)
C(3)-C(4)	1.521(2)
C(3)-H(3A)	0.96(2)
C(3)-H(3B)	0.99(2)
C(4)-C(5)	1.525(2)
C(4)-H(4A)	0.94(2)
C(4)-H(4B)	0.972(19)
C(5)-C(6)	1.526(2)
C(5)-H(5A)	0.989(19)
C(5)-H(5B)	0.943(19)
C(6)-C(7)	1.522(2)
C(6)-H(6A)	1.00(2)
C(6)-H(6B)	0.93(2)
C(7)-C(8)	1.515(2)
C(7)-H(7A)	1.00(2)
C(7)-H(7B)	0.92(2)
C(8)-C(9)	1.516(3)
C(8)-H(8A)	0.98(3)
C(8)-H(8B)	0.93(2)
C(9)-H(9A)	0.87(3)
C(9)-H(9B)	0.97(3)
C(9)-H(9C)	1.01(3)
O-S-O#1	120.14(9)
O-S-N(1)	107.01(6)
O#1-S-N(1)	105.40(6)
O-S-N(1)#1	105.40(6)

O#1-S-N(1)#1	107.01(6)
N(1)-S-N(1)#1	111.97(9)
C(2)-N(1)-S	118.62(10)
C(2)-N(1)-H	114.3(13)
S-N(1)-H	111.4(13)
N(1)-C(2)-C(3)	109.95(12)
N(1)-C(2)-H(2A)	106.8(12)
C(3)-C(2)-H(2A)	110.7(12)
N(1)-C(2)-H(2B)	110.7(11)
C(3)-C(2)-H(2B)	112.0(12)
H(2A)-C(2)-H(2B)	106.5(16)
C(2)-C(3)-C(4)	113.02(12)
C(2)-C(3)-H(3A)	109.4(11)
C(4)-C(3)-H(3A)	110.2(12)
C(2)-C(3)-H(3B)	109.5(11)
C(4)-C(3)-H(3B)	110.1(11)
H(3A)-C(3)-H(3B)	104.2(16)
C(3)-C(4)-C(5)	112.67(13)
C(3)-C(4)-H(4A)	107.1(12)
C(5)-C(4)-H(4A)	110.1(12)
C(3)-C(4)-H(4B)	109.9(11)
C(5)-C(4)-H(4B)	110.8(11)
H(4A)-C(4)-H(4B)	106.0(16)
C(4)-C(5)-C(6)	113.94(13)
C(4)-C(5)-H(5A)	110.3(11)
C(6)-C(5)-H(5A)	110.9(11)
C(4)-C(5)-H(5B)	109.5(12)
C(6)-C(5)-H(5B)	108.2(11)
H(5A)-C(5)-H(5B)	103.5(16)
C(7)-C(6)-C(5)	113.96(14)
C(7)-C(6)-H(6A)	109.1(12)
C(5)-C(6)-H(6A)	111.6(11)
C(7)-C(6)-H(6B)	109.3(13)
C(5)-C(6)-H(6B)	108.5(13)
H(6A)-C(6)-H(6B)	103.8(16)
C(8)-C(7)-C(6)	114.14(15)
C(8)-C(7)-H(7A)	108.9(12)

C(6)-C(7)-H(7A)	109.6(12)
C(8)-C(7)-H(7B)	108.9(13)
C(6)-C(7)-H(7B)	110.6(13)
H(7A)-C(7)-H(7B)	104.2(17)
C(7)-C(8)-C(9)	113.91(19)
C(7)-C(8)-H(8A)	106.5(14)
C(9)-C(8)-H(8A)	111.1(15)
C(7)-C(8)-H(8B)	107.5(15)
C(9)-C(8)-H(8B)	111.0(15)
H(8A)-C(8)-H(8B)	106(2)
C(8)-C(9)-H(9A)	110(2)
C(8)-C(9)-H(9B)	112.0(16)
H(9A)-C(9)-H(9B)	109(3)
C(8)-C(9)-H(9C)	110.5(14)
H(9A)-C(9)-H(9C)	113(2)
H(9B)-C(9)-H(9C)	102(2)

Symmetry transformations used to generate equivalent atoms:

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

Table 24. Anisotropic displacement parameters ($\approx^2$x 10^3)for **1f**. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S	23(1)	22(1)	31(1)	0	5(1)	0
O	31(1)	31(1)	42(1)	4(1)	7(1)	-6(1)
N(1)	23(1)	31(1)	33(1)	-3(1)	5(1)	-2(1)
C(2)	27(1)	33(1)	36(1)	-6(1)	7(1)	2(1)
C(3)	28(1)	34(1)	38(1)	-6(1)	5(1)	1(1)
C(4)	29(1)	37(1)	38(1)	-6(1)	6(1)	1(1)
C(5)	31(1)	36(1)	42(1)	-8(1)	4(1)	1(1)
C(6)	33(1)	40(1)	41(1)	-9(1)	5(1)	1(1)
C(7)	37(1)	39(1)	43(1)	-9(1)	3(1)	-1(1)
C(8)	53(1)	57(1)	53(1)	-22(1)	11(1)	-7(1)
C(9)	78(2)	59(1)	54(1)	-23(1)	7(1)	-15(1)

Table 25. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10^3) for **1f**.

	x	y	z	U(eq)
H	1620(20)	2980(30)	2740(5)	40(5)
H(2A)	-570(20)	2950(40)	3171(5)	52(5)
H(2B)	-740(20)	4800(40)	2870(5)	48(5)
H(3A)	1550(20)	6590(40)	3088(5)	50(5)
H(3B)	1820(20)	4670(30)	3376(5)	48(5)
H(4A)	-250(20)	6080(40)	3651(5)	50(5)
H(4B)	-610(20)	7970(30)	3368(5)	40(5)
H(5A)	2110(20)	7960(30)	3830(5)	43(5)
H(5B)	1680(20)	9870(30)	3557(5)	46(5)
H(6A)	-400(20)	11210(40)	3866(5)	51(5)
H(6B)	30(20)	9270(40)	4137(5)	54(6)
H(7A)	2360(20)	11090(40)	4321(5)	55(6)
H(7B)	1960(20)	13020(40)	4051(6)	53(6)
H(8A)	-100(30)	14230(40)	4344(6)	75(7)
H(8B)	350(30)	12350(40)	4627(6)	73(7)
H(9A)	1300(40)	15880(60)	4840(9)	113(11)
H(9B)	2150(30)	16370(50)	4521(7)	86(8)
H(9C)	2720(30)	14370(40)	4778(6)	72(7)

Table 26. Torsion angles [°] for **1f**.

O-S-N(1)-C(2)	-52.84(12)
O#1-S-N(1)-C(2)	178.18(11)
N(1)#1-S-N(1)-C(2)	62.18(10)
S-N(1)-C(2)-C(3)	-169.52(10)
N(1)-C(2)-C(3)-C(4)	-173.88(13)
C(2)-C(3)-C(4)-C(5)	-178.42(14)
C(3)-C(4)-C(5)-C(6)	-177.57(14)
C(4)-C(5)-C(6)-C(7)	179.85(14)
C(5)-C(6)-C(7)-C(8)	-178.66(17)
C(6)-C(7)-C(8)-C(9)	-177.69(19)

Symmetry transformations used to generate equivalent atoms:

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

Table 27. Hydrogen bonds for **1f** [Å and deg.].

D-H•••A	d(D-H)	d(D•••A)	d(D•••A)	<(DHA)
N(1)-H•••O#2	0.819(19)	2.212(19)	3.0184(16)	168.5(18)

Symmetry transformations used to generate equivalent atoms:

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