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Synthesis, characterization and crystal structure of 2-chloroethyl (methylsulfonyl) methanesulfonate

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Table S1. Crystal data and structure refinement results for2-chloro (methylsulfonyl) methanesulfonate.

Empirical formula	C4 H9 Cl O5 S2
Formula weight	236.68
Temperature	296(2) K
Wavelength	1.54184 Å
Crystal system	Monoclinic
Space group	P 2_1
Unit cell dimensions	a = 9.4479(7) Å
	b = 5.5001(3) Å
	c = 10.0650(7) Å
	β = 112.633(9)°
Volume	482.74(6) $Å^3$
Z, density (calculated)	2, 1.628 Mg/m^3
Absorption coefficient	7.485 mm^{-1}
F(000)	244
Crystal size	0.305 x 0.162 x 0.080 mm^3
ϑ-range for data collection	4.760 to 70.824°
Index ranges	$-11 \leq h \leq 10$, $-6 \leq k \leq 5$, $-11 \leq l \leq 12$
Reflections collected	1784
Independent reflections	1263 [R(int) = 0.0299]
Observed reflections [I>2σ(I)]	1186
Completeness to ϑ = 67.684°	99.9 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1263 / 1 / 110
Goodness-of-fit on F^2	1.081
Final R indices[a][I>2σ(I)]	R1 = 0.0427, wR2 = 0.1081
R indices (all data)	R1 = 0.0459, wR2 = 0.1149
Absolute structure parameter	0.09(5)

Largest diff. peak and hole	0.659 and -0.296 e.Å^{-3}

[a] $R_1=\Sigma||F_o|-|F_c||/\Sigma|F_o|$, $wR_2=[\Sigma w(|F_o|^2-|F_c|^2)^2/\Sigma w(|F_o|^2)^2]^{1/2}$

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 2-chloro (methylsulfonyl) methanesulfonate. To be deposited.

Atom	x	y	z	U(eq)
C(1)	6736(10)	8152(16)	5522(8)	69(2)
C(2)	7937(6)	5995(12)	3642(5)	42(1)
C(3)	7340(7)	3302(14)	468(6)	57(2)
C(4)	7466(7)	4540(15)	-783(6)	58(2)
O(3)	6392(5)	4877(9)	975(4)	55(1)
O(11)	7742(7)	3732(9)	5872(5)	70(1)
O(12)	9607(6)	7019(14)	6272(5)	84(2)
O(21)	6532(6)	1816(9)	2768(5)	64(1)
O(22)	4990(5)	5504(12)	2465(4)	67(2)
S(1)	8111(2)	6057(3)	5487(1)	49(1)
S(2)	6328(2)	4328(3)	2477(1)	46(1)
Cl	8599(2)	7216(5)	-285(3)	84(1)

Table S3. Anisotropic displacement parameters ($Å^2$ x 10^3) for 2-chloro (methylsulfonyl) methanesulfonate. To be deposited.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	97(5)	57(4)	72(4)	-9(4)	53(4)	-1(4)
C(2)	44(2)	45(3)	41(2)	-4(2)	20(2)	-2(2)
C(3)	69(3)	59(4)	40(3)	-6(3)	18(2)	4(3)
C(4)	53(3)	75(5)	45(2)	-3(3)	19(2)	1(3)
O(3)	59(2)	68(3)	36(2)	3(2)	17(2)	11(2)
O(11)	108(4)	52(3)	49(2)	5(2)	31(2)	-7(3)
O(12)	74(3)	117(5)	51(2)	-15(3)	12(2)	-34(3)
O(21)	85(3)	50(3)	52(2)	-1(2)	22(2)	-16(2)
O(22)	45(2)	99(4)	54(2)	3(2)	18(2)	1(2)
S(1)	58(1)	46(1)	38(1)	1(1)	16(1)	-8(1)
S(2)	47(1)	55(1)	35(1)	2(1)	14(1)	-5(1)
Cl	75(1)	79(1)	105(2)	-1(1)	41(1)	-10(1)

Table S4. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for the 2-chloro (methylsulfonyl) methanesulfonate. To be deposited.

Atom	x	y	z	U(eq)
H(1A)	5730	7529	4976	104
H(1B)	6878	9661	5110	104
H(1C)	6845	8418	6499	104
H(2A)	7864	7652	3293	51
H(2B)	8862	5286	3605	51
H(3A)	6861	1722	189	68
H(3B)	8348	3077	1221	68
H(4A)	6448	4961	-1462	69
H(4B)	7917	3433	-1260	69

Table S5.Theoretical and experimental geometric parameter of the Clomesone.

		Dimer R7		Monomer			
	X-ray		6-311G(3df)	6-311++(d,p)	6-311G(d,p)	6-31G(d)	
Distances/Å	298K[a]	Unit 1	Unit 2				
C(1) H prom	0,96	1,089	1,089	1,089	1,089	1,091	1,092
C(1) S(1)	1,747	1,78	1,78	1,782	1,802	1,803	1,804
S(19) O(11)	1,418	1,442	1,436	1,435	1,459	1,464	1,465
S(1) O(12)	1,429	1,437	1,450	1,439	1,464	1,468	1,468
S(1) C(2)	1,801	1,817	1,808	1,823	1,844	1,842	1,843
C(2) H(2A, 2B)	0,97	1,091	1,090	1,090	1,089	1,091	1,092
C(2) S(2)	1,776	1,798	1,799	1,797	1,817	1,815	1,816
S(2) O(21)	1,410	1,432	1,427	1,427	1,450	1,456	1,456
S(2) O(22)	1,416	1,427	1,427	1,427	1,451	1,457	1,457
S(2) O(3)	1,566	1,584	1,590	1,602	1,644	1,647	1,647
C(3) O(3)	1,471	1,458	1,452	1,445	1,450	1,448	1,447
C(3) H(3A, 3B)	0,97	1,09	1,091	1,091	1,092	1,094	1,094
C(3) C(4)	1,476	1,505	1,307	1,507	1,510	1,513	1,514
C(4) H(4A, 4B)	0,97	1,088	1,088	1,089	1,089	1,091	1,092
C(4) Cl	1,775	1,801	1,800	1,802	1,813	1,812	1,812
RMSD/Å							
Angles/º							
C(1) H prom	109,5	111	111,1	111,0	111,3	111,1	111,0
H C(1) S (1)	109,5	107,9	107,7	107,9	107,5	107,8	107,9
C(1) S(1) O(11)	109,1	108,6	109,5	108,9	109	108,7	108,7
C(1) S(1) O(12)	109,2	108,9	108	108,6	108,6	108,7	108,7
C(1) S(1) C(2)	105	105,4	105,1	104,7	104,9	104,3	104,3
O(11) S(1) C(2)	109,3	108	109,7	108,6	108,6	108,2	108,3
O(12) S(1) C(2)	104,8	104,9	104,3	104,0	103,5	104	104,0
S(1) C(5) S(2)	113,9	116	116,6	116,4	116,2	115,8	115,8
C(2) H(2A, 3B)	107,6	110,8	109,9	109,9	110,5	109,9	109,8
C(2) S(2) O(21)	110,8	109,6	109,3	110,2	110,8	110,8	110,7
C(2) S(2) O(22)	107,7	108,1	108,7	108,1	108,2	108,1	108,1
C(2) S(2) O(3)	101,3	100,7	99,9	99,4	98,0	97,9	97,8

O(21) S(2) O(3)	109,6	109,4	107,4	109,1	108,9	109,2	109,2
O(22) S(2) O(3)	105,6	105,9	105,9	105,7	106,2	106,1	106,1
S(2) O(3) C(3)	118,4	119,9	119,9	121,1	120,6	119,5	119,6
C(3) H(3A,3B)	108,7	109,3	110,1	108,9	109,1	109	109,0
O(3) C(3) C(4)	106,5	107,4	108,3	109,0	108,9	109,1	109,1
C(3) C(4) Cl	112,3	112,4	112,8	112,3	112,1	112,3	112,2
C(4) H(4A,4B)	107,9	109,7	109,7	109,6	109,7	109,5	109,5
S(1) O_2	118,5	120,1	119,3	120,9	121,1	121,5	121,5
S(2) O_2	120	121	127,7	122,0	122,0	122	122,0
RMSD/º							
Dihedral Angles/º							
C(1) S(1) C(2) S(2)	-76,50	-69,5	-70,9	-70,7	-69,7	-71,3	-71,5
S(1) C(2) S(2) O(3)	173,60	175,4	165,9	168,1	164,0	167,1	167,5
C(2) S(2) O(3) C(3)	87,20	87,6	78,4	106,6	115,2	115,1	115,3
S(2) O(3) C(3) C(4)	-167,60	-176,7	-152,2	-142,0	-137,7	-135,9	-136,2
O(3)C(3) C(4) Cl	69,40	68,3	67,7	69,8	69,5	69,2	69,2
O(11) S(1) C(2) S(2)	40,40	48,4	46,7	45,5	46,6	44,3	44,1
O(12) S(1) C(2) S(2)	168,40	177,6	175,5	175,4	176,5	174,8	174,6
0(21) S(2) C(2) S(1)	-70,10	-69,2	-78,9	-77,4	-82,2	-78,8	-78,4
O(22) S(2) C(2) S(1)	63,00	64,5	56,1	58,0	53,9	57,3	57,6
O(21) S(2) O(3) C(3)	-29,90	-27,8	-36,4	-8,7	0,0	0,2	0,2
O(22) S(2) O(3) C(3)	-160,50	-159,8	-169	-141,5	-133,1	-133,5	-133,3
RSMD/º							
RSMD/total		2.17	3.49	5,09	6,78	6,63	6,61

Table S6. Theoretical inter / intramolecular hydrogen bonds of the dimer form for Clomesone, calculated at B3LYP/6-311(3df) level.

B3LYP/6-311G(3df)	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(1)-H(1A)...O(22)	1.08	2.47	3.21	124.1
C(2)-H(2B)...O(12)#3	1.09	2.22	3.27	149.0
C(3)-H(3B)...O(12)#3	1.09	2.57	3.55	160.2

Table S7. Observed and Calculated Frequencies, Infrared and Raman Intensities for Clomesone.

Mode	*Observed*		*Calculated*				*Approximate description of mode*[f]
	IR[a]	*Raman*[b]	*B3LYP/6-311(3df)*	*Scaled*[c]	*IR intensity*[d]	*Raman activity*[e]	
	3438 (w,br)						ν_a O----H
1	3038 (w)	3038(31)	3183	3042	1.8	27	ν_a C(1)H_3
2	3038 (w)	3038(31)	3167	3028	2.0	32	ν_a C(2)H_2
3	3038 (w)	3038 (31)	3156	3020	1,2	58	ν_a C(1)H_3
4	3022 (vw)	3022(56)	3152	3016	3.1	49	ν_a C(4)H_2
5	3022(vw)	3022(56)	3132	2998	2,5	44	ν_a C(3)H_2
6	2990(m)	2991(35)	3096	2970	2,4	66	ν_s C(2)H_2
7	2990(m)	2977(100)	3091	2963	16	165	ν_s C(4)H_2
8	2938(vw)	2939(83)	3065	2936	1.1	133	ν_s C(1)H_3
9	2928(w)	2925(76)	3057	2932	20	122	ν_s C(3)H_2
10	1462(w)	1459(14)	1502	1476	6	7	δ C(3)H_2
11	1430(w)	1430(25)	1469	1442	6	6	δ C(4)H_2
12	1414(vw)	1413(96)	1459	1431	5	3	δ_a C(1)H_3
13	1390(vw)	1390(5)	1451	1410	5	6	δ_a C(1)H_3
14	1370(m)	1372(13)	1425	1406	165	3	ν_a S(2)O_2
15	1361(m)	1365(13)	1405	1383	14	5	δ C(2)H_2
16	1354(sh)	1357(6)	1402	1377	29	3	ω C(3)H_2
17	1318(s)	1318(16)	1385	1367	180	5	ν_a S(1)O_2
18	1301(sh)	1300(10)	1350	1330	23	1	δs C(1)H_3
19	1253(vw)	1253 (10)	1333	1315	24	1	ω C(4)H_2
20	1237(w)	1241(sh)	1271	1240	0,5	6	tω C(3)H_2
21	1200(w)	1199(4)	1231	1205	55	2	ω C(2)H_2
22	1186(m)	-	1223	1195	14	1,5	tω C(4)H_2
23	1177(m)	1177(84)	1202	1189	97	19	ν_s S(2)O_2
24	1166(m)	1160(20)	1179	1166	112	6	ν_s S(1)O_2

25	1125(w)	1121(28)	1120	1108	9	5	tω $C(2)H_2$
26	1072(w)	1072(4)	1087	1075	60	2	νC(4)-C(3)
27	988(m)	988 sh	1033	1012	151	3	νC(3)-O(3)
28	982(sh)	982(22)	984	966	32	0,8	ρ C(1)H3
29	956(m)	954(7)	977	967	4	2	ρ C(1)H3
30	916(m)	913(9)	964	956	68	3	$\rho C(3)H_2$
31	870(m)	870(5)	890	886	136	0,8	ρ $C(4)H_2$
32	802(sh)	804 sh	863	856	45	1	ρ $C(2)H_2$
33	790(m)	794(24)	763	762	170	5	νC(2)-S(2)
34	766(w)	765sh	747	746	128	9	νC(1)-S(1)
35	758(w)	758(40)	730	730	12	5	νS(2)-O(3)
36	699(vw)	702(77)	670	670	7	24	ν C(2)-S(1)
37	667(w)	666(79)	660	660	26	14	νC(3)-Cl
38	598(w)	600 (5)	600	600	26	1	ω $S(2)O_2$
39	528(m)	528(15)	516	516	37	2	δ $S(2)O_2$
40	507(m)	506(34)	500	500	38	1,3	δ $S(1)O_2$
41	464(m)	464 sh	456	456	25	1	ω $S(1)O_2$
42	459(w)	459(31)	453	449	30	3	ρ $S(2)O_2$
43	419(w)	418(23)	406	403	16	4	δ Cl-C(4)-C(3)
44	-	364(25)	342	340	1	2	ρ $S(1)O_2$
45	-	352 sh	332	332	3	0,6	tω S(2) O2
46	-	294(11)	281	281	3	2	δ O(3)-C(3)-C(4)
47	-	271(83)	260	260	1,6	5	tω $S(1)O_2$
48	-	246(57)	232	232	1,6	1,4	δ C(1)S(1)C(2)
49	-	202(11)	222	122	1,4	4	δ C(2)-S(2)-O(3)
50	-	193 sh	206	106	1,2	1	δ S(2)-O(3)-C(3)
51	-	169(7)	184	-	1,3	0	τ C(1)-S(1)
52	-	138 sh	145	-	3	1	δ S(2)-C(2)-S(1)
53	-	108(30)	106	-	4	1	τ C(4)-C(3)
54	-	-	84	-	3	0	τ C(3)-S(1)

55	-	-	38	-	5	0	τ S(1)-C(2)
56	-	-	32	-	1	0	τ C(3)-O(3)
57	-	-	20	-	3	0	τ S(2)-O(3)

[a] sh, shoulder; br, broad; s, strong; w, weak; m, medium; v, very. [b] Relative band heights in parentheses. [c] Yoshida scaled method. [d] Units are km mol^{-1}. [e] Units are $Å^4$ amu^{-1}. [f] Coordinate numbers correspond to Fig. 1

Table S8. Topological properties of Clomesone calculated at B3LYP/6-311G(3df) level.

Bond (X-Y)	ρ	$\nabla^2\rho$ (L)	λ_1/λ_3	$\varepsilon = \lvert\lambda_1/\lambda_2\rvert - 1$	q (X)	q (Y)	q (X)	q (Y)
					Mulliken		NPA	
C(1A)-S(1A)	0.209	-0.422	1.25	0.034	-0.834	0.966	-0.797	2.172
S(1A)- C(2A)	0.194	-0.368	1.31	0.025	0.966	-0.663	2.172	-0.861
C(2A)-S(2A)	0.205	-0.412	1.22	0.009	-0.633	1.132	-0.861	2.402
S(2A)- O(3A)	0.230	-0.130	0.63	0.115	1.132	-0.541	2.402	-0.770
O(3A)- C(3A)	0.235	-0.440	1.04	0.043	-0.541	0.103	-0.770	-0.036
C(3A)- C(4A)	0.260	-0.624	1.29	0.042	0.103	-0.580	-0.036	-0.340
C(4A)-Cl(A)	0.177	-0.220	0.85	0.022	-0.580	-0.152	-0.340	-0.076
S(1A)-O(11A)	0.316	-0.806	0.30	0.017	0.966	-0.507	2.172	-0.936
S(1A)-O(12A)	0.314	-0.787	0.30	0.041	0.966	-0.474	2.172	-0.945
S(2A)-O(21A)	0.322	-0.860	0.27	0.05	1.132	-0.495	2.402	-0.919
S(2A)-O(22A)	0.323	-0.844	0.30	0.02	1.132	-0.502	2.402	-0.931

Figure S1. Infrared and Raman spectra of Clomesone.

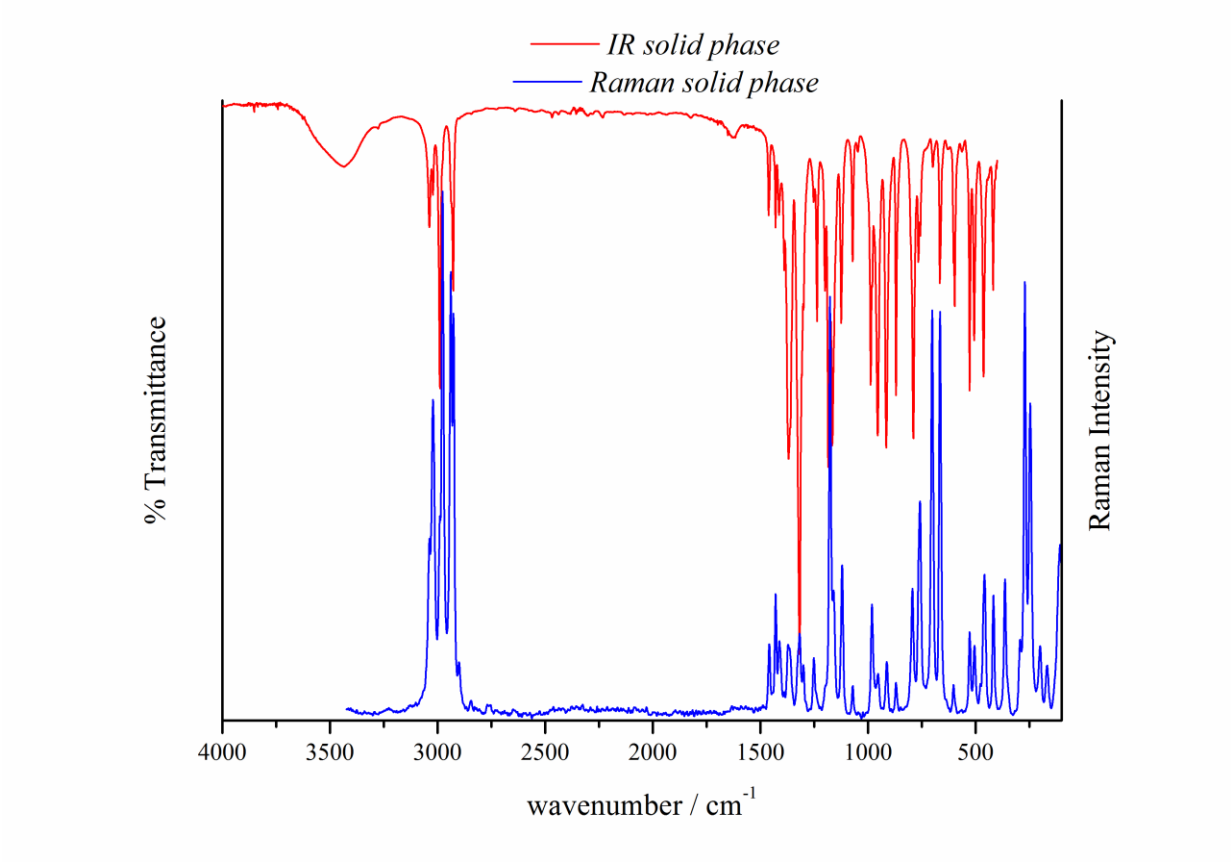

Figure S2. View of Clomesone, N-hexanoyl-L-homoserine lactone, $CH_3SO_2CH_2SO_2O^-$ and Colistin methansulfonate (CMS).