Appendix I : Supplementary Data for the Crystal Structure of N-phenylsalicylidene sulfonic acid guanidinium

I.1 Tables

Table 1. Crystal data and structure refinement for JEM1.

Empirical formula	$C_{14}H_{16}N_4O_4S$
Formula weight	336.37

Data Collection

Type of diffractometer	CCD area detector
Wavelength	0.71073 Å MoKα
Data Collection Temperature	293 K
θ range for reflections used in lattice determination	2.10 to 23.34°
Unit cell dimensions	a = 7.3650(15) Å b = 17.578(4) Å β= 98.48(3)° c = 11.727(2) Å
Volume	1501.7(5) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.488 Mg/m^3
F(000)	704
θ range for data collection	2.10 to 23.34°
Completeness to θ = 23.34°	99.1 %
Index ranges	$-8 \le h \le 8, -18 \le k \le 19, -11 \le l \le 13$
Data collection scan type	ω scans at 3 ϕ settings
Reflections collected	6820
Independent reflections	2168 [R_{int}= 0.0805]
Absorption coefficient	0.243 mm^{-1}
Absorption correction	None

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	2168 / 0 / 209
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	3.704
Final R indices [I>2σ(I)]	R1 = 0.1101, *w*R2 = 0.1841
R indices (all data)	R1 = 0.1350, *w*R2 = 0.1858
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Largest diff. peak and hole	1.188 and -0.381 e.Å^{-3}

Special Refinement Details

These crystals of N-phenylsalicylidene sulfonic acid guanidinium are twinned. It was not possible to obtain satisfactory refinement results when applying the twinning algorithms. Therefore the twinning was ignored. The overall structure is correct but is not of sufficient quality to publish.

Refinement of F^2 is against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for JEM1. U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
S(1)	2243(3)	7275(1)	894(2)	46(1)
O(1)	3405(8)	4325(3)	3275(4)	56(2)
O(2)	1770(7)	7079(3)	-322(4)	56(2)
O(3)	3937(7)	7703(3)	1071(4)	57(1)
O(4)	768(7)	7644(3)	1338(4)	60(2)
N(1)	2543(8)	4956(3)	5072(4)	45(2)
C(1)	3173(11)	4999(4)	2766(7)	48(2)
C(2)	3504(11)	5081(4)	1660(7)	47(2)
C(3)	3213(11)	5757(4)	1081(6)	52(2)
C(4)	2643(10)	6409(4)	1643(6)	43(2)
C(5)	2397(10)	6342(4)	2775(6)	45(2)
C(6)	2652(10)	5661(4)	3339(6)	41(2)
C(7)	2360(10)	5587(5)	4531(6)	49(2)
C(8)	2224(11)	4861(4)	6242(7)	50(2)
C(9)	1695(11)	5455(4)	6931(6)	51(2)
C(10)	1433(11)	5250(5)	8033(7)	57(2)
C(11)	1642(12)	4550(6)	8466(7)	62(2)
C(12)	2165(12)	3963(5)	7772(7)	68(3)
C(13)	2454(11)	4130(5)	6676(6)	53(2)
N(20)	7078(9)	7606(4)	4961(6)	66(2)
N(21)	8097(10)	8069(4)	3347(6)	64(2)
N(22)	5016(9)	8089(4)	3501(5)	60(2)
C(20)	6747(13)	7925(4)	3936(8)	52(2)

Table 3. Bond lengths [Å] and angles [°] for JEM1.

S(1)-O(4)	1.427(5)	C(2)-C(1)-C(6)	117.5(7)
S(1)-O(3)	1.445(5)	C(1)-C(2)-C(3)	121.7(7)
S(1)-O(2)	1.459(5)	C(1)-C(2)-H(2B)	119.1
S(1)-C(4)	1.761(8)	C(3)-C(2)-H(2B)	119.1
O(1)-C(1)	1.327(8)	C(2)-C(3)-C(4)	120.4(7)
O(1)-H(1A)	0.8200	C(2)-C(3)-H(3B)	119.8
N(1)-C(7)	1.276(8)	C(4)-C(3)-H(3B)	119.8
N(1)-C(8)	1.436(8)	C(5)-C(4)-C(3)	118.1(7)
C(1)-C(2)	1.363(9)	C(5)-C(4)-S(1)	121.5(6)
C(1)-C(6)	1.424(10)	C(3)-C(4)-S(1)	120.4(6)
C(2)-C(3)	1.370(10)	C(6)-C(5)-C(4)	121.1(7)
C(2)-H(2B)	0.9300	C(6)-C(5)-H(5A)	119.5
C(3)-C(4)	1.416(10)	C(4)-C(5)-H(5A)	119.5
C(3)-H(3B)	0.9300	C(5)-C(6)-C(1)	120.9(7)
C(4)-C(5)	1.372(8)	C(5)-C(6)-C(7)	121.0(7)
C(5)-C(6)	1.366(10)	C(1)-C(6)-C(7)	118.0(7)
C(5)-H(5A)	0.9300	N(1)-C(7)-C(6)	122.5(7)
C(6)-C(7)	1.452(9)	N(1)-C(7)-H(7A)	118.8
C(7)-H(7A)	0.9300	C(6)-C(7)-H(7A)	118.8
C(8)-C(13)	1.382(10)	C(13)-C(8)-C(9)	120.4(7)
C(8)-C(9)	1.410(10)	C(13)-C(8)-N(1)	115.5(7)
C(9)-C(10)	1.382(10)	C(9)-C(8)-N(1)	124.0(7)
C(9)-H(9A)	0.9300	C(10)-C(9)-C(8)	115.5(8)
C(10)-C(11)	1.330(11)	C(10)-C(9)-H(9A)	122.2
C(10)-H(10A)	0.9300	C(8)-C(9)-H(9A)	122.2
C(11)-C(12)	1.403(11)	C(11)-C(10)-C(9)	125.0(8)
C(11)-H(11A)	0.9300	C(11)-C(10)-H(10A)	117.5
C(12)-C(13)	1.365(9)	C(9)-C(10)-H(10A)	117.5
C(12)-H(12A)	0.9300	C(10)-C(11)-C(12)	119.0(8)
C(13)-H(13A)	0.9300	C(10)-C(11)-H(11A)	120.5
N(20)-C(20)	1.316(10)	C(12)-C(11)-H(11A)	120.5
N(20)-H(20A)	0.8600	C(13)-C(12)-C(11)	118.7(8)
N(20)-H(20B)	0.8600	C(13)-C(12)-H(12A)	120.6
N(21)-C(20)	1.316(9)	C(11)-C(12)-H(12A)	120.6
N(21)-H(21A)	0.8600	C(12)-C(13)-C(8)	121.3(8)
N(21)-H(21B)	0.8600	C(12)-C(13)-H(13A)	119.4
N(22)-C(20)	1.333(10)	C(8)-C(13)-H(13A)	119.4
N(22)-H(22A)	0.8600	C(20)-N(20)-H(20A)	120.0
N(22)-H(22B)	0.8600	C(20)-N(20)-H(20B)	120.0
O(4)-S(1)-O(3)	113.5(3)	H(20A)-N(20)-H(20B)	120.0
O(4)-S(1)-O(2)	112.3(3)	C(20)-N(21)-H(21A)	120.0
O(3)-S(1)-O(2)	109.9(3)	C(20)-N(21)-H(21B)	120.0
O(4)-S(1)-C(4)	106.9(3)	H(21A)-N(21)-H(21B)	120.0
O(3)-S(1)-C(4)	107.5(3)	C(20)-N(22)-H(22A)	120.0
O(2)-S(1)-C(4)	106.4(3)	C(20)-N(22)-H(22B)	120.0
C(1)-O(1)-H(1A)	109.5	H(22A)-N(22)-H(22B)	120.0
C(7)-N(1)-C(8)	123.8(6)	N(21)-C(20)-N(20)	120.6(9)
O(1)-C(1)-C(2)	119.5(7)	N(21)-C(20)-N(22)	120.7(8)
O(1)-C(1)-C(6)	122.9(7)	N(20)-C(20)-N(22)	118.7(8)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for JEM1. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	582(13)	425(12)	370(12)	26(10)	35(9)	-42(12)
O(1)	940(40)	360(30)	370(30)	-40(30)	50(30)	70(30)
O(2)	700(40)	540(40)	400(30)	0(30)	-30(30)	-50(30)
O(3)	670(30)	570(30)	470(30)	70(30)	40(30)	-240(30)
O(4)	750(40)	480(30)	590(30)	90(30)	230(30)	180(30)
N(1)	750(50)	300(40)	270(40)	10(30)	10(30)	0(40)
C(1)	550(50)	370(50)	520(50)	80(40)	50(40)	0(40)
C(2)	640(50)	370(50)	390(50)	-90(40)	60(40)	50(40)
C(3)	640(50)	500(60)	430(50)	-90(40)	100(40)	-50(50)
C(4)	500(50)	390(50)	390(50)	-40(40)	20(40)	-60(40)
C(5)	600(50)	350(50)	400(50)	-40(40)	70(40)	-60(40)
C(6)	540(50)	400(50)	280(40)	-50(40)	10(40)	-50(40)
C(7)	590(50)	440(50)	440(50)	-60(40)	20(40)	-10(40)
C(8)	570(50)	380(50)	510(50)	90(40)	-10(40)	-80(40)
C(9)	860(60)	400(50)	270(40)	-150(40)	90(40)	-80(50)
C(10)	690(60)	640(60)	350(50)	-100(40)	-60(40)	10(50)
C(11)	720(60)	790(70)	360(50)	-90(50)	100(40)	-180(60)
C(12)	850(70)	620(60)	550(60)	190(50)	0(50)	-120(50)
C(13)	710(60)	420(50)	410(50)	20(40)	-30(40)	20(40)
N(20)	620(40)	610(50)	760(50)	60(40)	140(40)	80(40)
N(21)	700(50)	690(50)	550(40)	40(40)	130(40)	0(40)
N(22)	650(50)	720(50)	430(40)	20(30)	60(30)	0(40)
C(20)	640(60)	270(50)	670(60)	-100(40)	150(50)	-40(40)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for JEM1.

	x	y	z	U_{iso}
H(1A)	3177	4357	3937	84
H(20A)	8183	7488	5251	79
H(20B)	6188	7514	5340	79
H(21A)	9207	7954	3631	77
H(21B)	7870	8278	2678	77
H(22A)	4771	8291	2827	72
H(22B)	4144	7993	3893	72
H(2B)	3940	4667	1288	56
H(3B)	3390	5789	313	62
H(5A)	2051	6766	3166	54
H(7A)	2028	6017	4916	59
H(9A)	1534	5952	6662	61
H(10A)	1083	5630	8508	69
H(11A)	1444	4452	9217	74
H(12A)	2312	3468	8053	82
H(13A)	2811	3745	6212	63

Table 6. Hydrogen bonds for JEM1 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1A)...N(1)	0.82	1.81	2.543(7)	147.9
N(20)-H(20A)...O(4)#1	0.86	2.14	2.983(9)	165.5
N(20)-H(20B)...O(3)#2	0.86	2.01	2.868(8)	174.1
N(21)-H(21A)...O(2)#1	0.86	2.10	2.928(8)	162.9
N(21)-H(21B)...O(1)#3	0.86	2.28	3.017(8)	143.3
N(22)-H(22A)...O(3)	0.86	2.31	2.923(7)	128.8
N(22)-H(22B)...O(2)#2	0.86	2.10	2.947(8)	169.6

Symmetry transformations used to generate equivalent atoms:
#1 x+1,-y+3/2,z+1/2 #2 x,-y+3/2,z+1/2 #3 -x+1,y+1/2,-z+1/2

I.2 Figures

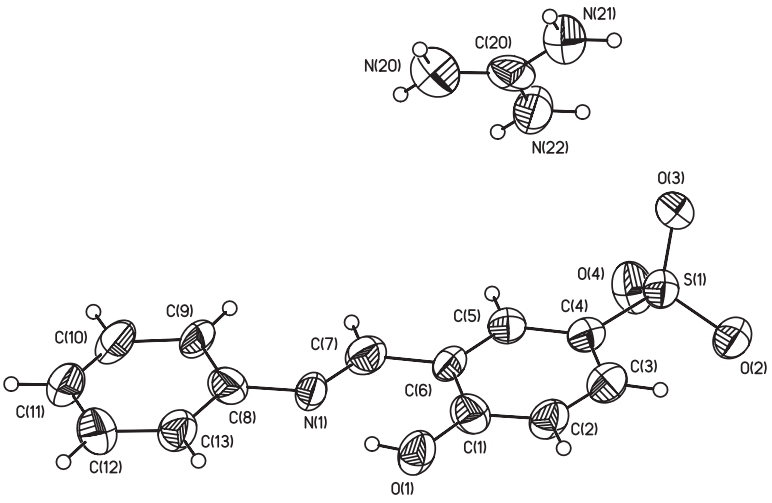

Figure I.1: Structural components of N-phenylsalicylidene sulfonic acid guanidinium.

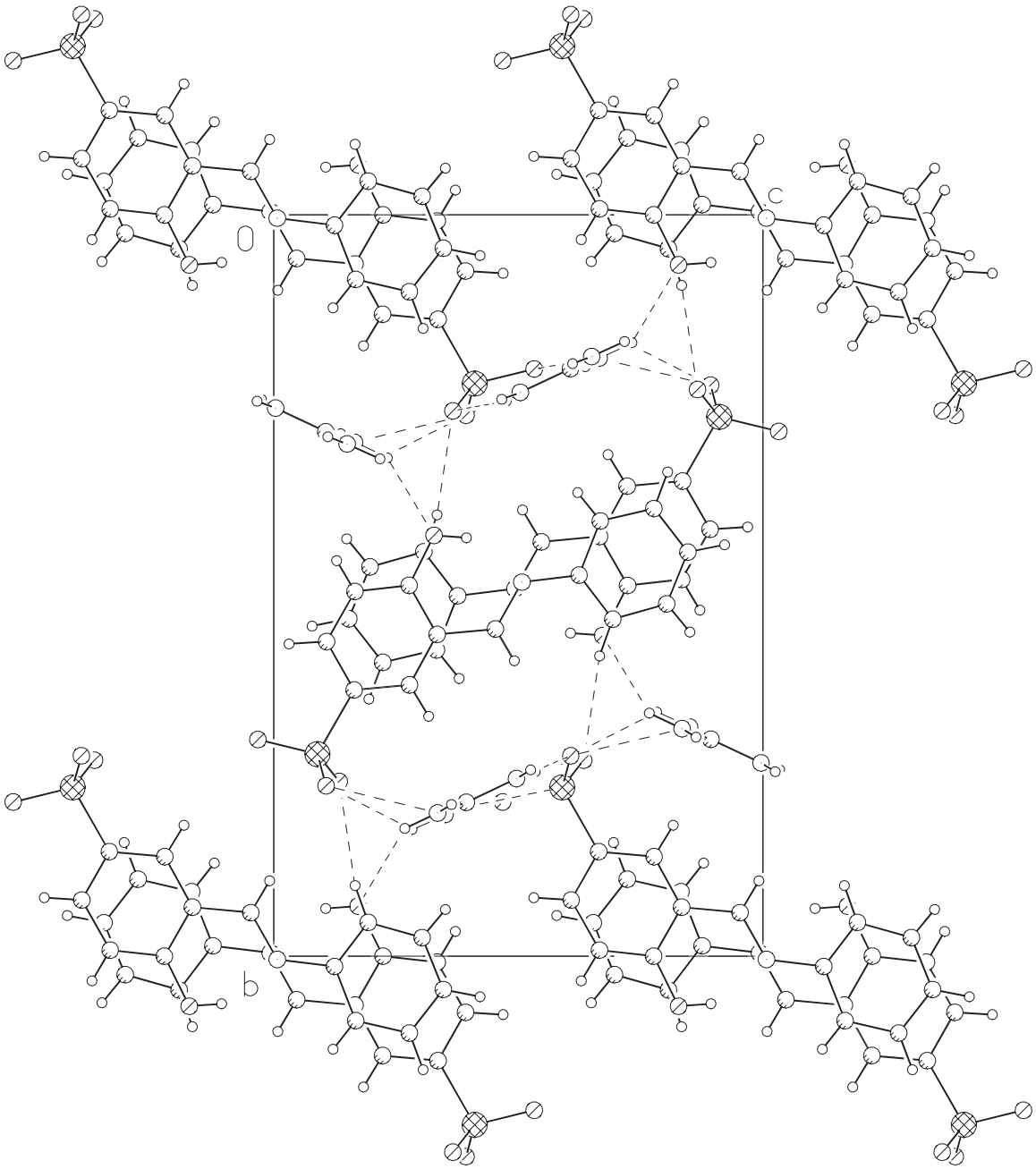

Figure I.2: View of N-phenylsalicylidene sulfonic acid guanidinium along the bc plane.

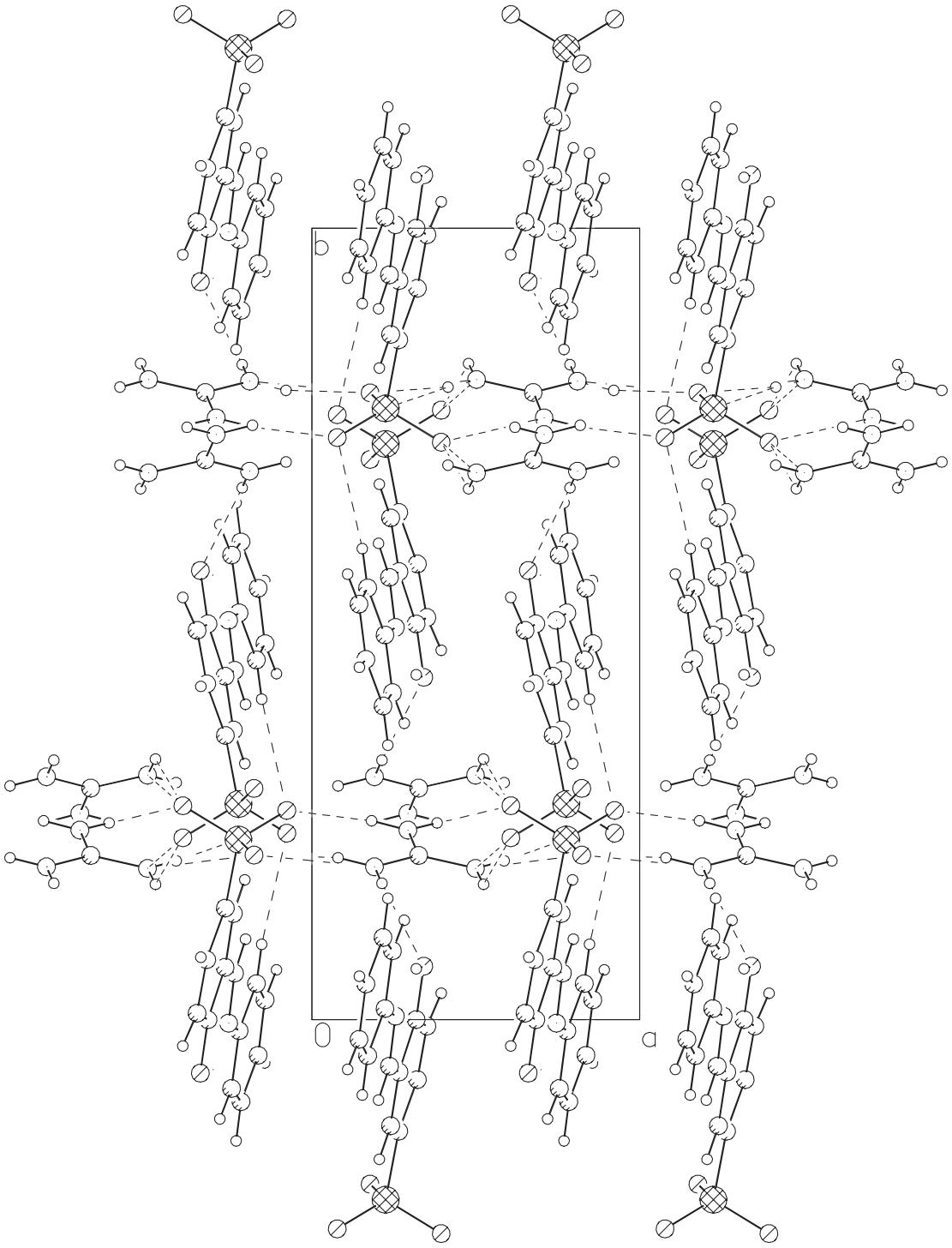

Figure I.3: View of the N-phenylsalicylidene sulfonic acid guanidinium structure along the ab plane.

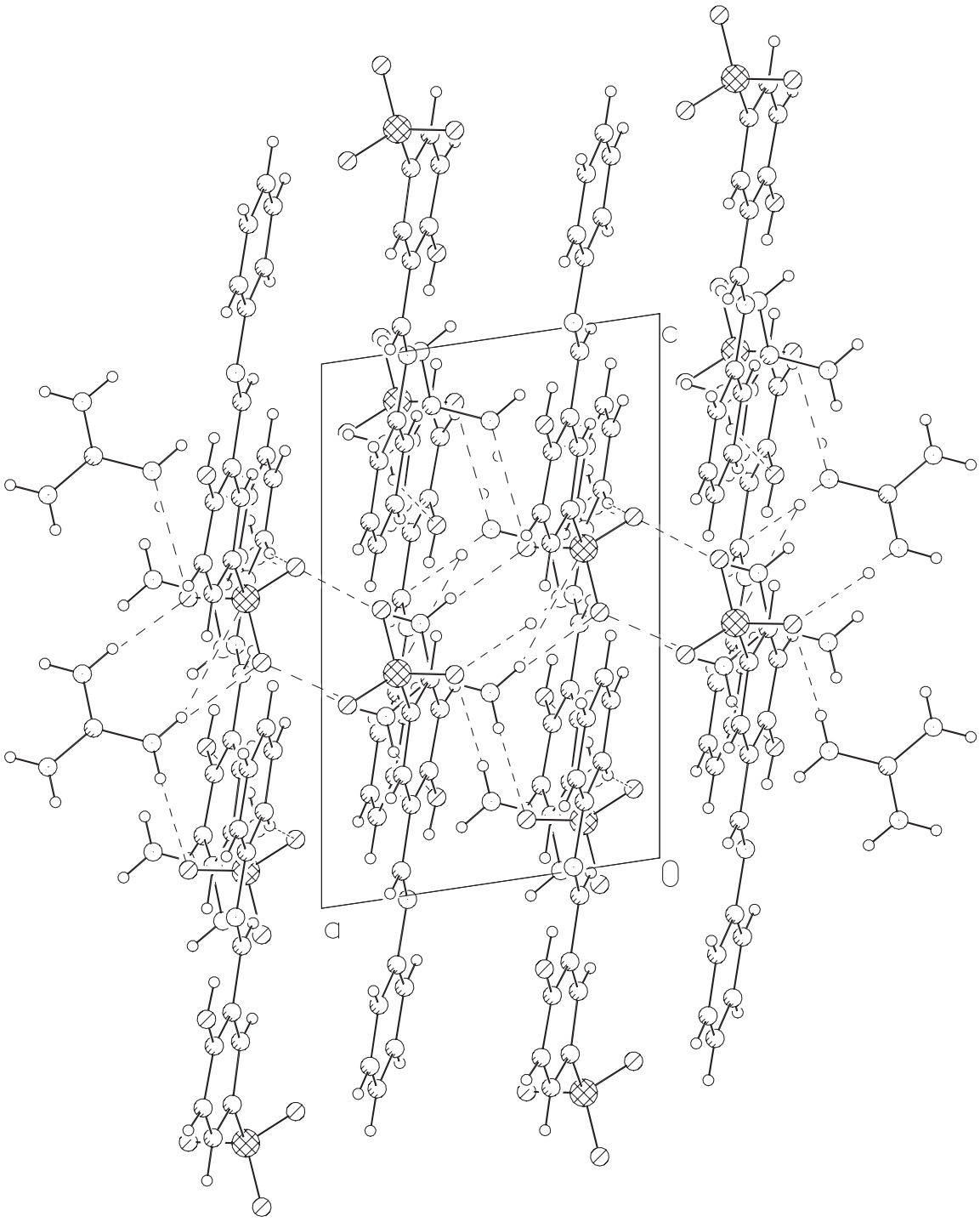

Figure I.4: View of the N-phenylsalicylidene sulfonic acid guanidinium structure along the ac plane.