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Ring stacking and laddering in ammonium carboxylate salts: extension to secondary ammonium salts

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Supplementary Information

Contents

Table S1. Hydrogen bond parameters in salts 1-7.

Salt	$N^+ \cdots O^-$ distance /Å	Symmetry operator	$N^+H\ O^-$ angle /°
1	2.727(1) 2.709(1)	x,y,z x,1/2-y,-1/2+z	179(2) 173(1)
2	2.760(4) 2.713(5) 2.773(4) 2.737(5)	x,y,z x,y,z x,y,z x,1+y,z	162(4) 176.0 176.9 174(4)
3	2.726(2) 2.678(2)	x,y,z 1-x,1-y,-z	155(2) 177(2)
4	2.711(1) 2.710(1)	x,y,z 1/2-x,1/2-y,-z	165(1) 166(1)
5	2.970(2) 2.979(2) 2.744(2)	x,y,z x,y,z 1+x,y,z	144(2) 156(2) 170(2)
6	2.708(2) 2.688(2)	x,y,z 1+x,y,z	173(3) 172(2)
7	2.765(3) 2.719(3)	x,y,z 1+x,y,z	173(3) 173(3)

[†] Deceased 2 January 2020.

Table S2. Selected crystallographic data for salts 1-7.

Structure number	**1**	**2**	**3**	**4**	**5**	**6**	**7**
Empirical formula	$C_{18}H_{23}NO_2$	$C_{22}H_{31}NO_2$	$C_{20}H_{35}NO_2$	$C_{13}H_{25}NO_2$	$C_{13}H_{29}NO_4$	$C_{20}H_{31}NO_2$	$C_{26}H_{35}NO_2$
Formula weight	285.37	341.48	321.49	227.34	263.37	317.46	393.55
Temperature (K)	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)
Wavelength (Å)	0.71073	0.71073	0.71073	0.71073	0.71073	0.71073	0.71073
Crystal system	monoclinic	orthorhombic	triclinic	monoclinic	monoclinic	triclinic	triclinic
Space group	$P2_1/c$	$Pca2_1$	P-1	$C2/c$	$P2_1/n$	P-1	$P1$
Unit cell dimensions (Å, °)	a = 8.3067(7) α = 90 b = 16.4097(13) β = 96.3880(10) c = 11.4319(9) γ = 90	a = 21.610(3) α = 90 b = 8.5291(11) β = 90 c = 21.825(3) γ = 90	a = 8.5570(14) α = 77.147(3) b = 9.8287(16) β = 75.054(3) c = 12.292(2) γ = 83.311(3)	a = 19.216(3) α = 90 b = 8.1118(9) β = 94.372(4) c = 16.8987(19) γ = 90	a = 5.1775(7) α = 90 b = 17.585(2) β = 95.783(2) c = 16.534(2) γ = 90	a = 5.5919(10) α = 93.444(3) b = 10.782(2) β = 93.522(3) c = 15.271(3) γ = 94.507(3)	a = 5.7270(7) α = 95.286(2) b = 9.4987(12) β = 90.783(2) c = 10.4310(13) γ = 103.644(2)
Volume (Å)	1548.6(2)	4022.6(9)	971.9(3)	2626.4(5)	1497.7(3)	914.2(3)	548.71(12)
Z	4	8	2	8	4	2	1
Calculated density (g cm^{-3})	1.224	1.128	1.099	1.15	1.168	1.153	1.191
Absorption coefficient (mm^{-1})	0.079	0.071	0.069	0.076	0.085	0.073	0.074
F_{000}	616	1488	356	1008	584	348	214
Crystal size (mm^3)	0.50 × 0.39 × 0.16	0.24 × 0.11 × 0.08	0.47 × 0.10 × 0.10	0.60 × 0.51 × 0.45	0.400 × 0.190 × 0.100	1.05 × 0.52 × 0.26	0.20 × 0.10 × 0.03
θ range for data collection (°)	2.180 to 28.357	1.866 to 28.292	1.750 to 28.278	2.126 to 28.305	1.695 to 28.267	1.339 to 25.085	1.962 to 26.383
Miller index ranges	-11 ≤ h ≤ 11, -20 ≤ k ≤ 20, -15 ≤ l ≤ 15	-25 ≤ h ≤ 27, -10 ≤ k ≤ 11, -29 ≤ l ≤ 15	-11 ≤ h ≤ 11, -12 ≤ k ≤ 12, -16 ≤ l ≤ 16	-25 ≤ h ≤ 21, -8 ≤ k ≤ 10, -22 ≤ l ≤ 17	-6 ≤ h ≤ 6, -22 ≤ k ≤ 22, -11 ≤ l ≤ 21	-6 ≤ h ≤ 6, -12 ≤ k ≤ 12, -18 ≤ l ≤ 18	-7 ≤ h ≤ 7, -11 ≤ k ≤ 11, -13 ≤ l ≤ 13
Reflections collected	17669	23175	11235	8059	9233	8842	6017
Independent reflections	3681 [R_{int} = 0.0332]	7928 [R_{int} = 0.0835]	4442 [R_{int} = 0.0343]	3060 [R_{int} = 0.0267]	3496 [R_{int} = 0.0315]	3227 [R_{int} = 0.0281]	4351 [R_{int} = 0.0185]
Completeness to θ_{max} (%)	0.951	0.946	0.919	0.936	0.944	0.994	0.992
Max. and min. transmission	0.9976 and 0.9994	0.9976 and 0.9994	0.789 and 0.993	0.812 and 0.967	0.823 and 0.993	0.738 and 0.981	0.860 and 0.998
Data / restraints / parameters	3681 / 0 / 200	7928 / 1 / 463	4442 / 0 / 218	3060 / 0 / 153	3496 / 6 / 187	3227 / 0 / 216	4351 / 3 / 270
Goodness-of-fit on F^2	1.032	1.057	1.09	1.026	1.063	1.058	0.952
Final R indices [$I > 2\sigma(I)$]	$R1$ = 0.0463, $wR2$ = 0.1098	$R1$ = 0.0745, $wR2$ = 0.1342	$R1$ = 0.0620, $wR2$ = 0.1324	$R1$ = 0.0460, $wR2$ = 0.1201	$R1$ = 0.0555, $wR2$ = 0.1317	$R1$ = 0.0432, $wR2$ = 0.1076	$R1$ = 0.0440, $wR2$ = 0.1161
R indices (all data)	$R1$ = 0.0537, $wR2$ = 0.1147	$R1$ = 0.1038, $wR2$ = 0.1464	$R1$ = 0.0865, $wR2$ = 0.1426	$R1$ = 0.0553, $wR2$ = 0.1266	$R1$ = 0.0693, $wR2$ = 0.1397	$R1$ = 0.0534, $wR2$ = 0.1166	$R1$ = 0.0464, $wR2$ = 0.1188
Largest diff. peak and hole (e Å^{-3})	0.414 and -0.193	0.260 and -0.230	0.299 and -0.231	0.403 and -0.174	0.382 and -0.180	0.263 and -0.194	0.235 and -0.197

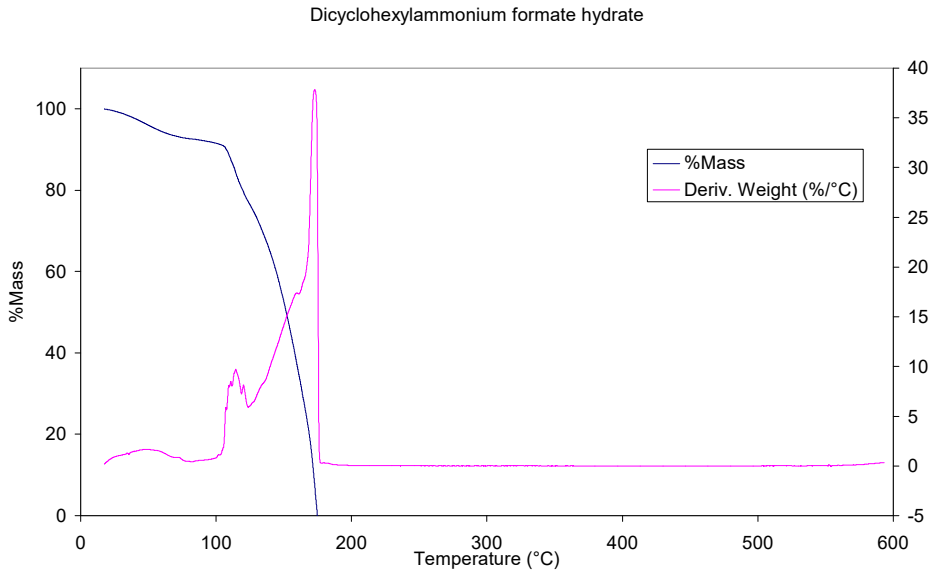

Figure S1. TGA of salt **5**. Observed mass loss is 10.27%, larger than the calculated mass loss of 6.83% for two waters, presumably due to surface solvent on the crystals.

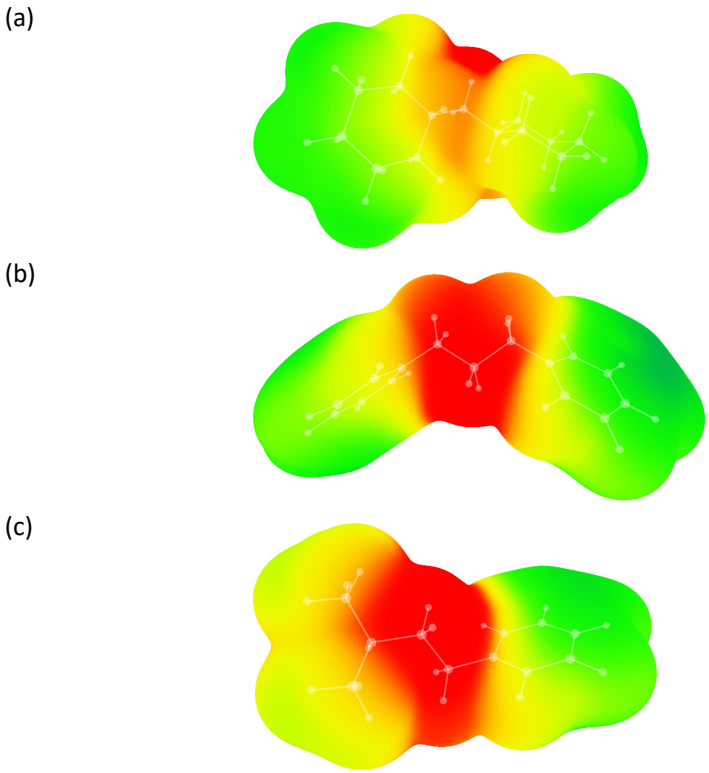

Figure S2. Electrostatic potential maps (0.01 a.u. isosurface) for the dicyclohexylammonium ion from (a) COBPEO and (b) PEQJEB, showing how the positive potential extends over the cation. Part (c) shows the electrostatic potential map for the N-benzylpropan-2-aminium cation from GEPNAT, demonstrating that this is not restricted to dicycloheylammonium ions. Wavefunctions and ESPs were calculated using Gaussian09 v D.01.[1] Hydrogen atom positions were optimised, and the wavefunction calculated using population analysis at the B3LYP-D3/6-311++G(d,p) level of theory. ESPs were read from output files generated in Surface Analysis Suite using WFA, and graphics generated in SurRender.[2]

Refcode lists

Secondary ammonium carboxylates (130 structures)

ABADAK
ABUQAR
AGAPOP
AKOGOY03
AWUQEP
BEPTUN
CADMAY
CENXAX
CENXIF
CENXOL
CENXUR
CENYEC
CENYIG
CENYOM
CEPFIP
CEPFUB
CEPGAI
CINZIJ
COBPEO01
ELIZAC
ELIZEG
EQEWII
EVIHOH
FAFLII
FIWZUI
GASCHA
GEPNAT
GEPYAE
GIGDUX
GISJIB
GIZCIC
GODFUA
HAPQEX02
ICOPAS
IROVUI
JAMWIE
JAMWOK
JAMWUQ
JAMXAX
JAMXEB
JAMXIF
JAMXUR
JAMYAY
JAMYEC
JAMYIG
JAMYOM
JAMYUS
JAVDOC
JAVNOM
JAVNUS
JAVPEE
JAVPII
JEFYAV
JEFYEZ
JEFYUP
JEFZIE
LESRIL
LIGZOR
MABVES
MAKJUG
MASQOM
MATNUQ
MEVXUG
MEVYAN
MEVYER
MISXIW
MORPLN11
NELTAZ
NENFES
NUKDOO
PEDGIO
PEQGEY
PEQGIC
PEQGOI
PEQGUO
PEQHAV
PEQHEZ
PEQHID
PEQHOJ
PEQHUP
PEQJAX
PEQJEB
PEQJIF
PEQJOL
PEQJUR
PEQKAY
PEQKEC
PEQKIG
PEZMUC
PIPBBZ
PIPCBZ01
PIPTAC
QUQJET
RATYAN
RATYER
RATYIV
RATYOB
RATYUH
RATZAO
RATZES
RATZIW
SAMLAU
SAWXEW
SUQNOI
TIYFUE
UHOBIC
ULIMIM
ULUPIA
VITTAV
VUKQOJ
VUPTUW
VUXNAF
VUXNEJ
VUXNIN
VUXNOT
VUXVOB
WUBHAC
XAFTAC
XEBGOC
XIMBZA
XIMBZB01
XISWEE
XONSUQ
YEJYAN
YEJYER
YIDJON
YUFREX
ZANPOW
ZIVLUL
ZUTFIF

Discrete rings (55 structures)

ABADAK
AWUQEP
CENYEC
ELIZAC
ELIZEG
EVIHOH
FAFLII
GIGDUX
GISJIB
GIZCIC
GODFUA
JAMXIF
JAMXUR
JAMYAY
JAMYEC
JAMYIG
JAMYOM
JAMYUS
JAVDOC
JAVNUS
JAVPEE
JAVPII
JEFYAV
JEFYEZ
JEFYUP
LESRIL
LIGZOR
MABVES
MATNUQ
MEVYAN
MEVYER
MISXIW
MORPLN11
NELTAZ
PEDGIO
PEZMUC
QUQJET
RATYAN
RATYER
RATYIV
RATYOB
RATYUH
RATZAO
SAWXEW
UHOBIC
VUPTUW
VUXNOT
WUBHAC
XEBGOC
XISWEE
YEJYAN
YEJYER
YIDJON
ZIVLUL
ZUTFIF

Ladders (9 structures)

EQEWII
PEQGIC
PEQGOI
PEQHAV
VUXNAF
VUXNEJ
VUXVOB
XAFTAC
YUFREX

1D chains (66 structures, ladders with one long rung in bold)

ABUQAR	PEQGEY
AGAPOP	PEQGUO
AKOGOY03	**PEQHEZ**
BEPTUN	**PEQHID**
CADMAY	**PEQHOJ**
CENXAX	**PEQHUP**
CENXIF	**PEQJAX**
CENXOL	**PEQJEB**
CENXUR	**PEQJIF**
CENYIG	**PEQJOL**
CENYOM	**PEQJUR**
CEPFIP	PEQKAY
CEPFUB	**PEQKEC**
CEPGAI	**PEQKIG**
CINZIJ	**PIPBBZ**
COBPEO01	**PIPCBZ01**
FIWZUI	**PIPTAC**
GASCHA	RATZES
GEPNAT	RATZIW
GEPYAE	**SAMLAU**
HAPQEX02	**SUQNOI**
ICOPAS	**TIYFUE**
IROVUI	**ULIMIM**
JAMWIE	**ULUPIA**
JAMWOK	**VITTAV**
JAMWUQ	VUKQOJ
JAMXAX	VUXNIN
JAMXEB	XIMBZA
JAVNOM	**XIMBZB01**
JEFZIE	**XONSUQ**
MAKJUG	**ZANPOW**
MASQOM	
MEVXUG	
NENFES	
NUKDOO	

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