Electronic Supplementary Information

Synthesis and Supramolecular Organization of Iodide and Triiodide of a Polycyclic Adamantane-Based Diammonium Cation: The Effect of Hydrogen Bonds and Weak I···I Interactions

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Table S1. Geometry of 1,3-Diazaadamantanes from the CSD Database

No.	CSD ref. code	d(N–CH_2), Å	d(N…N), Å	∠(N–C–N), deg.	Ref.
Neutral					
1	BOGROE	1.461; 1.473; 1.474; 1.484	2.478; 2.483	114.14; 115.29	[1]
2	BOGRUK	1.477; 1.490	2.454	111.63	[1]
3	BOQTOQ	1.471; 1.472	2.471	114.18	[2]
4	BOVWEP	1.471; 1.475	2.478	114.55	[3]
5	FOYRAM	1.457; 1.472	2.466	114.68	[4]
6	HULJAZ	1.467; 1.472; 1.472; 1.488	2.461; 2.474	114.15; 114.40	[5]
7	MOWYED	1.474; 1.474	2.478	114.34	[6]
8	PUYYAJ	1.480; 1.483	2.488	114.27	[7]
9	QIBGOX	1.468; 1.476	2.473	114.31	[8]
10	VOGVUL	1.466; 1.466	2.457	113.88	[9]
Monoprotonated Cation					
11	ANGSTF	1.417; 1.541	2.447	111.57	[10]
12	JADVIT	1.419; 1.534	2.416	109.91	[11]
13	KEQSEG	1.472; 1.511	2.446	110.19	[12]
14	VOJRUH	1.418; 1.426; 1.507; 1.511	2.293; 2.402	109.28; 110.15	[13]
Monomethylated Cation					
15	PAZADO10	1.359; 1.593	2.401	108.58	[14]
16	VOJRIV	1.422; 1.537	2.417	110.99	[13]
17	VOJROB	1.414; 1.538	2.439	109.77	[13]
Dimethylated Dication					
18	JIGJOY	1.464; 1.519	2.451	109.37	[15]

Table S2. Crystallographic Data and Structure Refinement Parameters for the Powder Sample of Compound **2**

Formula	$C_{10}H_{20}N_2I_4$
formula weight ($g \cdot mol^{-1}$)	675.9
crystal system	Triclinic
space group	$P\bar{1}$
a (Å)	8.15439(9)
b (Å)	8.70457(9)
c (Å)	13.0980(2)
α (°)	95.7365(11)
β (°)	98.8213(9)
γ (°)	96.0694(10)
V ($Å^3$)	907.15(2)
Z	2
ρ_{calcd} ($g \cdot cm^{-3}$)	2.4745
temperature (K)	293
radiation, λ, Å	Cu $K\alpha_1$, 1.54060
2θ range (deg)	4.005–84.915
R_1	0.0291
wR_2	0.0387
GoF	1.42
R_{prof}	0.0316
wR_{prof}	0.0401

Table S3. Selected Interatomic Distances and Angles in the Cations

Atoms	Distance, Å	Atoms	Angle, °
$[(C_{10}N_2H_{20})I]I$ (1)			
N(1)–C(3)	1.506(3)	C(4)–N(1)–C(3)	110.84(18)
N(1)–C(4)	1.483(3)	C(4)–N(1)–C(6)	111.05(19)
N(1)–C(6)	1.506(3)	C(6)–N(1)–C(3)	111.36(19)
		N(1)–C(3)–C(9)	108.52(19)
		N(1)–C(6)–C(2)	108.35(19)
N(2)–C(1)	1.505(3)	C(1)–N(2)–C(8)	111.03(19)
N(2)–C(4)	1.488(3)	C(4)–N(2)–C(1)	110.25(18)
N(2)–C(8)	1.509(3)	C(4)–N(2)–C(8)	111.34(19)
		N(2)–C(8)–C(2)	108.25(19)
		N(2)–C(1)–C(9)	108.99(18)
C(2)–C(5)	1.538(3)	C(6)–C(2)–C(5)	109.30(19)
C(2)–C(6)	1.533(3)	C(6)–C(2)–C(8)	108.19(19)
C(2)–C(7)	1.531(3)	C(7)–C(2)–C(5)	111.5(2)
C(2)–C(8)	1.537(3)	C(7)–C(2)–C(6)	109.6(2)
		C(7)–C(2)–C(8)	109.4(2)
		C(8)–C(2)–C(5)	108.84(19)
C(9)–C(1)	1.530(3)	C(1)–C(9)–C(3)	108.5(2)
C(9)–C(3)	1.530(3)	C(1)–C(9)–C(5)	108.6(2)
C(9)–C(5)	1.540(3)	C(3)–C(9)–C(5)	108.7(2)
C(9)–C(10)	1.524(3)	C(10)–C(9)–C(1)	109.1(2)
		C(10)–C(9)–C(3)	109.9(2)
		C(10)–C(9)–C(5)	112.0(2)
		C(2)–C(5)–C(9)	110.34(19)
		N(1)–C(4)–N(2)	107.67(18)
$[(C_{10}N_2H_{20})I]I_3$ (2)			
N(1)–C(1)	1.43(3)	C(1)–N(1)–C(5)	112(2)
N(1)–C(4)	1.54(3)	C(1)–N(1)–C(4)	111.4(19)
N(1)–C(5)	1.49(3)	C(5)–N(1)–C(4)	107(2)
		C(8)–N(2)–C(5)	112.6(19)
		C(8)–N(2)–C(6)	110.9(18)
N(2)–C(8)	1.48(3)	C(5)–N(2)–C(6)	110(2)
N(2)–C(5)	1.45(3)	N(1)–C(1)–C(7)	112(2)
N(2)–C(6)	1.57(3)	C(7)–C(2)–C(3)	110(2)
		C(8)–C(3)–C(4)	111.8(19)
		C(8)–C(3)–C(2)	109.5(19)
C(7)–C(1)	1.45(3)	C(4)–C(3)–C(2)	107.4(19)
C(7)–C(2)	1.51(3)	C(8)–C(3)–C(9)	108(2)
C(7)–C(6)	1.51(3)	C(4)–C(3)–C(9)	110(2)

C(7)–C(10)	1.51(4)	C(2)–C(3)–C(9)	110(2)
		C(3)–C(4)–N(1)	107.4(18)
		N(1)–C(5)–N(2)	107(2)
C(3)–C(2)	1.53(3)	C(7)–C(6)–N(2)	106.1(19)
C(3)–C(4)	1.48(3)	C(2)–C(7)–C(6)	111(2)
C(3)–C(8)	1.49(3)	C(2)–C(7)–C(1)	109(2)
C(3)–C(9)	1.52(3)	C(6)–C(7)–C(1)	107(2)
		C(2)–C(7)–C(10)	111(2)
		C(6)–C(7)–C(10)	107(2)
		C(1)–C(7)–C(10)	112(2)
		N(2)–C(8)–C(3)	107.4(19)

$[(C_{10}N_2H_{20})(H_2O)](I_3)_2$ (3)

N(1)–C(1)	1.537(11)	C(5)–N(1)–C(1)	109.7(7)
N(1)–C(5)	1.478(12)	C(5)–N(1)–C(10)	109.9(7)
N(1)–C(10)	1.522(12)	C(10)–N(1)–C(1)	110.6(7)
N(2)–C(2)	1.501(11)	C(2)–N(2)–C(6)	111.2(7)
N(2)–C(5)	1.481(11)	C(5)–N(2)–C(2)	110.7(7)
N(2)–C(6)	1.507(12)	C(5)–N(2)–C(6)	110.5(7)
C(9)–C(1)	1.523(12)	C(1)–C(9)–C(2)	108.5(8)
C(9)–C(4)	1.525(13)	C(1)–C(9)–C(4)	109.6(7)
C(9)–C(2)	1.525(13)	C(1)–C(9)–C(7)	108.4(7)
C(9)–C(7)	1.544(13)	C(2)–C(9)–C(7)	109.1(7)
C(3)–C(6)	1.535(13)	C(6)–C(3)–C(7)	109.9(7)
C(3)–C(7)	1.548(13)	C(10)–C(3)–C(6)	108.2(8)
C(3)–C(10)	1.521(12)	C(10)–C(3)–C(7)	107.6(7)
C(3)–C(8)	1.524(13)	C(10)–C(3)–C(8)	108.7(8)
N(3)–C(11)	1.461(13)	C(11)–N(3)–C(13)	110.7(8)
N(3)–C(13)	1.502(13)	C(11)–N(3)–C(18)	110.6(7)
N(3)–C(18)	1.532(12)	C(13)–N(3)–C(18)	111.2(8)
N(4)–C(11)	1.492(12)	C(11)–N(4)–C(14)	110.0(8)
N(4)–C(14)	1.495(13)	C(11)–N(4)–C(15)	110.5(8)
N(4)–C(15)	1.516(12)	C(14)–N(4)–C(15)	112.0(8)
C(17)–C(16)	1.510(14)	C(12)–C(17)–C(14)	109.6(8)
C(17)–C(13)	1.527(14)	C(13)–C(17)–C(14)	106.8(8)
C(17)–C(14)	1.543(13)	C(16)–C(17)–C(13)	109.6(8)
C(17)–C(12)	1.527(14)	C(16)–C(17)–C(14)	110.0(8)
C(20)–C(15)	1.531(14)	C(15)–C(20)–C(18)	108.6(8)
C(20)–C(16)	1.521(14)	C(15)–C(20)–C(19)	110.3(8)
C(20)–C(18)	1.540(14)	C(16)–C(20)–C(15)	109.9(9)
C(20)–C(19)	1.552(14)	C(16)–C(20)–C(18)	109.1(8)
O(1)–H(1C)	0.80(8)	C(16)–C(20)–C(19)	111.8(9)
O(1)–H(1D)	0.87(8)	C(18)–C(20)–C(19)	107.0(9)
O(2)–H(2C)	0.91(8)	H(1C)–O(1)–H(1D)	119(10)

O(2)–H(2D)	0.93(8)	H(2C)–O(2)–H(2D)	114(10)
		N(1)–C(5)–N(2)	109.7(8)
		C(9)–C(1)–N(1)	108.7(7)
		N(2)–C(2)–C(9)	109.4(7)
		N(2)–C(6)–C(3)	109.2(7)
		C(9)–C(7)–C(3)	108.4(7)
		C(3)–C(10)–N(1)	110.5(7)
		N(3)–C(11)–N(4)	108.5(8)
		N(3)–C(13)–C(17)	108.7(8)
		N(4)–C(14)–C(17)	108.3(8)
		N(4)–C(15)–C(20)	107.6(8)
		C(17)–C(16)–C(20)	110.8(8)
		N(3)–C(18)–C(20)	107.1(8)

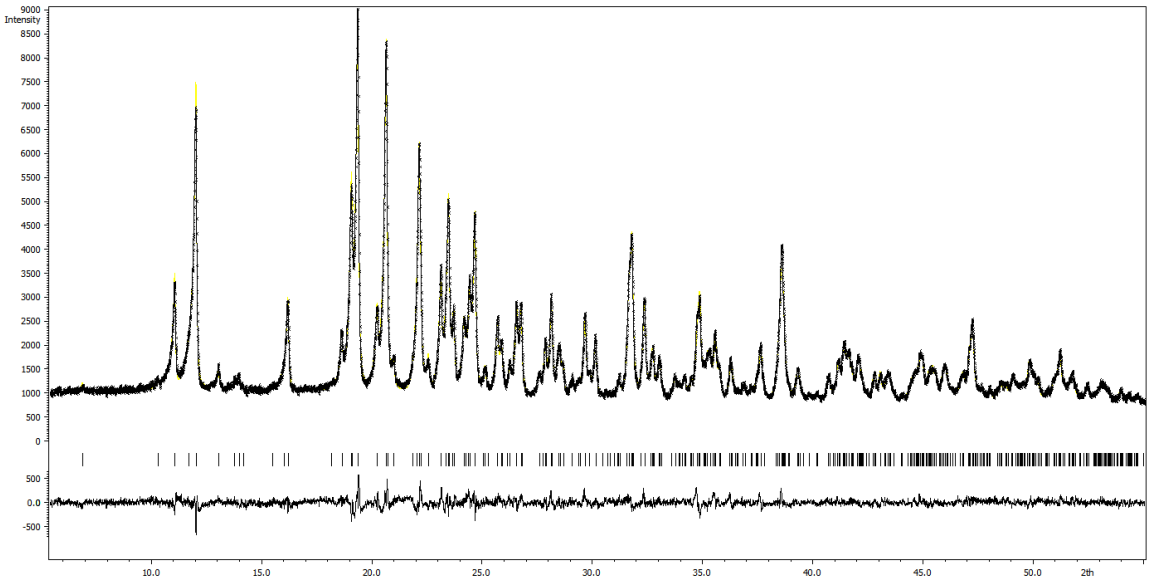

Figure S1. Powder X-ray diffraction pattern and Rietveld refinement for compound **2**. The upper curve represents the experimental diffraction pattern, the ticks show calculated peak positions, and the lower curve is the difference between the experimental and calculated patterns. The most significant area from 5 to 55° 2theta is shown.

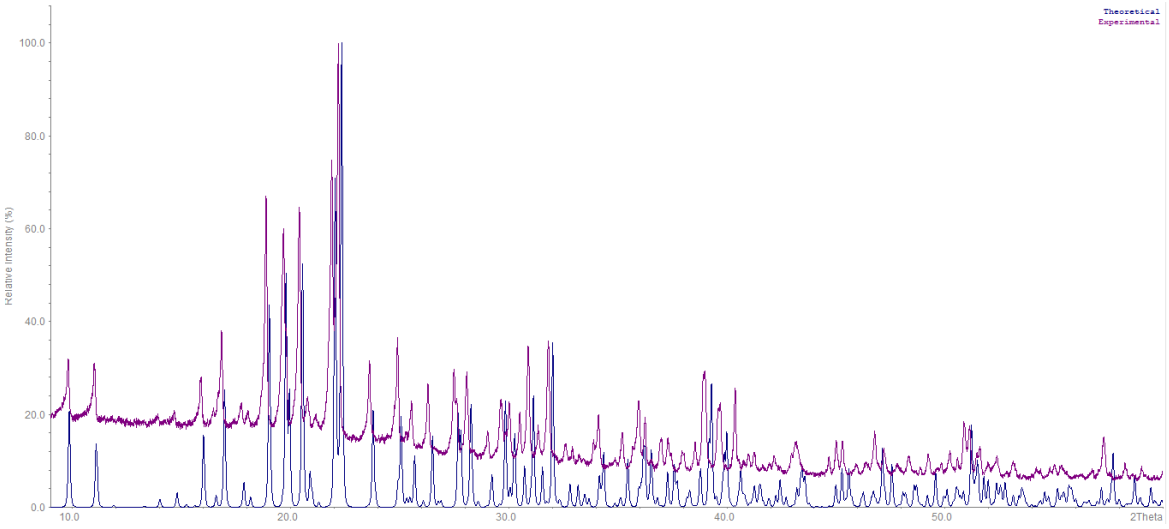

Figure S2. Powder X-ray diffraction pattern for compound **1**. Shown are experimental (purple) and simulated from the crystal data patterns. The most significant area from 8 to 60° 2theta is shown.

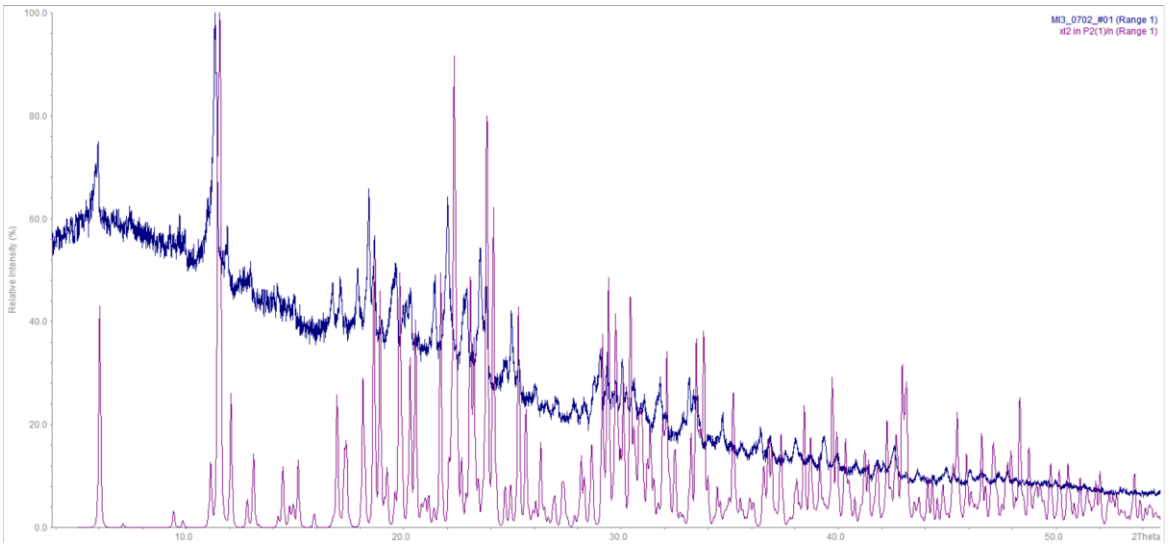

Figure S3. Powder X-ray diffraction pattern for compound **3** Shown are experimental (purple) and simulated from the crystal data patterns. The most significant area from 5 to 55° 2theta is shown.

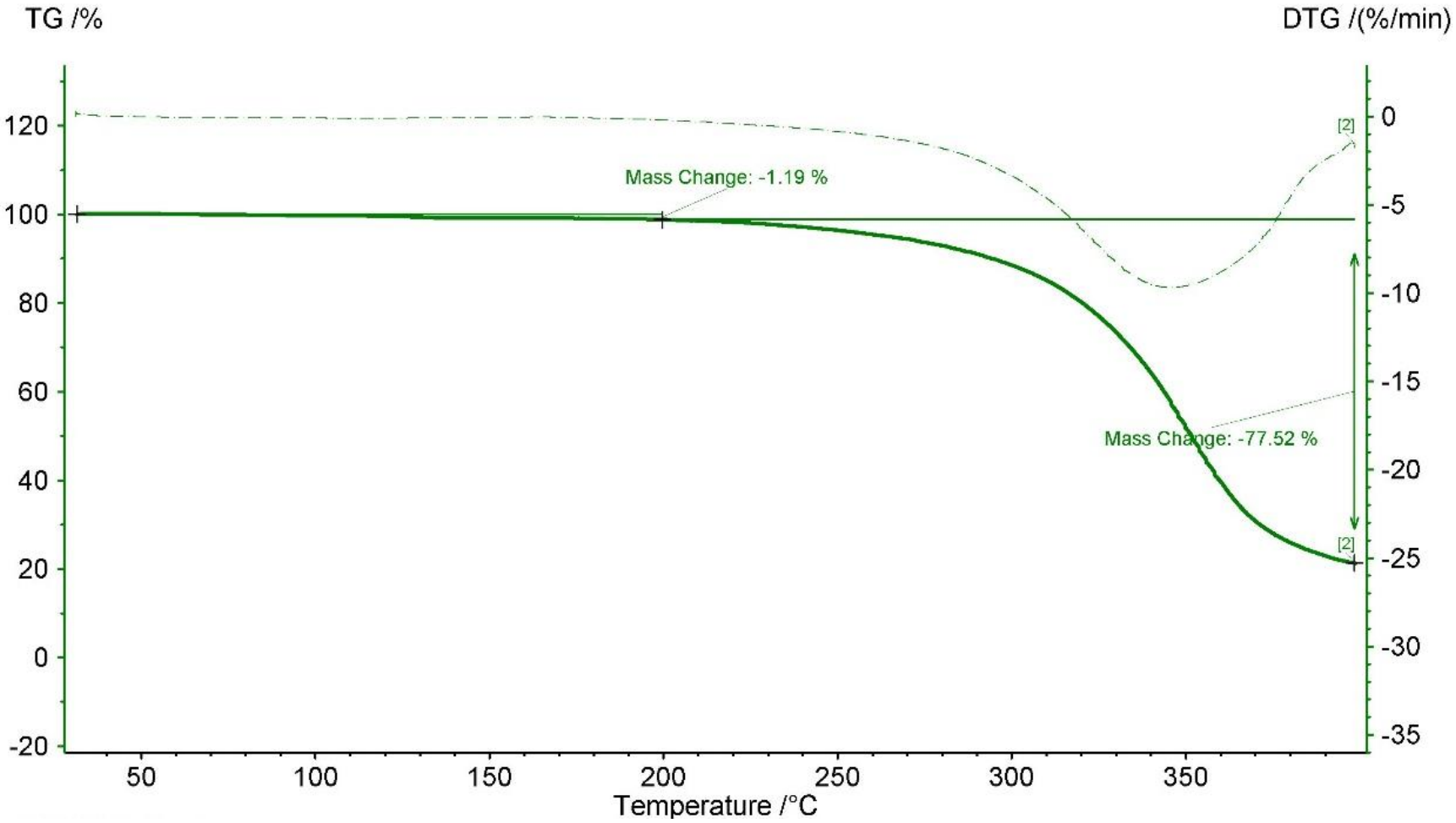

Figure S4. TGA diagram for compound **1**.

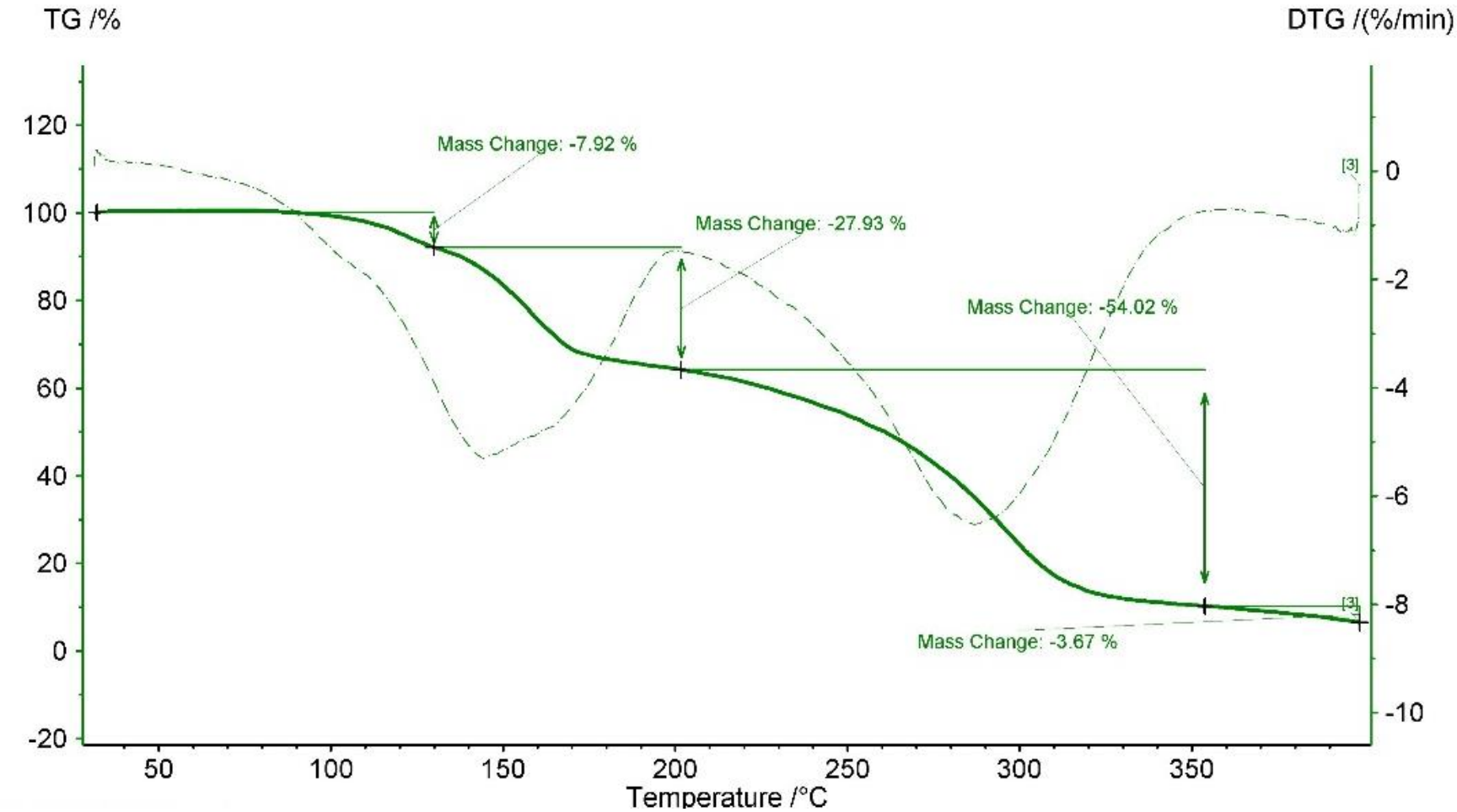

Figure S5. TGA diagram for compound **2**.

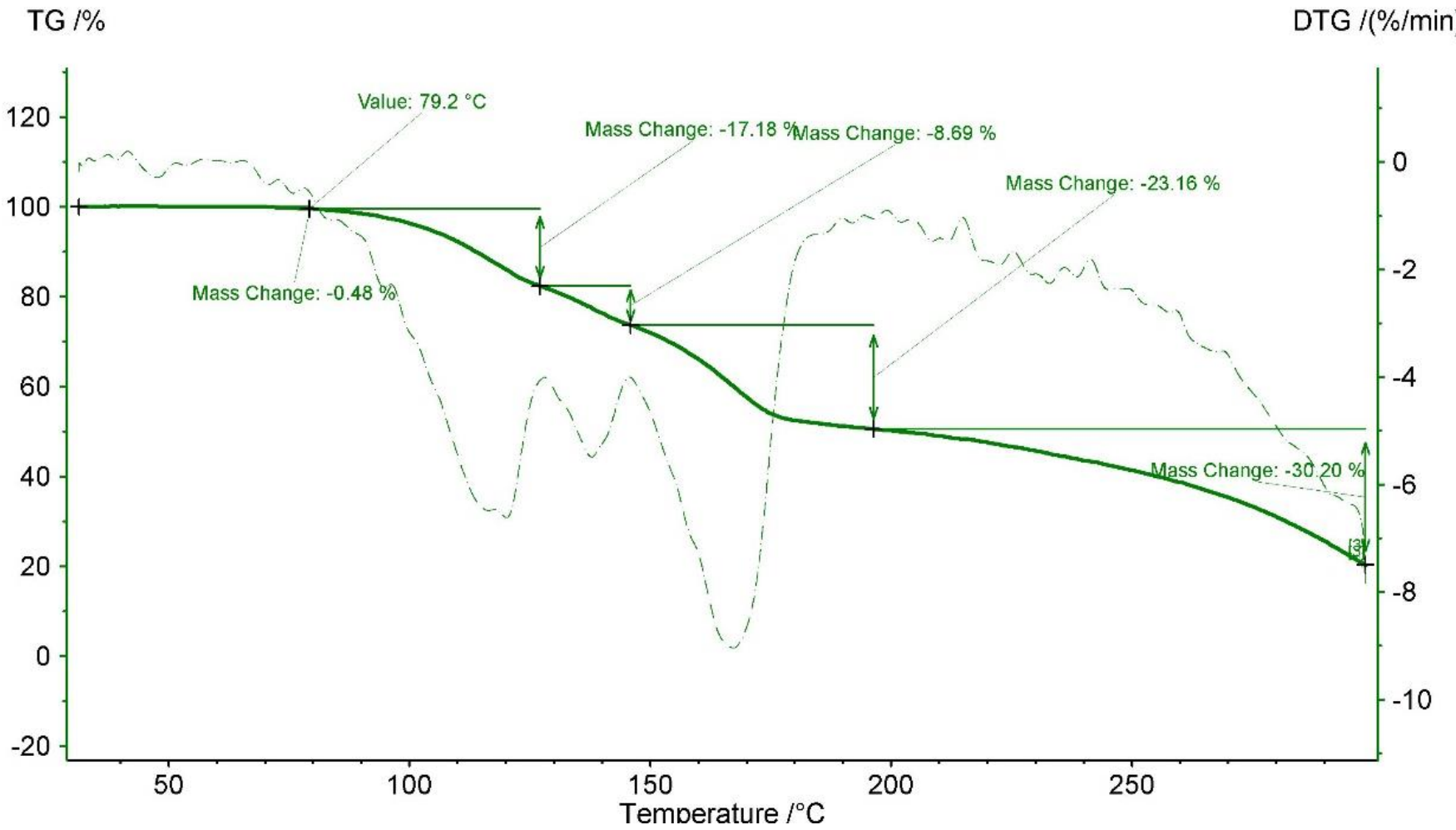

Figure S6. TGA diagram for compound **3**.

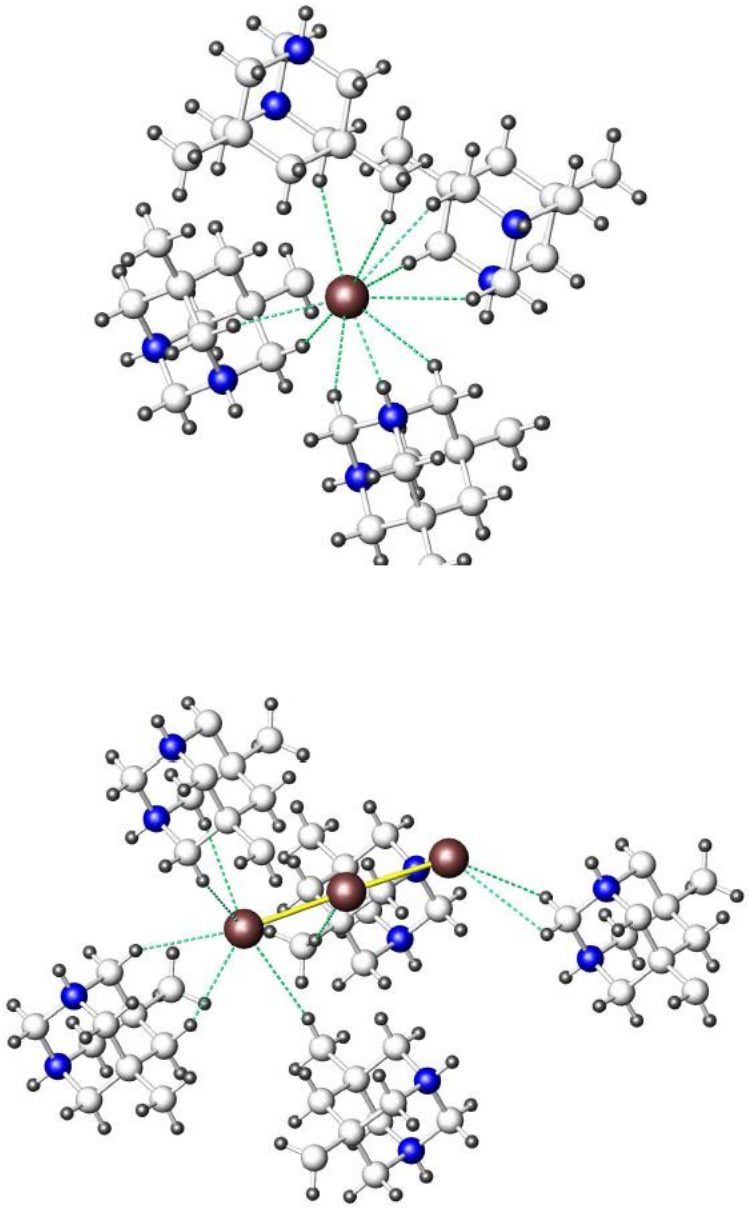

Figure S7. Surrounding of the guest I^- anions in the crystal structure of **1** (top) and of I_3^- anions in the crystal structure of **2** (bottom). Iodine, brown; nitrogen, blue; carbon, light grey; hydrogen, dark grey. Hydrogen (C)H…I bonds are shown by dashed green lines.

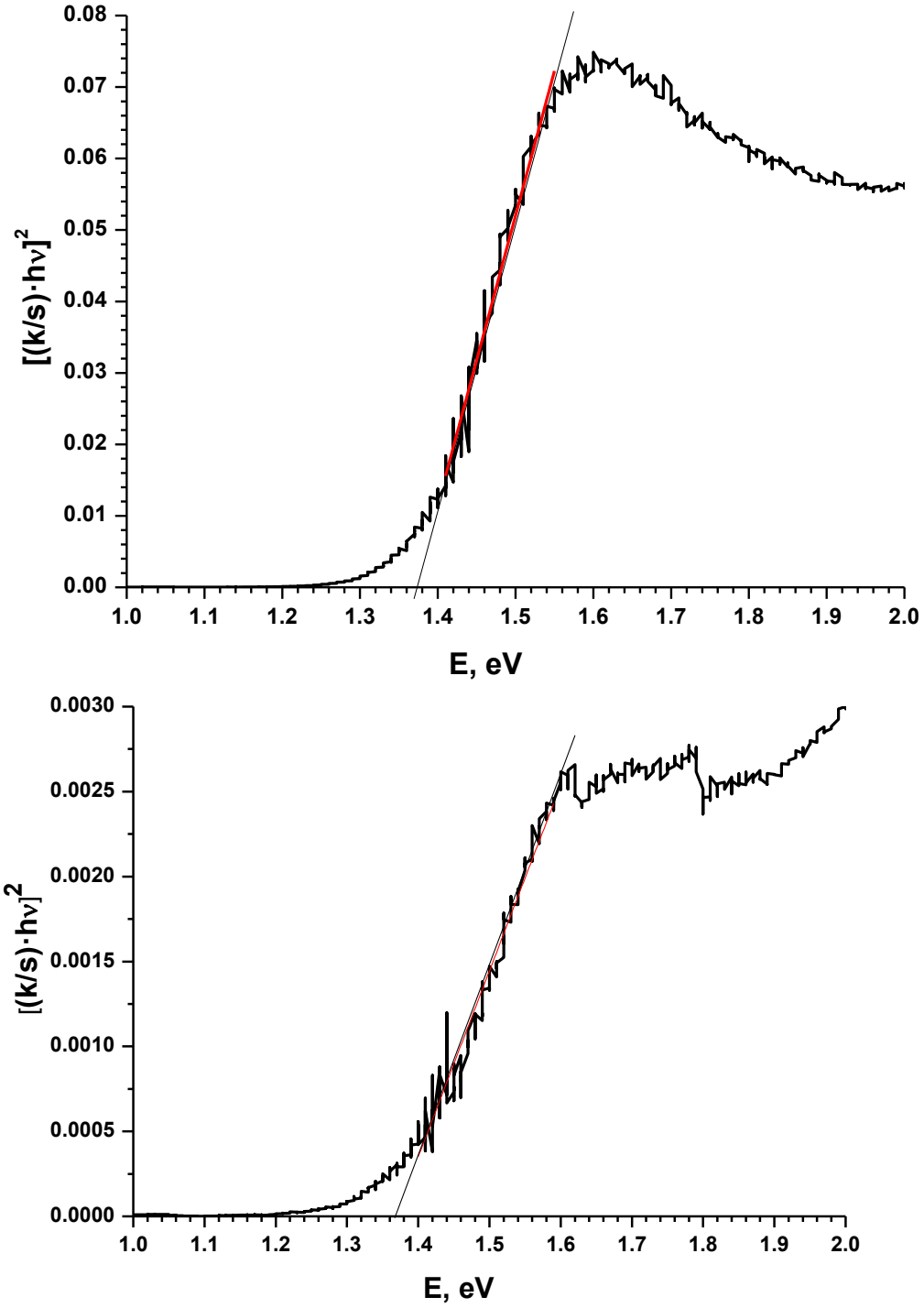

Figure S8. Kubelka-Munk plots for compounds **2** (top) and **3** (bottom).

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