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SUPPLEMENTARY DATA

Solution and solid state characterization of "sparteine surrogate" (+)-(1*R*,5*S*,11a*S*)-tetrahydrodeoxocytisine

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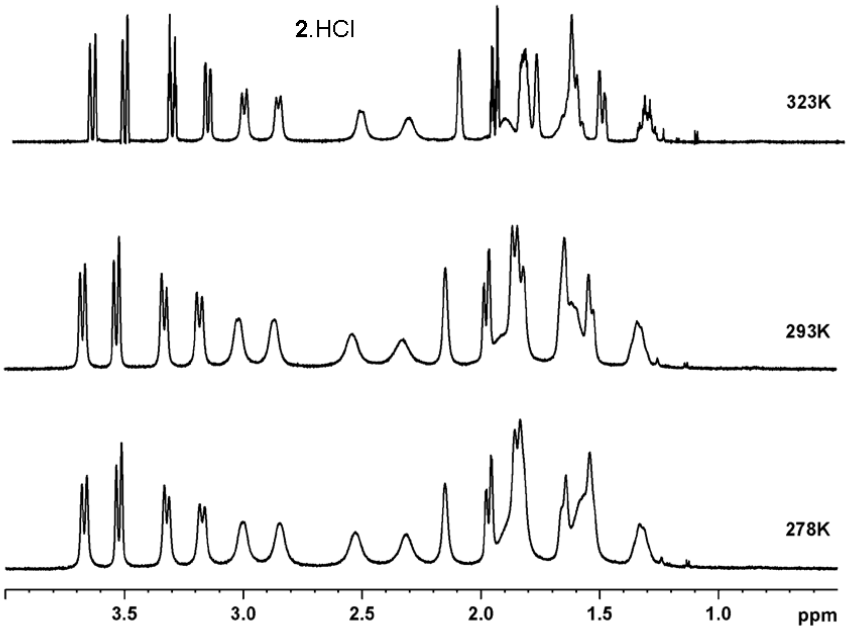

Fig. S1. Proton NMR spectra of **2**.HCl at different temperatures.

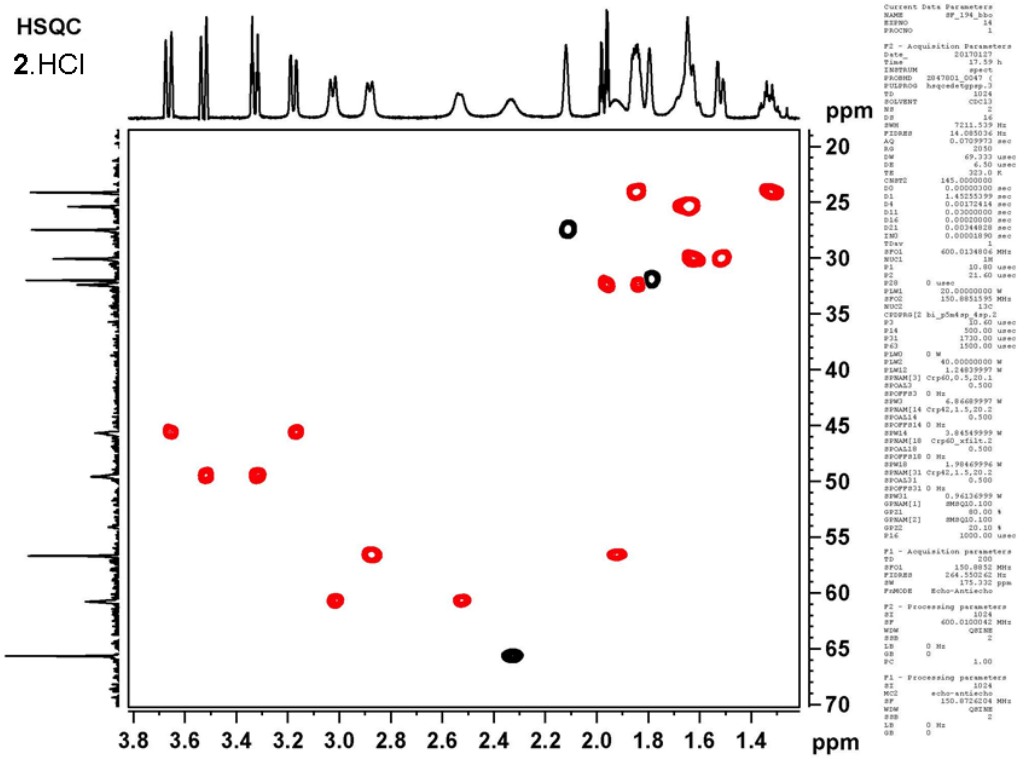

Fig. S2. HSQC spectrum of **2**.HCl.

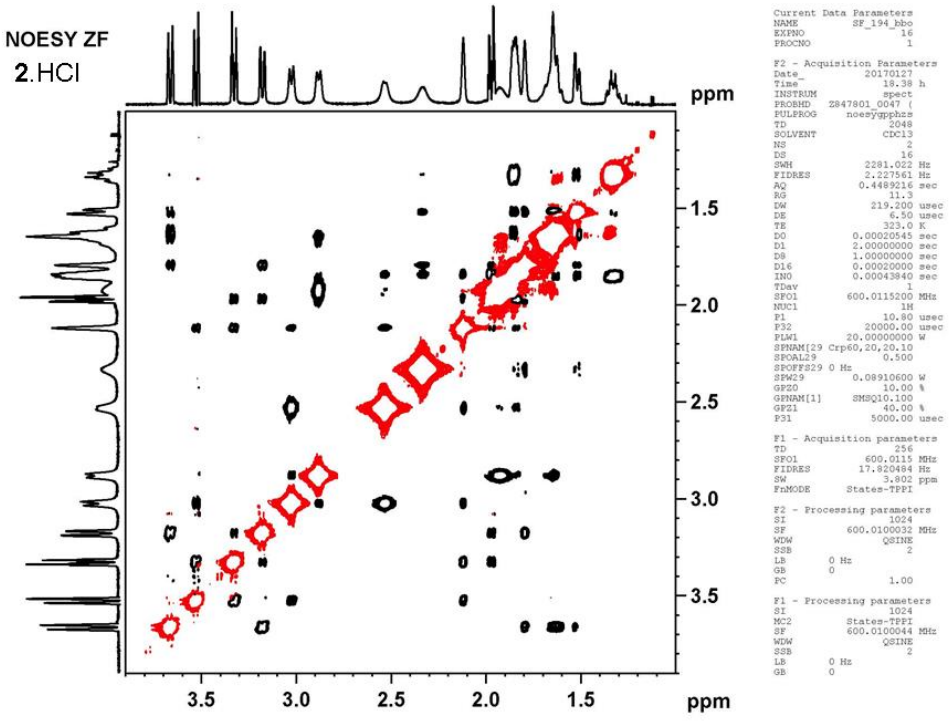

Fig. S3. NOESY ZF spectrum of **2**.HCl.

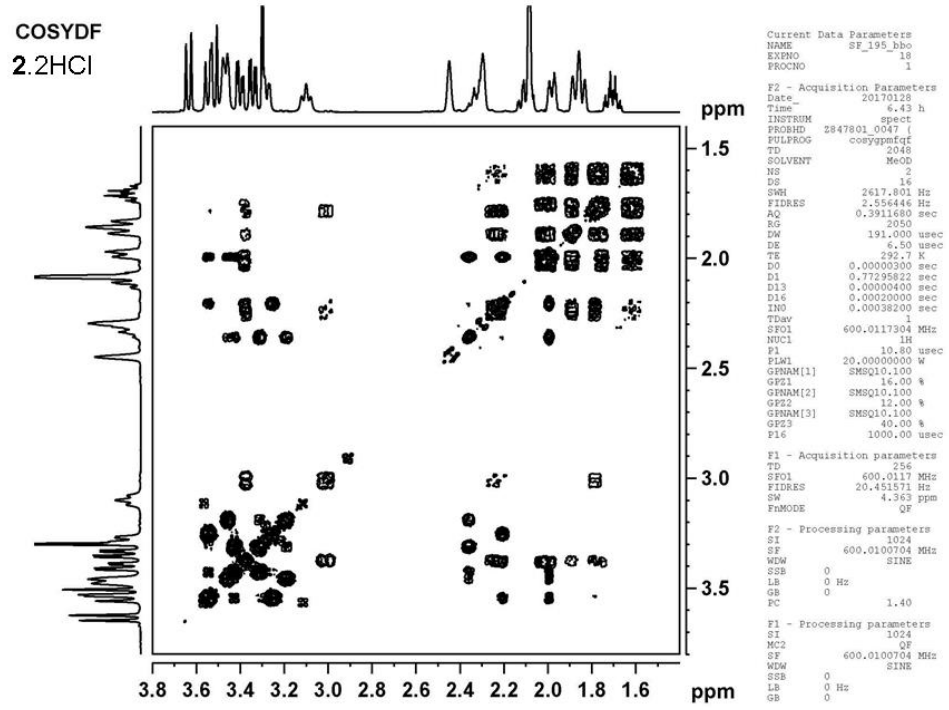

Fig. S4. COSYDF spectrum of **2**.2HCl.

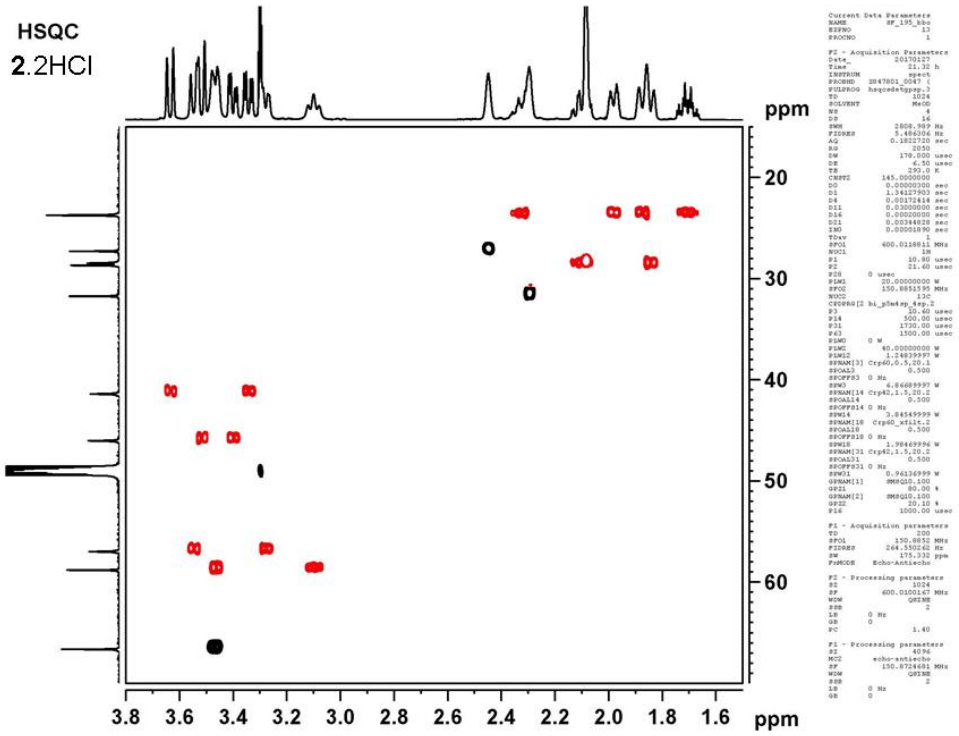

Fig. S5. HSQC spectrum of **2**.2HCl.

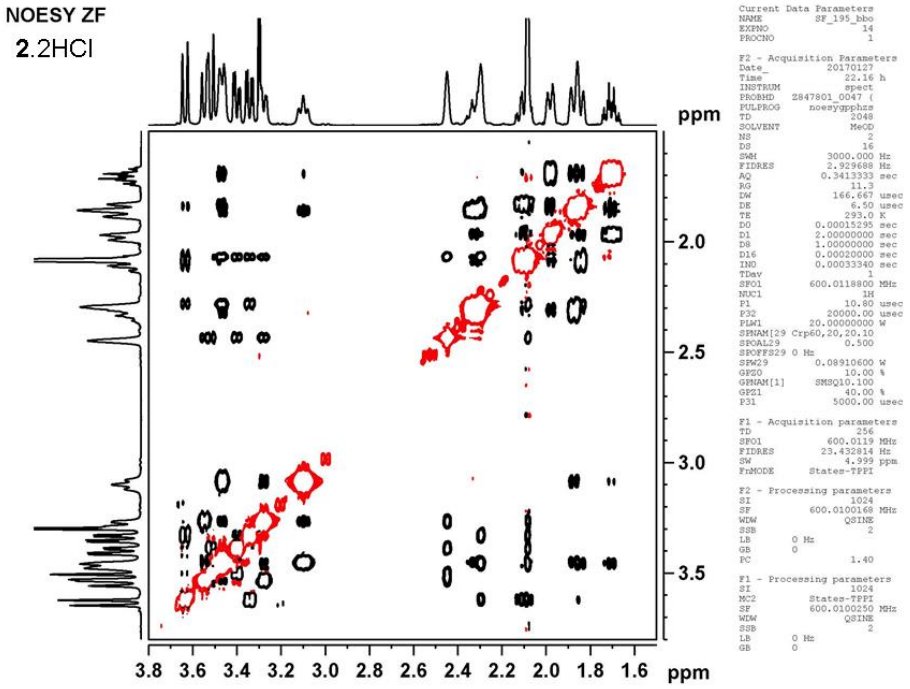

Fig. S6. NOESY ZF spectrum of **2**.2HCl.

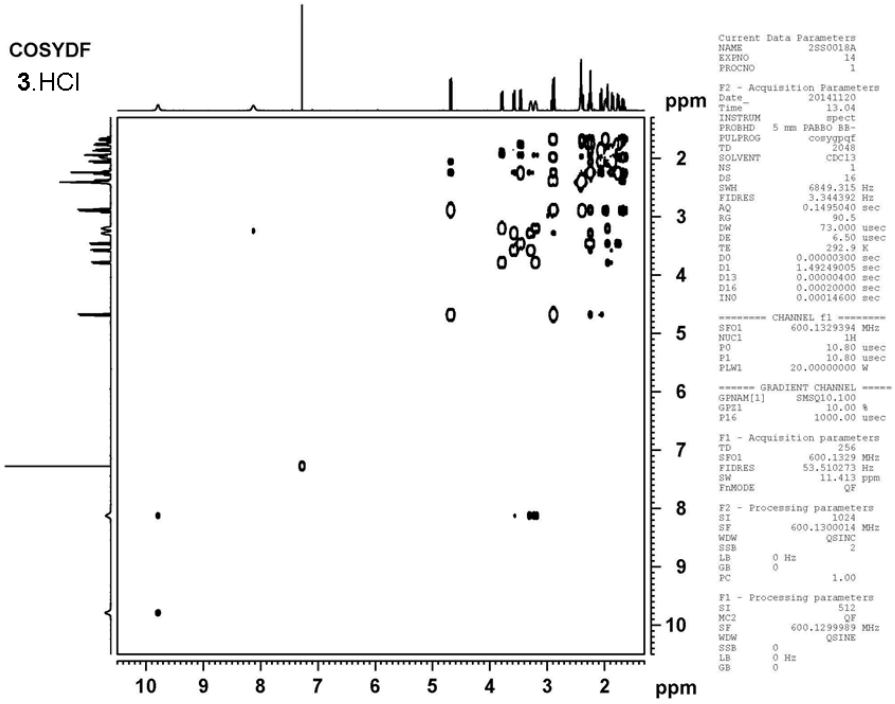

Fig. S7. COSYDF spectrum of **3**.HCl.

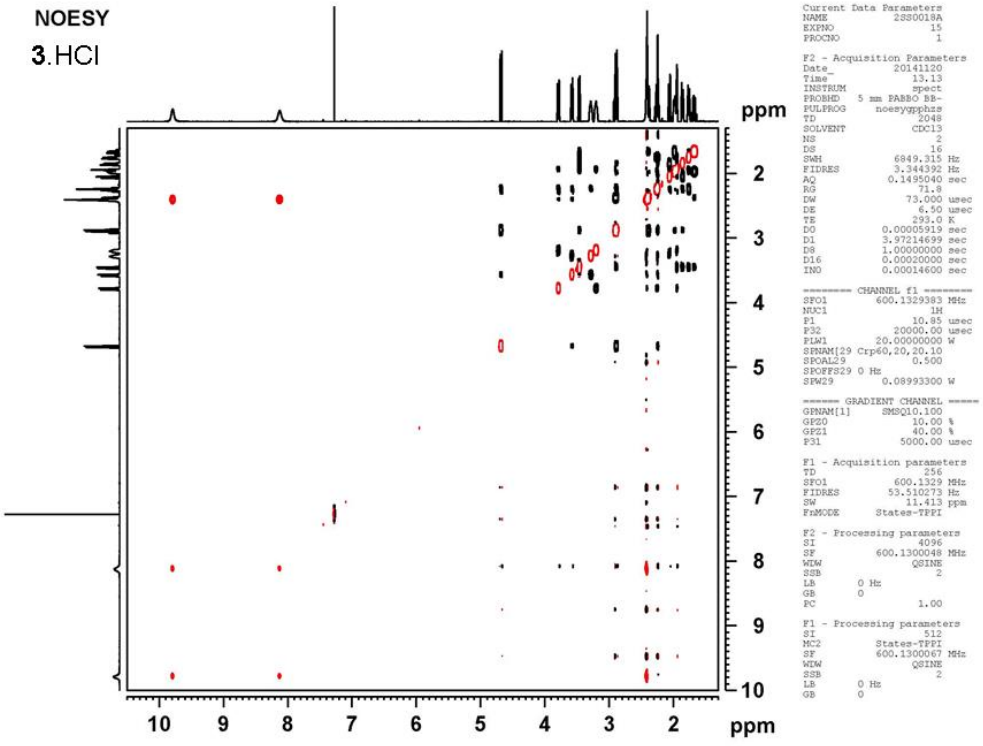

Fig. S8. NOESY ZF spectrum of **3**.HCl.

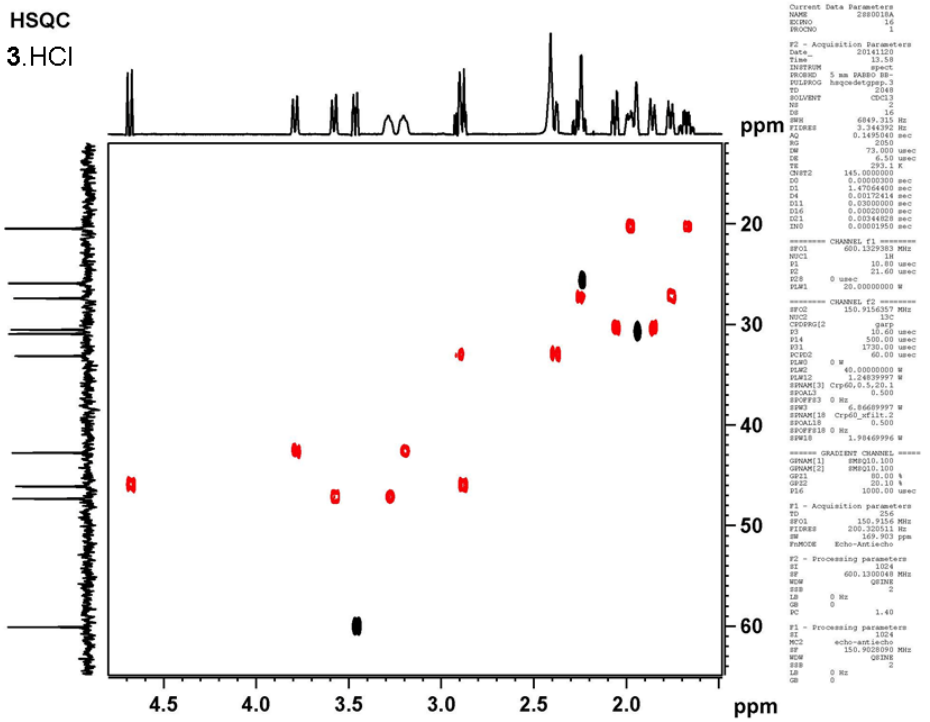

Fig. S9. HSQC spectrum of **3**.HCl.

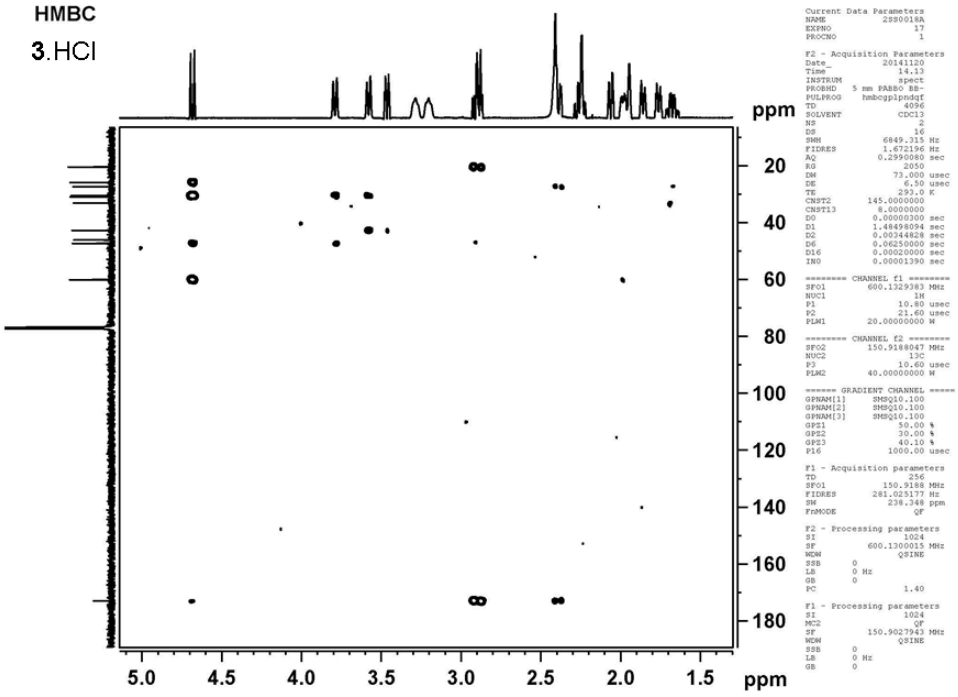

Fig. S10. HMBC spectrum of **3**.HCl.

Table S1. Most important data collection and refinement indicators for **2**.2HCl, **2**.HCl, and **3**.HCl

	2.2HCl	**2**.HCl	**3**.HCl
Chemical formula	$2(Cl)\cdot C_{11}H_{22}N_2\cdot 0.28(H_2O)$	$Cl\cdot C_{11}H_{21}N_2\cdot 0.41(H_2O)$	$C_{11}H_{19}N_2O\cdot H_2O\cdot Cl$
M_r	258.25	224.13	248.75
Crystal system, space group	Orthorhombic, $P2_12_12_1$	Monoclinic, $C2$	Orthorhombic, $P2_12_12_1$
Temperature (K)	290	290	290
a, b, c (Å)	7.7173(11), 9.3085(12), 18.737(3)	11.794(2), 7.7031(9), 13.9152(19)	7.602(3), 11.840(5), 13.840(3)
α, β, γ (°)	90, 90, 90	90, 97.170(15), 90	90, 90, 90
V (Å^3)	1346.0 (3)	1254.3 (3)	1245.8 (8)
Z	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.46	0.28	0.30
Crystal size (mm)	0.3 × 0.2 × 0.15	0.28 × 0.16 × 0.14	0.3 × 0.25 × 0.22
Data collection			
Diffractometer	SuperNova, Dual, Cu at zero, Atlas	SuperNova, Dual, Cu at zero, Atlas	CAD4 Enraf nonius
Absorption correction	Multi-scan	Multi-scan	None
T_{min}, T_{max} or decay	0.513, 1.000	0.592, 1.000	0.935
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4492, 2807, 1717	3752, 2578, 1912	2815, 2644, 1782
R_{int}	0.076	0.032	0.056
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.691	0.690	0.660
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.075, 0.195, 1.03	0.057, 0.180, 1.05	0.063, 0.175, 1.01
No. of reflections	2807	2578	2644
No. of parameters	170	157	168
No. of restraints	0	1	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement		
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.53, −0.41	0.29, −0.30	0.36, −0.34
Absolute structure	Flack x determined using 371 quotients [(I+)-(I-)]/[(I+)+(I-)][1]	Flack x determined using 604 quotients [(I+)-(I-)]/[(I+)+(I-)][1]	Flack x determined using 528 quotients [(I+)-(I-)]/[(I+)+(I-)] [1]
Absolute structure parameter	−0.29 (17)	0.02 (14)	−0.23 (14)

Table S2. Bond lengths for **2**.2HCl, **2**.HCl, and **3**.HCl

	2.2HCl	**2**.HCl	**3**.HCl
N7A—C11A	1.526(8)	1.456(5)	1.483(7)
N7A—C7	1.494(9)	1.471(6)	1.487(7)
N7A—C8	1.506(9)	1.459(6)	1.365(8)
N7A—H7A	0.93(6)	-	-
N3—H3A	1.00 (7)	0.89 (7)	0.94 (7)
N3—H3B	1.09 (7)	0.79 (6)	0.82 (8)
N3—C4	1.480 (8)	1.484 (7)	1.490 (8)
N3—C2	1.473 (8)	1.479 (6)	1.492 (8)
C11A—C1	1.500 (9)	1.525 (6)	1.545 (8)
C11A—C11	1.514 (9)	1.510 (6)	1.508 (8)
C11A—H11A	1.05 (7)	0.96 (4)	1.05 (5)
C1—C2	1.520 (9)	1.523 (6)	1.517 (8)
C1—C6	1.516 (10)	1.509 (7)	1.527 (7)
C1—H1	1.02 (6)	0.96 (7)	1.047 (7)
C9—C10	1.512 (11)	1.508 (7)	1.510 (9)
C9—C8	1.492 (11)	1.500 (7)	1.504 (8)
C5—H5	0.93 (6)	0.94 (6)	1.06 (6)
C11—C10	1.534 (11)	1.514(7)	1.509 (9)
C6—C5	1.521 (10)	1.501(8)	1.520 (9)
C7—C5	1.511 (10)	1.510 (8)	1.519 (8)
C5—C4	1.544 (10)	1.525(8)	1.517 (9)
O8—C8	-	-	1.216 (7)
O10W—H10C	0.9321	0.93	0.8496
O10W—H10D	0.9265	0.9291	0.8498

Table S3. Hydrogen Bonds geometry for **2**.2HCl, **2**.HCl, and **3**.HCl

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
Compound **2**.2HCl				
C2—H2*B*···Cl7^{i}	0.97	2.82	3.761 (7)	164
C11—H11*B*···O10*W*^{ii}	0.97	2.59	3.40 (3)	140
C8—H8*A*···Cl3^{iii}	0.97	2.80	3.679 (7)	152
C8—H8*B*···Cl3^{iv}	0.97	2.84	3.766 (8)	159
C4—H4*A*···Cl7	0.97	2.79	3.494 (6)	130
C4—H4*B*···O10*W*^{v}	0.97	2.64	3.43 (3)	139
O10*W*—H10*D*···Cl7^{vi}	0.93	2.17	3.10 (2)	179
N3—H3*A*···Cl7^{vii}	1.00 (7)	2.14 (7)	3.094 (6)	158 (5)
N7*A*—H7*A*···Cl7^{vii}	0.93 (6)	2.18 (7)	3.099 (5)	170 (5)
N3—H3*B*···Cl3^{v}	1.09 (7)	1.97 (7)	3.021 (6)	160 (5)
C1—H1···O10*W*^{ii}	1.02 (6)	2.32 (7)	3.29 (4)	158 (5)
C11*A*—H11*A*···Cl3^{iii}	1.05 (7)	2.68 (7)	3.666 (7)	156 (5)

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) *x*−1, *y*, *z*; (iii) *x*−1/2, −*y*+1/2, −*z*+1; (iv) *x*, *y*+1, *z*; (v) −*x*+2, *y*+1/2, −*z*+3/2; (vi) *x*+1, *y*−1, *z*; (vii) −*x*+2, *y*−1/2, −*z*+3/2.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
Compound **2**.HCl				
C2—H2*A*···Cl3^{i}	0.97	2.93	3.820 (5)	153
C4—H4*B*···O10*W*	0.97	2.53	3.382 (14)	147
O10*W*—H10*C*···Cl3^{i}	0.93	2.21	3.137 (17)	179
O10*W*—H10*D*···Cl3^{ii}	0.93	2.37	3.299 (15)	179
N3—H3*A*···Cl3^{iii}	0.89 (7)	2.21 (7)	3.068 (5)	160 (6)
N3—H3*B*···Cl3^{iv}	0.70 (6)	2.57 (6)	3.217 (5)	156 (6)
N3—H3*B*···N7*A*	0.70 (6)	2.45 (5)	2.806 (6)	113 (5)

Symmetry codes: (i) *x*+1/2, *y*−1/2, *z*; (ii) −*x*+1/2, *y*−1/2, −*z*+1; (iii) −*x*+1, *y*, −*z*+1; (iv) *x*+1/2, *y*+1/2, *z*.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
Compound **3**.HCl				
C9—H9*A*···O10*W*^{i}	0.97	2.57	3.332 (8)	135
C4—H4*A*···O10*W*^{ii}	0.97	2.55	3.370 (8)	143
O10*W*—H10*C*···Cl3^{iii}	0.85	2.38	3.230 (5)	174
O10*W*—H10*D*···O8	0.85	1.92	2.770 (7)	177
N3—H3*A*···Cl3	0.94 (7)	2.20 (7)	3.090 (6)	157 (6)
N3—H3*B*···Cl3^{iv}	0.82 (8)	2.46 (8)	3.178 (6)	147 (6)
C5—H5···O8^{ii}	1.06 (6)	2.42 (6)	3.427 (7)	157 (5)
C1—H1···O10*W*^{v}	1.05 (7)	2.65 (7)	3.639 (8)	157 (5)

Symmetry codes: (i) −*x*, *y*−1/2, −*z*−1/2; (ii) *x*−1/2, −*y*−3/2, −*z*; (iii) −*x*−1/2, −*y*−2, *z*−1/2; (iv) *x*+1/2, −*y*−5/2, −*z*; (v) −*x*−1, *y*−1/2, −*z*−1/2.

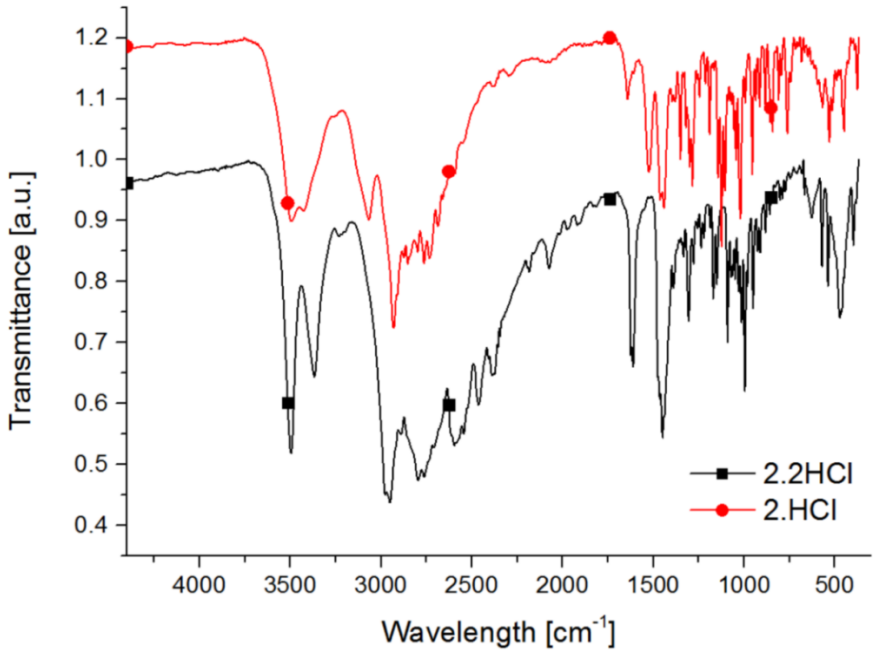

Fig. S11. FTIR spectrum of **2**.2HCl and **2**.HCl (KBr).

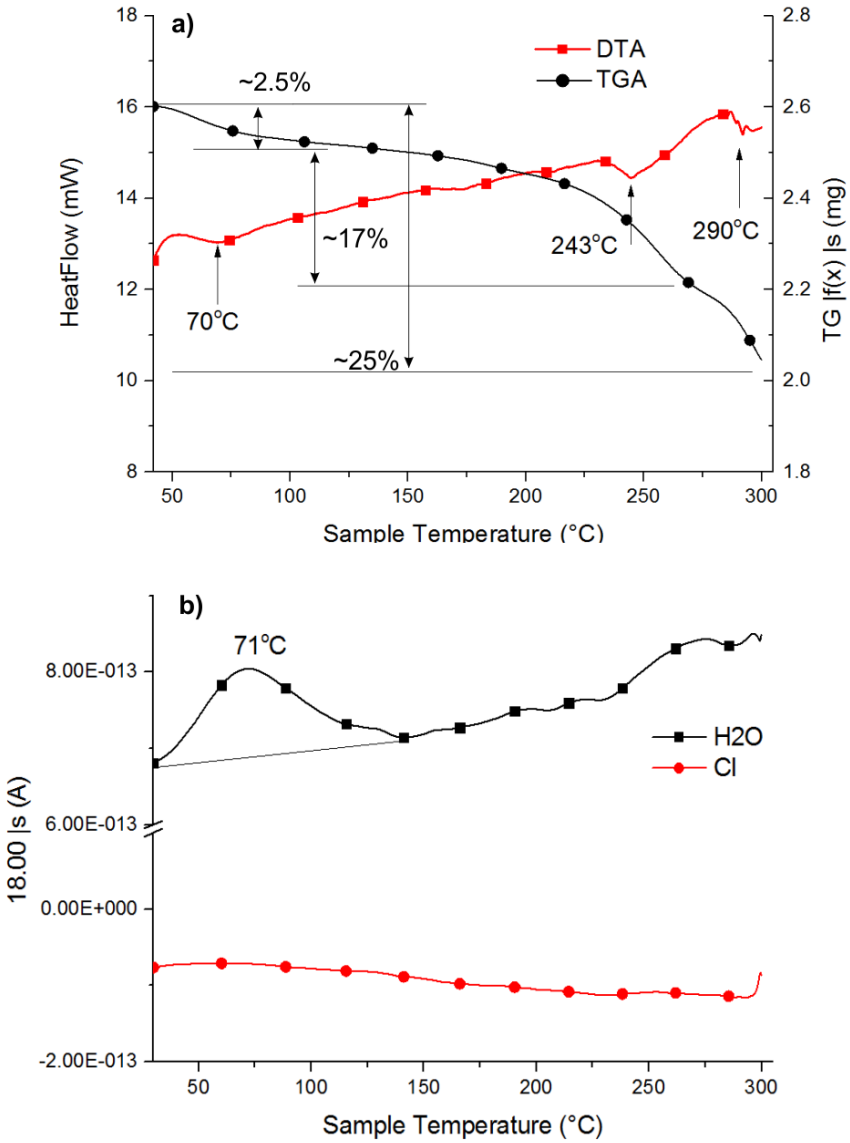

Fig. S12. Representation of a) DTA and TGA curves and b) gaz mass evolving detection of 18 u (water) and 35 u (chlorine) of **2**.2HCl; detection of 17 u (OH^-) and 36 u (HCl) was not observed.

References

[1] Parsons, Flack and Wagner, *Acta Cryst*. **B69** 249 (2013).