Return of the lysergamides. Part IV: Analytical and pharmacological characterization of lysergic acid morpholide (LSM-775)

Simon D. Brandt,[a] Pierce V. Kavanagh,[b] Brendan Twamley,[c] Folker Westphal,[d] Simon P. Elliott,[e] Jason Wallach,[f] Alexander Stratford,[g] Landon M. Klein,[h] John D. McCorvy,[i] David E. Nichols,[j] Adam L. Halberstadt[k,*]

[a] *School of Pharmacy and Biomolecular Sciences, Liverpool John Moores University, Byrom Street, Liverpool L3 3AF, UK*

[b] *Department of Pharmacology and Therapeutics, School of Medicine, Trinity Centre for Health Sciences, St. James Hospital, Dublin 8, Ireland*

[c] *School of Chemistry, Trinity College Dublin, Dublin 2, Ireland*

[d] *State Bureau of Criminal Investigation Schleswig-Holstein, Section Narcotics/Toxicology, Mühlenweg 166, D-24116 Kiel, Germany*

[e] *Alere Forensics (Forensics Ltd), Malvern Hills Science Park, Geraldine Road, WR14 3SZ, UK*

[f] *Department of Pharmaceutical Sciences, Philadelphia College of Pharmacy, University of the Sciences, 600 South 43rd Street, Philadelphia, PA 19104, USA*

[g] *Synex Synthetics BV, Poortweg 4, 2612 PA Delft, The Netherlands*

[h] *Department of Neurosciences, University of California San Diego, 9500 Gilman Drive, La Jolla, CA 92093, USA*

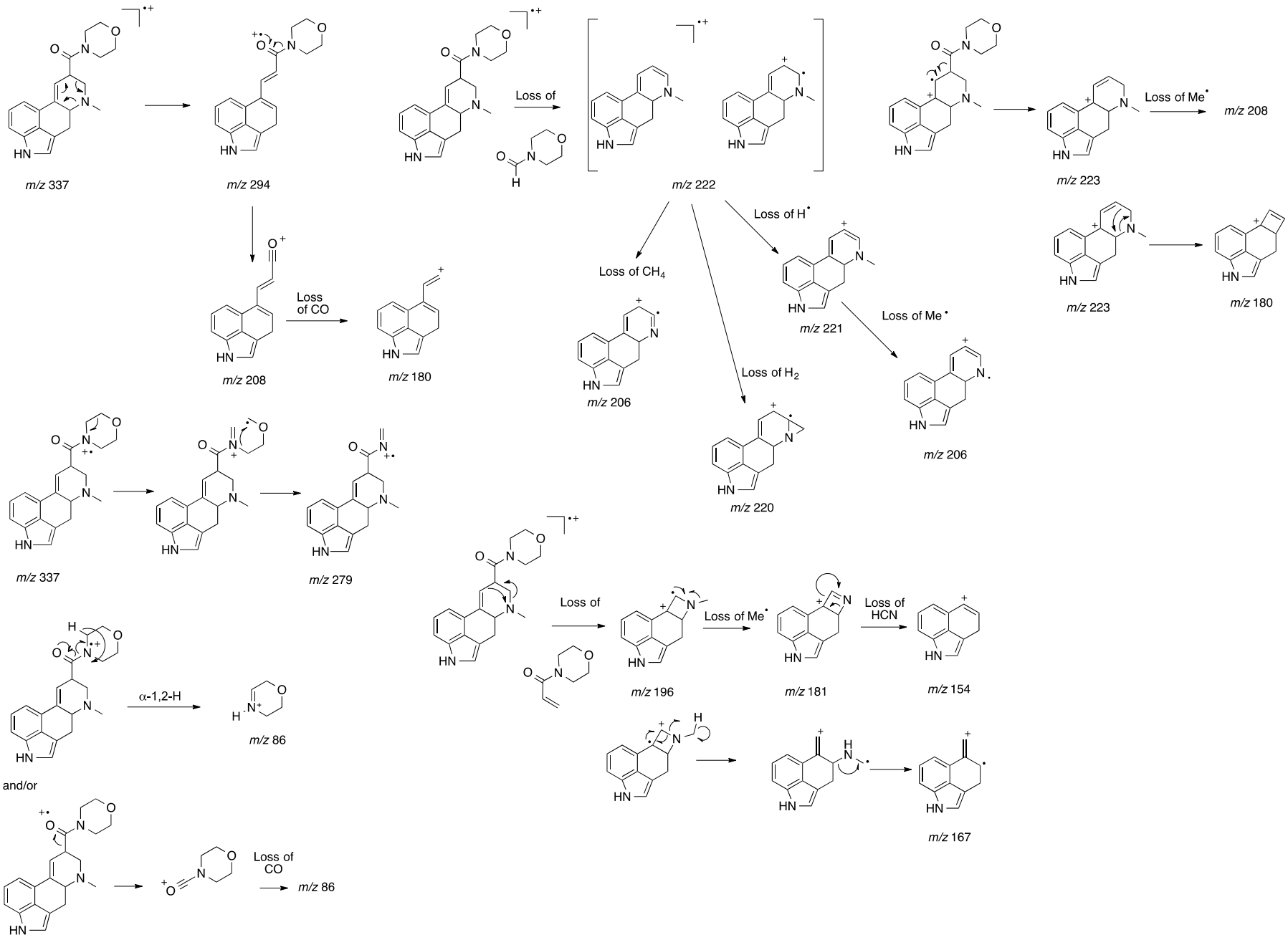
[i] *Department of Pharmacology, University of North Carolina, Genetic Medicine Building, 120 Mason Farm Road, Chapel Hill, NC 27599-7365, USA*

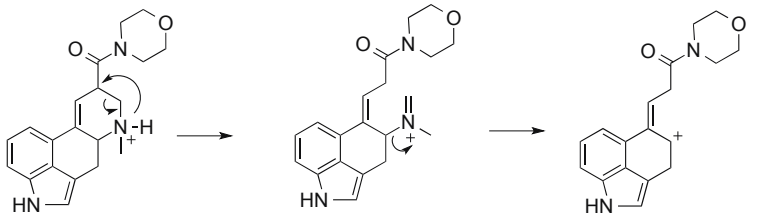
[i] *Division of Chemical Biology and Medicinal Chemistry, University of North Carolina, Genetic Medicine Building, 120 Mason Farm Road, Chapel Hill, NC 27599, USA*

[k] *Department of Psychiatry, University of California San Diego, 9500 Gilman Drive, La Jolla, CA 92093, USA*

* Correspondence to: Adam L. Halberstadt, Department of Psychiatry, University of California San Diego, 9500 Gilman Dr., La Jolla, CA 92093-0804 USA. E-Mail: ahalberstadt@ucsd.edu

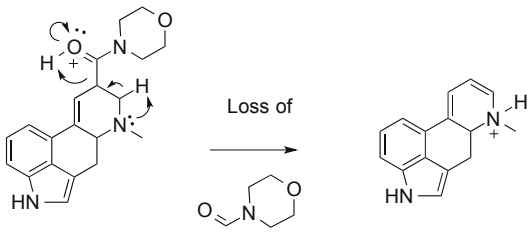
Content	Page
Photograph of powdered LSM-775 tartrate sample	S2
Proposed LSM-775 fragmentation pathways (EI-MS)	S3
Proposed LSM-775 dissociation pathways (QTOF-MS/MS)	S4
HPLC-DAD analysis of LSM-775 powder	S5
^{1}H NMR of LSM-775 hemitartrate (d_6-DMSO)	S6–S8
^{13}C and DEPTQ of LSM-775 hemitartrate (d_6-DMSO)	S9
HSQC of LSM-775 hemitartrate (d_6-DMSO)	S10–S12
HMBC of LSM-775 hemitartrate (d_6-DMSO)	S13–S18
^{1}H NMR of LSM-775 hemitartrate (CD_3OD)	S19
^{1}H and ^{13}C NMR data for LSM-775 hemitartrate in CD_3OD at 400 / 100 MHz (20 mg/mL)	S20
^{13}C and PENDANT NMR of LSM-775 hemitartrate (CD_3OD)	S21
^{1}H/^{1}H COSY of LSM-775 hemitartrate (CD_3OD)	S22
HMQC of LSM-775 hemitartrate (CD_3OD)	S23
HMBC of LSM-775 hemitartrate (CD_3OD)	S24
X-ray data for recrystallized LSM-775	S25–S43


m/z 337
m/z 294
Loss of
m/z 222
Loss of Me•
m/z 208
m/z 223
Loss of CO
m/z 208
m/z 180
Loss of CH4
Loss of H•
m/z 221
m/z 223
m/z 180
m/z 206
Loss of Me•
m/z 206
Loss of H2
m/z 220
m/z 337
m/z 279
Loss of
m/z 196
Loss of Me•
m/z 181
Loss of HCN
m/z 154
α-1,2-H
m/z 86
and/or
m/z 167
Loss of CO
m/z 86



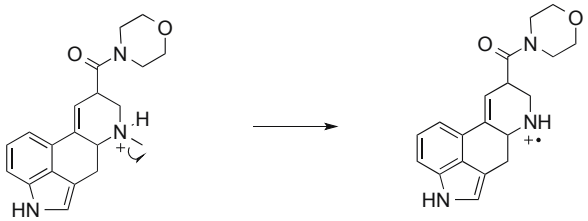
Chemical Formula: $C_{20}H_{24}N_3O_2^+$
Exact Mass: 338.18630

Chemical Formula: $C_{18}H_{19}N_2O_2^+$
Exact Mass: 295.14410



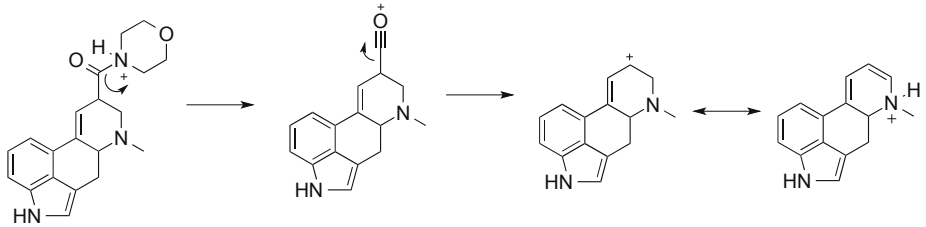
Chemical Formula: $C_{20}H_{24}N_3O_2^+$
Exact Mass: 338.18630

Chemical Formula: $C_{15}H_{15}N_2^+$
Exact Mass: 223.12297



Chemical Formula: $C_{20}H_{24}N_3O_2^+$
Exact Mass: 338.18630

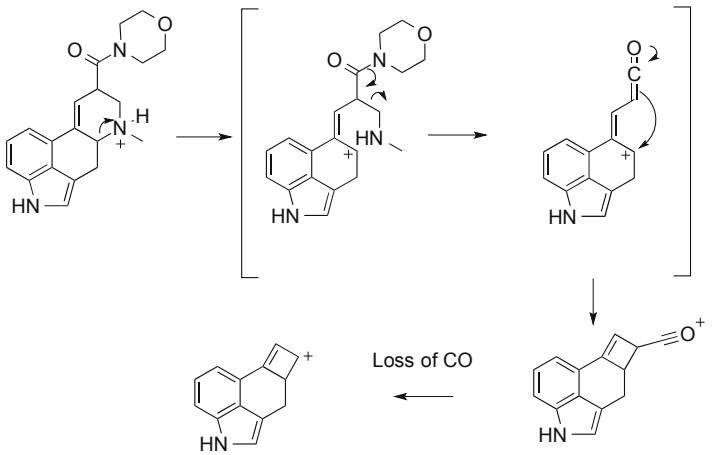
Chemical Formula: $C_{19}H_{21}N_3O_2^{\bullet+}$
Exact Mass: 323.16283



Chemical Formula: $C_{20}H_{24}N_3O_2^+$
Exact Mass: 338.18630

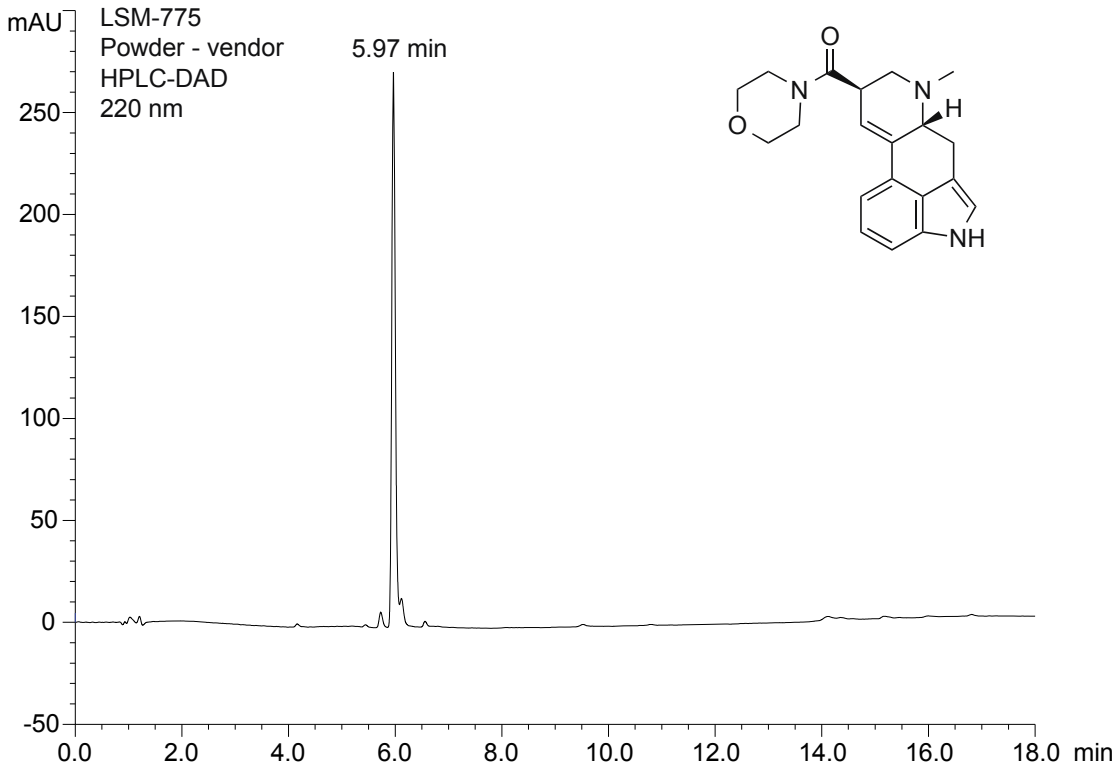
Chemical Formula: $C_{16}H_{15}N_2O^+$
Exact Mass: 251.11789

Chemical Formula: $C_{15}H_{15}N_2^+$
Exact Mass: 223.12297

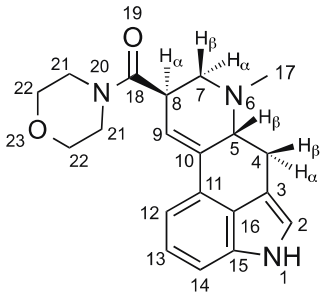


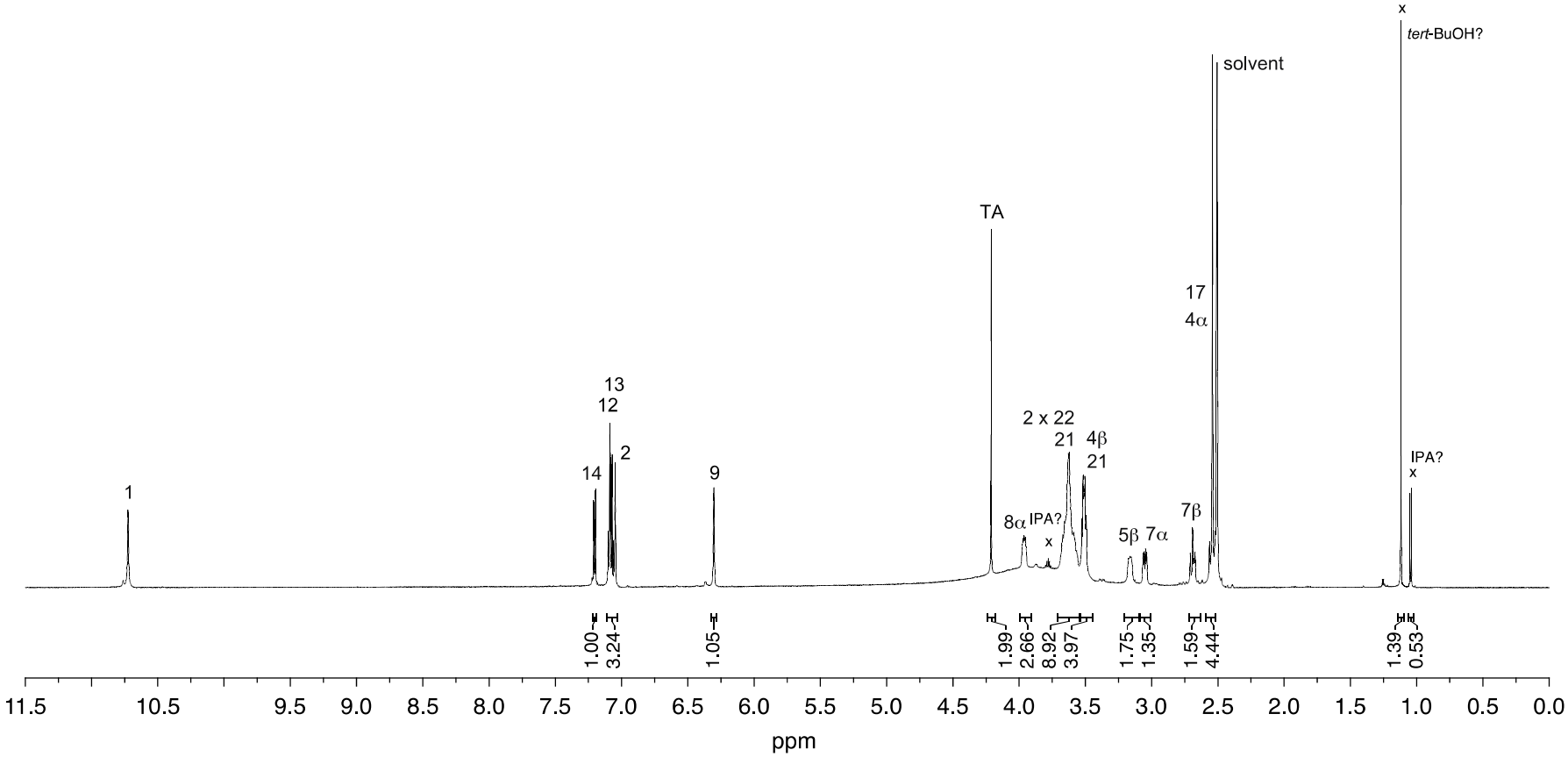
Chemical Formula: $C_{13}H_{10}N^+$
Exact Mass: 180.08078

Chemical Formula: $C_{14}H_{10}NO^+$
Exact Mass: 208.07569


mAU
LSM-775
Powder - vendor
HPLC-DAD
220 nm
5.97 min
250
200
150
100
50
0
-50
0.0
2.0
4.0
6.0
8.0
10.0
12.0
14.0
16.0
18.0 min
O
N
O
N
H
NH

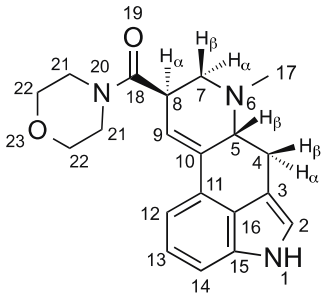
LSM-775 hemitartrate
^{1}H NMR / 600 MHz
d_6 - DMSO

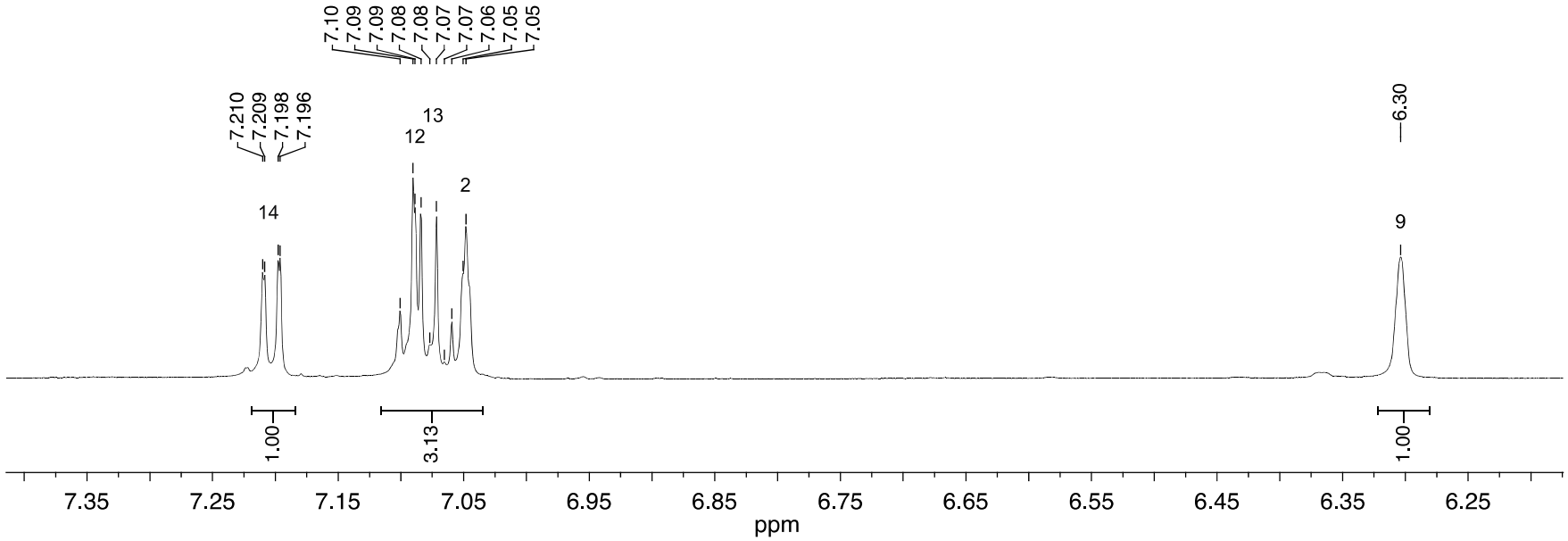




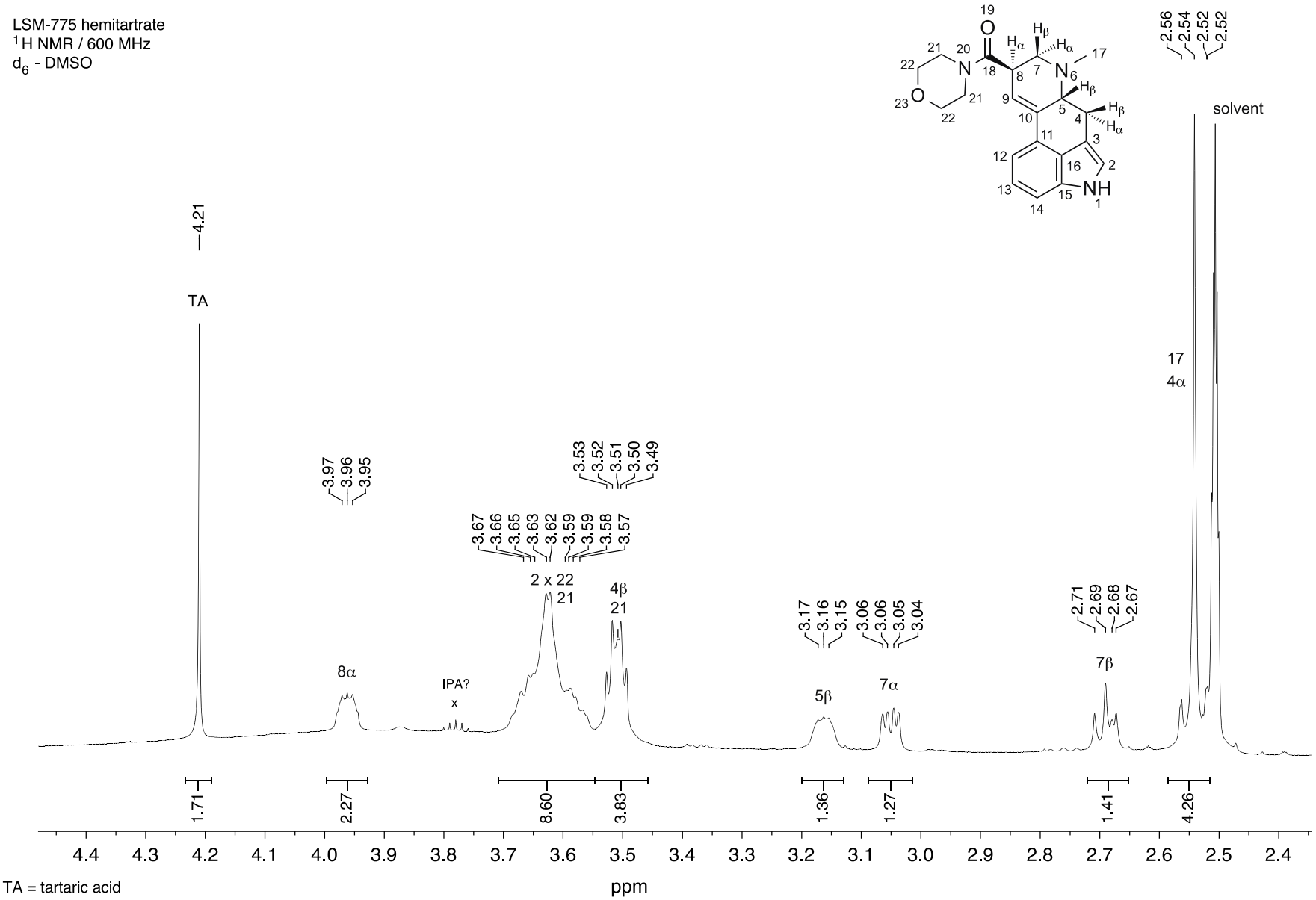
TA = tartaric acid

LSM-775 hemitartrate
^{1}H NMR / 600 MHz
d_6 - DMSO





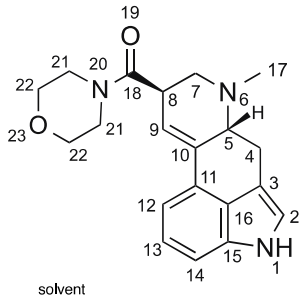
TA = tartaric acid

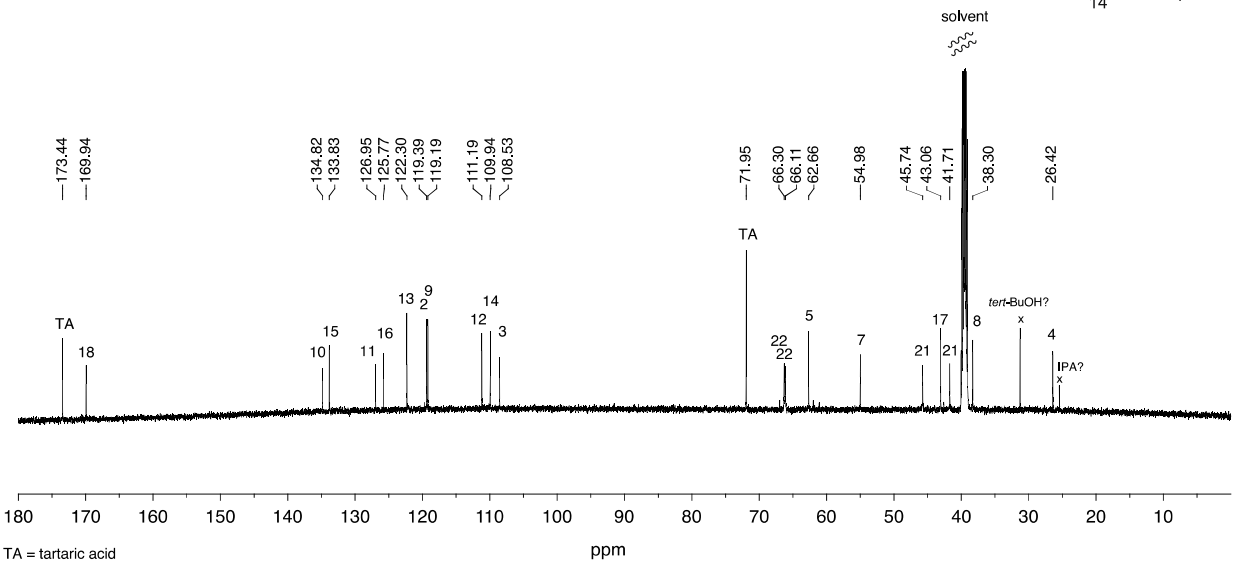


TA = tartaric acid

Increased baseline led to inaccurate integration values, particularly between 4.0 and 2.7 ppm

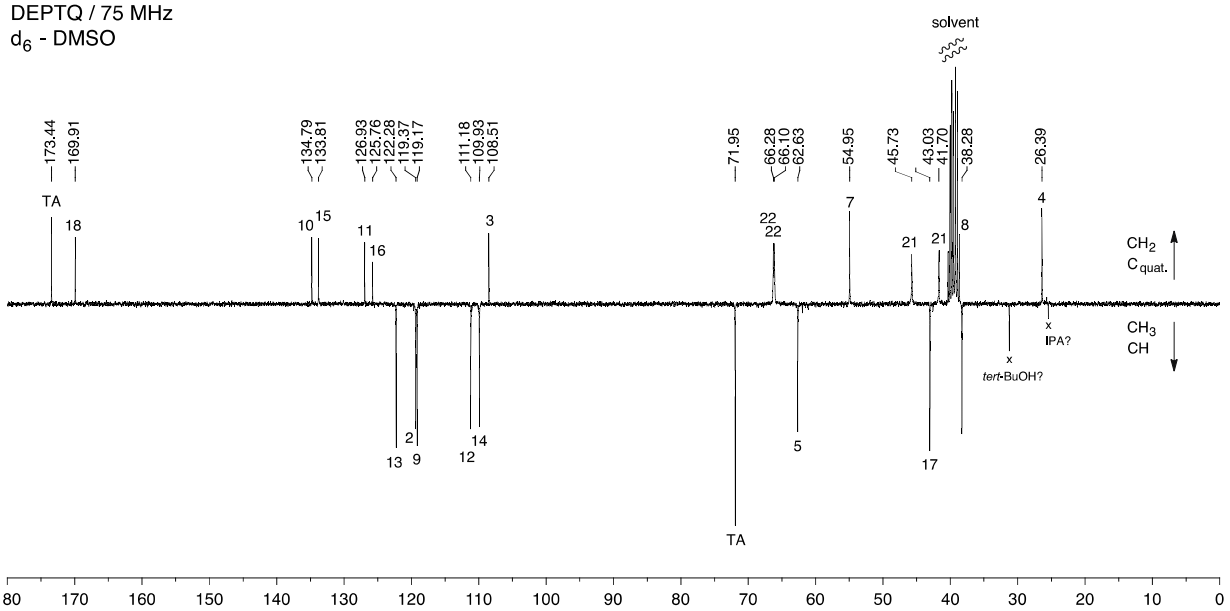
LSM-775 hemitartrate
^{13}C NMR / 150 MHz
d_6 - DMSO



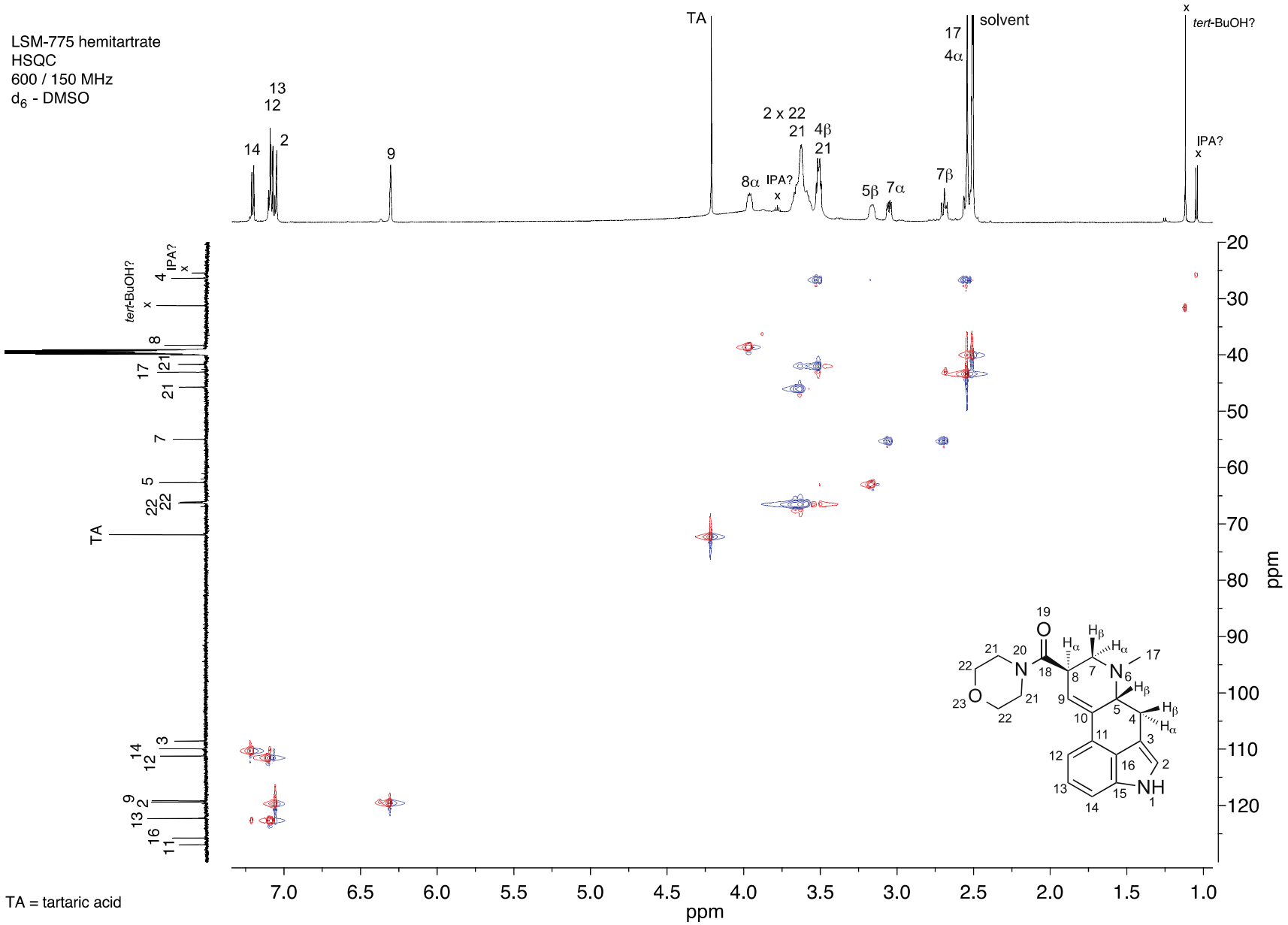


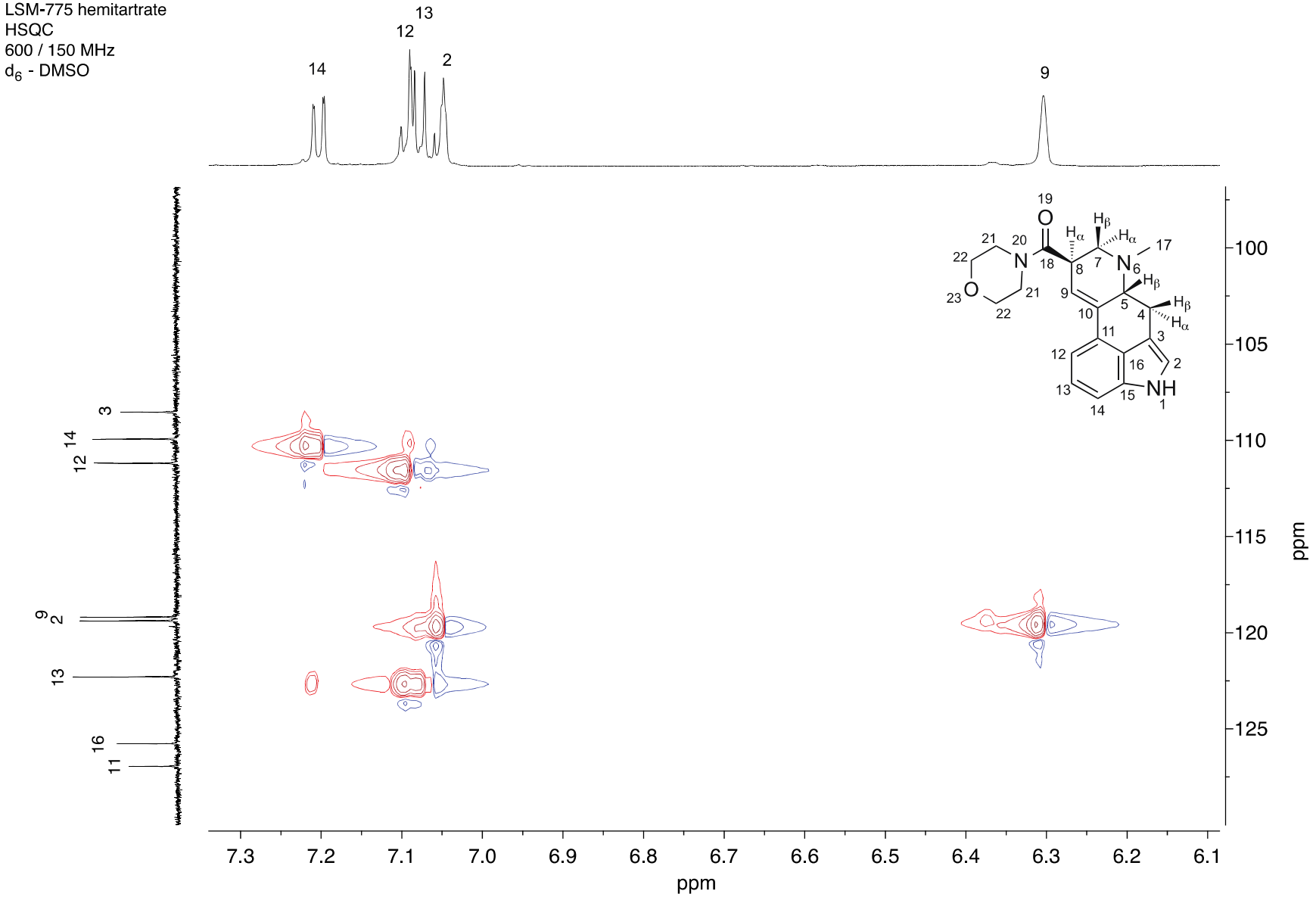
TA = tartaric acid

LSM-775 hemitartrate
DEPTQ / 75 MHz
d_6 - DMSO

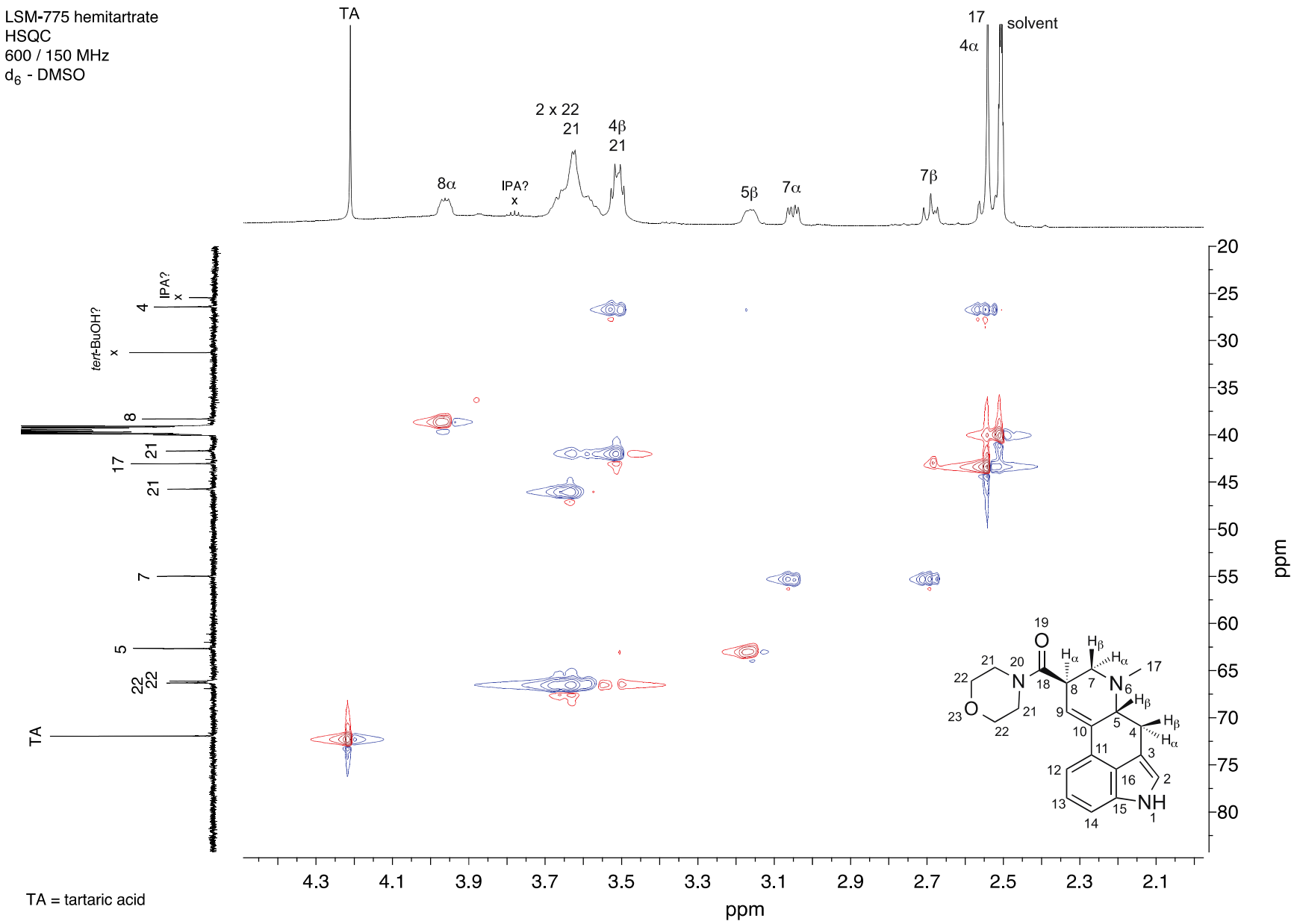


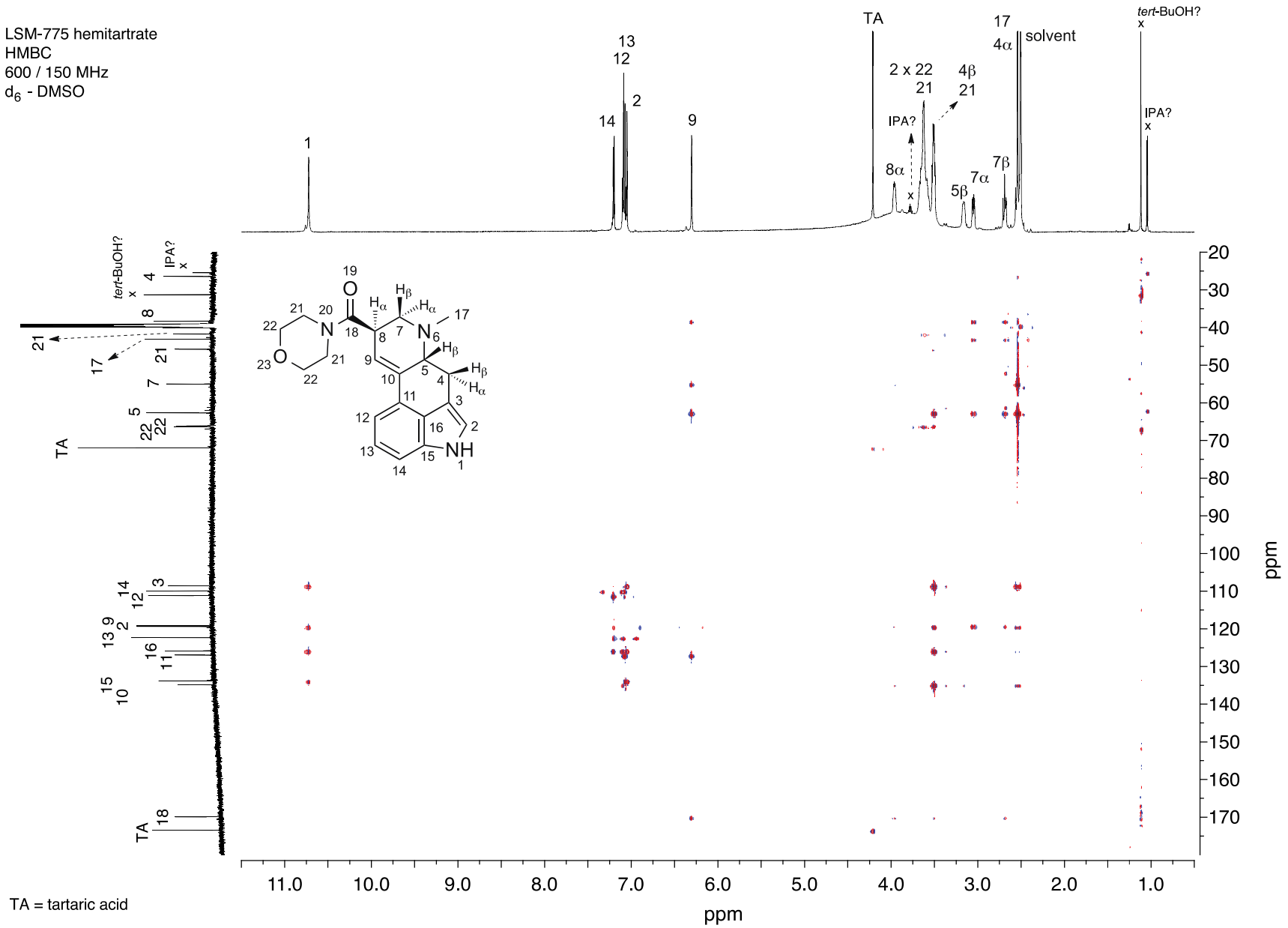
TA = tartaric acid


LSM-775 hemitartrate
HSQC
600 / 150 MHz
d6 - DMSO
TA
solvent
tert-BuOH?
IPA?
ppm
TA = tartaric acid

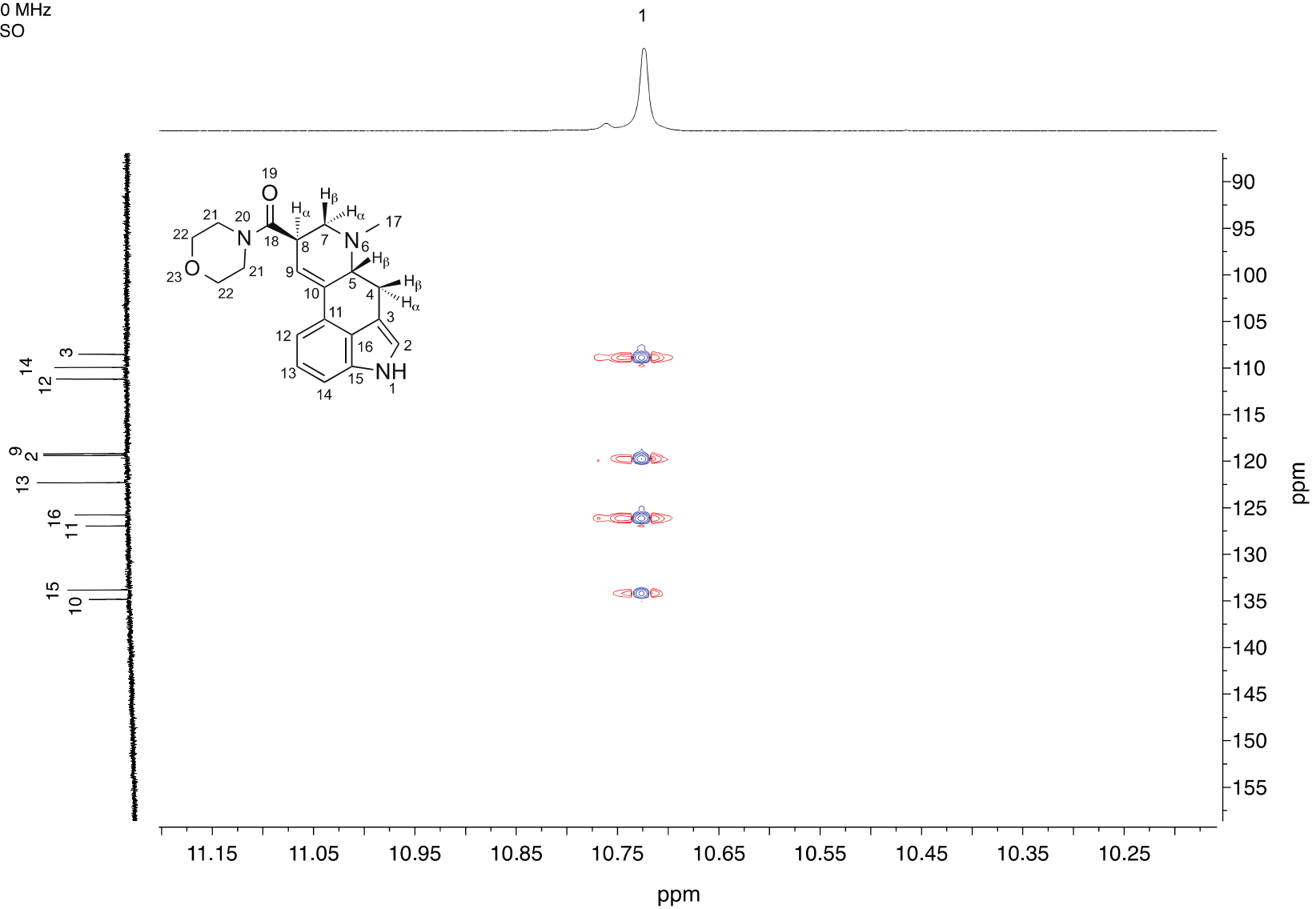


TA = tartaric acid

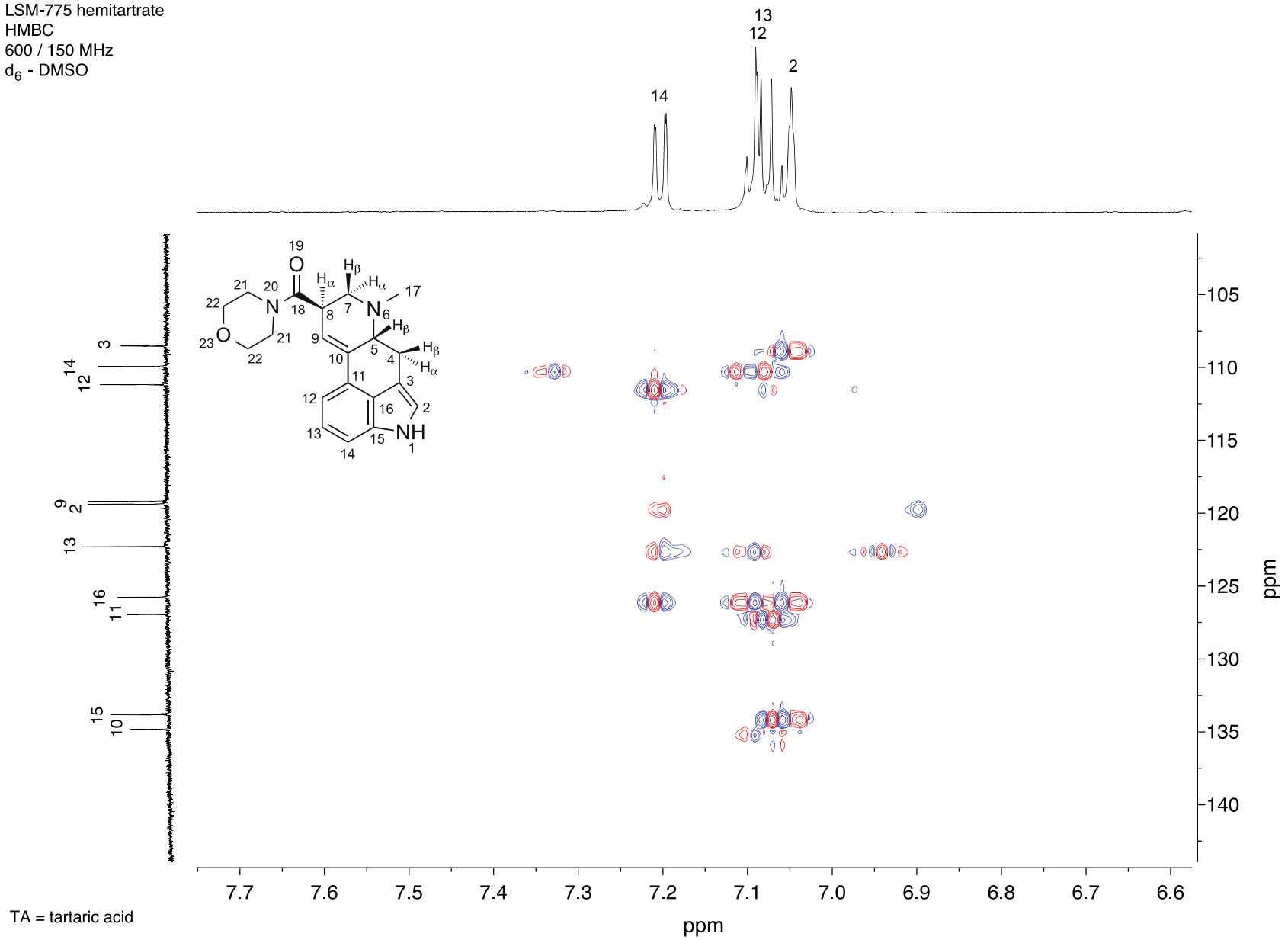

LSM-775 hemitartrate
HSQC
600 / 150 MHz
d6 - DMSO
TA
8α
IPA?
x
2 x 22
21
4β
21
5β
7α
7β
17
4α
solvent
tert-BuOH?
x
IPA?
x
4
8
17
21
21
7
5
22
22
TA
ppm
4.3
4.1
3.9
3.7
3.5
3.3
3.1
2.9
2.7
2.5
2.3
2.1
20
25
30
35
40
45
50
55
60
65
70
75
80
ppm
TA = tartaric acid


LSM-775 hemitartrate
HMBC
600 / 150 MHz
d_6 - DMSO
TA = tartaric acid
ppm
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
TA
solvent
IPA?
tert-BuOH?

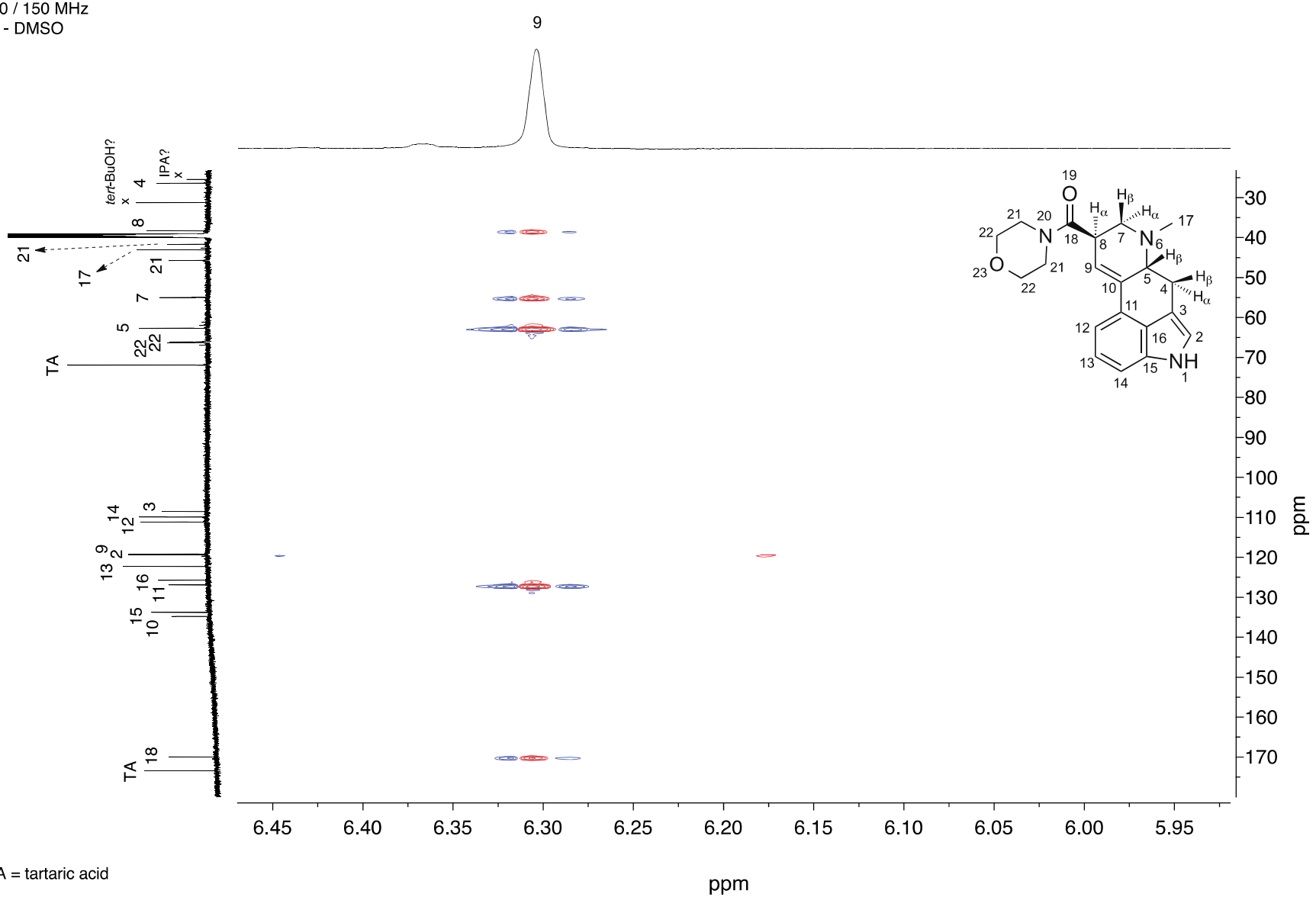
LSM-775 hemitartrate
HMBC
600 / 150 MHz
d_6 - DMSO



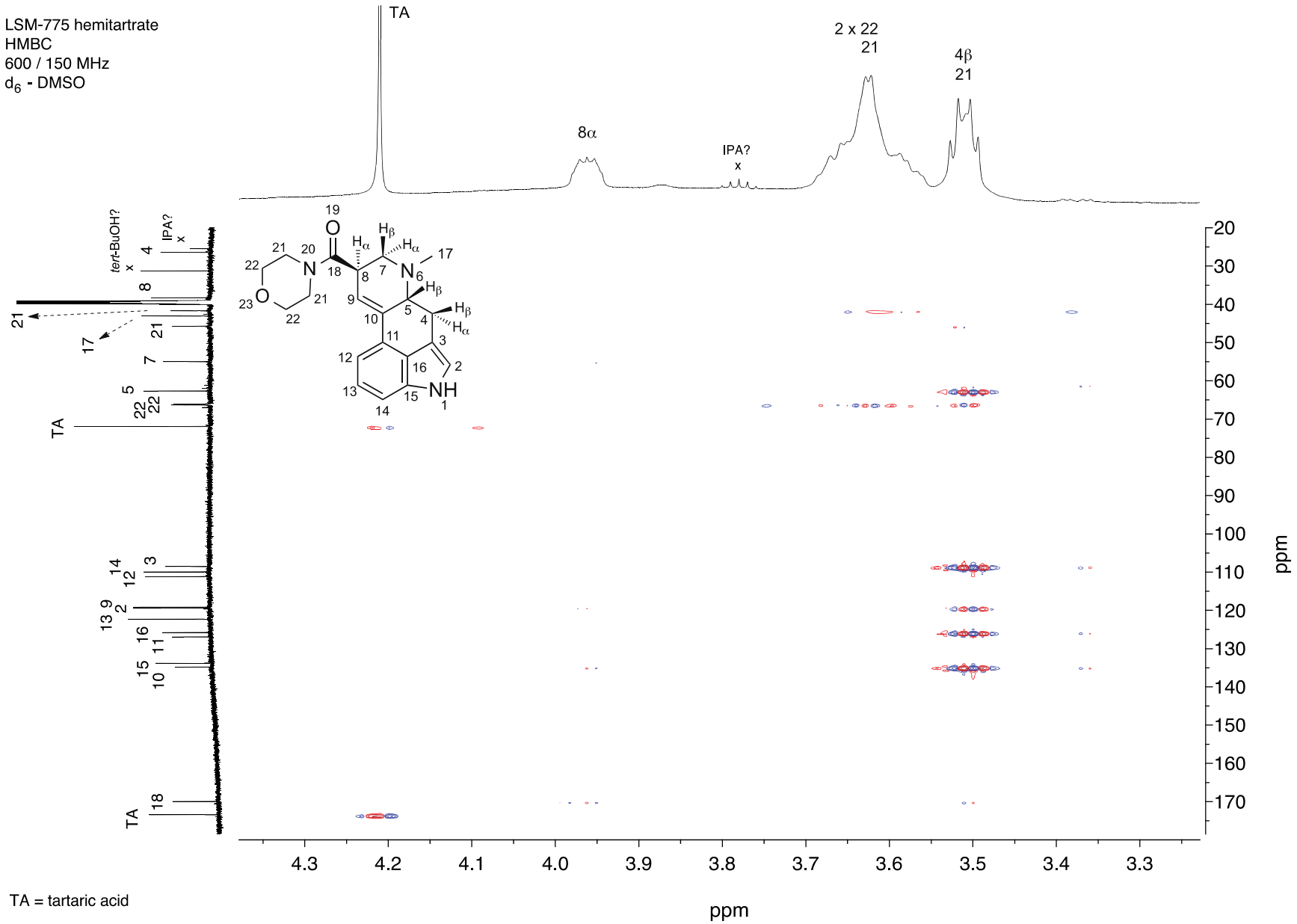
TA = tartaric acid


LSM-775 hemitartrate
HMBC
600 / 150 MHz
d_6 - DMSO
13
12
2
14
3
14
12
9
2
13
16
11
15
10
105
110
115
120
125
130
135
140
ppm
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
6.6
ppm
TA = tartaric acid

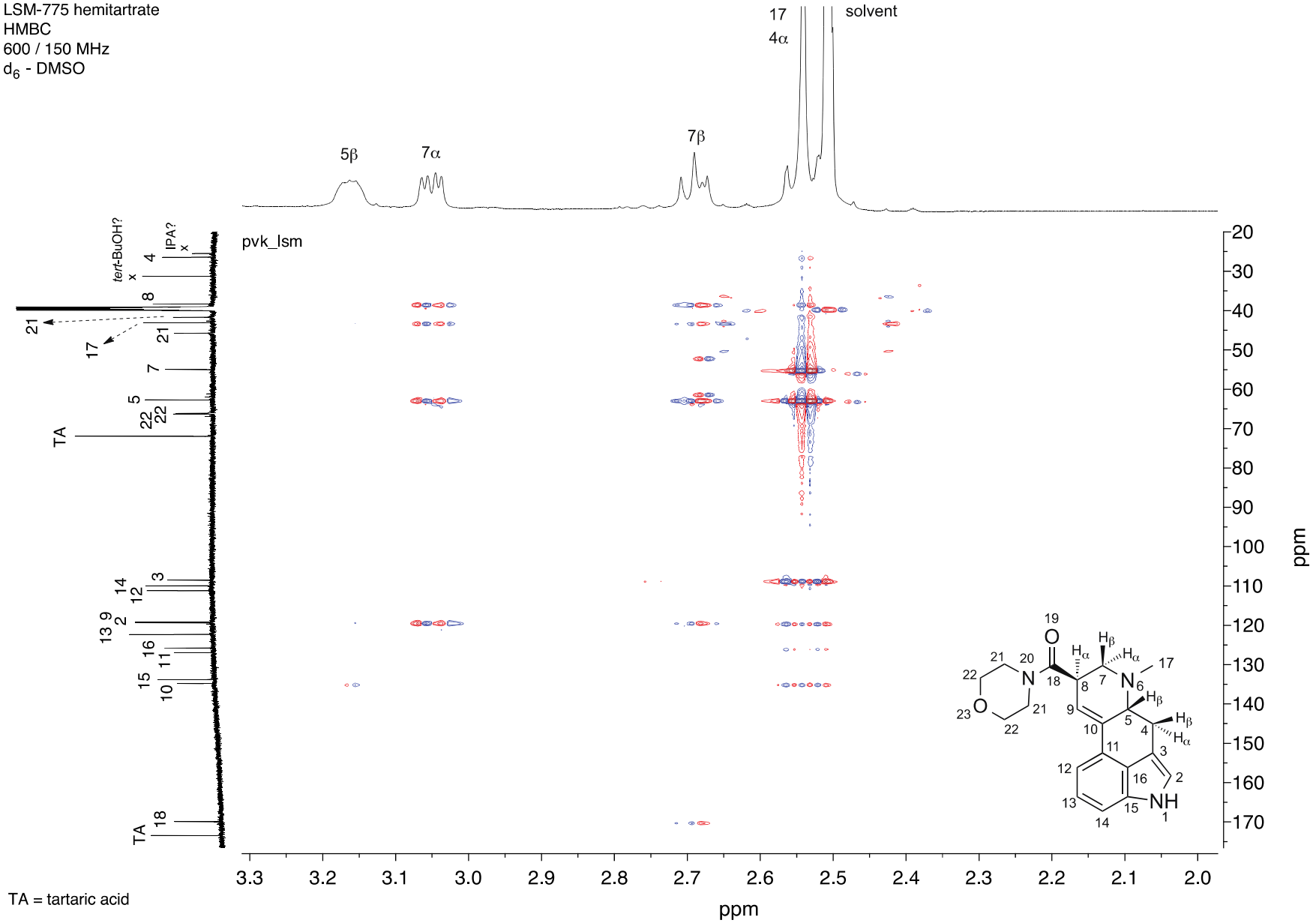
LSM-775 hemitartrate
HMBC
600 / 150 MHz
d_6 - DMSO

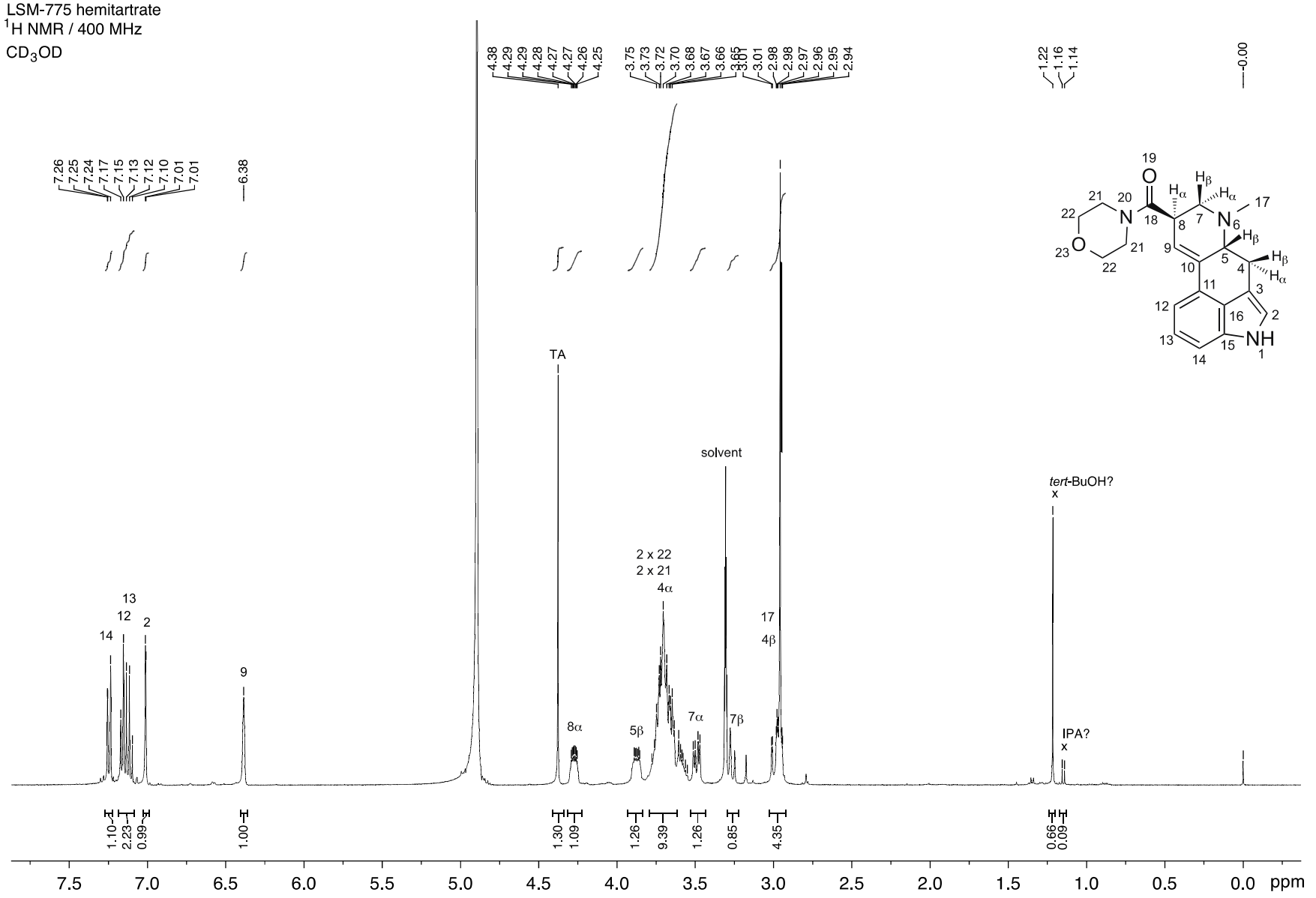


TA = tartaric acid

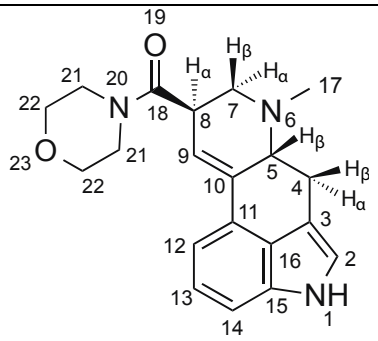


TA = tartaric acid


LSM-775 hemitartrate
HMBC
600 / 150 MHz
d6 - DMSO
pvk_lsm
5β
7α
7β
17
4α
solvent
tert-BuOH?
IPA?
TA
TA
ppm
ppm
TA = tartaric acid


LSM-775 hemitartrate
1H NMR / 400 MHz
CD3OD
TA
solvent
tert-BuOH?
x
IPA?
x
2 x 22
2 x 21
4α
17
4β
14
13
12
2
9
8α
5β
7α
7β
ppm
TA = tartaric acid

^{1}H and ^{13}C NMR data for LSM-775 hemitartrate in CD_3OD at 400 / 100 MHz (20 mg/mL)



No.	^{13}C [δ / ppm]	^{1}H [δ / ppm]
1	–	–
2	120.86	6.99 (d, *J* = 1.3 Hz, 1H)
3	108.53	–
4	26.32	3.80–3.60 (m, 4β-H, 1H) [a] 3.00–2.89 (m, 4α-H, 1H) [b]
5	64.09	3.90–3.80 (m, 5β-H, 1H)
6	–	–
7	55.18	3.47 (dd, *J* = 11.8, 5.1 Hz, 7α-H, 1H) 3.27–3.21 (m, 7β-H, 1H)
8	38.66	4.36–4.20 (m, 8α-H, 1H)
9	118.79	6.36 (s, 1H)
10	135.74	–
11	127.26	–
12	113.22	7.13 (dd, *J* = 7.4, 0.9 Hz, 1H)
13	124.01	7.09 (t, *J* = 7.5 Hz, 1H)
14	111.86	7.22 (dd, *J* = 7.8, 0.9 Hz, 1H)
15	135.60	–
16	127.15	–
17	42.82	2.94 (s, 3H)[c]
18	171.43	–
19	–	–
20	–	–
21	47.70	3.80–3.52 (m, 2H) [d, e]
21	43.82	3.80–3.52 (m, 2H) [d, e]
22	68.03	3.80–3.52 (m, 2H) [e, f]
22	67.82	3.80–3.52 (m, 2H) [e, f]
23	–	–
TA [g]	74.44	4.35 (s, 1H)
TA [g]	177.49	–

[a] Overlapping with 22-CH_2 and 21-CH_2 (4H)

[b] Overlapping with 17-CH_3 (3H)

[c] Overlapping with 4α-H (1H)

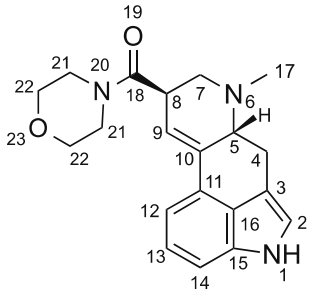
[d] Overlapping with 2 x 22-CH_2 (4H)

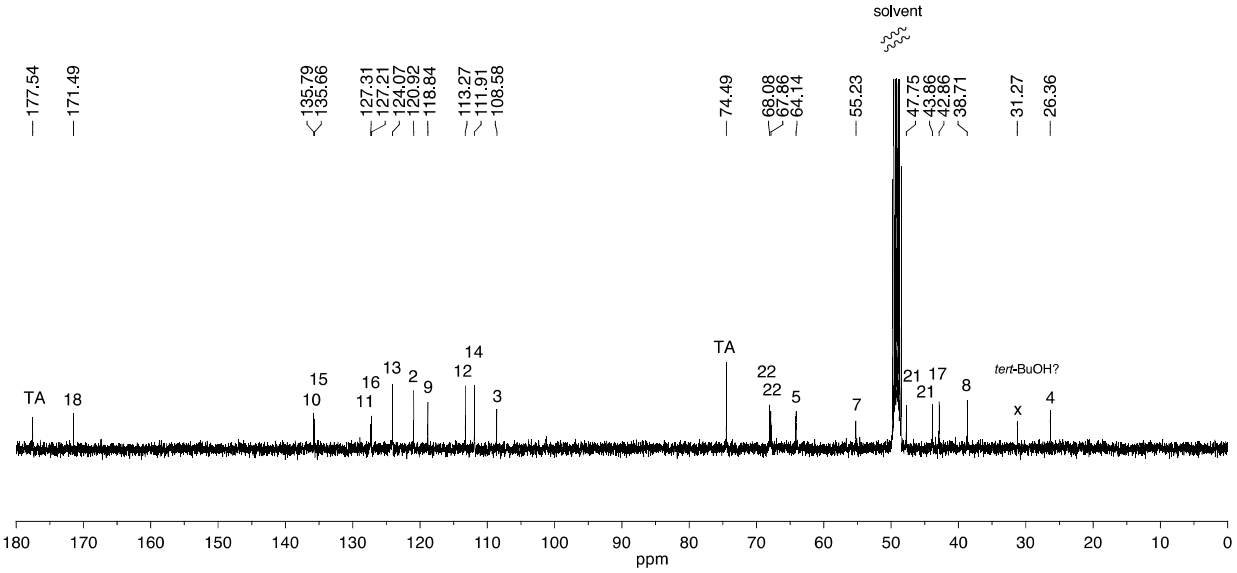
[e] Overlapping with 4β-H (1H)

[f] Overlapping with 1 x 21-CH_2 (2H)

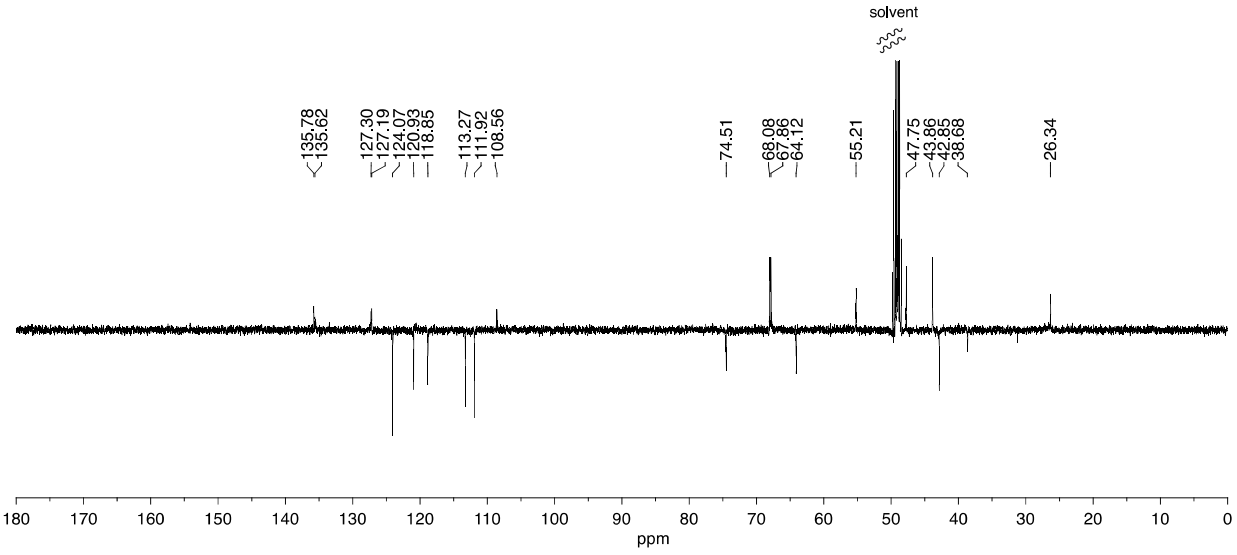
[g] TA: tartaric acid

LSM-775 hemitartrate
^{13}C NMR / 100 MHz
CD_3OD



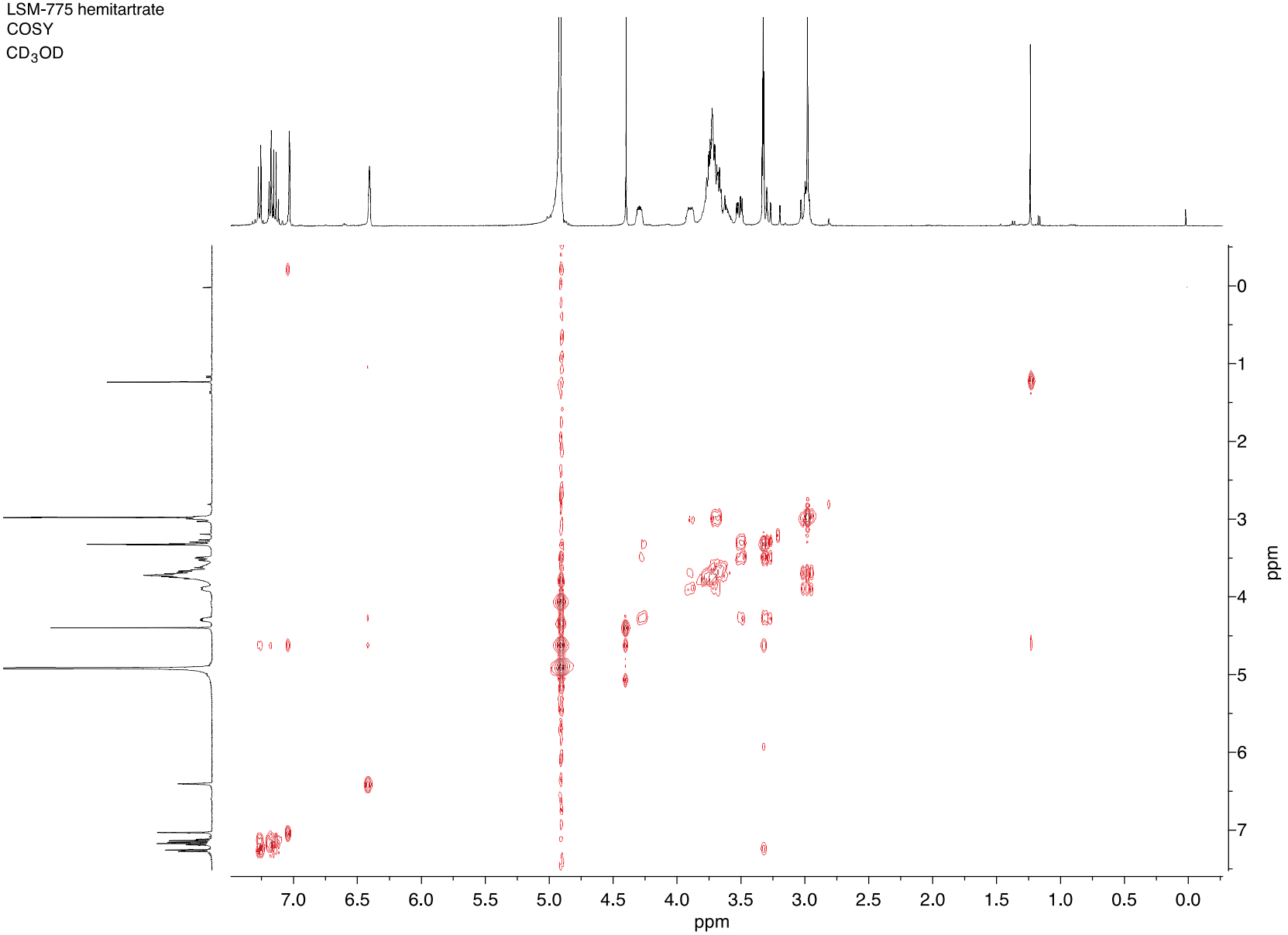


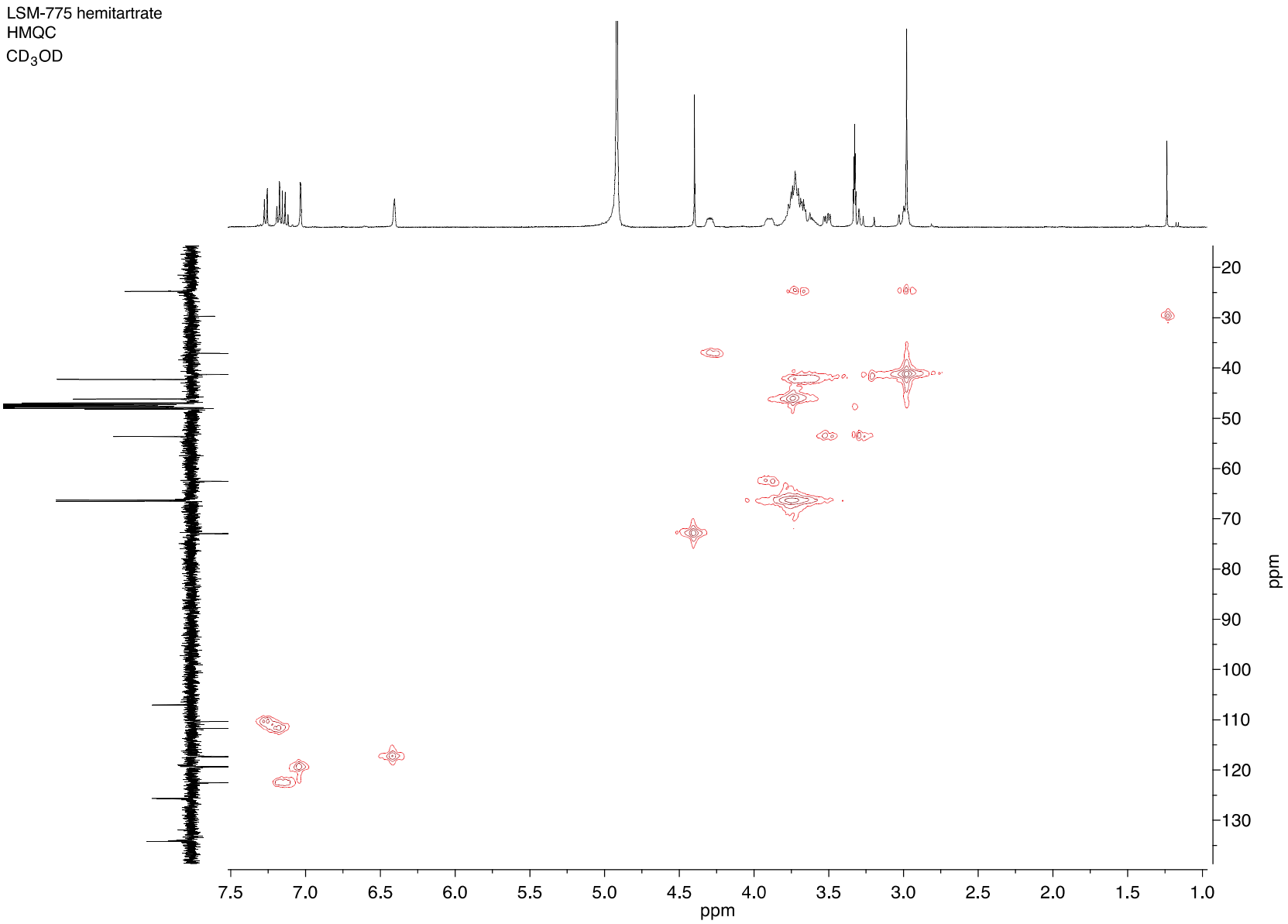
LSM-775 hemitartrate
PENDANT / 100 MHz
CD_3OD



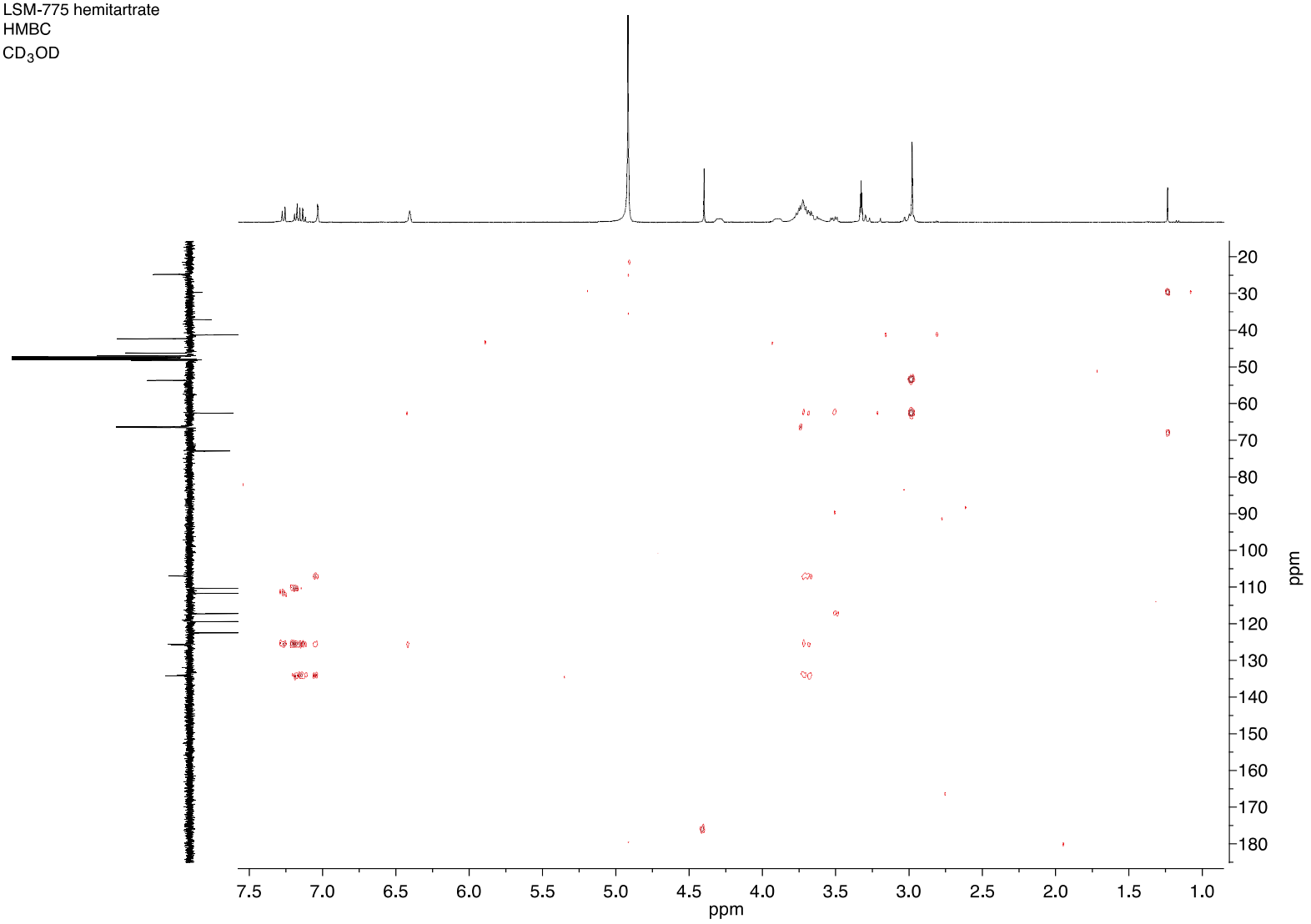
TA = tartaric acid

LSM-775 hemitartrate
COSY
CD_3OD




LSM-775 hemitartrate
HMQC
CD3OD
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
20
30
40
50
60
70
80
90
100
110
120
130
ppm

LSM-775 hemitartrate
HMBC
CD_3OD



Small Molecule X-ray Facility
School Of Chemistry

Structure Report

Filename: TCD173

Submitted by: Pierce Kavanagh

Reference: PKSBLSM

Group: Kavanagh

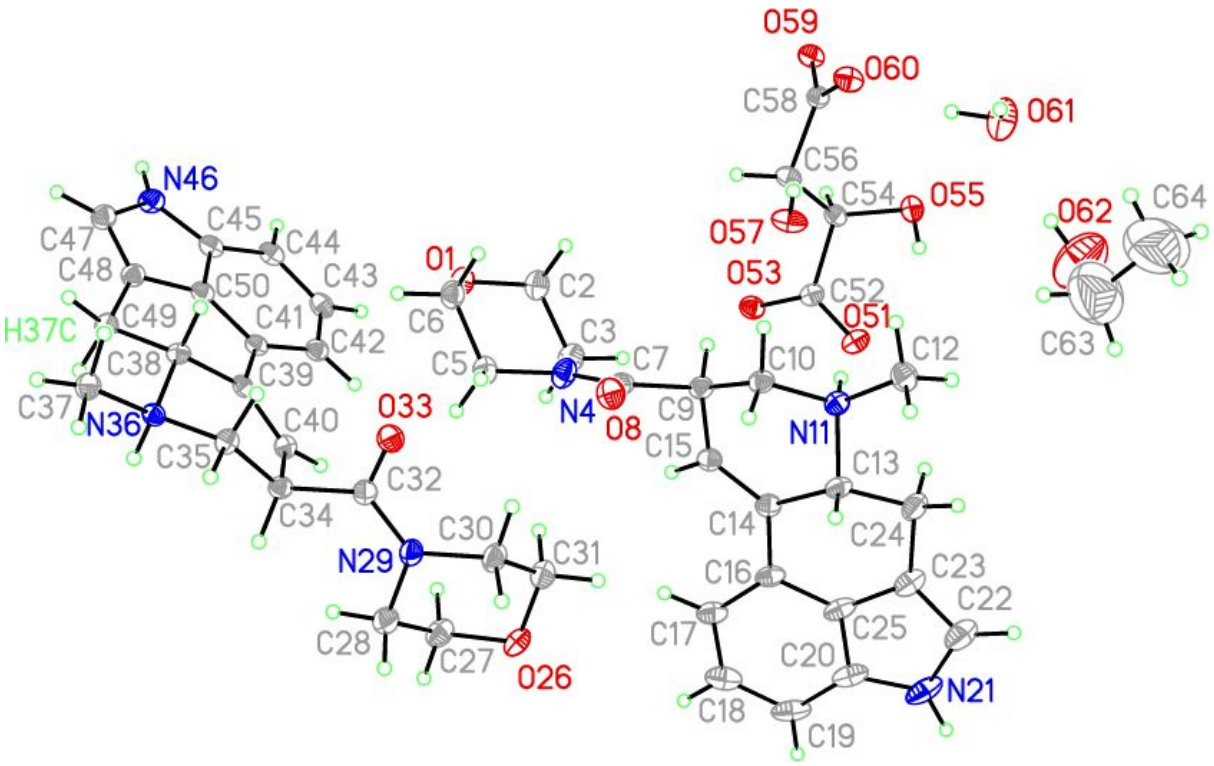


Fig. 1. Asymmetric unit of TCD173 showing both molecules with the tartrate anion as well as solvates H_2O and EtOH. Displacement ellipsoids shown at 50%.

11/12/14

Brendan Twamley

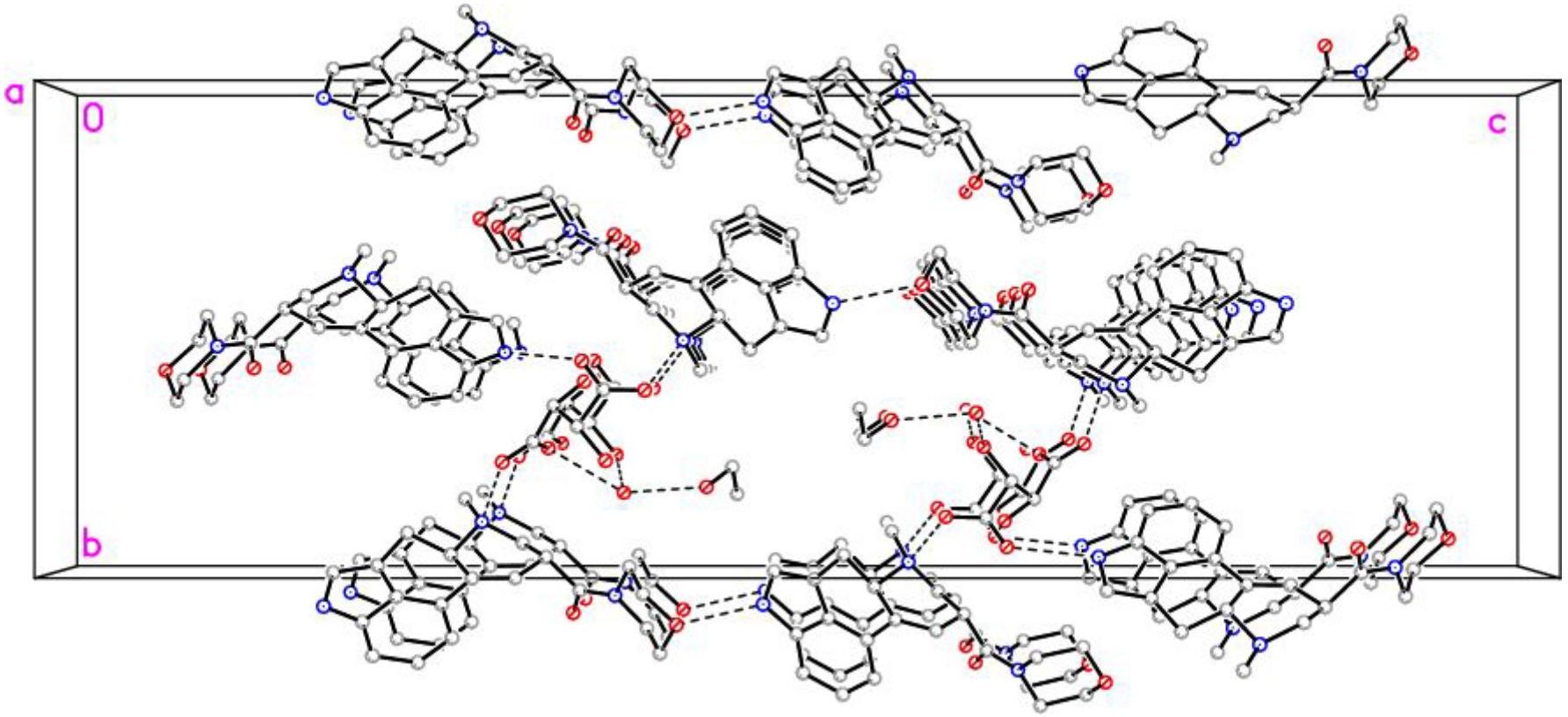


Figure 2. Packing diagram of TCD173 viewed down the a-axis. Hydrogen atoms omitted for clarity.

Crystal Structure Report for TCD173

A specimen of $C_{46}H_{60}N_6O_{12}$, approximate dimensions 0.060 mm x 0.060 mm x 0.210 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 100(2)K using an Oxford Cryosystems Cobra low temperature device using a MiTeGen micromount. See Table 1 for collection parameters and exposure time. Bruker APEX software was used to correct for Lorentz and polarization effects.

A total of 5863 frames were collected. The total exposure time was 29.31 hours. The integration of the data using an orthorhombic unit cell yielded a total of 57701 reflections to a maximum θ angle of 68.30° (0.83 Å resolution), of which 8027 were independent (average redundancy 7.188, completeness = 99.9%, R_{int} = 4.44%, R_{sig} = 2.32%) and 7723 (96.21%) were greater than $2\sigma(F^2)$. The final cell constants of a = 5.9621(2) Å, b = 15.5087(6) Å, c = 47.4811(19) Å, volume = 4390.3(3) $Å^3$, are based upon the refinement of the XYZ-centroids of reflections above 20 σ(I). Data were corrected for absorption effects using the numerical method (SADABS). The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.8490 and 0.9424.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group $P2_12_12_1$, with Z = 4 for the formula unit, $C_{46}H_{60}N_6O_{12}$. The final anisotropic full-matrix least-squares refinement on F^2 with 607 variables converged at R1 = 3.71%, for the observed data and wR2 = 9.92% for all data. The goodness-of-fit was 1.050. The largest peak in the final difference electron density synthesis was 0.557 $e^-/Å^3$ and the largest hole was -0.395 $e^-/Å^3$ with an RMS deviation of 0.049 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.345 g/cm^3 and F(000), 1896 e^-.
Refinement Note: All donor hydrogen atoms were located and refined with restraints (SADI). The solvent EtOH molecule C-C distance was also restrained (DFIX). Absolute configuration was established by anomalous-dispersion effects in diffraction measurements on the crystal.

References:
Bruker APEX v2012.12-0, Bruker AXS Inc., Madison, Wisconsin, USA.

SADABS (2014/3) Bruker AXS Inc., Madison, Wisconsin, USA; Sheldrick, G. M. University of Göttingen, Germany.

SHELXL-2014, (2014), Bruker AXS Inc., Madison, Wisconsin, USA; Sheldrick, G. M. University of Göttingen, Germany.

Acknowledgement:
Facility funded by PRTLI and ERDF.

Table 1: Data collection details for TCD173.

Axis	dx/mm	2θ/°	ω/°	φ/°	χ/°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Omega	45.000	-59.59	184.68	360.00	54.74	0.60	209	18.00	1.54184	45	0.6	100
Omega	45.000	104.59	343.28	270.00	64.87	0.60	215	18.00	1.54184	45	0.6	100
Phi	45.000	104.92	82.49	0.00	-23.00	0.60	600	18.00	1.54184	45	0.6	100
Omega	45.000	-74.59	169.68	270.00	54.74	0.60	209	18.00	1.54184	45	0.6	100
Phi	45.000	104.92	4.83	180.20	23.00	0.60	306	18.00	1.54184	45	0.6	100
Omega	45.000	-39.60	312.65	0.00	-64.88	0.60	165	18.00	1.54184	45	0.6	100
Omega	45.000	-39.60	312.65	270.00	-64.88	0.60	165	18.00	1.54184	45	0.6	100
Phi	45.000	89.92	80.25	360.00	-57.06	0.60	600	18.00	1.54184	45	0.6	100
Omega	45.000	104.59	95.82	189.00	-54.74	0.60	206	18.00	1.54184	45	0.6	100
Phi	45.000	-36.55	333.12	0.00	57.06	0.60	600	18.00	1.54184	45	0.6	100
Omega	45.000	104.59	95.82	270.00	-54.74	0.60	206	18.00	1.54184	45	0.6	100
Omega	45.000	104.59	343.28	27.00	64.88	0.60	215	18.00	1.54184	45	0.6	100
Phi	45.000	104.92	95.25	0.00	-57.06	0.60	600	18.00	1.54184	45	0.6	100
Omega	45.000	104.59	95.82	108.00	-54.74	0.60	206	18.00	1.54184	45	0.6	100
Phi	45.000	-74.92	42.49	360.00	-57.06	0.60	600	18.00	1.54184	45	0.6	100
Omega	45.000	104.59	95.82	216.00	-54.74	0.60	206	18.00	1.54184	45	0.6	100
Omega	45.000	104.59	95.82	81.00	-54.74	0.60	206	18.00	1.54184	45	0.6	100
Omega	45.000	-26.40	325.84	153.00	-64.88	0.60	143	18.00	1.54184	45	0.6	100
Omega	45.000	104.59	95.82	135.00	-54.74	0.60	206	18.00	1.54184	45	0.6	100

Table 2. Crystal data and structure refinement for TCD173.

Identification code	tcd173	
Empirical formula	$C_{46}H_{60}N_6O_{12}$	
Formula weight	889.00	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 5.9621(2) Å	α= 90°.
	b = 15.5087(6) Å	β= 90°.
	c = 47.4811(19) Å	γ = 90°.
Volume	4390.3(3) $Å^3$	
Z	4	
Density (calculated)	1.345 Mg/m^3	
Absorption coefficient	0.807 mm^{-1}	
F(000)	1896	
Crystal size	0.210 x 0.060 x 0.060 mm^3	
Theta range for data collection	3.404 to 68.296°.	
Index ranges	$-7\leq h\leq 7$, $-18\leq k\leq 17$, $-57\leq l\leq 56$	
Reflections collected	57701	
Independent reflections	8027 [R(int) = 0.0444]	
Completeness to theta = 67.679°	99.9 %	
Absorption correction	Numerical	
Max. and min. transmission	1.000 and 0.9424	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8027 / 10 / 607	
Goodness-of-fit on F^2	1.050	
Final R indices [$I>2\sigma(I)$]	R1 = 0.0371, wR2 = 0.0979	
R indices (all data)	R1 = 0.0387, wR2 = 0.0992	
Absolute structure parameter	0.08(4)	
Largest diff. peak and hole	0.557 and -0.395 $e.Å^{-3}$	

Table 3. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for TCD173. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(2)	1323(5)	6429(2)	8112(1)	23(1)
C(3)	1086(5)	6623(2)	8423(1)	24(1)
C(5)	3978(5)	7721(2)	8369(1)	25(1)
C(6)	4120(5)	7500(2)	8061(1)	22(1)
C(7)	4580(4)	6612(2)	8727(1)	19(1)
C(9)	3663(4)	5830(2)	8885(1)	18(1)
C(10)	5566(4)	5415(2)	9049(1)	18(1)
C(12)	6346(5)	4182(2)	9358(1)	25(1)
C(13)	3382(4)	5257(2)	9486(1)	19(1)
C(14)	1694(4)	5881(2)	9352(1)	18(1)
C(15)	1808(4)	6107(2)	9080(1)	18(1)
C(16)	-29(4)	6233(2)	9543(1)	20(1)
C(17)	-1369(5)	6954(2)	9501(1)	24(1)
C(18)	-2928(5)	7215(2)	9708(1)	29(1)
C(19)	-3193(5)	6778(2)	9963(1)	32(1)
C(20)	-1854(5)	6054(2)	10006(1)	28(1)
C(22)	-26(6)	4861(2)	10153(1)	34(1)
C(23)	772(5)	5037(2)	9889(1)	27(1)
C(24)	2382(5)	4591(2)	9693(1)	25(1)
C(25)	-331(5)	5788(2)	9797(1)	24(1)
C(27)	1209(5)	9790(2)	9010(1)	25(1)
C(28)	3271(5)	10270(2)	8909(1)	24(1)
C(30)	5484(5)	8983(2)	9010(1)	22(1)
C(31)	3341(5)	8553(2)	9107(1)	26(1)
C(32)	6146(4)	9726(2)	8563(1)	17(1)
C(34)	5447(4)	10415(2)	8347(1)	16(1)
C(35)	7495(4)	10653(2)	8173(1)	16(1)
C(37)	8721(4)	11531(2)	7774(1)	19(1)
C(38)	5612(4)	10463(2)	7720(1)	15(1)
C(39)	3693(4)	10032(1)	7882(1)	14(1)
C(40)	3621(4)	10043(2)	8164(1)	16(1)
C(41)	2025(4)	9565(2)	7707(1)	16(1)
C(42)	468(4)	8956(2)	7795(1)	17(1)

C(43)	-881(4)	8522(2)	7598(1)	20(1)
C(44)	-725(4)	8648(2)	7308(1)	19(1)
C(45)	857(4)	9255(2)	7217(1)	18(1)
C(47)	3170(5)	10164(2)	6990(1)	21(1)
C(48)	3557(4)	10296(2)	7271(1)	18(1)
C(49)	4902(5)	10914(2)	7444(1)	19(1)
C(50)	2141(4)	9713(2)	7415(1)	17(1)
C(52)	871(4)	3803(2)	8758(1)	16(1)
C(54)	1905(4)	3078(2)	8583(1)	16(1)
C(56)	3838(4)	3469(2)	8414(1)	17(1)
C(58)	5120(4)	2742(2)	8266(1)	16(1)
N(4)	3248(4)	6958(2)	8526(1)	24(1)
N(11)	4583(4)	4782(1)	9253(1)	18(1)
N(21)	-1628(5)	5475(2)	10223(1)	36(1)
N(29)	4943(4)	9659(1)	8804(1)	20(1)
N(36)	6767(3)	11102(1)	7910(1)	14(1)
N(46)	1536(4)	9539(1)	6957(1)	20(1)
O(1)	2049(3)	7167(1)	7957(1)	23(1)
O(8)	6414(3)	6921(1)	8789(1)	24(1)
O(26)	1789(3)	9168(1)	9221(1)	24(1)
O(33)	7685(3)	9226(1)	8507(1)	21(1)
O(51)	1287(3)	3790(1)	9021(1)	21(1)
O(53)	-234(3)	4364(1)	8630(1)	20(1)
O(55)	2678(3)	2390(1)	8759(1)	22(1)
O(57)	5193(3)	3952(1)	8601(1)	23(1)
O(59)	4066(3)	2344(1)	8076(1)	19(1)
O(60)	7074(3)	2589(1)	8347(1)	21(1)
O(61)	8262(4)	1668(2)	8840(1)	37(1)
O(62)	6805(7)	1807(3)	9398(1)	89(1)
C(63)	8661(13)	2189(5)	9564(2)	110(2)
C(64)	10528(16)	1603(6)	9591(2)	142(4)

Table 4. Bond lengths [Å] and angles [°] for TCD173.

C(2)-O(1)	1.429(3)	C(17)-C(18)	1.413(4)
C(2)-C(3)	1.512(4)	C(17)-H(17)	0.9500
C(2)-H(2A)	0.9900	C(18)-C(19)	1.395(5)
C(2)-H(2B)	0.9900	C(18)-H(18)	0.9500
C(3)-N(4)	1.475(4)	C(19)-C(20)	1.393(5)
C(3)-H(3A)	0.9900	C(19)-H(19)	0.9500
C(3)-H(3B)	0.9900	C(20)-N(21)	1.373(4)
C(5)-N(4)	1.466(3)	C(20)-C(25)	1.406(4)
C(5)-C(6)	1.506(4)	C(22)-C(23)	1.368(4)
C(5)-H(5A)	0.9900	C(22)-N(21)	1.389(4)
C(5)-H(5B)	0.9900	C(22)-H(22)	0.9500
C(6)-O(1)	1.426(3)	C(23)-C(25)	1.407(4)
C(6)-H(6A)	0.9900	C(23)-C(24)	1.505(4)
C(6)-H(6B)	0.9900	C(24)-H(24A)	0.9900
C(7)-O(8)	1.230(3)	C(24)-H(24B)	0.9900
C(7)-N(4)	1.351(4)	C(27)-O(26)	1.436(3)
C(7)-C(9)	1.529(3)	C(27)-C(28)	1.515(4)
C(9)-C(15)	1.505(3)	C(27)-H(27A)	0.9900
C(9)-C(10)	1.517(4)	C(27)-H(27B)	0.9900
C(9)-H(9)	1.0000	C(28)-N(29)	1.463(3)
C(10)-N(11)	1.498(3)	C(28)-H(28A)	0.9900
C(10)-H(10A)	0.9900	C(28)-H(28B)	0.9900
C(10)-H(10B)	0.9900	C(30)-N(29)	1.469(3)
C(12)-N(11)	1.490(3)	C(30)-C(31)	1.514(4)
C(12)-H(12A)	0.9800	C(30)-H(30A)	0.9900
C(12)-H(12B)	0.9800	C(30)-H(30B)	0.9900
C(12)-H(12C)	0.9800	C(31)-O(26)	1.434(4)
C(13)-N(11)	1.512(3)	C(31)-H(31A)	0.9900
C(13)-C(14)	1.535(4)	C(31)-H(31B)	0.9900
C(13)-C(24)	1.544(4)	C(32)-O(33)	1.231(3)
C(13)-H(13)	1.0000	C(32)-N(29)	1.354(3)
C(14)-C(15)	1.338(4)	C(32)-C(34)	1.538(3)
C(14)-C(16)	1.475(4)	C(34)-C(40)	1.508(3)
C(15)-H(15)	0.9500	C(34)-C(35)	1.521(3)
C(16)-C(17)	1.389(4)	C(34)-H(34)	1.0000
C(16)-C(25)	1.400(4)	C(35)-N(36)	1.494(3)

C(35)-H(35A)	0.9900	C(56)-H(56)	1.0000
C(35)-H(35B)	0.9900	C(58)-O(60)	1.250(3)
C(37)-N(36)	1.488(3)	C(58)-O(59)	1.262(3)
C(37)-H(37A)	0.9800	N(11)-H(11)	0.89(2)
C(37)-H(37B)	0.9800	N(21)-H(21)	0.90(2)
C(37)-H(37C)	0.9800	N(36)-H(36)	0.90(2)
C(38)-N(36)	1.505(3)	N(46)-H(46)	0.89(2)
C(38)-C(39)	1.531(3)	O(55)-H(55)	0.84(3)
C(38)-C(49)	1.544(3)	O(57)-H(57)	0.85(3)
C(38)-H(38)	1.0000	O(61)-H(61A)	0.94(3)
C(39)-C(40)	1.341(3)	O(61)-H(61B)	0.90(3)
C(39)-C(41)	1.485(3)	O(62)-C(63)	1.484(8)
C(40)-H(40)	0.9500	O(62)-H(62)	0.86(3)
C(41)-C(42)	1.389(4)	C(63)-C(64)	1.443(8)
C(41)-C(50)	1.405(3)	C(63)-H(63A)	0.9900
C(42)-C(43)	1.405(4)	C(63)-H(63B)	0.9900
C(42)-H(42)	0.9500	C(64)-H(64A)	0.9800
C(43)-C(44)	1.394(4)	C(64)-H(64B)	0.9800
C(43)-H(43)	0.9500	C(64)-H(64C)	0.9800
C(44)-C(45)	1.401(4)		
C(44)-H(44)	0.9500	O(1)-C(2)-C(3)	111.9(2)
C(45)-N(46)	1.373(3)	O(1)-C(2)-H(2A)	109.2
C(45)-C(50)	1.404(4)	C(3)-C(2)-H(2A)	109.2
C(47)-C(48)	1.368(4)	O(1)-C(2)-H(2B)	109.2
C(47)-N(46)	1.383(4)	C(3)-C(2)-H(2B)	109.2
C(47)-H(47)	0.9500	H(2A)-C(2)-H(2B)	107.9
C(48)-C(50)	1.413(4)	N(4)-C(3)-C(2)	108.2(2)
C(48)-C(49)	1.497(3)	N(4)-C(3)-H(3A)	110.0
C(49)-H(49A)	0.9900	C(2)-C(3)-H(3A)	110.0
C(49)-H(49B)	0.9900	N(4)-C(3)-H(3B)	110.0
C(52)-O(53)	1.249(3)	C(2)-C(3)-H(3B)	110.0
C(52)-O(51)	1.270(3)	H(3A)-C(3)-H(3B)	108.4
C(52)-C(54)	1.531(3)	N(4)-C(5)-C(6)	109.2(2)
C(54)-O(55)	1.432(3)	N(4)-C(5)-H(5A)	109.8
C(54)-C(56)	1.529(3)	C(6)-C(5)-H(5A)	109.8
C(54)-H(54)	1.0000	N(4)-C(5)-H(5B)	109.8
C(56)-O(57)	1.416(3)	C(6)-C(5)-H(5B)	109.8
C(56)-C(58)	1.531(3)	H(5A)-C(5)-H(5B)	108.3

O(1)-C(6)-C(5)	111.7(2)	C(9)-C(15)-H(15)	118.1
O(1)-C(6)-H(6A)	109.3	C(17)-C(16)-C(25)	116.5(2)
C(5)-C(6)-H(6A)	109.3	C(17)-C(16)-C(14)	127.6(2)
O(1)-C(6)-H(6B)	109.3	C(25)-C(16)-C(14)	115.9(2)
C(5)-C(6)-H(6B)	109.3	C(16)-C(17)-C(18)	120.6(3)
H(6A)-C(6)-H(6B)	107.9	C(16)-C(17)-H(17)	119.7
O(8)-C(7)-N(4)	122.4(2)	C(18)-C(17)-H(17)	119.7
O(8)-C(7)-C(9)	120.6(2)	C(19)-C(18)-C(17)	122.7(3)
N(4)-C(7)-C(9)	116.9(2)	C(19)-C(18)-H(18)	118.7
C(15)-C(9)-C(10)	110.9(2)	C(17)-C(18)-H(18)	118.7
C(15)-C(9)-C(7)	109.8(2)	C(20)-C(19)-C(18)	116.9(3)
C(10)-C(9)-C(7)	108.7(2)	C(20)-C(19)-H(19)	121.6
C(15)-C(9)-H(9)	109.1	C(18)-C(19)-H(19)	121.6
C(10)-C(9)-H(9)	109.1	N(21)-C(20)-C(19)	133.8(3)
C(7)-C(9)-H(9)	109.1	N(21)-C(20)-C(25)	105.9(3)
N(11)-C(10)-C(9)	108.4(2)	C(19)-C(20)-C(25)	120.3(3)
N(11)-C(10)-H(10A)	110.0	C(23)-C(22)-N(21)	108.7(3)
C(9)-C(10)-H(10A)	110.0	C(23)-C(22)-H(22)	125.7
N(11)-C(10)-H(10B)	110.0	N(21)-C(22)-H(22)	125.7
C(9)-C(10)-H(10B)	110.0	C(22)-C(23)-C(25)	106.7(3)
H(10A)-C(10)-H(10B)	108.4	C(22)-C(23)-C(24)	134.1(3)
N(11)-C(12)-H(12A)	109.5	C(25)-C(23)-C(24)	119.1(2)
N(11)-C(12)-H(12B)	109.5	C(23)-C(24)-C(13)	109.4(2)
H(12A)-C(12)-H(12B)	109.5	C(23)-C(24)-H(24A)	109.8
N(11)-C(12)-H(12C)	109.5	C(13)-C(24)-H(24A)	109.8
H(12A)-C(12)-H(12C)	109.5	C(23)-C(24)-H(24B)	109.8
H(12B)-C(12)-H(12C)	109.5	C(13)-C(24)-H(24B)	109.8
N(11)-C(13)-C(14)	108.25(19)	H(24A)-C(24)-H(24B)	108.2
N(11)-C(13)-C(24)	108.9(2)	C(16)-C(25)-C(20)	123.1(3)
C(14)-C(13)-C(24)	115.6(2)	C(16)-C(25)-C(23)	128.0(3)
N(11)-C(13)-H(13)	107.9	C(20)-C(25)-C(23)	108.9(3)
C(14)-C(13)-H(13)	107.9	O(26)-C(27)-C(28)	110.9(2)
C(24)-C(13)-H(13)	107.9	O(26)-C(27)-H(27A)	109.5
C(15)-C(14)-C(16)	122.1(2)	C(28)-C(27)-H(27A)	109.5
C(15)-C(14)-C(13)	122.1(2)	O(26)-C(27)-H(27B)	109.5
C(16)-C(14)-C(13)	115.8(2)	C(28)-C(27)-H(27B)	109.5
C(14)-C(15)-C(9)	123.7(2)	H(27A)-C(27)-H(27B)	108.1
C(14)-C(15)-H(15)	118.1	N(29)-C(28)-C(27)	110.0(2)

N(29)-C(28)-H(28A)	109.7	N(36)-C(38)-C(39)	109.23(19)
C(27)-C(28)-H(28A)	109.7	N(36)-C(38)-C(49)	109.59(19)
N(29)-C(28)-H(28B)	109.7	C(39)-C(38)-C(49)	114.7(2)
C(27)-C(28)-H(28B)	109.7	N(36)-C(38)-H(38)	107.7
H(28A)-C(28)-H(28B)	108.2	C(39)-C(38)-H(38)	107.7
N(29)-C(30)-C(31)	109.4(2)	C(49)-C(38)-H(38)	107.7
N(29)-C(30)-H(30A)	109.8	C(40)-C(39)-C(41)	123.0(2)
C(31)-C(30)-H(30A)	109.8	C(40)-C(39)-C(38)	121.3(2)
N(29)-C(30)-H(30B)	109.8	C(41)-C(39)-C(38)	115.7(2)
C(31)-C(30)-H(30B)	109.8	C(39)-C(40)-C(34)	124.0(2)
H(30A)-C(30)-H(30B)	108.2	C(39)-C(40)-H(40)	118.0
O(26)-C(31)-C(30)	111.6(2)	C(34)-C(40)-H(40)	118.0
O(26)-C(31)-H(31A)	109.3	C(42)-C(41)-C(50)	116.2(2)
C(30)-C(31)-H(31A)	109.3	C(42)-C(41)-C(39)	127.6(2)
O(26)-C(31)-H(31B)	109.3	C(50)-C(41)-C(39)	116.0(2)
C(30)-C(31)-H(31B)	109.3	C(41)-C(42)-C(43)	120.5(2)
H(31A)-C(31)-H(31B)	108.0	C(41)-C(42)-H(42)	119.8
O(33)-C(32)-N(29)	122.0(2)	C(43)-C(42)-H(42)	119.8
O(33)-C(32)-C(34)	119.6(2)	C(44)-C(43)-C(42)	123.6(2)
N(29)-C(32)-C(34)	118.2(2)	C(44)-C(43)-H(43)	118.2
C(40)-C(34)-C(35)	111.0(2)	C(42)-C(43)-H(43)	118.2
C(40)-C(34)-C(32)	108.35(19)	C(43)-C(44)-C(45)	116.2(2)
C(35)-C(34)-C(32)	108.3(2)	C(43)-C(44)-H(44)	121.9
C(40)-C(34)-H(34)	109.7	C(45)-C(44)-H(44)	121.9
C(35)-C(34)-H(34)	109.7	N(46)-C(45)-C(44)	133.6(2)
C(32)-C(34)-H(34)	109.7	N(46)-C(45)-C(50)	106.2(2)
N(36)-C(35)-C(34)	109.6(2)	C(44)-C(45)-C(50)	120.2(2)
N(36)-C(35)-H(35A)	109.8	C(48)-C(47)-N(46)	109.5(2)
C(34)-C(35)-H(35A)	109.8	C(48)-C(47)-H(47)	125.3
N(36)-C(35)-H(35B)	109.8	N(46)-C(47)-H(47)	125.3
C(34)-C(35)-H(35B)	109.8	C(47)-C(48)-C(50)	106.0(2)
H(35A)-C(35)-H(35B)	108.2	C(47)-C(48)-C(49)	136.2(2)
N(36)-C(37)-H(37A)	109.5	C(50)-C(48)-C(49)	117.6(2)
N(36)-C(37)-H(37B)	109.5	C(48)-C(49)-C(38)	108.9(2)
H(37A)-C(37)-H(37B)	109.5	C(48)-C(49)-H(49A)	109.9
N(36)-C(37)-H(37C)	109.5	C(38)-C(49)-H(49A)	109.9
H(37A)-C(37)-H(37C)	109.5	C(48)-C(49)-H(49B)	109.9
H(37B)-C(37)-H(37C)	109.5	C(38)-C(49)-H(49B)	109.9

H(49A)-C(49)-H(49B)	108.3	C(20)-N(21)-H(21)	126(3)
C(45)-C(50)-C(41)	123.3(2)	C(22)-N(21)-H(21)	124(3)
C(45)-C(50)-C(48)	109.0(2)	C(32)-N(29)-C(28)	126.7(2)
C(41)-C(50)-C(48)	127.7(2)	C(32)-N(29)-C(30)	120.0(2)
O(53)-C(52)-O(51)	126.3(2)	C(28)-N(29)-C(30)	112.7(2)
O(53)-C(52)-C(54)	117.3(2)	C(37)-N(36)-C(35)	110.07(19)
O(51)-C(52)-C(54)	116.4(2)	C(37)-N(36)-C(38)	113.16(18)
O(55)-C(54)-C(56)	111.1(2)	C(35)-N(36)-C(38)	109.05(18)
O(55)-C(54)-C(52)	110.90(19)	C(37)-N(36)-H(36)	106(3)
C(56)-C(54)-C(52)	107.3(2)	C(35)-N(36)-H(36)	106(3)
O(55)-C(54)-H(54)	109.2	C(38)-N(36)-H(36)	112(3)
C(56)-C(54)-H(54)	109.2	C(45)-N(46)-C(47)	109.3(2)
C(52)-C(54)-H(54)	109.2	C(45)-N(46)-H(46)	127(3)
O(57)-C(56)-C(54)	108.1(2)	C(47)-N(46)-H(46)	124(3)
O(57)-C(56)-C(58)	113.1(2)	C(6)-O(1)-C(2)	111.96(19)
C(54)-C(56)-C(58)	108.9(2)	C(31)-O(26)-C(27)	109.79(19)
O(57)-C(56)-H(56)	108.9	C(54)-O(55)-H(55)	101(2)
C(54)-C(56)-H(56)	108.9	C(56)-O(57)-H(57)	108(2)
C(58)-C(56)-H(56)	108.9	H(61A)-O(61)-H(61B)	106.7
O(60)-C(58)-O(59)	126.3(2)	C(63)-O(62)-H(62)	110.9
O(60)-C(58)-C(56)	117.7(2)	C(64)-C(63)-O(62)	111.8(6)
O(59)-C(58)-C(56)	116.1(2)	C(64)-C(63)-H(63A)	109.3
C(7)-N(4)-C(5)	120.4(2)	O(62)-C(63)-H(63A)	109.3
C(7)-N(4)-C(3)	127.5(2)	C(64)-C(63)-H(63B)	109.3
C(5)-N(4)-C(3)	112.0(2)	O(62)-C(63)-H(63B)	109.3
C(12)-N(11)-C(10)	110.5(2)	H(63A)-C(63)-H(63B)	107.9
C(12)-N(11)-C(13)	113.09(19)	C(63)-C(64)-H(64A)	109.5
C(10)-N(11)-C(13)	109.92(19)	C(63)-C(64)-H(64B)	109.5
C(12)-N(11)-H(11)	106(2)	H(64A)-C(64)-H(64B)	109.5
C(10)-N(11)-H(11)	108(2)	C(63)-C(64)-H(64C)	109.5
C(13)-N(11)-H(11)	109(2)	H(64A)-C(64)-H(64C)	109.5
C(20)-N(21)-C(22)	109.7(2)	H(64B)-C(64)-H(64C)	109.5

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for TCD173. 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}
C(2)	21(1)	24(1)	25(1)	4(1)	-3(1)	-3(1)
C(3)	21(1)	28(1)	24(1)	3(1)	-2(1)	-4(1)
C(5)	30(2)	20(1)	24(1)	3(1)	1(1)	-2(1)
C(6)	25(1)	20(1)	22(1)	3(1)	0(1)	-3(1)
C(7)	21(1)	18(1)	18(1)	-2(1)	3(1)	-1(1)
C(9)	19(1)	18(1)	16(1)	-2(1)	0(1)	-2(1)
C(10)	17(1)	19(1)	17(1)	-2(1)	3(1)	-3(1)
C(12)	23(1)	25(1)	26(1)	2(1)	-1(1)	6(1)
C(13)	19(1)	25(1)	14(1)	-4(1)	-1(1)	2(1)
C(14)	18(1)	20(1)	18(1)	-3(1)	-2(1)	-2(1)
C(15)	17(1)	18(1)	18(1)	-2(1)	0(1)	-1(1)
C(16)	17(1)	27(1)	17(1)	-7(1)	-1(1)	-2(1)
C(17)	21(1)	30(1)	21(1)	-8(1)	-2(1)	1(1)
C(18)	20(1)	35(2)	33(2)	-15(1)	-2(1)	3(1)
C(19)	22(1)	47(2)	26(1)	-18(1)	4(1)	0(1)
C(20)	23(1)	42(2)	19(1)	-11(1)	4(1)	-5(1)
C(22)	34(2)	49(2)	21(1)	3(1)	4(1)	2(2)
C(23)	24(1)	40(2)	17(1)	-1(1)	1(1)	-1(1)
C(24)	26(1)	32(1)	18(1)	5(1)	3(1)	4(1)
C(25)	20(1)	36(2)	15(1)	-6(1)	0(1)	-3(1)
C(27)	24(1)	32(1)	19(1)	4(1)	-1(1)	3(1)
C(28)	27(2)	24(1)	22(1)	2(1)	4(1)	4(1)
C(30)	25(1)	25(1)	17(1)	5(1)	-2(1)	2(1)
C(31)	34(2)	24(1)	20(1)	4(1)	-1(1)	-1(1)
C(32)	18(1)	17(1)	15(1)	-1(1)	-5(1)	-3(1)
C(34)	16(1)	17(1)	15(1)	-1(1)	-1(1)	0(1)
C(35)	15(1)	18(1)	15(1)	1(1)	-2(1)	0(1)
C(37)	18(1)	19(1)	21(1)	0(1)	2(1)	-3(1)
C(38)	15(1)	16(1)	15(1)	-3(1)	0(1)	0(1)
C(39)	13(1)	11(1)	18(1)	1(1)	-2(1)	2(1)
C(40)	15(1)	16(1)	17(1)	1(1)	-1(1)	0(1)
C(41)	16(1)	14(1)	17(1)	-2(1)	-2(1)	2(1)
C(42)	17(1)	17(1)	18(1)	0(1)	-1(1)	2(1)

C(43)	16(1)	16(1)	27(1)	-1(1)	-2(1)	1(1)
C(44)	17(1)	17(1)	22(1)	-4(1)	-5(1)	1(1)
C(45)	19(1)	16(1)	19(1)	-4(1)	-4(1)	3(1)
C(47)	22(1)	22(1)	18(1)	0(1)	-2(1)	-1(1)
C(48)	18(1)	19(1)	18(1)	0(1)	0(1)	2(1)
C(49)	20(1)	20(1)	17(1)	1(1)	-1(1)	-3(1)
C(50)	17(1)	15(1)	18(1)	-1(1)	-1(1)	3(1)
C(52)	13(1)	19(1)	17(1)	-2(1)	1(1)	-4(1)
C(54)	15(1)	16(1)	19(1)	-1(1)	-2(1)	1(1)
C(56)	15(1)	16(1)	20(1)	-5(1)	-1(1)	-1(1)
C(58)	16(1)	16(1)	16(1)	0(1)	1(1)	1(1)
N(4)	27(1)	25(1)	19(1)	4(1)	-1(1)	-9(1)
N(11)	16(1)	20(1)	16(1)	0(1)	0(1)	-1(1)
N(21)	35(2)	57(2)	16(1)	-3(1)	8(1)	0(1)
N(29)	22(1)	19(1)	18(1)	3(1)	0(1)	3(1)
N(36)	14(1)	14(1)	16(1)	-1(1)	0(1)	-1(1)
N(46)	22(1)	23(1)	16(1)	-3(1)	-4(1)	-1(1)
O(1)	23(1)	23(1)	21(1)	4(1)	-1(1)	-1(1)
O(8)	22(1)	22(1)	28(1)	1(1)	0(1)	-5(1)
O(26)	26(1)	31(1)	14(1)	3(1)	1(1)	-1(1)
O(33)	21(1)	21(1)	21(1)	1(1)	-1(1)	4(1)
O(51)	21(1)	27(1)	16(1)	-2(1)	1(1)	-3(1)
O(53)	18(1)	21(1)	21(1)	-2(1)	-1(1)	2(1)
O(55)	25(1)	19(1)	22(1)	3(1)	-1(1)	2(1)
O(57)	14(1)	26(1)	30(1)	-13(1)	1(1)	-4(1)
O(59)	17(1)	20(1)	20(1)	-4(1)	-4(1)	3(1)
O(60)	14(1)	24(1)	24(1)	-5(1)	-3(1)	2(1)
O(61)	34(1)	41(1)	36(1)	13(1)	-8(1)	-9(1)
O(62)	91(3)	112(3)	66(2)	13(2)	12(2)	-28(3)
C(63)	121(6)	97(5)	112(5)	-1(4)	-34(5)	29(5)
C(64)	159(8)	160(7)	107(5)	-21(5)	-19(5)	94(7)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for TCD173.

	x	y	z	U(eq)
H(2A)	2419	5956	8087	28
H(2B)	-139	6231	8038	28
H(3A)	-111	7056	8453	29
H(3B)	677	6092	8527	29
H(5A)	5464	7913	8438	30
H(5B)	2897	8198	8397	30
H(6A)	4528	8022	7953	27
H(6B)	5316	7066	8032	27
H(9)	3056	5404	8747	21
H(10A)	6597	5116	8918	21
H(10B)	6426	5861	9151	21
H(12A)	7244	3973	9199	38
H(12B)	5634	3692	9453	38
H(12C)	7319	4487	9491	38
H(13)	4518	5607	9591	23
H(15)	653	6463	9006	21
H(17)	-1237	7275	9331	29
H(18)	-3832	7707	9673	35
H(19)	-4238	6966	10101	38
H(22)	440	4396	10269	41
H(24A)	1589	4135	9587	30
H(24B)	3601	4318	9803	30
H(27A)	116	10206	9088	30
H(27B)	494	9494	8848	30
H(28A)	2849	10676	8757	29
H(28B)	3913	10609	9066	29
H(30A)	6274	9239	9173	27
H(30B)	6486	8550	8923	27
H(31A)	2635	8252	8946	31
H(31B)	3701	8117	9253	31
H(34)	4874	10937	8448	19
H(35A)	8491	11035	8283	19

H(35B)	8345	10125	8124	19
H(37A)	9548	11864	7915	29
H(37B)	8190	11918	7625	29
H(37C)	9712	11093	7692	29
H(38)	6726	10005	7671	18
H(40)	2342	9803	8254	19
H(42)	313	8832	7990	21
H(43)	-1957	8122	7666	24
H(44)	-1640	8338	7179	22
H(47)	3910	10456	6841	25
H(49A)	3994	11431	7488	23
H(49B)	6249	11099	7338	23
H(54)	749	2849	8449	20
H(56)	3206	3866	8268	20
H(11)	3590(50)	4460(20)	9160(7)	39(10)
H(21)	-2380(70)	5490(30)	10387(6)	58(12)
H(36)	5850(60)	11530(20)	7965(8)	55(12)
H(46)	1000(60)	9370(20)	6790(6)	42(10)
H(55)	2990(60)	2660(20)	8908(6)	33
H(57)	6550(50)	3860(20)	8560(7)	35
H(61A)	7610(20)	1970(11)	8689(5)	59(13)
H(61B)	9710(50)	1838(6)	8850(1)	73(15)
H(62)	7108(13)	1816(3)	9222(6)	134
H(63A)	9175	2725	9472	132
H(63B)	8101	2341	9754	132
H(64A)	9968	1021	9630	213
H(64B)	11494	1791	9747	213
H(64C)	11392	1600	9416	213

Table 7. Torsion angles [°] for TCD173.

O(1)-C(2)-C(3)-N(4)	-56.0(3)	N(21)-C(20)-C(25)-C(16)	-178.5(3)
N(4)-C(5)-C(6)-O(1)	55.7(3)	C(19)-C(20)-C(25)-C(16)	1.6(4)
O(8)-C(7)-C(9)-C(15)	105.9(3)	N(21)-C(20)-C(25)-C(23)	1.1(3)
N(4)-C(7)-C(9)-C(15)	-71.5(3)	C(19)-C(20)-C(25)-C(23)	-178.8(3)
O(8)-C(7)-C(9)-C(10)	-15.6(3)	C(22)-C(23)-C(25)-C(16)	178.0(3)
N(4)-C(7)-C(9)-C(10)	167.0(2)	C(24)-C(23)-C(25)-C(16)	-4.5(5)
C(15)-C(9)-C(10)-N(11)	46.5(3)	C(22)-C(23)-C(25)-C(20)	-1.5(3)
C(7)-C(9)-C(10)-N(11)	167.32(19)	C(24)-C(23)-C(25)-C(20)	175.9(3)
N(11)-C(13)-C(14)-C(15)	-16.7(3)	O(26)-C(27)-C(28)-N(29)	56.5(3)
C(24)-C(13)-C(14)-C(15)	-139.1(3)	N(29)-C(30)-C(31)-O(26)	-56.2(3)
N(11)-C(13)-C(14)-C(16)	164.7(2)	O(33)-C(32)-C(34)-C(40)	91.1(3)
C(24)-C(13)-C(14)-C(16)	42.3(3)	N(29)-C(32)-C(34)-C(40)	-85.0(3)
C(16)-C(14)-C(15)-C(9)	174.0(2)	O(33)-C(32)-C(34)-C(35)	-29.3(3)
C(13)-C(14)-C(15)-C(9)	-4.5(4)	N(29)-C(32)-C(34)-C(35)	154.6(2)
C(10)-C(9)-C(15)-C(14)	-10.8(3)	C(40)-C(34)-C(35)-N(36)	43.9(3)
C(7)-C(9)-C(15)-C(14)	-130.9(3)	C(32)-C(34)-C(35)-N(36)	162.72(18)
C(15)-C(14)-C(16)-C(17)	-16.3(4)	N(36)-C(38)-C(39)-C(40)	-19.0(3)
C(13)-C(14)-C(16)-C(17)	162.2(3)	C(49)-C(38)-C(39)-C(40)	-142.5(2)
C(15)-C(14)-C(16)-C(25)	164.1(3)	N(36)-C(38)-C(39)-C(41)	164.38(19)
C(13)-C(14)-C(16)-C(25)	-17.3(3)	C(49)-C(38)-C(39)-C(41)	40.9(3)
C(25)-C(16)-C(17)-C(18)	0.5(4)	C(41)-C(39)-C(40)-C(34)	171.9(2)
C(14)-C(16)-C(17)-C(18)	-179.0(3)	C(38)-C(39)-C(40)-C(34)	-4.4(4)
C(16)-C(17)-C(18)-C(19)	0.6(4)	C(35)-C(34)-C(40)-C(39)	-8.1(3)
C(17)-C(18)-C(19)-C(20)	-0.7(4)	C(32)-C(34)-C(40)-C(39)	-126.9(2)
C(18)-C(19)-C(20)-N(21)	179.7(3)	C(40)-C(39)-C(41)-C(42)	-13.4(4)
C(18)-C(19)-C(20)-C(25)	-0.3(4)	C(38)-C(39)-C(41)-C(42)	163.1(2)
N(21)-C(22)-C(23)-C(25)	1.3(4)	C(40)-C(39)-C(41)-C(50)	171.4(2)
N(21)-C(22)-C(23)-C(24)	-175.6(3)	C(38)-C(39)-C(41)-C(50)	-12.1(3)
C(22)-C(23)-C(24)-C(13)	-156.1(3)	C(50)-C(41)-C(42)-C(43)	0.3(3)
C(25)-C(23)-C(24)-C(13)	27.4(4)	C(39)-C(41)-C(42)-C(43)	-175.0(2)
N(11)-C(13)-C(24)-C(23)	-167.7(2)	C(41)-C(42)-C(43)-C(44)	1.7(4)
C(14)-C(13)-C(24)-C(23)	-45.6(3)	C(42)-C(43)-C(44)-C(45)	-1.1(4)
C(17)-C(16)-C(25)-C(20)	-1.6(4)	C(43)-C(44)-C(45)-N(46)	178.1(3)
C(14)-C(16)-C(25)-C(20)	178.0(2)	C(43)-C(44)-C(45)-C(50)	-1.5(4)
C(17)-C(16)-C(25)-C(23)	178.9(3)	N(46)-C(47)-C(48)-C(50)	1.4(3)
C(14)-C(16)-C(25)-C(23)	-1.5(4)	N(46)-C(47)-C(48)-C(49)	-173.2(3)

C(47)-C(48)-C(49)-C(38)	-150.8(3)	C(6)-C(5)-N(4)-C(3)	-56.9(3)
C(50)-C(48)-C(49)-C(38)	35.1(3)	C(2)-C(3)-N(4)-C(7)	-118.7(3)
N(36)-C(38)-C(49)-C(48)	-174.4(2)	C(2)-C(3)-N(4)-C(5)	56.8(3)
C(39)-C(38)-C(49)-C(48)	-51.1(3)	C(9)-C(10)-N(11)-C(12)	163.7(2)
N(46)-C(45)-C(50)-C(41)	-176.1(2)	C(9)-C(10)-N(11)-C(13)	-70.8(2)
C(44)-C(45)-C(50)-C(41)	3.6(4)	C(14)-C(13)-N(11)-C(12)	177.5(2)
N(46)-C(45)-C(50)-C(48)	1.8(3)	C(24)-C(13)-N(11)-C(12)	-56.1(3)
C(44)-C(45)-C(50)-C(48)	-178.6(2)	C(14)-C(13)-N(11)-C(10)	53.5(3)
C(42)-C(41)-C(50)-C(45)	-2.9(4)	C(24)-C(13)-N(11)-C(10)	179.9(2)
C(39)-C(41)-C(50)-C(45)	172.9(2)	C(19)-C(20)-N(21)-C(22)	179.7(3)
C(42)-C(41)-C(50)-C(48)	179.7(2)	C(25)-C(20)-N(21)-C(22)	-0.3(3)
C(39)-C(41)-C(50)-C(48)	-4.5(4)	C(23)-C(22)-N(21)-C(20)	-0.7(4)
C(47)-C(48)-C(50)-C(45)	-2.0(3)	O(33)-C(32)-N(29)-C(28)	172.2(3)
C(49)-C(48)-C(50)-C(45)	173.8(2)	C(34)-C(32)-N(29)-C(28)	-11.8(4)
C(47)-C(48)-C(50)-C(41)	175.8(2)	O(33)-C(32)-N(29)-C(30)	1.1(4)
C(49)-C(48)-C(50)-C(41)	-8.5(4)	C(34)-C(32)-N(29)-C(30)	177.0(2)
O(53)-C(52)-C(54)-O(55)	-167.1(2)	C(27)-C(28)-N(29)-C(32)	134.7(3)
O(51)-C(52)-C(54)-O(55)	15.1(3)	C(27)-C(28)-N(29)-C(30)	-53.6(3)
O(53)-C(52)-C(54)-C(56)	71.4(3)	C(31)-C(30)-N(29)-C(32)	-134.6(2)
O(51)-C(52)-C(54)-C(56)	-106.4(2)	C(31)-C(30)-N(29)-C(28)	53.1(3)
O(55)-C(54)-C(56)-O(57)	-72.3(2)	C(34)-C(35)-N(36)-C(37)	165.7(2)
C(52)-C(54)-C(56)-O(57)	49.1(2)	C(34)-C(35)-N(36)-C(38)	-69.6(2)
O(55)-C(54)-C(56)-C(58)	50.9(3)	C(39)-C(38)-N(36)-C(37)	177.78(19)
C(52)-C(54)-C(56)-C(58)	172.3(2)	C(49)-C(38)-N(36)-C(37)	-55.8(3)
O(57)-C(56)-C(58)-O(60)	8.1(3)	C(39)-C(38)-N(36)-C(35)	54.9(2)
C(54)-C(56)-C(58)-O(60)	-112.1(2)	C(49)-C(38)-N(36)-C(35)	-178.63(19)
O(57)-C(56)-C(58)-O(59)	-173.1(2)	C(44)-C(45)-N(46)-C(47)	179.5(3)
C(54)-C(56)-C(58)-O(59)	66.7(3)	C(50)-C(45)-N(46)-C(47)	-0.9(3)
O(8)-C(7)-N(4)-C(5)	1.3(4)	C(48)-C(47)-N(46)-C(45)	-0.4(3)
C(9)-C(7)-N(4)-C(5)	178.6(2)	C(5)-C(6)-O(1)-C(2)	-56.6(3)
O(8)-C(7)-N(4)-C(3)	176.5(2)	C(3)-C(2)-O(1)-C(6)	57.2(3)
C(9)-C(7)-N(4)-C(3)	-6.2(4)	C(30)-C(31)-O(26)-C(27)	60.5(3)
C(6)-C(5)-N(4)-C(7)	119.0(3)	C(28)-C(27)-O(26)-C(31)	-60.2(3)

Table 8. Hydrogen bonds for TCD173 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(5)-H(5A)...O(33)	0.99	2.45	3.280(3)	141
C(9)-H(9)...O(53)	1.00	2.60	3.469(3)	145
C(10)-H(10A)...O(57)	0.99	2.49	3.117(3)	121
C(12)-H(12A)...O(51)#1	0.98	2.57	3.407(3)	143
C(22)-H(22)...O(62)#2	0.95	2.58	3.527(5)	177
C(27)-H(27B)...O(33)#3	0.99	2.37	3.299(3)	156
C(30)-H(30B)...O(8)	0.99	2.60	3.411(3)	139
C(35)-H(35A)...O(60)#4	0.99	2.57	3.125(3)	115
C(44)-H(44)...O(59)#5	0.95	2.44	3.373(3)	169
C(49)-H(49B)...O(1)#6	0.99	2.40	3.273(3)	147
C(54)-H(54)...O(60)#3	1.00	2.28	3.181(3)	149
N(11)-H(11)...O(51)	0.89(2)	1.84(2)	2.729(3)	171(3)
N(21)-H(21)...O(26)#7	0.90(2)	2.00(3)	2.859(3)	160(4)
N(36)-H(36)...O(59)#4	0.90(2)	1.73(2)	2.631(3)	179(4)
N(36)-H(36)...O(60)#4	0.90(2)	2.55(4)	3.109(3)	120(3)
N(46)-H(46)...O(53)#5	0.89(2)	2.05(2)	2.908(3)	161(3)
O(55)-H(55)...O(51)	0.84(3)	2.09(3)	2.634(3)	122(3)
O(57)-H(57)...O(53)#1	0.85(3)	2.10(3)	2.804(3)	141(3)
O(57)-H(57)...O(60)	0.85(3)	2.23(4)	2.679(3)	113(3)
O(61)-H(61A)...O(60)	0.94	1.91	2.831(3)	165
O(61)-H(61B)...O(55)#1	0.90	2.01	2.887(3)	162
O(62)-H(62)...O(61)	0.86	1.95	2.795(4)	169

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z #2 x-1/2,-y+1/2,-z+2 #3 x-1,y,z

#4 x,y+1,z #5 -x,y+1/2,-z+3/2 #6 -x+1,y+1/2,-z+3/2

#7 x-1/2,-y+3/2,-z+2