SUPPORTING INFORMATION

Model peptides containing the 3-sulfanyl-norbornene amino acid, a conformationally constrained cysteine analogue effective inducer of 3_{10}-helix secondary structures †

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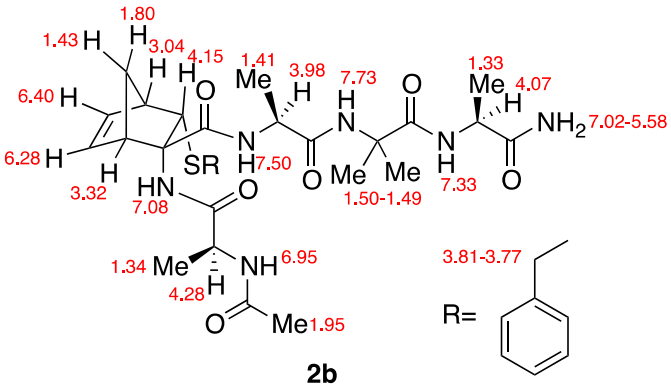
General Experimental Methods

Chemicals were obtained from commercial sources and used without further purification. Preparative RP-HPLC analyses were performed using a DENALI C-18 column (10 mm, 250_22 mm). Two mobile phases were used: A=94.9% water, 5% MeCN, 0.1% TFA; B=95% MeCN, 4.9% water, 0.1% TFA. ESI mass spectra were recorded on an LCQ Advantage spectrometer. NMR spectroscopic analysis: NMR spectroscopic experiments were carried out on either 200 MHz spectrometer (200 and 50 MHz for ^{1}H and ^{13}C, respectively), 500 MHz spectrometer (500 and 125 MHz for ^{1}H and ^{13}C, respectively) or 300 MHz spectrometer (300 and 75 MHz for ^{1}H and ^{13}C, respectively) To take advantage of the magnetic field value, measurements that required temperatures higher than room temperature for observing coalescence were performed in an apparatus with a ^{1}H resonance frequency of 300 MHz spectrometer. 2D-NOESY experiments on peptides **2a** and **2b** were performed at different mixing times (300, 500 ms). Chemical shifts are given in ppm relative to $CDCl_3$ or CD_3CN, CD_3OD as internal standards, and coupling constants J are reported in hertz (Hz). The MW mediated reaction were performed using MW reactor with IR temperature detector.

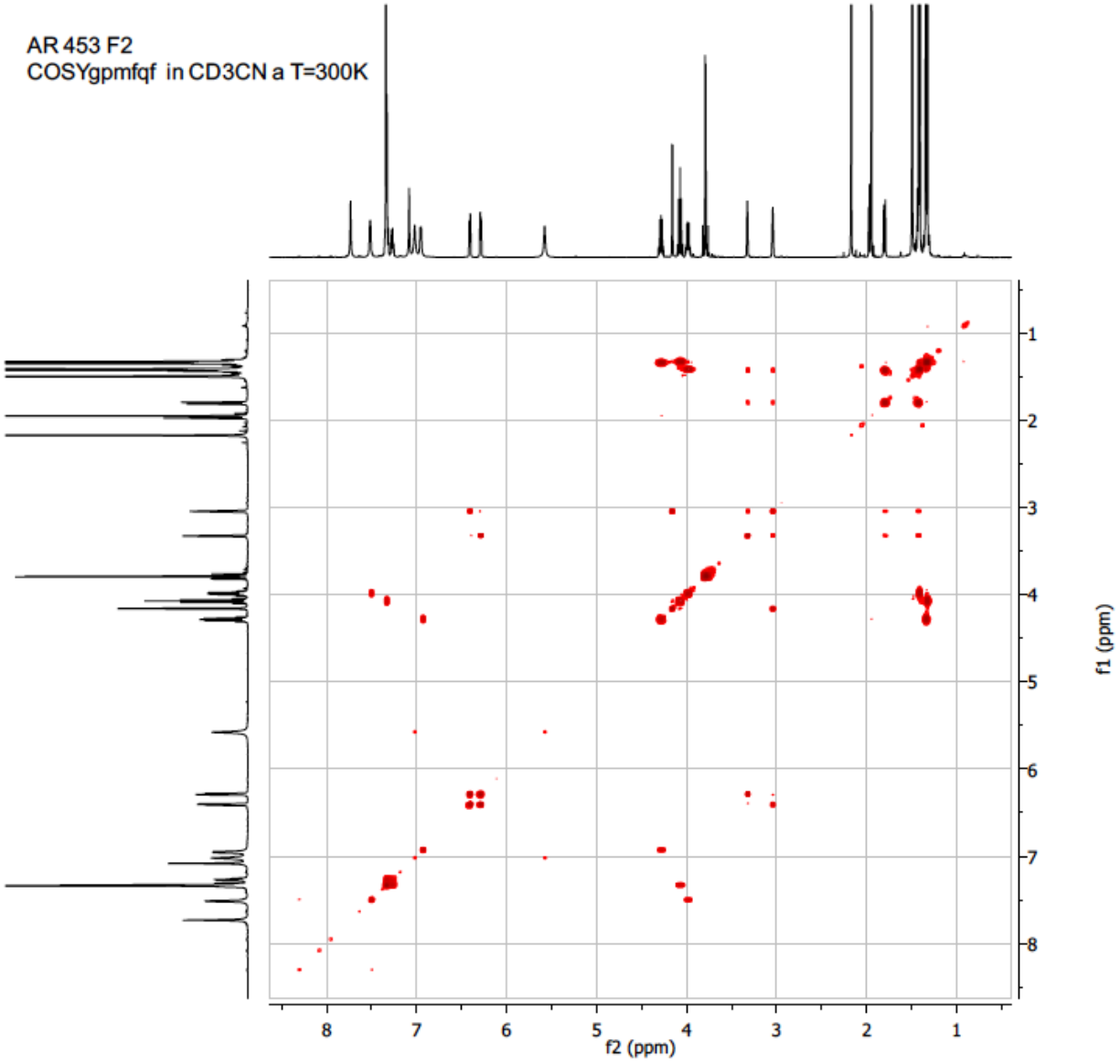
Two dimensional NMR experiments

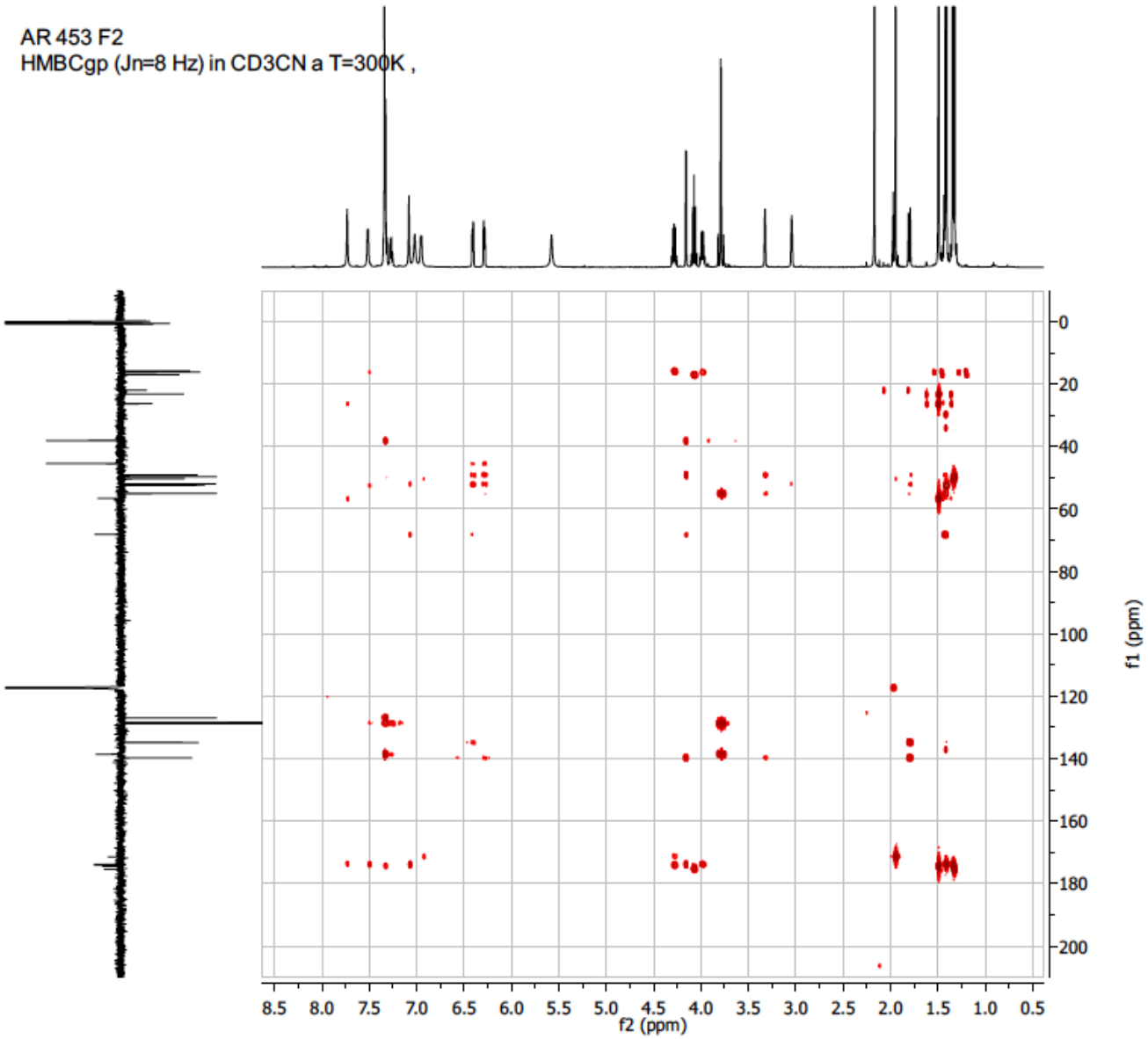
Compound 2b

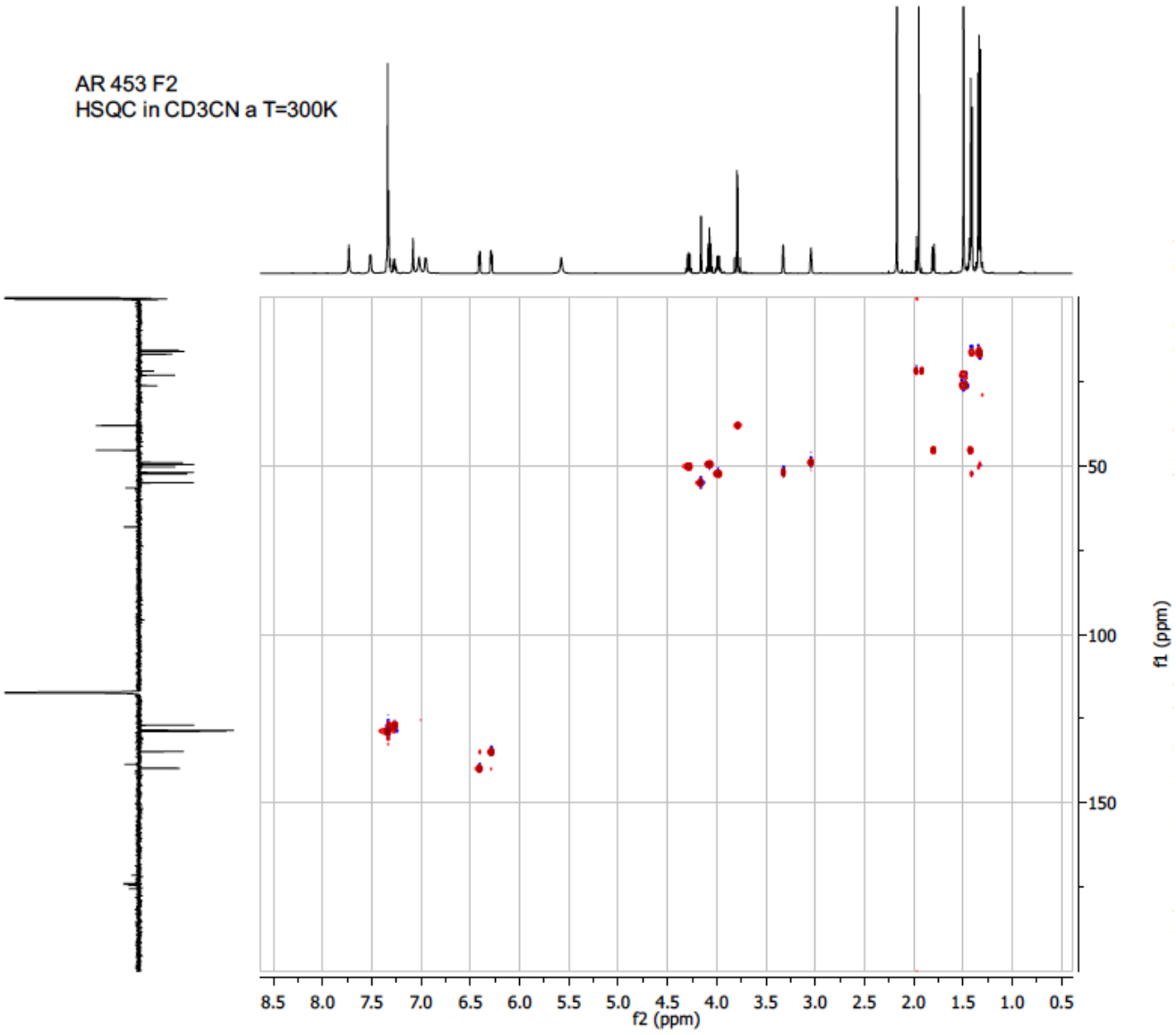
Protons signal assignment:

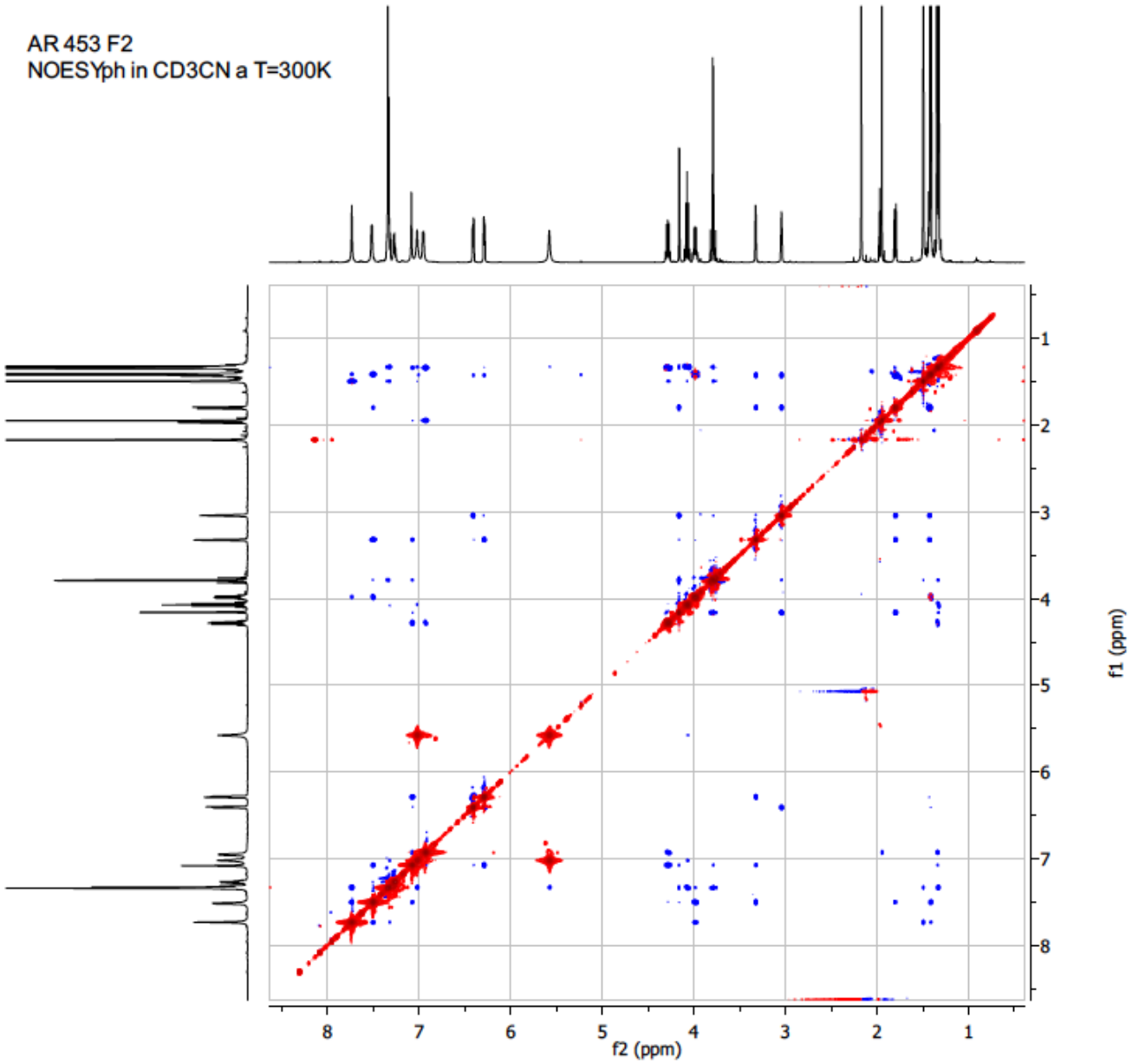


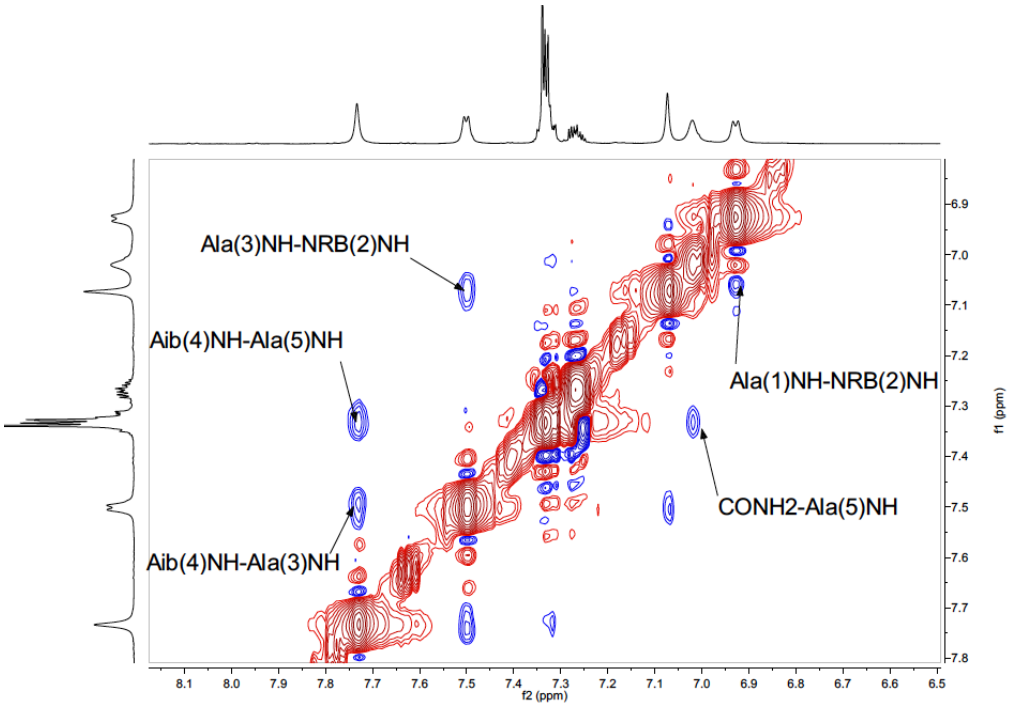
2b




AR 453 F2
HMBCgp (Jn=8 Hz) in CD3CN a T=300K ,
f1 (ppm)
f2 (ppm)

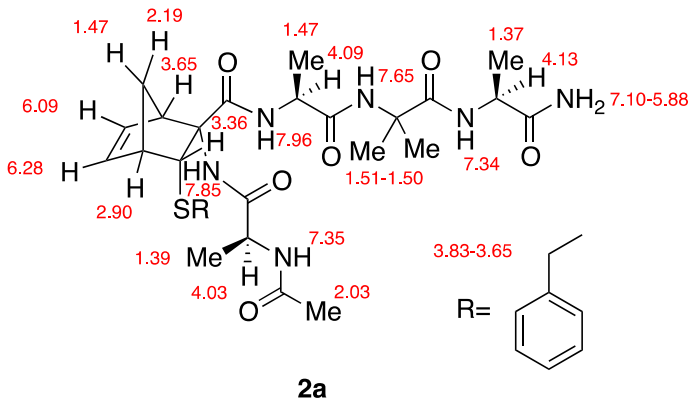

AR 453 F2
HSQC in CD3CN a T=300K
50
100
150
f1 (ppm)
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f2 (ppm)


AR 453 F2
NOESYph in CD3CN a T=300K
f2 (ppm)
f1 (ppm)

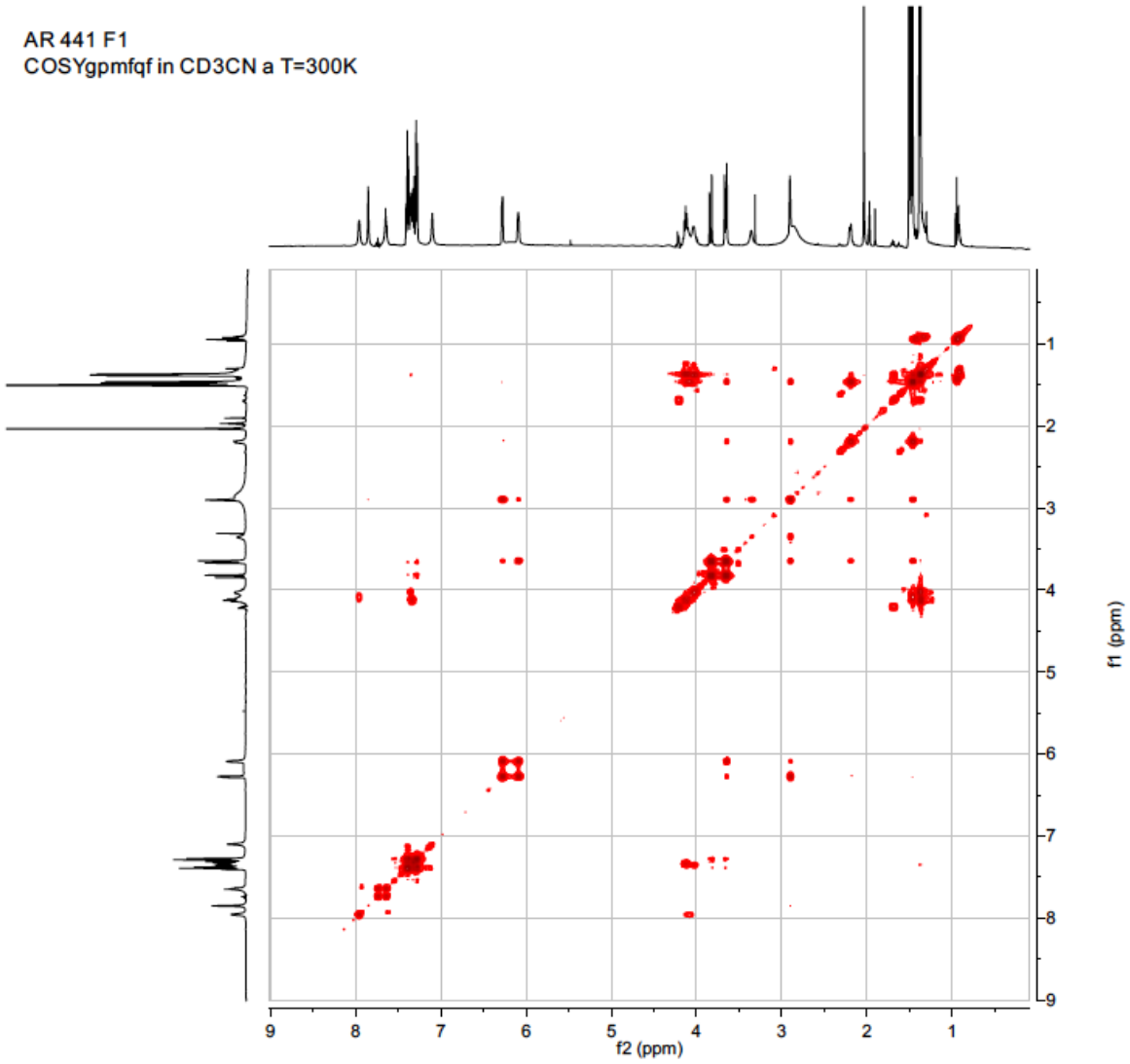


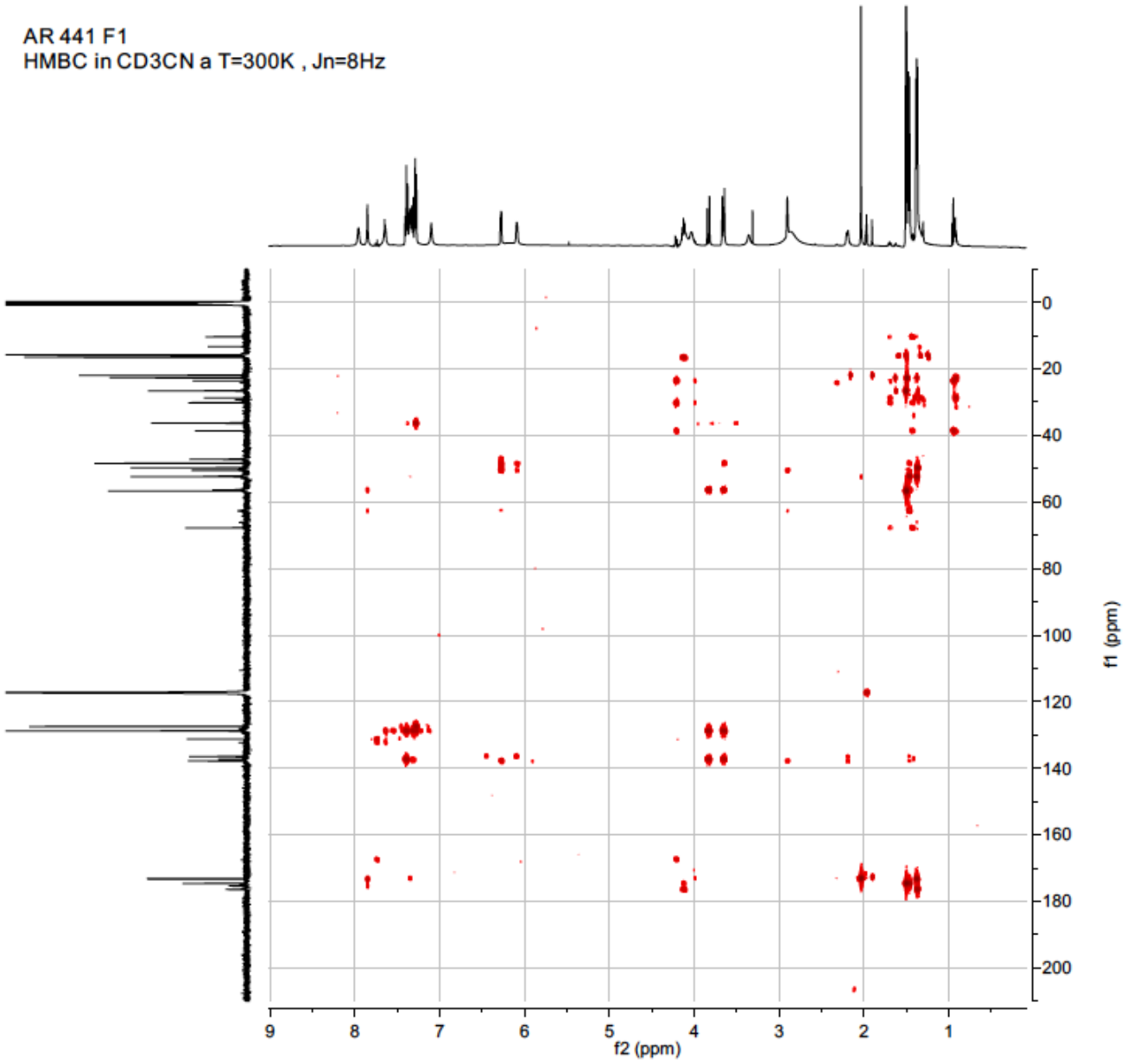
Amide protons region of the NOESY spectrum (τ_m 500ms, BBI probe, 300K, 500MHz) of peptide **2b** in CD_3CN (20mM). Sequential short range NH_i-NH_{i+1} signals.

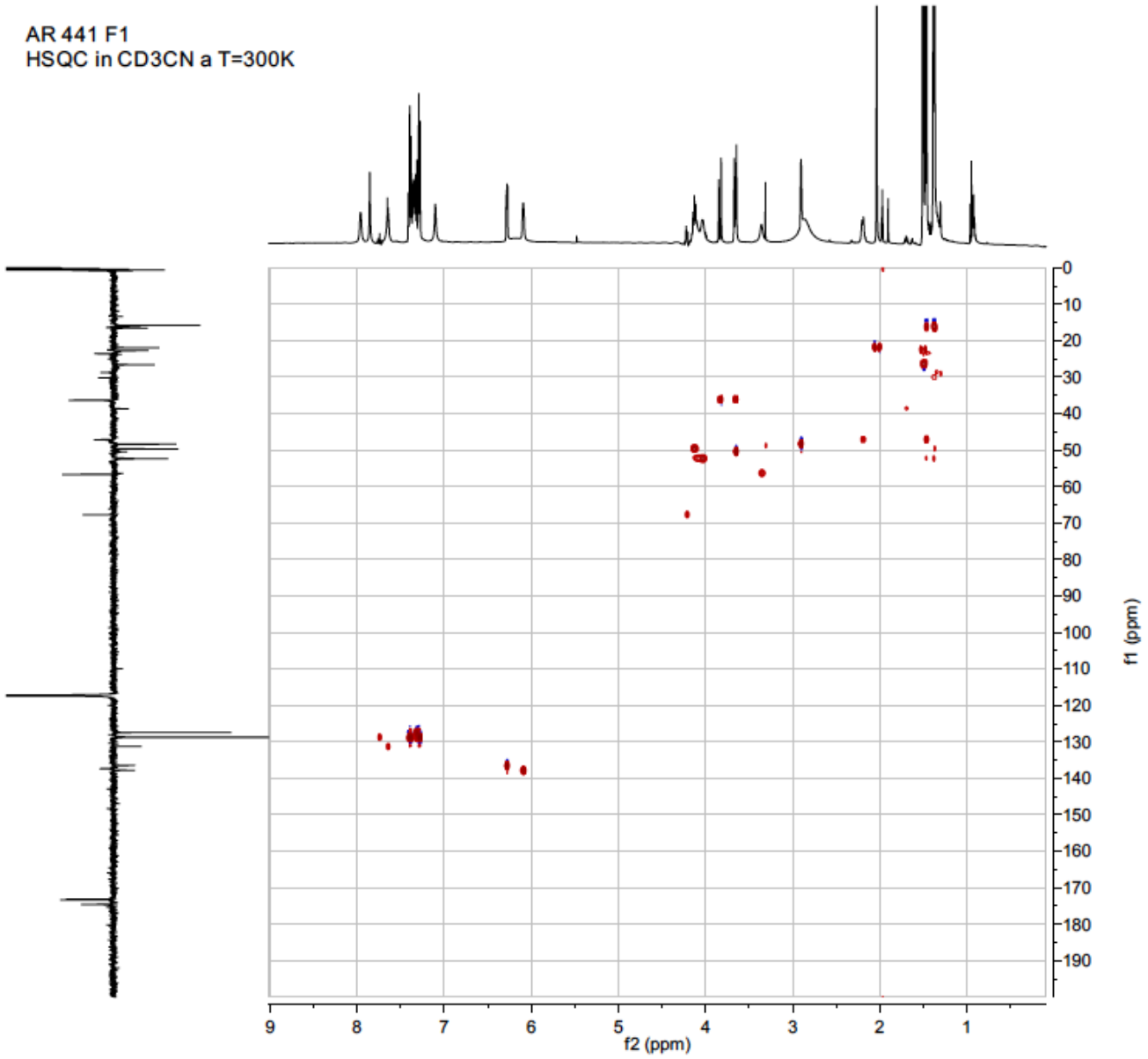
Compound 2a
Protons signals assignments:

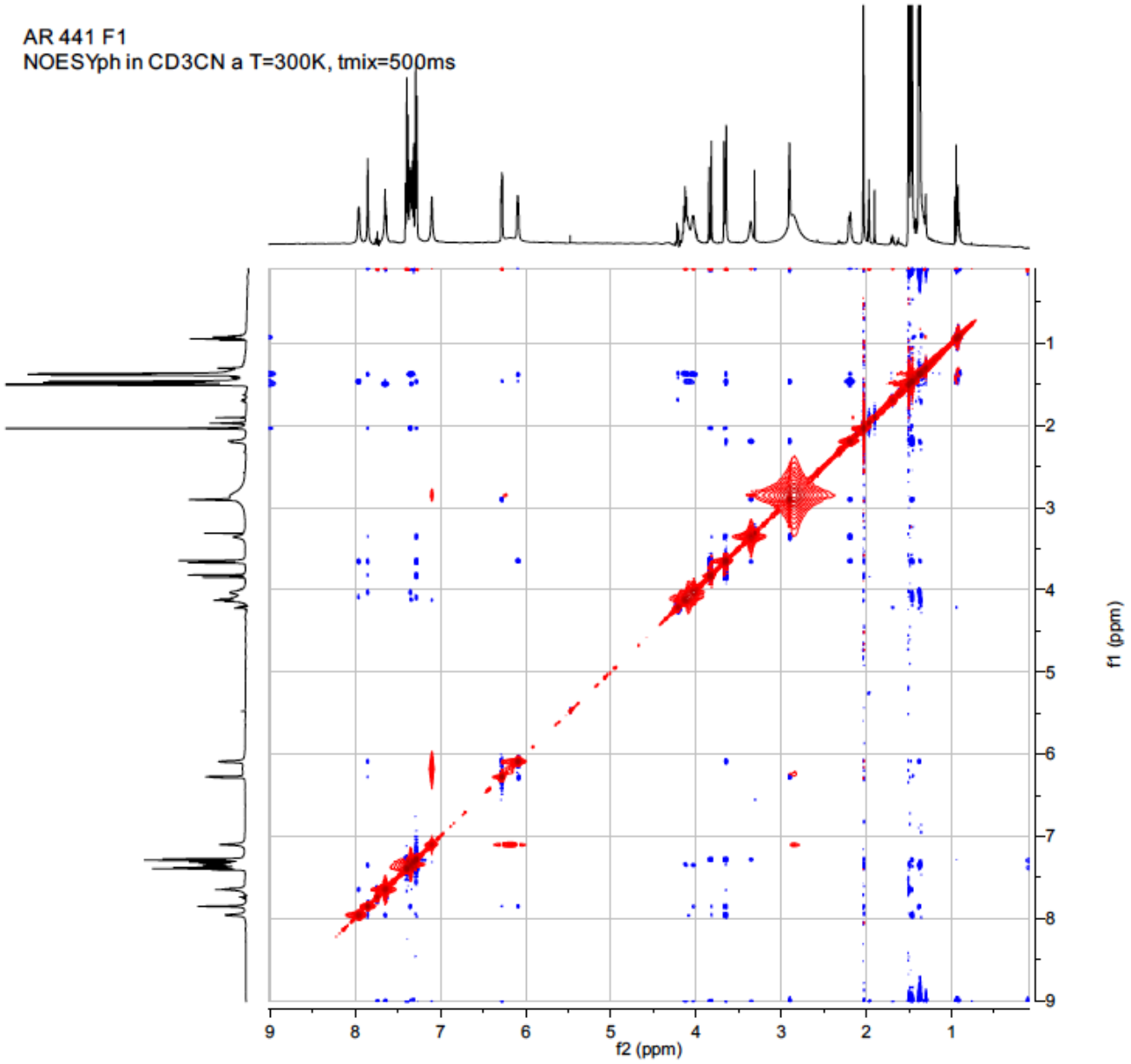


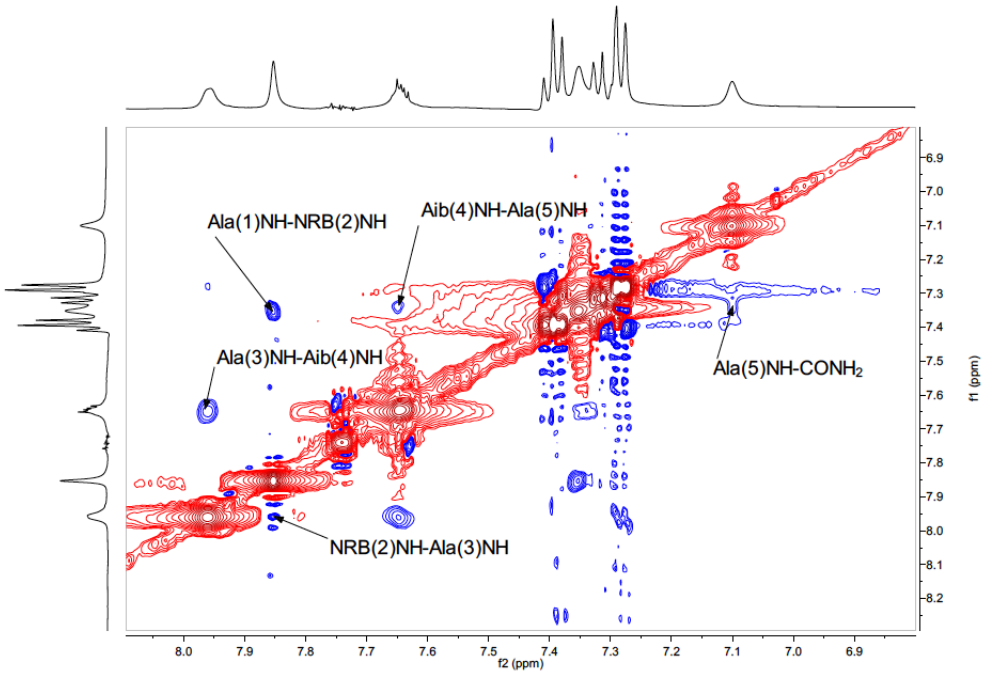
2a



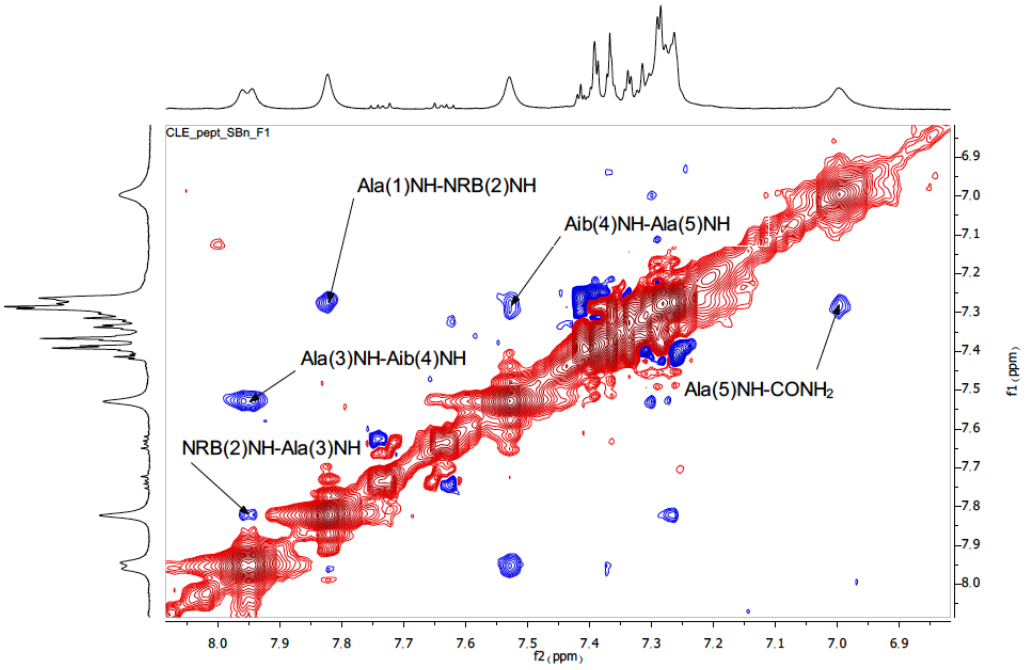

AR 441 F1
HMBC in CD3CN a T=300K , Jn=8Hz
f2 (ppm)
f1 (ppm)


AR 441 F1
HSQC in CD3CN a T=300K
f2 (ppm)
f1 (ppm)


AR 441 F1
NOESYph in CD3CN a T=300K, tmix=500ms
f2 (ppm)
f1 (ppm)



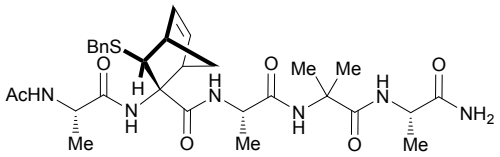
1) Amide protons region of the NOESY spectrum (τ_m 500 ms, BBI probe, 300 K, 500 MHz) of peptide **2a** in CD_3CN (20 mM). Sequential short range NH_i-NH_{i+1} signals.



2) Amide protons region of the NOESY spectrum (τ_m 300 ms, BBI probe, 300 K, 300 MHz) of peptide **2a** in CD_3CN (8 mM). Sequential short range NH_i-NH_{i+1} signals.

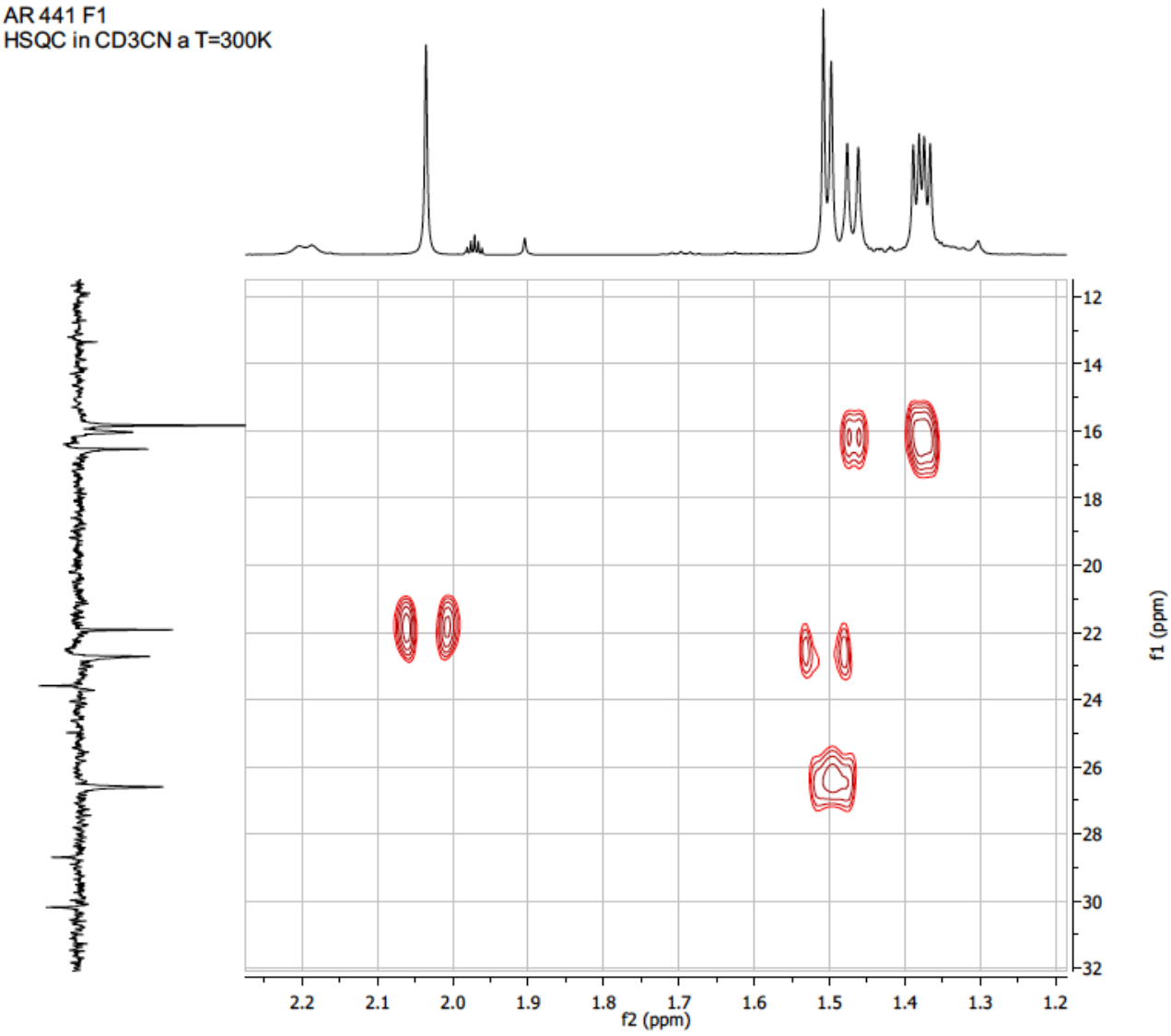
Non magnetic equivalence (NME) evaluation

Compound 2a

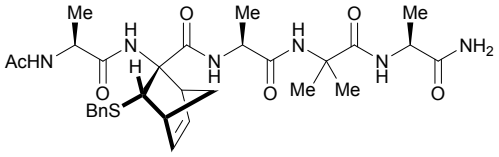


2a

Aib chemical shift (26ppm.60-22.72 ppm,), NME = 3.88 ppm;

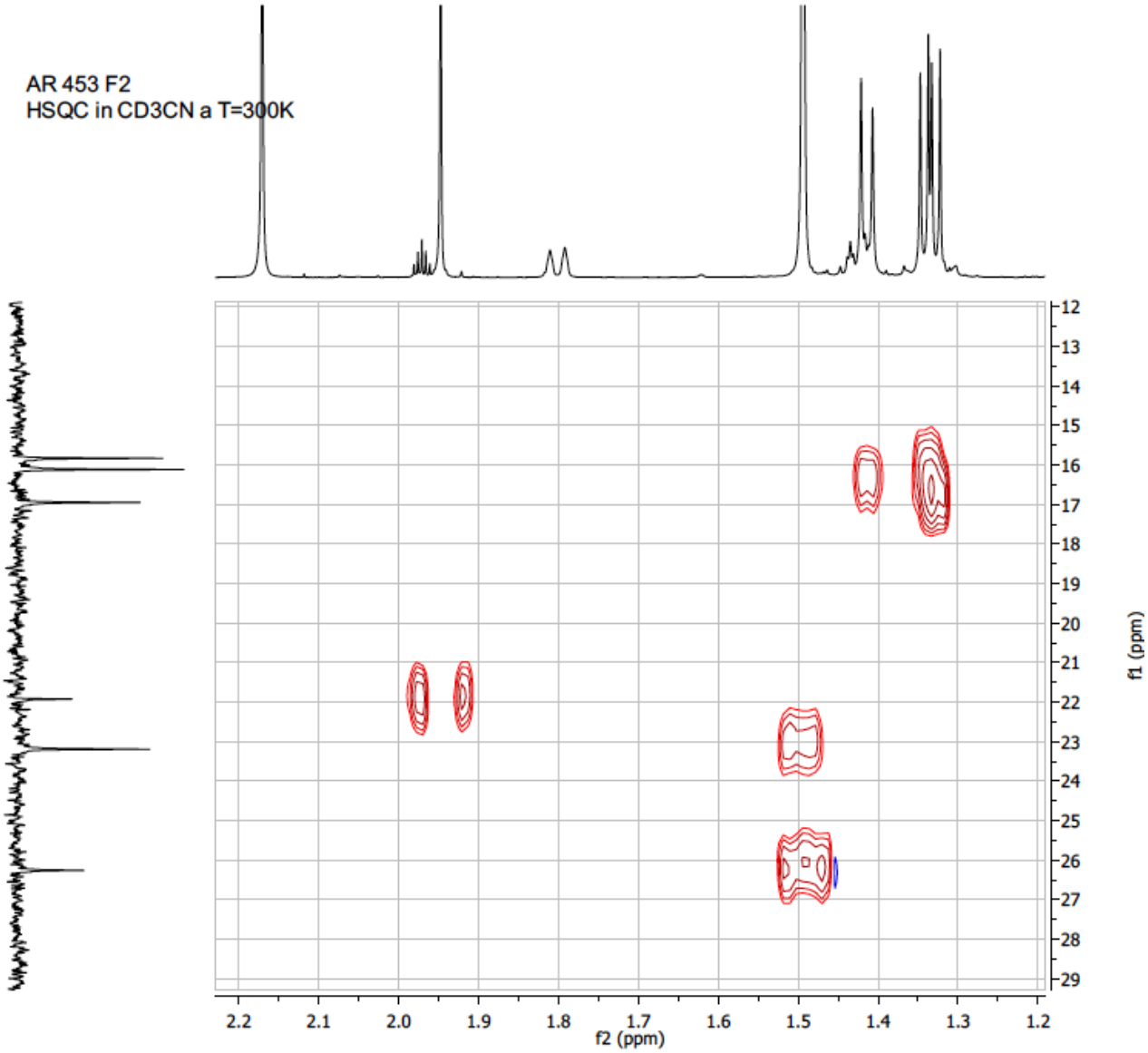


Compound 2b



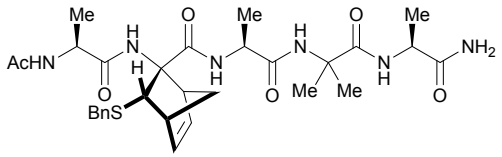
2b

Aib (26.26ppm-23.19 ppm), NME = 3.07 ppm;

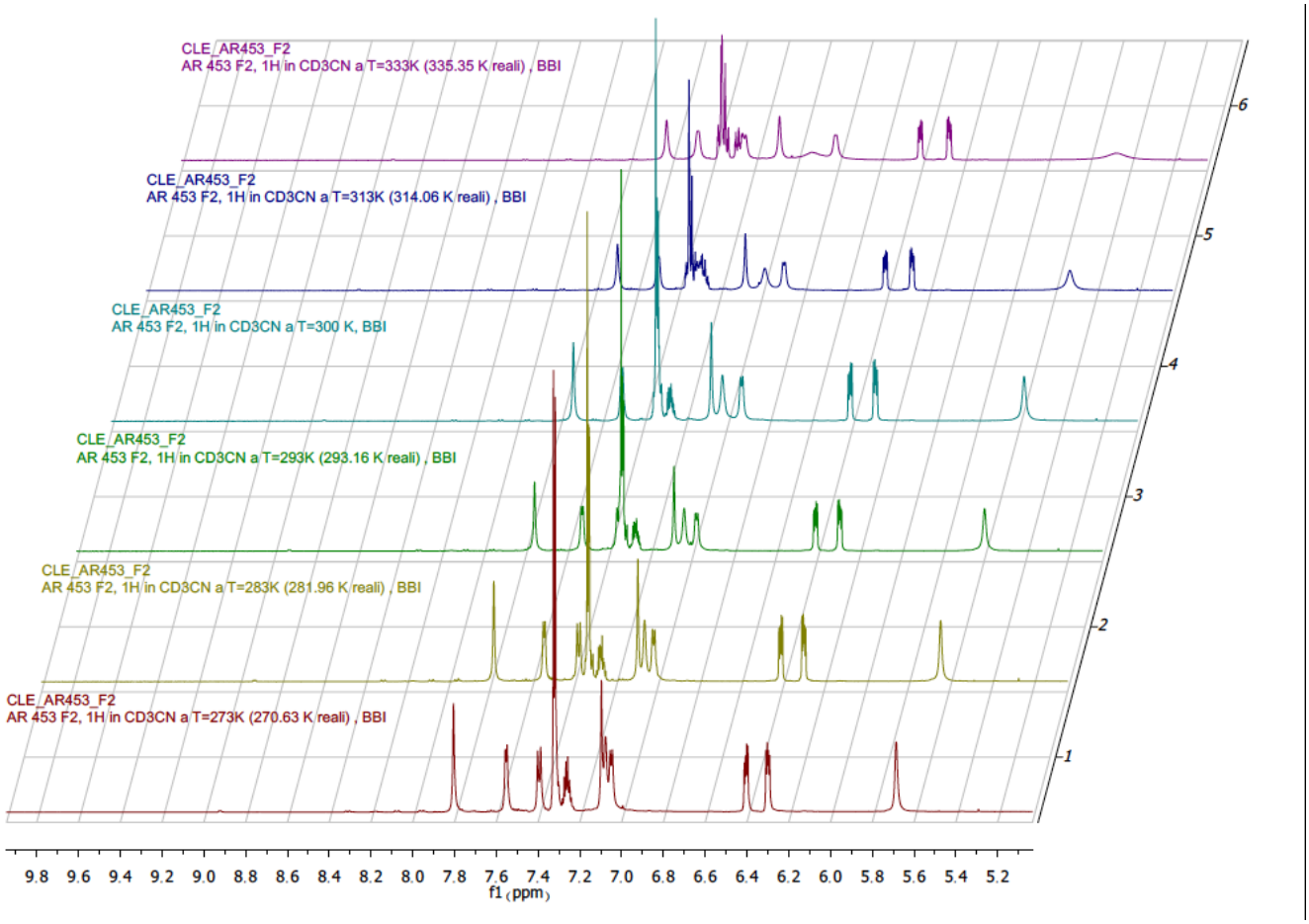


VT-NMR, DMSO$_{d6}$ titration

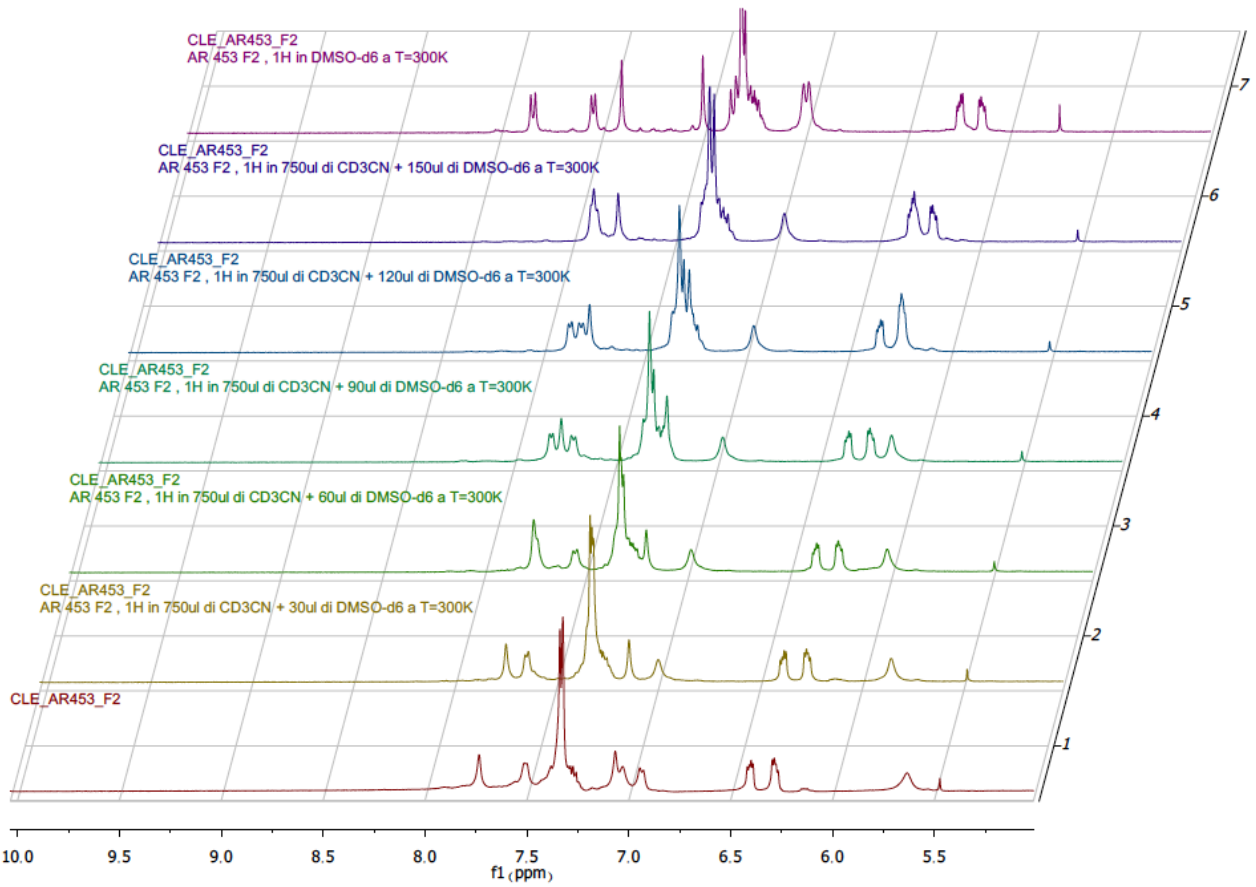
Compound 2b



2b

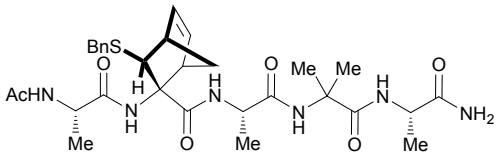


Variation temperature NMR for compound **2b**

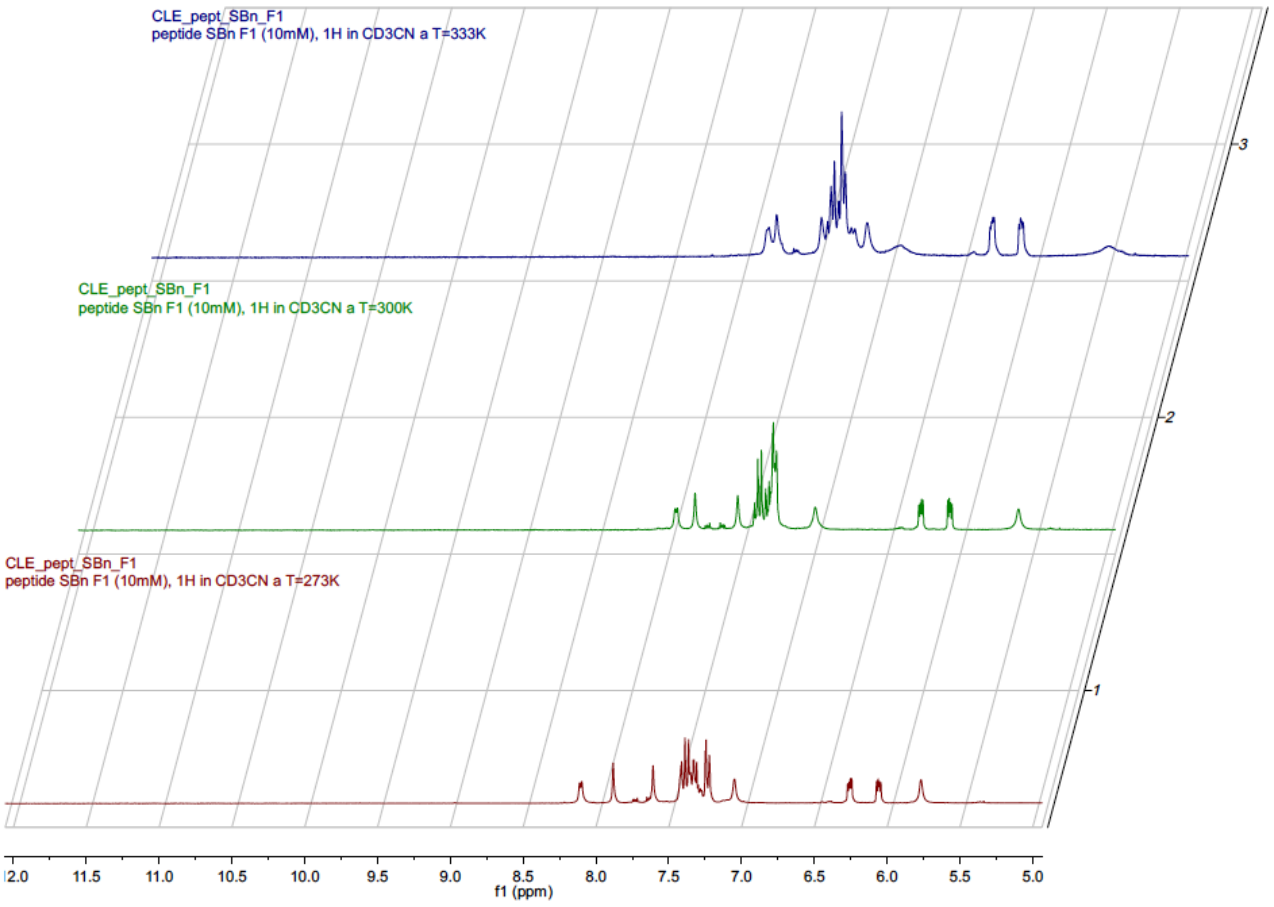


DMSO$_{d6}$ titration NMR of compound **2b**

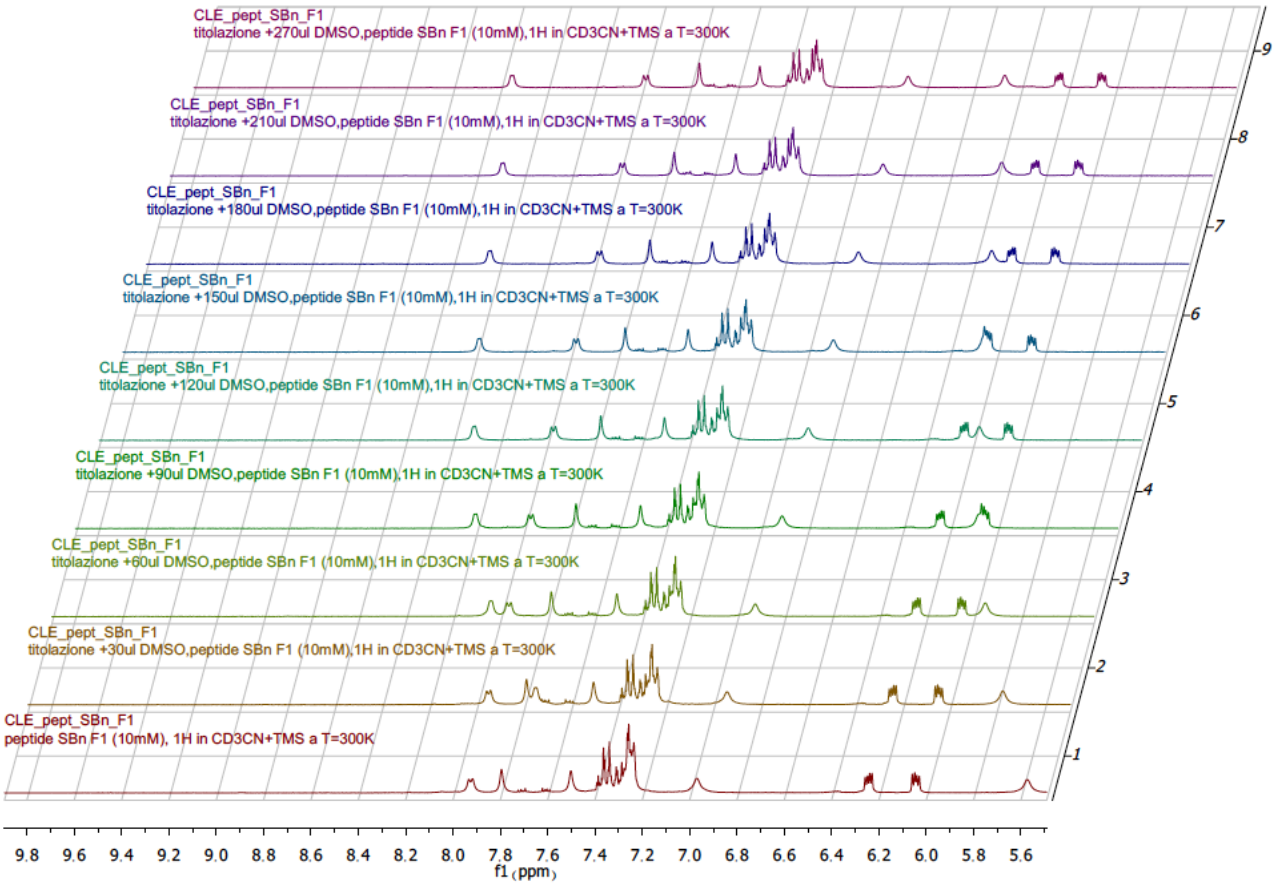
Compound 2a



2a



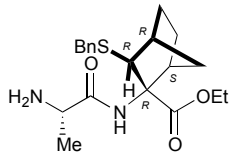
Variation temperature NMR of compound **2a**



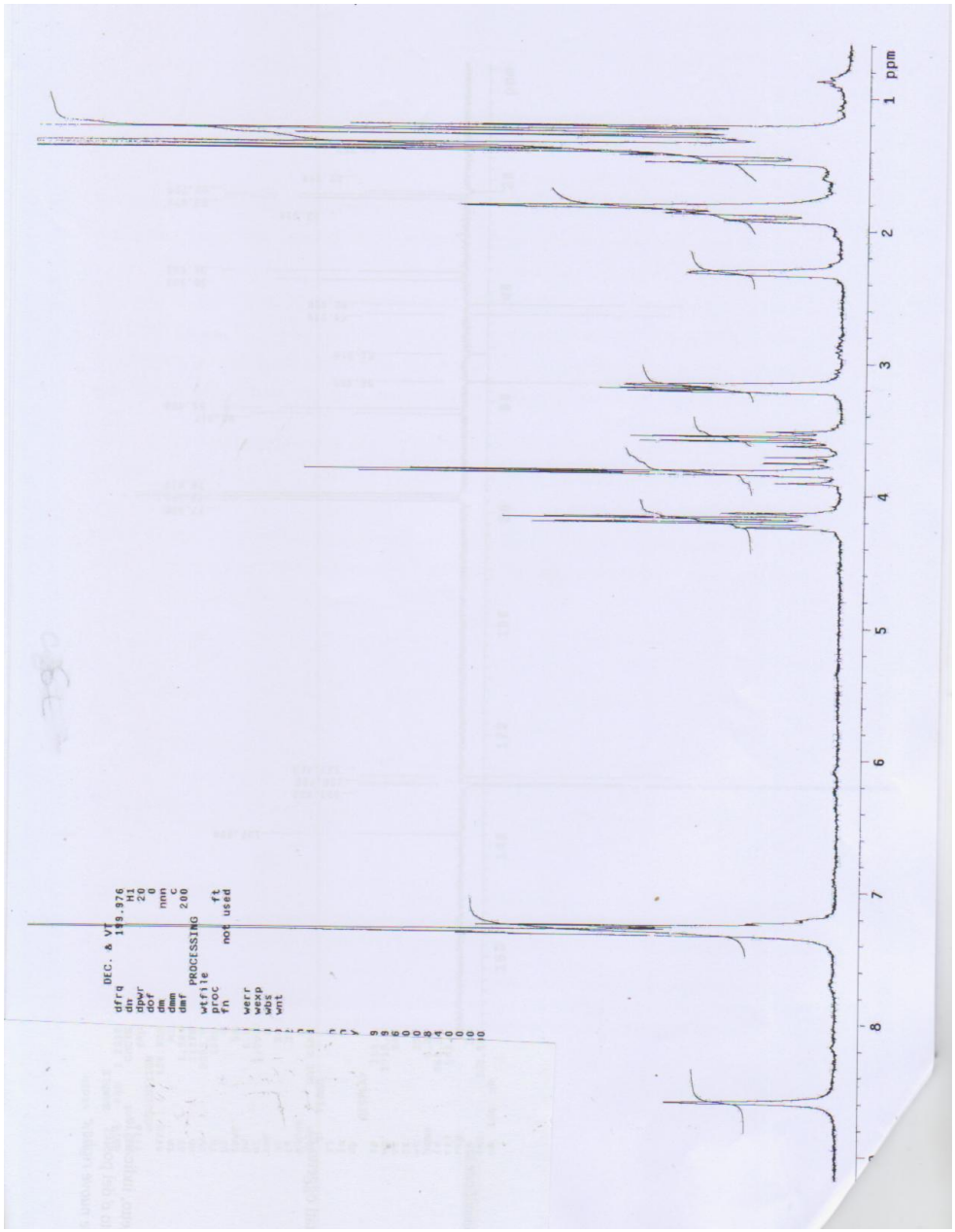
DMSO$_{d6}$ titration NMR of compound **2a**

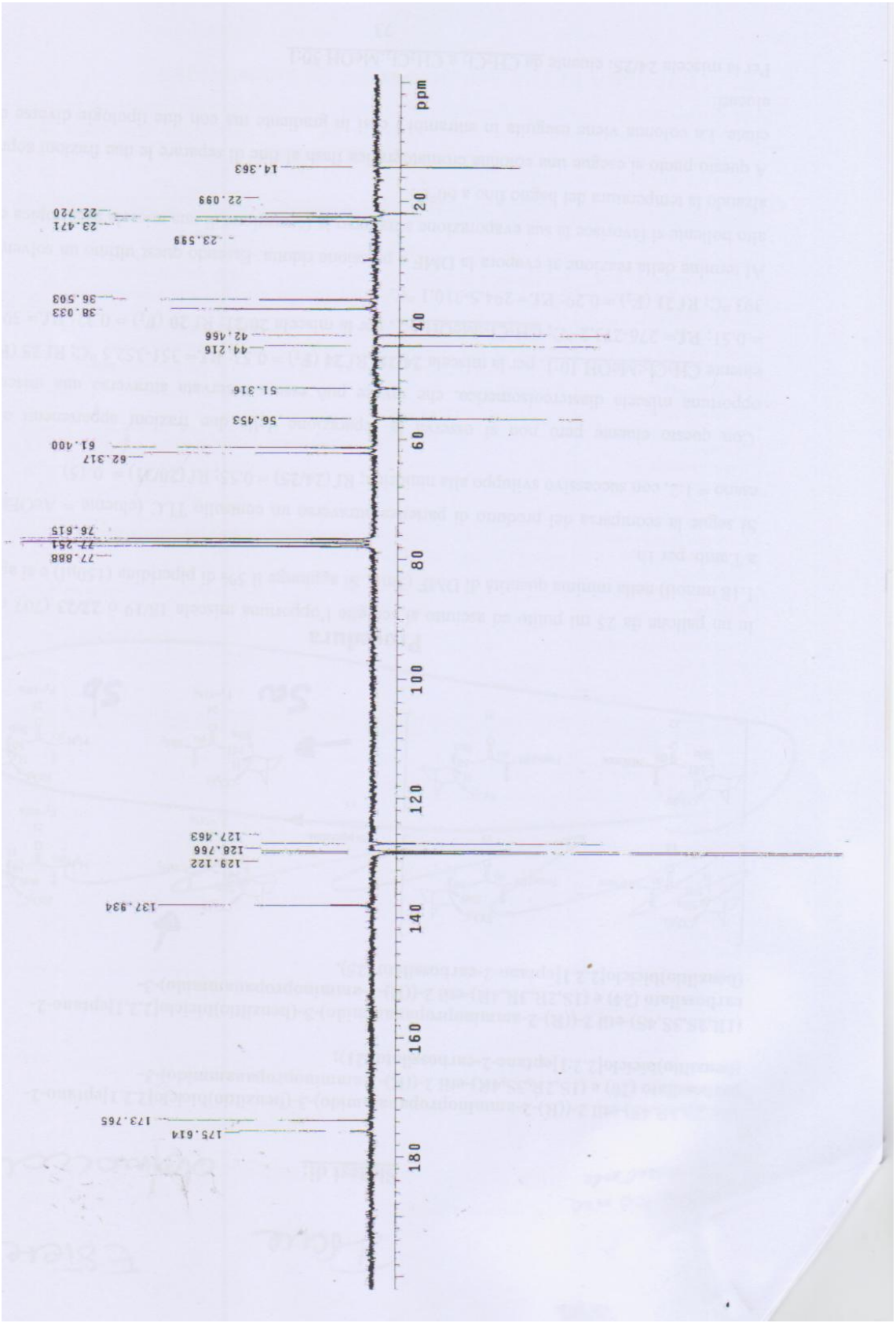
NMR

Compound 6a

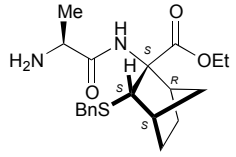


6a

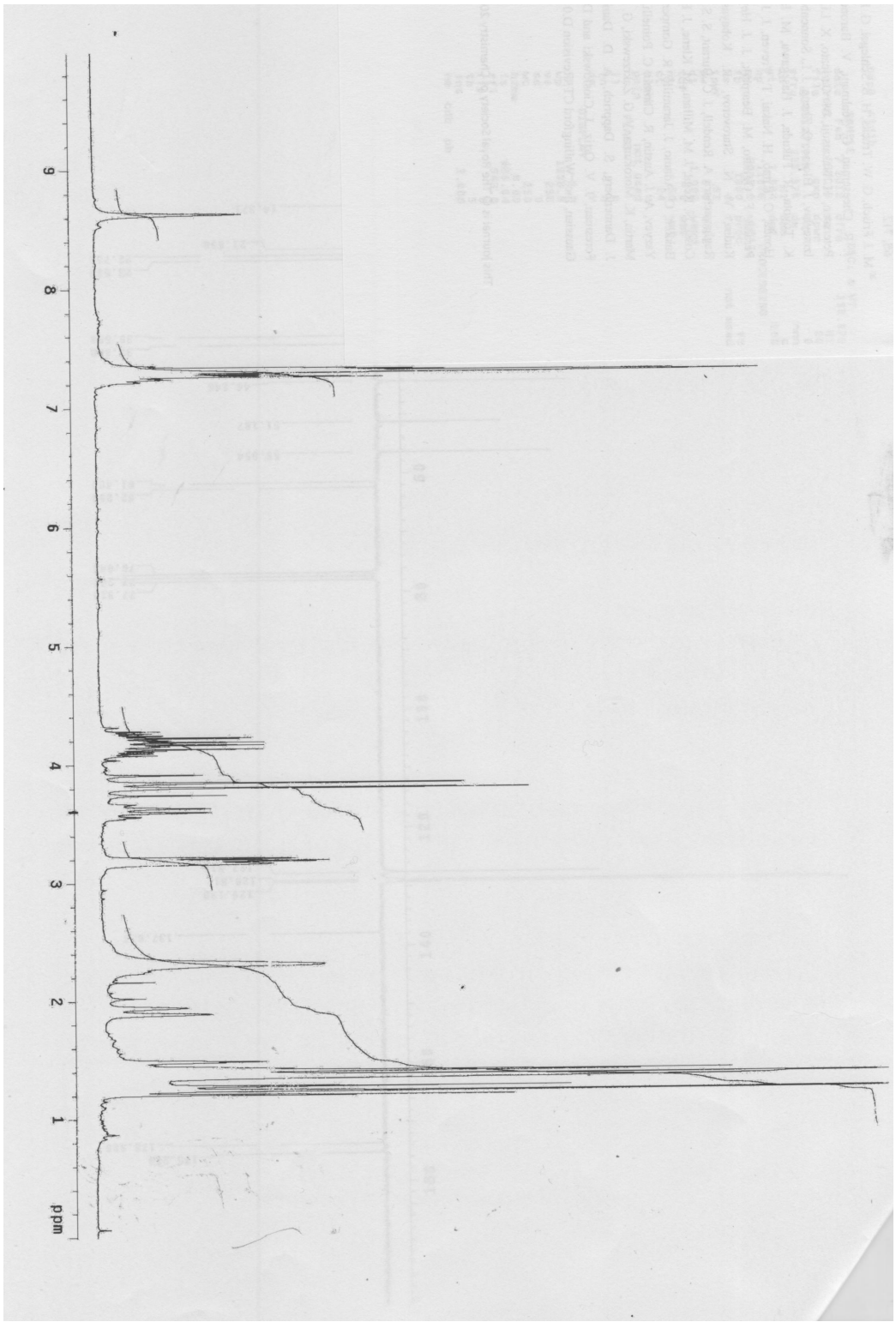


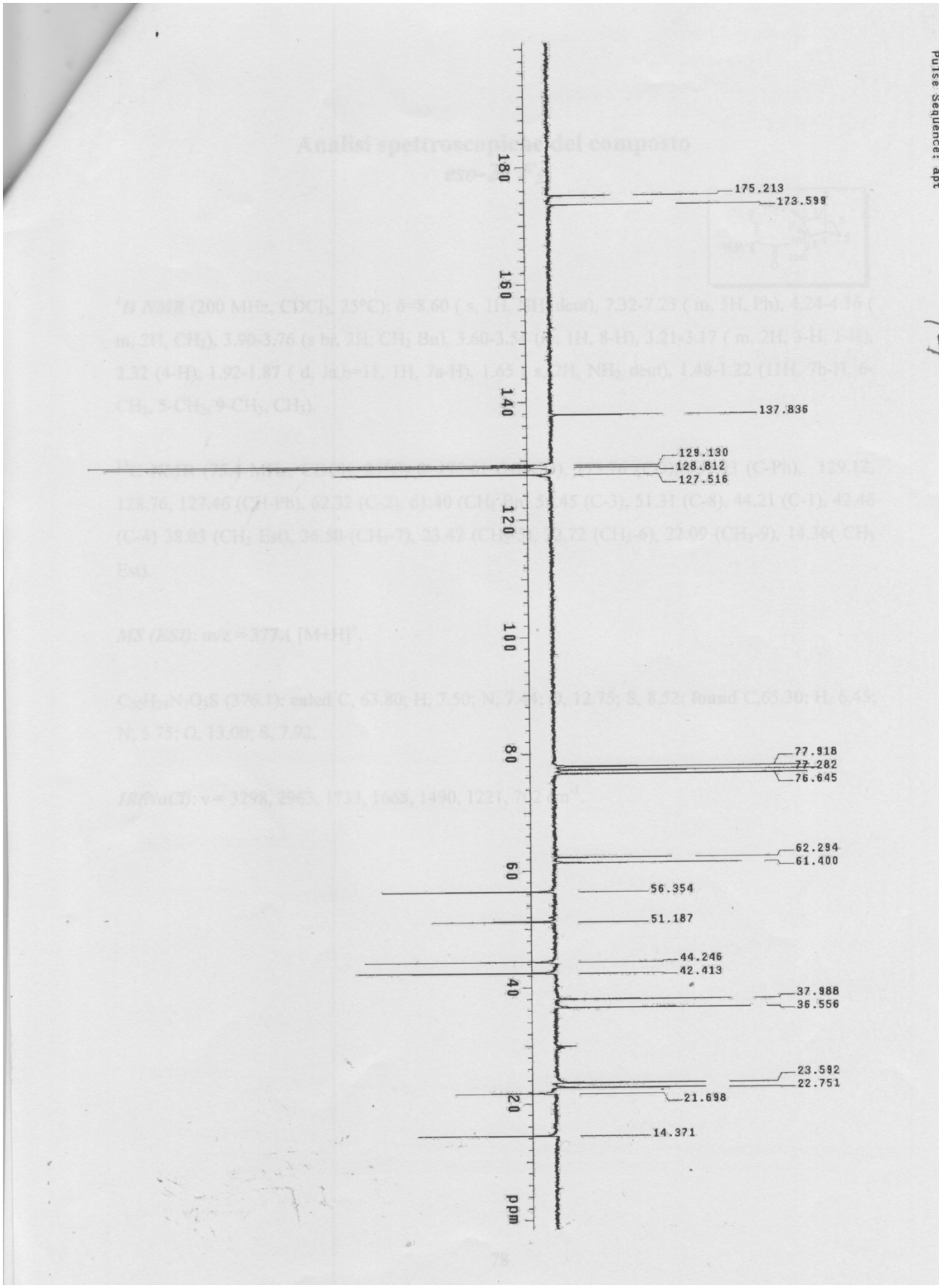

ppm
20
40
60
80
100
120
140
160
180
14.363
22.099
22.720
23.471
23.599
36.503
38.033
42.466
44.216
51.316
56.453
61.400
62.317
76.615
77.251
77.888
127.463
128.766
129.122
137.934
173.765
175.614

Compound 6b

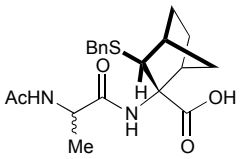


6b

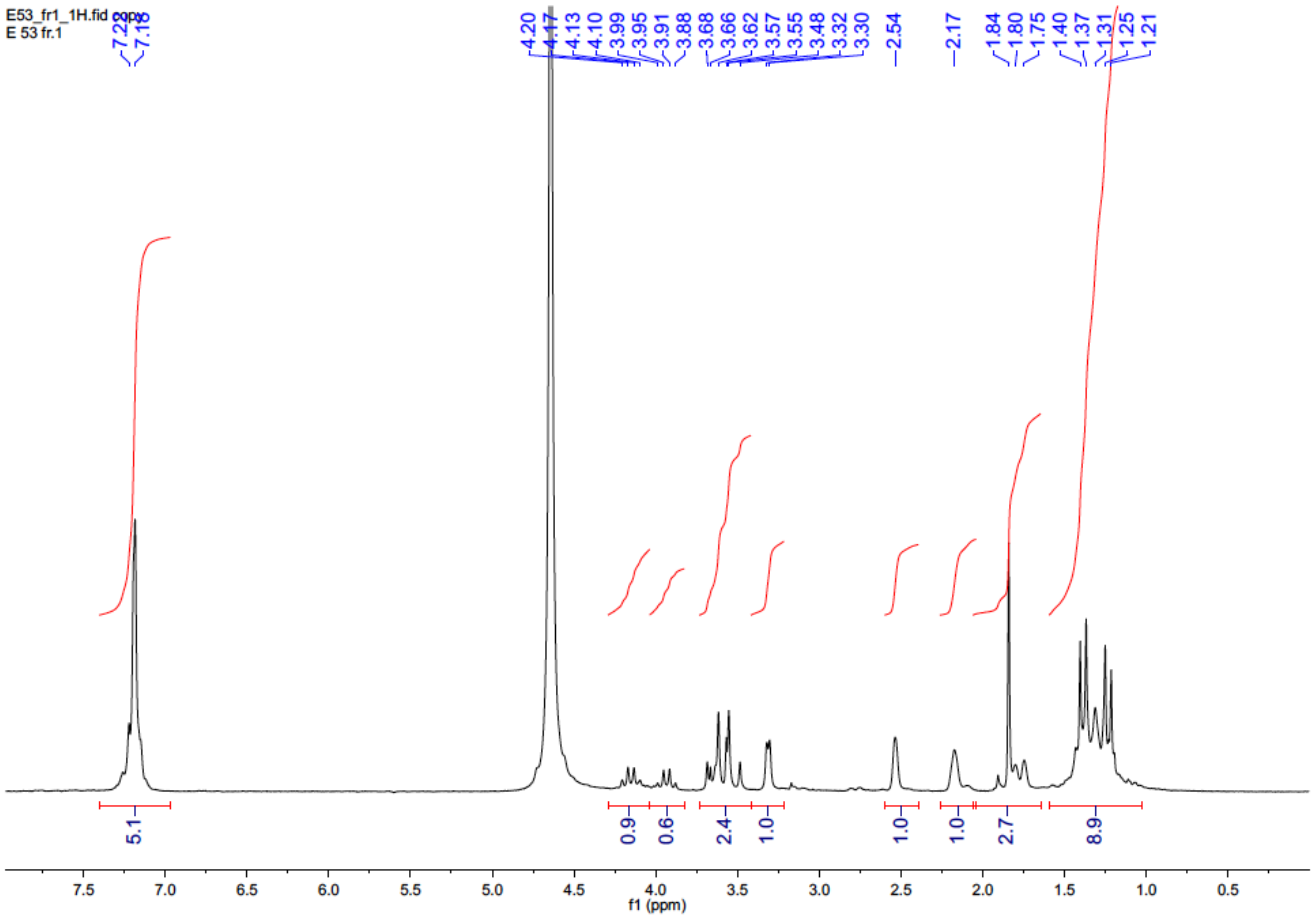



Pulse Sequence: apt
175.213
173.599
137.836
129.130
128.812
127.516
77.918
77.282
76.645
62.294
61.400
56.354
51.187
44.246
42.413
37.988
36.556
23.592
22.751
21.698
14.371
ppm

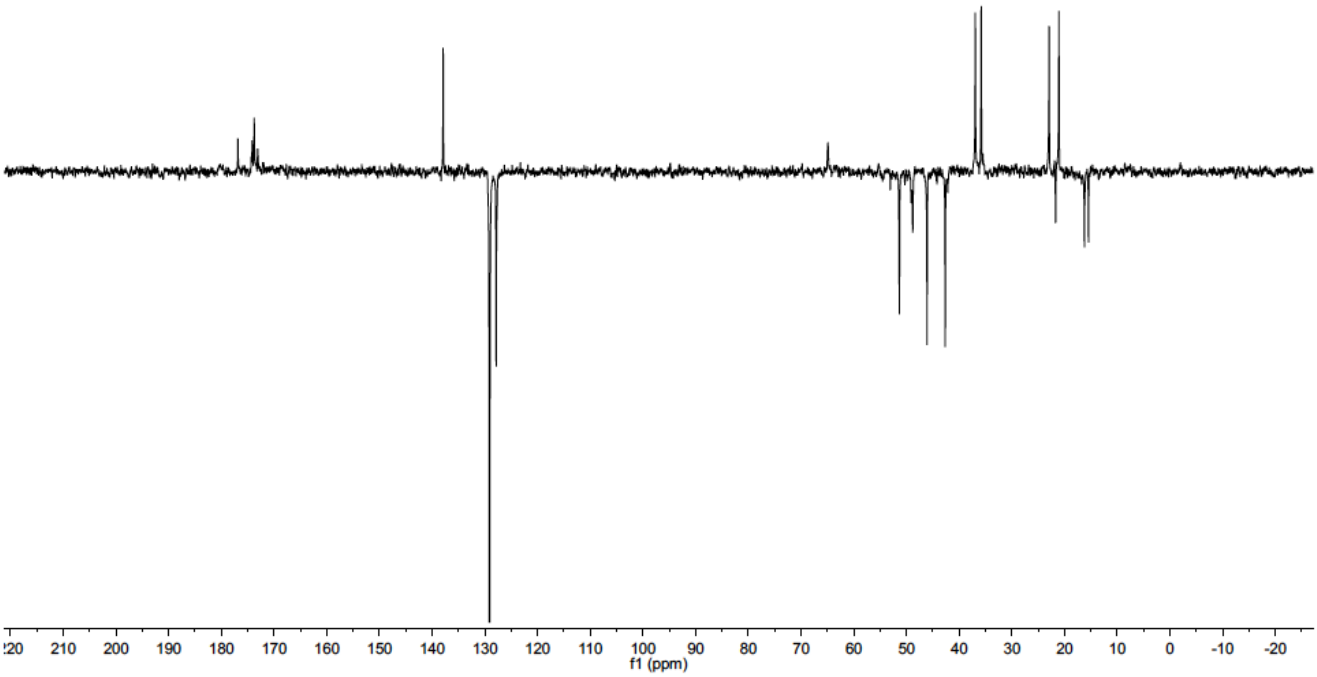
Compound 7a



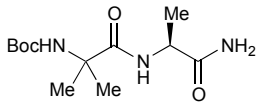
7a



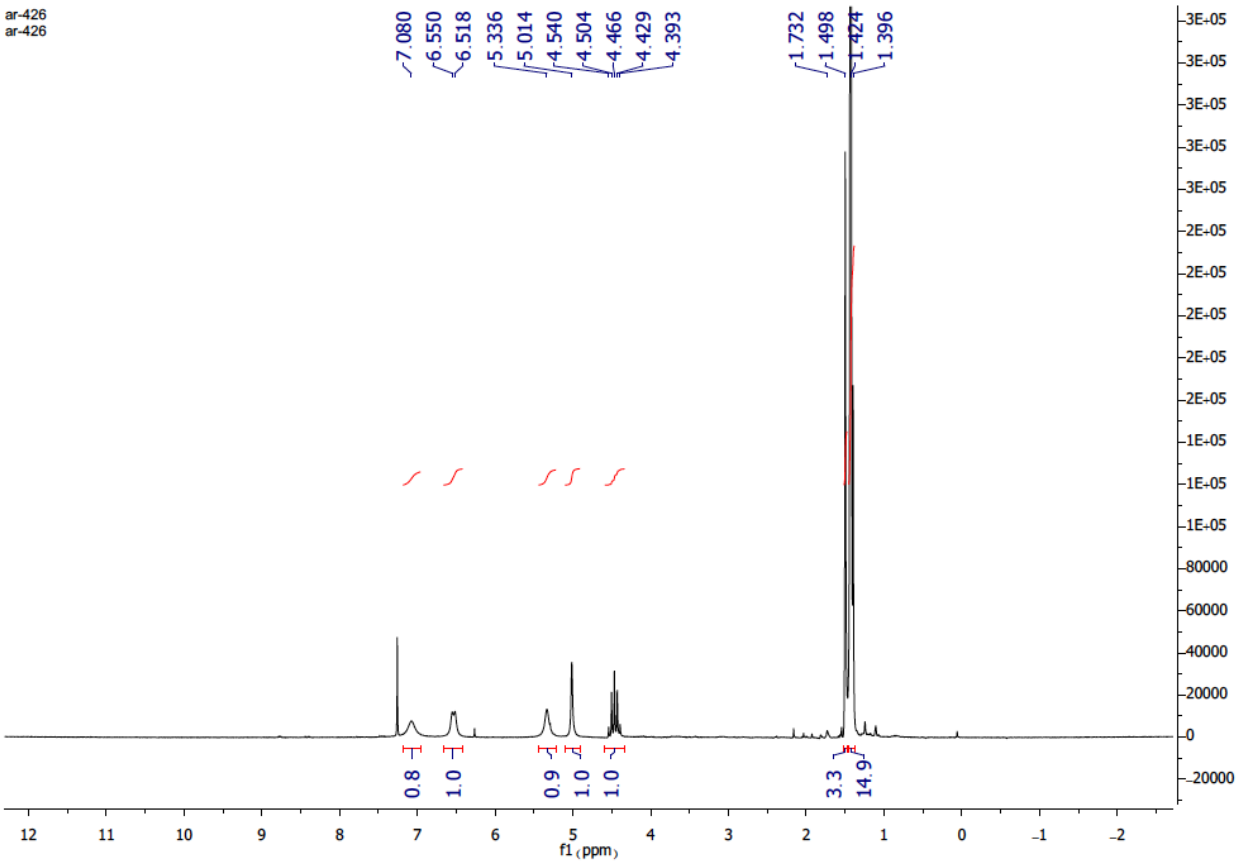
E53_fr1_13C.fid copy
E 53 fr.1

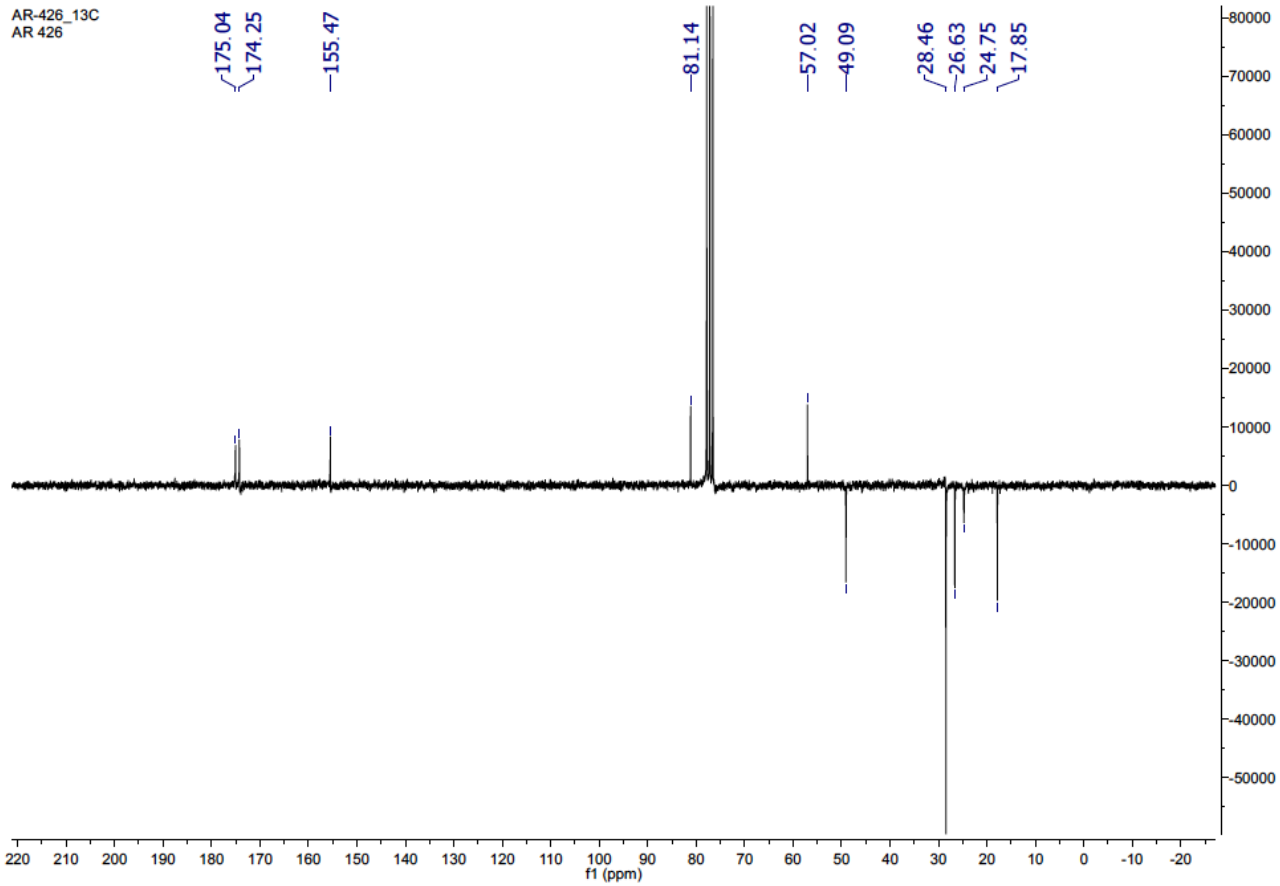


Compound 10

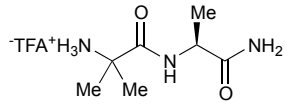


10

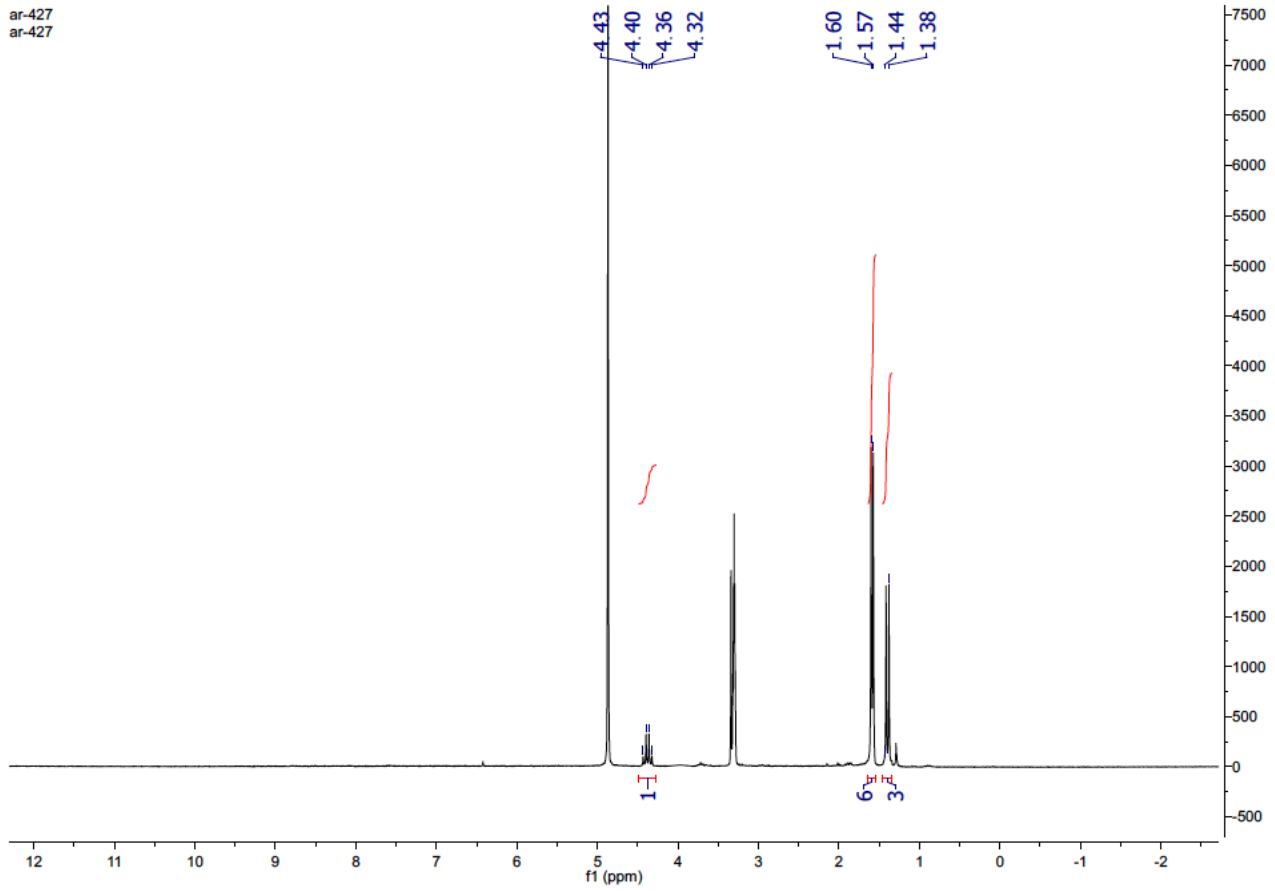


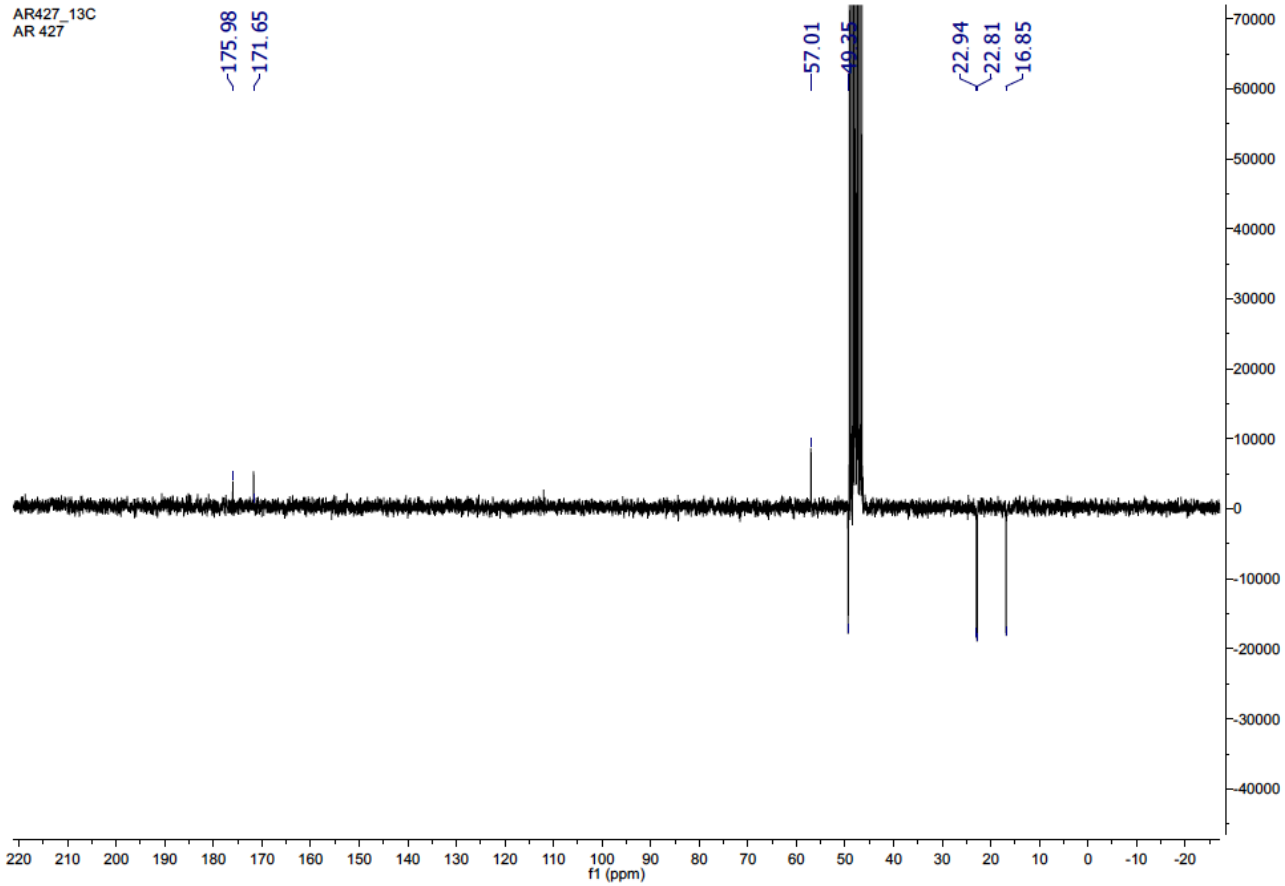

AR-426_13C
AR 426
175.04
174.25
155.47
81.14
57.02
49.09
28.46
26.63
24.75
17.85
f1 (ppm)

Compound 11

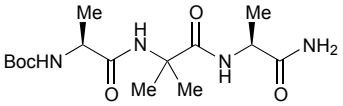


11

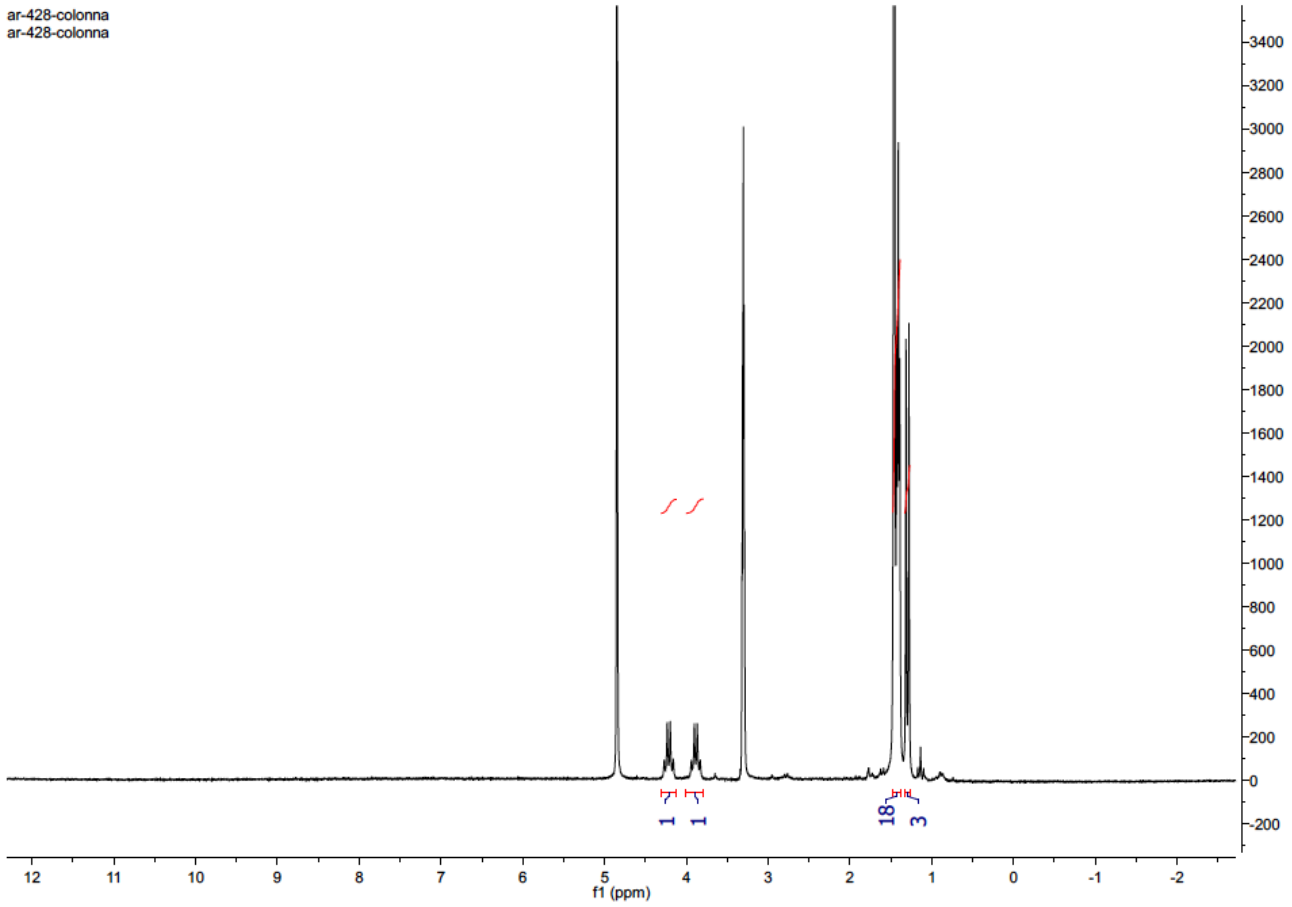


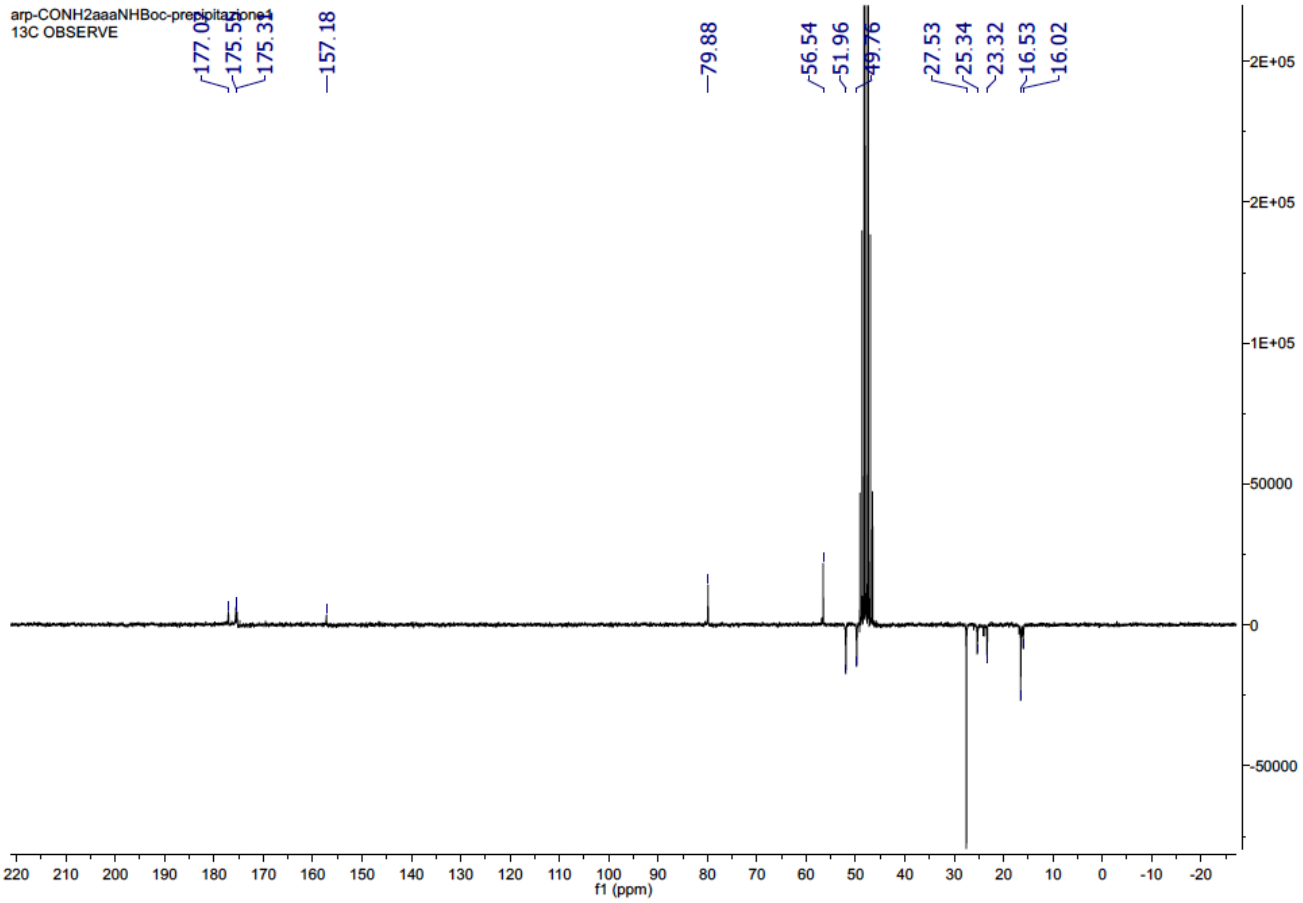

AR427_13C
AR 427
175.98
171.65
57.01
49.25
22.94
22.81
16.85
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
-30000
-40000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

Compound 13

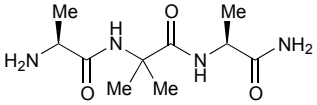


13

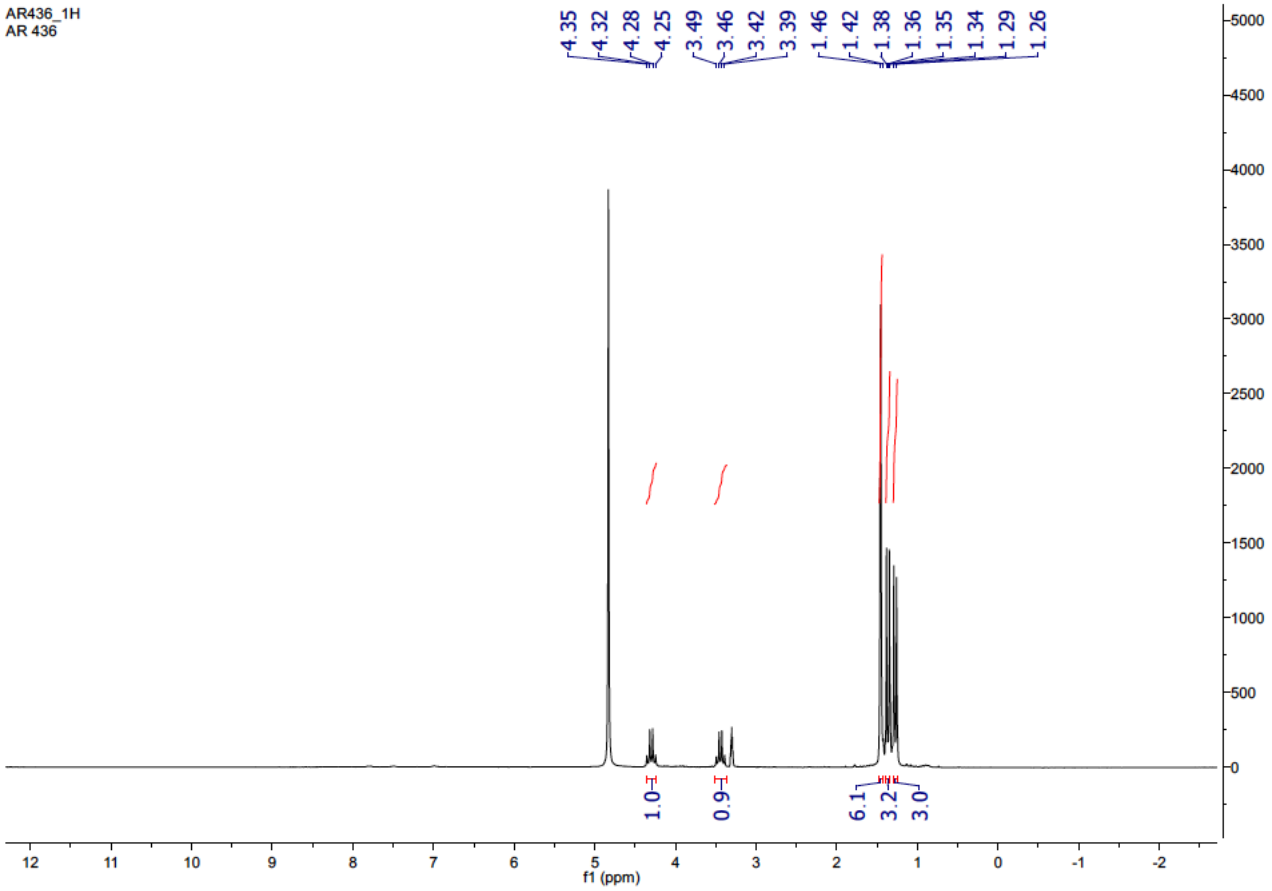


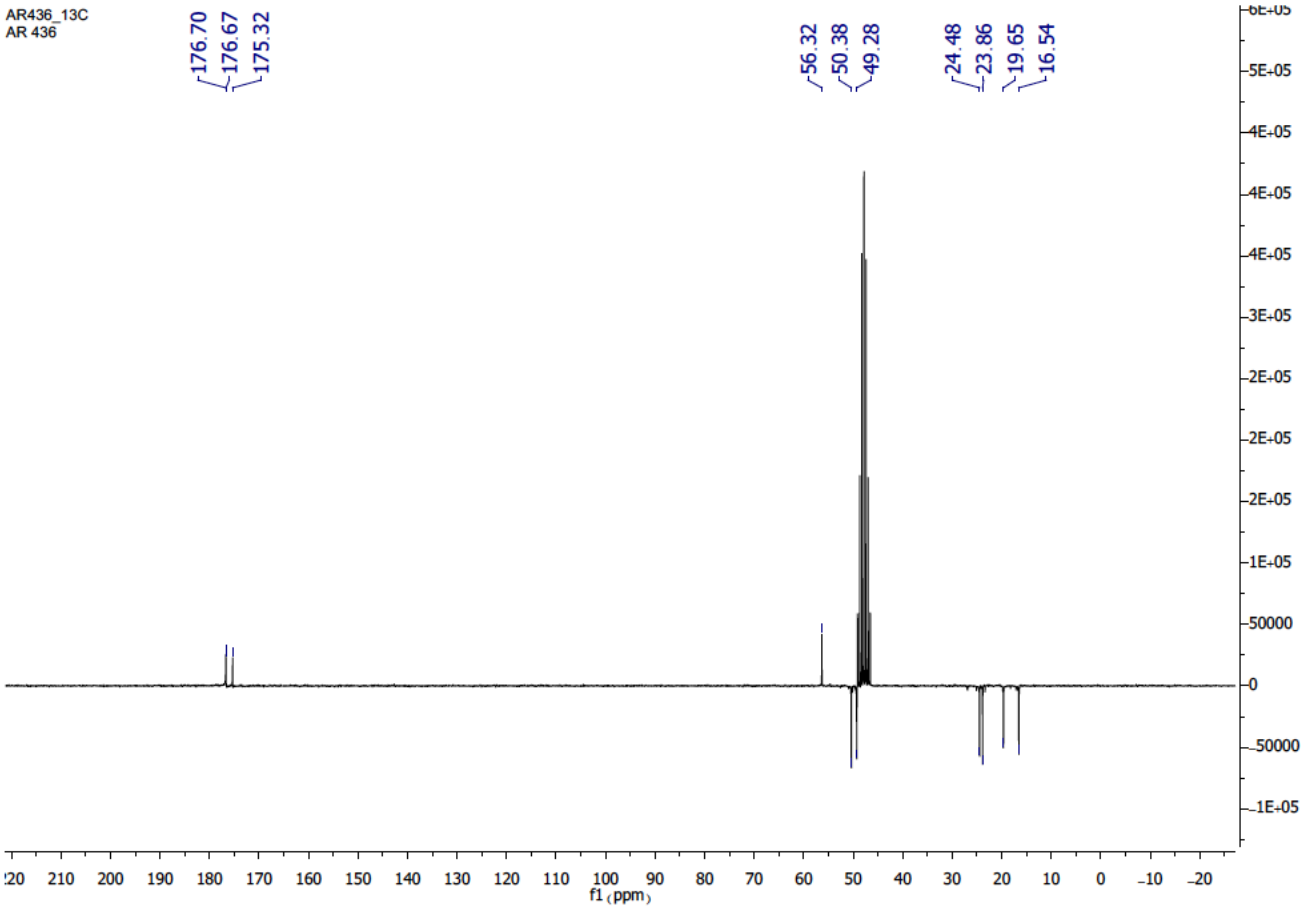

arp-CONH2aaaNHBoc-precipitazione1
13C OBSERVE
177.05
175.55
175.31
157.18
79.88
56.54
51.96
49.76
27.53
25.34
23.32
16.53
16.02
2E+05
2E+05
1E+05
50000
0
-50000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

Compound 4

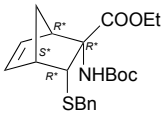

Me
H
N
O
Me
H2N
NH2
O
Me
Me
N
H
O

4

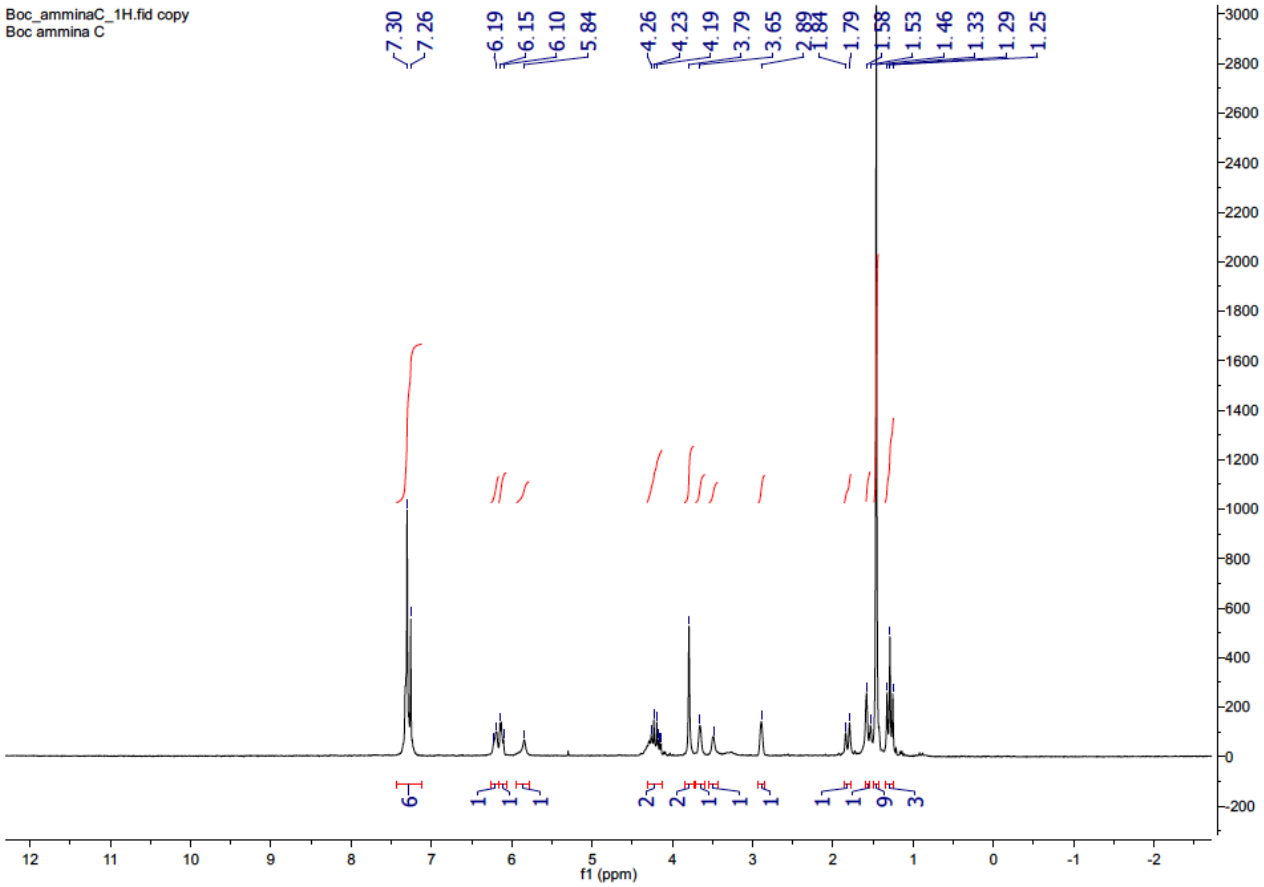

AR436_1H
AR 436
4.35
4.32
4.28
4.25
3.49
3.46
3.42
3.39
1.46
1.42
1.38
1.36
1.35
1.34
1.29
1.26
1.0
0.9
6.1
3.2
3.0
f1 (ppm)

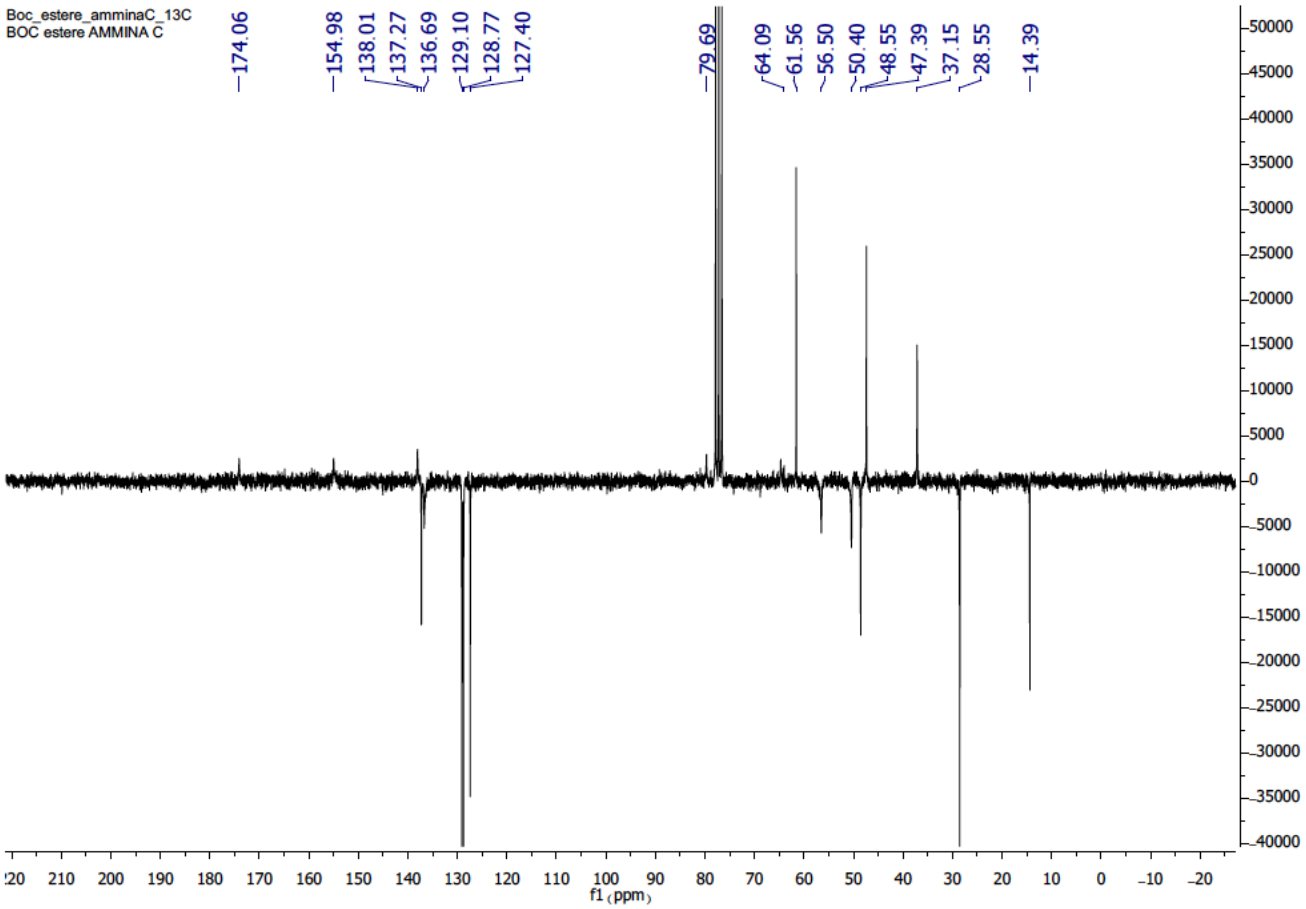

AR436_13C
AR 436
176.70
176.67
175.32
56.32
50.38
49.28
24.48
23.86
19.65
16.54
5E-05
4E-05
4E-05
4E-05
3E-05
2E-05
2E-05
2E-05
1E-05
50000
0
-50000
-1E-05
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

Compound 14

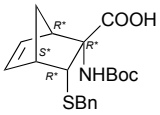


14

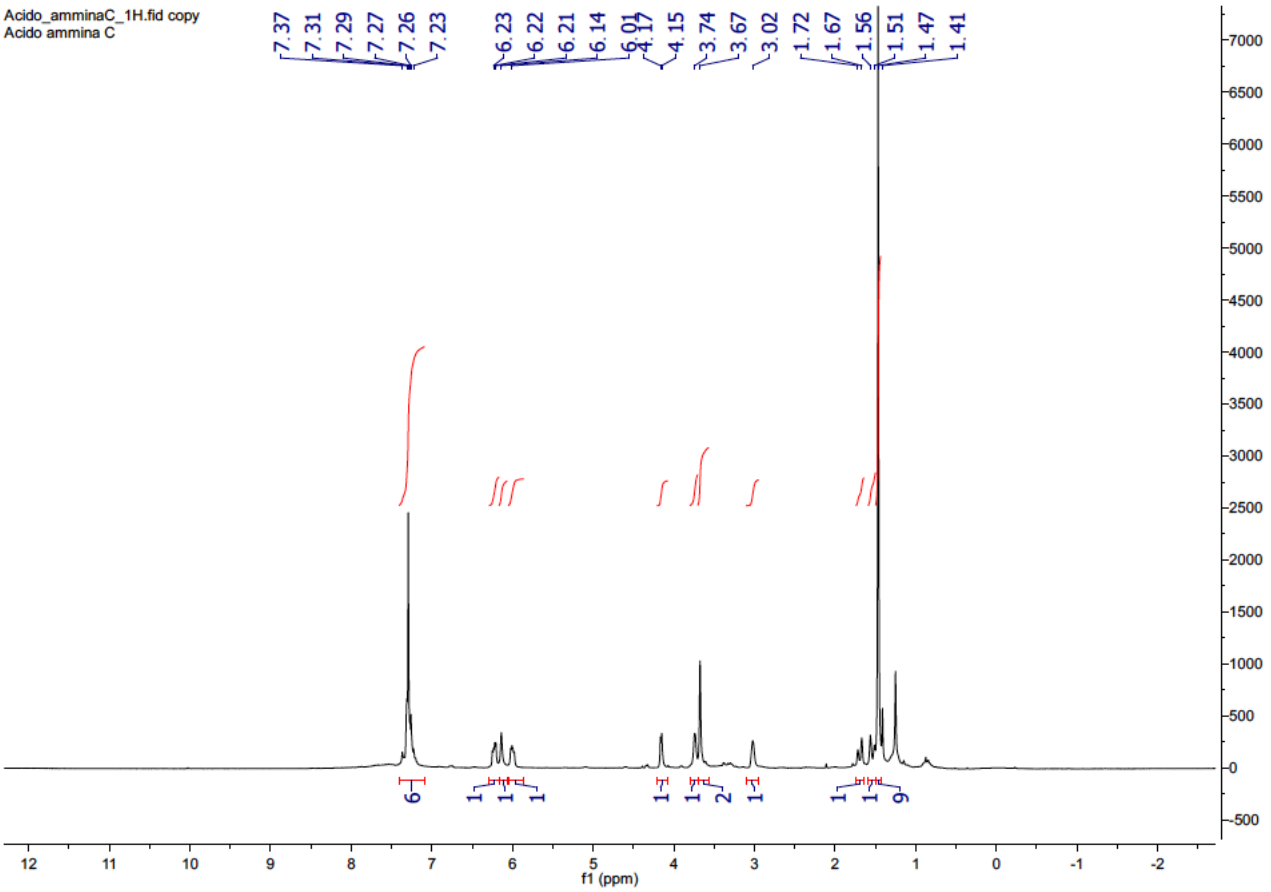


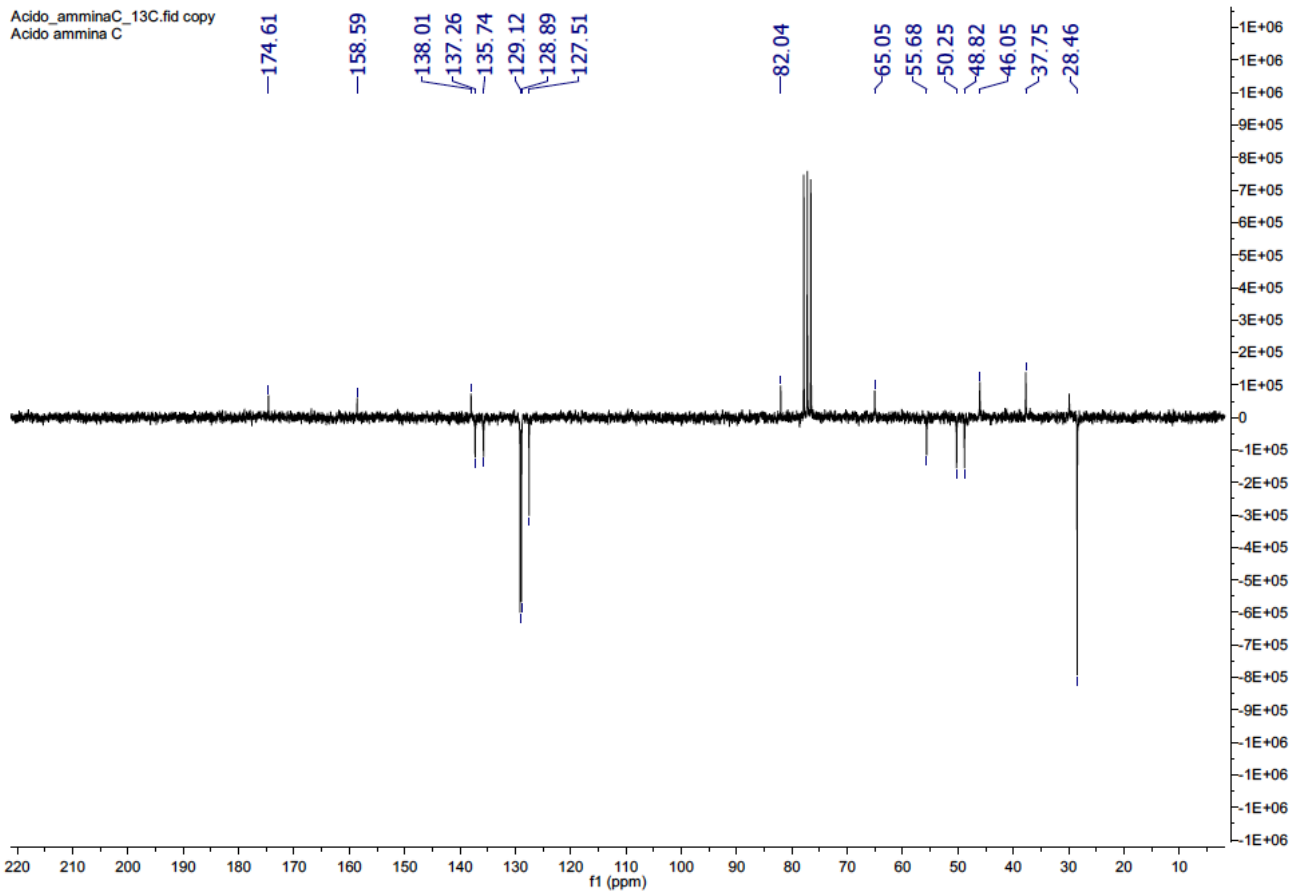

Boc_estere_amminaC_13C
BOC estere AMMINA C
174.06
154.98
138.01
137.27
136.69
129.10
128.77
127.40
79.69
64.09
61.56
56.50
50.40
48.55
47.39
37.15
28.55
14.39
f1 (ppm)

Compound 15

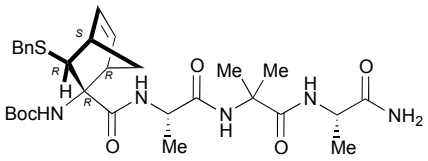


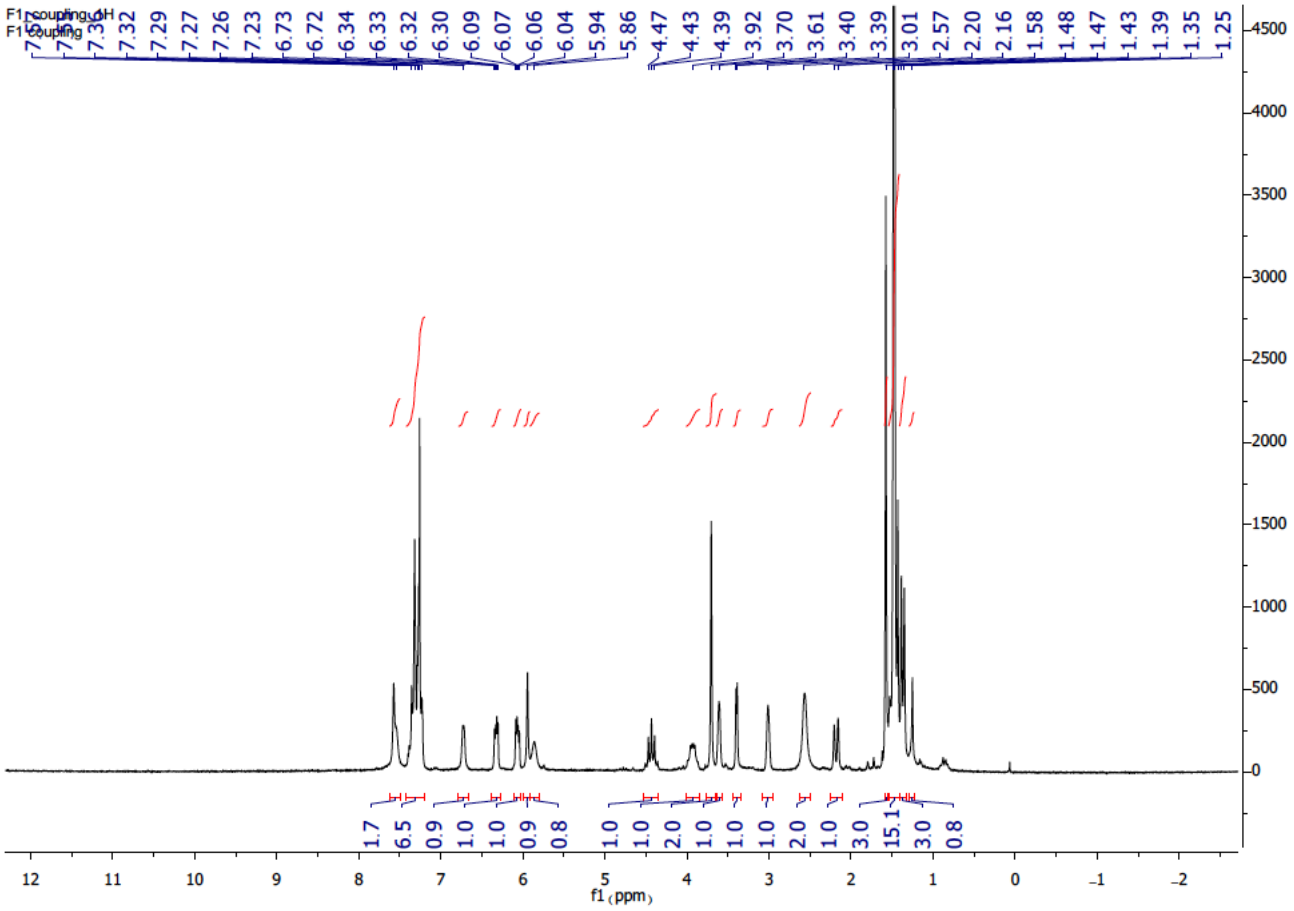
15

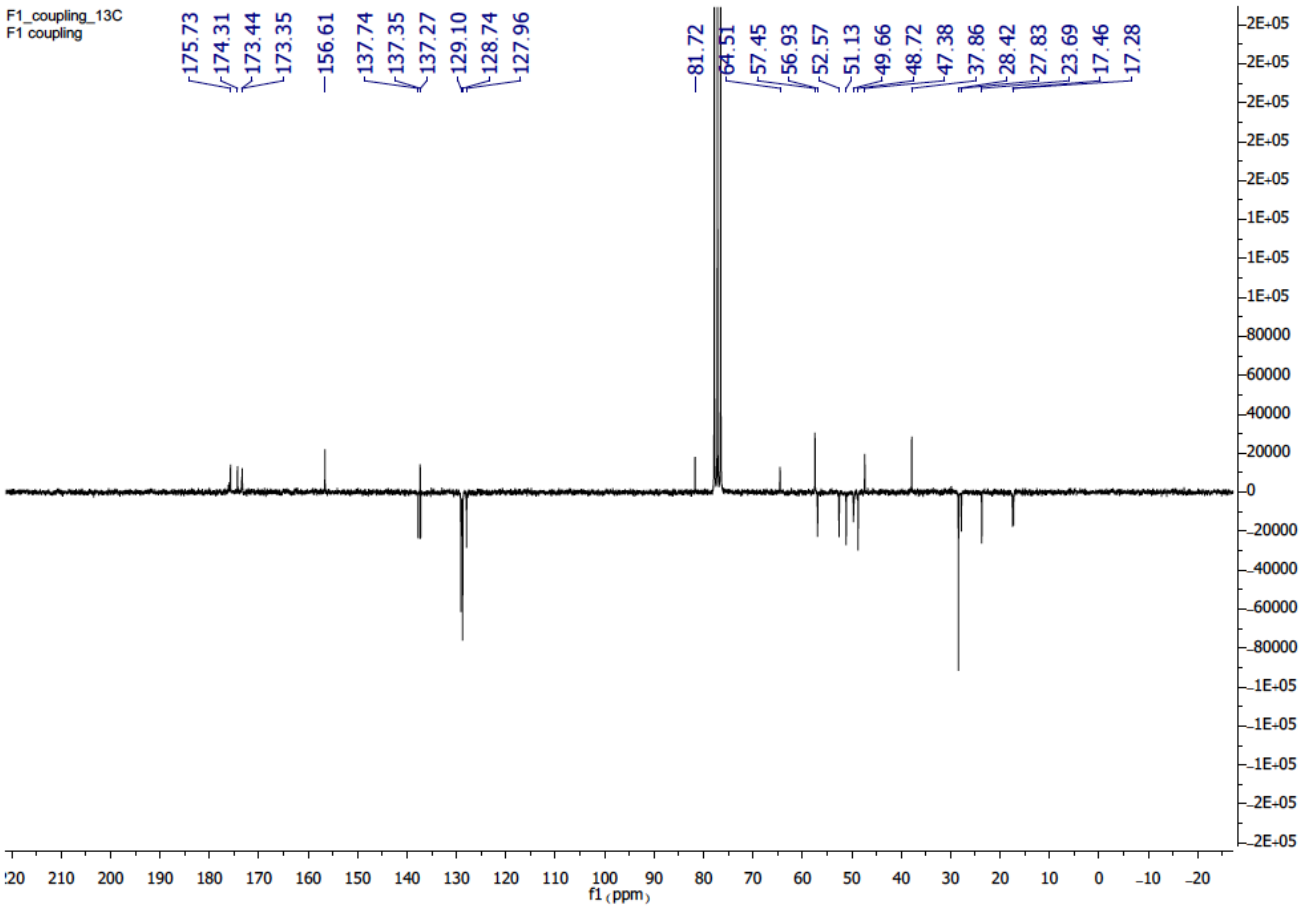



Acido_amminaC_13C.fid copy
Acido ammina C
174.61
158.59
138.01
137.26
135.74
129.12
128.89
127.51
82.04
65.05
55.68
50.25
48.82
46.05
37.75
28.46
f1 (ppm)

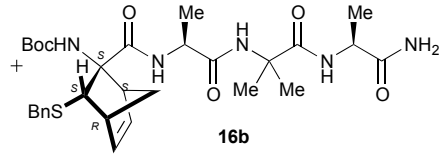
Compound 16a



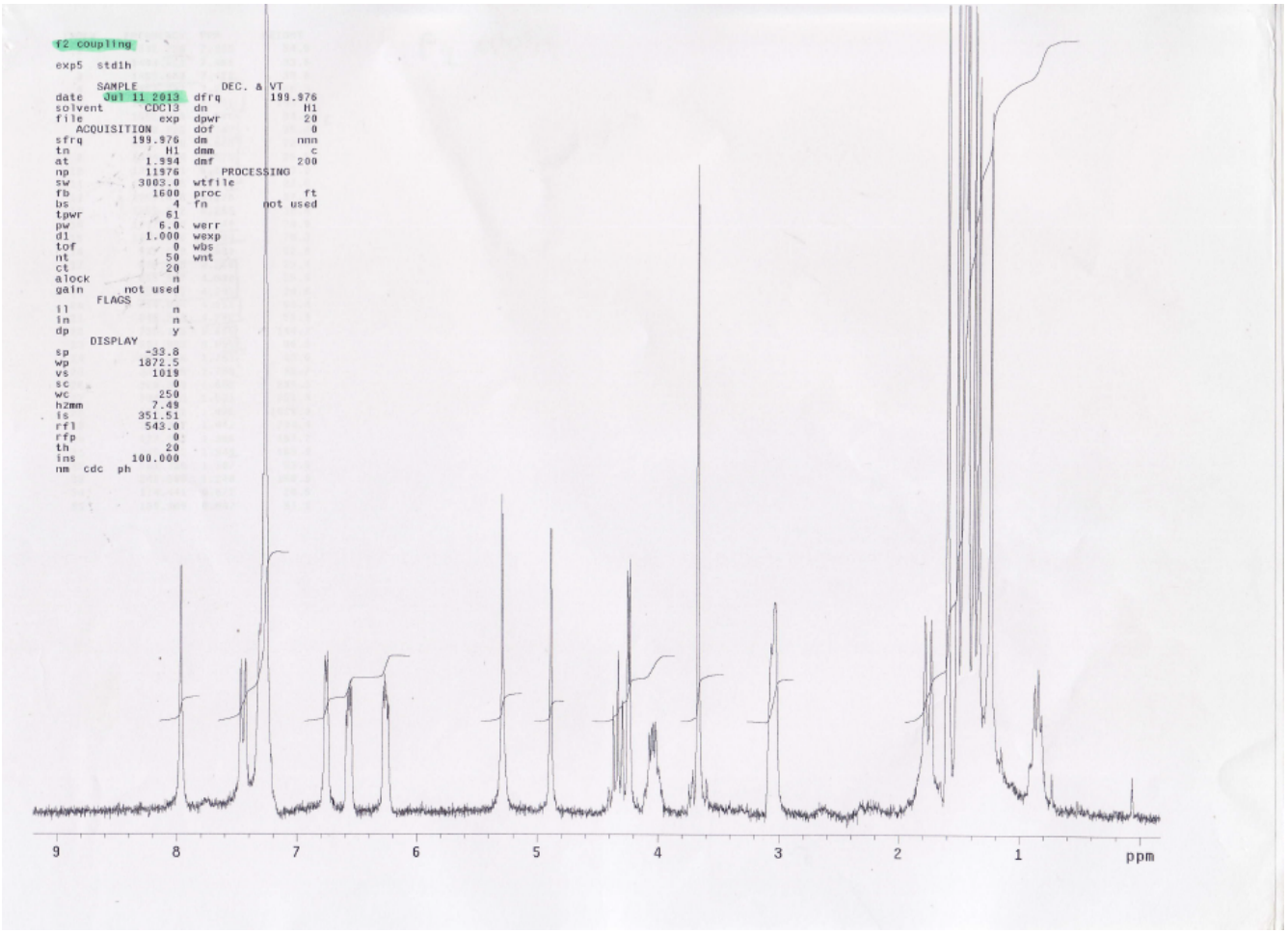


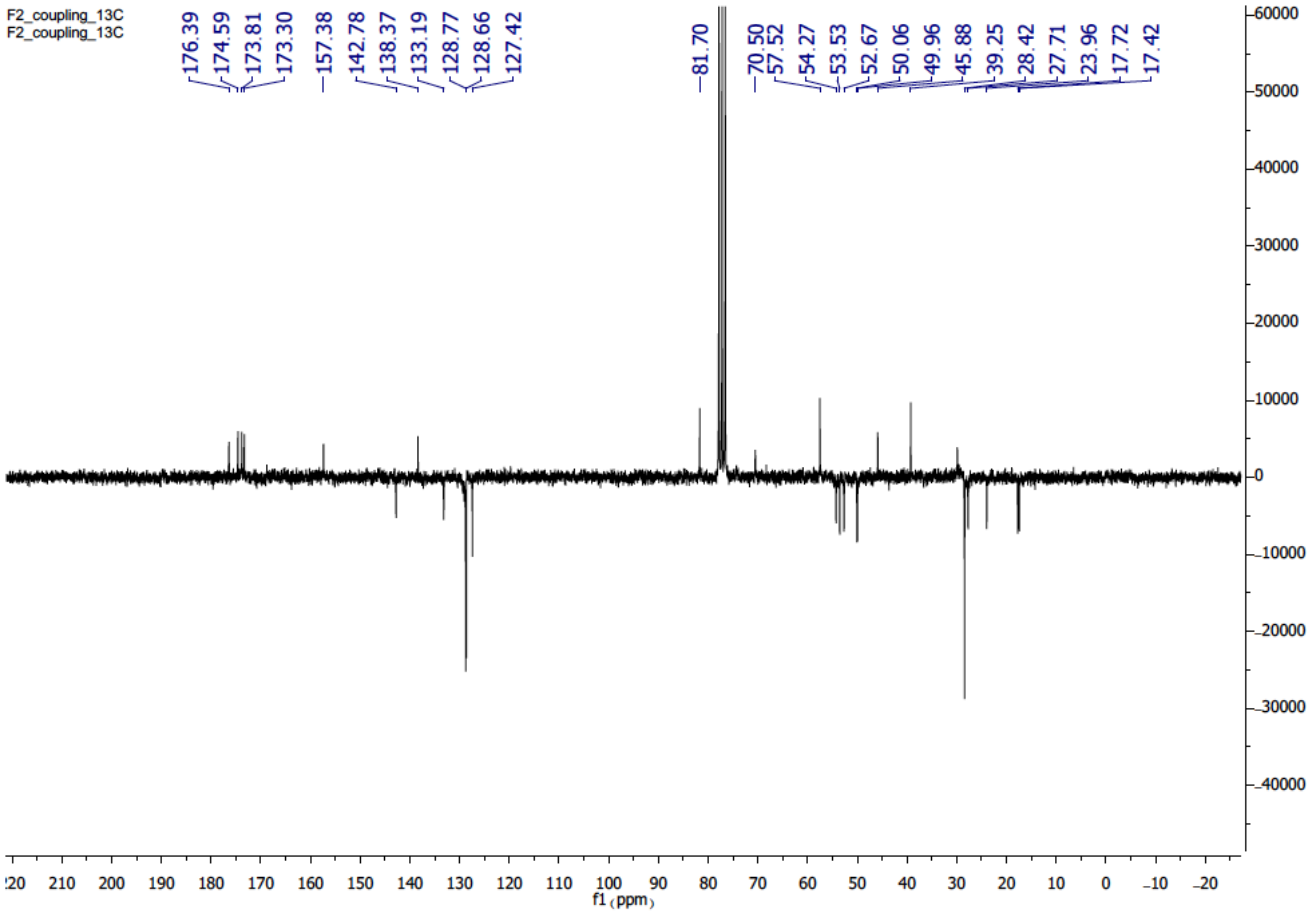

F1_coupling_13C
F1 coupling
175.73
174.31
173.44
173.35
156.61
137.74
137.35
137.27
129.10
128.74
127.96
81.72
64.51
57.45
56.93
52.57
51.13
49.66
48.72
47.38
37.86
28.42
27.83
23.69
17.46
17.28
f1 (ppm)

Compound 16b

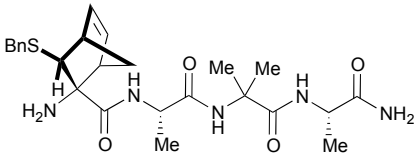


16b

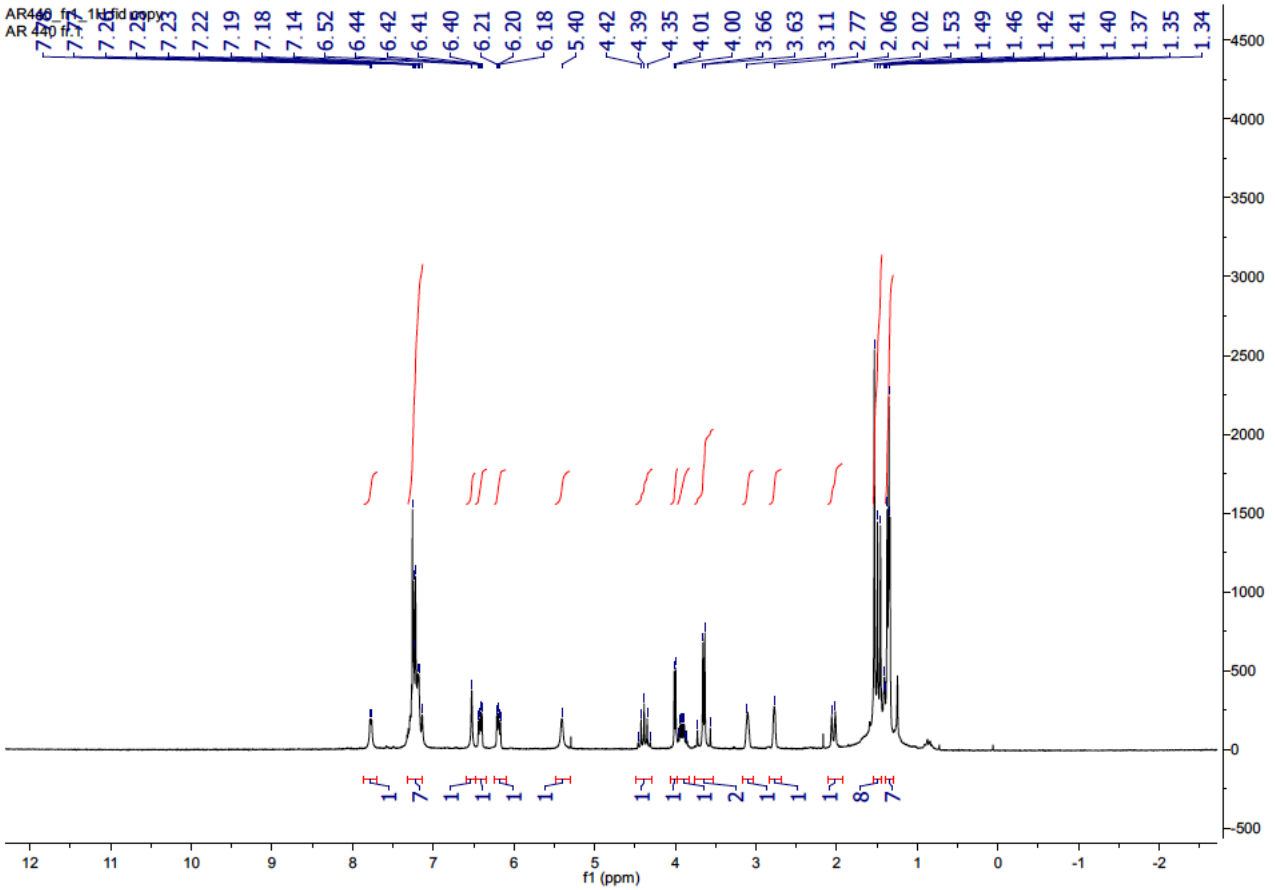


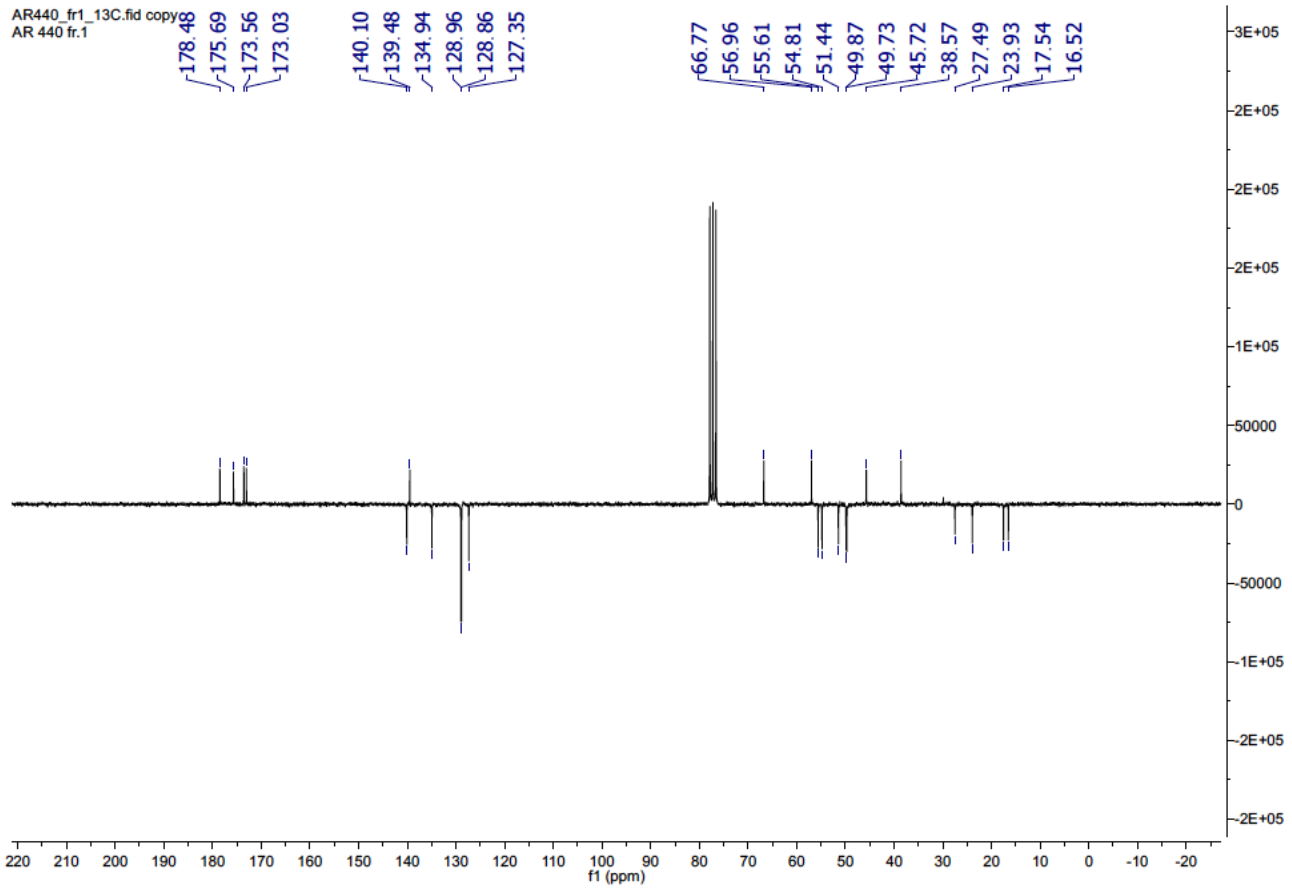

F2_coupling_13C
F2_coupling_13C
176.39
174.59
173.81
173.30
157.38
142.78
138.37
133.19
128.77
128.66
127.42
81.70
70.50
57.52
54.27
53.53
52.67
50.06
49.96
45.88
39.25
28.42
27.71
23.96
17.72
17.42
60000
50000
40000
30000
20000
10000
0
-10000
-20000
-30000
-40000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

Compound 17a

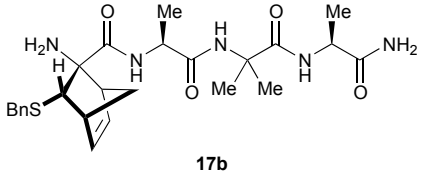


17a

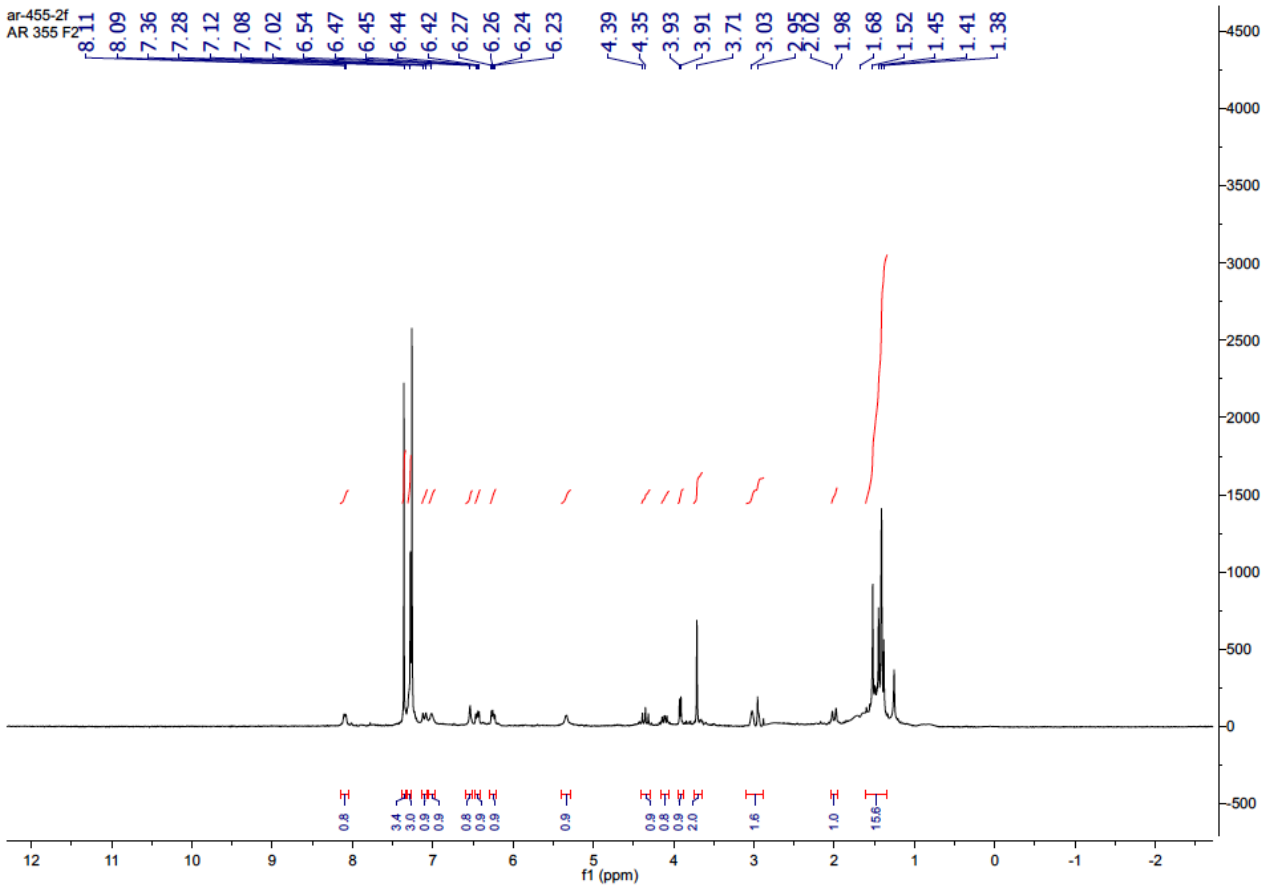


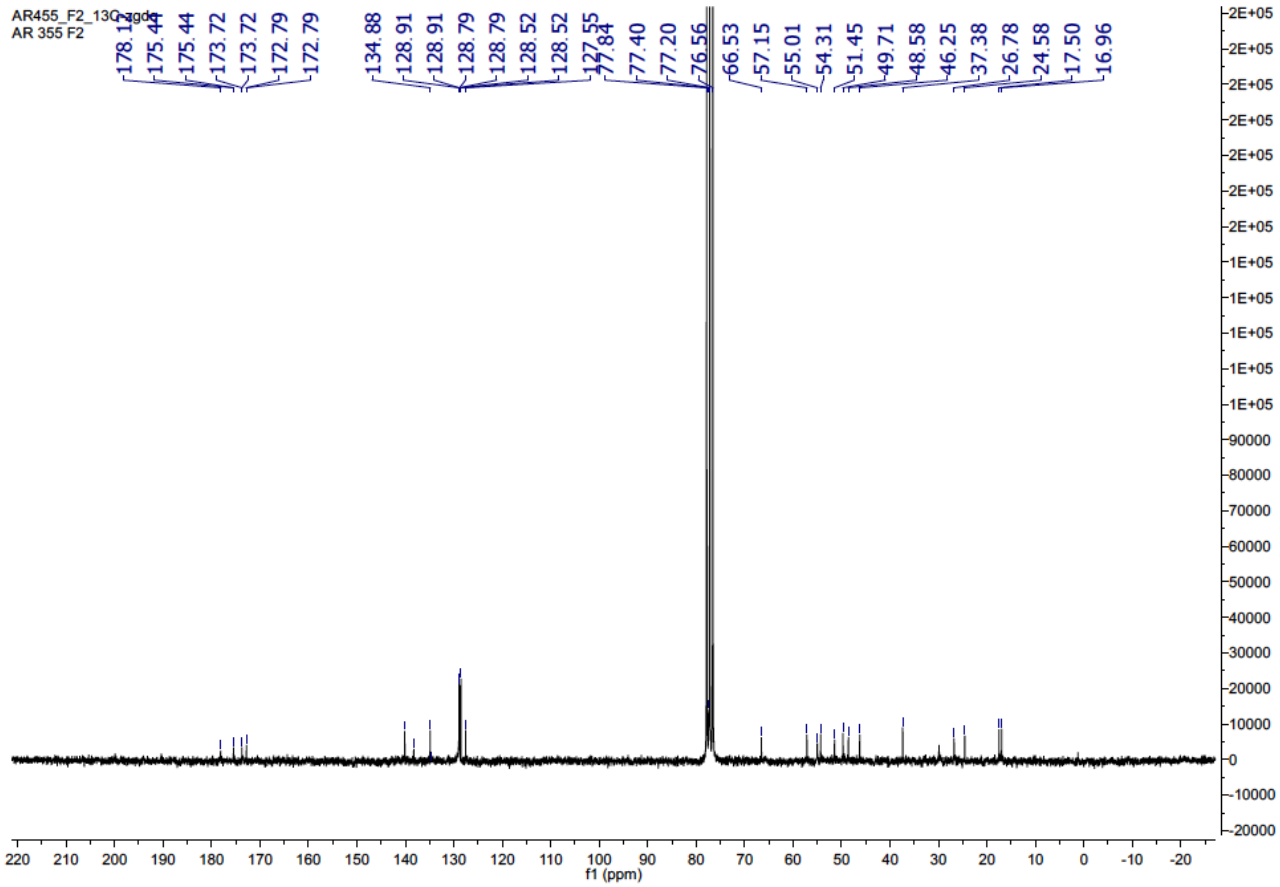

AR440_fr1_13C.fid copy
AR 440 fr.1
178.48
175.69
173.56
173.03
140.10
139.48
134.94
128.96
128.86
127.35
66.77
56.96
55.61
54.81
51.44
49.87
49.73
45.72
38.57
27.49
23.93
17.54
16.52
3E+05
2E+05
2E+05
2E+05
1E+05
50000
0
-50000
-1E+05
-2E+05
-2E+05
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

Compound 17b

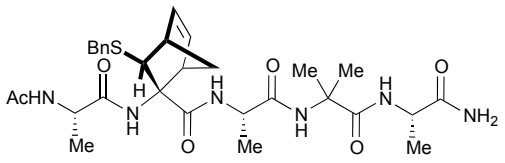


17b

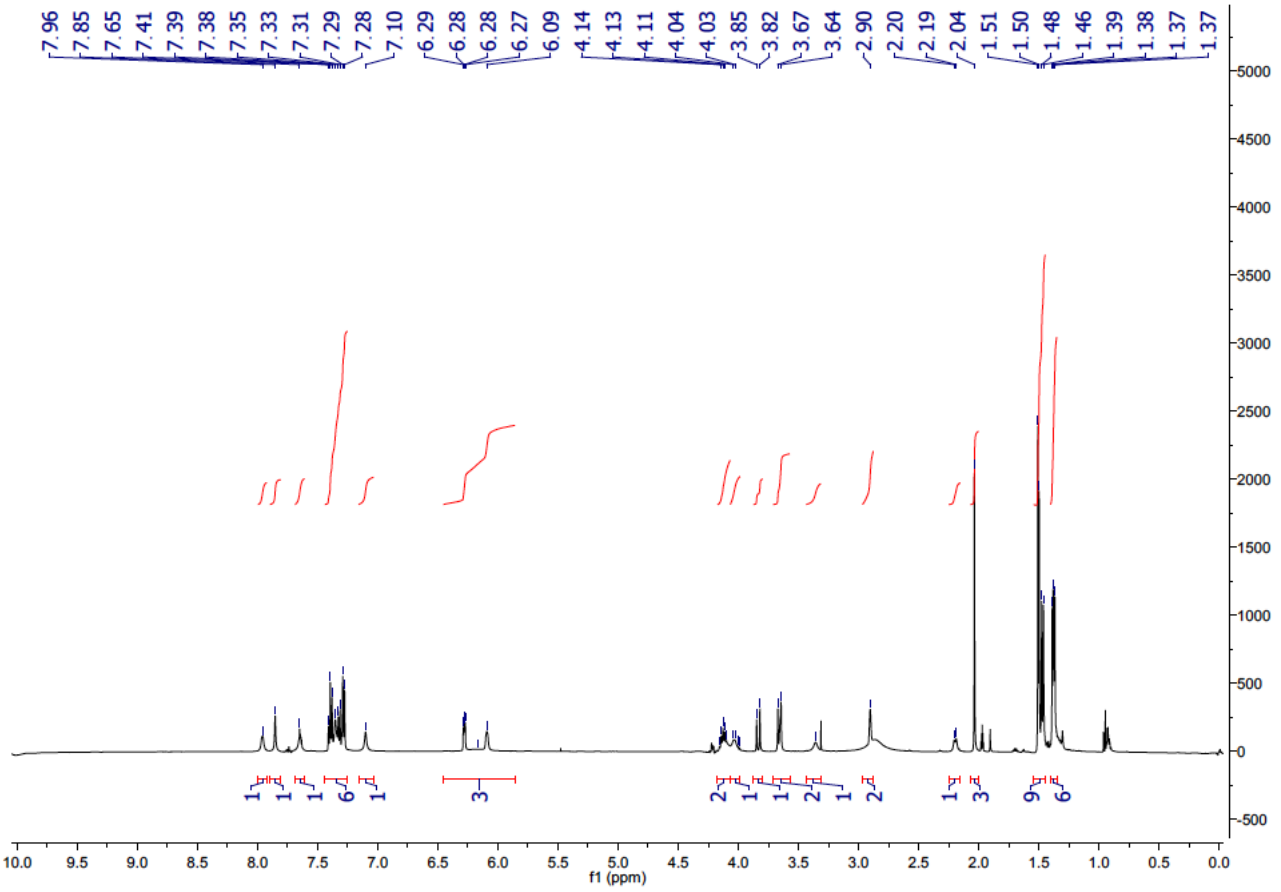



AR455_F2_13C-zgdc
AR 355 F2
178.12
175.44
175.44
173.72
173.72
172.79
172.79
134.88
128.91
128.91
128.79
128.79
128.52
128.52
127.55
77.84
77.40
77.20
76.56
66.53
57.15
55.01
54.31
51.45
49.71
48.58
46.25
37.38
26.78
24.58
17.50
16.96
f1 (ppm)

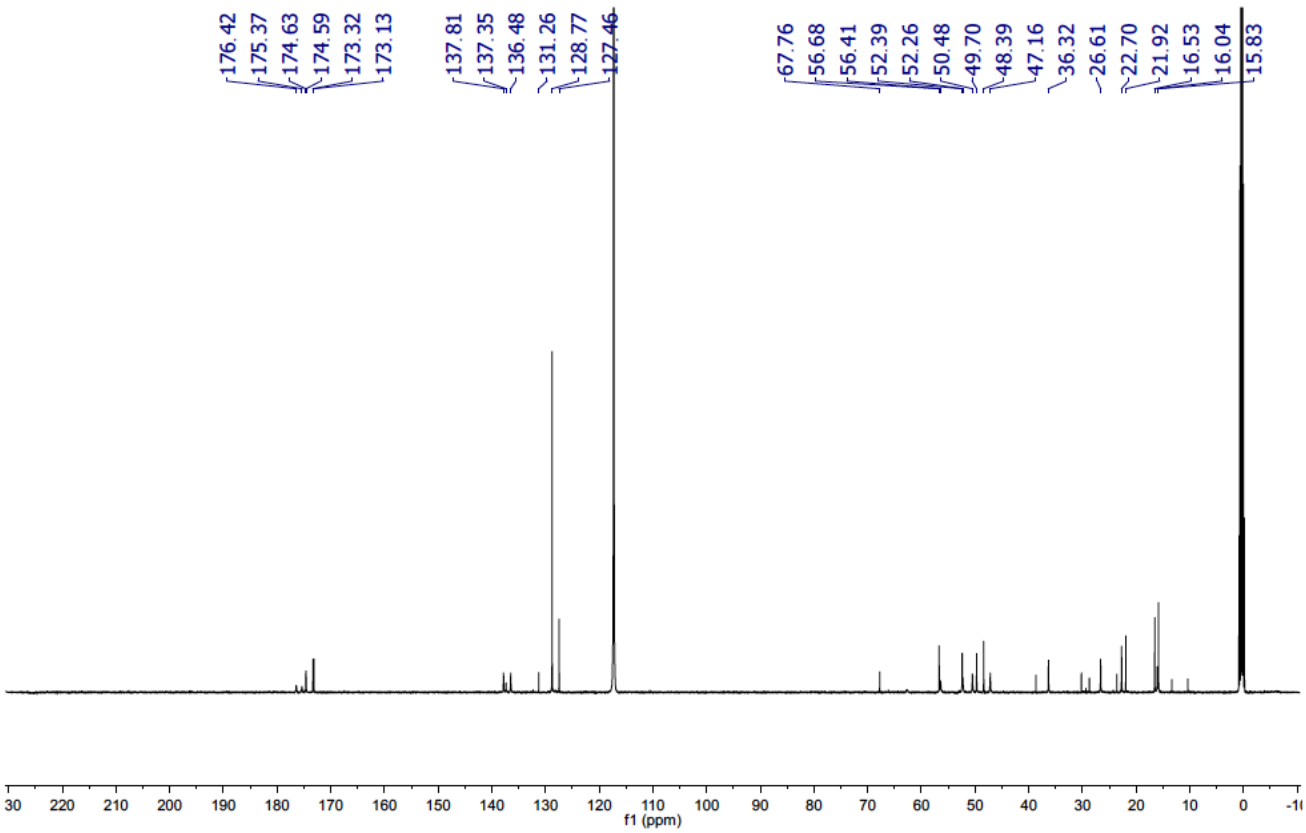
Compound 2a



2a

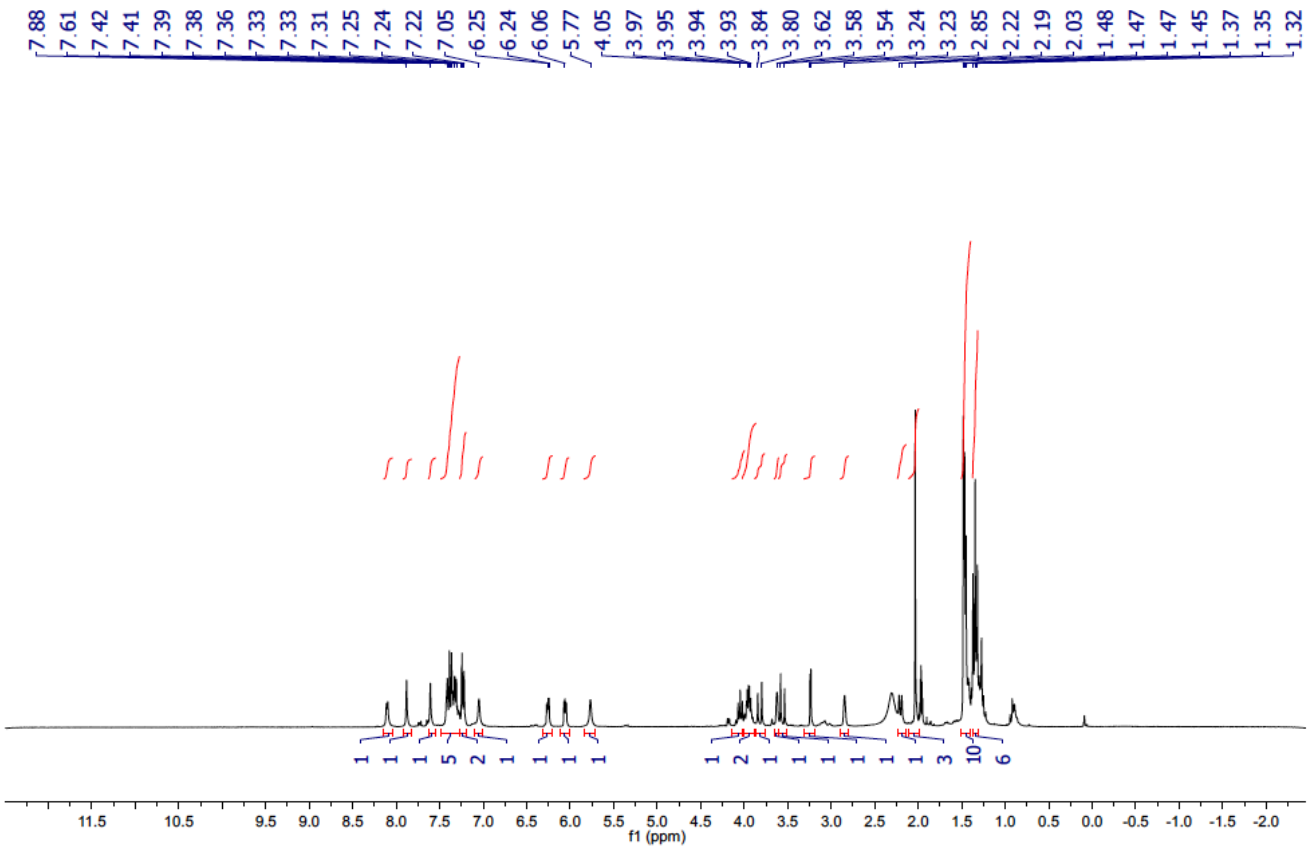


AR 441 F1
13C-zgdc in CD3CN a T=300K

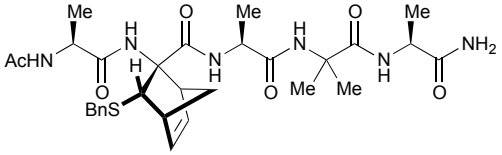


Compound 2a (0.08 mM)

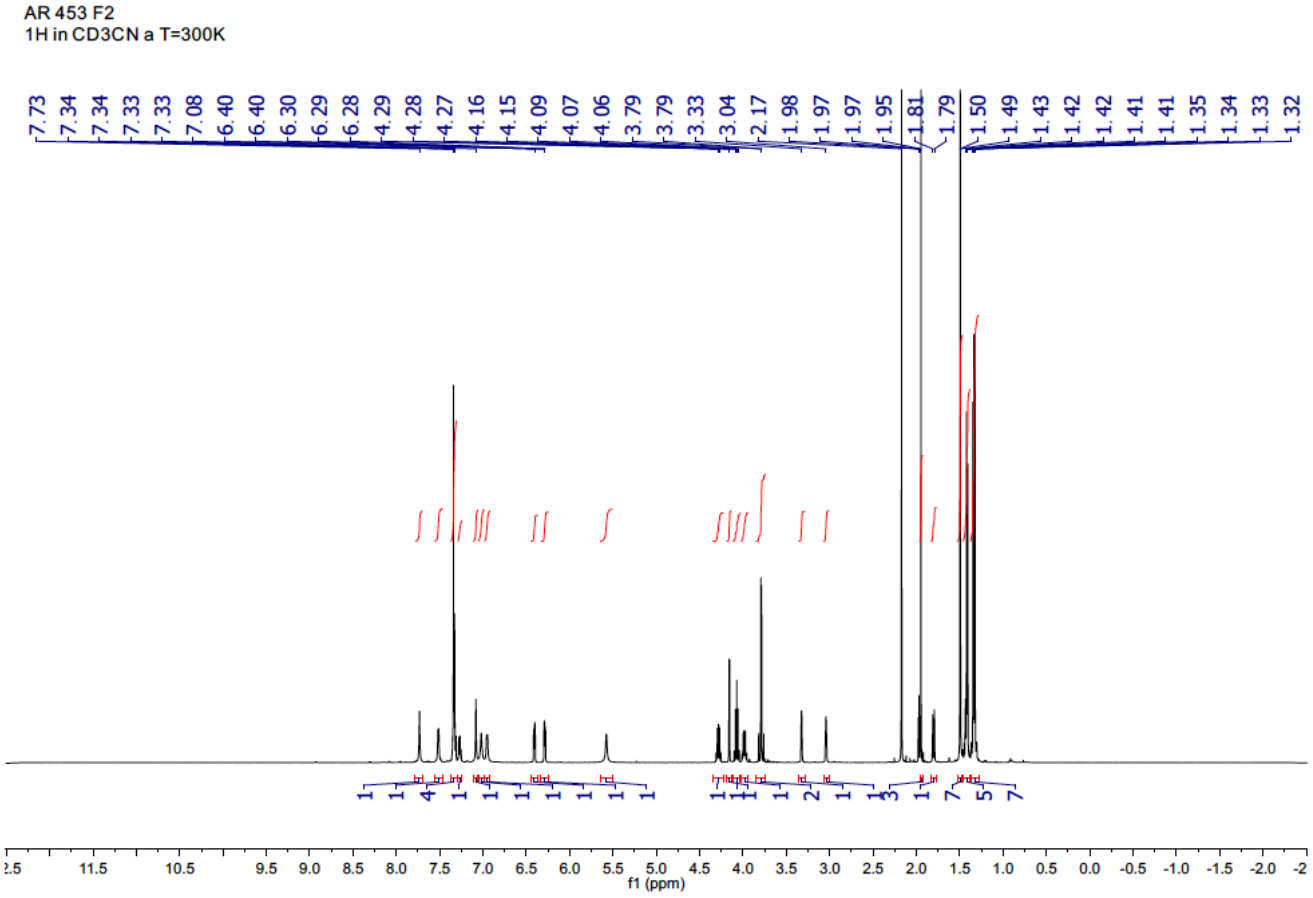
Acquisition in 0.08mM concentration use in VT-NMR and $DMSO_{d6}$ titration present different chemical shifts for amide protons and is reported below



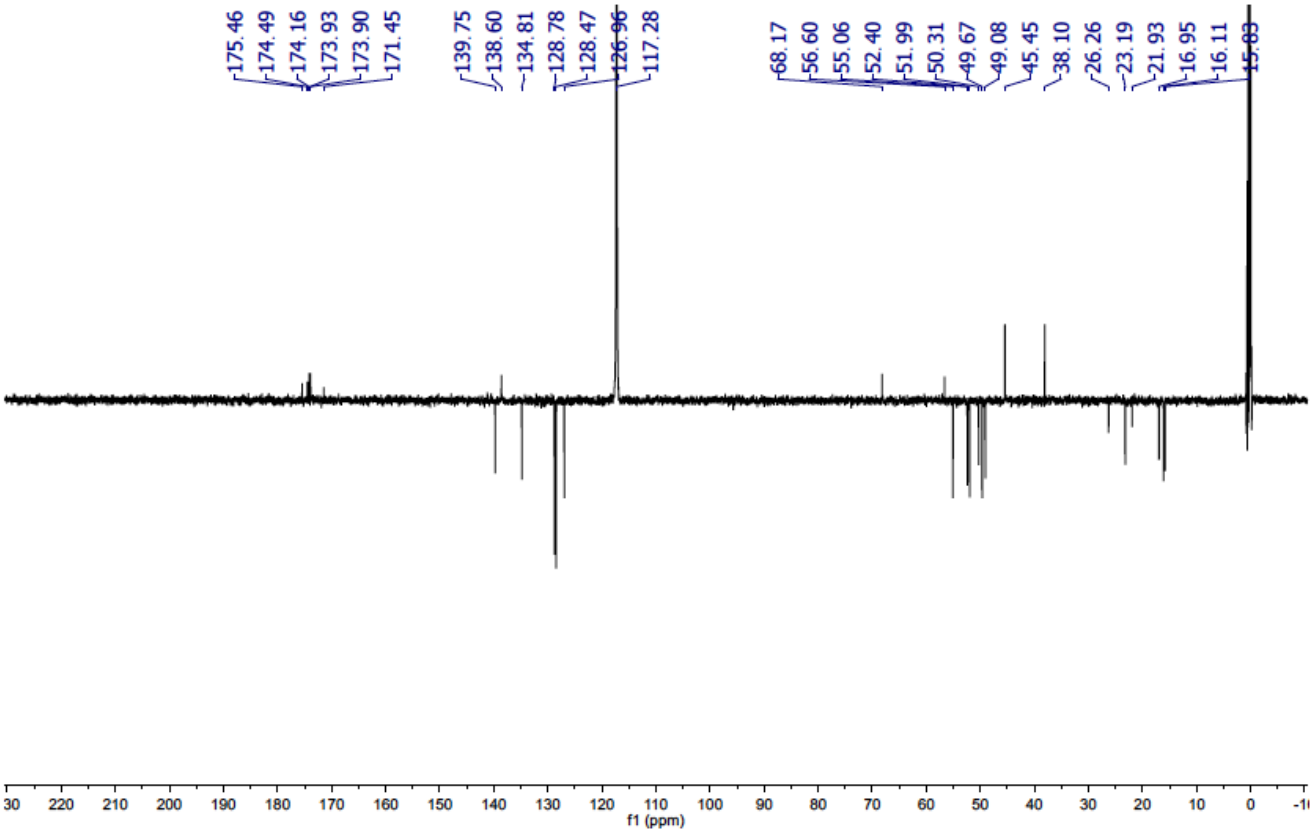
Compound 2b



2b



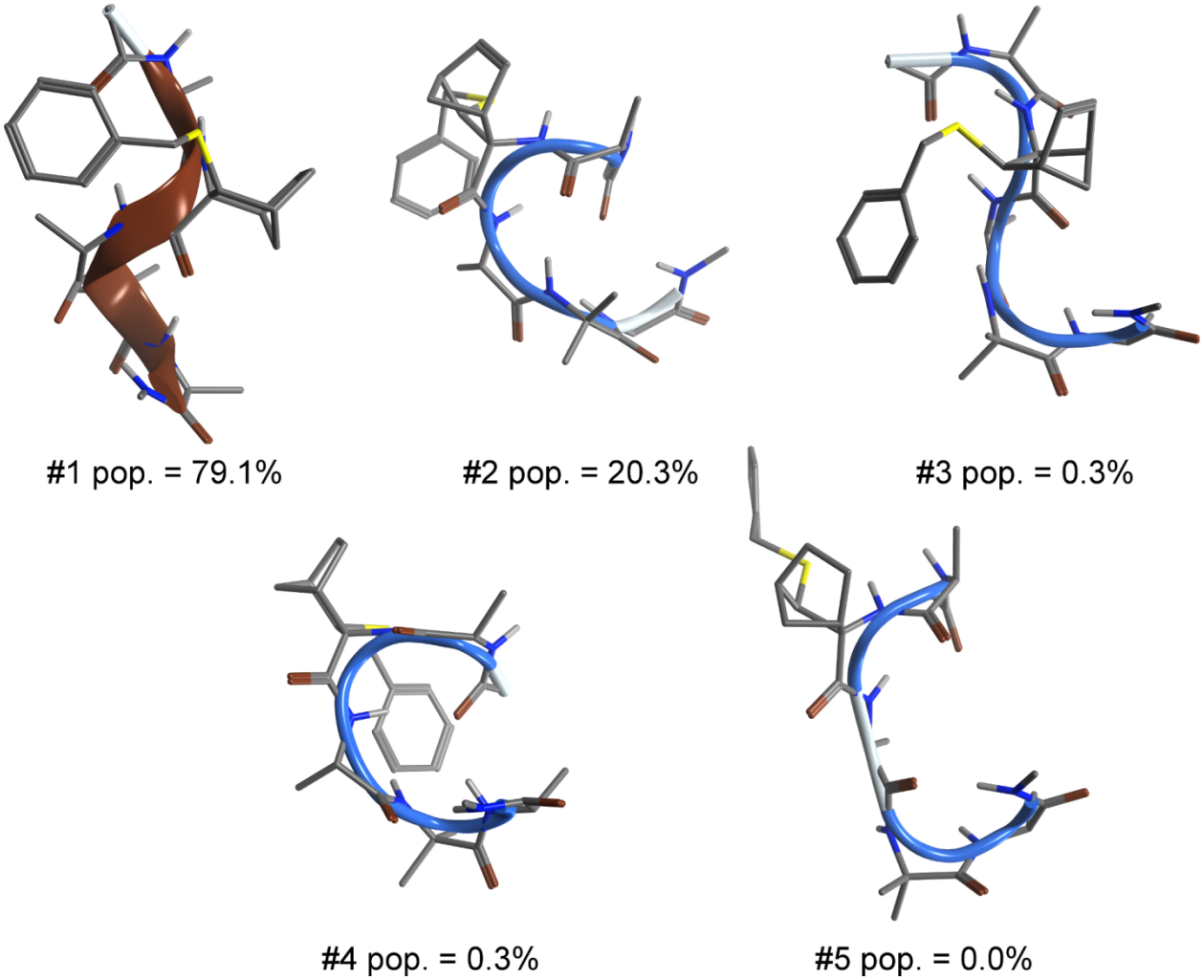
AR 453 F2
13C-APT in CD3CN a T=300K



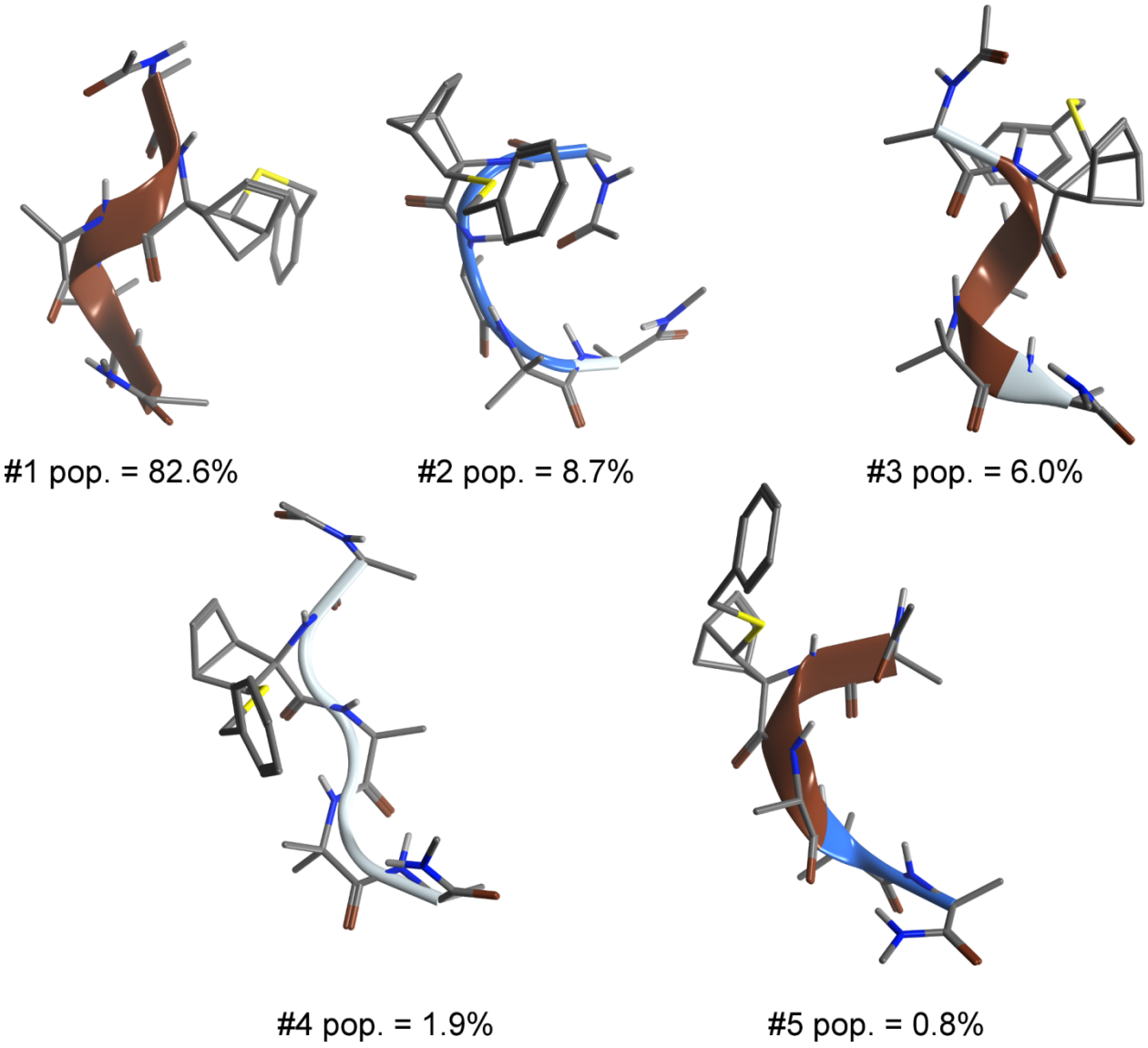
Computational Analysis

Methods

Theoretical calculations A preliminary Low Mode conformational search was performed for Ac-(*R,R,R,S*)-NRB-NMe using MOE software with the MMFF94x force field and the Born solvation model.[1] To obtain a charge set compatible with the *ff99SB* force field,[2] the two lowest energy conformations were optimized at the HF/6-31G(d) level and two different spatial orientation were used to derive orientation- and conformation-independent Resp charges, using the Gaussian 09 package.[3] Accordingly to standard amino acids, charge restraints of −0.4157, 0.2719, 0.5973 and −0.5679 were imposed to backbone nitrogen, hydrogen, carbonyl carbon and oxygen, respectively. The REMD simulation protocol has been previously optimized.[4] The simulations (12 replicas distributed over the following temperatures: 260.00, 283.25, 308.53, 335.98, 365.78, 398.13, 433.19, 471.31, 512.66, 557.47, 606.14 and 658.94 K) were originated from the extended configuration of peptides **2a** and **2b**. A short equilibration run (200 ps) was performed on each replica prior to the actual production run, then REMD simulations were conducted on each peptide for 50 ns at constant temperature by using Langevin dynamics (*ntt* = 3) with different seeds (*ig*) for every simulation.[5] A time step of 0.002 ps and an infinite cut-off for electrostatic were requested, and the SHAKE algorithm was used in order to constrain all bonds involving hydrogens.[6] Exchanges were attempted every 2 ps and were on average accepted with a 55% probability. For H-bond analyses the following parameters were used: donor-acceptor distance: 4.0 Å, angle cutoff: 30°. Cluster analyses were performed with *ptraj* (AmberTools13) by using the average-linkage algorithm, as suggested by Shao and coworkers,[7] by sampling one every two frames and using the pairwise mass weighted rmsd between frames as a metric. A total of 5 cluster were requested on the basis of the pseudo-F statistical analysis, and SSR/SST ratio (R-squared value).[8] Pushing the REMD runs up to 250 ns did not provide significant variations to the results of trajectory analyses, proofing that the simulation was fully converged for the parameters of interest.



Most representative geometries obtained from the cluster analysis of the final 25 ns of the 308.5 K *ff99SB* REMD trajectory for **2a** peptide having (*R,R,R,S*)-NRB at position 2



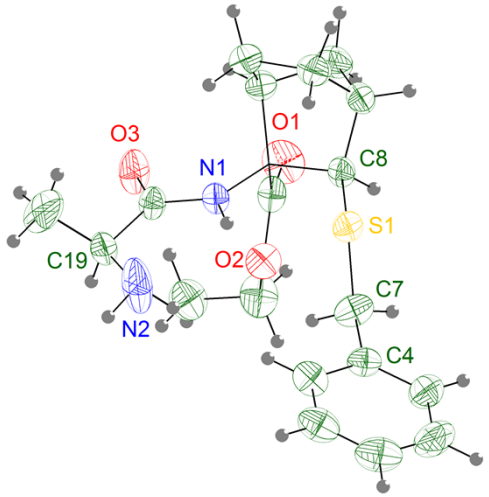
Most representative geometries obtained from the cluster analysis of the final 25 ns of the 308.5 K *ff99SB* REMD trajectory for **2b** peptide having (*S,S,S,R*)-NRB at position 2.

X-ray diffraction

Single crystals of **6a** were grown by slow evaporation from a mixture 1:1 of ethyl acetoacetate and chloroform, while those of compound **2b** by slow evaporation from a solution of acetonitrile. X-ray diffraction data were collected at room temperature and 150(2) K, for **6a** and **2b**, respectively, on a three-circle Bruker SMART APEX II diffractometer equipped with a CCD area detector and an Oxford Cryostream N_2 device. Diffraction data were recorded for both compounds using ω-scans (0.5 deg/frame) with graphite monochromated Mo Ka radiation (λ = 0.71073 Å) up to a 2ϑ Bragg angle of 55.0°. The *SAINT-Plus*[9] program package was used to perform the data reduction, whereas the collected structure factor amplitudes were corrected for beam anisotropy effects and for absorption using the program *SADAB*S.[10] The structures were solved by direct methods with the *SHELXS97*[11] program and refined by full-matrix least-squares procedures on F^2, using all data, by application of the SHELXL-97[4] and SHELXL2013[4] programs, for compound **6a** and **2b** respectively, with all non-H atoms anisotropic. For compound **6a** the coordinates of the H atoms bonded to nitrogen atoms and to atom C19 were refined, while all the other were included in the model at geometrically calculated positions and in riding modes, as it was done for all H atoms of compound **2b**. Crystal structure data have been deposited at the Cambridge Crystallographic Data Centre, and allocated the deposition numbers CCDC 989313 and CCDC 989314 for **6a** and **2b**, respectively.

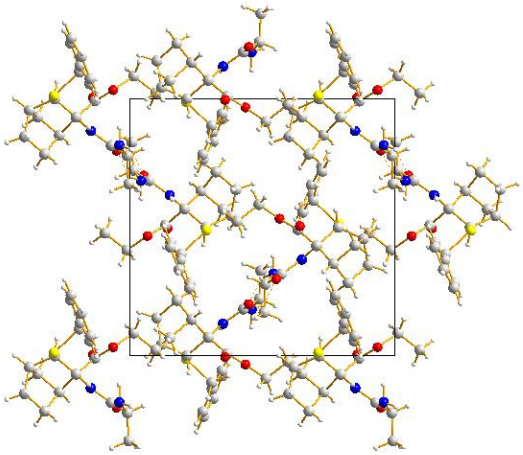
Crystal data for compound **6a**: $C_{20}H_{28}N_2O_3S$, M = 376.50, orthorhombic, space group $P2_12_12_1$, a =11.545(2) Å, b = 13.276(3) Å, c =13.627(3) Å, V = 2088.6(7) Å^3, Z = 4, Z'=1; ρ_{calcd} = 1.197 Mg m^{-3}, μ = 0.18 mm^{-1}, 27663 reflections measured, 4800 independent reflections (R_{int} = 0.044), θ_{max} = 27.51°, T = 293(2) K. Flack parameter: -0.03(9). The final R_1 values were 0.0468 [$I > 2\sigma(I)$] and 0.0738 (all data). The final w$R(F^2)$ values were 0.1082 [$I > 2\sigma(I)$] and 0.1231 (all data). Data/restraints/parameters 4800/0/242. Goodness-of-fit on F^2 = 1.019. The largest difference peak and hole were 0.286 and -0.243 e Å^{-3}, respectively.

Stereoview of the molecular structure of compound **6a** at 298 K, showing 30% probability displacement ellipsoids

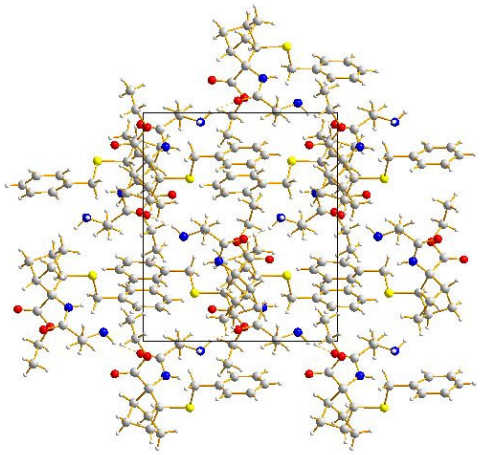


.

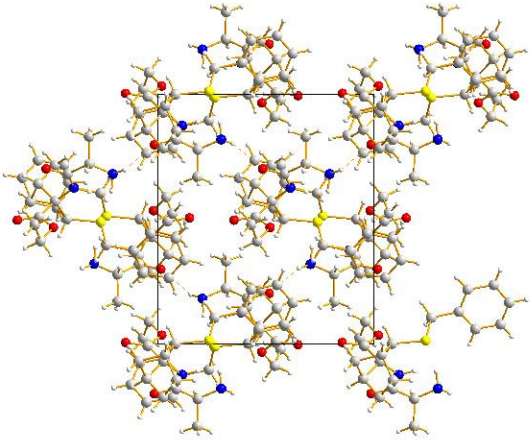
Packing of **6a**.



a) projection along cell axis *a*.



b) projection along cell axis *b*;



c) projection along cell axis *c*;

Crystal data for compound **2b**: $C_{30}H_{42}N_6O_6S$, M = 614.75, monoclinic, space group $P2_1$, a = 11.829(2)Å, b = 9.0852(18) Å, c = 31.098(6) Å, V = 3340.7(12) Å^3, Z = 4, Z'=2; ρ_{calcd} = 1.222 Mg m^{-3}, μ = 0.15 mm^{-1}, 72594 reflections measured, 15364 independent reflections (R_{int} = 0.045), θ_{max} = 27.51°, T = 150(2) K. Flack parameter: 0.01(2). The final R_1 values were 0.0502 [$I > 2\sigma(I)$] and 0.0795 (all data). The final w$R(F^2)$ values were 0.1113 [$I > 2\sigma(I)$] and 0.1235 (all data). Data/restraints/parameters 15364/1/788. Goodness-of-fit on F2 = 1.039. The largest difference peak and hole were 0.566 and -0.392 e Å-3, respectively.

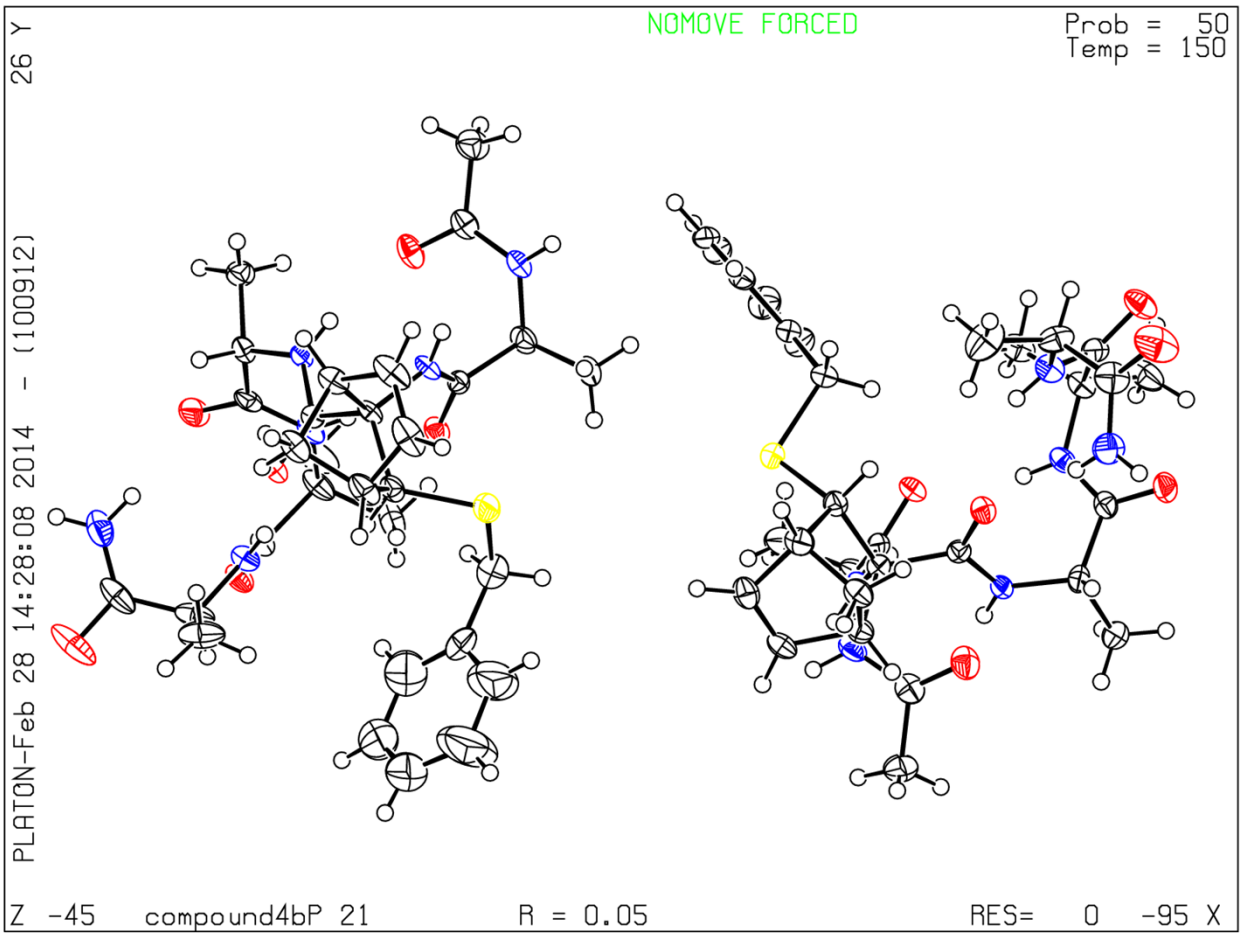


Figure S 1 Stereoview of the molecular structure of compound **2b** at 150 K, showing 50% probability displacement ellipsoids, 4i and 4ii two different conformers of peptide **2b** in the crystal cell

CIF for 6a (CCDC 989313)

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_audit_creation_method SHELXL-97

_audit_conform_dict_name cif_core.dic

_audit_conform_dict_version 2.4

_audit_conform_dict_location ftp://ftp.iucr.org/pub/cif_core.dic

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loop_

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#---------- CHEMICAL INFORMATION ----------#

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_chemical_formula_moiety 'C20 H28 N2 O3 S'
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_chemical_formula_weight 376.50
_chemical_absolute_configuration ad

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'H'  'H'   0.0000   0.0000
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'N'  'N'   0.0061   0.0033
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'S'  'S'   0.1246   0.1234
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting           orthorhombic
_symmetry_space_group_name_H-M    'P 21 21 21'
_symmetry_space_group_name_Hall   'P 2ac 2ab'

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x+1/2, -y, z+1/2'
 '-x, y+1/2, -z+1/2'
 'x+1/2, -y+1/2, -z'

_cell_length_a                 11.545(2)
_cell_length_b                 13.276(3)
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_cell_length_c                  13.627(3)
_cell_angle_alpha               90.00
_cell_angle_beta                90.00
_cell_angle_gamma               90.00
_cell_volume                    2088.6(7)
_cell_formula_units_Z           4
_cell_measurement_temperature   293(2)
_cell_measurement_reflns_used   4277
_cell_measurement_theta_min     2.3
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_exptl_crystal_description      'prism'
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_exptl_crystal_size_min         0.10
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_exptl_crystal_density_method   'not measured'
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_exptl_absorpt_correction_T_min 0.663
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_diffrn_measurement_method '\w-scan'

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_diffrn_reflns_av_R_equivalents 0.0438

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_diffrn_reflns_limit_l_min -17

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_reflns_number_total 4800

_reflns_number_gt 3447

_reflns_threshold_expression >2sigma(I)

_computing_data_collection 'SAINT+ (Bruker AXS, Madison (USA) 1994-1996)'

_computing_cell_refinement 'SAINT+ (Bruker AXS, Madison (USA) 1994-1996)'

_computing_data_reduction 'SAINT+ (Bruker AXS, Madison (USA) 1994-1996)'

_computing_structure_solution 'SHELXS-97 (Sheldrick, 1997)'

_computing_structure_refinement 'SHELXL-97 (Sheldrick, 1997)'

_computing_molecular_graphics 'Diamond v3.2i (Brandenburg, 2012)'

_refine_special_details

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Refinement of F^2^ against ALL reflections. The weighted R-factor wR 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^ > 2sigma(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.

;

_refine_ls_structure_factor_coef Fsqd

_refine_ls_matrix_type full

_refine_ls_weighting_scheme calc

_refine_ls_weighting_details

'calc w=1/[\s^2^(Fo^2^)+(0.0553P)^2^+0.3574P] where P=(Fo^2^+2Fc^2^)/3'

_atom_sites_solution_primary direct

_atom_sites_solution_secondary difmap

_atom_sites_solution_hydrogens geom

_refine_ls_hydrogen_treatment mixed

_refine_ls_extinction_method none

_refine_ls_extinction_coef ?

_refine_ls_abs_structure_details

'Flack H D (1983), Acta Cryst. A39, 876-881'

_refine_ls_abs_structure_Flack -0.03(9)

_refine_ls_number_reflns 4800

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_refine_ls_number_restraints 0

_refine_ls_R_factor_all 0.0738

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loop_

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_atom_site_symmetry_multiplicity

_atom_site_calc_flag

_atom_site_refinement_flags

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_atom_site_disorder_group

C1 C -0.1234(3) -0.1645(4) 0.6847(3) 0.1030(13) Uani 1 1 d . . .

H1 H -0.2026 -0.1773 0.6813 0.124 Uiso 1 1 calc R . .

C2 C -0.0517(4) -0.2357(3) 0.7237(3) 0.1089(13) Uani 1 1 d . . .

H2 H -0.0816 -0.2956 0.7486 0.131 Uiso 1 1 calc R . .

C3 C 0.0688(3) -0.2168(2) 0.7256(3) 0.0843(9) Uani 1 1 d . . .

H3 H 0.1195 -0.2647 0.7509 0.101 Uiso 1 1 calc R . .

C4 C 0.1097(2) -0.1281(2) 0.6903(2) 0.0625(7) Uani 1 1 d . . .

C5 C 0.0327(3) -0.0582(3) 0.6532(2) 0.0769(8) Uani 1 1 d . . .

H5 H 0.0602 0.0028 0.6291 0.092 Uiso 1 1 calc R . .

C6 C -0.0831(3) -0.0777(3) 0.6515(3) 0.0919(10) Uani 1 1 d . . .

H6 H -0.1341 -0.0298 0.6269 0.110 Uiso 1 1 calc R . .

C7 C 0.2371(2) -0.1042(2) 0.6924(2) 0.0768(8) Uani 1 1 d . . .

H7A H 0.2613 -0.0773 0.6295 0.092 Uiso 1 1 calc R . .

H7B H 0.2813 -0.1649 0.7054 0.092 Uiso 1 1 calc R . .

C8 C 0.41805(18) 0.00043(19) 0.78580(18) 0.0565(6) Uani 1 1 d . . .

H8 H 0.4513 -0.0670 0.7775 0.068 Uiso 1 1 calc R . .

C9 C 0.47324(18) 0.07184(18) 0.70475(19) 0.0527(5) Uani 1 1 d . . .

C10 C 0.5508(2) 0.1413(2) 0.7681(2) 0.0670(7) Uani 1 1 d . . .

H10 H 0.6150 0.1726 0.7322 0.080 Uiso 1 1 calc R . .

C11 C 0.4748(3) 0.2152(2) 0.8240(2) 0.0774(8) Uani 1 1 d . . .

H11A H 0.4195 0.2476 0.7807 0.093 Uiso 1 1 calc R . .

H11B H 0.5212 0.2665 0.8562 0.093 Uiso 1 1 calc R . .

C12 C 0.4130(3) 0.1474(2) 0.8999(2) 0.0790(8) Uani 1 1 d . . .

H12A H 0.4284 0.1700 0.9664 0.095 Uiso 1 1 calc R . .

H12B H 0.3299 0.1467 0.8891 0.095 Uiso 1 1 calc R . .

C13 C 0.4653(3) 0.0446(2) 0.8815(2) 0.0721(8) Uani 1 1 d . . .

H13 H 0.4605 -0.0013 0.9377 0.087 Uiso 1 1 calc R . .

C14 C 0.5885(3) 0.0701(3) 0.8505(2) 0.0852(9) Uani 1 1 d . . .

H14A H 0.6310 0.0120 0.8266 0.102 Uiso 1 1 calc R . .

H14B H 0.6321 0.1040 0.9016 0.102 Uiso 1 1 calc R . .

C15 C 0.5460(2) 0.0087(2) 0.6336(2) 0.0645(7) Uani 1 1 d . . .

C16 C 0.5402(4) -0.0886(3) 0.4869(3) 0.1033(12) Uani 1 1 d . . .

H16A H 0.4915 -0.1436 0.4645 0.124 Uiso 1 1 calc R . .

H16B H 0.6114 -0.1173 0.5125 0.124 Uiso 1 1 calc R . .

C17 C 0.5672(4) -0.0225(3) 0.4041(2) 0.1042(11) Uani 1 1 d . . .

H17A H 0.6082 -0.0599 0.3549 0.156 Uiso 1 1 calc R . .

H17B H 0.6146 0.0324 0.4264 0.156 Uiso 1 1 calc R . .

H17C H 0.4966 0.0035 0.3768 0.156 Uiso 1 1 calc R . .

C18 C 0.4141(2) 0.18177(19) 0.5720(2) 0.0587(6) Uani 1 1 d . . .

C19 C 0.3148(2) 0.2215(2) 0.5112(2) 0.0648(7) Uani 1 1 d . . .

C20 C 0.3163(4) 0.3311(3) 0.5058(5) 0.159(3) Uani 1 1 d . . .

H20A H 0.2524 0.3538 0.4665 0.238 Uiso 1 1 calc R . .

H20B H 0.3877 0.3530 0.4766 0.238 Uiso 1 1 calc R . .

H20C H 0.3098 0.3587 0.5707 0.238 Uiso 1 1 calc R . .

N1 N 0.38628(17) 0.12523(16) 0.64919(16) 0.0531(5) Uani 1 1 d . . .

N2 N 0.2064(3) 0.1807(3) 0.5412(3) 0.1516(19) Uani 1 1 d . . .

H2A H 0.2125 0.1128 0.5411 0.227 Uiso 1 1 d . . .

H2B H 0.1527 0.1975 0.4964 0.227 Uiso 1 1 d . . .

O1 O 0.64769(16) -0.0045(2) 0.64149(19) 0.1020(7) Uani 1 1 d . . .

O2 O 0.48084(18) -0.03372(16) 0.56474(15) 0.0770(5) Uani 1 1 d . . .

O3 O 0.51496(17) 0.20144(18) 0.55176(18) 0.0911(7) Uani 1 1 d . . .

S1 S 0.26240(5) -0.01211(5) 0.78841(5) 0.06035(18) Uani 1 1 d . . .

HN1 H 0.315(2) 0.110(2) 0.6581(19) 0.064(8) Uiso 1 1 d . . .

H19 H 0.326(3) 0.194(2) 0.450(2) 0.077 Uiso 1 1 d . . .

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C2 0.107(3) 0.104(3) 0.116(3) -0.005(3) 0.015(3) -0.048(2)

C3 0.083(2) 0.0757(19) 0.094(2) 0.0003(18) -0.0028(19) -0.0108(16)

C4 0.0500(13) 0.0746(17) 0.0629(16) -0.0078(13) 0.0041(12) -0.0044(12)

C5 0.0645(18) 0.098(2) 0.0683(17) 0.0025(16) 0.0029(14) 0.0049(16)

C6 0.063(2) 0.127(3) 0.086(2) -0.007(2) -0.0078(17) 0.006(2)

C7 0.0524(14) 0.0796(17) 0.099(2) -0.0161(16) 0.0113(15) -0.0039(14)

C8 0.0452(11) 0.0601(14) 0.0643(13) 0.0131(14) -0.0042(11) -0.0003(10)

C9 0.0366(11) 0.0579(13) 0.0637(14) 0.0138(12) -0.0016(11) 0.0021(9)

C10 0.0484(13) 0.0747(17) 0.0779(18) 0.0101(15) -0.0082(13) -0.0129(12)

C11 0.084(2) 0.0711(18) 0.0768(19) -0.0032(15) -0.0056(16) -0.0151(16)

C12 0.085(2) 0.086(2) 0.0664(17) -0.0010(15) -0.0066(16) -0.0126(17)

C13 0.0707(17) 0.0797(19) 0.0660(17) 0.0191(14) -0.0147(13) -0.0105(15)

C14 0.0634(18) 0.104(2) 0.088(2) 0.0130(19) -0.0248(16) -0.0106(17)

C15 0.0468(13) 0.0650(16) 0.0816(18) 0.0176(15) 0.0094(12) 0.0085(12)

C16 0.126(3) 0.099(3) 0.086(2) -0.004(2) 0.033(2) 0.018(2)

C17 0.116(3) 0.117(3) 0.080(2) 0.004(2) 0.019(2) -0.006(2)

C18 0.0499(14) 0.0577(14) 0.0685(16) 0.0141(13) 0.0050(12) 0.0009(11)

C19 0.0651(16) 0.0676(17) 0.0616(16) 0.0159(14) -0.0045(13) 0.0055(13)

C20 0.140(4) 0.060(2) 0.276(7) 0.038(3) -0.109(4) 0.005(2)

N1 0.0357(10) 0.0608(12) 0.0629(12) 0.0142(10) 0.0011(9) 0.0023(9)

N2 0.0591(17) 0.194(4) 0.201(4) 0.137(3) -0.034(2) -0.0205(19)

O1 0.0510(11) 0.1279(19) 0.1272(18) 0.0032(17) 0.0059(11) 0.0307(13)

O2 0.0735(12) 0.0810(13) 0.0765(12) -0.0062(10) 0.0151(11) 0.0031(10)

O3 0.0503(11) 0.1113(17) 0.1116(16) 0.0517(14) 0.0140(11) -0.0028(11)

S1 0.0471(3) 0.0685(4) 0.0654(3) 0.0066(3) 0.0063(3) -0.0062(3)

_geom_special_details

;

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.

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_geom_bond_atom_site_label_2

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C1 C6 1.322(5) . ?

C1 C2 1.365(6) . ?

C1 H1 0.9300 . ?

C2 C3 1.415(5) . ?

C2 H2 0.9300 . ?

C3 C4 1.357(4) . ?

C3 H3 0.9300 . ?

C4 C5 1.381(4) . ?

C4 C7 1.504(4) . ?

C5 C6 1.362(4) . ?

C5 H5 0.9300 . ?

C6 H6 0.9300 . ?

C7 S1 1.814(3) . ?

C7 H7A 0.9700 . ?

C7 H7B 0.9700 . ?

C8 C13 1.530(4) . ?

C8 C9 1.589(3) . ?

C8 S1 1.805(2) . ?

C8 H8 0.9800 . ?

C9 N1 1.443(3) . ?

C9 C15 1.532(4) . ?

C9 C10 1.548(4) . ?

C10 C11 1.521(4) . ?

C10 C14 1.531(4) . ?

C10 H10 0.9800 . ?

C11 C12 1.546(4) . ?

C11 H11A 0.9700 . ?

C11 H11B 0.9700 . ?

C12 C13 1.514(4) . ?

C12 H12A 0.9700 . ?

C12 H12B 0.9700 . ?

C13 C14 1.522(4) . ?

C13 H13 0.9800 . ?

C14 H14A 0.9700 . ?

C14 H14B 0.9700 . ?

C15 O1 1.192(3) . ?

C15 O2 1.328(3) . ?

C16 O2 1.459(4) . ?

C16 C17 1.463(5) . ?

C16 H16A 0.9700 . ?

C16 H16B 0.9700 . ?

C17 H17A 0.9600 . ?

C17 H17B 0.9600 . ?

C17 H17C 0.9600 . ?

C18 O3 1.225(3) . ?

C18 N1 1.331(3) . ?

C18 C19 1.509(4) . ?

C19 N2 1.423(4) . ?

C19 C20 1.457(5) . ?

C19 H19 0.92(3) . ?

C20 H20A 0.9600 . ?

C20 H20B 0.9600 . ?

C20 H20C 0.9600 . ?

N1 HN1 0.85(3) . ?

N2 H2A 0.904 . ?

N2 H2B 0.898 . ?

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C6 C1 H1 119.2 . . ?

C2 C1 H1 119.2 . . ?

C1 C2 C3 118.7(4) . . ?

C1 C2 H2 120.6 . . ?

C3 C2 H2 120.6 . . ?

C4 C3 C2 119.3(3) . . ?

C4 C3 H3 120.3 . . ?

C2 C3 H3 120.3 . . ?

C3 C4 C5 119.3(3) . . ?

C3 C4 C7 121.1(3) . . ?

C5 C4 C7 119.7(3) . . ?

C6 C5 C4 120.7(3) . . ?

C6 C5 H5 119.6 . . ?

C4 C5 H5 119.6 . . ?

C1 C6 C5 120.4(4) . . ?

C1 C6 H6 119.8 . . ?

C5 C6 H6 119.8 . . ?

C4 C7 S1 108.27(19) . . ?

C4 C7 H7A 110.0 . . ?

S1 C7 H7A 110.0 . . ?

C4 C7 H7B 110.0 . . ?

S1 C7 H7B 110.0 . . ?

H7A C7 H7B 108.4 . . ?

C13 C8 C9 102.8(2) . . ?

C13 C8 S1 111.92(18) . . ?

C9 C8 S1 117.91(15) . . ?

C13 C8 H8 107.9 . . ?

C9 C8 H8 107.9 . . ?

S1 C8 H8 107.9 . . ?

N1 C9 C15 108.5(2) . . ?

N1 C9 C10 113.7(2) . . ?

C15 C9 C10 111.2(2) . . ?

N1 C9 C8 112.25(17) . . ?

C15 C9 C8 109.45(19) . . ?

C10 C9 C8 101.53(19) . . ?

C11 C10 C14 101.2(3) . . ?

C11 C10 C9 109.3(2) . . ?

C14 C10 C9 101.8(2) . . ?

C11 C10 H10 114.3 . . ?

C14 C10 H10 114.3 . . ?

C9 C10 H10 114.3 . . ?

C10 C11 C12 103.1(2) . . ?

C10 C11 H11A 111.2 . . ?

C12 C11 H11A 111.2 . . ?

C10 C11 H11B 111.2 . . ?

C12 C11 H11B 111.2 . . ?

H11A C11 H11B 109.1 . . ?

C13 C12 C11 103.3(3) . . ?

C13 C12 H12A 111.1 . . ?

C11 C12 H12A 111.1 . . ?

C13 C12 H12B 111.1 . . ?

C11 C12 H12B 111.1 . . ?

H12A C12 H12B 109.1 . . ?

C12 C13 C14 102.6(3) . . ?

C12 C13 C8 110.2(2) . . ?

C14 C13 C8 100.5(2) . . ?

C12 C13 H13 114.1 . . ?

C14 C13 H13 114.1 . . ?

C8 C13 H13 114.1 . . ?

C13 C14 C10 94.3(2) . . ?

C13 C14 H14A 112.9 . . ?

C10 C14 H14A 112.9 . . ?

C13 C14 H14B 112.9 . . ?

C10 C14 H14B 112.9 . . ?

H14A C14 H14B 110.3 . . ?

O1 C15 O2 124.0(3) . . ?

O1 C15 C9 124.2(3) . . ?

O2 C15 C9 111.7(2) . . ?

O2 C16 C17 111.2(3) . . ?

O2 C16 H16A 109.4 . . ?

C17 C16 H16A 109.4 . . ?

O2 C16 H16B 109.4 . . ?

C17 C16 H16B 109.4 . . ?

H16A C16 H16B 108.0 . . ?

C16 C17 H17A 109.5 . . ?

C16 C17 H17B 109.5 . . ?

H17A C17 H17B 109.5 . . ?

C16 C17 H17C 109.5 . . ?

H17A C17 H17C 109.5 . . ?

H17B C17 H17C 109.5 . . ?

O3 C18 N1 121.8(2) . . ?

O3 C18 C19 121.6(2) . . ?

N1 C18 C19 116.6(2) . . ?

N2 C19 C20 113.9(4) . . ?

N2 C19 C18 112.2(2) . . ?

C20 C19 C18 111.6(3) . . ?

N2 C19 H19 103(2) . . ?

C20 C19 H19 110.6(19) . . ?

C18 C19 H19 104.6(19) . . ?

C19 C20 H20A 109.5 . . ?

C19 C20 H20B 109.5 . . ?

H20A C20 H20B 109.5 . . ?

C19 C20 H20C 109.5 . . ?

H20A C20 H20C 109.5 . . ?

H20B C20 H20C 109.5 . . ?

C18 N1 C9 121.6(2) . . ?

C18 N1 HN1 118.5(18) . . ?

C9 N1 HN1 118.6(19) . . ?

C19 N2 H2A 108.1 . . ?

C19 N2 H2B 108.5 . . ?

H2A N2 H2B 107.5 . . ?

C15 O2 C16 117.4(3) . . ?

C8 S1 C7 102.03(12) . . ?

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_geom_torsion_site_symmetry_4

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C6 C1 C2 C3 -2.0(6) . . . . ?

C1 C2 C3 C4 1.1(6) . . . . ?

C2 C3 C4 C5 0.0(5) . . . . ?

C2 C3 C4 C7 179.0(3) . . . . ?

C3 C4 C5 C6 -0.2(5) . . . . ?

C7 C4 C5 C6 -179.3(3) . . . . ?

C2 C1 C6 C5 1.7(6) . . . . ?

C4 C5 C6 C1 -0.6(5) . . . . ?

C3 C4 C7 S1 -105.9(3) . . . . ?

C5 C4 C7 S1 73.1(3) . . . . ?

C13 C8 C9 N1 -118.0(2) . . . . ?

S1 C8 C9 N1 5.6(3) . . . . ?

C13 C8 C9 C15 121.5(2) . . . . ?

S1 C8 C9 C15 -115.0(2) . . . . ?

C13 C8 C9 C10 3.8(2) . . . . ?

S1 C8 C9 C10 127.40(19) . . . . ?

N1 C9 C10 C11 47.1(3) . . . . ?

C15 C9 C10 C11 170.0(2) . . . . ?

C8 C9 C10 C11 -73.6(2) . . . . ?

N1 C9 C10 C14 153.7(2) . . . . ?

C15 C9 C10 C14 -83.4(3) . . . . ?

C8 C9 C10 C14 32.9(2) . . . . ?

C14 C10 C11 C12 -37.0(3) . . . . ?

C9 C10 C11 C12 69.9(3) . . . . ?

C10 C11 C12 C13 2.7(3) . . . . ?

C11 C12 C13 C14 32.9(3) . . . . ?

C11 C12 C13 C8 -73.4(3) . . . . ?

C9 C8 C13 C12 68.3(3) . . . . ?

S1 C8 C13 C12 -59.2(3) . . . . ?

C9 C8 C13 C14 -39.4(3) . . . . ?

S1 C8 C13 C14 -166.9(2) . . . . ?

C12 C13 C14 C10 -54.5(3) . . . . ?

C8 C13 C14 C10 59.1(3) . . . . ?

C11 C10 C14 C13 55.9(3) . . . . ?

C9 C10 C14 C13 -56.9(3) . . . . ?

N1 C9 C15 O1 142.9(3) . . . . ?

C10 C9 C15 O1 17.1(4) . . . . ?

C8 C9 C15 O1 -94.3(3) . . . . ?

N1 C9 C15 O2 -40.1(3) . . . . ?

C10 C9 C15 O2 -165.9(2) . . . . ?

C8 C9 C15 O2 82.7(2) . . . . ?

O3 C18 C19 N2 173.5(4) . . . . ?

N1 C18 C19 N2 -7.1(4) . . . . ?

O3 C18 C19 C20 -57.3(5) . . . . ?

N1 C18 C19 C20 122.1(4) . . . . ?

O3 C18 N1 C9 -9.2(4) . . . . ?

C19 C18 N1 C9 171.4(2) . . . . ?

C15 C9 N1 C18 -52.7(3) . . . . ?

C10 C9 N1 C18 71.7(3) . . . . ?

C8 C9 N1 C18 -173.8(2) . . . . ?

O1 C15 O2 C16 -8.4(4) . . . . ?

C9 C15 O2 C16 174.6(2) . . . . ?

C17 C16 O2 C15 -90.3(4) . . . . ?

C13 C8 S1 C7 -159.9(2) . . . . ?

C9 C8 S1 C7 81.2(2) . . . . ?

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CIF for 2b (CCDC 989314)

data_compound2b

_audit_creation_date 15/02/2014

_audit_creation_method SHELXL-97

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#---------- CHEMICAL INFORMATION ----------#

_chemical_name_systematic

;

(1S,2S,3S,4R)-2-((S)-2-acetamidopropanamido)-N-((S)-1-((1-(((S)-1-amino-1-oxopropan-2-yl)amino)-2-methyl-1-oxopropan-2-yl)amino)-1-oxopropan-2-yl)-3-(benzylthio)bicyclo[2.2.1]hept-5-ene-2-carboxamide

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_chemical_melting_point ?

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'C30 H42 N6 O6 S'

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'C30 H42 N6 O6 S'

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_atom_type_scat_dispersion_real

_atom_type_scat_dispersion_imag

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'C' 'C' 0.0033 0.0016

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'S' 'S' 0.1246 0.1234

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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loop_

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'x, y, z'

'-x, y+1/2, -z'

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_reflns_threshold_expression 'I > 2\s(I)'

_reflns_Friedel_coverage 0.881

_reflns_special_details

;

Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

_reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

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_computing_cell_refinement 'SAINT+ (Bruker AXS, Madison (USA) 1994-1996)'

_computing_data_reduction 'SAINT+ (Bruker AXS, Madison (USA) 1994-1996)'

_computing_structure_solution 'SHELXS97 (Sheldrick, 1997)'

_computing_structure_refinement 'SHELXL-97 (Sheldrick, 1997)'

_computing_molecular_graphics 'Diamond v3.2i (Brandenburg, 2012)'

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_refine_ls_structure_factor_coef Fsqd

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_refine_ls_weighting_scheme calc

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'w=1/[\s^2^(Fo^2^)+(0.0650P)^2^+0.1731P] where P=(Fo^2^+2Fc^2^)/3'

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_atom_sites_solution_secondary ?

_atom_sites_solution_hydrogens geom

_refine_ls_hydrogen_treatment constr

_refine_ls_extinction_method SHELXL

_refine_ls_extinction_coef 0.0021(6)

_refine_ls_extinction_expression

'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'

_refine_ls_abs_structure_details

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Flack x determined using 4302 quotients [(I+)-(I-)]/[(I+)+(I-)]

(Parsons and Flack (2004), Acta Cryst. A60, s61).

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_refine_ls_abs_structure_Flack 0.01(2)

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_refine_ls_number_reflns 15364

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loop_

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_atom_site_refinement_flags_occupancy

_atom_site_disorder_assembly

_atom_site_disorder_group

S1A S 0.55782(8) 0.23683(12) 0.79807(3) 0.0300(2) Uani 1 1 d . . . . .

O1A O 0.77319(19) 0.3001(3) 0.91003(8) 0.0236(6) Uani 1 1 d . . . . .

O2A O 0.9310(2) 0.0395(3) 0.98508(8) 0.0326(6) Uani 1 1 d . . . . .

O3A O 0.59932(18) -0.0058(3) 0.87904(8) 0.0239(6) Uani 1 1 d . . . . .

O4A O 1.0932(2) -0.0637(3) 0.89809(9) 0.0347(7) Uani 1 1 d . . . . .

O5A O 0.4534(2) 0.0156(3) 0.97587(8) 0.0355(7) Uani 1 1 d . . . . .

O6A O 1.2037(2) 0.3181(4) 0.91189(11) 0.0529(9) Uani 1 1 d . . . . .

N1A N 0.6563(2) 0.1838(3) 0.95431(9) 0.0214(7) Uani 1 1 d . . . . .

HN1A H 0.5887 0.1555 0.9596 0.026 Uiso 1 1 calc R U . . .

N2A N 0.8110(2) -0.0351(3) 0.93143(9) 0.0228(7) Uani 1 1 d . . . . .

HN2A H 0.7424 -0.0357 0.9215 0.027 Uiso 1 1 calc R U . . .

N3A N 0.4864(2) 0.1902(3) 0.89192(9) 0.0203(6) Uani 1 1 d . . . . .

HN3A H 0.4185 0.2185 0.8968 0.024 Uiso 1 1 calc R U . . .

N4A N 0.9657(2) 0.1138(3) 0.88238(10) 0.0250(7) Uani 1 1 d . . . . .

HN4A H 0.8952 0.1366 0.8804 0.030 Uiso 1 1 calc R U . . .

N5A N 0.3239(2) -0.0047(4) 0.92221(10) 0.0278(7) Uani 1 1 d . . . . .

HN5A H 0.2548 0.0095 0.9139 0.033 Uiso 1 1 calc R U . . .

N6A N 1.0280(3) 0.3594(4) 0.93553(11) 0.0362(8) Uani 1 1 d . . . . .

HN6A1 H 1.0522 0.4070 0.9579 0.043 Uiso 1 1 calc R U . . .

HN6A2 H 0.9566 0.3469 0.9311 0.043 Uiso 1 1 calc R U . . .

C1A C 0.6789(3) 0.1179(5) 0.78889(12) 0.0297(9) Uani 1 1 d . . . . .

H1A1 H 0.7406 0.1759 0.7779 0.036 Uiso 1 1 calc R U . . .

H1A2 H 0.7043 0.0737 0.8159 0.036 Uiso 1 1 calc R U . . .

C2A C 0.6474(3) -0.0005(4) 0.75731(11) 0.0260(8) Uani 1 1 d . . . . .

C3A C 0.6641(3) 0.0192(4) 0.71366(11) 0.0254(8) Uani 1 1 d . . . . .

H3A H 0.6980 0.1053 0.7043 0.031 Uiso 1 1 calc R U . . .

C4A C 0.6316(3) -0.0862(4) 0.68408(12) 0.0264(8) Uani 1 1 d . . . . .

H4A H 0.6430 -0.0705 0.6550 0.032 Uiso 1 1 calc R U . . .

C5A C 0.5818(3) -0.2158(4) 0.69753(13) 0.0312(9) Uani 1 1 d . . . . .

H5A H 0.5594 -0.2873 0.6777 0.037 Uiso 1 1 calc R U . . .

C6A C 0.5659(3) -0.2370(5) 0.74115(13) 0.0376(10) Uani 1 1 d . . . . .

H6A H 0.5327 -0.3235 0.7506 0.045 Uiso 1 1 calc R U . . .

C7A C 0.5985(3) -0.1319(5) 0.77035(13) 0.0349(10) Uani 1 1 d . . . . .

H7A H 0.5878 -0.1485 0.7995 0.042 Uiso 1 1 calc R U . . .

C8A C 0.6146(3) 0.3430(4) 0.84279(11) 0.0239(8) Uani 1 1 d . . . . .

H8A H 0.6972 0.3344 0.8428 0.029 Uiso 1 1 calc R U . . .

C9A C 0.5740(3) 0.3015(4) 0.88945(11) 0.0192(7) Uani 1 1 d . . . . .

C10A C 0.9955(3) -0.0194(4) 0.89735(11) 0.0242(8) Uani 1 1 d . . . . .

C11A C 0.6761(3) 0.2589(4) 0.91851(11) 0.0196(7) Uani 1 1 d . . . . .

C12A C 0.8374(3) 0.0454(4) 0.96700(11) 0.0231(8) Uani 1 1 d . . . . .

C13A C 0.7492(3) 0.1492(4) 0.98465(11) 0.0244(8) Uani 1 1 d . . . . .

H13A H 0.7877 0.2419 0.9918 0.029 Uiso 1 1 calc R U . . .

C14A C 0.7012(4) 0.0892(5) 1.02628(12) 0.0387(11) Uani 1 1 d . . . . .

H14A H 0.6515 0.1610 1.0383 0.058 Uiso 1 1 calc R U . . .

H14B H 0.7620 0.0687 1.0465 0.058 Uiso 1 1 calc R U . . .

H14C H 0.6598 0.0003 1.0202 0.058 Uiso 1 1 calc R U . . .

C15A C 0.5055(3) 0.0444(4) 0.88705(11) 0.0205(7) Uani 1 1 d . . . . .

C16A C 0.4049(3) -0.0585(4) 0.89130(12) 0.0266(8) Uani 1 1 d . . . . .

H16A H 0.4333 -0.1541 0.9014 0.032 Uiso 1 1 calc R U . . .

C17A C 0.3549(3) 0.0229(4) 0.96314(13) 0.0294(9) Uani 1 1 d . . . . .

C18A C 1.0496(3) 0.2210(5) 0.86943(12) 0.0317(9) Uani 1 1 d . . . . .

H18A H 1.1107 0.1675 0.8556 0.038 Uiso 1 1 calc R U . . .

C19A C 0.8971(3) -0.1217(4) 0.90930(12) 0.0249(8) Uani 1 1 d . . . . .

C20A C 0.5839(3) 0.5093(4) 0.83930(12) 0.0288(9) Uani 1 1 d . . . . .

H20A H 0.6216 0.5642 0.8167 0.035 Uiso 1 1 calc R U . . .

C21A C 0.4564(3) 0.5187(4) 0.83844(13) 0.0315(9) Uani 1 1 d . . . . .

H21A H 0.4105 0.5439 0.8148 0.038 Uiso 1 1 calc R U . . .

C22A C 0.6104(3) 0.5605(4) 0.88554(12) 0.0281(9) Uani 1 1 d . . . . .

H22A H 0.6881 0.5414 0.8947 0.034 Uiso 1 1 calc R U . . .

H22B H 0.5915 0.6630 0.8902 0.034 Uiso 1 1 calc R U . . .

C23A C 1.1013(3) 0.3050(5) 0.90783(14) 0.0343(10) Uani 1 1 d . . . . .

C24A C 0.5264(3) 0.4544(4) 0.90544(12) 0.0236(8) Uani 1 1 d . . . . .

H24A H 0.5171 0.4638 0.9365 0.028 Uiso 1 1 calc R U . . .

C25A C 0.4217(3) 0.4847(4) 0.87745(13) 0.0271(8) Uani 1 1 d . . . . .

H25A H 0.3471 0.4802 0.8860 0.032 Uiso 1 1 calc R U . . .

C26A C 0.8442(3) -0.1781(5) 0.86755(13) 0.0310(9) Uani 1 1 d . . . . .

H26A H 0.7807 -0.2397 0.8737 0.047 Uiso 1 1 calc R U . . .

H26B H 0.8990 -0.2341 0.8523 0.047 Uiso 1 1 calc R U . . .

H26C H 0.8193 -0.0964 0.8502 0.047 Uiso 1 1 calc R U . . .

C27A C 0.9414(3) -0.2507(5) 0.93625(15) 0.0397(10) Uani 1 1 d . . . . .

H27A H 0.9739 -0.2145 0.9628 0.060 Uiso 1 1 calc R U . . .

H27B H 0.9979 -0.3025 0.9207 0.060 Uiso 1 1 calc R U . . .

H27C H 0.8801 -0.3162 0.9422 0.060 Uiso 1 1 calc R U . . .

C28A C 0.2610(3) 0.0703(5) 0.99226(13) 0.0392(10) Uani 1 1 d . . . . .

H28A H 0.2676 0.0177 1.0190 0.059 Uiso 1 1 calc R U . . .

H28B H 0.1891 0.0492 0.9786 0.059 Uiso 1 1 calc R U . . .

H28C H 0.2669 0.1741 0.9977 0.059 Uiso 1 1 calc R U . . .

C29A C 0.9990(4) 0.3270(6) 0.83687(15) 0.0494(12) Uani 1 1 d . . . . .

H29A H 0.9726 0.2736 0.8119 0.074 Uiso 1 1 calc R U . . .

H29B H 1.0554 0.3969 0.8287 0.074 Uiso 1 1 calc R U . . .

H29C H 0.9368 0.3781 0.8493 0.074 Uiso 1 1 calc R U . . .

C30A C 0.3451(3) -0.0812(6) 0.84813(14) 0.0432(11) Uani 1 1 d . . . . .

H30A H 0.2843 -0.1500 0.8512 0.065 Uiso 1 1 calc R U . . .

H30B H 0.3977 -0.1189 0.8279 0.065 Uiso 1 1 calc R U . . .

H30C H 0.3153 0.0111 0.8379 0.065 Uiso 1 1 calc R U . . .

S1B S 0.28924(8) 0.64460(13) 0.68729(3) 0.0376(3) Uani 1 1 d . . . . .

O1B O 0.04079(18) 0.5500(3) 0.58327(8) 0.0216(5) Uani 1 1 d . . . . .

O2B O -0.10883(19) 0.2206(3) 0.53216(9) 0.0375(7) Uani 1 1 d . . . . .

O3B O 0.20864(19) 0.3293(3) 0.64102(8) 0.0258(6) Uani 1 1 d . . . . .

O4B O -0.27424(19) 0.1873(3) 0.62409(8) 0.0255(6) Uani 1 1 d . . . . .

O5B O 0.3436(2) 0.1707(3) 0.55663(9) 0.0321(6) Uani 1 1 d . . . . .

O6B O -0.4359(2) 0.5320(4) 0.57267(12) 0.0606(10) Uani 1 1 d . . . . .

N1B N 0.1608(2) 0.3994(3) 0.54922(9) 0.0185(6) Uani 1 1 d . . . . .

HN1B H 0.2294 0.3702 0.5466 0.022 Uiso 1 1 calc R U . . .

N2B N 0.0103(2) 0.2142(3) 0.59006(10) 0.0243(7) Uani 1 1 d . . . . .

HN2B H 0.0781 0.2300 0.5997 0.029 Uiso 1 1 calc R U . . .

N3B N 0.3301(2) 0.4736(3) 0.60607(9) 0.0197(6) Uani 1 1 d . . . . .

HN3B H 0.3970 0.4856 0.5965 0.024 Uiso 1 1 calc R U . . .

N4B N -0.1675(2) 0.3884(3) 0.61273(9) 0.0227(7) Uani 1 1 d . . . . .

HN4B H -0.1008 0.4238 0.6096 0.027 Uiso 1 1 calc R U . . .

N5B N 0.4790(2) 0.2174(3) 0.60724(10) 0.0239(7) Uani 1 1 d . . . . .

HN5B H 0.5501 0.2307 0.6124 0.029 Uiso 1 1 calc R U . . .

N6B N -0.2945(3) 0.4297(4) 0.53643(12) 0.0385(9) Uani 1 1 d . . . . .

HN6B1 H -0.3338 0.4298 0.5127 0.046 Uiso 1 1 calc R U . . .

HN6B2 H -0.2266 0.3956 0.5370 0.046 Uiso 1 1 calc R U . . .

C1B C 0.1857(4) 0.5609(6) 0.71954(15) 0.0518(13) Uani 1 1 d . . . . .

H1B1 H 0.1487 0.4827 0.7033 0.062 Uiso 1 1 calc R U . . .

H1B2 H 0.2227 0.5171 0.7447 0.062 Uiso 1 1 calc R U . . .

C2B C 0.0959(4) 0.6712(8) 0.73428(14) 0.0644(17) Uani 1 1 d . . . . .

C3B C -0.0101(5) 0.6694(9) 0.7235(2) 0.0809(19) Uani 1 1 d . . . . .

H3B H -0.0343 0.5961 0.7045 0.097 Uiso 1 1 calc R U . . .

C4B C -0.0917(6) 0.7671(10) 0.7377(2) 0.094(3) Uani 1 1 d . . . . .

H4B H -0.1682 0.7527 0.7314 0.113 Uiso 1 1 calc R U . . .

C5B C -0.0553(6) 0.8820(11) 0.7607(2) 0.095(3) Uani 1 1 d . . . . .

H5B H -0.1059 0.9565 0.7672 0.114 Uiso 1 1 calc R U . . .

C6B C 0.0562(6) 0.8935(11) 0.7754(3) 0.131(4) Uani 1 1 d . . . . .

H6B H 0.0785 0.9674 0.7945 0.157 Uiso 1 1 calc R U . . .

C7B C 0.1333(6) 0.7919(10) 0.7607(2) 0.106(3) Uani 1 1 d . . . . .

H7B H 0.2097 0.8021 0.7680 0.128 Uiso 1 1 calc R U . . .

C8B C 0.2104(3) 0.6818(4) 0.63765(12) 0.0254(8) Uani 1 1 d . . . . .

H8B H 0.1294 0.6705 0.6426 0.031 Uiso 1 1 calc R U . . .

C9B C 0.2432(3) 0.5842(4) 0.59762(12) 0.0194(7) Uani 1 1 d . . . . .

C10B C -0.1813(3) 0.2428(4) 0.61748(11) 0.0213(7) Uani 1 1 d . . . . .

C11B C 0.1389(3) 0.5079(4) 0.57710(11) 0.0181(7) Uani 1 1 d . . . . .

C12B C -0.0162(3) 0.2492(4) 0.54944(12) 0.0240(8) Uani 1 1 d . . . . .

C13B C 0.0720(3) 0.3304(4) 0.52342(11) 0.0218(8) Uani 1 1 d . . . . .

H13B H 0.0330 0.4075 0.5067 0.026 Uiso 1 1 calc R U . . .

C14B C 0.1246(3) 0.2236(4) 0.49190(12) 0.0280(8) Uani 1 1 d . . . . .

H14D H 0.1794 0.2745 0.4753 0.042 Uiso 1 1 calc R U . . .

H14E H 0.0666 0.1840 0.4730 0.042 Uiso 1 1 calc R U . . .

H14F H 0.1609 0.1448 0.5076 0.042 Uiso 1 1 calc R U . . .

C15B C 0.3052(3) 0.3530(4) 0.62884(11) 0.0189(7) Uani 1 1 d . . . . .

C16B C 0.3996(3) 0.2464(4) 0.64121(12) 0.0276(8) Uani 1 1 d . . . . .

H16B H 0.3643 0.1525 0.6486 0.033 Uiso 1 1 calc R U . . .

C17B C 0.4445(3) 0.1704(4) 0.56808(12) 0.0262(8) Uani 1 1 d . . . . .

C18B C -0.2649(3) 0.4875(4) 0.61275(14) 0.0339(10) Uani 1 1 d . . . . .

H18B H -0.3119 0.4602 0.6369 0.041 Uiso 1 1 calc R U . . .

C19B C -0.0725(3) 0.1497(4) 0.61881(13) 0.0301(9) Uani 1 1 d . . . . .

C20B C 0.2331(3) 0.8401(4) 0.62173(14) 0.0332(10) Uani 1 1 d . . . . .

H20B H 0.1969 0.9189 0.6379 0.040 Uiso 1 1 calc R U . . .

C21B C 0.3600(3) 0.8535(4) 0.61749(16) 0.0386(11) Uani 1 1 d . . . . .

H21B H 0.4083 0.9096 0.6350 0.046 Uiso 1 1 calc R U . . .

C22B C 0.1975(3) 0.8284(4) 0.57394(13) 0.0301(9) Uani 1 1 d . . . . .

H22C H 0.2123 0.9176 0.5579 0.036 Uiso 1 1 calc R U . . .

H22D H 0.1194 0.7981 0.5695 0.036 Uiso 1 1 calc R U . . .

C23B C -0.3383(3) 0.4816(4) 0.57182(16) 0.0374(11) Uani 1 1 d . . . . .

C24B C 0.2825(3) 0.7044(4) 0.56515(13) 0.0259(8) Uani 1 1 d . . . . .

H24B H 0.2869 0.6729 0.5351 0.031 Uiso 1 1 calc R U . . .

C25B C 0.3897(3) 0.7722(4) 0.58484(15) 0.0359(10) Uani 1 1 d . . . . .

H25B H 0.4629 0.7585 0.5754 0.043 Uiso 1 1 calc R U . . .

C26B C -0.0234(3) 0.1570(6) 0.66498(15) 0.0514(14) Uani 1 1 d . . . . .

H26D H 0.0421 0.0950 0.6675 0.077 Uiso 1 1 calc R U . . .

H26E H -0.0792 0.1240 0.6846 0.077 Uiso 1 1 calc R U . . .

H26F H -0.0025 0.2567 0.6717 0.077 Uiso 1 1 calc R U . . .

C27B C -0.0999(4) -0.0089(5) 0.6068(2) 0.0595(15) Uani 1 1 d . . . . .

H27D H -0.1330 -0.0121 0.5783 0.089 Uiso 1 1 calc R U . . .

H27E H -0.1523 -0.0484 0.6268 0.089 Uiso 1 1 calc R U . . .

H27F H -0.0317 -0.0662 0.6078 0.089 Uiso 1 1 calc R U . . .

C28B C 0.5345(3) 0.1170(5) 0.53856(13) 0.0369(10) Uani 1 1 d . . . . .

H28D H 0.5303 0.0119 0.5361 0.055 Uiso 1 1 calc R U . . .

H28E H 0.6076 0.1445 0.5501 0.055 Uiso 1 1 calc R U . . .

H28F H 0.5231 0.1608 0.5107 0.055 Uiso 1 1 calc R U . . .

C29B C -0.2232(4) 0.6443(5) 0.62013(15) 0.0455(12) Uani 1 1 d . . . . .

H29D H -0.1891 0.6522 0.6484 0.068 Uiso 1 1 calc R U . . .

H29E H -0.2859 0.7112 0.6175 0.068 Uiso 1 1 calc R U . . .

H29F H -0.1683 0.6685 0.5991 0.068 Uiso 1 1 calc R U . . .

C30B C 0.4617(4) 0.3020(6) 0.68121(14) 0.0481(13) Uani 1 1 d . . . . .

H30D H 0.5206 0.2340 0.6893 0.072 Uiso 1 1 calc R U . . .

H30E H 0.4097 0.3108 0.7042 0.072 Uiso 1 1 calc R U . . .

H30F H 0.4942 0.3966 0.6755 0.072 Uiso 1 1 calc R U . . .

loop_

_atom_site_aniso_label

_atom_site_aniso_U_11

_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

S1A 0.0302(5) 0.0379(6) 0.0217(5) -0.0027(4) -0.0024(4) 0.0097(5)

O1A 0.0199(13) 0.0236(14) 0.0272(14) 0.0029(11) 0.0001(10) -0.0024(11)

O2A 0.0196(13) 0.0488(18) 0.0292(14) 0.0050(13) -0.0031(11) 0.0012(12)

O3A 0.0167(12) 0.0242(14) 0.0311(14) -0.0009(11) 0.0026(10) 0.0023(11)

O4A 0.0175(14) 0.0365(16) 0.0503(18) -0.0042(14) 0.0028(12) -0.0001(12)

O5A 0.0244(14) 0.0453(18) 0.0369(16) 0.0106(14) 0.0007(12) -0.0050(13)

O6A 0.0305(17) 0.050(2) 0.078(2) -0.0174(18) 0.0065(15) -0.0154(15)

N1A 0.0160(14) 0.0264(17) 0.0217(15) 0.0016(13) 0.0009(11) 0.0020(12)

N2A 0.0152(14) 0.0228(17) 0.0306(17) 0.0027(14) 0.0028(12) -0.0011(13)

N3A 0.0159(14) 0.0187(16) 0.0264(16) 0.0016(13) 0.0010(12) 0.0002(12)

N4A 0.0204(15) 0.0220(17) 0.0328(18) 0.0010(14) 0.0029(13) -0.0080(13)

N5A 0.0153(14) 0.0341(18) 0.0343(18) 0.0002(16) 0.0048(13) 0.0028(14)

N6A 0.0283(18) 0.043(2) 0.037(2) -0.0093(17) -0.0010(15) -0.0078(16)

C1A 0.026(2) 0.037(2) 0.026(2) -0.0034(18) 0.0029(16) 0.0094(18)

C2A 0.0240(18) 0.033(2) 0.0216(19) -0.0016(17) 0.0021(15) 0.0079(17)

C3A 0.0232(18) 0.024(2) 0.029(2) 0.0025(17) 0.0046(15) 0.0053(16)

C4A 0.028(2) 0.031(2) 0.0208(19) 0.0003(17) 0.0064(15) 0.0075(17)

C5A 0.031(2) 0.031(2) 0.032(2) -0.0044(18) -0.0012(17) 0.0018(17)

C6A 0.039(2) 0.040(3) 0.034(2) 0.008(2) 0.0046(18) -0.005(2)

C7A 0.036(2) 0.043(3) 0.026(2) 0.0043(19) 0.0044(17) 0.0012(19)

C8A 0.0231(18) 0.025(2) 0.024(2) 0.0028(16) 0.0009(15) 0.0038(16)

C9A 0.0206(17) 0.0172(18) 0.0198(18) -0.0002(14) 0.0006(14) -0.0008(14)

C10A 0.0198(18) 0.028(2) 0.0246(19) -0.0066(16) 0.0020(14) -0.0034(16)

C11A 0.0224(18) 0.0168(18) 0.0198(18) -0.0013(14) 0.0010(14) 0.0004(14)

C12A 0.0212(18) 0.0233(19) 0.0249(19) 0.0091(16) 0.0013(15) 0.0001(15)

C13A 0.0254(18) 0.028(2) 0.0194(18) 0.0027(16) -0.0043(14) 0.0011(16)

C14A 0.039(2) 0.054(3) 0.023(2) 0.010(2) 0.0044(17) 0.015(2)

C15A 0.0224(18) 0.0203(19) 0.0187(18) 0.0020(15) 0.0001(14) 0.0034(15)

C16A 0.0177(18) 0.022(2) 0.040(2) -0.0058(17) 0.0045(16) -0.0022(15)

C17A 0.026(2) 0.027(2) 0.035(2) 0.0064(18) 0.0035(17) -0.0037(17)

C18A 0.033(2) 0.032(2) 0.031(2) -0.0042(19) 0.0123(17) -0.0134(19)

C19A 0.0207(19) 0.022(2) 0.033(2) 0.0020(17) 0.0028(15) 0.0015(15)

C20A 0.033(2) 0.023(2) 0.031(2) 0.0108(17) 0.0073(16) 0.0055(17)

C21A 0.033(2) 0.024(2) 0.037(2) 0.0058(18) -0.0034(17) 0.0073(17)

C22A 0.028(2) 0.0196(19) 0.037(2) 0.0024(17) 0.0019(16) 0.0020(16)

C23A 0.029(2) 0.030(2) 0.044(3) -0.002(2) 0.0056(18) -0.0128(18)

C24A 0.0242(19) 0.0200(19) 0.027(2) 0.0002(15) 0.0021(15) 0.0019(15)

C25A 0.0239(19) 0.0189(19) 0.039(2) -0.0007(17) 0.0036(16) 0.0060(16)

C26A 0.0233(19) 0.025(2) 0.045(2) -0.0084(18) 0.0055(17) -0.0055(17)

C27A 0.031(2) 0.033(2) 0.056(3) 0.012(2) 0.0114(19) 0.0096(19)

C28A 0.036(2) 0.048(3) 0.034(2) 0.003(2) 0.0119(18) 0.001(2)

C29A 0.065(3) 0.044(3) 0.039(3) 0.007(2) 0.004(2) -0.020(3)

C30A 0.025(2) 0.056(3) 0.049(3) -0.023(2) 0.0039(19) -0.009(2)

S1B 0.0327(6) 0.0463(7) 0.0333(6) -0.0136(5) -0.0087(4) 0.0104(5)

O1B 0.0155(12) 0.0200(13) 0.0293(14) 0.0004(11) -0.0010(10) 0.0030(10)

O2B 0.0172(13) 0.0537(19) 0.0413(16) -0.0192(15) -0.0043(11) -0.0022(13)

O3B 0.0159(12) 0.0315(15) 0.0299(15) 0.0059(12) 0.0017(10) -0.0012(11)

O4B 0.0161(12) 0.0221(14) 0.0386(15) 0.0065(11) 0.0035(11) -0.0009(10)

O5B 0.0214(13) 0.0269(15) 0.0475(17) -0.0014(13) -0.0090(12) -0.0004(11)

O6B 0.0250(16) 0.046(2) 0.111(3) 0.026(2) 0.0187(17) 0.0146(15)

N1B 0.0141(14) 0.0177(15) 0.0237(16) -0.0036(12) -0.0011(11) 0.0018(12)

N2B 0.0144(14) 0.0201(16) 0.0383(18) 0.0010(14) 0.0018(12) -0.0009(12)

N3B 0.0122(14) 0.0142(15) 0.0328(17) 0.0007(13) 0.0019(12) 0.0021(12)

N4B 0.0208(15) 0.0152(16) 0.0323(18) 0.0036(13) 0.0052(13) -0.0002(12)

N5B 0.0168(14) 0.0213(16) 0.0334(17) 0.0003(14) -0.0019(12) 0.0028(13)

N6B 0.0242(17) 0.046(2) 0.045(2) 0.0121(18) -0.0051(16) -0.0001(16)

C1B 0.046(3) 0.072(4) 0.037(3) 0.000(2) -0.002(2) 0.008(3)

C2B 0.056(3) 0.117(5) 0.021(2) 0.003(3) 0.002(2) 0.021(3)

C3B 0.070(4) 0.103(5) 0.070(4) 0.008(4) 0.001(3) 0.006(4)

C4B 0.089(5) 0.126(7) 0.066(4) -0.038(4) -0.020(4) 0.047(5)

C5B 0.071(5) 0.155(8) 0.059(4) 0.012(5) 0.013(3) 0.039(5)

C6B 0.080(5) 0.161(9) 0.150(8) -0.088(7) 0.001(5) 0.040(5)

C7B 0.072(4) 0.147(8) 0.099(5) -0.067(5) -0.007(4) 0.027(5)

C8B 0.0222(18) 0.022(2) 0.031(2) -0.0074(16) -0.0055(15) 0.0043(15)

C9B 0.0137(16) 0.0135(17) 0.031(2) -0.0010(15) 0.0012(14) 0.0026(13)

C10B 0.0180(17) 0.0186(18) 0.0271(19) 0.0005(16) -0.0004(14) 0.0011(15)

C11B 0.0199(17) 0.0140(17) 0.0204(17) 0.0044(14) 0.0028(14) 0.0002(14)

C12B 0.0174(17) 0.0197(19) 0.035(2) -0.0106(17) 0.0007(15) 0.0034(15)

C13B 0.0191(17) 0.0175(18) 0.029(2) -0.0045(15) -0.0049(14) 0.0017(15)

C14B 0.0247(19) 0.030(2) 0.029(2) -0.0084(18) 0.0006(15) -0.0003(17)

C15B 0.0184(18) 0.0186(18) 0.0195(18) -0.0027(15) -0.0020(14) 0.0000(14)

C16B 0.0268(19) 0.023(2) 0.033(2) 0.0043(17) 0.0008(16) 0.0080(17)

C17B 0.026(2) 0.0146(18) 0.038(2) 0.0012(16) -0.0015(17) 0.0009(15)

C18B 0.037(2) 0.019(2) 0.048(3) 0.0058(19) 0.0243(19) 0.0052(18)

C19B 0.0168(18) 0.020(2) 0.054(3) 0.0075(19) 0.0056(17) 0.0007(16)

C20B 0.027(2) 0.018(2) 0.054(3) -0.0113(18) -0.0057(18) 0.0049(16)

C21B 0.028(2) 0.016(2) 0.071(3) -0.003(2) -0.012(2) -0.0047(17)

C22B 0.0242(19) 0.0173(19) 0.049(3) 0.0040(18) -0.0006(17) 0.0002(16)

C23B 0.0196(19) 0.022(2) 0.071(3) 0.019(2) 0.008(2) 0.0028(17)

C24B 0.0239(18) 0.0151(18) 0.039(2) 0.0016(16) 0.0051(16) -0.0008(15)

C25B 0.023(2) 0.015(2) 0.070(3) 0.004(2) 0.005(2) -0.0022(16)

C26B 0.021(2) 0.076(4) 0.057(3) 0.043(3) -0.0009(19) 0.001(2)

C27B 0.038(3) 0.014(2) 0.128(5) 0.002(3) 0.028(3) -0.001(2)

C28B 0.026(2) 0.042(3) 0.043(2) -0.008(2) 0.0016(18) -0.0046(19)

C29B 0.063(3) 0.021(2) 0.054(3) -0.004(2) 0.028(2) 0.002(2)

C30B 0.040(2) 0.070(3) 0.034(2) -0.006(2) -0.0096(19) 0.032(2)

_geom_special_details

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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.

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loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

S1A C8A 1.806(4) . ?

S1A C1A 1.823(4) . ?

O1A C11A 1.243(4) . ?

O2A C12A 1.229(4) . ?

O3A C15A 1.232(4) . ?

O4A C10A 1.222(4) . ?

O5A C17A 1.221(4) . ?

O6A C23A 1.221(5) . ?

N1A C11A 1.332(4) . ?

N1A C13A 1.462(4) . ?

N1A HN1A 0.8600 . ?

N2A C12A 1.355(5) . ?

N2A C19A 1.473(5) . ?

N2A HN2A 0.8600 . ?

N3A C15A 1.352(5) . ?

N3A C9A 1.451(4) . ?

N3A HN3A 0.8600 . ?

N4A C10A 1.341(5) . ?

N4A C18A 1.456(5) . ?

N4A HN4A 0.8600 . ?

N5A C17A 1.338(5) . ?

N5A C16A 1.460(5) . ?

N5A HN5A 0.8600 . ?

N6A C23A 1.334(5) . ?

N6A HN6A1 0.8600 . ?

N6A HN6A2 0.8600 . ?

C1A C2A 1.497(5) . ?

C1A H1A1 0.9700 . ?

C1A H1A2 0.9700 . ?

C2A C3A 1.389(5) . ?

C2A C7A 1.392(6) . ?

C3A C4A 1.375(5) . ?

C3A H3A 0.9300 . ?

C4A C5A 1.387(6) . ?

C4A H4A 0.9300 . ?

C5A C6A 1.388(5) . ?

C5A H5A 0.9300 . ?

C6A C7A 1.365(6) . ?

C6A H6A 0.9300 . ?

C7A H7A 0.9300 . ?

C8A C20A 1.557(5) . ?

C8A C9A 1.587(5) . ?

C8A H8A 0.9800 . ?

C9A C11A 1.537(5) . ?

C9A C24A 1.584(5) . ?

C10A C19A 1.543(5) . ?

C12A C13A 1.520(5) . ?

C13A C14A 1.529(5) . ?

C13A H13A 0.9800 . ?

C14A H14A 0.9600 . ?

C14A H14B 0.9600 . ?

C14A H14C 0.9600 . ?

C15A C16A 1.522(5) . ?

C16A C30A 1.513(5) . ?

C16A H16A 0.9800 . ?

C17A C28A 1.516(5) . ?

C18A C29A 1.509(6) . ?

C18A C23A 1.530(6) . ?

C18A H18A 0.9800 . ?

C19A C26A 1.515(5) . ?

C19A C27A 1.525(5) . ?

C20A C21A 1.509(5) . ?

C20A C22A 1.535(5) . ?

C20A H20A 0.9800 . ?

C21A C25A 1.329(5) . ?

C21A H21A 0.9300 . ?

C22A C24A 1.527(5) . ?

C22A H22A 0.9700 . ?

C22A H22B 0.9700 . ?

C24A C25A 1.519(5) . ?

C24A H24A 0.9800 . ?

C25A H25A 0.9300 . ?

C26A H26A 0.9600 . ?

C26A H26B 0.9600 . ?

C26A H26C 0.9600 . ?

C27A H27A 0.9600 . ?

C27A H27B 0.9600 . ?

C27A H27C 0.9600 . ?

C28A H28A 0.9600 . ?

C28A H28B 0.9600 . ?

C28A H28C 0.9600 . ?

C29A H29A 0.9600 . ?

C29A H29B 0.9600 . ?

C29A H29C 0.9600 . ?

C30A H30A 0.9600 . ?

C30A H30B 0.9600 . ?

C30A H30C 0.9600 . ?

S1B C1B 1.776(5) . ?

S1B C8B 1.813(4) . ?

O1B C11B 1.243(4) . ?

O2B C12B 1.234(4) . ?

O3B C15B 1.233(4) . ?

O4B C10B 1.232(4) . ?

O5B C17B 1.236(4) . ?

O6B C23B 1.243(5) . ?

N1B C11B 1.343(4) . ?

N1B C13B 1.446(4) . ?

N1B HN1B 0.8600 . ?

N2B C12B 1.331(5) . ?

N2B C19B 1.467(5) . ?

N2B HN2B 0.8600 . ?

N3B C15B 1.342(4) . ?

N3B C9B 1.456(4) . ?

N3B HN3B 0.8600 . ?

N4B C10B 1.342(5) . ?

N4B C18B 1.462(5) . ?

N4B HN4B 0.8600 . ?

N5B C17B 1.343(5) . ?

N5B C16B 1.458(5) . ?

N5B HN5B 0.8600 . ?

N6B C23B 1.317(6) . ?

N6B HN6B1 0.8600 . ?

N6B HN6B2 0.8600 . ?

C1B C2B 1.539(7) . ?

C1B H1B1 0.9700 . ?

C1B H1B2 0.9700 . ?

C2B C3B 1.288(7) . ?

C2B C7B 1.433(9) . ?

C3B C4B 1.392(9) . ?

C3B H3B 0.9300 . ?

C4B C5B 1.330(11) . ?

C4B H4B 0.9300 . ?

C5B C6B 1.387(10) . ?

C5B H5B 0.9300 . ?

C6B C7B 1.384(9) . ?

C6B H6B 0.9300 . ?

C7B H7B 0.9300 . ?

C8B C20B 1.546(6) . ?

C8B C9B 1.585(5) . ?

C8B H8B 0.9800 . ?

C9B C11B 1.537(5) . ?

C9B C24B 1.567(5) . ?

C10B C19B 1.539(5) . ?

C12B C13B 1.528(5) . ?

C13B C14B 1.525(5) . ?

C13B H13B 0.9800 . ?

C14B H14D 0.9600 . ?

C14B H14E 0.9600 . ?

C14B H14F 0.9600 . ?

C15B C16B 1.519(5) . ?

C16B C30B 1.513(6) . ?

C16B H16B 0.9800 . ?

C17B C28B 1.506(5) . ?

C18B C23B 1.522(6) . ?

C18B C29B 1.523(6) . ?

C18B H18B 0.9800 . ?

C19B C26B 1.535(6) . ?

C19B C27B 1.521(6) . ?

C20B C21B 1.516(6) . ?

C20B C22B 1.537(6) . ?

C20B H20B 0.9800 . ?

C21B C25B 1.312(6) . ?

C21B H21B 0.9300 . ?

C22B C24B 1.540(5) . ?

C22B H22C 0.9700 . ?

C22B H22D 0.9700 . ?

C24B C25B 1.522(5) . ?

C24B H24B 0.9800 . ?

C25B H25B 0.9300 . ?

C26B H26D 0.9600 . ?

C26B H26E 0.9600 . ?

C26B H26F 0.9600 . ?

C27B H27D 0.9600 . ?

C27B H27E 0.9600 . ?

C27B H27F 0.9600 . ?

C28B H28D 0.9600 . ?

C28B H28E 0.9600 . ?

C28B H28F 0.9600 . ?

C29B H29D 0.9600 . ?

C29B H29E 0.9600 . ?

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C13A N1A HN1A 119.9 . . ?

C12A N2A C19A 121.6(3) . . ?

C12A N2A HN2A 119.2 . . ?

C19A N2A HN2A 119.2 . . ?

C15A N3A C9A 123.7(3) . . ?

C15A N3A HN3A 118.2 . . ?

C9A N3A HN3A 118.2 . . ?

C10A N4A C18A 121.7(3) . . ?

C10A N4A HN4A 119.2 . . ?

C18A N4A HN4A 119.2 . . ?

C17A N5A C16A 121.5(3) . . ?

C17A N5A HN5A 119.3 . . ?

C16A N5A HN5A 119.3 . . ?

C23A N6A HN6A1 120.0 . . ?

C23A N6A HN6A2 120.0 . . ?

HN6A1 N6A HN6A2 120.0 . . ?

C2A C1A S1A 110.3(2) . . ?

C2A C1A H1A1 109.6 . . ?

S1A C1A H1A1 109.6 . . ?

C2A C1A H1A2 109.6 . . ?

S1A C1A H1A2 109.6 . . ?

H1A1 C1A H1A2 108.1 . . ?

C3A C2A C7A 117.9(4) . . ?

C3A C2A C1A 120.6(4) . . ?

C7A C2A C1A 121.6(3) . . ?

C4A C3A C2A 121.2(4) . . ?

C4A C3A H3A 119.4 . . ?

C2A C3A H3A 119.4 . . ?

C3A C4A C5A 120.2(3) . . ?

C3A C4A H4A 119.9 . . ?

C5A C4A H4A 119.9 . . ?

C4A C5A C6A 118.9(4) . . ?

C4A C5A H5A 120.6 . . ?

C6A C5A H5A 120.6 . . ?

C7A C6A C5A 120.6(4) . . ?

C7A C6A H6A 119.7 . . ?

C5A C6A H6A 119.7 . . ?

C6A C7A C2A 121.2(4) . . ?

C6A C7A H7A 119.4 . . ?

C2A C7A H7A 119.4 . . ?

C20A C8A C9A 102.6(3) . . ?

C20A C8A S1A 112.5(3) . . ?

C9A C8A S1A 117.5(3) . . ?

C20A C8A H8A 107.9 . . ?

C9A C8A H8A 107.9 . . ?

S1A C8A H8A 107.9 . . ?

N3A C9A C11A 110.1(3) . . ?

N3A C9A C24A 109.5(3) . . ?

C11A C9A C24A 108.4(3) . . ?

N3A C9A C8A 116.7(3) . . ?

C11A C9A C8A 110.1(3) . . ?

C24A C9A C8A 101.4(3) . . ?

O4A C10A N4A 122.9(3) . . ?

O4A C10A C19A 121.1(3) . . ?

N4A C10A C19A 115.7(3) . . ?

O1A C11A N1A 121.1(3) . . ?

O1A C11A C9A 121.0(3) . . ?

N1A C11A C9A 117.8(3) . . ?

O2A C12A N2A 122.2(3) . . ?

O2A C12A C13A 118.7(3) . . ?

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N1A C13A C12A 114.3(3) . . ?

N1A C13A C14A 109.4(3) . . ?

C12A C13A C14A 111.1(3) . . ?

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C13A C14A H14B 109.5 . . ?

H14A C14A H14B 109.5 . . ?

C13A C14A H14C 109.5 . . ?

H14A C14A H14C 109.5 . . ?

H14B C14A H14C 109.5 . . ?

O3A C15A N3A 122.7(3) . . ?

O3A C15A C16A 120.1(3) . . ?

N3A C15A C16A 117.3(3) . . ?

N5A C16A C30A 109.3(3) . . ?

N5A C16A C15A 112.4(3) . . ?

C30A C16A C15A 110.7(3) . . ?

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C30A C16A H16A 108.1 . . ?

C15A C16A H16A 108.1 . . ?

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O5A C17A C28A 122.0(4) . . ?

N5A C17A C28A 115.8(3) . . ?

N4A C18A C29A 110.6(3) . . ?

N4A C18A C23A 112.2(3) . . ?

C29A C18A C23A 110.3(4) . . ?

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C29A C18A H18A 107.9 . . ?

C23A C18A H18A 107.9 . . ?

N2A C19A C26A 107.9(3) . . ?

N2A C19A C27A 112.6(3) . . ?

C26A C19A C27A 109.8(3) . . ?

N2A C19A C10A 109.1(3) . . ?

C26A C19A C10A 107.1(3) . . ?

C27A C19A C10A 110.2(3) . . ?

C21A C20A C22A 100.2(3) . . ?

C21A C20A C8A 106.7(3) . . ?

C22A C20A C8A 100.8(3) . . ?

C21A C20A H20A 115.7 . . ?

C22A C20A H20A 115.7 . . ?

C8A C20A H20A 115.7 . . ?

C25A C21A C20A 107.8(3) . . ?

C25A C21A H21A 126.1 . . ?

C20A C21A H21A 126.1 . . ?

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C24A C22A H22A 112.9 . . ?

C20A C22A H22A 112.9 . . ?

C24A C22A H22B 112.9 . . ?

C20A C22A H22B 112.9 . . ?

H22A C22A H22B 110.3 . . ?

O6A C23A N6A 123.9(4) . . ?

O6A C23A C18A 120.2(4) . . ?

N6A C23A C18A 115.9(3) . . ?

C25A C24A C22A 100.5(3) . . ?

C25A C24A C9A 105.7(3) . . ?

C22A C24A C9A 100.7(3) . . ?

C25A C24A H24A 115.9 . . ?

C22A C24A H24A 115.9 . . ?

C9A C24A H24A 115.9 . . ?

C21A C25A C24A 107.3(3) . . ?

C21A C25A H25A 126.4 . . ?

C24A C25A H25A 126.4 . . ?

C19A C26A H26A 109.5 . . ?

C19A C26A H26B 109.5 . . ?

H26A C26A H26B 109.5 . . ?

C19A C26A H26C 109.5 . . ?

H26A C26A H26C 109.5 . . ?

H26B C26A H26C 109.5 . . ?

C19A C27A H27A 109.5 . . ?

C19A C27A H27B 109.5 . . ?

H27A C27A H27B 109.5 . . ?

C19A C27A H27C 109.5 . . ?

H27A C27A H27C 109.5 . . ?

H27B C27A H27C 109.5 . . ?

C17A C28A H28A 109.5 . . ?

C17A C28A H28B 109.5 . . ?

H28A C28A H28B 109.5 . . ?

C17A C28A H28C 109.5 . . ?

H28A C28A H28C 109.5 . . ?

H28B C28A H28C 109.5 . . ?

C18A C29A H29A 109.5 . . ?

C18A C29A H29B 109.5 . . ?

H29A C29A H29B 109.5 . . ?

C18A C29A H29C 109.5 . . ?

H29A C29A H29C 109.5 . . ?

H29B C29A H29C 109.5 . . ?

C16A C30A H30A 109.5 . . ?

C16A C30A H30B 109.5 . . ?

H30A C30A H30B 109.5 . . ?

C16A C30A H30C 109.5 . . ?

H30A C30A H30C 109.5 . . ?

H30B C30A H30C 109.5 . . ?

C1B S1B C8B 102.4(2) . . ?

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C15B N3B C9B 119.7(3) . . ?

C15B N3B HN3B 120.2 . . ?

C9B N3B HN3B 120.2 . . ?

C10B N4B C18B 120.6(3) . . ?

C10B N4B HN4B 119.7 . . ?

C18B N4B HN4B 119.7 . . ?

C17B N5B C16B 121.9(3) . . ?

C17B N5B HN5B 119.0 . . ?

C16B N5B HN5B 119.0 . . ?

C23B N6B HN6B1 120.0 . . ?

C23B N6B HN6B2 120.0 . . ?

HN6B1 N6B HN6B2 120.0 . . ?

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S1B C1B H1B1 109.1 . . ?

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H1B1 C1B H1B2 107.8 . . ?

C3B C2B C7B 116.1(6) . . ?

C3B C2B C1B 126.1(6) . . ?

C7B C2B C1B 117.7(5) . . ?

C2B C3B C4B 126.0(7) . . ?

C2B C3B H3B 117.0 . . ?

C4B C3B H3B 117.0 . . ?

C5B C4B C3B 117.0(7) . . ?

C5B C4B H4B 121.5 . . ?

C3B C4B H4B 121.5 . . ?

C4B C5B C6B 121.7(7) . . ?

C4B C5B H5B 119.1 . . ?

C6B C5B H5B 119.1 . . ?

C7B C6B C5B 118.1(8) . . ?

C7B C6B H6B 121.0 . . ?

C5B C6B H6B 121.0 . . ?

C6B C7B C2B 120.3(6) . . ?

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N3B C9B C11B 108.5(3) . . ?

N3B C9B C24B 112.1(3) . . ?

C11B C9B C24B 107.2(3) . . ?

N3B C9B C8B 115.6(3) . . ?

C11B C9B C8B 111.5(3) . . ?

C24B C9B C8B 101.5(3) . . ?

O4B C10B N4B 122.3(3) . . ?

O4B C10B C19B 121.3(3) . . ?

N4B C10B C19B 116.1(3) . . ?

O1B C11B N1B 121.6(3) . . ?

O1B C11B C9B 122.6(3) . . ?

N1B C11B C9B 115.6(3) . . ?

O2B C12B N2B 123.1(4) . . ?

O2B C12B C13B 118.8(3) . . ?

N2B C12B C13B 118.1(3) . . ?

N1B C13B C14B 109.2(3) . . ?

N1B C13B C12B 114.3(3) . . ?

C14B C13B C12B 109.3(3) . . ?

N1B C13B H13B 107.9 . . ?

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H14D C14B H14E 109.5 . . ?

C13B C14B H14F 109.5 . . ?

H14D C14B H14F 109.5 . . ?

H14E C14B H14F 109.5 . . ?

O3B C15B N3B 121.5(3) . . ?

O3B C15B C16B 119.5(3) . . ?

N3B C15B C16B 118.9(3) . . ?

N5B C16B C30B 110.4(3) . . ?

N5B C16B C15B 114.6(3) . . ?

C30B C16B C15B 109.4(3) . . ?

N5B C16B H16B 107.4 . . ?

C30B C16B H16B 107.4 . . ?

C15B C16B H16B 107.4 . . ?

O5B C17B N5B 121.8(3) . . ?

O5B C17B C28B 121.3(3) . . ?

N5B C17B C28B 116.9(3) . . ?

N4B C18B C23B 114.2(3) . . ?

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C23B C18B C29B 109.5(3) . . ?

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C23B C18B H18B 108.0 . . ?

C29B C18B H18B 108.0 . . ?

N2B C19B C27B 111.8(4) . . ?

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C27B C19B C26B 110.0(4) . . ?

N2B C19B C10B 109.7(3) . . ?

C27B C19B C10B 110.0(3) . . ?

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C22B C20B C8B 101.5(3) . . ?

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C22B C20B H20B 115.7 . . ?

C8B C20B H20B 115.7 . . ?

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C25B C21B H21B 126.0 . . ?

C20B C21B H21B 126.0 . . ?

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C20B C22B H22C 113.0 . . ?

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C24B C22B H22D 113.0 . . ?

H22C C22B H22D 110.4 . . ?

O6B C23B N6B 122.5(4) . . ?

O6B C23B C18B 118.6(4) . . ?

N6B C23B C18B 118.9(3) . . ?

C25B C24B C22B 100.0(3) . . ?

C25B C24B C9B 106.4(3) . . ?

C22B C24B C9B 100.8(3) . . ?

C25B C24B H24B 115.8 . . ?

C22B C24B H24B 115.8 . . ?

C9B C24B H24B 115.8 . . ?

C21B C25B C24B 107.5(3) . . ?

C21B C25B H25B 126.2 . . ?

C24B C25B H25B 126.2 . . ?

C19B C26B H26D 109.5 . . ?

C19B C26B H26E 109.5 . . ?

H26D C26B H26E 109.5 . . ?

C19B C26B H26F 109.5 . . ?

H26D C26B H26F 109.5 . . ?

H26E C26B H26F 109.5 . . ?

C19B C27B H27D 109.5 . . ?

C19B C27B H27E 109.5 . . ?

H27D C27B H27E 109.5 . . ?

C19B C27B H27F 109.5 . . ?

H27D C27B H27F 109.5 . . ?

H27E C27B H27F 109.5 . . ?

C17B C28B H28D 109.5 . . ?

C17B C28B H28E 109.5 . . ?

H28D C28B H28E 109.5 . . ?

C17B C28B H28F 109.5 . . ?

H28D C28B H28F 109.5 . . ?

H28E C28B H28F 109.5 . . ?

C18B C29B H29D 109.5 . . ?

C18B C29B H29E 109.5 . . ?

H29D C29B H29E 109.5 . . ?

C18B C29B H29F 109.5 . . ?

H29D C29B H29F 109.5 . . ?

H29E C29B H29F 109.5 . . ?

C16B C30B H30D 109.5 . . ?

C16B C30B H30E 109.5 . . ?

H30D C30B H30E 109.5 . . ?

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H30D C30B H30F 109.5 . . ?

H30E C30B H30F 109.5 . . ?

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C1A C2A C7A C6A 177.4(4) . . . . ?

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C1A S1A C8A C9A -102.4(3) . . . . ?

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C20A C8A C9A N3A 118.5(3) . . . . ?

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S1A C8A C9A C11A 121.0(3) . . . . ?

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S1A C8A C9A C24A -124.4(3) . . . . ?

C18A N4A C10A O4A -5.6(5) . . . . ?

C18A N4A C10A C19A -179.3(3) . . . . ?

C13A N1A C11A O1A 0.5(5) . . . . ?

C13A N1A C11A C9A -175.8(3) . . . . ?

N3A C9A C11A O1A 151.7(3) . . . . ?

C24A C9A C11A O1A -88.5(4) . . . . ?

C8A C9A C11A O1A 21.7(4) . . . . ?

N3A C9A C11A N1A -32.0(4) . . . . ?

C24A C9A C11A N1A 87.8(4) . . . . ?

C8A C9A C11A N1A -162.1(3) . . . . ?

C19A N2A C12A O2A -7.2(5) . . . . ?

C19A N2A C12A C13A 171.4(3) . . . . ?

C11A N1A C13A C12A -65.3(4) . . . . ?

C11A N1A C13A C14A 169.4(3) . . . . ?

O2A C12A C13A N1A 161.5(3) . . . . ?

N2A C12A C13A N1A -17.1(5) . . . . ?

O2A C12A C13A C14A -74.1(4) . . . . ?

N2A C12A C13A C14A 107.2(4) . . . . ?

C9A N3A C15A O3A -1.7(5) . . . . ?

C9A N3A C15A C16A 179.0(3) . . . . ?

C17A N5A C16A C30A 179.3(4) . . . . ?

C17A N5A C16A C15A -57.4(5) . . . . ?

O3A C15A C16A N5A 147.9(3) . . . . ?

N3A C15A C16A N5A -32.9(5) . . . . ?

O3A C15A C16A C30A -89.6(4) . . . . ?

N3A C15A C16A C30A 89.7(4) . . . . ?

C16A N5A C17A O5A 5.7(6) . . . . ?

C16A N5A C17A C28A -176.8(4) . . . . ?

C10A N4A C18A C29A 154.7(4) . . . . ?

C10A N4A C18A C23A -81.7(4) . . . . ?

C12A N2A C19A C26A -171.4(3) . . . . ?

C12A N2A C19A C27A 67.3(4) . . . . ?

C12A N2A C19A C10A -55.4(4) . . . . ?

O4A C10A C19A N2A 145.1(3) . . . . ?

N4A C10A C19A N2A -41.1(4) . . . . ?

O4A C10A C19A C26A -98.5(4) . . . . ?

N4A C10A C19A C26A 75.3(4) . . . . ?

O4A C10A C19A C27A 20.9(5) . . . . ?

N4A C10A C19A C27A -165.3(3) . . . . ?

C9A C8A C20A C21A -67.3(3) . . . . ?

S1A C8A C20A C21A 60.0(3) . . . . ?

C9A C8A C20A C22A 36.9(3) . . . . ?

S1A C8A C20A C22A 164.2(2) . . . . ?

C22A C20A C21A C25A -33.8(4) . . . . ?

C8A C20A C21A C25A 70.8(4) . . . . ?

C21A C20A C22A C24A 50.1(3) . . . . ?

C8A C20A C22A C24A -59.3(3) . . . . ?

N4A C18A C23A O6A 131.4(4) . . . . ?

C29A C18A C23A O6A -104.8(5) . . . . ?

N4A C18A C23A N6A -48.0(5) . . . . ?

C29A C18A C23A N6A 75.7(5) . . . . ?

C20A C22A C24A C25A -49.5(3) . . . . ?

C20A C22A C24A C9A 58.8(3) . . . . ?

N3A C9A C24A C25A -56.1(3) . . . . ?

C11A C9A C24A C25A -176.3(3) . . . . ?

C8A C9A C24A C25A 67.8(3) . . . . ?

N3A C9A C24A C22A -160.4(3) . . . . ?

C11A C9A C24A C22A 79.4(3) . . . . ?

C8A C9A C24A C22A -36.5(3) . . . . ?

C20A C21A C25A C24A 1.0(4) . . . . ?

C22A C24A C25A C21A 32.4(4) . . . . ?

C9A C24A C25A C21A -72.0(4) . . . . ?

C8B S1B C1B C2B -71.1(4) . . . . ?

S1B C1B C2B C3B 115.0(6) . . . . ?

S1B C1B C2B C7B -61.7(6) . . . . ?

C7B C2B C3B C4B -5.0(10) . . . . ?

C1B C2B C3B C4B 178.3(6) . . . . ?

C2B C3B C4B C5B 7.8(11) . . . . ?

C3B C4B C5B C6B -9.2(12) . . . . ?

C4B C5B C6B C7B 8.5(14) . . . . ?

C5B C6B C7B C2B -5.5(14) . . . . ?

C3B C2B C7B C6B 3.9(11) . . . . ?

C1B C2B C7B C6B -179.2(7) . . . . ?

C1B S1B C8B C20B 133.7(3) . . . . ?

C1B S1B C8B C9B -109.9(3) . . . . ?

C15B N3B C9B C11B -53.4(4) . . . . ?

C15B N3B C9B C24B -171.6(3) . . . . ?

C15B N3B C9B C8B 72.7(4) . . . . ?

C20B C8B C9B N3B 122.8(3) . . . . ?

S1B C8B C9B N3B 1.9(4) . . . . ?

C20B C8B C9B C11B -112.7(3) . . . . ?

S1B C8B C9B C11B 126.4(3) . . . . ?

C20B C8B C9B C24B 1.3(3) . . . . ?

S1B C8B C9B C24B -119.7(3) . . . . ?

C18B N4B C10B O4B -3.6(5) . . . . ?

C18B N4B C10B C19B -177.6(3) . . . . ?

C13B N1B C11B O1B 2.9(5) . . . . ?

C13B N1B C11B C9B -172.1(3) . . . . ?

N3B C9B C11B O1B 147.2(3) . . . . ?

C24B C9B C11B O1B -91.5(4) . . . . ?

C8B C9B C11B O1B 18.8(4) . . . . ?

N3B C9B C11B N1B -37.8(4) . . . . ?

C24B C9B C11B N1B 83.4(3) . . . . ?

C8B C9B C11B N1B -166.2(3) . . . . ?

C19B N2B C12B O2B -5.3(5) . . . . ?

C19B N2B C12B C13B 173.8(3) . . . . ?

C11B N1B C13B C14B 174.2(3) . . . . ?

C11B N1B C13B C12B -63.1(4) . . . . ?

O2B C12B C13B N1B 161.2(3) . . . . ?

N2B C12B C13B N1B -17.9(5) . . . . ?

O2B C12B C13B C14B -76.0(4) . . . . ?

N2B C12B C13B C14B 104.8(4) . . . . ?

C9B N3B C15B O3B 3.8(5) . . . . ?

C9B N3B C15B C16B -173.9(3) . . . . ?

C17B N5B C16B C30B -178.3(3) . . . . ?

C17B N5B C16B C15B -54.3(5) . . . . ?

O3B C15B C16B N5B 140.2(3) . . . . ?

N3B C15B C16B N5B -42.0(5) . . . . ?

O3B C15B C16B C30B -95.2(4) . . . . ?

N3B C15B C16B C30B 82.5(4) . . . . ?

C16B N5B C17B O5B 11.2(5) . . . . ?

C16B N5B C17B C28B -168.8(3) . . . . ?

C10B N4B C18B C23B -73.8(4) . . . . ?

C10B N4B C18B C29B 163.5(3) . . . . ?

C12B N2B C19B C26B -166.9(4) . . . . ?

C12B N2B C19B C27B 71.8(4) . . . . ?

C12B N2B C19B C10B -50.4(4) . . . . ?

O4B C10B C19B N2B 151.2(3) . . . . ?

N4B C10B C19B N2B -34.8(4) . . . . ?

O4B C10B C19B C26B -91.7(4) . . . . ?

N4B C10B C19B C26B 82.4(4) . . . . ?

O4B C10B C19B C27B 27.9(5) . . . . ?

N4B C10B C19B C27B -158.0(4) . . . . ?

C9B C8B C20B C21B -68.0(4) . . . . ?

S1B C8B C20B C21B 56.1(4) . . . . ?

C9B C8B C20B C22B 35.7(3) . . . . ?

S1B C8B C20B C22B 159.9(2) . . . . ?

C22B C20B C21B C25B -35.1(4) . . . . ?

C8B C20B C21B C25B 70.1(4) . . . . ?

C21B C20B C22B C24B 51.0(3) . . . . ?

C8B C20B C22B C24B -58.2(3) . . . . ?

N4B C18B C23B O6B 162.3(3) . . . . ?

C29B C18B C23B O6B -75.3(5) . . . . ?

N4B C18B C23B N6B -20.7(5) . . . . ?

C29B C18B C23B N6B 101.6(4) . . . . ?

C20B C22B C24B C25B -50.2(3) . . . . ?

C20B C22B C24B C9B 58.8(3) . . . . ?

N3B C9B C24B C25B -57.7(4) . . . . ?

C11B C9B C24B C25B -176.7(3) . . . . ?

C8B C9B C24B C25B 66.2(3) . . . . ?

N3B C9B C24B C22B -161.6(3) . . . . ?

C11B C9B C24B C22B 79.4(3) . . . . ?

C8B C9B C24B C22B -37.7(3) . . . . ?

C20B C21B C25B C24B 1.6(4) . . . . ?

C22B C24B C25B C21B 32.5(4) . . . . ?

C9B C24B C25B C21B -72.0(4) . . . . ?

_refine_diff_density_max 0.566

_refine_diff_density_min -0.392

_refine_diff_density_rms 0.054

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