Supporting Information

Atropisomeric and Conformational Properties of 6*N*-Benzoyl- and 6*N*-*p*-Tosyl-1,6-benzodiazocines: Comparison with Those of 1,5-Benzodiazepines

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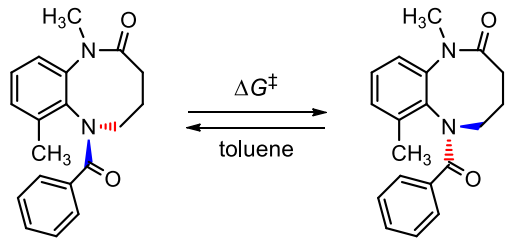
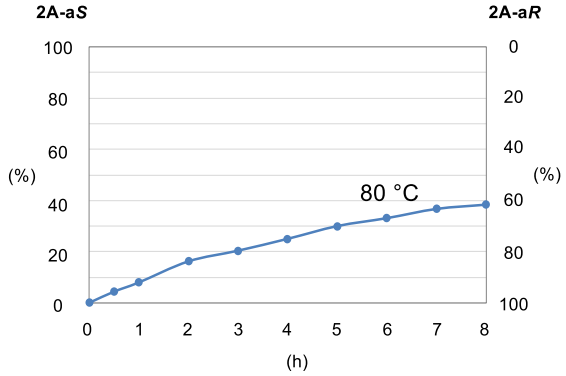
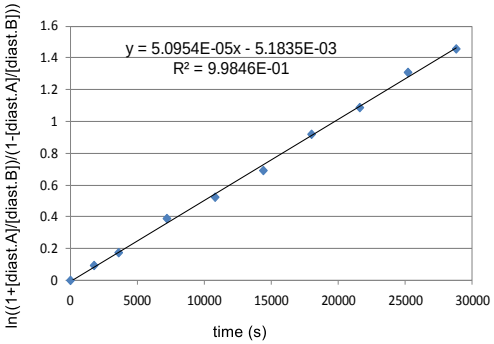
1. Stereochemical stability of 2A and 2B (determination of $\Delta G^{\ddagger}$ value)

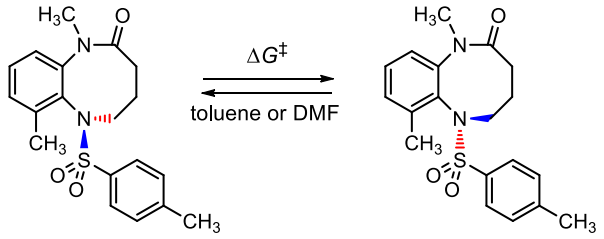


(a*S*)-**2A** (a*R*)-**2A**

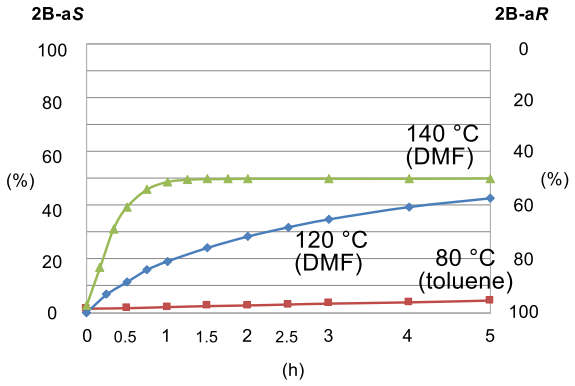


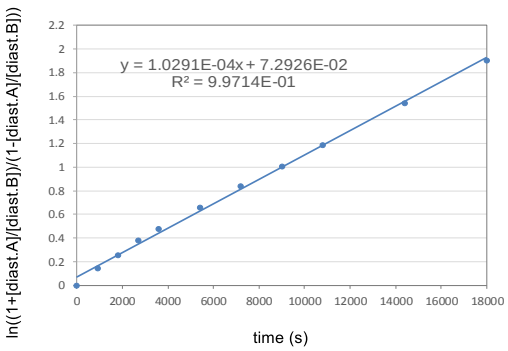


T = 353
k = 1/2 slope = 2.548×10^{-5}
K = kh/*k*T = 3.463×10^{-18}
$\Delta G^{\ddagger}$ = -RTlnK = 118 kJ/mol



(a*S*)-**2B** (a*R*)-**2B**





T = 393
k = 1/2 slope = 0.515×10^{-4}
K = kh/*k*T = 6.288×10^{-18}
$\Delta G^{\ddagger}$ = -RTlnK = 129 kJ/mol

2. Computational study of 1B and 2B.

(a) General computational methodology.

Conformer generations and geometry optimizations were performed for **1B** and **2B**. As for **1B**, the 12 conformers were generated starting from the 2D chemical structures of **1B** using the RDKit[s1] with MMFF94 force feild. Among them, the six conformers with a^1*R* stereochemistry were analyzed to obtain the stable conformations by DFT calculation at 298 K in the gas phase and CH_2Cl_2 solution (SMD solvation model[s2]) using Gaussian 09[s3] at the RB3LYP/6-31+G (d,p) level. No imaginary frequencies were confirmed by vibrational analyses. Similarly, the conformer analysis was performed for **2B**: the 45 conformers of **2B** were generated starting from the 2D chemical structures using the RDKit, and among them, the 22 conformers with a^1*R* stereochemistry were analyzed in detail.

(b) Energy differences between the conformers of 1B (Table S1, Figure S1, and Figure S2).

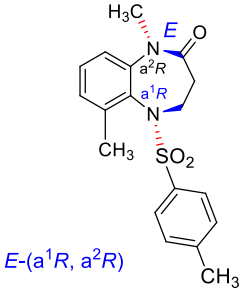
Table S1 shows the analytical data of the conformers of **1B** by DFT calculation, and Figure S1 illustrates the typical conformers of (a^1*R*)-**1B.** The lowest energy conformer in CH_2Cl_2 solution was ID-No7 (the folded form), whose schematic conformation is shown in Figure S2 along with that of ID-No.6 (the extended form), and the X-ray structure of (a^1*R*)-**1B**.

Table S1. Molecular Mechanics and DFT energies of copmpuond **1B** (selected 12 conformers)

1B	*R*/*S*	MMFF94	RB3LYP/6-31+G(d,p)					
			Gas phase (298K)			CH_2Cl_2 solution (298K)		
ID No	at axis1	E / kcal/mol	E / hartree	G / hartree	ΔG / kJ/mol	E / hartree	G / hartree	ΔG / kJ/mol
1	*S*	32.73012274	n.c.*[1]			n.c.		
2	*R*	32.73012276	-1431.341098	-1431.039987	0.00	-1431.37447666	-1431.072330	1.61
3	*R*	36.24419860	-1431.338528	-1431.039498	1.28	-1431.37285796	-1431.071073	4.91
4	*S*	36.24419862	n.c.			n.c.		
5	*S*	37.04962325	n.c.			n.c.		
6	*R*	37.04962326	-1431.338528	-1431.039539	1.18	-1431.37292665	-1431.071811	2.97
7*[2]	*R*	43.53033628	-1431.341098	-1431.039983	0.01	-1431.37442578	-1431.072944	0.00
8	*S*	43.58380145	n.c.			n.c.		
9	*R*	44.64853814	-1431.338528	-1431.039497	1.29	-1431.37283389	-1431.071225	4.51
10	*S*	44.64853815	n.c.			n.c.		
11	*R*	45.61012280	-1431.338528	-1431.039517	1.23	-1431.37292665	-1431.071812	2.97
12	*S*	45.61012282	n.c.			n.c.		

*[1] n.c. = not calculated

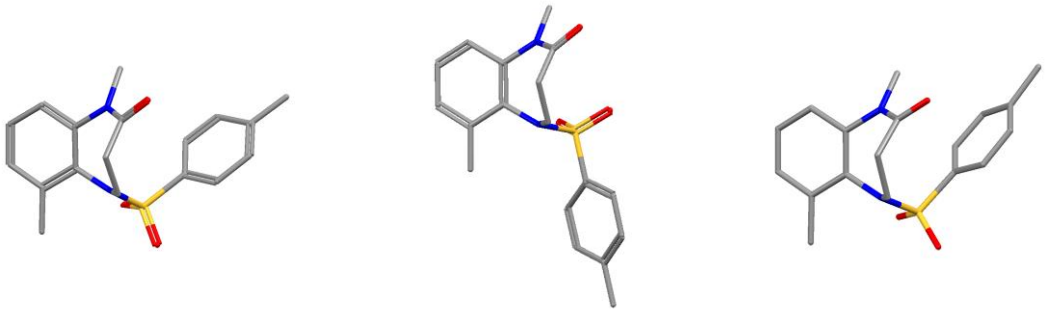
*[2] ID-No.7 is the lowest energy conformer in CH_2Cl_2 solution.



ID-No	*	ΔG kJ/mol	
2	f	1.61	
3	e	4.91	
6	e	2.97	
7	f	0	most stable
9	e	4.51	
11	e	2.97	

* orientation of the tolyl group
f: folded
e: extended

Figure S1. Conformers of (a^1*R*)-**1B in CH_2Cl_2**



ID-No.7
(the lowest energy conformer)

ID-No.6
(Δ*G*, 2.97 kJ/mol)

X-ray structure

Figure S2. Typical conformers of 1B: ID-No.7 (the lowest energy conformer with the folded form), ID-No.6 (the extended form), and the X-ray crystal structure

(c) Energy differences between the conformers of 2B (Table S2, Figure S3, and Figure S4).

Table S2 shows the analytical data of the conformers of **2B** by DFT calculation, and Figure S3 illustrates the typical conformers of (a^1*R*)-**2B.** The lowest energy conformer in CH_2Cl_2 solution was ID No5 (the extended form), whose schematic conformation is shown in Figure S3 along with those of ID-No.2 (the 2nd lowest form with the folded one ID-No.17 (a diazocine-ring conformer with the extended form), and the X-ray structure of (a^1*R*)-**2B**.

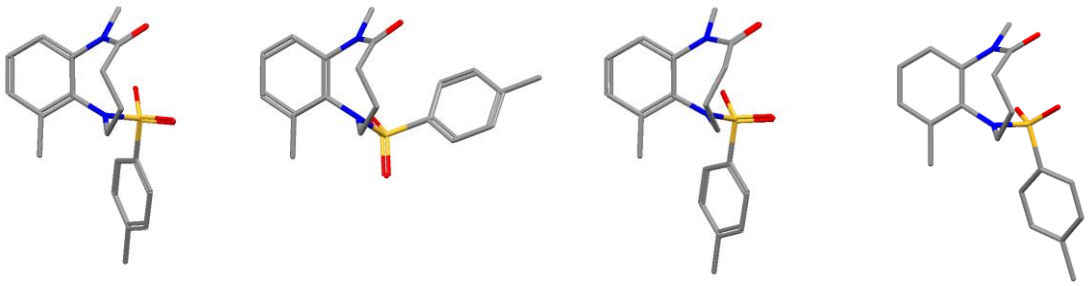
Table S2. Molecular Mechanics and DFT energies of copmpuond **2B** (selected 45 conformers)

2B	*R*/*S*	MMFF94	RB3LYP/6-31+G(d,p)					
			Gas phase (298K)			CH_2Cl_2 solution (298K)		
ID No	at axis1	E / kcal/mol	E / hartree	G / hartree	ΔG / kJ/mol	E / hartree	G / hartree	ΔG / kJ/mol
1	*S*	40.36420112	n.c.*[1]			n.c.		
2	*R*	40.36420113	-1470.65314372	-1470.325237	0.00	-1470.68739065	-1470.359364	1.76
3	*R*	40.69751499	-1470.65300997	-1470.324035	3.16	-1470.68740034	-1470.358467	4.12
4	*S*	40.69751525	n.c.			n.c.		
5[*2]	*R*	42.25275037	-1470.65286680	-1470.325142	0.25	-1470.68812403	-1470.360035	0.00
6	*S*	42.25275037	n.c.			n.c.		
7	*S*	42.70929128	n.c.			n.c.		
8	*R*	42.75975862	-1470.64774255	-1470.318474	17.76	-1470.68141778	-1470.351159	23.30
9	*S*	45.10954357	n.c.			n.c.		
10	*R*	45.14686079	-1470.64643847	-1470.319008	16.35	-1470.68054526	-1470.349218	28.40
11	*S*	45.89826824	n.c.			n.c.		
12	*R*	45.94537679	-1470.64646417	-1470.318414	17.91	-1470.68137798	-1470.352886	18.77
13	*S*	46.23678913	n.c.			n.c.		
14	*R*	46.24565146	-1470.64727478	-1470.320383	12.74	-1470.68209503	-1470.354432	14.71
15	*R*	47.11637784	-1470.64756005	-1470.320469	12.52	-1470.68211203	-1470.353870	16.19
16	*S*	47.11637785	n.c.			n.c.		
17	*R*	48.86943039	-1470.64556813	-1470.318241	18.37	-1470.68141589	-1470.353357	17.53
18	*S*	48.92998564	n.c.			n.c.		
19	*R*	55.77443029	-1470.64556813	-1470.318241	18.37	-1470.68146239	-1470.352778	19.05
20	*S*	55.77443030	n.c.			n.c.		
21	*R*	55.99928828	-1470.62550357	-1470.295933	76.94	-1470.65999544	-1470.330734	76.93
22	*S*	55.99928831	n.c.			n.c.		
23	*R*	57.14505121	-1470.62764270	-1470.298998	68.89	-1470.66139534	-1470.331220	75.65

24	*S*	57.19606458	n.c.			n.c.		
25	*R*	58.92634727	-1470.62550358	-1470.295949	76.90	-1470.65970757	-1470.329715	79.61
26	*S*	59.00093002	n.c.			n.c.		
27	*S*	59.71770114	n.c.			n.c.		
28	*R*	59.71770118	-1470.62769619	-1470.298608	69.91	-1470.66138465	-1470.331370	75.26
29	*R*	59.75684571	-1470.62764788	-1470.299239	68.26	-1470.66141053	-1470.331442	75.07
30	*S*	59.75684581	n.c.			n.c.		
31	*S*	61.55967908	n.c.			n.c.		
32	*R*	61.55967936	-1470.62670092	-1470.298320	70.67	-1470.66139481	-1470.331681	74.44
33	*R*	62.59126295	-1470.62684773	-1470.296052	76.63	-1470.65927172	-1470.327174	86.28
34	*S*	62.65998084	n.c.			n.c.		
35	*S*	62.89803614	n.c.			n.c.		
36	*R*	62.96301032	-1470.62550357	-1470.295929	76.95	-1470.65970872	-1470.329748	79.52
37	*R*	63.28530628	-1470.62540457	-1470.296247	76.11	-1470.65996022	-1470.331065	76.06
38	*R*	64.25371970	-1470.62549227	-1470.295195	78.88	-1470.65886406	-1470.328660	82.38
39	*S*	64.31304977	n.c.			n.c.		
40	*S*	64.48816318	n.c.			n.c.		
41	*S*	65.92282633	n.c.			n.c.		
42	*R*	65.98601707	-1470.61890274	-1470.292504	85.94	-1470.65320313	-1470.323684	95.44
43	*R*	67.35437263	-1470.61284146	-1470.284470	107.03	-1470.64678001	-1470.316830	113.43
44	*S*	67.42134436	n.c.			n.c.		
45	*S*	83.43899880	n.c.			n.c.		

*[1] n.c. = not calculated

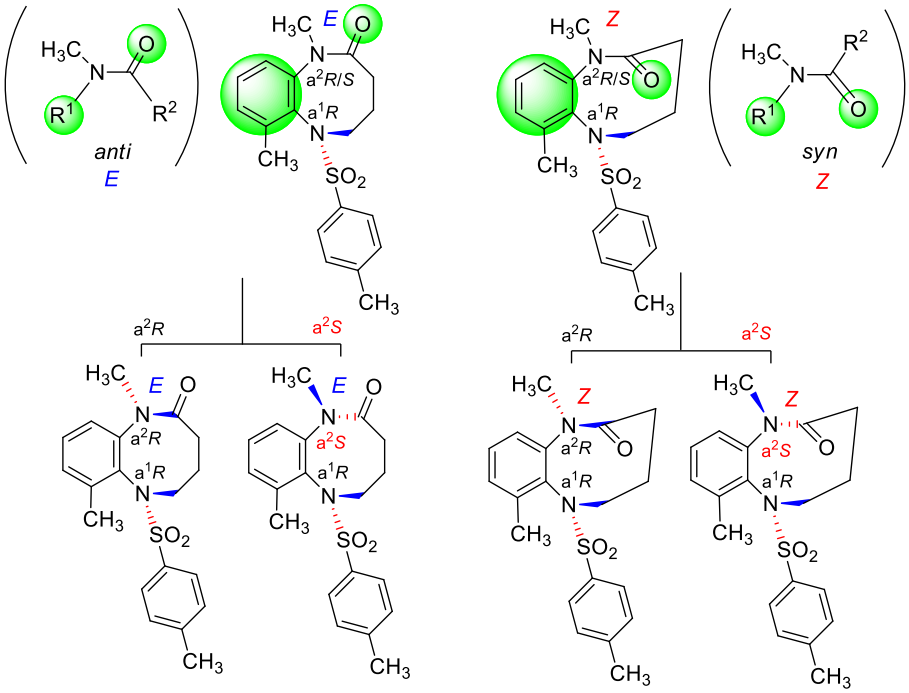
*[2] ID-No.5 is the lowest energy conformer in CH_2Cl_2 solution.



ID-No.5 (the lowest energy conformer) | ID-No.2 (Δ*G*, 1.76 kJ/mol) | ID-No.17 (Δ*G*, 17.53 kJ/mol) | X-ray structure

Figure S3. Typical conformers of 2B: ID-No.5 (the lowest energy conformer with the extended form), ID-No.2 (the folded form), ID-No.17 (a diazocine-ring conformer with the extended form), and the X-ray crystal structure of (a[1]*R*)-**2B**.

In the eight-membered ring conformers of **2B**, in addition to the expected Type A conformer, two types of unexpected diastereomer (Type B and Type C) appeared in the calculation study, as shown in Figure S4. The first one (Type B) is the unfavored diastereomers (a^1R, a^2S), which appeared in two of the 22 conformers of **2B** analyzed. However, the energy difference (ΔG) compared with the diastereomer (a^1R, a^2R) was shown to be very large (>82 kJ/mol). The second one (Type C) had the *Z*-form around the lactam moiety (>N^1-C^2=O), which appeared in 10 of the 22 conformers of **2B** analyzed. These conformers, however, were also shown to be very unstable with the ΔG values of >74 kJ/mol compared with the lowest conformer. Since the energy differences of these Type B and C conformers are very large, these conformers would not occur in practice.



E-(a^1R, a^2R) stable

ID-No	*	ΔG kJ/mol
2	f	1.76
3	f	4.12
most stable 5	e	0
8	f	23.3
10	e	28.4
12	e	18.77
14	f	14.71
15	f	16.19
17	e	17.53
19	e	19.05

E-(a^1R, a^2S) unstable

ID-No	*	ΔG kJ/mol
33	f	86.28
38	e	82.38

Z-(a^1R, a^2R) unstable

ID-No	*	ΔG kJ/mol
21	f	76.93
23	e	75.65
25	f	79.61
28	e	75.26
29	e	75.07
32	e	74.44
36	f	79.52
39	f	76.06
42	e	95.44
43	f	113.43

Z-(a^1R, a^2S) unstable

not appeared in the calculation study

* orientation of the tolyl group
f: folded
e: extended

Figure S4. Conformers of (a^1R)**-2B in CH_2Cl_2.** In the DFT analysis, conformers of Type B and Type C appeared in **2B** in addition to the expected Type A conformer.

(d) Atomic coordinates for the typical conformers of 1B and 2B by DFT calculation (Tables S3-S7).

Atomic coordinates obtained by the calculation using the RB3LYP/6-31+G (d,p) level are shown for **1B** [ID-No.7 (the lowest-energy conformer), ID-No.6 (the extended form) shown in Figure S2] and **2B** [ID-No.5 (the lowest-energy conformer), ID-No.2 (the folded form), and ID-No.17 (a diazocine-ring conformer with the extended form) shown in Figure S3].

Table S3. Atomic coordinate for compound **1B**-ID-No.7 (the lowest energy conformer) in CH_2Cl_2 solution

Gibbs Free Energy (298K, 1 atm)	-1431.072944 hartree		
Total Energy	-1431.37442578 hartree		
Atom	x	y	z
S	0.08085	-1.86110	-0.02575
O	-0.48101	-2.13651	-1.35691
O	0.30691	-2.95996	0.93059
N	-0.97570	-0.78703	0.72584
C	1.66755	-1.05174	-0.26409
C	-1.95780	-0.05826	-0.02084
C	-0.75600	-0.46921	2.15367
C	1.90314	-0.34110	-1.44481
C	2.65780	-1.16018	0.71846
C	-1.74443	1.31379	-0.28694
C	-3.15261	-0.68857	-0.43005
C	-0.97735	1.02221	2.43389
C	3.14327	0.26974	-1.63331
C	3.88910	-0.53839	0.51306
N	-0.57545	1.97704	0.19023
C	-2.69103	2.03276	-1.02709
C	-4.07942	0.05676	-1.17302
C	-3.44251	-2.13072	-0.09733
C	-0.18031	1.92434	1.50667
C	4.15493	0.18163	-0.66335
C	0.10881	2.93540	-0.68881
C	-3.84591	1.39714	-1.48111
O	0.77628	2.59314	1.91391
C	5.50504	0.81465	-0.88850
H	0.26675	-0.74425	2.41984
H	-1.43257	-1.06354	2.77894
H	1.13789	-0.27671	-2.21077
H	2.47748	-1.73338	1.62117
H	-2.04307	1.26444	2.36218
H	-0.65475	1.23511	3.45511
H	3.32955	0.81725	-2.55341
H	4.65810	-0.62235	1.27653
H	-2.52940	3.08549	-1.23289
H	-4.99691	-0.42254	-1.50316
H	-2.96063	-2.80523	-0.81297
H	-3.07118	-2.39751	0.89634
H	-4.51947	-2.31875	-0.12887
H	1.17879	2.90465	-0.48268
H	-0.24585	3.95872	-0.52057
H	-0.07035	2.65678	-1.72718
H	-4.57783	1.95897	-2.05438
H	6.23436	0.06342	-1.2178
H	5.89328	1.26155	0.0325
H	5.45931	1.59055	-1.65812

Table S4. Atomic coordinate for compound **1B**-ID-No.6 (the extended form) in CH_2Cl_2 solution

Gibbs Free Energy (298K, 1 atm)	-1431.071811 hartree		
Total Energy	-1431.37292665 hartree		
Atom	x	y	z
S	-0.49767	-0.55306	-0.88822
O	-0.30323	-2.00254	-1.08129
O	-0.15050	0.38020	-1.97224
N	0.36619	-0.03354	0.47771
C	-2.22791	-0.31852	-0.46587
C	1.43266	0.91307	0.29675
C	0.44488	-0.95136	1.63596
C	-2.86166	0.88003	-0.81054
C	-2.92831	-1.34118	0.17797
C	2.71704	0.44509	-0.05709
C	1.20346	2.28558	0.51037
C	1.88701	-1.38904	1.94281
C	-4.20760	1.05118	-0.49338
C	-4.27505	-1.14793	0.49407
N	2.95682	-0.94954	-0.23657
C	3.75971	1.35817	-0.25244
C	2.26836	3.17780	0.31592
C	-0.15427	2.80753	0.90741
C	2.63692	-1.88547	0.71826
C	-4.93611	0.04514	0.16629
C	3.72710	-1.39574	-1.40485
C	3.52773	2.72198	-0.07487
O	2.95878	-3.07112	0.58473
C	-6.39423	0.24691	0.49512
H	-0.16688	-1.82908	1.42056
H	0.02326	-0.45450	2.51708
H	-2.31578	1.65924	-1.33056
H	-2.44116	-2.28003	0.41685
H	2.43830	-0.55944	2.39850
H	1.85798	-2.20722	2.66565
H	-4.70314	1.97856	-0.76904
H	-4.81999	-1.94384	0.99409
H	4.74535	0.99984	-0.53107
H	2.10170	4.24000	0.47282
H	-0.80061	2.92933	0.03065
H	-0.06097	3.78684	1.38538
H	-0.66582	2.12916	1.59519
H	3.63004	-0.65252	-2.19641
H	3.32307	-2.34770	-1.75250
H	4.78637	-1.53663	-1.16192
H	4.33929	3.42859	-0.22317
H	-6.98493	0.39145	-0.41722
H	-6.80489	-0.61247	1.03222
H	-6.53823	1.13913	1.11549

Table S5. Atomic coordinate for compound **2B-**ID-No.5 (the lowest energy conformer) in CH_2Cl_2 solution

Gibbs Free Energy (298K, 1 atm)	-1470.360035 hartree		
Total Energy	-1470.68812403 hartree		
Atom	x	y	z
S	-0.70735	0.02140	-1.13156
O	-0.44982	-1.24022	-1.85488
O	-0.50821	1.29354	-1.84679
N	0.23806	0.11065	0.29159
C	-2.39540	-0.05951	-0.52468
C	1.44841	0.88869	0.18257
C	0.15253	-1.11949	1.13161
C	-3.07020	-1.28166	-0.53661
C	-3.03178	1.11037	-0.09467
C	2.66725	0.30537	-0.22509
C	1.40030	2.26761	0.48847
C	1.11698	-1.15041	2.31884
C	-4.39650	-1.32998	-0.10040
C	-4.35433	1.04205	0.33650
N	2.76894	-1.10836	-0.44243
C	3.80846	1.09458	-0.39470
C	2.56239	3.03379	0.31547
C	0.13966	2.93772	0.97112
C	2.62016	-1.40918	1.99714
C	-5.05929	-0.17527	0.34043
C	2.85869	-1.98354	0.60857
C	3.04391	-1.60207	-1.79792
C	3.75117	2.46372	-0.13970
C	-6.49846	-0.22346	0.78783
O	3.09425	-3.18516	0.42420
H	0.29693	-2.01318	0.51469
H	-0.86709	-1.15640	1.52759
H	-2.57240	-2.17883	-0.88749
H	-2.50897	2.05984	-0.10370
H	0.75638	-1.96766	2.95171
H	1.01815	-0.23231	2.90659
H	-4.92223	-2.28089	-0.10869
H	-4.85117	1.94998	0.66835
H	4.73511	0.62433	-0.70974
H	2.52758	4.09545	0.54476
H	0.38240	3.84045	1.53942
H	-0.48968	3.23758	0.12552
H	-0.45536	2.27328	1.60233
H	3.19375	-0.48608	2.11196
H	3.02304	-2.135	2.70619
H	2.47942	-2.51971	-1.97323
H	4.11002	-1.81704	-1.93614
H	2.73155	-0.84563	-2.51756
H	4.63705	3.07851	-0.27061
H	-7.15182	0.24856	0.04337
H	-6.83894	-1.25323	0.92704
H	-6.63883	0.31827	1.72945

Table S6. Atomic coordinate for compound **2B-**ID-No.2 (the folded form) in CH_2Cl_2 solution

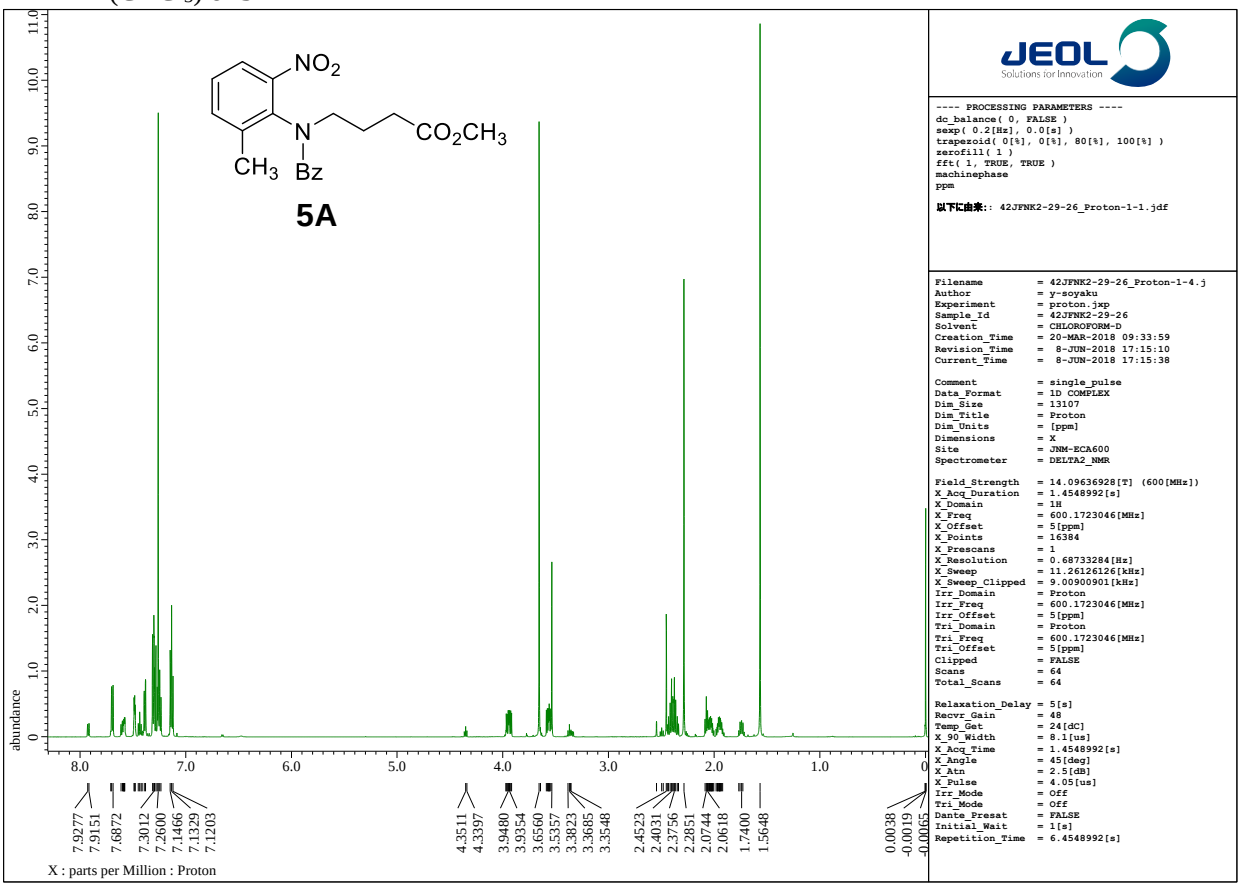
Gibbs Free Energy (298K, 1 atm)	-1470.359364 hartree		
Total Energy	-1470.68739065 hartree		
Atom	x	y	z
S	0.21611	-1.75008	-0.37447
O	-0.32105	-1.69918	-1.74493
O	0.35645	-3.05296	0.30548
N	-0.76846	-0.73710	0.55165
C	1.84850	-1.00321	-0.37881
C	-1.98825	-0.21840	-0.00886
C	-0.63121	-0.76520	2.02858
C	2.14890	-0.02481	-1.32978
C	2.80690	-1.42553	0.54712
C	-2.05647	1.13998	-0.38483
C	-3.13196	-1.04317	-0.12309
C	-0.07471	0.52718	2.62835
C	3.42221	0.54434	-1.33823
C	4.07416	-0.84264	0.52451
N	-0.92127	2.00296	-0.29953
C	-3.25035	1.66353	-0.89926
C	-4.31099	-0.48930	-0.63857
C	-3.10006	-2.50183	0.26179
C	-0.82135	1.80750	2.19376
C	4.40147	0.15311	-0.41063
C	-0.31489	2.37005	0.87430
C	-0.47734	2.67692	-1.53042
C	-4.37139	0.84903	-1.03075
C	5.76443	0.79865	-0.40400
O	0.63681	3.16437	0.86411
H	-1.62315	-0.97046	2.44647
H	0.00605	-1.61020	2.30017
H	1.40599	0.27949	-2.05839
H	2.57409	-2.20651	1.26249
H	0.99088	0.63044	2.39565
H	-0.15199	0.42990	3.71790
H	3.65965	1.30103	-2.08101
H	4.82186	-1.17385	1.24037
H	-3.29124	2.71007	-1.18441
H	-5.19083	-1.11970	-0.73301
H	-2.55994	-3.09559	-0.48395
H	-4.11661	-2.89910	0.32753
H	-2.60552	-2.66745	1.22290
H	-1.90150	1.63129	2.17426
H	-0.62648	2.59428	2.92680
H	0.61287	2.69157	-1.56291
H	-0.83624	3.71170	-1.56977
H	-0.85994	2.12959	-2.39172
H	-5.29607	1.25997	-1.42574
H	5.99267	1.26011	-1.36910
H	6.54996	0.07260	-0.17128
H	5.81249	1.58609	0.35914

Table S7. Atomic coordinate for compound **2B**-ID-No.17 (a diazocine-ring conformer with the extended form) in CH_2Cl_2 solution

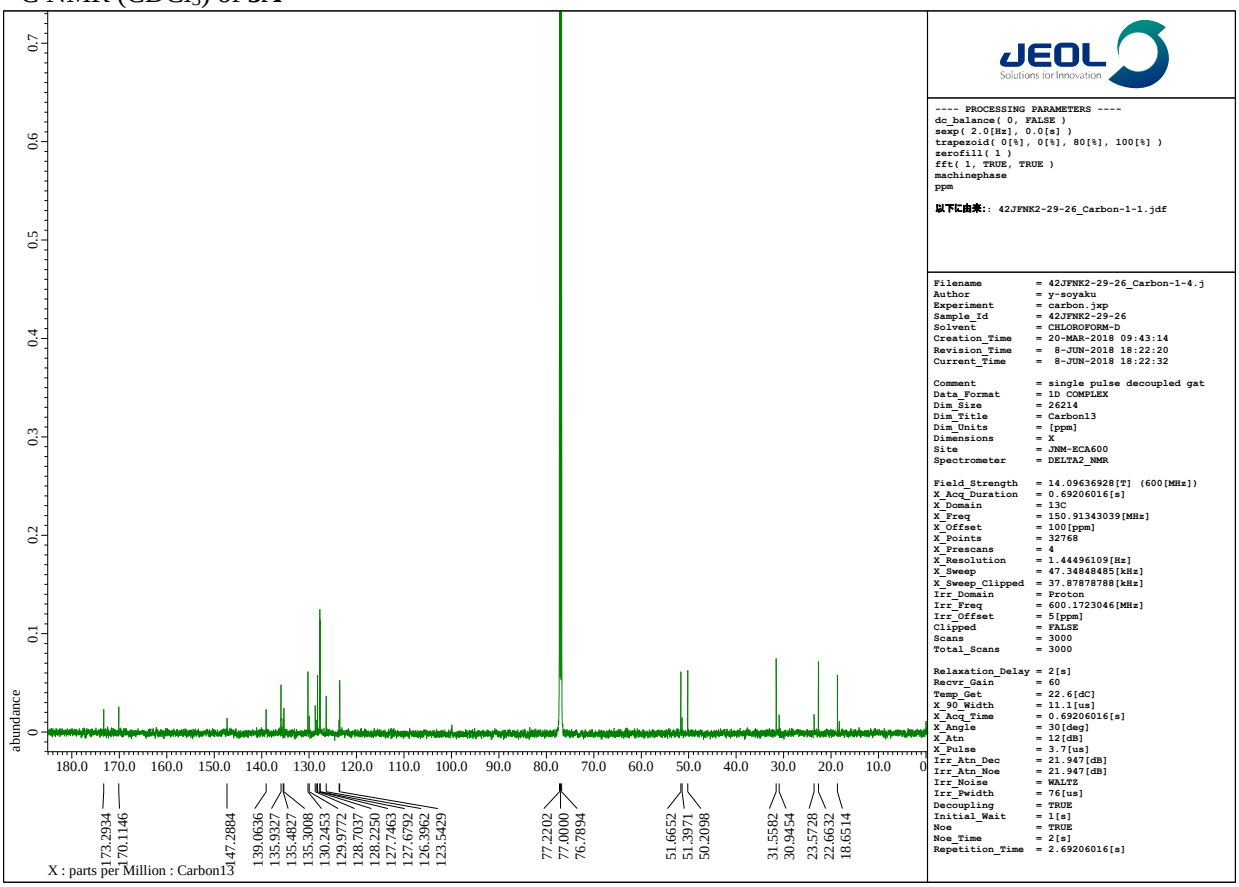
Gibbs Free Energy (298K, 1 atm)	-1470.353357 hartree		
Total Energy	-1470.68141589 hartree		
Atom	x	y	z
S	-0.50163	-1.03628	-0.68011
O	-0.09708	-0.47219	-1.97735
O	-0.37607	-2.48231	-0.42424
N	0.41164	-0.25141	0.48776
C	-2.23241	-0.61941	-0.42804
C	1.09940	0.97140	0.19420
C	0.50786	-0.79122	1.86387
C	-2.97467	-1.30781	0.53722
C	-2.81602	0.39269	-1.19466
C	2.41949	0.88021	-0.30493
C	0.54068	2.22660	0.50583
C	1.91432	-0.57880	2.42573
C	-4.31174	-0.96458	0.73804
C	-4.15638	0.71830	-0.98272
N	2.99371	-0.38008	-0.65133
C	3.15482	2.04493	-0.54323
C	1.30180	3.37843	0.24912
C	-0.82828	2.37969	1.12429
C	3.03432	-1.35877	1.71558
C	-4.92527	0.04933	-0.01650
C	3.20600	-1.43968	0.19378
C	3.37087	-0.58552	-2.06213
C	2.59001	3.29251	-0.27487
C	-6.38167	0.38655	0.18360
O	3.66941	-2.49360	-0.26747
H	0.27204	-1.85687	1.82328
H	-0.22584	-0.29732	2.51445
H	-2.52298	-2.10556	1.11713
H	-2.23369	0.91460	-1.94613
H	1.89750	-0.92240	3.46680
H	2.14018	0.49009	2.46600
H	-4.88917	-1.49848	1.48825
H	-4.61143	1.50353	-1.58074
H	4.16464	1.96739	-0.93413
H	0.87436	4.35183	0.47598
H	-1.47462	2.99796	0.49165
H	-0.75042	2.88544	2.09398
H	-1.32460	1.42263	1.28067
H	2.97408	-2.40950	2.01206
H	3.99925	-0.99321	2.09310
H	2.89518	-1.49016	-2.44708
H	4.45534	-0.69346	-2.16537
H	3.03318	0.27082	-2.64366
H	3.16333	4.19608	-0.46161
H	-7.01502	-0.23943	-0.45834
H	-6.69285	0.21210	1.21827
H	-6.58871	1.43050	-0.07046

3. ^{1}H- and ^{13}C-NMR, and 2D-NMR spectra

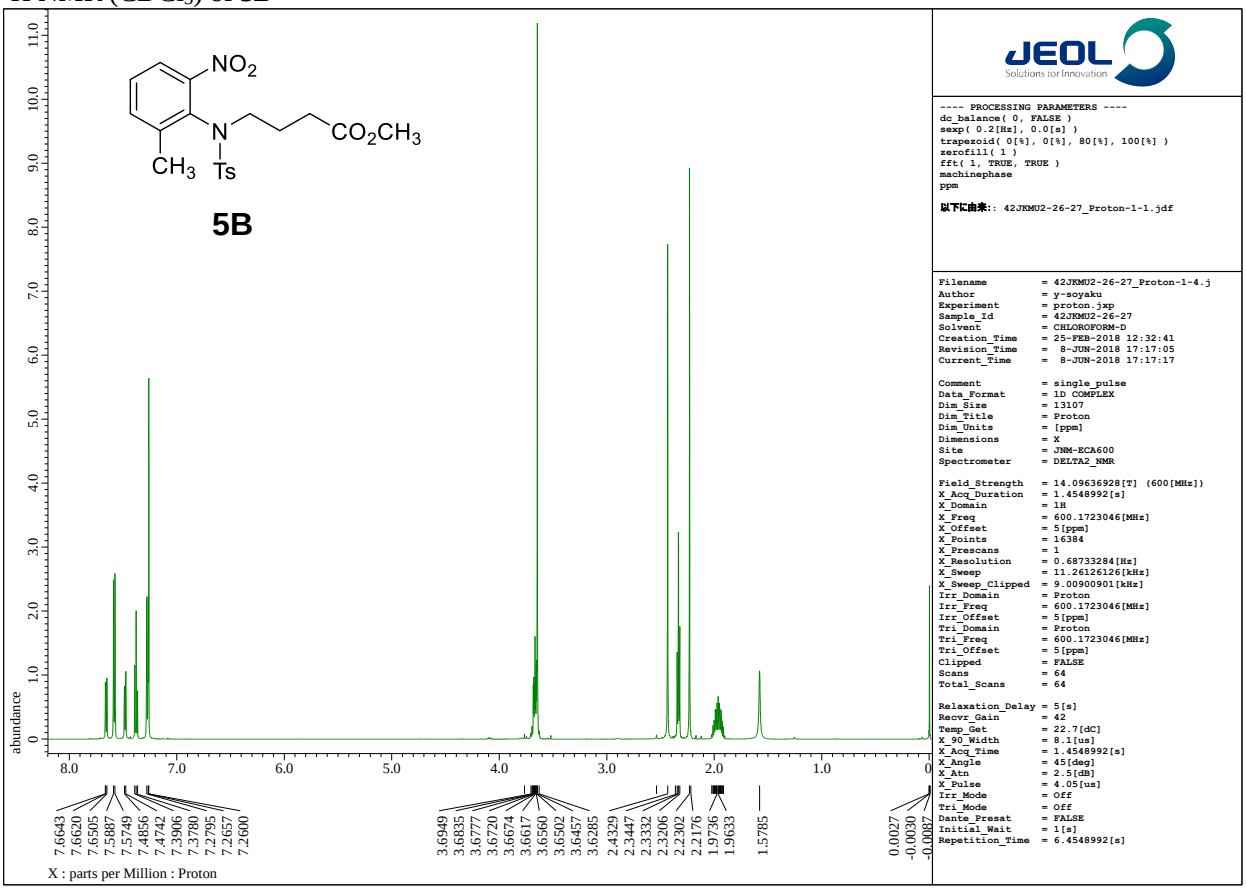
^{1}H NMR ($CDCl_3$) of **5A**



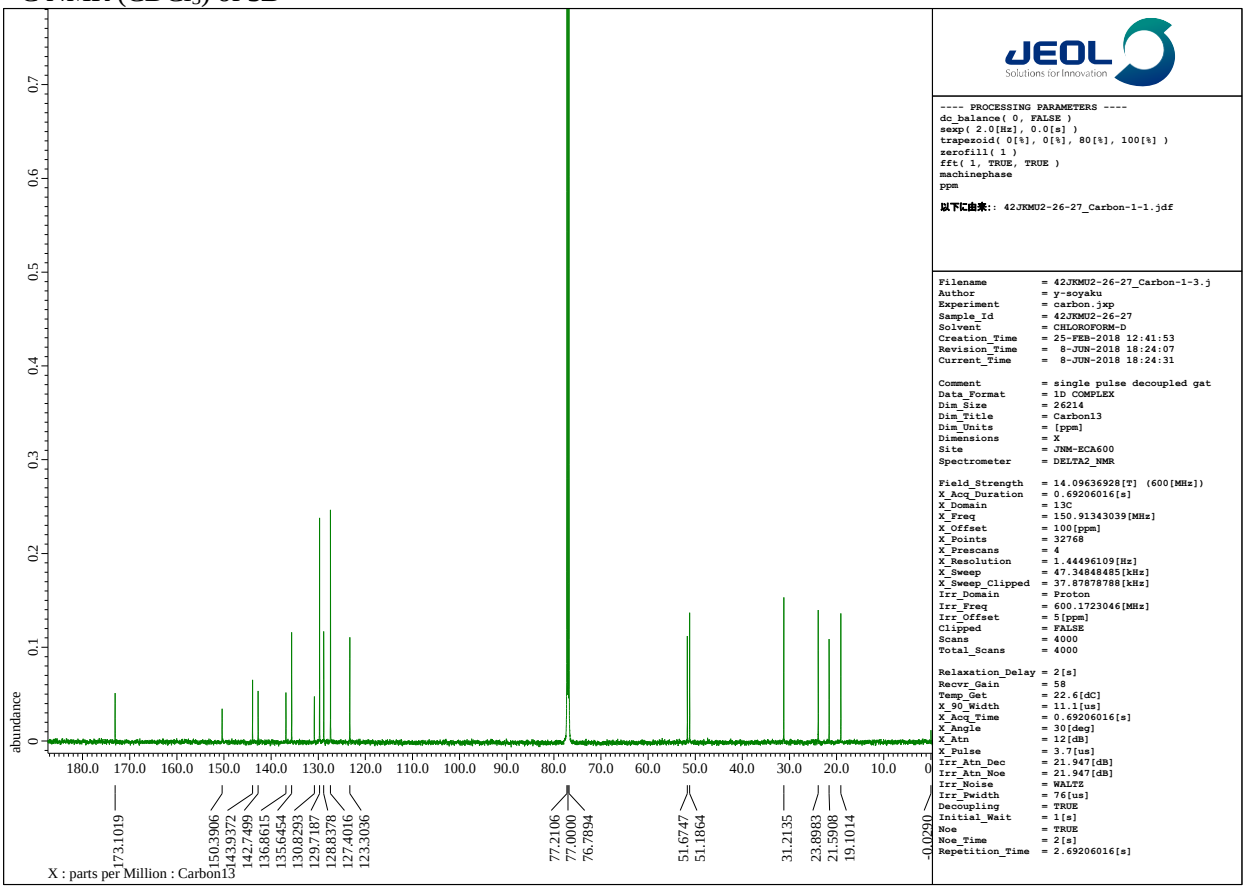
^{13}C NMR ($CDCl_3$) of **5A**



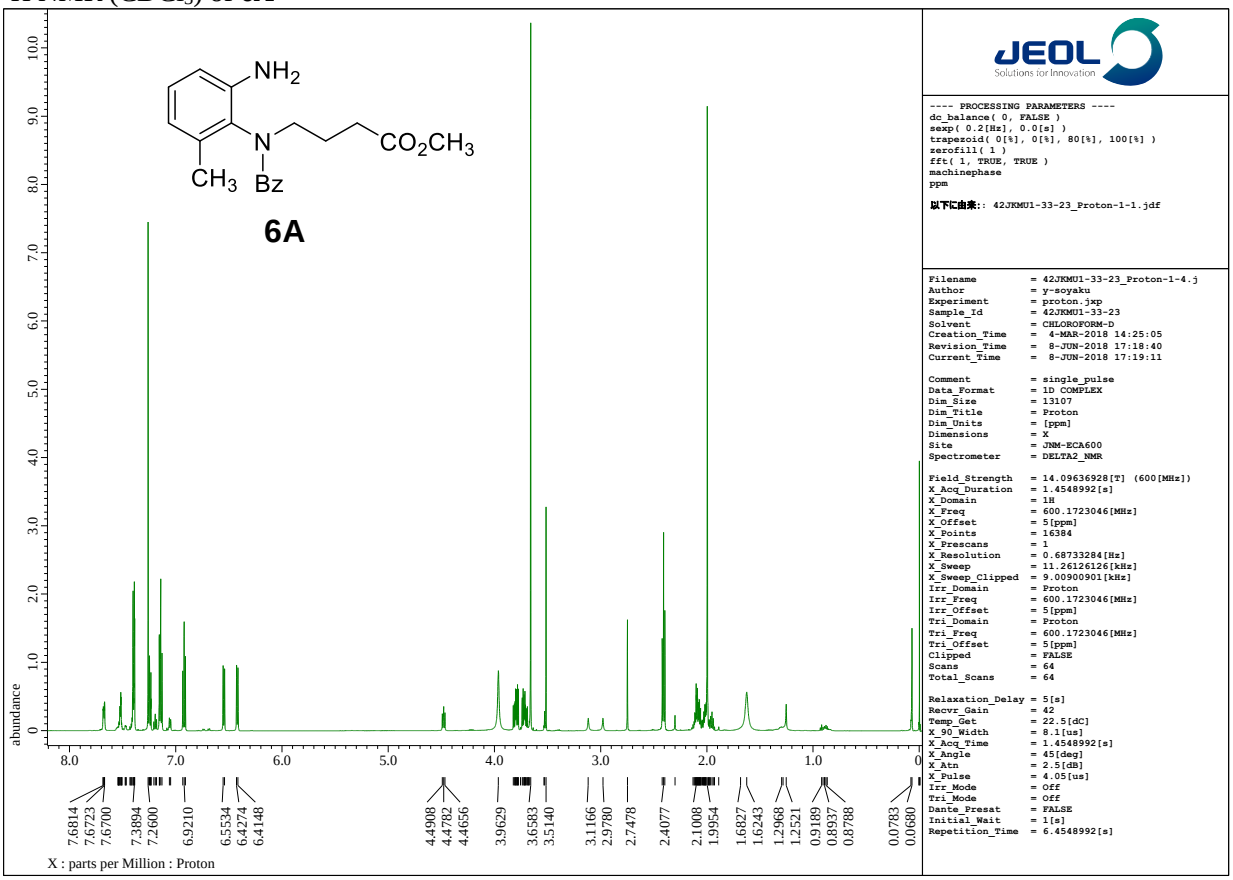
^{1}H NMR ($CDCl_3$) of **5B**



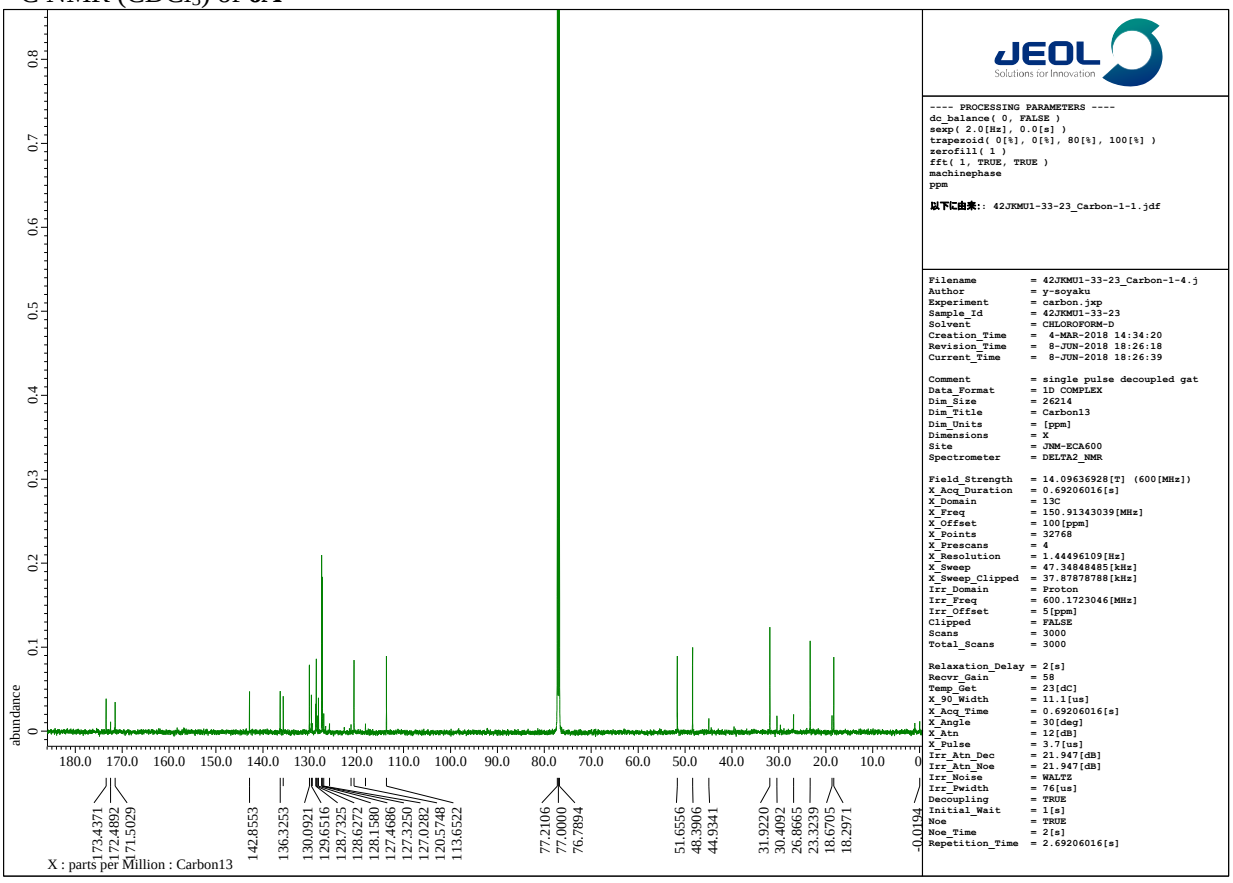
^{13}C NMR ($CDCl_3$) of **5B**



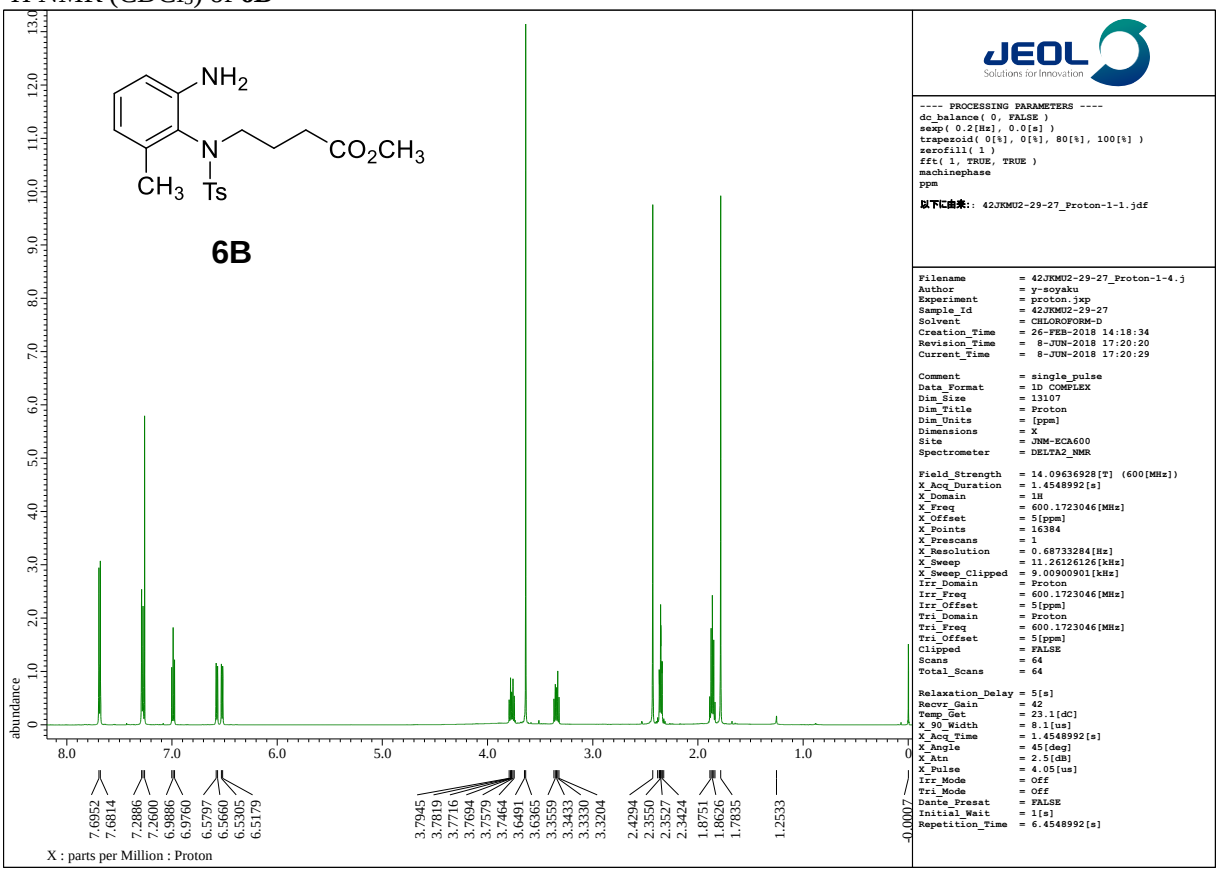
^{1}H NMR ($CDCl_3$) of **6A**



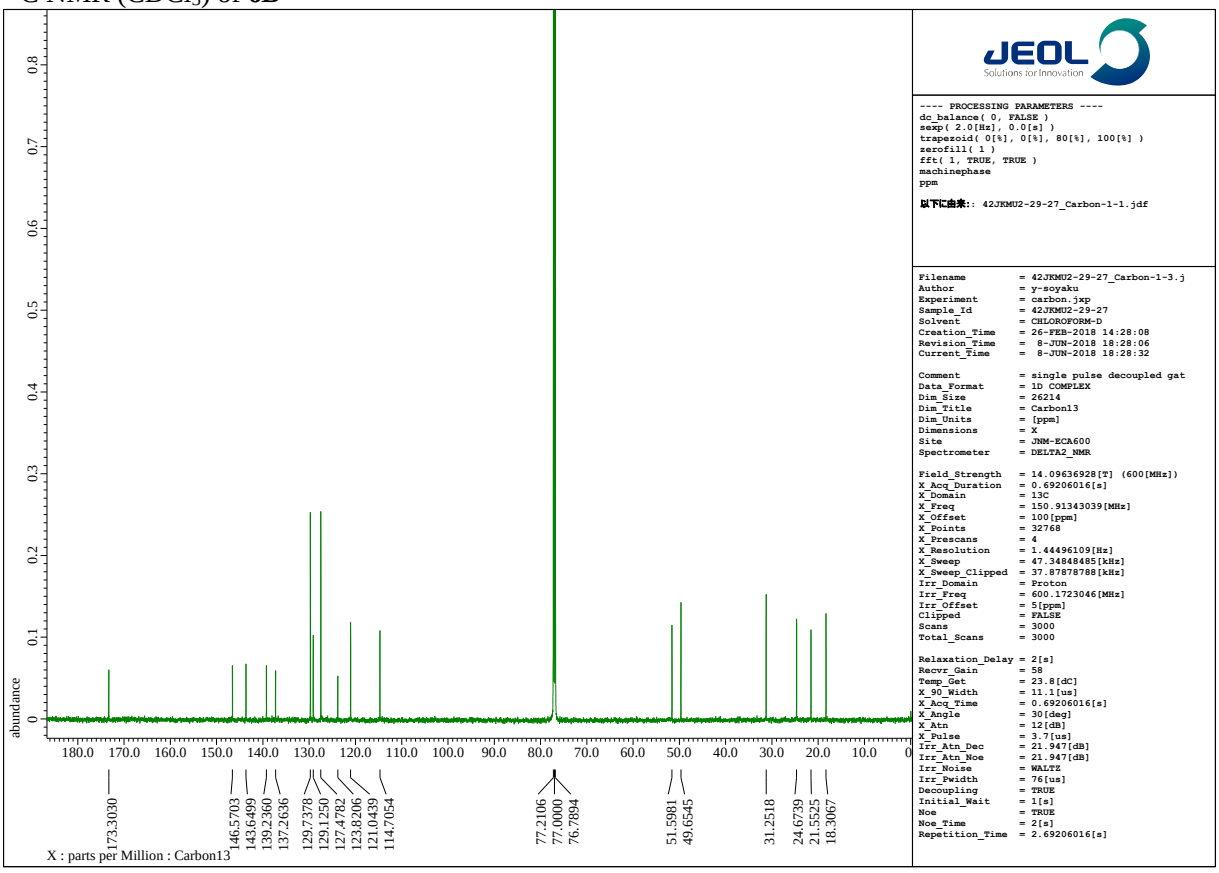
^{13}C NMR ($CDCl_3$) of **6A**



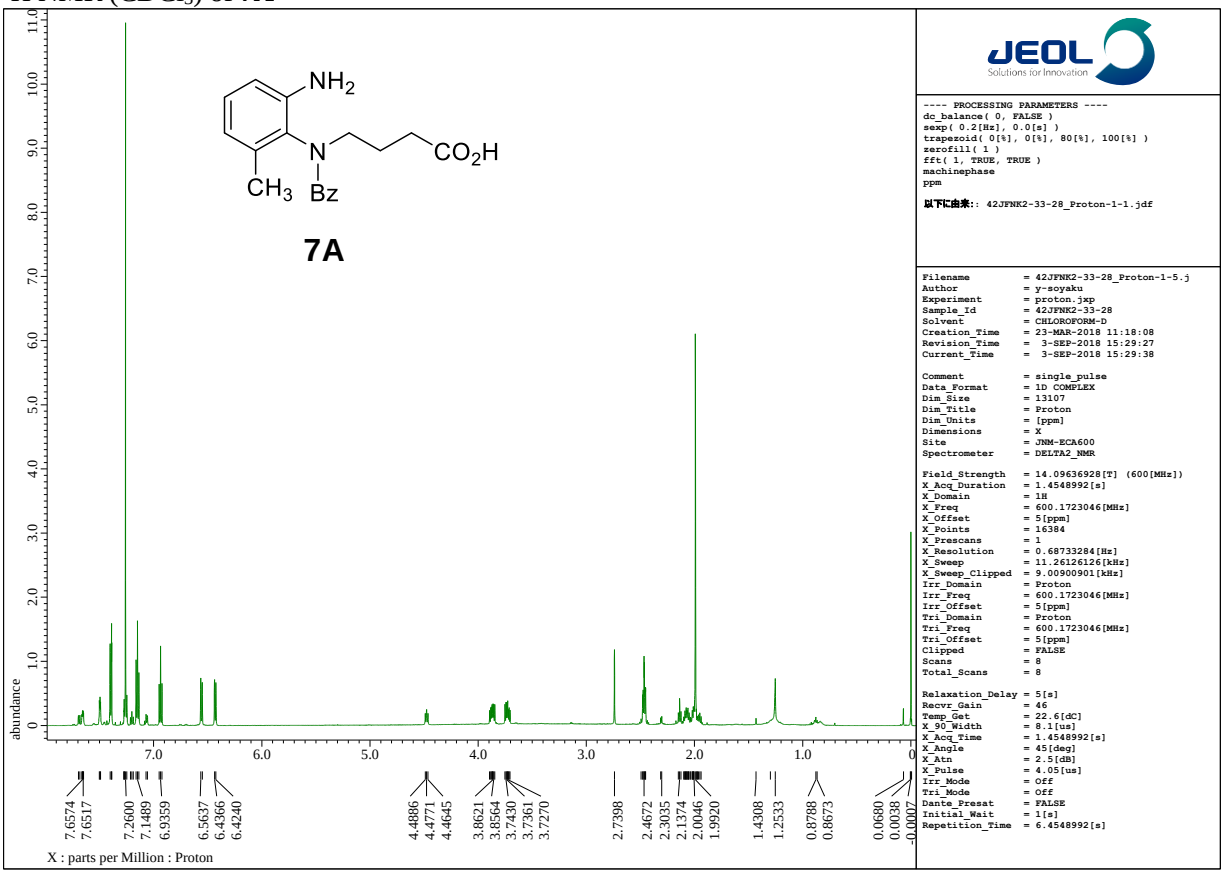
^{1}H NMR ($CDCl_3$) of **6B**



^{13}C NMR ($CDCl_3$) of **6B**



^{1}H NMR ($CDCl_3$) of **7A**

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---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: 42JFNK2-33-28_Proton-1-1.jdf
```

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Filename          = 42JFNK2-33-28_Proton-1-5.j
Author            = y-soyaku
Experiment        = proton.jxp
Sample_Id         = 42JFNK2-33-28
Solvent           = CHLOROFORM-D
Creation_Time     = 23-MAR-2018 11:18:08
Revision_Time     =  3-SEP-2018 15:29:27
Current_Time      =  3-SEP-2018 15:29:38

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 1.4548992[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.68733284[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 46
Temp_Get          = 22.6[dC]
X_90_Width        = 8.1[us]
X_Acq_Time        = 1.4548992[s]
X_Angle           = 45[deg]
X_Atn             = 2.5[dB]
X_Pulse           = 4.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.4548992[s]
```
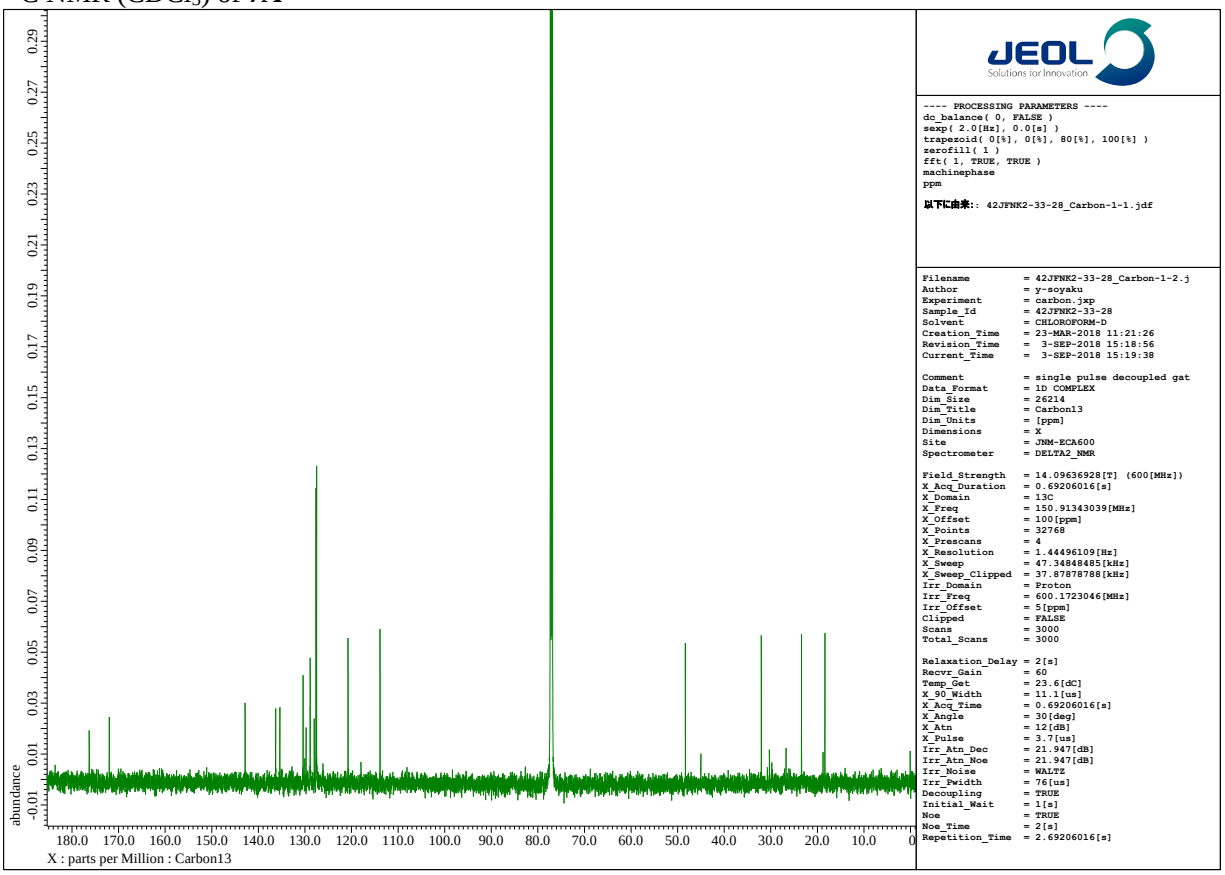
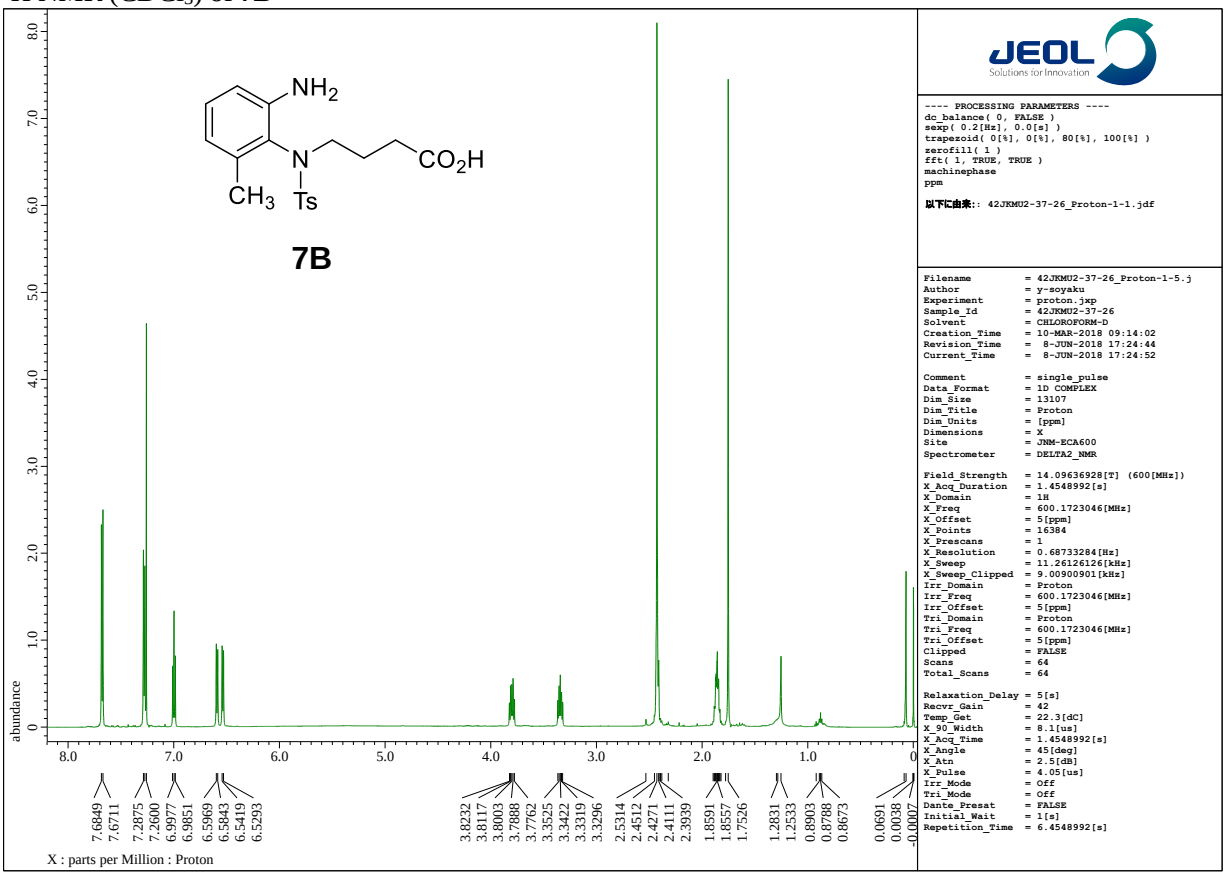

^{13}C NMR ($CDCl_3$) of **7A**




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: 42JFNK2-33-28_Carbon-1-1.jdf
```

```
Filename          = 42JFNK2-33-28_Carbon-1-2.j
Author            = y-soyaku
Experiment        = carbon.jxp
Sample_Id         = 42JFNK2-33-28
Solvent           = CHLOROFORM-D
Creation_Time     = 23-MAR-2018 11:21:26
Revision_Time     =  3-SEP-2018 15:18:56
Current_Time      =  3-SEP-2018 15:19:38

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 3000
Total_Scans       = 3000

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.6[dC]
X_90_Width        = 11.1[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 12[dB]
X_Pulse           = 3.7[us]
Irr_Atn_Dec       = 21.947[dB]
Irr_Atn_Noe       = 21.947[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
```

^{1}H NMR ($CDCl_3$) of **7B**



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```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: 42JKMU2-37-26_Proton-1-1.jdf
```

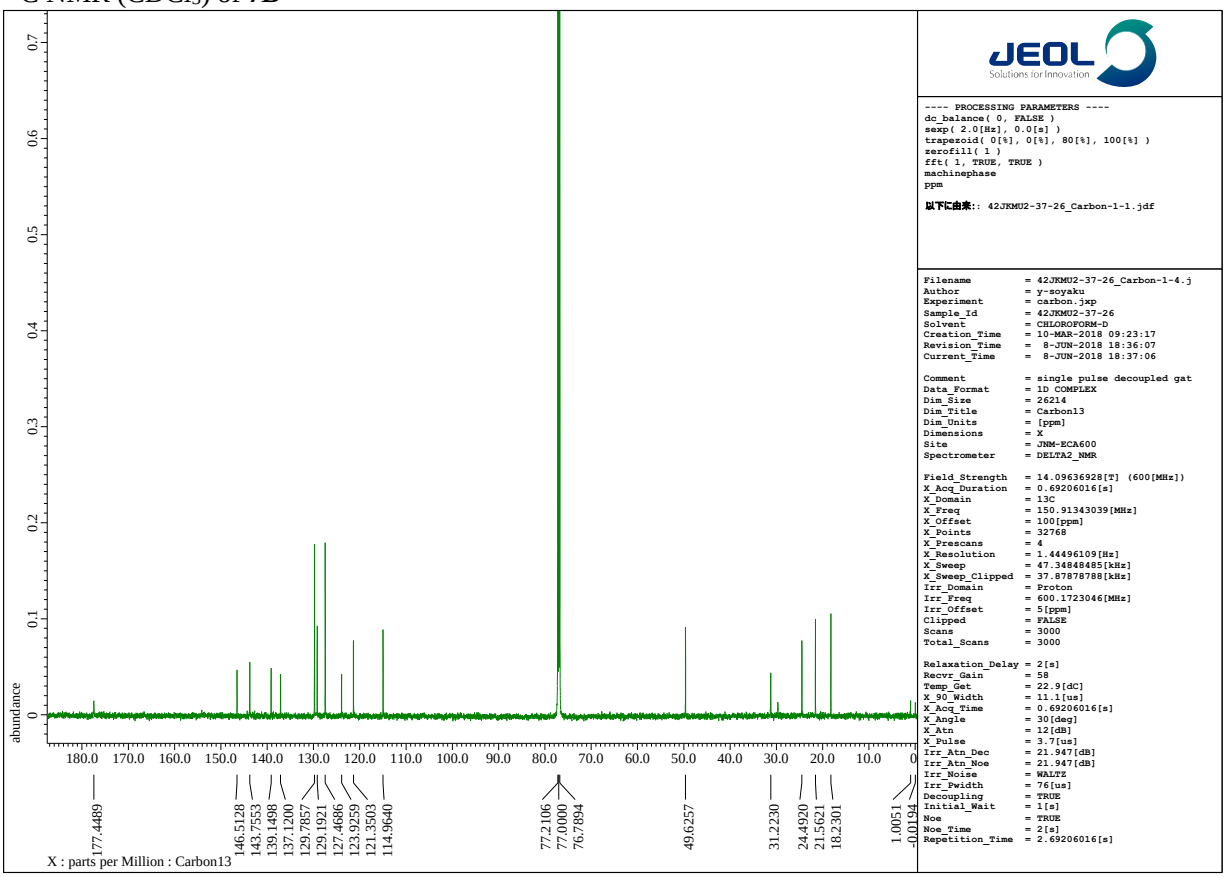
```
Filename          = 42JKMU2-37-26_Proton-1-5.j
Author            = y-soyaku
Experiment        = proton.jxp
Sample_Id         = 42JKMU2-37-26
Solvent           = CHLOROFORM-D
Creation_Time     = 10-MAR-2018 09:14:02
Revision_Time     =  8-JUN-2018 17:24:44
Current_Time      =  8-JUN-2018 17:24:52

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 1.4548992[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.68733284[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 64
Total_Scans       = 64

Relaxation_Delay = 5[s]
Recvr_Gain        = 42
Temp_Get          = 22.3[dC]
X_90_Width        = 8.1[us]
X_Acq_Time        = 1.4548992[s]
X_Angle           = 45[deg]
X_Atn             = 2.5[dB]
X_Pulse           = 4.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.4548992[s]
```

^{13}C NMR ($CDCl_3$) of **7B**



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```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: 42JKMU2-37-26_Carbon-1-1.jdf
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```
Filename          = 42JKMU2-37-26_Carbon-1-4.j
Author            = y-soyaku
Experiment        = carbon.jxp
Sample_Id         = 42JKMU2-37-26
Solvent           = CHLOROFORM-D
Creation_Time     = 10-MAR-2018 09:23:17
Revision_Time     =  8-JUN-2018 18:36:07
Current_Time      =  8-JUN-2018 18:37:06

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 3000
Total_Scans       = 3000

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 22.9[dC]
X_90_Width        = 11.1[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 12[dB]
X_Pulse           = 3.7[us]
Irr_Atn_Dec       = 21.947[dB]
Irr_Atn_Noe       = 21.947[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
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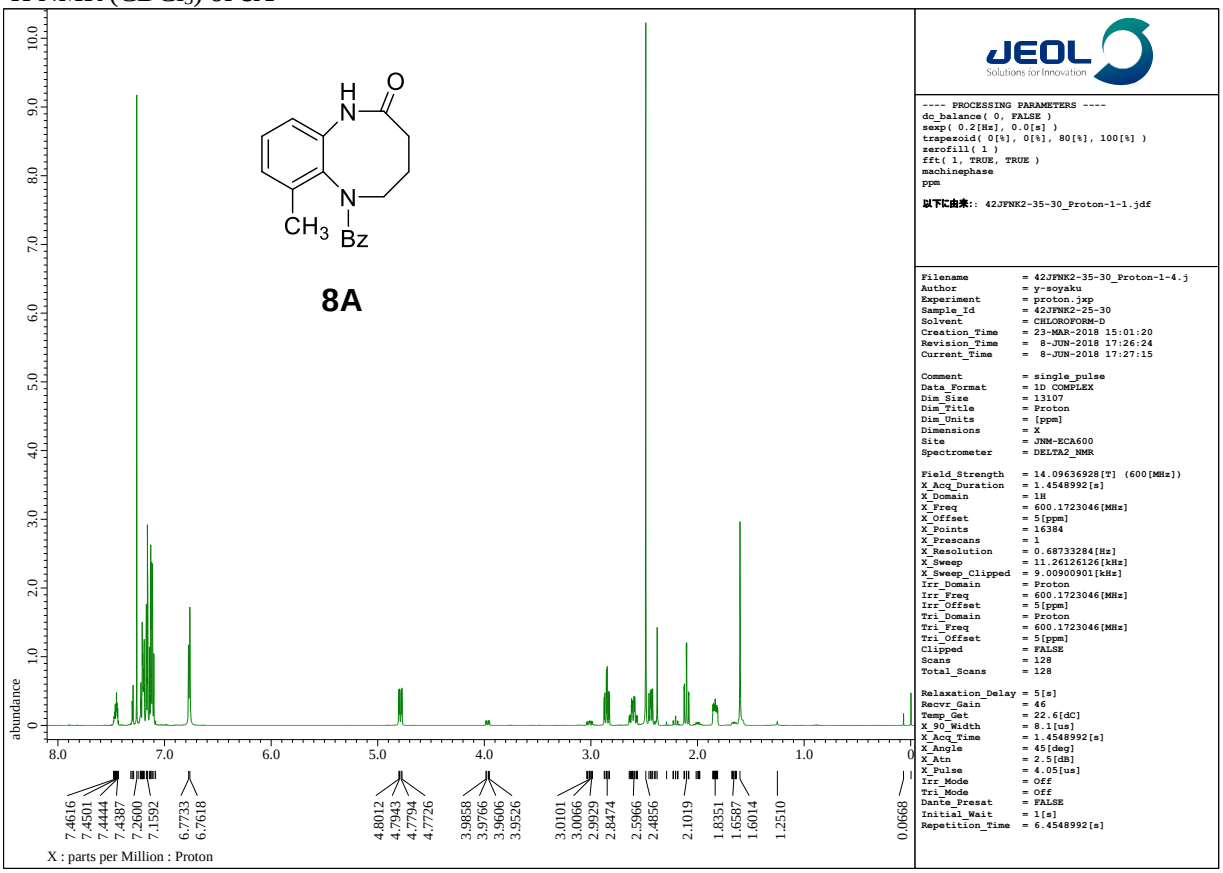
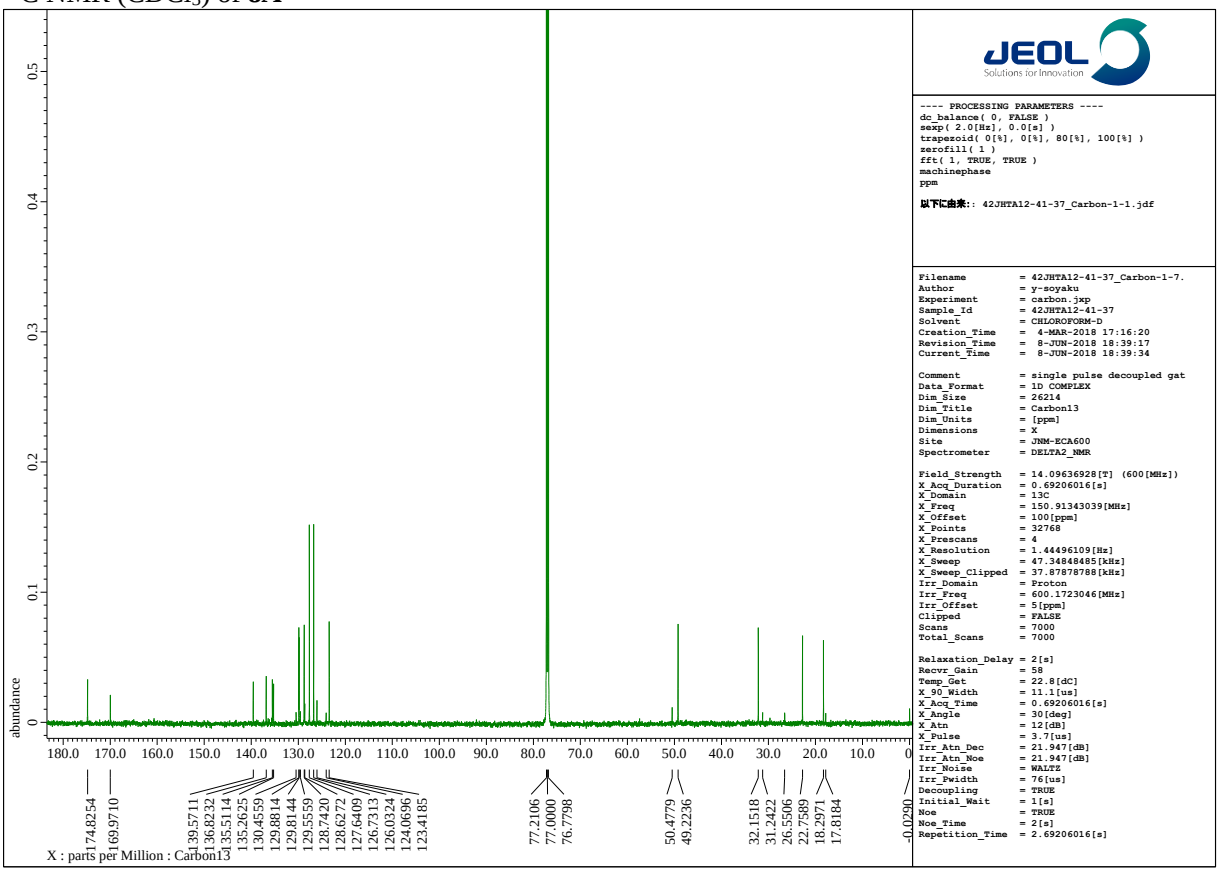
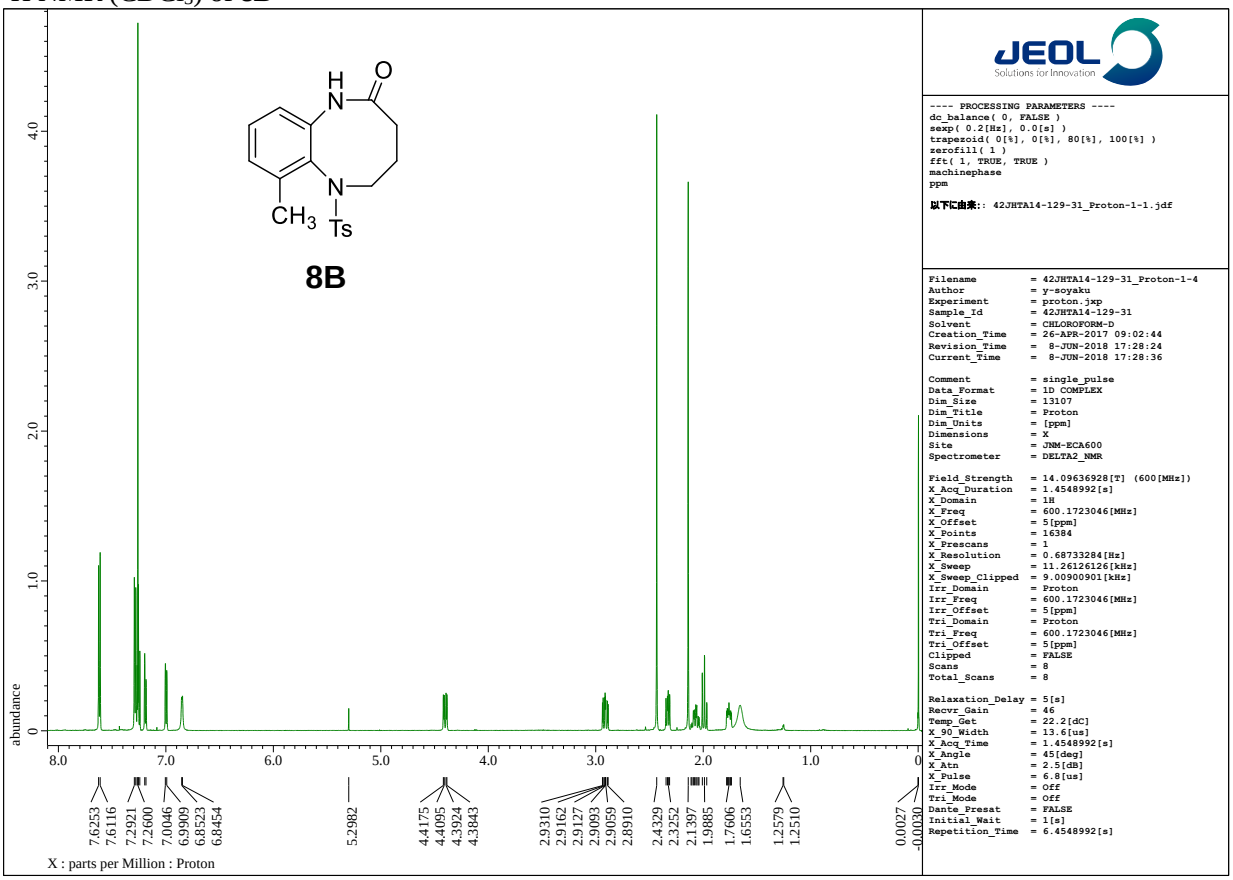
^{1}H NMR ($CDCl_3$) of **8A**



^{13}C NMR ($CDCl_3$) of **8A**



^{1}H NMR ($CDCl_3$) of **8B**



---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

以下に由来:: 42JHTA14-129-31_Proton-1-1.jdf

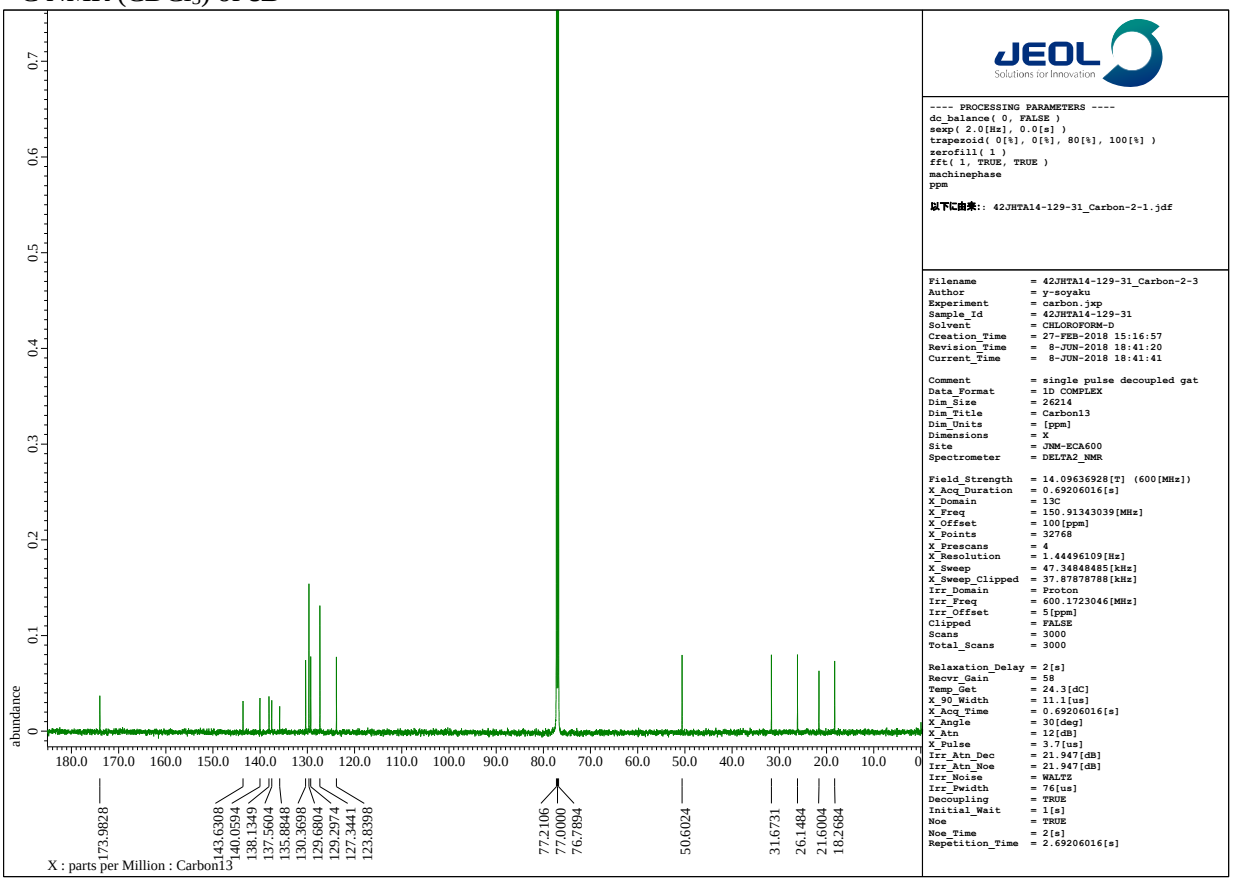
Filename = 42JHTA14-129-31_Proton-1-4
Author = y-soyaku
Experiment = proton.jxp
Sample_Id = 42JHTA14-129-31
Solvent = CHLOROFORM-D
Creation_Time = 26-APR-2017 09:02:44
Revision_Time = 8-JUN-2018 17:28:24
Current_Time = 8-JUN-2018 17:28:36

Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR

Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 1.4548992[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.68733284[Hz]
X_Sweep = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 600.1723046[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8

Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 22.2[dC]
X_90_Width = 13.6[us]
X_Acq_Time = 1.4548992[s]
X_Angle = 45[deg]
X_Atn = 2.5[dB]
X_Pulse = 6.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.4548992[s]

^{13}C NMR ($CDCl_3$) of **8B**



---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

以下に由来:: 42JHTA14-129-31_Carbon-2-1.jdf

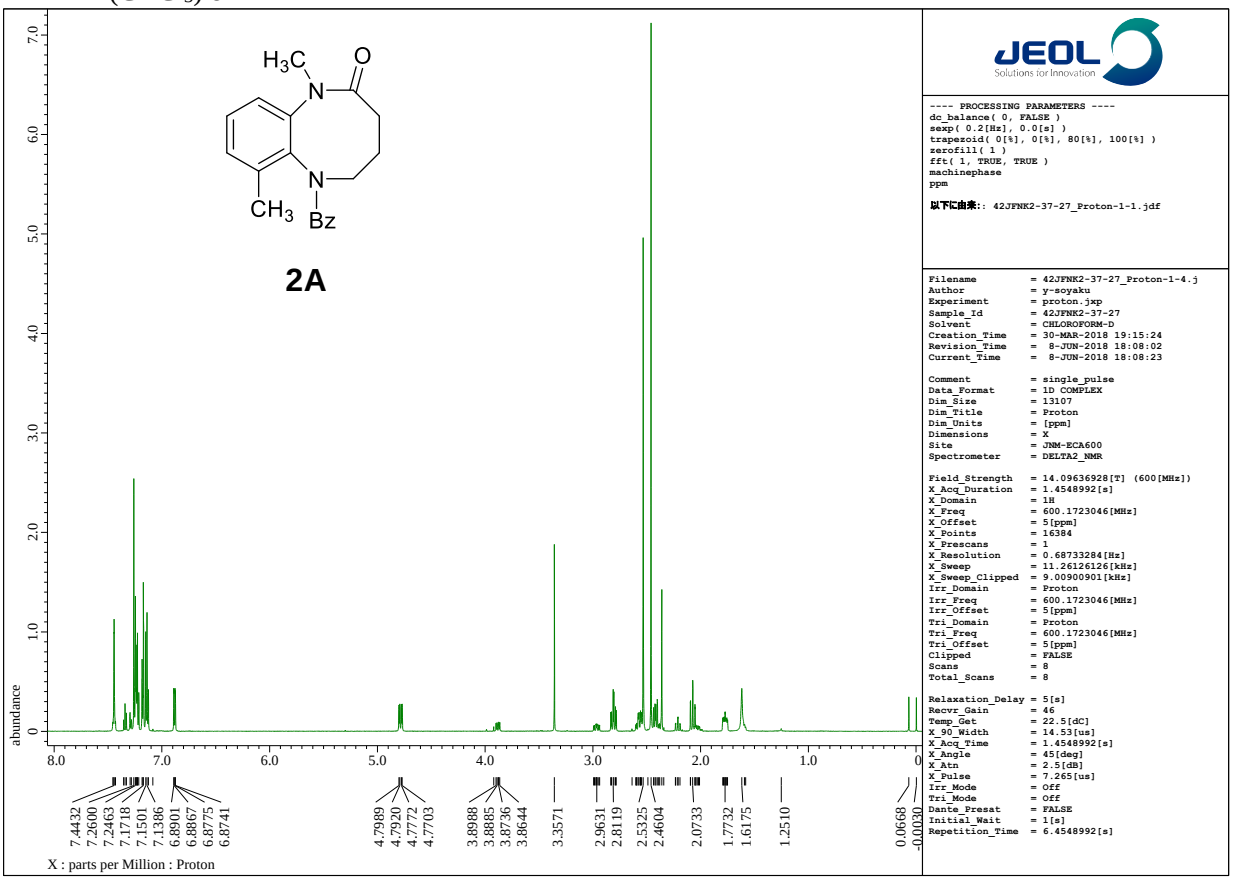
Filename = 42JHTA14-129-31_Carbon-2-3
Author = y-soyaku
Experiment = carbon.jxp
Sample_Id = 42JHTA14-129-31
Solvent = CHLOROFORM-D
Creation_Time = 27-FEB-2018 15:16:57
Revision_Time = 8-JUN-2018 18:41:20
Current_Time = 8-JUN-2018 18:41:41

Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR

Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 0.69206016[s]
X_Domain = 13C
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 3000
Total_Scans = 3000

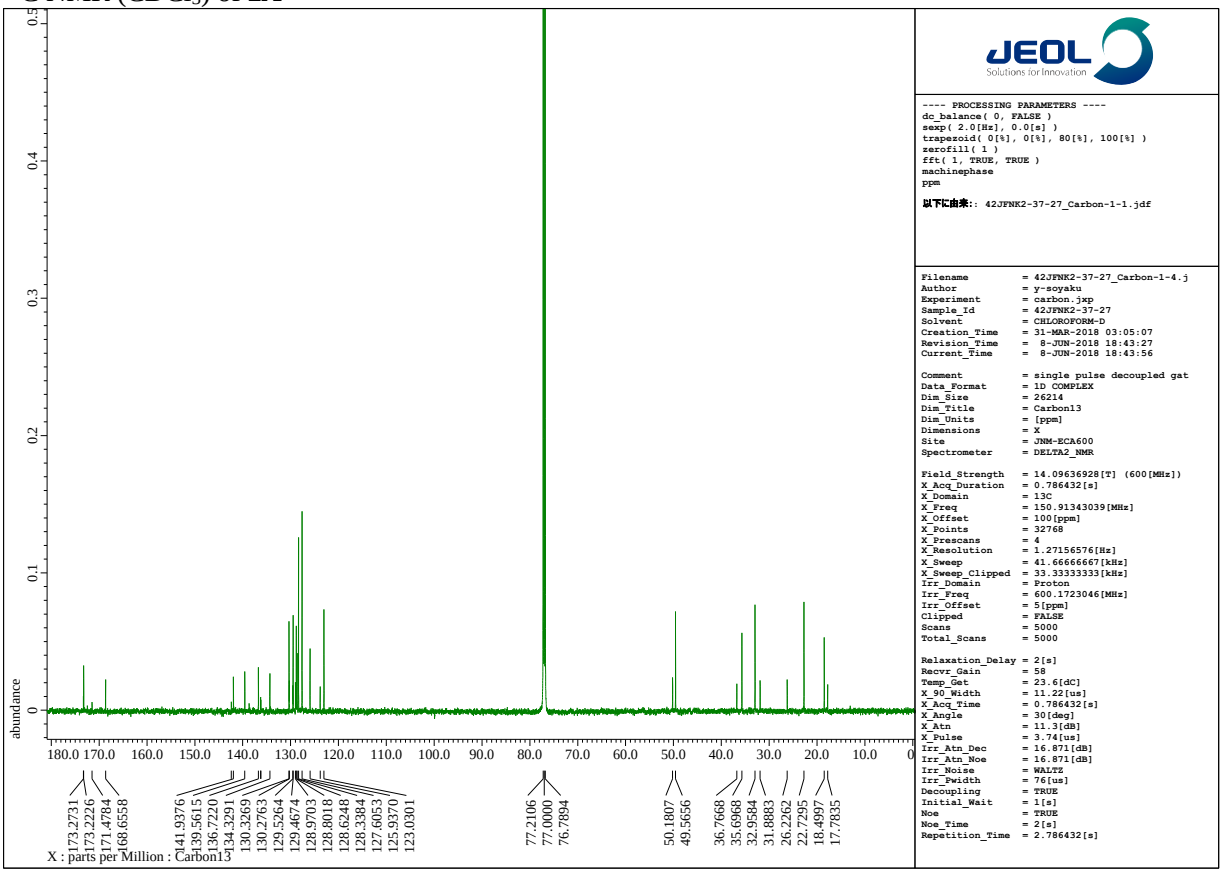
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 24.3[dC]
X_90_Width = 11.1[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.7[us]
Irr_Atn_Dec = 21.947[dB]
Irr_Atn_Noe = 21.947[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69206016[s]

^{1}H NMR ($CDCl_3$) of **2A**



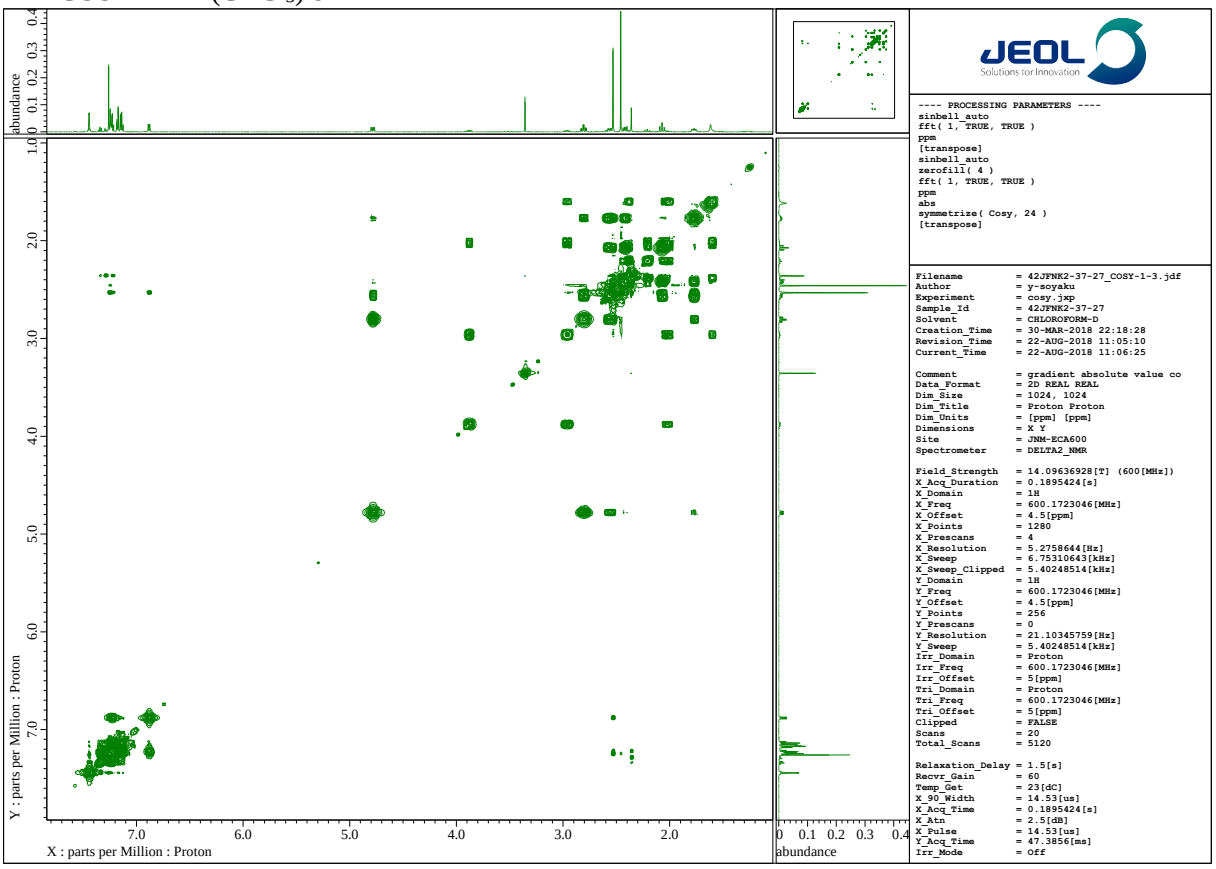

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: 42JFNK2-37-27_Proton-1-1.jdf

Filename          = 42JFNK2-37-27_Proton-1-4.j
Author            = y-soyaku
Experiment        = proton.jxp
Sample_Id         = 42JFNK2-37-27
Solvent           = CHLOROFORM-D
Creation_Time     = 30-MAR-2018 19:15:24
Revision_Time     =  8-JUN-2018 18:08:02
Current_Time      =  8-JUN-2018 18:08:23

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 1.4548992[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.68733284[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 46
Temp_Get          = 22.5[dC]
X_90_Width        = 14.53[us]
X_Acq_Time        = 1.4548992[s]
X_Angle           = 45[deg]
X_Atn             = 2.5[dB]
X_Pulse           = 7.265[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.4548992[s]
```

^{13}C NMR ($CDCl_3$) of **2A**




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: 42JFNK2-37-27_Carbon-1-1.jdf

Filename          = 42JFNK2-37-27_Carbon-1-4.j
Author            = y-soyaku
Experiment        = carbon.jxp
Sample_Id         = 42JFNK2-37-27
Solvent           = CHLOROFORM-D
Creation_Time     = 31-MAR-2018 03:05:07
Revision_Time     =  8-JUN-2018 18:43:27
Current_Time      =  8-JUN-2018 18:43:56

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.786432[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.27156576[Hz]
X_Sweep           = 41.66666667[kHz]
X_Sweep_Clipped   = 33.33333333[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 5000
Total_Scans       = 5000

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 23.6[dC]
X_90_Width        = 11.22[us]
X_Acq_Time        = 0.786432[s]
X_Angle           = 30[deg]
X_Atn             = 11.3[dB]
X_Pulse           = 3.74[us]
Irr_Atn_Dec       = 16.871[dB]
Irr_Atn_Noe       = 16.871[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.786432[s]
```

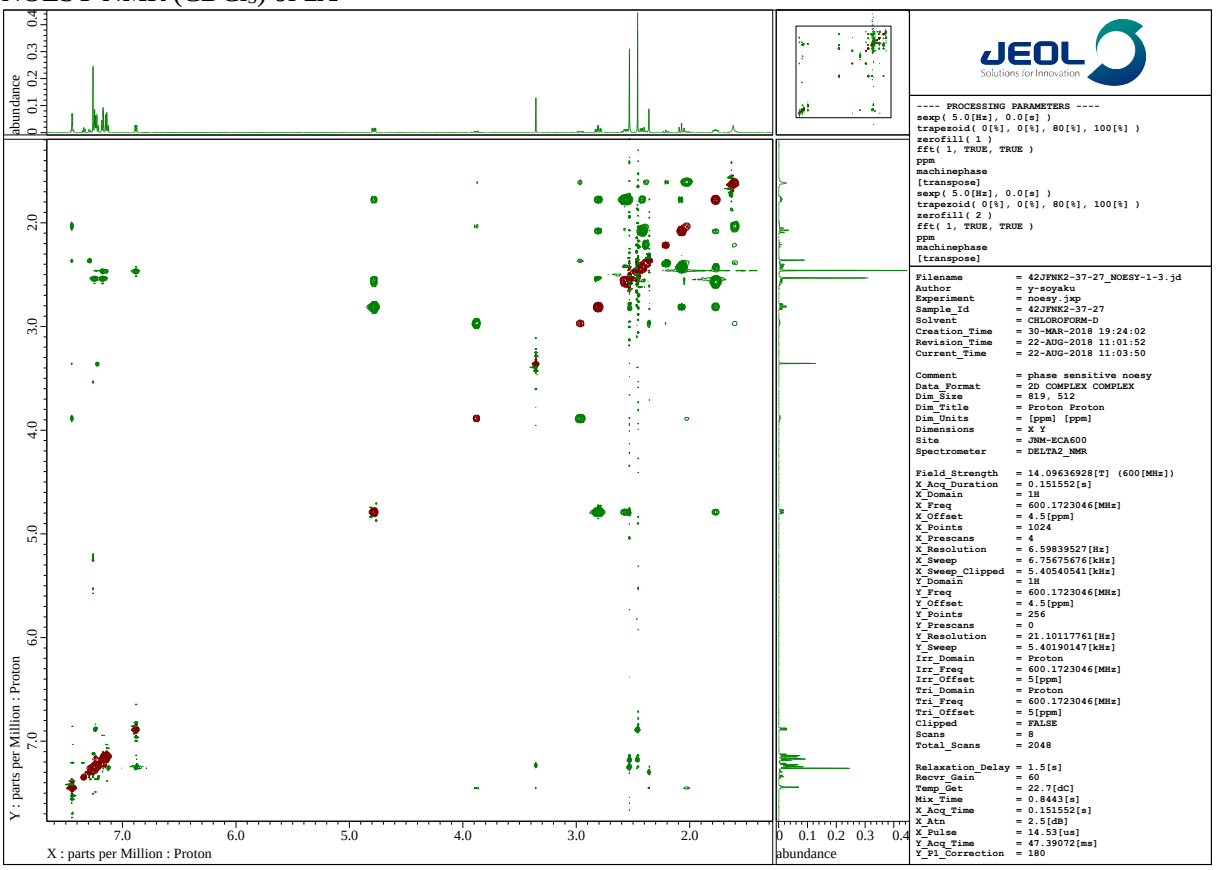
H-H COSY NMR ($CDCl_3$) of **2A**




```
---- PROCESSING PARAMETERS ----
sinbell_auto
fft( 1, TRUE, TRUE )
ppm
[transpose]
sinbell_auto
zerofill( 4 )
fft( 1, TRUE, TRUE )
ppm
abs
symmetrize( Cosy, 24 )
[transpose]
```

```
Filename            = 42JFNK2-37-27_COSY-1-3.jdf
Author              = y-soyaku
Experiment          = cosy.jxp
Sample_Id           = 42JFNK2-37-27
Solvent             = CHLOROFORM-D
Creation_Time       = 30-MAR-2018 22:18:28
Revision_Time       = 22-AUG-2018 11:05:10
Current_Time        = 22-AUG-2018 11:06:25

Comment             = gradient absolute value co
Data_Format         = 2D REAL REAL
Dim_Size            = 1024, 1024
Dim_Title           = Proton Proton
Dim_Units           = [ppm] [ppm]
Dimensions          = X Y
Site                = JNM-ECA600
Spectrometer        = DELTA2_NMR

Field_Strength      = 14.09636928[T] (600[MHz])
X_Acq_Duration      = 0.1895424[s]
X_Domain            = 1H
X_Freq              = 600.1723046[MHz]
X_Offset            = 4.5[ppm]
X_Points            = 1280
X_Prescans          = 4
X_Resolution        = 5.2758644[Hz]
X_Sweep             = 6.75310643[kHz]
X_Sweep_Clipped     = 5.40248514[kHz]
Y_Domain            = 1H
Y_Freq              = 600.1723046[MHz]
Y_Offset            = 4.5[ppm]
Y_Points            = 256
Y_Prescans          = 0
Y_Resolution        = 21.10345759[Hz]
Y_Sweep             = 5.40248514[kHz]
Irr_Domain          = Proton
Irr_Freq            = 600.1723046[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 600.1723046[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 20
Total_Scans         = 5120

Relaxation_Delay    = 1.5[s]
Recvr_Gain          = 60
Temp_Get            = 23[dC]
X_90_Width          = 14.53[us]
X_Acq_Time          = 0.1895424[s]
X_Atn               = 2.5[dB]
X_Pulse             = 14.53[us]
Y_Acq_Time          = 47.3856[ms]
Irr_Mode            = Off
```

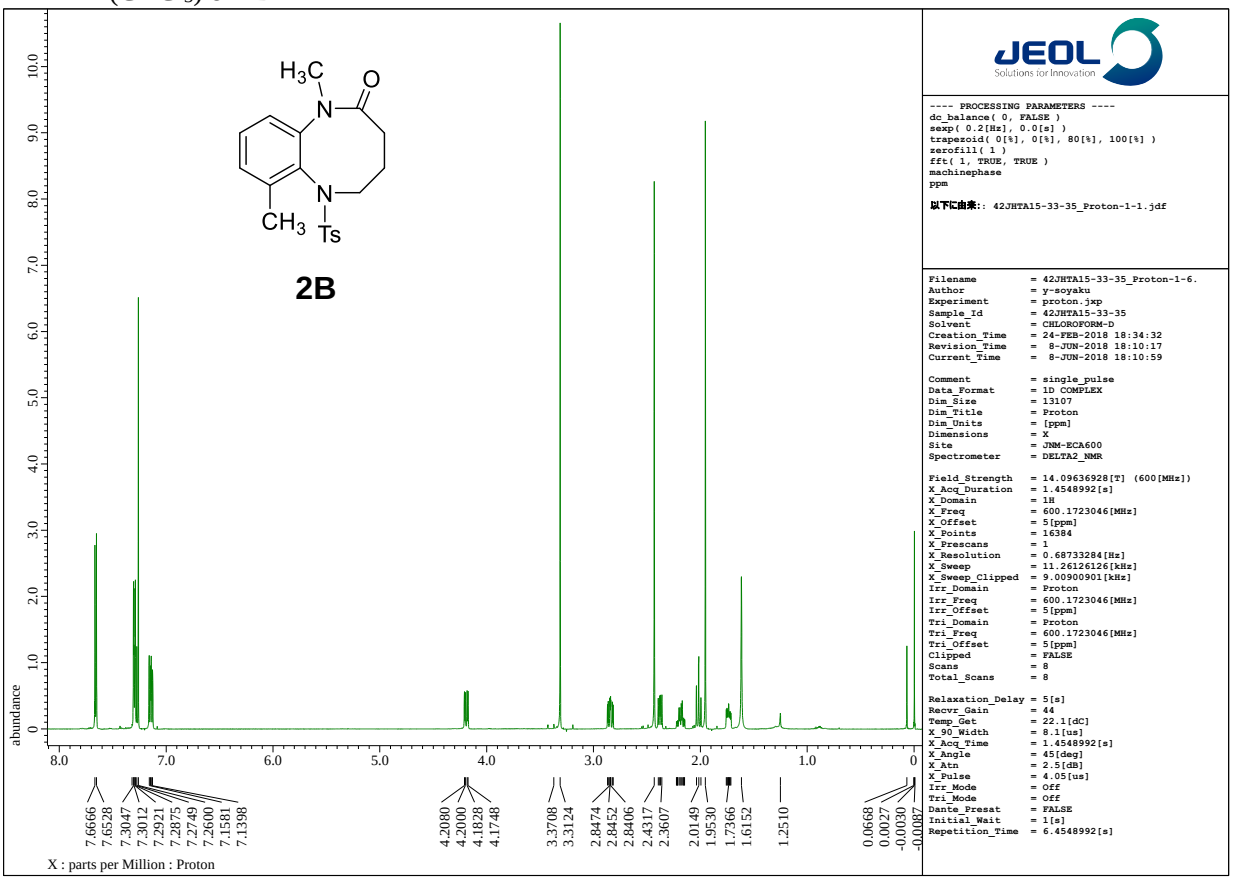
NOESY NMR ($CDCl_3$) of **2A**




```
---- PROCESSING PARAMETERS ----
sexp( 5.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
[transpose]
sexp( 5.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2 )
fft( 1, TRUE, TRUE )
ppm
machinephase
[transpose]
```

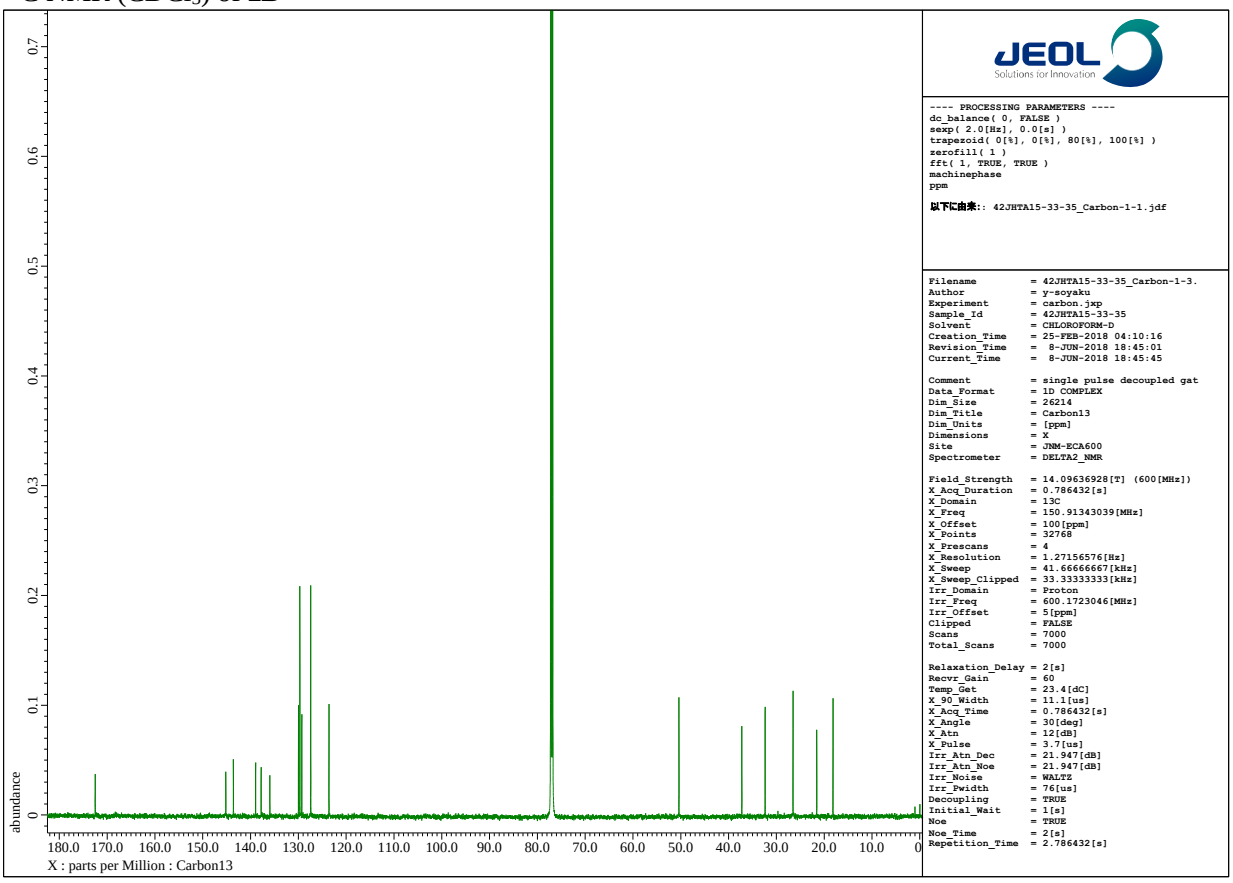
```
Filename            = 42JFNK2-37-27_NOESY-1-3.jd
Author              = y-soyaku
Experiment          = noesy.jxp
Sample_Id           = 42JFNK2-37-27
Solvent             = CHLOROFORM-D
Creation_Time       = 30-MAR-2018 19:24:02
Revision_Time       = 22-AUG-2018 11:01:52
Current_Time        = 22-AUG-2018 11:03:50

Comment             = phase sensitive noesy
Data_Format         = 2D COMPLEX COMPLEX
Dim_Size            = 819, 512
Dim_Title           = Proton Proton
Dim_Units           = [ppm] [ppm]
Dimensions          = X Y
Site                = JNM-ECA600
Spectrometer        = DELTA2_NMR

Field_Strength      = 14.09636928[T] (600[MHz])
X_Acq_Duration      = 0.151552[s]
X_Domain            = 1H
X_Freq              = 600.1723046[MHz]
X_Offset            = 4.5[ppm]
X_Points            = 1024
X_Prescans          = 4
X_Resolution        = 6.59839527[Hz]
X_Sweep             = 6.75675676[kHz]
X_Sweep_Clipped     = 5.40540541[kHz]
Y_Domain            = 1H
Y_Freq              = 600.1723046[MHz]
Y_Offset            = 4.5[ppm]
Y_Points            = 256
Y_Prescans          = 0
Y_Resolution        = 21.10117761[Hz]
Y_Sweep             = 5.40190147[kHz]
Irr_Domain          = Proton
Irr_Freq            = 600.1723046[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 600.1723046[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 2048

Relaxation_Delay    = 1.5[s]
Recvr_Gain          = 60
Temp_Get            = 22.7[dC]
Mix_Time            = 0.8443[s]
X_Acq_Time          = 0.151552[s]
X_Atn               = 2.5[dB]
X_Pulse             = 14.53[us]
Y_Acq_Time          = 47.39072[ms]
Y_P1_Correction     = 180
```

^{1}H NMR ($CDCl_3$) of **2B**



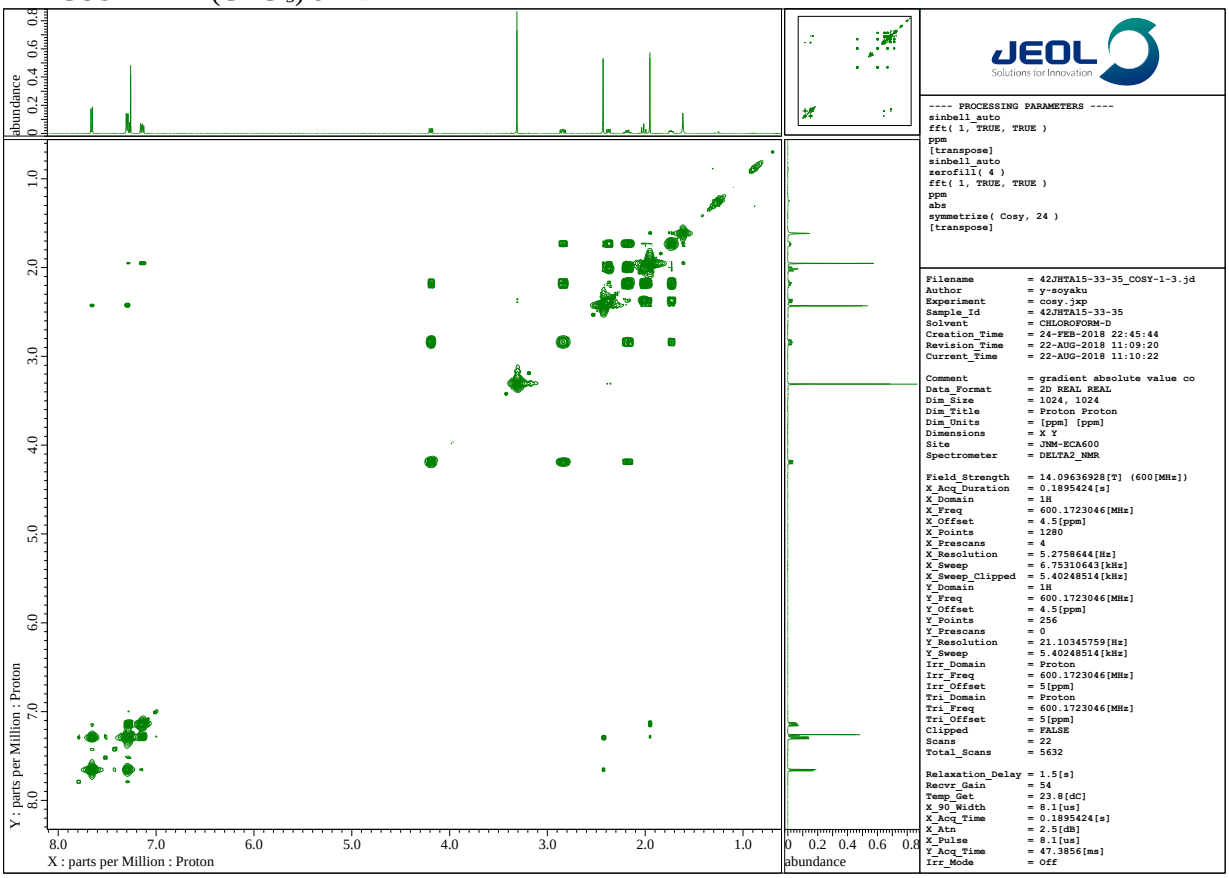

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下の由来:: 42JHTA15-33-35_Proton-1-1.jdf

Filename          = 42JHTA15-33-35_Proton-1-6.
Author            = y-soyaku
Experiment        = proton.jxp
Sample_Id         = 42JHTA15-33-35
Solvent           = CHLOROFORM-D
Creation_Time     = 24-FEB-2018 18:34:32
Revision_Time     =  8-JUN-2018 18:10:17
Current_Time      =  8-JUN-2018 18:10:59

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 1.4548992[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.68733284[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 22.1[dC]
X_90_Width        = 8.1[us]
X_Acq_Time        = 1.4548992[s]
X_Angle           = 45[deg]
X_Atn             = 2.5[dB]
X_Pulse           = 4.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.4548992[s]
```

^{13}C NMR ($CDCl_3$) of **2B**




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下の由来:: 42JHTA15-33-35_Carbon-1-1.jdf

Filename          = 42JHTA15-33-35_Carbon-1-3.
Author            = y-soyaku
Experiment        = carbon.jxp
Sample_Id         = 42JHTA15-33-35
Solvent           = CHLOROFORM-D
Creation_Time     = 25-FEB-2018 04:10:16
Revision_Time     =  8-JUN-2018 18:45:01
Current_Time      =  8-JUN-2018 18:45:45

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.786432[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.27156576[Hz]
X_Sweep           = 41.66666667[kHz]
X_Sweep_Clipped   = 33.33333333[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 7000
Total_Scans       = 7000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.4[dC]
X_90_Width        = 11.1[us]
X_Acq_Time        = 0.786432[s]
X_Angle           = 30[deg]
X_Atn             = 12[dB]
X_Pulse           = 3.7[us]
Irr_Atn_Dec       = 21.947[dB]
Irr_Atn_Noe       = 21.947[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.786432[s]
```

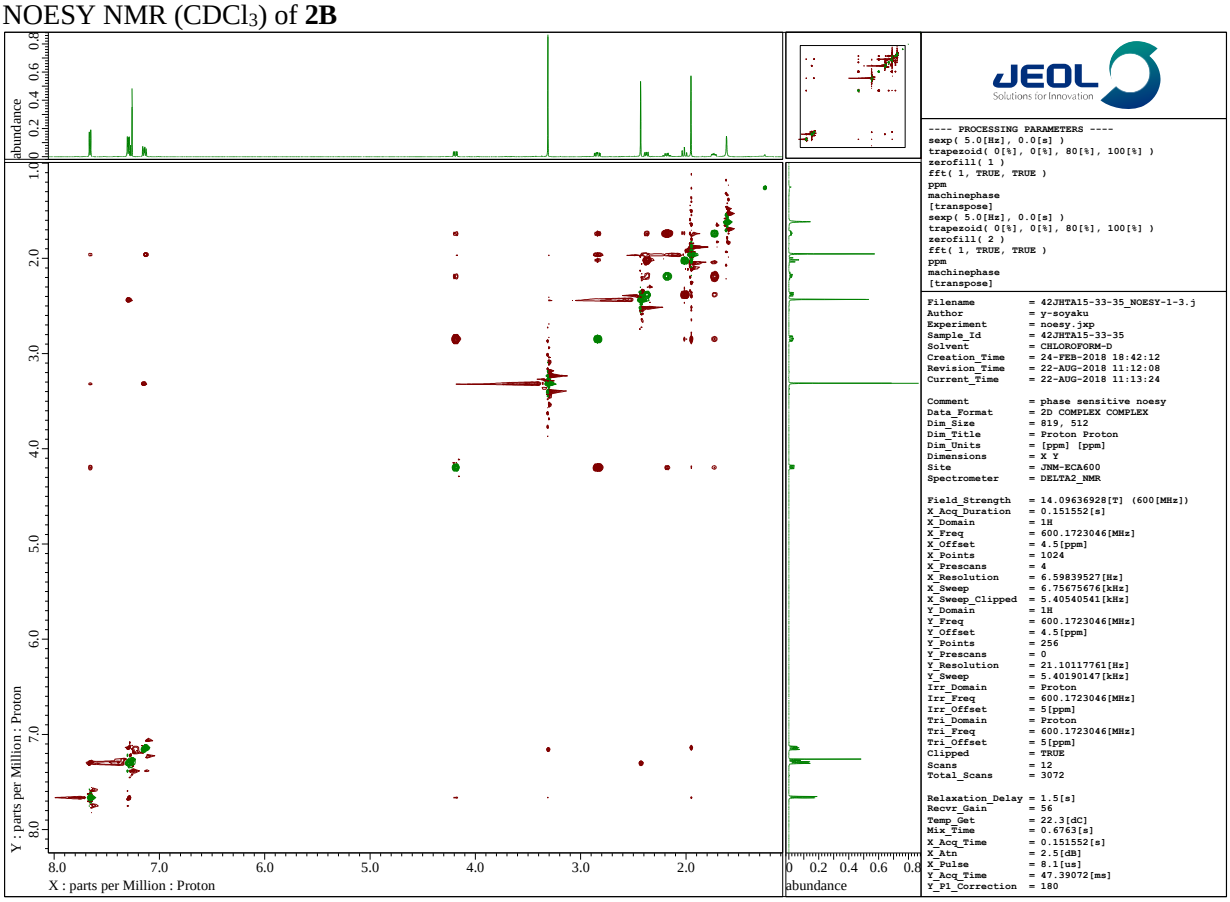
H-H COSY NMR ($CDCl_3$) of **2B**




```
---- PROCESSING PARAMETERS ----
sinbell_auto
fft( 1, TRUE, TRUE )
ppm
[transpose]
sinbell_auto
zerofill( 4 )
fft( 1, TRUE, TRUE )
ppm
abs
symmetrize( Cosy, 24 )
[transpose]
```

```
Filename          = 42JHTA15-33-35_COSY-1-3.jd
Author            = y-soyaku
Experiment        = cosy.jxp
Sample_Id         = 42JHTA15-33-35
Solvent           = CHLOROFORM-D
Creation_Time     = 24-FEB-2018 22:45:44
Revision_Time     = 22-AUG-2018 11:09:20
Current_Time      = 22-AUG-2018 11:10:22

Comment           = gradient absolute value co
Data_Format       = 2D REAL REAL
Dim_Size          = 1024, 1024
Dim_Title         = Proton Proton
Dim_Units         = [ppm] [ppm]
Dimensions        = X Y
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.1895424[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 4.5[ppm]
X_Points          = 1280
X_Prescans        = 4
X_Resolution      = 5.2758644[Hz]
X_Sweep           = 6.75310643[kHz]
X_Sweep_Clipped   = 5.40248514[kHz]
Y_Domain          = 1H
Y_Freq            = 600.1723046[MHz]
Y_Offset          = 4.5[ppm]
Y_Points          = 256
Y_Prescans        = 0
Y_Resolution      = 21.10345759[Hz]
Y_Sweep           = 5.40248514[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 22
Total_Scans       = 5632

Relaxation_Delay  = 1.5[s]
Recvr_Gain        = 54
Temp_Get          = 23.8[dC]
X_90_Width        = 8.1[us]
X_Acq_Time        = 0.1895424[s]
X_Atn             = 2.5[dB]
X_Pulse           = 8.1[us]
Y_Acq_Time        = 47.3856[ms]
Irr_Mode          = Off
```

NOESY NMR ($CDCl_3$) of **2B**




```
---- PROCESSING PARAMETERS ----
sexp( 5.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
[transpose]
sexp( 5.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2 )
fft( 1, TRUE, TRUE )
ppm
machinephase
[transpose]
```

```
Filename          = 42JHTA15-33-35_NOESY-1-3.j
Author            = y-soyaku
Experiment        = noesy.jxp
Sample_Id         = 42JHTA15-33-35
Solvent           = CHLOROFORM-D
Creation_Time     = 24-FEB-2018 18:42:12
Revision_Time     = 22-AUG-2018 11:12:08
Current_Time      = 22-AUG-2018 11:13:24

Comment           = phase sensitive noesy
Data_Format       = 2D COMPLEX COMPLEX
Dim_Size          = 819, 512
Dim_Title         = Proton Proton
Dim_Units         = [ppm] [ppm]
Dimensions        = X Y
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.151552[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 4.5[ppm]
X_Points          = 1024
X_Prescans        = 4
X_Resolution      = 6.59839527[Hz]
X_Sweep           = 6.75675676[kHz]
X_Sweep_Clipped   = 5.40540541[kHz]
Y_Domain          = 1H
Y_Freq            = 600.1723046[MHz]
Y_Offset          = 4.5[ppm]
Y_Points          = 256
Y_Prescans        = 0
Y_Resolution      = 21.10117761[Hz]
Y_Sweep           = 5.40190147[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 12
Total_Scans       = 3072

Relaxation_Delay  = 1.5[s]
Recvr_Gain        = 56
Temp_Get          = 22.3[dC]
Mix_Time          = 0.6763[s]
X_Acq_Time        = 0.151552[s]
X_Atn             = 2.5[dB]
X_Pulse           = 8.1[us]
Y_Acq_Time        = 47.39072[ms]
Y_P1_Correction   = 180
```

4. References

[s1] RDKit: Open-source cheminformatics, http://www.rdkit.org.

[s2] For predicting free energy in solution, the SMD (Standard Molecular Data) solvation model was used, see; A. V. Marenich, C. J. Cramer, and D. G. Truhlar, *J. Phys. Chem. B*, 2009, **113**, 6378.

[s3] *Gaussian 09, Revision C.01*: M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2010.