On the prevalence of bridged macrocyclic pyrroloindolines formed in regiodivergent alkylations of tryptophan

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Supporting Material

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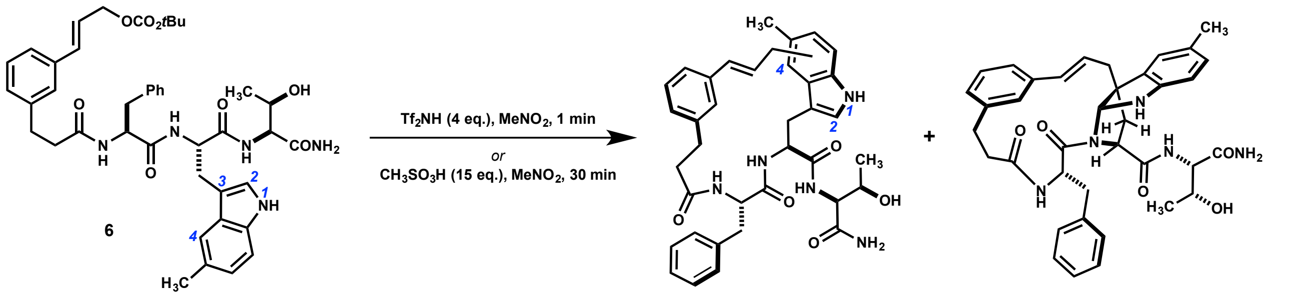
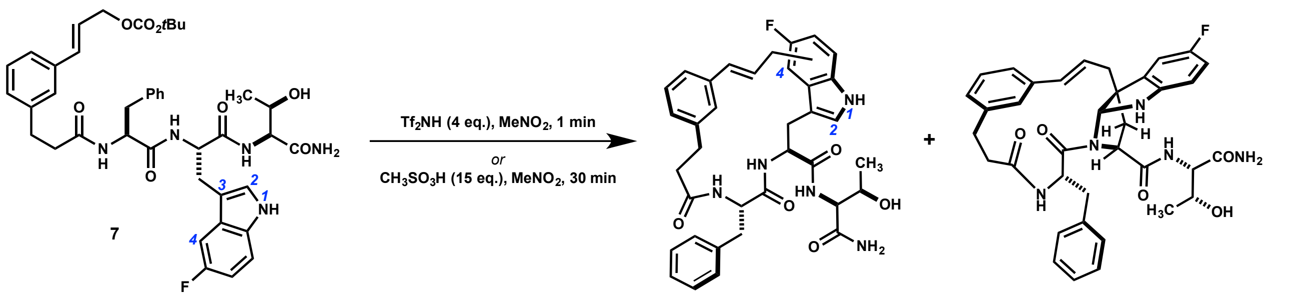
A. Supplementary Figures

Figure S1. **Phe-Trp(5Me)-Thr**. Comparative performance of Tf_2NH and $MeSO_3H$ in cyclization of linear precursor **6**.



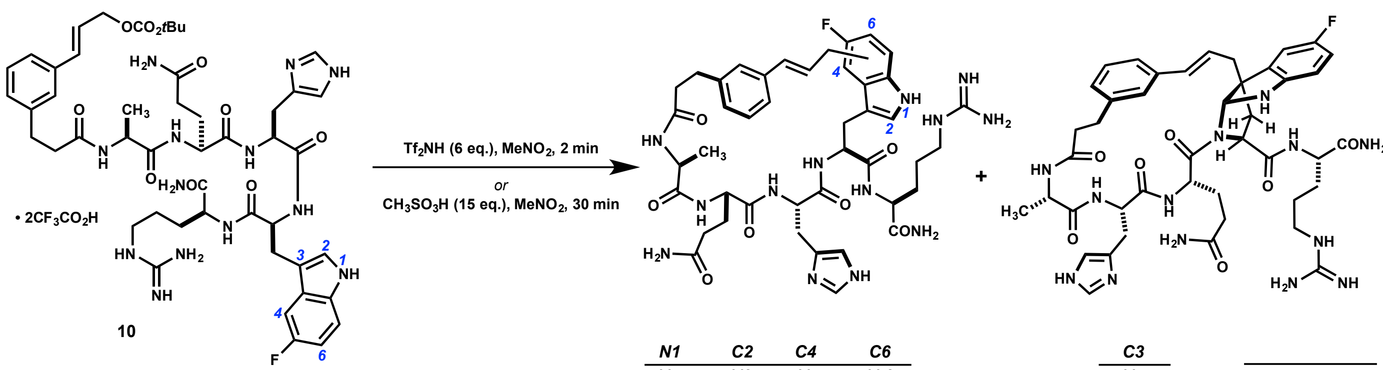
	N1	C2	C4	C3	Combined Yield
	8a	**8b** (% yield)	**8c**	**8d** (% yield)	
Tf_2NH	*n.d.*	*11*	*30*	*8*	***50 %***
CH_3SO_3H	*n.d.*	*8*	*27*	*8*	***43 %***

Figure S2. **Phe-Trp(5F)-Thr**. Comparative performance of Tf_2NH and $MeSO_3H$ in cyclization of linear precursor **7**.



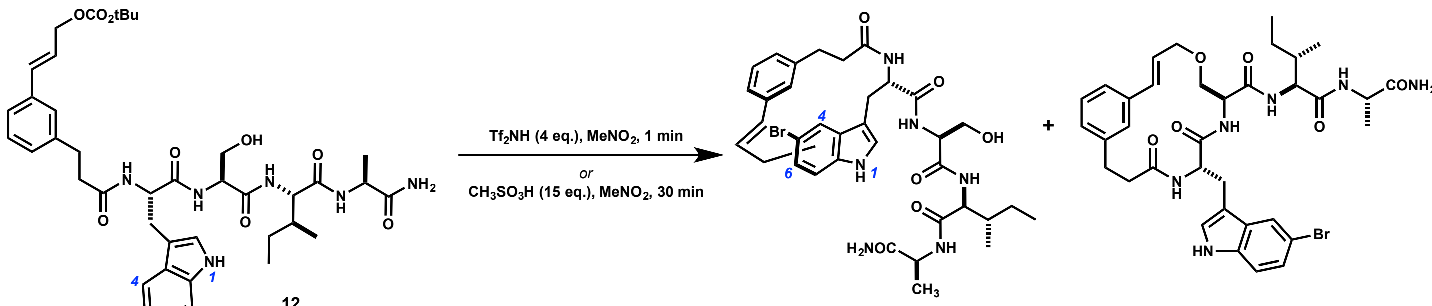
	N1	C2	C4	C3	Combined Yield
	9a	**9b** (% yield)	**9c**	**9e** (% yield)	
Tf_2NH	*10*	*17*	*14*	*22*	***62 %***
CH_3SO_3H	*7*	*7*	*16*	*15*	***45 %***

Figure S3. **Ala-Gln-His-Trp(5F)-Arg**. Comparative performance of Tf_2NH and $MeSO_3H$ in cyclization of linear precursor **10**.



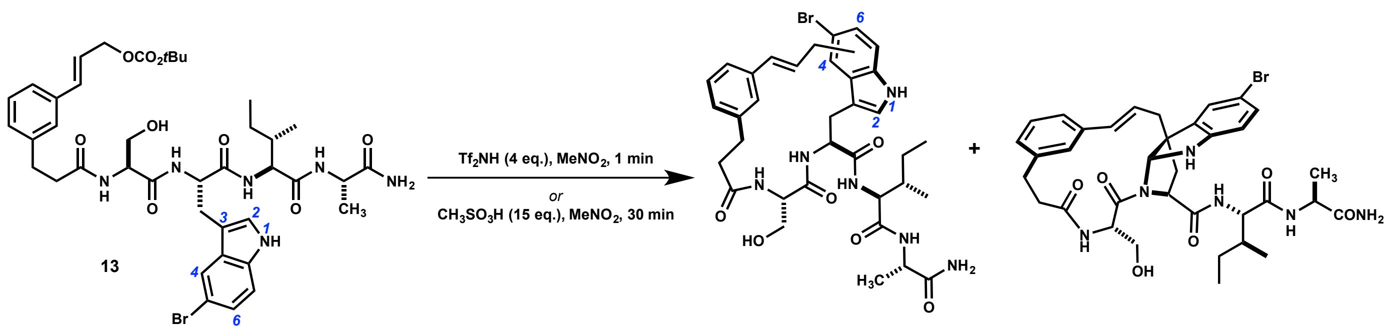
	N1	C2	C4	C6	C3	Combined Yield
	11a	**11b** (% yield)	**11c**	**11d**	**11e** (% yield)	
Tf_2NH	*17*	*12*	*6*	*48*	*8*	***91 %***
CH_3SO_3H	*6*	*4*	*3*	*19*	*5*	***37 %***

Figure S4. **Trp(5Br)-Ser-Ile-Ala**. Comparative performance of Tf_2NH and $MeSO_3H$ in cyclization of linear precursor **12**.



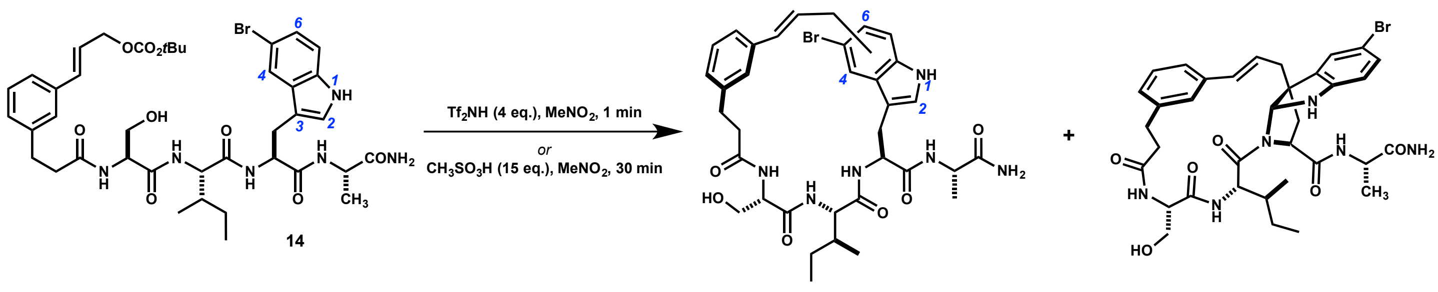
	N1	C6	C7	Ser	Combined Yield
	16d	**16a**	**16c**	**16b**	
	% yield			% yield	
Tf_2NH	*11*	*25*	*9*	*6*	***52 %***
CH_3SO_3H	*6*	*27*	*14*	*0*	***47 %***

Figure S5. **Ser-Trp(5Br)-Ile-Ala**. Comparative performance of Tf_2NH and $MeSO_3H$ in cyclization of linear precursor **13**.



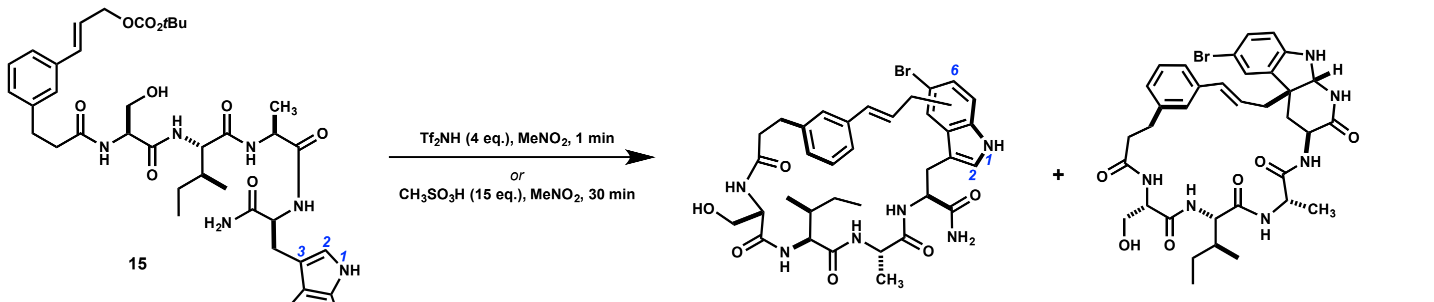
	N1	C2	C4	C6	C3	Combined Yield
	17d	**17a**	**17b**	**17c**	**17e**	
	% yield				% yield	
Tf_2NH	*6*	*14*	*14*	*7*	*14*	***63 %***
CH_3SO_3H	*0*	*12*	*13*	*10*	*8*	***42 %***

Figure S6. **Ser-Ile-Trp(5Br)-Ala**. Comparative performance of Tf_2NH and $MeSO_3H$ in cyclization of linear precursor **14**.



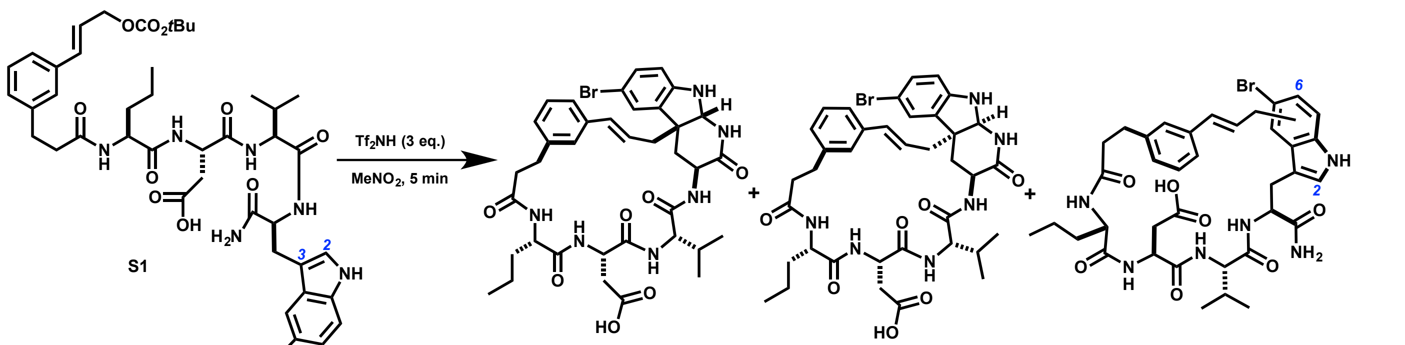
	N1	C2	C4	C6	C3	Combined Yield
	18e	**18a**	**18b**	**18d**	**18c**	
	% yield				% yield	
Tf_2NH	*6*	*19*	*4*	*21*	*27*	***77 %***
CH_3SO_3H	*7*	*5*	*2*	*26*	*7*	***48 %***

Figure S7. **Ser-Ile-Ala-Trp(5Br)**. Comparative performance of Tf_2NH and $MeSO_3H$ in cyclization of linear precursor **15**.



	N1	*C2*	*C6*	*C3*	
	19d	**19a**	**19c**	**19b**	***Combined Yield***
		% yield		% yield	
Tf_2NH	*11*	*13*	*39*	*10*	***72 %***
CH_3SO_3H	*8*	*10*	*40*	*10*	***68 %***

Figure S8**. Nva-Asp-Val-Trp(5Br).** Cyclization of **S1** promoted by Tf_2NH forms diastereomeric pyridoindolines **S2a**&**b**.

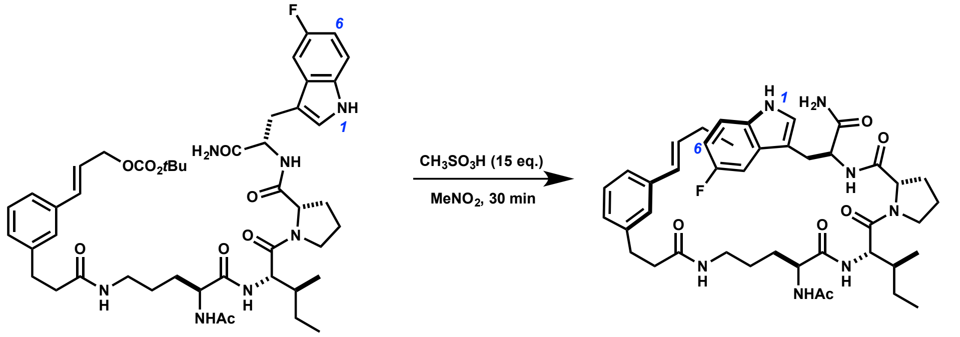


S2a (*11%*)

S2b (*6%*)

C2	*C6*	
S2c	**S2d**	***Combined Yield***
% yield		
26	*18*	***61%***

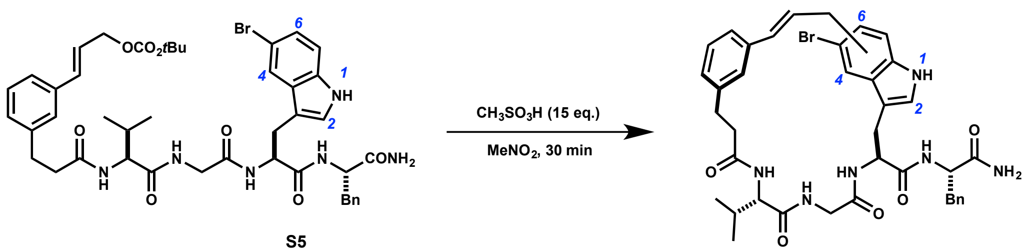
Figure S9. **Ac-Orn(H)-Ile-Pro-Trp(5F)**. Cyclization of **S3** promoted by $MeSO_3H$ did not yield an analogous pyrroloindoline.



S3

N1	*C6*	
S4b	**S4a**	***Combined Yield***
% isolated yield		
19	*34*	***53 %***

Figure S10. **Val-Gly-Trp(5Br)-Phe**. Cyclization of **S5** promoted by $MeSO_3H$ did not yield an analogous pyrroloindoline.



N1	C2	C4	C6
S6d	S6a	S6c	S6b
	% isolated yield		
14	*6*	*8*	*20*

Combined Yield
48 %

B. Computational Results and Discussion

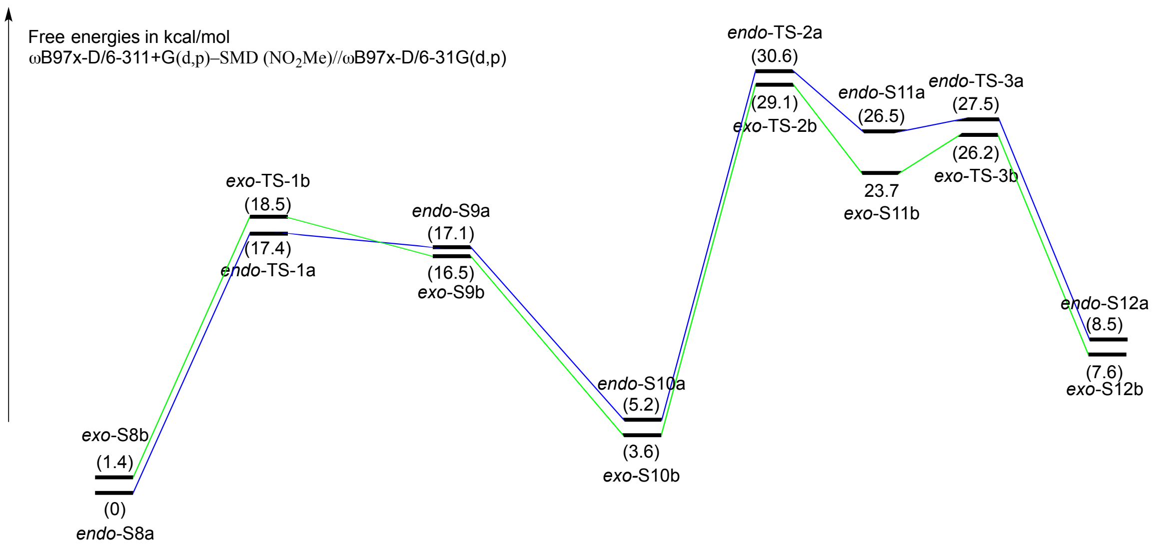


Figure S11. Free energy profile for the 1,2-rearrangement of *exo-* and *endo*-pyrroloindolines shown in Figure 3 (where R=Me). (ωB97x-D/6-311+G(d,p)–SMD (nitromethane) // ωB97x-D/6-31G(d,p)) The relative free energies are compared with *endo*-**S8a** and are reported in kcal/mol. The blue pathway represents the *endo* reaction profile, and the green pathway represents the *exo* reaction profile.

The free energy profile in Figure S11 was calculated for the cinnamyl 1,2-rearrangement shown in Figure 3 (where R=Me). The neutral pyrroloindolines are protonated at the acetyl oxygen to form *endo*-**S8a** and *exo*-**S8b**, shown in Figure S12. The protonation transition state was not determined. *Endo*-**S8a** is the lowest-energy intermediate of the free energy profile. The *exo*-pyrroloindolinium, *exo*-**S8b**, is higher in energy by 1.4 kcal/mol. The calculated geometries of intermediates **S8**-**S10**, and **S12** are shown in Figure S12. The protonated species proceed through the ring opening transition structures, *endo*-**TS-1a** and *exo*-**TS-1b**, shown in Figure S13.

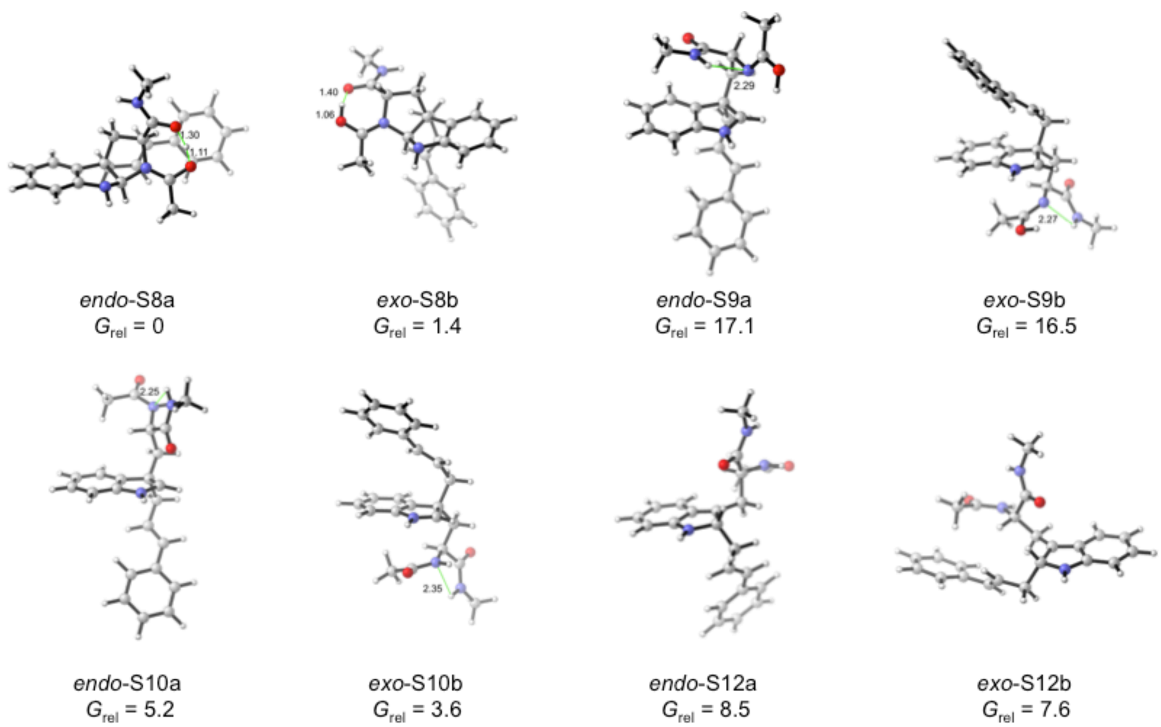
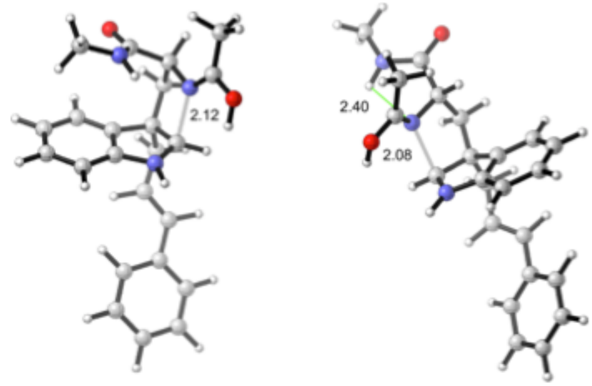


Figure S12. Lowest-energy intermediates S8-S10, and S12 for the free energy profile in Figure S11. (ωB97x-D/6-311+G(d,p)–SMD (nitromethane) // ωB97x-D/6-31G(d,p)). The relative free energies are compared with *endo*-**S8a** and are reported in kcal/mol.



endo-TS-1a
$\Delta G^{\ddagger}$ = 17.4

exo-TS-1b
$\Delta G^{\ddagger}$ = 17.1

Figure S13. Lowest-energy transition structures *endo*-TS-1a and *exo*-TS-1b for the ring opening reaction of *endo*-S8a and *exo*-S8b. (ωB97x-D/6-311+G(d,p)–SMD (nitromethane) // ωB97x-D/6-31G(d,p)). The free energy of activation barrier for *endo*-**TS-1a** is compared to *endo*-**S8a**, and the free energy of activation barrier for *exo*-**TS-1b** is compared to *exo*-**S8b**. The free energies are reported in kcal/mol.

The *endo* transition structure *endo*-**TS-1a** has a barrier of 17.4 kcal/mol, relative to *endo*-**S8a**. The *exo* transition structure *exo*-**TS-1b** has a barrier of 17.1 kcal/mol, relative to *exo*-**S8b**. Intermediates *endo*-**S9a** and *exo*-**S9b** are iminol tautomers, which tautomerize to amides *endo*-**S10a** and *exo*-**S10b**. The transition states for these tautomerizations were not calculated. The pyrroloindoliniums *endo*-**S10a** and *exo*-**S10b** undergo 1,2-rearrangement through a stepwise mechanism of disassociation, leading to a complex between the indolinium and cinnamyl group, followed by addition to indole C2. The transition structures *endo*-**TS-2a** and *exo*-**TS-2b** have barriers of 30.6 and 27.7 kcal/mol, respectively, and are shown in Figure S14. The dissociation transition structures lead to complexes *endo*-**S11a** and *exo*-**S11b**. These complexes undergo addition to indole C2 via transition structures *endo*-**TS-3a** and *exo*-**TS-3b** with barriers of 27.5 kcal/mol and 24.8 kcal/mol, respectively. These transition structures lead to cations *endo*-**S12a** and *exo*-**S12b**, which are deprotonated to form the neutral C2-cinnamyl indole **S13**.

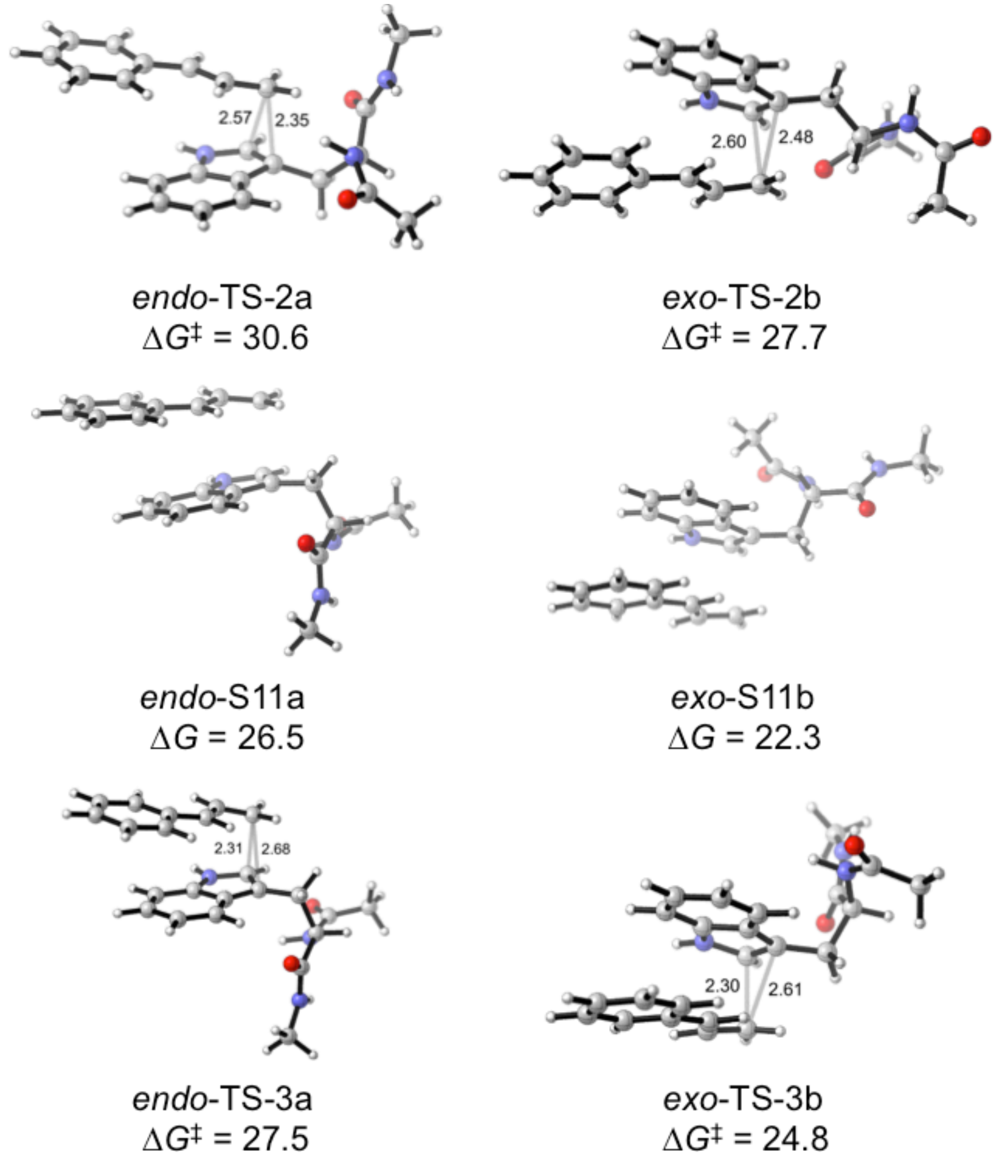


Figure S14. Lowest-energy transition structures *endo*-TS-2a, *exo*-TS-2b, *endo*-TS-3a, and *exo*-TS-3b for the stepwise 1,2-rearrangement of *endo*-**S10a** and *exo*-**S10b**, and the complexes *endo*-**S11a** and *exo*-**S11b** interceding these transition structures (ωB97x-D/6-311+G(d,p)–SMD (nitromethane) // ωB97x-D/6-31G(d,p)). The free energy of activation barrier for *endo*-**TS-2a** and *endo*-**TS-3a** is compared to *endo*-**S8a**, and the free energy of activation barrier for *exo*-**TS-2b** and *exo*-**TS-3b** is compared to *exo*-**S8b**. The difference in free energy for *endo*-**S11a** is compared to *endo*-**S8a**, and *exo*-**S11b** is compared with *exo*-**S8b**. The free energies are reported in kcal/mol.

It was found that the dissociation transition structures *endo*-**TS-2a** and *exo*-**TS-2b** are the highest-energy barriers and are rate-determining. The difference in free energy of activation for the *exo*- and *endo*- processes is 2.9 kcal/mol, where the *exo*- is lower in energy. The difference in energy arises from two factors: (1) The greater stability of intermediate *endo*-**S8a** over *exo*-**S8b** by 1.4 kcal/mol. (2) The stability of the *exo*-**TS-2b** over *endo*-**TS-2a** by 1.5 kcal/mol. The *exo*- transition structure is lower in energy due to stabilizing electrostatic interactions between the carbonyl oxygen of the acetamide group and electropositive indolinium ring. This stabilizing electrostatic effect does not occur in *endo*-**TS-2a**, due to the configuration of the alanyl moiety.

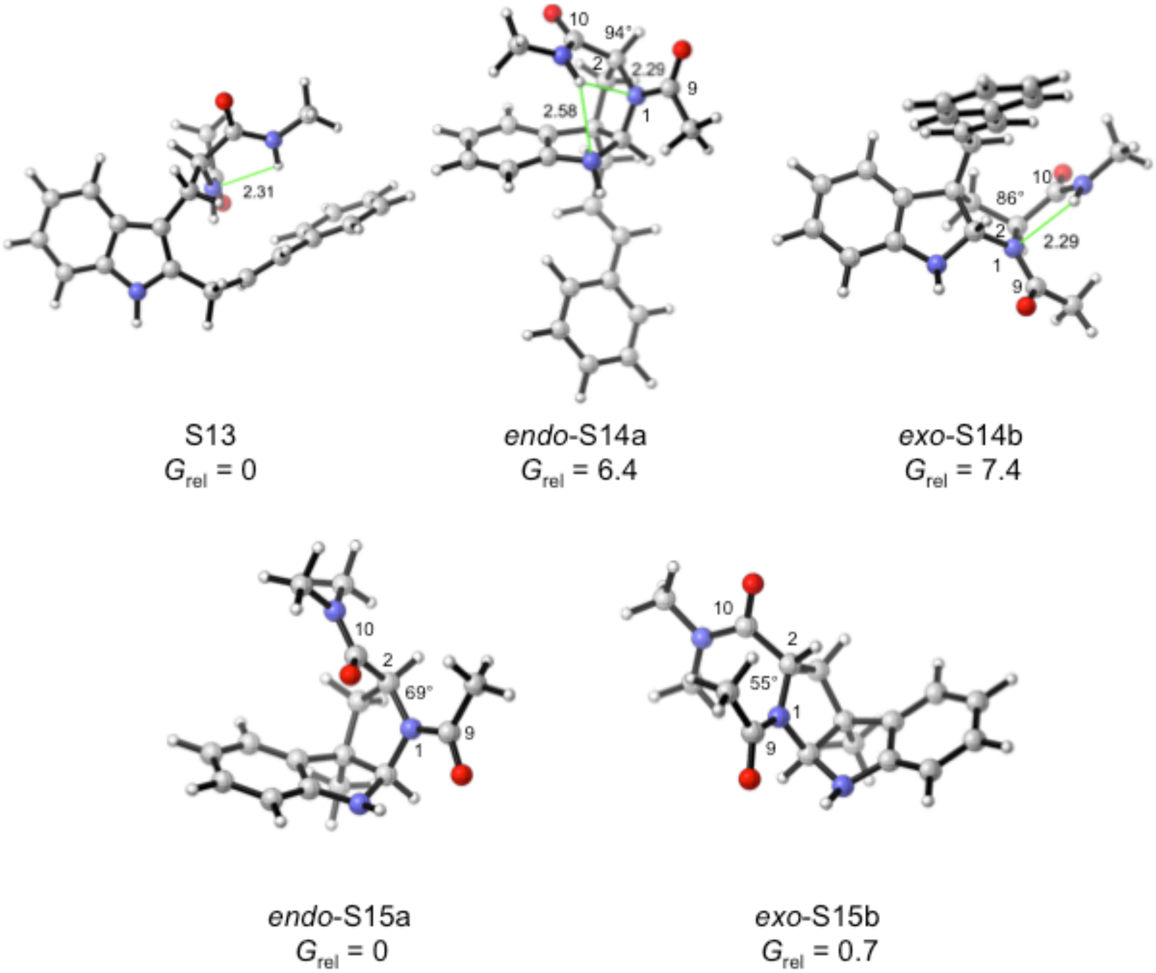
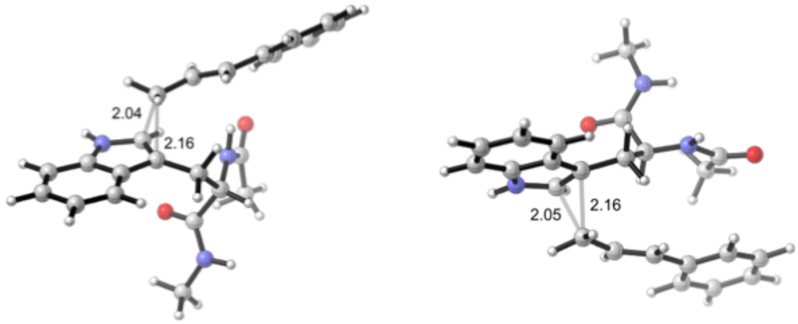


Figure S15. Lowest-energy ground states for the neutral pyrroloindolines S14, S15, and C2-linked product S13. (ωB97x-D/6-311+G(d,p)–SMD (nitromethane) // ωB97x-D/6-31G(d,p)). The relative free energies of *endo*-**S14a** and *exo*-**S14b** are compared to **S13**. The relative free energy of *N*,*N*-dimethylated variant *exo*-**S15b** is compared to *endo*-**S15a**. The relative free energies are reported in kcal/mol.

The neutral pyrroloindolines, shown in Figure S15, were calculated and it was found that the aromatized C2-cinnamyl indole **S13** was the thermodynamic product. The *endo*-pyrroloindoline *endo*-**S14a** is 6.4 kcal/mol higher in energy than **S13**. The *exo*-**S14b** is 7.4 kcal/mol higher in energy than **S13**. Consistent with findings of Crich,[1] the *exo*-**S14b** is less stable compared to *endo*-**S14a** due primarily to 1,3-allylic strain. The 1.0 kcal/mol difference arises from the steric interactions between the N1-acetyl group and C2-acetamidyl group. As shown in Figure S15, the exocyclic dihedral angle between C9-N1-C2-C10 is 94° for *endo*-**S14a** and 86° for *exo*-**S14b**. To test whether the difference in energy was due to an intramolecular hydrogen bond we calculated a model where the acetamide group was *N,N*-dimethylated (the cinnamyl group was truncated to methyl). From this, the lowest-energy model pyrroloindolines *endo*-**S15a** and *exo*-**S15b**, shown in Figure **S15**. The *endo*-model **S15a** is 0.7 kcal/mol more stable than the *exo*-model **S15b**. The dihedral angles of the C9-N1-C2-C10 bonds are 69° for the *endo*-**S15a** and 55° for the *exo*-**S15b**, consistent with the findings for *endo*-**S14a** and *exo*-**S14b**.

[1] D. Crich, M. Bruncko, S. Natarajan, B. Teo and D. Tocher, *Tetrahedron* 1995, **51**, 2215.

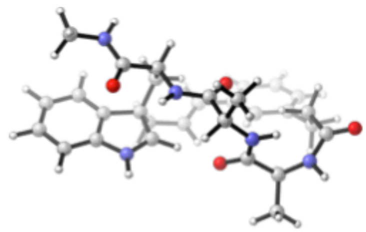


endo-TS-4a
$\Delta G^{\ddagger}$ = 33.8

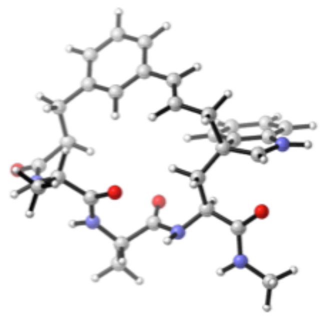
exo-TS-4b
$\Delta G^{\ddagger}$ = 28.0

Figure S16. Lowest-energy transition structures *endo*-TS-4a and *exo*-TS-4b for the concerted 1,2-rearrangement of *endo*-S10a and *exo*-S10b. (ωB97x-D/6-311+G(d,p)–SMD (nitromethane) // ωB97x-D/6-31G(d,p)). The free energy of activation barrier for *endo*-**TS-4a** is compared to *endo*-**S8a**, and the free energy of activation barrier for *exo*-**TS-4b** is compared to *exo*-**S8b**. The free energies are reported in kcal/mol.

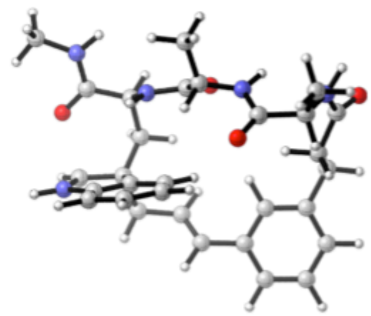
In addition to the stepwise 1,2-rearrangement, a concerted pathway was also located, and is shown in Figure S16. The transition structures *endo*-**TS-4a** and *exo*-**TS-4b** were the lowest-energy structures located for the *exo* and *endo* pathways. Both transition structures are concerted 1,2-shifts of the cinnamyl group from indole C3 to C2. The free energy of activation barrier for *endo*-**TS-4a** is 33.8 kcal/mol, and 28.0 kcal/mol for *exo*-**TS-4b**. The stabilization of the *exo* transition structure over the *endo* is due to similar effects observed for the **TS-2** series, where there is a stabilizing electrostatic interaction in *exo*-**TS-4b** not present in *endo*-**TS-4a**. The activation barrier for *endo*-**TS-4a** is 3.2 kcal/mol higher than *endo*-**TS-2a**, and thus the *endo* pyrroloindolines react via stepwise 1,2-rearrangements. However, *exo*-**TS-4b** is only 0.3 kcal/mol higher in energy than *exo*-**TS-2b**. Thus, *exo*-pyrroloindolines can react through either stepwise or concerted 1,2-rearrangements.



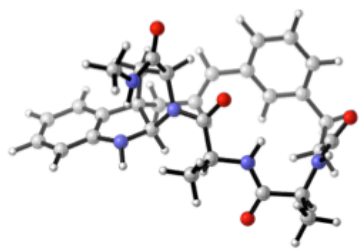
endo-S16a
G_{rel} = 0



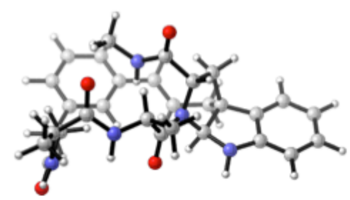
endo-S16a-2
G_{rel} = 5.4



exo-S16b
G_{rel} = 4.5



endo-S17a
G_{rel} = 0



exo-S17b
G_{rel} = 1.1

Figure S17. Lowest-energy ground states of truncated macrocycles S16-S17. (ωB97x-D/6-311+G(d,p)–SMD (nitromethane) // ωB97x-D/6-31G(d,p)). These models are truncated, neutral variants of macrocyclic pyrroloindolines **18c** (for **S17**) and **20** (for **S16**) where peptide side chains were replaced with methyl groups. The relative free energies of *endo*-**S14a** and *exo*-**S14b** are compared to **S13**. The relative free energy of *exo*-**S15b** is compared to *endo*-**S15a**. The relative free energies are reported in kcal/mol.

Truncated models of macrocyclic *endo*-pyrroloindoline **18c** and its open-chain iminol tautomer **20** – as well as diastereomers corresponding to *exo*-**18c** (not isolated) – were calculated to estimate the feasibility of stepwise 1,2-rearrangement in macrocyclic systems. Peptide side chains were replaced with methyl groups for calculation. *Endo*-**S16a** and *exo*-**S16b** are the lowest-energy calculated intermediates, analogous to *endo*-**S10a** and *exo*-**S10b**. It was found that *endo*-**S16a** is preferred over *exo*-**S16b** by 4.5 kcal/mol. The cinnamyl group in *endo*-**S16a** does not overlap with the indolinium ring. However, we located a conformation higher in energy by 5.4 kcal/mol, *endo*-**S16a-2**, where the cinnamyl group can overlap with the indolinium ring analogously to complexes **S11a,b**. *Exo*-**S16b** adopts a conformation where the cinnamyl group is proximal to the indolinium ring and is 4.5 kcal/mol higher in energy than *endo*-**S16a**. Thus, these conformations suggest that the dissociated complex geometry in the stepwise 1,2-rearrangement of model pyrroloindolines is also feasible in macrocyclic variants. For neutral pyrroloindolines, *endo*-**S17a** was found to be lower in energy than *exo*-**S17b** by 1.1 kcal/mol. This energy difference was found to arise from 1,3-allylic strain, similarly to the model substrates *endo*-**S14a** and *exo*-**S14b**.

Computational Methods

All quantum chemical calculations were performed with Gaussian 09.[2] Geometry optimizations and frequencies were calculated with the ωB97x-D[3] (*in vacuo*) density functional with the 6-31G(d,p) basis set. Optimized geometries were verified by frequency calculations as minima (zero imaginary frequencies) or transition structures (a single imaginary frequency). Free energy corrections were determined using unscaled ωB97x-D/6-31G(d,p) vibrational frequencies assuming a standard state of 1 atm and 298.15 K. Errors in the treatment of low modes as harmonic oscillations were mitigated by use of the quasiharmonic approximation proposed by Truhlar and coworkers.[4] Single point energy calculations were performed on optimized geometries with ωB97x-D/6-311+G(d,p). The free energies reported herein were determined by adding zero-point energy and thermal correction determined using ωB97x-D/6-31G(d,p) to electronic energies computed at the ωB97x-D/6-311+G(d,p) level of theory.

Monte Carlo conformational searches were performed on the intermediates using the OPLS-2005 force field[5] in Maestro/Macromodel.[6] Reactive conformations with the distance between the bond-forming atoms shorter than 4.0 Å were used as input geometries for transition structure optimizations.

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[2] Gaussian 09, Revision D.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. Jr. Montgomery, J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, 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, Ö. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2009**.**

[3] J.-D. Chai, and M. Head-Gordon, *Phys. Chem. Chem. Phys.* 2008, **10**, 6615.

[4] Y. Zhao, and D. G. Truhlar, *Phys. Chem. Chem. Phys.* 2008, **10**, 2813.

[5] J. L. Banks, H. S. Beard, X. Y. Cao, A. E. Cho, W. Damm, R. Farid, A. K. Felts, T. A. Halgren, D. T. Mainz, J. R. Maple, R. Murphy, D. M. Philipp, M. P. Repasky, L. Y. Zhang, B. J. Berne, R. A. Friesner, E. Gallicchio, and R. M. Levy, *J. Comp. Chem.* 2005, **26**, 1752.

[6] Schrödinger Release 2015-3: MacroModel, version 10.9, Schrödinger, LLC, New York, NY, 2015.

C. General Considerations

Fmoc-5-bromo-L-tryptophan, Fmoc-5-fluoro-L-tryptophan, and Fmoc-5-methyl-L-tryptophan were synthesized by kinetic enzymatic resolution of their racemates according to published procedures.[7] Triflimide was purchased from Oakwood and handled under a dry atmosphere of argon to prepare stock solutions in $MeNO_2$ (1 mg/mL). Methanesulfonic acid ≥99.5% was purchased from Aldrich.

Nitromethane Purification

Pre-treatment of commercial grade nitromethane with either 3Å molecular sieves (7 days) or activated neutral alumina (Aldrich, 58 Å, activated Brockman I, 150 mesh, 12 hrs) is essential for optimal results in Friedel-Crafts cyclizations. Adding H_2O (up to 1000 ppm) to the resultant dry nitromethane has no deleterious effects. For further discussions see: Rose, T. E. Ph.D. Dissertation [Online], University of California, Los Angeles, 2015. pp. 158-160. http://escholarship.org/uc/item/0mx7x1st (Accessed Oct 2, 2015). UMI: 3706064.

HPLC Analysis and Purification

Purification of acidolysis products was performed on an Agilent 1100/1200 HPLC system equipped with G1361A preparative pumps, a G1314A autosampler, a G1314A VWD, and a G1364B automated fraction collector. Analytical HPLC was performed using an identical system, but with a G1312A binary pump. Mass spectra were recorded using an Agilent 6130 LC/MS system equipped with an ESI source. Stationary phase and gradient profile are noted for individual reactions below.

NMR Methods

NMR spectra were recorded on Bruker Advance (500 or 600 MHz) or DRX (500 MHz) spectrometers. 2D NMR data were acquired as previously detailed.[8]

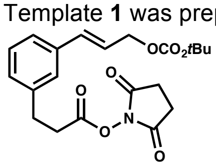
D. Experimental Procedures

Peptide Synthesis

All peptides were synthesized via standard Fmoc solid phase peptide synthesis conditions using Rink Amide MBHA resin (polystyrene, 1% DVB, 0.7 mmol/g).[9]

Linear Precursors Synthesis

Template **1** was prepared as described.[10]



1

General procedure A – Acylation of peptide by template 1: A round bottom flask was charged with peptide (1.1 equiv.), DMF (10 mL), and *i*Pr_2NEt (4.0 – 6.0 eq.), followed by template **1** (1.0 eq.). Reaction progress was monitored by analytical HPLC-UV/MS. Reactions were worked up and purified by column chromatography, trituration, or by preparative HPLC (25%→78% [7 min.] ACN + 0.1% TFA, 18 mL/min, Sunfire C_{18} 19x250 mm) - see details for individual examples below.

General procedure B – Macrocyclization

Using Tf_2NH*:*

A flask was charged with linear precursor (1 eq.) and nitromethane (5 mM in substrate). The heterogeneous mixture was flushed with argon for 10 mins. A stock solution of Tf_2NH in $MeNO_2$ (4.0 – 6.0 eq., 1 mg/mL stock) was then quickly added. The heterogeneous slurry homogenized and became purple in color. The reaction was stirred for 1 minute (2 minutes for **10**). The reaction was quenched with excess *i*Pr_2NEt and concentrated *in vacuo*. The mixture was concentrated, further dried *in vacuo*, diluted with DMSO, and an aliquot was removed and spiked with an equal concentration of internal standard (starting linear precursor). This aliquot was analyzed by HPLC-UV (254 nm) and product peaks were integrated and divided by the internal standard area to provide a yield – uncharacterized products were *not* included towards total yield. Product mixtures were resolved by preparative HPLC purification — see details per example, below.

Using $MeSO_3H$*:*

Reactions were carried out in the same manner as for Tf_2NH but using instead $MeSO_3H$ (75 mM in $MeNO_2$, 5 mM in substrate), and were stirred for 30 mins, then neutralized by the addition of *i*Pr_2NEt.

Isomerization of macrocyclic pyrroloindoline ***18c*:**

Purified **18c** was dissolved in a vigorously stirred solution of 1:4 TFA/CH_3NO_2 at room tempertature. Aliquots were removed, quenched with excess *i*Pr_2NEt, taken to dryness, reconstituted in DMSO (75 µL) and analyzed by HPLC-UV (254 nm). Product yield and isomer distribution were determined by peak integration relative to starting **18c**. The pseudo-first order rate constant was determined by least-squares fitting of the time-course data to the first-order rate law.

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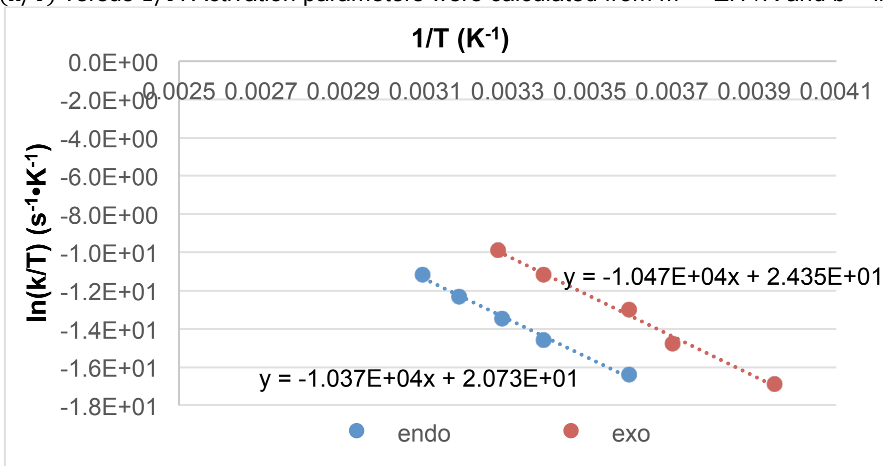
7. Porter, J.; Dykert, J.; Rivier, J. *Int. J. Peptide Protein Res.* **1987**, *30*, 13–21.
8. Rose, T. E.; Lawson, K. V.; Harran, P. G. *Chem. Sci.* **2015**, *6*, 2219–2223
9. Chan, W. C.; White, P. D. *Fmoc Solid Phase Peptide Synthesis: A Practical Approach*, Oxford University Press, Oxford, 2000
10. Lawson, K. V.; Rose, T. E.; Harran, P. G. *Proc. Natl. Acad. Sci. U. S. A.*, **2013**, *110*, E3753.

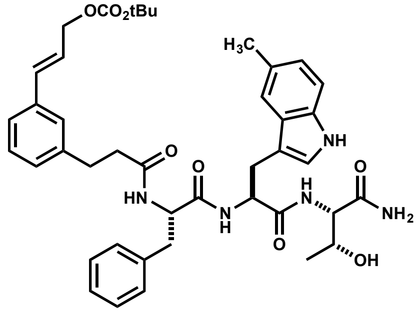
*Determination of kinetic barriers for 1,2-rearrangement of pyrroloindoline endo-**21a** and exo-**21b***

Purified pyrroloindolines **21a** and **21b** were individual subjected to reactions in 20 vol% TFA solution in $MeNO_2$, as for macrocycle **18c**. Reaction temperature was maintained using a heated or cooled water bath, and solutions of substrate in $MeNO_2$ were pre-incubated before adding TFA, which had also been equilibrated to the reaction temperature. Aliquots (25 μL) were removed periodically, chilled briefly in a dry ice acetone bath, and promptly concentrated *in vacuo*. The resulting dried residues were reconstituted in MeOH containing iPr_2EtN (0.1 vol%, 300 μL) and analyzed by HPLC-UV. Pseudo-first order rate constants were determined by least-squares fitting of the time-course peak area data (254 nm) to the first-order rate law, and these data were used to construct the corresponding Eyring plots (below) of $\ln{(k/T)}$ versus $1/T$. Activation parameters were calculated from m = $-\Delta H^{\ddagger}/R$ and b = $\ln(K_B/h) + \Delta S^{\ddagger}/R$.

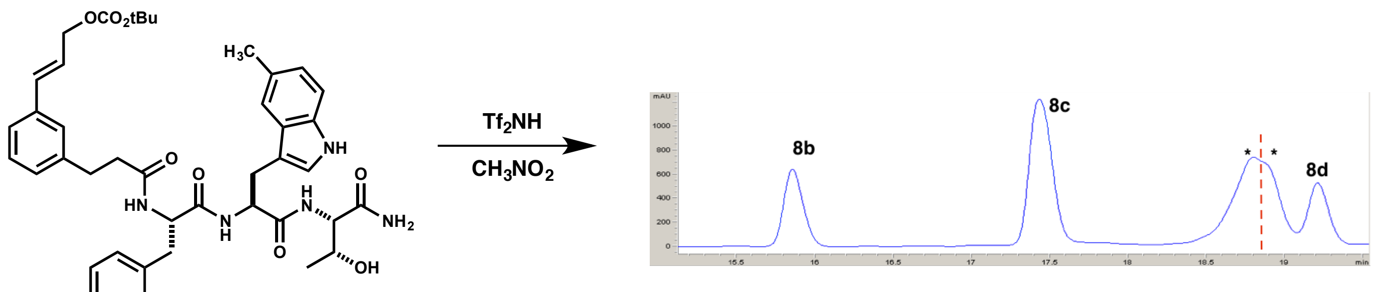


	***exo*-21a**	***endo*-21b**
$\Delta H^{\ddagger}$ (kcal•mol^{-1})	**20.8**	**20.6**
$\Delta S^{\ddagger}$ (kcal•K^{-1}•mol^{-1})	**1.2E-03**	**-6.0E-03**
$\Delta G^{\ddagger}$ (kcal•mol^{-1})	**20.5**	**22.4**

***D*.1.** Acyclic precursors and macrocyclization products

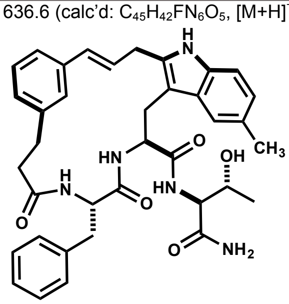


Acyclic Cinnamyl Carbonate 6: Synthesized according to Procedure A. After completion of the reaction, the solution was diluted with 100 mL EtOAc and washed 3x50 mL $NaHCO_3$, 3x50 mL NH_4Cl, 1x50 mL brine. Dried with $MgSO_4$ and concentrated *in vacuo*. Chromatographed on SiO_2 with a gradient from 0% to 5% MeOH in $CHCl_3$. White Solid. 81% yield. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 10.70 (d, *J* = 1.8 Hz, 1H), 8.27 (d, *J* = 7.5 Hz, 1H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.63 (d, *J* = 8.5 Hz, 1H), 7.36 (s, 1H), 7.25 (d, *J* = 7.8 Hz, 1H), 7.21-7.12 (m, 10H), 7.04 (br s, 1H), 6.99 (d, *J* = 7.5 Hz, 1H), 6.88 (dd, *J* = 8.2, 1.1 Hz, 1H), 6.61 (d, *J* = 15.9 Hz, 1H), 6.32 (ddd, *J* = 15.9, 6.3, 6.2 Hz, 1H), 4.90 (d, *J* = 5.4 Hz, 1H), 4.66 (dd, *J* = 6.2, 0.8 Hz, 2H), 4.58 (ddd, *J* = 8.5, 7.5, 4.9 Hz, 1H), 4.52 (ddd, *J* = 10.0, 8.4, 3.9 Hz, 1H), 4.13 (dd, *J* = 8.6, 3.2 Hz, 1H), 4.07-4.04 (m, 1H), 3.18-3.14 (m, 1H), 3.03-2.95 (m, 2H), 2.70 (dd, *J* = 13.9, 10.3 Hz, 1H), 2.62 (apt t, *J* = 7.9 Hz, 2H), 2.38 (s, 3H), 2.45-2.24 (m, 2H), 1.43 (s, 9H), 1.00 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (DMSO-d_6, 126 MHz): δ 172.0, 171.6, 171.4, 152.8, 141.7, 137.9, 135.8, 134.4, 133.4, 129.1, 128.6, 127.9, 127.6, 126.6, 126.4, 126.1, 124.1, 123.7, 123.2, 122.5, 118.0, 111.0, 109.3, 81.5, 66.9, 66.4, 57.9, 53.8, 53.7, 37.5, 36.7, 30.9, 27.4, 27.1, 21.3, 19.9. MS *m/z* 753.4 (calc'd: $C_{42}H_{51}N_5O_8$, $[M+H]^+$, 753.4).



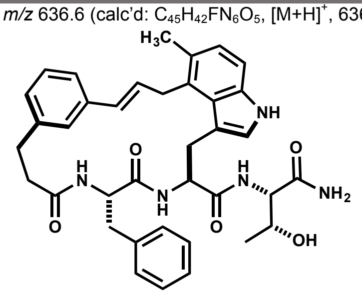
*Unidentified isomeric products

MS *m/z* 636.6 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 636.3).



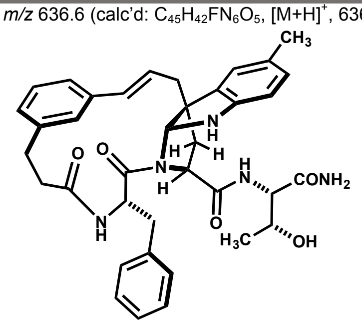
8b

MS *m/z* 636.6 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 636.3).



8c

MS *m/z* 636.6 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 636.3).



8d

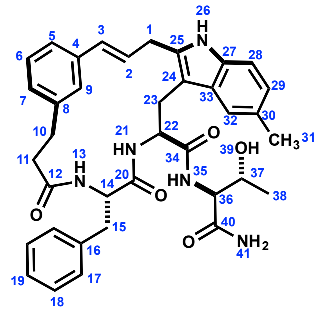
Analytical HPLC Method
Column: Waters Sunfire™ C_{18}, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	40
2.5	30
24	86
29	30

Preparative HPLC Method
Column: Waters Sunfire™ C_{18}, 19x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B ACN + 0.1% TFA
Flow rate: 18.0 mL/min

Time	%B
0	40
2	40
30	50

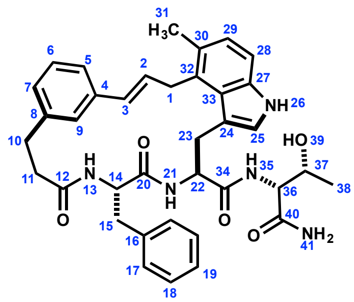
*Macrocyclic Product **8b***



(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	29.8	3.56 (dd, J = 15.6, 6.9 Hz, 1H), 3.78 (dd, J = 15.6, 5.9 Hz, 1H)	HMBC 1->24,25
2	127.8	6.08 (apt dt, J = 15.7, 6.8 Hz, 1H)	COSY 2->1, HMBC 2->4
3	130.3	6.40 (d, J = 15.7 Hz, 1H)	
4	136.6	-	
5	123.9	7.09-7.12 (m, 1H) overlap	
6	127.8	7.14 (dd, J = 7.4, 7.4 Hz, 1H) overlap	
7	127.1	6.95 (br d, J = 7.4 Hz, 1H)	HMBC 7->5
8	141.0	-	
9	124.4	6.98-7.00 (m, 1H) overlap	
10	30.0	2.59-2.65 (m, 1H) overlap, 2.06-2.92 (m, 1H)	HMBC 10->8
11	35.8	2.00 (ddd, J = 14.0, 7.7, 3.1 Hz, 1H), 2.37-2.42 (m, 1H) overlap	
12	171.0	-	
13	-	8.08 (d, J = 8.8 Hz, 1H)	TOCSY 13->14,15, HMBC 13->12
14	52.5	4.79 (ddd, J = 9.4, 9.4, 3.8 Hz, 1H)	
15	38.2	2.61-2.66 (m, 1H) overlap, 2.97-3.02 (m, 1H) overlap	HMBC 15->16
16	137.6	-	
17	129.1	7.17-7.19 (m, 2H) overlap	HMBC 17->19
18	127.4	7.17-7.20 (m, 2H) overlap	HMBC 18->16
19	125.7	7.12-7.15 (m, 1H) overlap	HMBC 19->17
20	172.0	-	
21	-	8.62 (d, J = 7.6 Hz, 1H)	TOCSY 21->22,23, HMBC 21->20
22	54.2	4.67 (ddd, J = 10.6, 7.6, 4.6 Hz, 1H)	
23	26.0	3.02 (dd, J = 14.9, 10.6 Hz, 1H), 3.10 (dd, J = 14.9, 4.6 Hz, 1H) overlap	HMBC 23->24,25
24	105.3	-	
25	133.9	-	
26	-	10.64 (s, 1H)	HMBC 26->24,25,33
27	133.3	-	
28	109.9	7.11 (d, J = 8.3 Hz, 1H) overlap	HMBC 28->30,33
29	121.6	6.83 (dd, J = 8.3, 1.3 Hz, 1H)	HMBC 29->32,31
30	126.3	-	
31	21.1	2.39 (s, 3H)	HMBC 31->29,30,32
32	117.6	7.30 (br s, 1H)	HMBC 32->29,31
33	129.0	-	
34	171.9	-	
35	-	7.66 (d, J = 8.5 Hz, 1H)	HMBC 35->34
36	57.5	4.16 (dd, J = 8.5, 3.1 Hz, 1H)	HMBC 36->40
37	66.0	4.08-4.13 (m, 1H) overlap	
38	19.7	1.08 (d, J = 6.4 Hz, 3H)	HMBC 38->36,37
39	-	not observed	
40	171.8	-	
41	-	6.98-7.00 (m, 1H) overlap, 7.10-7.12 (m, 1H) overlap	HMBC 41->40, TOCSY 41->41'

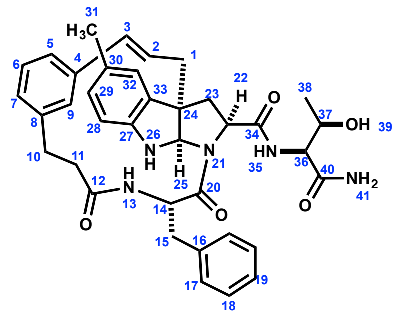
*Macrocyclic Product **8c***



(500 MHz, DMSO-d$_6$, 298K)

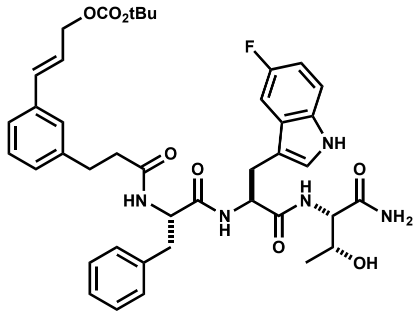
	13C	1H	key correlation
1	32.0	3.82-3.91 (m, 2H)	
2	128.9	6.37 (dt, J = 16.0, 5.4 Hz, 1H)	HMBC 2->4,32, COSY 2->1
3	129.6	6.07 (d, J = 16.0 Hz, 1H)	HMBC 3->5,9
4	136.7	-	
5	123.0	7.02 (d, J = 8.0 Hz, 1H)	
6	127.7	7.09 (dd, J = 8.0, 8.0 Hz, 1H) overlap	HMBC 6->4,8
7	126.8	6.94 (d, J = 8.0 Hz, 1H) overlap	
8	141.1	-	
9	125.3	7.07 (br s, 1H) overlap	
10	29.7	2.54-2.59 (m, 1H) obscured, 2.91-2.97 (m, 1H) overlap	HMBC 10->7,9,12
11	35.9	2.09-2.15 (m, 1H), 2.33-2.39 (m, 1H) overlap	HMBC 11->8,12
12	170.6	-	
13	-	7.94 (d, J = 9.1 Hz, 1H)	HMBC 13->12
14	52.5	4.80-4.88 (m, 1H)	
15	37.9	2.66-2.72 (m, 1H), 2.92-2.97 (m, 1H) overlap	HMBC15->16,17
16	137.7	-	
17	128.8	7.23-7.24 (m, 2H) overlap	HMBC 17->15
18	127.5	7.23-7.25 (m, 2H) overlap	
19	125.6	7.14-7.19 (m, 1H)	
20	171.0	-	
21	-	8.44-8.48 (m, 1H)	HMBC 21->20
22	53.4	4.63-4.70 (m, 1H)	
23	29.5	3.14-3.19 (m, 1H), 3.39 (dd, J = 13.9, 9.9 Hz, 1H)	HMBC 23->24
24	109.5	-	
25	123.3	7.06-7.08 (m, 1H) overlap	HMBC 25->24,27,33
26	-	10.66 (br s, 1H)	COSY 26->25, HMBC 26->24,25,27,33
27	135.4	-	
28	109.2	7.12 (d, J = 8.3 Hz, 1H) overlap	HMBC 28->30,33
29	123.6	6.91 (d, J = 8.3 Hz, 1H)	HMBC 29->27,31,32
30	125.2	-	
31	18.4	2.34 (s, 3H)	HMBC 31->29,30,32
32	128.4	-	
33	126.0	-	
34	170.7	-	
35	-	7.59 (d, J = 8.4 Hz, 1H)	
36	57.6	4.13 (dd, J = 8.4, 3.0 Hz, 1H)	HMBC 36->40
37	65.8	4.04-4.09 (m, 1H)	
38	19.6	1.05 (d, J = 6.2 Hz, 1H)	COSY 38->37, HMBC 38->36,37
39	-	not observed	
40	171.6	-	
41	-	6.87 (br s, 1H), 6.95 (br s, 1H) overlap	

Macrocyclic Product ***8d***

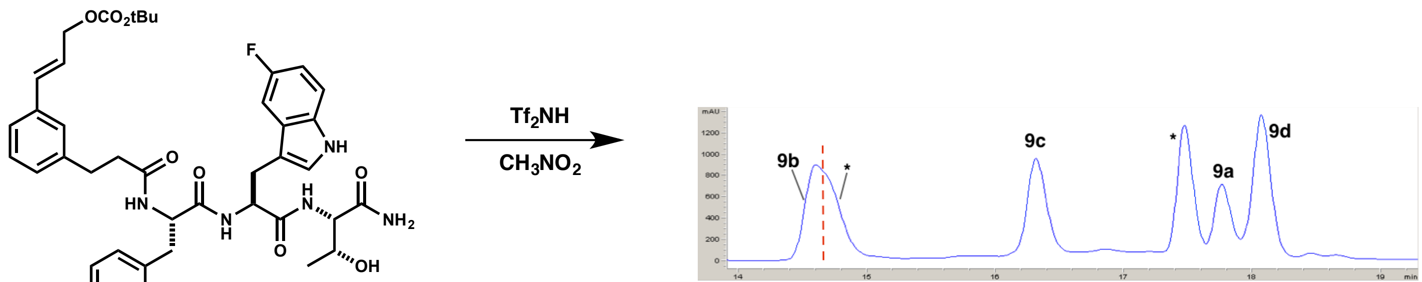


(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	39.3	2.51-2.57 (m, 1H), 2.80 (dd, J = 13.7, 10.0 Hz, 1H)	HMBC 1->24,25
2	126.3	6.05-6.14 (m, 1H)	
3	132.1	6.63 (d, J = 15.8 Hz, 1H)	TOCSY 3->2,1 HMBC 3->4
4	136.9	-	
5	124.3	7.04 (br d, J = 7.6 Hz, 1H)	HMBC 5->3,7
6	128.2	7.15 (dd, J = 7.6, 7.6 Hz, 1H)	HMBC 6->4,8
7	126.8	6.99 (br d, J = 7.6 Hz, 1H)	HMBC 7->5
8	140.5	-	
9	125.0	7.18 (br s, 1H)	
10	30.6	2.62-2.69 (m, 1H), 2.82-2.90 (m, 1H)	
11	37.1	2.01-2.08 (m, 1H), 2.32-3.39 (m, 1H)	HMBC 11->8 TOCSY 11->10,10',11'
12	171.1	-	
13	-	7.96 (d, J = 8.8 Hz, 1H)	HMBC 13->12
14	49.8	5.34 (ddd, J = 8.8, 8.8, 4.8 Hz, 1H)	HMBC 14->20
15	38.3	2.89 (dd, J = 13.9, 8.8 Hz, 1H), 3.09 (dd, J = 13.9, 4.8 Hz, 1H)	HMBC 15->16,17 TOCSY 14->15,13
16	136.5	-	
17	129.8	7.39 (d, J = 7.4 Hz, 2H)	TOCSY 17->18,19
18	127.8	7.28 (dd, J = 7.4, 7.4 Hz, 2H)	HMBC 18->16
19	126.0	7.20-7.24 (m, 1H)	HMBC 19->17
20	171.3	-	
21	-	-	
22	61.6	4.43 (dd, J = 10.4, 5.7 Hz, 1H)	HMBC 22->23,24
23	40.2	2.00-2.07 (m, 1H), 2.50-2.57 (m, 1H)	
24	57.3	-	
25	81.4	6.11 (s, 1H)	HMBC 25->22,24
26	-	not detected	
27	144.9	-	
28	109.9	6.45 (d, J = 7.8 Hz, 1H)	HMBC 28->33
29	128.0	6.84 (dd, J = 7.8, 0.9 Hz, 1H)	HMBC 29->32
30	127.3	-	
31	20.4	2.21 (s, 1H)	HMBC 31->28,30,32
32	122.0	6.91-6.93 (m, 1H)	HMBC 32->27,29
33	135.5	-	
34	170.4	-	
35	-	7.51 (d, J = 7.8 Hz, 1H)	
36	57.2	3.84 (dd, J = 7.8, 2.5 Hz, 1H)	HMBC 36->40
37	65.2	3.91-3.97 (m, 1H)	HMBC 37->40
38	19.3	0.78 (d, J = 6.6 Hz, 3H)	COSY 38->37 TOCSY 38->35,36,37
39	-	not detected	
40	171.5	-	
41	-	6.68 (br s, 1H), 7.20 (br s, 1H)	HMBC 41->40 TOCSY 41->41'

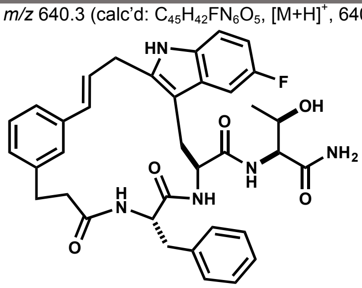


Acyclic Cinnamyl Carbonate 7: Synthesized according to Procedure A. Workup and chromatography conditions were the same as for linear precursor **6**. White Solid. 62% yield. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 10.97 (d, J = 2.2 Hz, 1H), 8.29 (d, J = 7.8 Hz, 1H), 8.08 (d, J = 8.1 Hz, 1H), 7.8 (d, J = 8.6 Hz, 1H), 7.41 (dd, J = 10.2, 2.4 Hz, 1H), 7.32 (dd, J = 8.8, 4.5 Hz, 1H), 7.29 (d, J = 2.2 Hz, 1H), 7.26 (br. d, J = 7.8 Hz, 1H), 7.23 (br. s, 1H), 7.21 (br. s, 1H), 7.2 (br. s, 1H), 7.14-7.18 (m, 4H), 7.09 (br. s, 1H), 7.01 (d, J = 8.1 Hz, 1H), 6.9 (ddd, J = 9.0, 9.0, 2.3 Hz, 1H), 6.63 (d, J = 15.7 Hz, 1H), 6.33 (dt, J = 15.9, 6.2 Hz, 1H), 4.67 (dd, J = 6.3, 1.1 Hz, 2H), 4.63 (ddd, J = 8.6, 8.0, 4.9 Hz, 1H), 4.54 (ddd, J = 9.9, 8.4, 4.0 Hz, 1H), 4.16 (dd, J = 8.7, 3.2 Hz, 1H), 4.08 (dddd, J = 6.2, 6.2, 6.2, 3.4 Hz, 1H), 3.17 (dd, J = 15.0, 4.6 Hz, 1H), 3.02 (dd, J = 15.3, 9.3 Hz, 1H), 2.97 (dd, J = 13.7, 4.0 Hz, 1H), 2.71 (dd, J = 13.9, 16.0 Hz, 1H), 2.65 (app t, J = 7.9 Hz, 2H), 2.23-2.38 (m, 2H), 1.4 (s, 9H), 1.02 (d, J = 6.4 Hz, 3H). ^{13}C NMR (DMSO-d_6, 126 MHz): δ 172.0, 171.5, 171.4, 171.3, 157.6, 155.8, 152.8, 141.7, 137.9, 135.8, 133.4, 132.7, 129.1, 128.6, 127.9, 127.6, 127.5, 126.4, 126.1, 125.9, 124.1, 123.2, 112.14, 112.06, 110.2, 110.2, 109.0, 108.8, 103.3, 103.1, 81.5, 66.9, 66.3, 58.0, 53.7, 53.5, 37.4, 36.7, 30.9, 27.3, 19.9. MS m/z 758.8 (calc'd: $C_{41}H_{48}FN_5O_8$, $[M+H]^+$, 758.4).



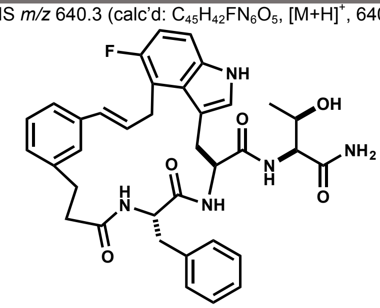
*Unidentified isomeric products

MS m/z 640.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 640.3).



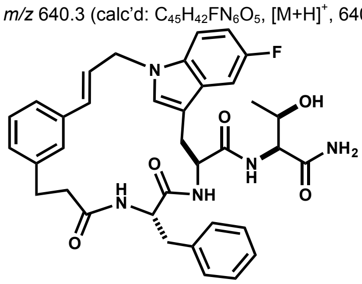
9b

MS m/z 640.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 640.3).



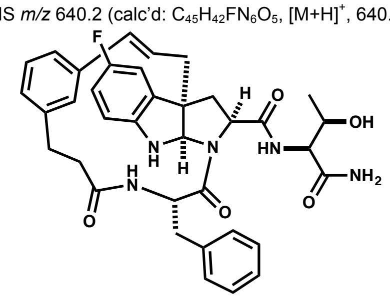
9c

MS m/z 640.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 640.3).



9a

MS m/z 640.2 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 640.3).



9d

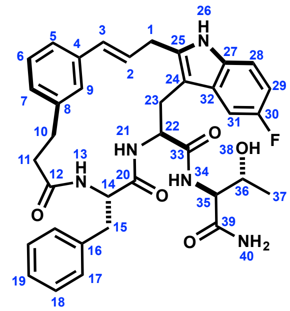
Analytical HPLC Method
Column: Waters Sunfire™ C_{18}, 4.6x250 mm, 5 µm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	30
2.5	30
24	86
29	30

Preparative HPLC Method
Column: Waters Sunfire™ C_{18}, 19x250 mm, 5 µm
Solvent A: H_2O + 0.1% TFA
Solvent B ACN + 0.1% TFA
Flow rate: 18.0 mL/min

Time	%B
0	40
2	40
30	60

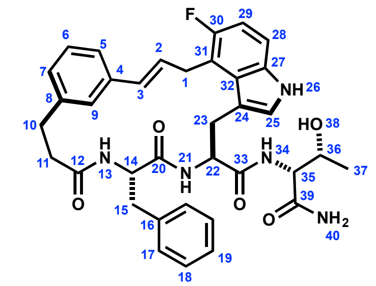
Macrocyclic Product ***9b***



(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	46.6	4.95 (ddd, J = 16.4, 4.7, 1.6 Hz, 1H), 4.83 (dd, J = 16.4,6.9 Hz, 1H)	TOCSY1->2,3 HMBC 1->2,3,25
2	125.5	6.07 (ddd, J = 15.8, 7.1, 4.5 Hz, 1H)	HMBC 2->1,3,4
3	131.0	6.22 (br d, J = 15.9 Hz, 1H)	
4	135.7	-	
5	124.7	7.11 (m, 1H) overlap	HMBC 5->9,7
6	127.8	7.15 (m, 1H) overlap	HMBC 6->4,8
7	128.2	6.96 (m, 1H) overlap	
8	142.0	-	
9	124.5	6.92 (m, 1H) overlap	HMBC 9->7
10	29.1	2.46 (m, 1H) overlap, 2.95 (m, 1H) overlap	HMBC 10->7,8,9,11,12
11	35.5	2.08 (ddd, J = 15.1, 7.2, 2.4 Hz, 1H), 2.40 (ddd, J = 15.2, 11.6, 2.3 Hz, 1H)	TOCSY 11->10
12	170.6	-	
13	-	7.68 (d, J = 8.5 Hz, 1H)	HMBC 13->12 TOCSY 13->14,15
14	52.6	4.71 (m, 1H) overlap	
15	38.9	2.70 (dd, J = 13.6, 8.0 Hz, 1H), 3.02 (dd, J = 13.6, 4.1 Hz, 1H)	HMBC 15->14,16,17,20
16	137.6	-	
17	129.4	7.08 (m, 1H) overlap	HMBC 17->15
18	127.8	7.15 (m, 1H) overlap	HMBC 18->16
19	126.4	7.11 (m, 1H) overlap	HMBC 19->17
20	170.6	-	
21	-	8.60 (d, J = 8.7 Hz, 1H)	TOCSY 21->22,23 HMBC 21->20
22	52.6	4.74 (m, 1H) overlap	HMBC 22->23
23	27.2	3.10 (br. d, J = 14.8 Hz, 1H), 2.90 (m, 1H) overlap	HMBC 23->22,24,25
24	110.8	-	
25	128.1	7.28 (s, 1H)	HMBC 25->1,32,38
26	-	-	
27	132.6	-	
28	110.9	7.45 (dd, J = 7.8, 4.5 Hz, 1H)	HMBC 28->32 TOCSY 28->29,31
29	109.2	6.94 (m, 1H) overlap	HMBC 29->27,30
30	157.0	-	
31	103.8	7.51 (dd J = 9.9, 2.4 Hz, 1H)	HMBC 31->27,30
32	127.8	-	
33	171.9	-	
34	-	7.96 (d, J = 8.8 Hz, 1H)	TOCSY 34->35,36 HMBC 34->33
35	58.0	4.21 (dd, J = 8.8, 3.1 Hz, 1H)	HMBC 35->36
36	66.4	4.10 (m, 1H)	
37	20.0	1.07 (d, J = 6.4 Hz, 3H)	HMBC 37->35,36
38	-	not observed	
39	172.2	-	
40	-	7.2 (br. s, 2H)	HMBC 40->39

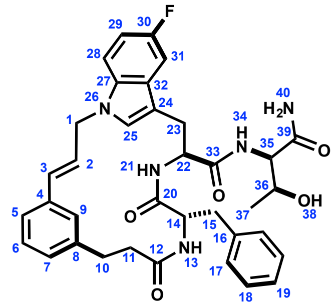
*Macrocyclic Product **9c***



(500 MHz, DMSO-d_6, 298K)

	13C	1H	key correlation
1	30.0	3.57 (dd, J = 15.3, 7.2 Hz, 1H), 3.80 (dd, J =15.3, 6.3 Hz, 1H)	TOCSY 1->2,3 HMBC 1->2,3,24,25
2	127.6	6.07 (dt, J = 15.6, 6.9 Hz, 1H)	HMBC 2->1,4
3	131.0	6.43 (d, J = 15.9 Hz, 1H)	HMBC 3->1,4,5,9
4	136.8	-	
5	123.6	7.10 (m, 1H) overlap	HMBC 5->9
6	127.9	7.13 (m, 1H) overlap	HMBC 6->4
7	124.0	7.10 (m, 1H) overlap	HMBC 7->5,9
8	141.2	-	
9	125.0	6.98 (br. s, 1)	HMBC 10->8,9 TOCSY 10->11
10	30.4	2.62 (m, 1H) overlap, 2.88 (ddd, J = 13.7, 11.0, 5.6 Hz, 1H)	HMBC 11->8,12
11	35.8	1.98 (ddd, J = 14.0, 7.6, 3.1 Hz, 1H), 2.40 (ddd, J = 13.6, 11.0, 2.6 Hz, 1H)	
12	171.2	-	
13	-	8.07 (d, J - 8.9 Hz, 1H)	COSY 13->14 TOCSY 13->14,15,15' HMBC 13->12
14	52.7	4.75 (ddd, J = 9.3, 9.5, 3.8 Hz, 1H)	HMBC 14->15
15	38.0	2.98 (m, 1H) overlap , 2.65 (m, 1H) overlap	HMBC 15->14,16,17
16	138.1	-	
17	129.5	7.16 (m, 1H) overlap	HMBC 17->18
18	127.7	7.17 (m, 1H) overlap	HMBC 18->16,17
19	128.0	7.14 (m, 1H) overlap	
20	172.1	-	
21	-	8.59 (d, J = 7.8 Hz, 1H)	COSY 21->22 TOCSY 21->22,23 HMBC 21->20
22	54.1	4.66 (ddd, J = 10.2, 7.6, 5.1 Hz, 1H)	HMBC 22->23
23	26.2	3.07 (dd, J - 15.1, 5.2 Hz, 1H), 2.97 (m, 1H) overlap	HMBC 23->22,24,32
24	106.4	-	
25	136.4	-	
26	-	10.91 (s, 1H)	HMBC 26->24,25,27,32
27	131.8	-	
28	111.2	7.18 (m, 1H) overlap	HMBC 28->30 TOCSY 28->29
29	108.4	6.81 (ddd, J = 9.3, 9.3, 2.5 Hz, 1H)	TOCSY 29->28,31 HMBC 29->27,30
30	156.6	-	
31	103.4	7.24 (dd, J = 10.3, 2.7 Hz, 1H)	HMBC 31->27,30
32	129.0	-	
33	172.1	-	
34	-	7.73 (d, J = 8.7 Hz, 1H)	HMBC 34->33 TOCSY 34->35,36,37
35	57.8	4.13 (dd, J = 8.7, 2.9 Hz, 1H)	HMBC 35->36,39
36	66.2	4.07 (m, 1H)	
37	19.5	1.05 (d, J = 6.4 Hz, 3H)	
38	-	4.96 (d, J = 4.9 Hz, 1H)	
39	172.0	-	
40	-	6.93 (m, 1H) overlap	HMBC 40->39

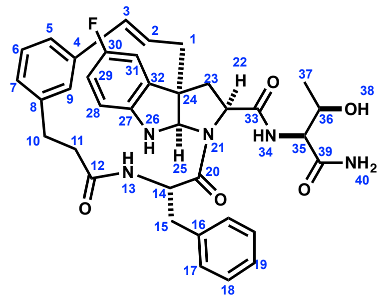
*Macrocyclic Product **9a***



(500 MHz, DMSO-d$_6$, 298K)

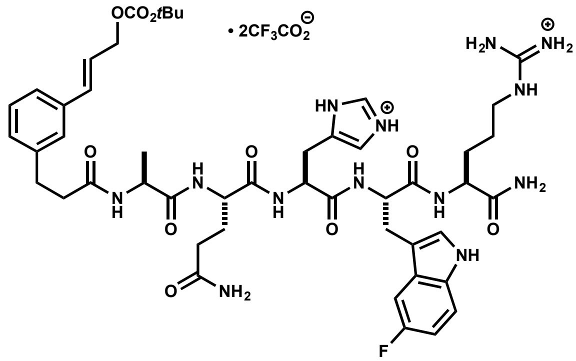
	13C	1H	key correlation
1	27.8	3.76 (dd, J = 16.6, 5.9 Hz, 1H), 3.90 (br. d, J = 16.0 Hz, 1H)	TOCSY1->2,3 HMBC 1->2,3,31
2	130.0	6.07 (d, J = 15.8 Hz, 1H)	HMBC 2->1,31
3	128.8	6.35 (dt, J = 16.0, 5.5 Hz, 1H)	HMBC 3->1,5,9,30,32
4	136.5	-	
5	122.9	7.00 (m, 1H) overlap	HMBC 5->9
6	127.8	7.06 (m, 1H) overlap	HMBC 6->4,8
7	127.1	7.06 (m, 1H) overlap	HMBC 7->9
8	141.3	-	
9	125.8	6.99 (br. s, 1H)	HMBC 9->5,7
10	29.6	2.91 (m, 1H) overlap, 2.52 (m, 1H) overlap	HMBC 10->7,8,9,11,12
11	35.7	2.32 (app t, 13.5 Hz, 1H), 2.08 (m, 1H) overlap	HMBC 11->8,12
12	170.7	-	
13	-	8.1 0 (d, J = 8.9 Hz, 1H)	HMBC 13->12 TOCSY 13->14,15,15'
14	52.6	4.82 (ddd, J = 9.7, 9.7, 4.1 Hz, 1H)	HMBC 14->15
15	38.0	2.89 (m, 1H) overlap, 2.64 (dd, J = 13.3, 10.5 Hz, 1H)	HMBC 15->14,16,17
16	137.7	-	
17	128.8	7.21 (m, 1H) overlap	
18	127.6	7.21 (m, 1H) overlap	
19	127.6	7.15 (m, 1H) overlap	HMBC 19->17
20	171.1	-	
21	-	8.62 (d, J = 6.6 Hz, 1H)	HMBC 21->20 TOCSY 21->22,23
22	53.2	4.66 (m, 1H)	
23	29.1	3.30 (m, 1H) overlap, 3.06 (br. d, J = 13.4 Hz, 1H)	HMBC 23->22,24
24	110.6	-	HMBC 25->27
25	125.8	7.13 (m, 1H) overlap	HMBC 26->24,25,27
26	-	10.96 (br. s, 1H)	
27	133.0	-	
28	110.0	7.18 (m, 1H) overlap	
29	108.8	6.89 (m, 1H) overlap	HMBC 29->27,30,31
30	154.5	-	
31	115.6	-	
32	125.8	-	
33	170.7	-	
34	-	7.74 (d, J = 8.3 Hz, 1H)	HMBC 34->33 TOCSY 34->35,36,37
35	57.8	4.08 (dd, J = 8.7, 3.0 Hz, 1H)	
36	65.8	4.02 (m, 1H)	
37	19.8	0.99 (d, J = 6.4 Hz, 3H)	HMBC 37->35,36
38	-	4.9 (d, J = 5.1 Hz, 1H)	HMBC 38->35,36,37
39	171.7	-	
40	-	7.03 (m, 1H) overlap	HMBC 40->39

*Macrocyclic Product **9d***

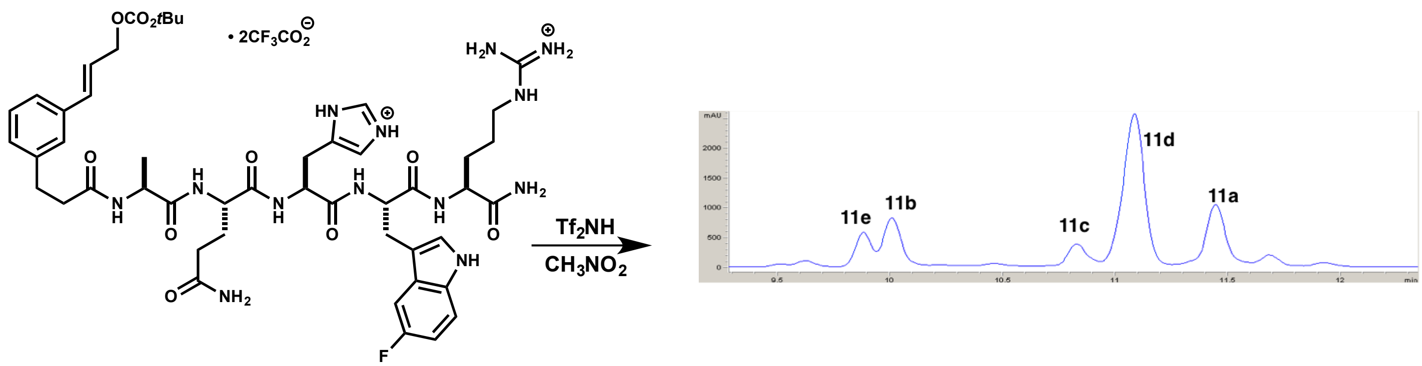


(500 MHz, DMSO-d_6, 298K)

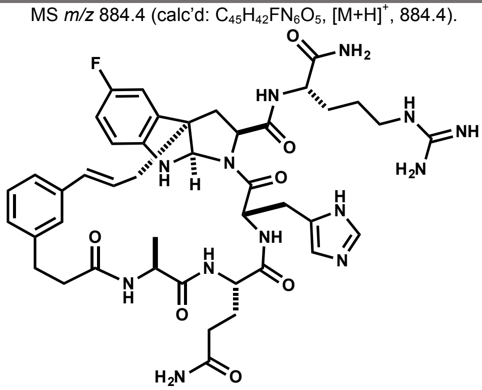
	13C	1H	key correlation
1	39.0	2.51-2.57 (m, 1H), 2.79-2.85 (m, 1H)	HMBC 1->24
2	125.0	6.09 (ddd, J = 15.8, 9.7, 6.2 Hz, 1H)	TOCSY 2->3,1
3	132.6	6.62 (d, J = 15.8 Hz, 1H)	HMBC 3->4
4	136.9	-	
5	124.2	7.05 (br d, J = 8.5 Hz, 1H)	HMBC 5->9,7
6	127.9	7.15 (dd, J = 8.5, 7.4 Hz, 1H)	HMBC 6->4,8
7	127.0	6.99 (br d, J = 7.4 Hz, 1H)	
8	140.5	-	
9	124.9	7.18 (br s, 1H)	HMBC 9->3
10	30.9	2.62-2.67 (m, 1H), 2.85-2.90 (m, 1H)	
11	37.4	2.05 (ddd, J = 13.5, 6.9, 3.8 Hz, 1H), 2.35 (ddd, J = 13.5, 10.8, 3.1 Hz, 1H)	
12	171.4	-	
13	-	7.96 (d, J = 8.8 Hz, 1H)	HMBC 13->12
14	50.1	5.33 (ddd, J = 8.9, 8.8, 4.8 Hz, 1H)	HMBC 14->20
15	38.6	2.82-2.87 (m, 1H), 3.08 (dd, J = 14.0, 4.8 Hz, 1H)	HMBC 15->16,17 TOCSY 15->14,13
16	136.7	-	
17	129.9	7.38 (d, J = 7.4 Hz, 2H)	HMBC 17->19
18	127.8	7.27 (dd, J = 7.4, 7.4 Hz, 2H)	HMBC 18->16
19	126.1	7.20-7.23 (m, 1H)	HMBC 18->17
20	171.2	-	
21	-	-	
22	61.5	4.48 (dd, J =10.3, 5.5 Hz, 1H)	HMBC 22->24 COSY 22->23
23	40.0	2.10 (dd, J = 13.6, 5.5 Hz, 1H), 2.51-2.55 (m, 1H)	HMBC 23->24
24	57.6	-	
25	81.6	6.16 (br s, 1H)	COSY 25->26 HMBC 25->27
26	-	6.32 (br s, 1H)	
27	143.8	-	
28	110.5	6.51 (dd, JHH = 8.6 Hz, JHF = 4.6 Hz, 1H)	HMBC 28->30,32
29	113.7	6.84 (ddd, JHF = 9.0 Hz, JHH = 8.6, 2.7 Hz, 1H)	HMBC 29->27,30
30	156.8 (d, J≈240 Hz)	-	
31	109.3	7.02 (dd, JHF = 8.4 Hz, JHH = 2.7 Hz, 1H)	HMBC 31->27,30
32	136.9	-	
33	170.4	-	
34	-	7.49 (d, J = 8.0 Hz, 1H)	HMBC 34->33
35	57.5	3.86 (ddd, J = 8.0, 2.6 Hz, 1H)	HMBC 35->39
36	65.6	3.90-3.96 (m, 1H)	
37	19.4	0.77 (d, J = 6.6 Hz, 3H)	COSY 37->36 TOCSY 37->36,35,34
38	-	not detected	
39	171.5	-	
40	-	6.73 (br s, 1H), 7.19 (br s, 1H)	TOCSY 40->40'



Acyclic Cinnamyl Carbonate 10: Synthesized according to Procedure B. White Powder. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 10.98 (d, J = 2.3 Hz, 1H), 8.96 (d, J = 1.2 Hz, 1H), 8.24 (d, J = 7.9 Hz, 1H), 8.06-8.16 (m, 4H), 7.74 (t, J = 5.7 Hz, 1H), 7.39 (dd, J = 16.0, 2.5 Hz, 1H), 7.36 (br. s, 1H), 7.23-7.34 (m, 7H), 7.09-7.14 (m, 2H), 6.87-6.93 (m, 2H), 6.65 (d, J = 15.9 Hz, 1H), 6.35 (dt, J = 16.0, 6.2 Hz, 1H), 4.68 (dd, J = 6.5, 1.0 Hz, 2H), 4.52-4.60 (m, 2H), 4.18-4.28 (m, 2H), 4.16 (ddd, J = 7.9, 5.6 Hz, 1H), 3.03-3.17 (m, 4H), 2.94 (dd, J = 15.9, 15.9, 9 Hz, 2H), 2.75-2.84 (m, 2H), 2.39-4.29 (m, 2H), 2.04-2.17 (m, 2H), 1.80-1.91 (m, 1H), 1.65-1.79 (m, 2H), 1.45-1.55 (m, 2H), 1.43 (s, 9H), 1.15 (d, J = 7 Hz, 3H). ^{13}C NMR (DMSO-d_6, 126 MHz): δ174.1, 173.1, 172.7, 171.6, 171.4, 171.3, 169.8, 157.6, 156.8, 155.8, 152.8, 141.7, 135.8, 133.7, 133.4, 132.7, 129.3, 129.1, 128.6, 128.0, 127.5, 127.4, 126.4, 125.9, 124.2, 123.3, 117.7, 116.8, 115.3, 115.2, 112.2, 112.1, 109.9, 109.9, 81.5, 66.9, 55.0, 53.4, 52.4, 52.2, 51.5, 48.3, 36.6, 31.3, 30.8, 29.1, 27.3, 25.0, 17.9. MS m/z 1002.7 (calc'd: $C_{48}H_{64}N_{13}O_{10}$, $[M+H]^+$, 1002.5).

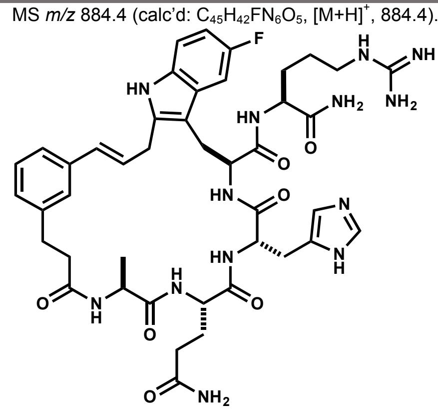


MS m/z 884.4 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 884.4).



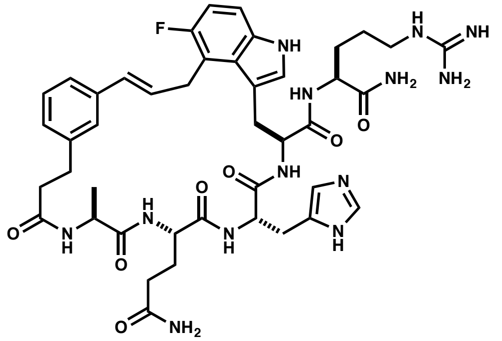
11e

MS m/z 884.4 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 884.4).



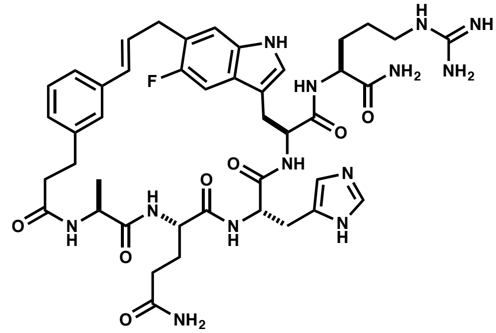
11b

MS m/z 884.4 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 884.4).



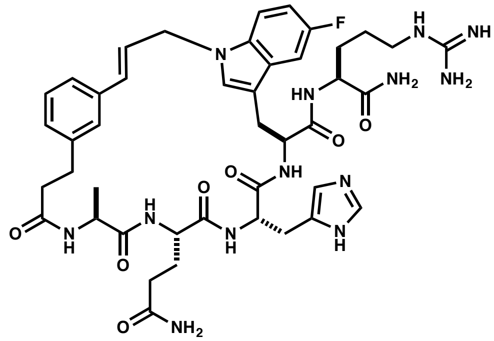
11c

MS m/z 884.4 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 884.4).



11d

MS m/z 884.4 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 884.4).



11a

Analytical HPLC Method
Column: Waters Sunfire™ C_{18}, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	10
0.5	10
2	25
17	64

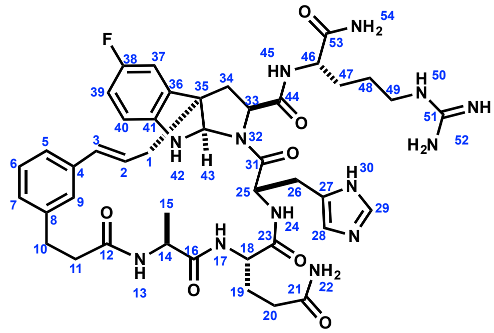
Preparative HPLC method A:
Column: Waters XBridge™ C_{18}, 19x250mm, 5μm.
Solvent A: H_2O + 0.1%v TFA
Solvent B: ACN + 0.1%v TFA
Flow rate: 18.00 ml/min

Time	%B
0	30
2	30
30	100

Preparative HPLC method B:
Same as A
Repurification of **11a, 11c, & 11d**

Time	%B
0	30
2	30
30	55

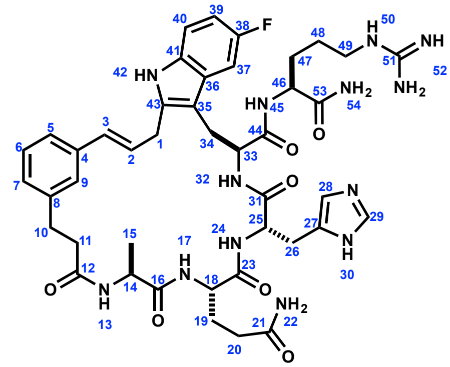
*Macrocyclic Product **11e***



(500 MHz, DMSO-d_6, 298K)

	13C	1H	key correlations
1	47.1	4.83 (dd, J 15.5, 6.4 Hz, 1H), 4.99 (dd, J = 15.5, 5.7 Hz, 1H)	HMBC 1->41,43
2	125.4	6.37 (ddd, J = 15.7, 6.4, 5.7 Hz, 1H)	HMBC 2->4
3	131.9	6.65 (br d, J = 15.7 Hz, 1H)	TOCSY 3->2,1
4	135.9	-	
5	125.1	7.15-7.20 (m, 1H) overlap	
6	128.5	7.19-7.24 (m, 1H) overlap	HMBC 6->4,8
7	127.8	7.09 (br d, J = 7.1 Hz, 1H)	
8	141.6	-	
9	125.6	7.25 (br s, 1H) overlap	
10	30.7	2.73-2.86 (m, 2H)	HMBC 10->8,12
11	36.3	2.36-2.51 (m, 2H)	HMBC 11->8,12
12	172.6	-	
13	-	8.24 (d, J = 6.4 Hz, 1H)	HMBC 13->12
14	49.6	7.94-7.98 (m, 1H)	HMBC 14->16
15	17.3	1.14 (d, J = 7.2 Hz, 3H)	TOCSY 15->14,13
16	173.2	-	
17	-	7.81-7.88 (m, 1H)	HMBC 17->16
18	49.6	4.08-4.16 (m, 1H)	
19	27.4	1.65-1.74 (m, 1H), 179-1.87 (m, 1H)	HMBC 19->21
20	31	1.95-2.11 (m, 2H)	HMBC 20->21
21	174.1	-	
22	-	6.81 (br s, 1H), 7.30 (br s, 1H)	HMBC 22->21
23	171.9	-	
24	-	8.12 (d, J = 7.6 Hz, 1H)	HMBC 24->23
25	51.4	4.55-4.62 (m, 1H) overlap	HMBC 25->31
26	26.8	2.92-3.01 (m, 1H) overlap, 3.06-3.12 (m, 1H)	
27	129.6	-	
28	116.9	7.25 (s, 1H) overlap	HMBC 28->29
29	134.1	8.95 (br s, 1H)	HMBC 29->27,28
30	-	Not detected	
31	170.7	-	
32	-	8.02 (d, J = 7.2 Hz, 1H)	HMBC 32->31
33	53.3	4.52-4.59 (m, 1H)	HMBC 33->34
34	27.3	2.92-3.01 (m, 1H) overlap, 3.12-3.22 (m, 1H)	HMBC 34->33,35
35	109.9	-	
36	128	-	
37	103.9	7.45 (dd, JHF = 9.9 Hz, JHH = 2.3 Hz, 1H)	HMBC 37->41
38	157.1 (d, J ≅ 220Hz)	-	
39	109.4	6.98 (ddd, JHF = 9.1 Hz, JHH = 9.1, 2.3 Hz, 1H)	HMBC 39->41
40	111.1	7.48 (dd, JHH = 9.1 Hz, JHF = 4.5 Hz, 1H)	HMBC 40->36
41	132.8	-	
42	-	-	
43	128.5	7.28 (s, 1H)	HMBC 43->1
44	171.9	-	
45	-	8.22 (d, J = 8.1 Hz, 1H)	
46	52.2	4.19-4.28 (m, 1H)	HMBC 46->53
47	29.1	1.54-1.64 m, 1H), 1.69-1.79 (m, 1H)	
48	25	1.43-1.57 (m, 2H)	
49	40.5	3.08-3.15 (m, 2H) overlap	HMBC 49->51
50	-	7.61 (t, J = 5.1 Hz, 1H)	
51	156.9	-	
52	-	14.03-14.44 (m, 3H)	
53	173.4	-	
54	-	7.15 (br s, 1H), 7.31 (br s, 1H)	HMBC 54->53

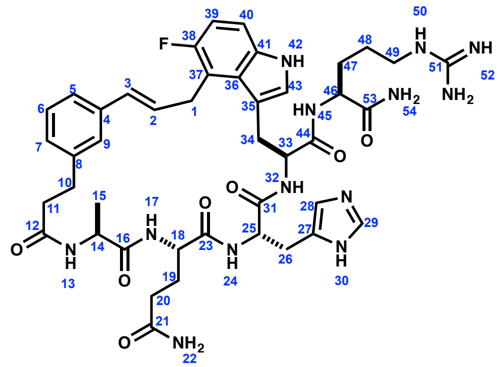
*Macrocyclic Product **11b***



(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlations
1	32.4	3.52 (dd, J = 15.3, 5.6 Hz, 1H), 3.64 (dd, J = 15.3, 6.3 Hz, 1H)	HMBC 1->38,39,40
2	129.3	6.39 (ddd, J = 15.8, 6.3, 5.6 Hz, 1H)	TOCSY 2->1,2 HMBC 2->4
3	129.9	6.30 (br d, J = 15.8 Hz, 1H)	HMBC 3->4
4	137	-	
5	124.1	7.13-7.17 (m, 1H) overlap	HMBC 5->7,9
6	128.3	7.15-7.19 (m, 1H) overlap	HMBC 6->4,8
7	127.3	7.10 (br d, J = 7.2 Hz, 1H)	HMBC 7->9
8	141.1	-	
9	124.5	7.10 (br s, 1H) overlap	HMBC 9->7
10	30.2	2.69-2.76 (m, 1H), 2.77-2.82 (m, 1H) overlap	HMBC 10->7,8,9,12
11	35.4	2.31 (ddd, J = 14.3, 6.5, 6.5 Hz, 1H), 2.44-2.51 (m, 1H) overlap	HMBC 11->8,12
12	171.4	-	
13	-	8.07 (d, J = 7.6 Hz, 1H)	HMBC 13->12
14	47.7	4.12 (qd, J = 7.6, 7.1 Hz, 1H)	HMBC 14->15,16
15	17.6	0.85 (d, J = 7.1 Hz, 3H)	HMBC 15->15,16
16	172.3	-	
17	-	7.86 (d, J = 7.7 Hz, 1H)	HMBC 17->16
18	52.3	3.98 (ddd, J = 8.0, 7.8, 5.4 Hz, 1H)	TOCSY 18->17,18,20 HMBC 18->19,20,23
19	27.3	1.50-1.58 (m, 1H) overlap, 1.70-1.81 (m, 1H) overlap	HMBC 19->20,21
20	30.9	1.91-2.00 (m, 2H)	HMBC 20->21
21	173.9	-	
22	-	6.78 (br s, 1H), 7.22 (br s, 1H) overlap	TOCSY 22->22'
23	171.2	-	
24	-	7.56 (br d, J = 6.8 Hz, 1H)	HMBC 24->23
25	50.7	4.45 (ddd, J = 7.2, 6.8, 5.8 Hz, 1H)	COSY 25->24
26	27.6	2.93 (dd, J = 15.3, 7.6 Hz, 1H), 3.05-3.12 (m, 1H) overlap	HMBC 26->27
27	128.9	-	
28	116.8	7.28 (s, 1H)	HMBC 28->27,30
29	-	not observed	
30	134	8.95 (br s, 1H)	HMBC 30->27,28
31	170	-	
32	-	8.07 (d, J = 7.4 Hz, 1H)	HMBC 32->31
33	53.7	4.60 (ddd, J = 10.9, 7.4, 3.3 Hz, 1H)	TOCSY 33->32,34
34	27.7	2.85 (dd, J = 14.4, 11.1 Hz, 1H), 3.13-3.20 (m, 1H) overlap	HMBC 34->33,35
35	109.8	-	
36	133	-	
37	103.8	7.57 (d, JHF = 11.1 Hz, 1H)	HMBC 37->35,41,38,39
38	155.3 (d, J ≅ 230Hz)	-	
39	120.1	-	
40	112.8	7.21 (d, JHF = 6.4 Hz, 1H)	HMBC 40->38
41	133	-	
42	-	10.83 (d, J = 1.3 Hz, 1H)	HMBC 42->35,36,41
43	125.7	7.17-7.19 (m, 1H) overlap	
44	172.2	-	
45	-	8.70 (br d, J = 7.8 Hz, 1H)	HMBC 45->44
46	52.2	4.25 (ddd, J = 7.8, 7.8, 6.1 Hz, 1H)	TOCSY 46->45,47,48,49
47	28.9	1.55-1.63 (m, 1H) overlap, 1.71-1.78 (m, 1H) overlap	HMBC 47->46
48	25.1	1.47-1.57 (m, 2H) overlap	HMBC 48->49
49	40.3	3.09-3.16 (m, 2H) overlap	HMBC 49->47,48,51
50	-	14.17 (br s) overlap	
51	156.9	-	
52	-	14.17 (br s) overlap	
53	173.3	-	
54	-	7.14 (br s, 1H), 7.39 (br s, 1H)	TOCSY 54'->54

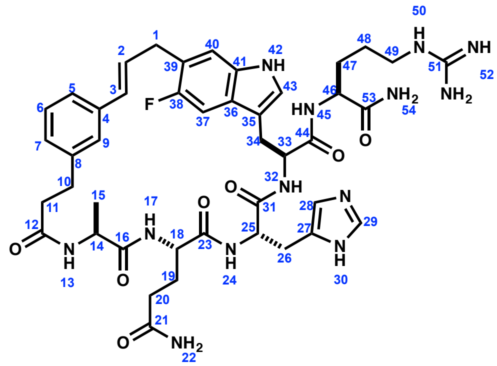
*Macrocyclic Product **11c***



(500 MHz, DMSO-d_6, 298K)

	13C	1H	key correlations
1	27.3	3.80-3.91 (m, 2H)	HMBC 1->2,3,37
2	129.2	6.44 (dt, J = 15.8, 6.0 Hz, 1H)	HMBC 2->4,37
3	129.9	6.20 (br d, J = 15.8 Hz, 1H)	HMBC 3->5,9 TOCSY 3->2,1
4	136.9	-	
5	124.4	6.99-7.02 (m, 1H) overlap	
6	128.1	7.13 (dd J = 7.6, 7.6 Hz, 1H)	HMBC 6->4,8
7	127.5	6.99-7.02 (m, 1H) overlap	
8	141.4	-	
9	125.7	7.35 (br s, 1H)	HMBC 9->3,5,7
10	30.4	2.76-2.87 (m, 2H)	HMBC 10->8,12
11	35.9	2.44 (ddd, J = 14.6, 5.7, 5.7 Hz, 1H), 2.56 (ddd, J = 14.6, 9.3, 5.8 Hz, 1H)	HMBC 11->8,12
12	172.6	-	
13	-	8.11 (d, J = 6.0 Hz, 1H)	HMBC 13->12
14	49.3	3.97-4.03 (m, 1H)	HMBC 14->16
15	17.3	1.09 (d, J = 7.2 Hz, 3H)	HMBC 15->14 TOCSY 15->14,13
16	173.2	-	
17	-	8.01 (d, J = 7.5 Hz, 1H)	
18	53	4.00-4.06 (m, 1H) overlap	HMBC 18->23
19	26.8	1.62-1.70 (m, 1H) overlap, 1.73-1.82 (m, 1H) overlap	HMBC 19->21,23
20	30.9	1.95-2.03 (m, 1H), 2.04-2.11 (m, 1H)	HMBC 20->21
21	174.1	-	
22	-	6.87(br s, 1H), 7.30 (br s, 1H) overlap	
23	171.9	-	
24	-	8.27 (d, J = 7.9 Hz, 1H)	HMBC 24->23
25	51.5	4.60 (ddd, J = 9.3, 7.9, 5.1 Hz, 1H)	HMBC 25->31
26	26.2	3.03-3.09 (m, 1H) overlap, 3.22-3.27 (m, 1H)	HMBC 26->27,31
27	129.7	-	
28	116.3	7.30 (s, 1H) overlap	HMBC 28->29 TOCSY 28->29
29	134.1	8.97 (br s, 1H)	
30	-	Not observed	
31	169.7	-	
32	-	7.92 (d, J = 7.9 Hz, 1H)	HMBC 32->31
33	54.3	4.71 (ddd, J = 8.7, 7.9, 5.6 Hz, 1H)	HMBC 33->44
34	29.3	3.02-3.07 (m, 1H), 3.29 (dd, J = 14.8, 5.3 Hz, 1H)	HMBC 34->44
35	110.4	-	
36	125.7	-	
37	116.2	-	
38	154.9	-	
39	109.3	6.92 (dd, JHF = 9.7Hz, JHH = 8.9 Hz, 1H)	HMBC 39->37,41
40	110.7	7.21 (dd, JHH = 8.9 Hz, JHF = 4.4 Hz, 1H)	HMBC 40->36
41	133.4	-	
42	-	10.95 (d, J = 2.4 Hz, 1H)	TOCSY 42->43 HMBC 42->41
43	125.9	7.13 (d, J = 2.4 Hz, 1H)	
44	170.7	-	
45	-	8.01 (d, J = 7.5 Hz, 1H)	TOCSY 45->46,47,48,49,50 HMBC 45->44
46	51.9	4.14 (ddd, J = 8.1, 7.5, 6.0 Hz, 1H)	HMBC 46->53
47	28.3	1.44-1.53 (m, 1H), 1.62-1.70 (m,1 H)	
48	24.4	1.36-1.45 (m, 2H)	
49	40.2	3.03-3.09 (m, 2H) overlap	HMBC 49->47,51
50	-	7.47 (t, J = 5.5 Hz, 1H)	
51	156.6	-	
52	-	13.95-14.37 (m, 3H)	
53	173	-	
54	-	6.92 (br s, 1H) overlap, 6.99 (br s, 1H) overlap	TOCSY 54'->54

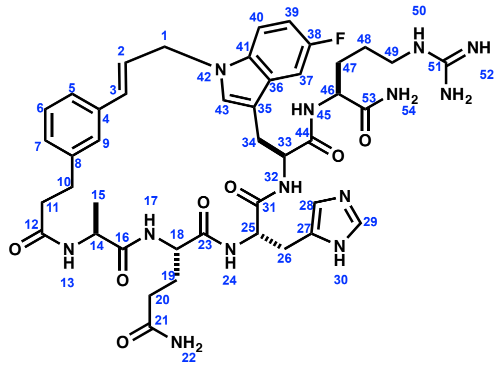
*Macrocyclic Product **11d***



(500 MHz, DMSO-d_6, 298K)

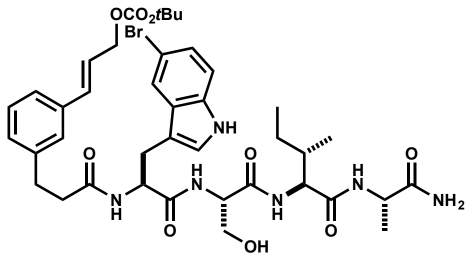
	13C	1H	key correlations
1	32.4	3.52 (dd, J = 15.3, 5.6 Hz, 1H), 3.64 (dd, J = 15.3, 6.3 Hz, 1H)	HMBC 1->38,39,40
2	129.3	6.39 (ddd, J = 15.8, 6.3, 5.6 Hz, 1H)	TOCSY 2->1,2 HMBC 2->4
3	129.9	6.30 (br d, J = 15.8 Hz, 1H)	HMBC 3->4
4	137	-	
5	124.1	7.13-7.17 (m, 1H) overlap	HMBC 5->7,9
6	128.3	7.15-7.19 (m, 1H) overlap	HMBC 6->4,8
7	127.3	7.10 (br d, J = 7.2 Hz, 1H)	HMBC 7->9
8	141.1	-	
9	124.5	7.10 (br s, 1H) overlap	HMBC 9->7
10	30.2	2.69-2.76 (m, 1H), 2.77-2.82 (m, 1H) overlap	HMBC 10->7,8,9,12
11	35.4	2.31 (ddd, J = 14.3, 6.5, 6.5 Hz, 1H), 2.44-2.51 (m, 1H) overlap	HMBC 11->8,12
12	171.4	-	
13	-	8.07 (d, J = 7.6 Hz, 1H)	HMBC 13->12
14	47.7	4.12 (qd, J = 7.6, 7.1 Hz, 1H)	HMBC 14->15,16
15	17.6	0.85 (d, J = 7.1 Hz, 3H)	HMBC 15->15,16
16	172.3	-	
17	-	7.86 (d, J = 7.7 Hz, 1H)	HMBC 17->16
18	52.3	3.98 (ddd, J = 8.0, 7.8, 5.4 Hz, 1H)	TOCSY 18->17,18,20 HMBC 18->19,20,23
19	27.3	1.50-1.58 (m, 1H) overlap, 1.70-1.81 (m, 1H) overlap	HMBC 19->20,21
20	30.9	1.91-2.00 (m, 2H)	HMBC 20->21
21	173.9	-	
22	-	6.78 (br s, 1H), 7.22 (br s, 1H) overlap	TOCSY 22->22'
23	171.2	-	
24	-	7.56 (br d, J = 6.8 Hz, 1H)	HMBC 24->23
25	50.7	4.45 (ddd, J = 7.2, 6.8, 5.8 Hz, 1H)	COSY 25->24
26	27.6	2.93 (dd, J = 15.3, 7.6 Hz, 1H), 3.05-3.12 (m, 1H) overlap	HMBC 26->27
27	128.9	-	
28	116.8	7.28 (s, 1H)	HMBC 28->27,30
29	-	not observed	
30	134	8.95 (br s, 1H)	HMBC 30->27,28
31	170	-	
32	-	8.07 (d, J = 7.4 Hz, 1H)	HMBC 32->31
33	53.7	4.60 (ddd, J = 10.9, 7.4, 3.3 Hz, 1H)	TOCSY 33->32,34
34	27.7	2.85 (dd, J = 14.4, 11.1 Hz, 1H), 3.13-3.20 (m, 1H) overlap	HMBC 34->33,35
35	109.8	-	
36	133	-	
37	103.8	7.57 (d, JHF = 11.1 Hz, 1H)	HMBC 37->35,41,38,39
38	155.3 (d, J ≅ 230Hz)	-	
39	120.1	-	
40	112.8	7.21 (d, JHF = 6.4 Hz, 1H)	HMBC 40->38
41	133	-	
42	-	10.83 (d, J = 1.3 Hz, 1H)	HMBC 42->35,36,41
43	125.7	7.17-7.19 (m, 1H) overlap	
44	172.2	-	
45	-	8.70 (br d, J = 7.8 Hz, 1H)	HMBC 45->44
46	52.2	4.25 (ddd, J = 7.8, 7.8, 6.1 Hz, 1H)	TOCSY 46->45,47,48,49
47	28.9	1.55-1.63 (m, 1H) overlap, 1.71-1.78 (m, 1H) overlap	HMBC 47->46
48	25.1	1.47-1.57 (m, 2H) overlap	HMBC 48->49
49	40.3	3.09-3.16 (m, 2H) overlap	HMBC 49->47,48,51
50	-	14.17 (br s) overlap	
51	156.9	-	
52	-	14.17 (br s) overlap	
53	173.3	-	
54	-	7.14 (br s, 1H), 7.39 (br s, 1H)	TOCSY 54'->54

*Macrocyclic Product **11a***

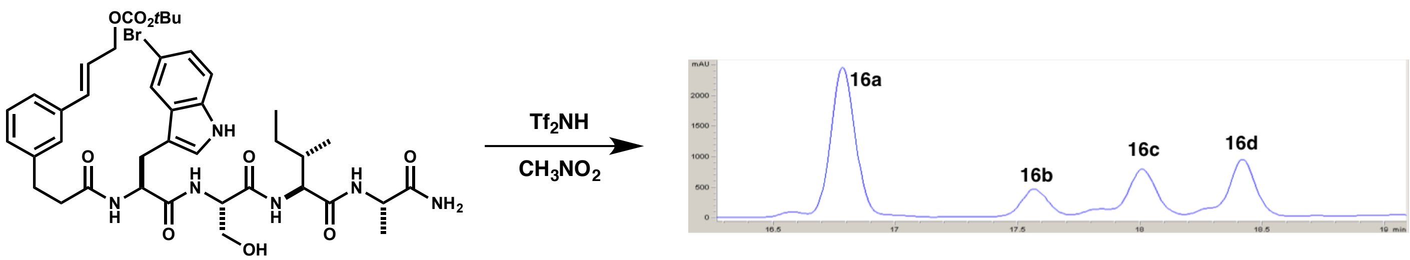


(500 & 600 MHz, DMSO-d_6, 298K)

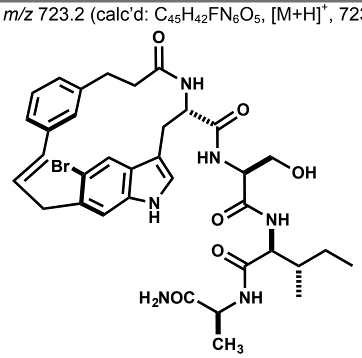
	13C	1H	key correlations
1	40.7	2.44 (dd, J = 14.3, 8.8 Hz, 1H), 2.68-2.73 (m, 1H)	HMBC 1->35,43
2	124.2	5.91 (ddd, J = 15.7, 8.8, 6.5 Hz, 1H)	COSY 2->1
3	134.2	6.55 (d, J = 15.7 Hz, 1H)	HMBC 3->1,4,5,9
4	136.7	-	
5	122.2	7.04-7.07 (m, 1H) overlap	
6	128.1	7.11-7.16 (m, 1H) overlap	HMBC 6->4,8
7	127.1	7.03-7.07 (m, 1H) overlap	
8	141.3	-	
9	126.2	7.13 (br s, 1H) overlap	HMBC 9->5,7
10	28.7	2.71-2.79 (m, 1H), 2.95-3.01 (m, 1H)	HMBC 10->8,12
11	34.3	2.51-2.57 (m, 1H), 2.59-2.66 (m, 1H)	HMBC 11->8,12
12	171.3	-	
13	-	8.09 (d, J = 8.1 Hz, 1H)	
14	48.3	4.13-4.19 (m, 1H) overlap	HMBC 14->16
15	17.6	1.20 (d, J = 7.3 Hz, 3H)	HMBC 15->14,16 COSY 15->14 TOCSY 15->14,13
16	171.8	-	
17	-	7.00-7.03 (m, 1H) overlap	HMBC 17->16
18	50.6	4.19 (ddd, J = 8.1, 7.8, 5.4 Hz, 1H)	HMBC 18->19,20
19	28.4	1.59-1.70 (m, 1H), 1.70-1.80 (m, 1H)	
20	31	1.99 (ddd, J = 15.5, 9.5, 5.8 Hz, 1H), 2.08 (ddd, J = 15.5, 9.9, 5.7 Hz, 1H)	HMBC 20->21
21	173.5	-	
22	-	6.84 (br s, 1H), 7.31 (br s, 1H)	HMBC 22->21
23	171	-	
24	-	8.77 (d, J = 8.1 Hz, 1H)	TOCSY 24->25,26 HMBC 24->23
25	48.3	5.07 (ddd, J = 9.7, 8.1, 5.2 Hz, 1H)	HMBC 25->31
26	25.6	3.17 (dd, J = 15.9, 5.1 Hz, 1H), 3.00 (dd, J = 15.9, 9.7 Hz, 1H)	HMBC 26->27,28,31
27	128.3	-	
28	117.3	7.50 (s, 1H)	HMBC 28->27,29
29	133.5	8.99 (s, 1H)	HMBC 29->27,28 TOCSY 29->28
30	-	Not detected	
31	170.2	-	
32	-	-	
33	60.1	4.62 (dd, J = 9.3, 4.5 Hz, 1H)	TOCSY 33->34 HMBC 33->1,34,35,44
34	40	2.21 (dd, J = 13.3, 4.5 Hz, 1H), 2.46-2.52 (m, 1H) overlap	HMBC 34->35
35	57.8	-	
36	135	-	
37	109.8	7.10-7.14 (m, 1H)	HMBC 37->38,41
38	156.5 (d, J ≅ 230 Hz)	-	
39	114	6.81-6.88 (m, 1H) overlap	HMBC 39->38,41
40	109.8	6.84 (dd, JHH = 9.1 Hz, JHF = 2.6 Hz, 1H)	HMBC 40->36,38 COSY 40->39
41	144.8	-	
42	-	Not detected	
43	80.3	6.19 (s, 1H)	
44	169.9	-	
45	-	7.56 (d, J = 8.2 Hz, 1H)	HMBC 45->44
46	50.6	4.04 (ddd, J = 8.2, 8.0, 6.0 Hz, 1H)	COSY 46->47 HMBC 46->44,54
47	29.9	0.97-1.06 (m, 1H) overlap, 1.33-1.42 (m, 1H) overlap	HMBC 47->49
48	24.4	1.00-1.11 (m, 1H) overlap, 1.36-1.46 (m, 1H) overlap	
49	39.8	2.89-2.97 (m, 1H) overlap, 3.03-3.11 (m, 1H)	HMBC 49->51
50	-	7.42 (apt t, J = 5.6 Hz, 1H)	
51	156.3	-	
52	-	14.11 (br s, 3H)	
53	172.4	-	
54	-	7.08 (br s, 1H), 7.40 (br s, 1H)	HMBC 55'->54 TOCSY 55'->55



Acyclic Cinnamyl Carbonate 12: Synthesized according to Procedure A with 0.41 mmol starting template. Purified via trituration with 3x5 mL methanol. Beige Solid. 170 mg (0.202 mmol) 49% yield. ^{1}H-NMR (500 MHz, DMSO-d$_6$) δ10.99 (d, *J* = 1.9 Hz, 1 H), 8.28 (d, *J* = 7.6 Hz, 1 H), 8.09 (d, *J* = 8.4 Hz. 1 H), 7.91 (d, *J* = 7.5 Hz, 1 H), 7.86 (d, *J* = 1.4 Hz, 1 H), 7.79 (d, *J* = 8.2 Hz, 1 H), 6.60 (d, *J* = 16.00 Hz, 1 H), 6.32 (dd, *J* = 15.9, 6.3, 6.3 Hz, 1 H), 5.08 (dd, *J* = 5.3, 5.3 Hz, 1 H), 4.66 (d, *J* = 5.95 Hz, 1 H), 4.60 (ddd, *J* = 9.0, 4.3, 4.3 Hz, 1 H), 4.39 (dd, *J* = 13.3, 6.1 Hz, 1 H), 4.22-4.17 (m, 2H), 3.67-3.55 (m, 2H), 3.09 (dd, *J* = 14.5, 3.8 Hz, 1 H), 3.09 (dd, *J* = 14.5, 3.8 Hz, 1 H), 2.85 (dd, *J* = 15.6, 9.9 Hz, 1 H), 2.68-2.57 (m, 2H), 2.32 (dd, *J* = 8.0, 8.0 Hz, 1 H), 1.81-1.76 (m, 1H), 1.43 (s, 9H), 1.20 (d, *J* = 7.2 Hz, 3 H), 1.17-1.12 (m, 1H), 1.10-1.04 (m, 1H), 0.86 (d, *J* = 6.7 Hz, 3 H), 0.82 (dd, *J* = 7.4, 7.4 Hz, 3 H). ^{13}C-NMR (126 MHz, d$_6$-DMSO) δ 174.1, 171.9, 171.3, 170.3, 170.2, 152.8, 141.7, 135.8, 134.7, 133.4, 129.3, 128.6, 127.9, 126.4, 126.0, 125.7, 124.2, 123.3, 123.2, 121.0, 113.2, 111.0, 110.1, 81.5, 66.9, 61.5, 57.1, 54.9, 53.2, 48.0, 36.9, 36.7, 31.0, 27.4, 24.1, 18.1, 15.4, 11.4. MS *m/z* [M-OCO$_2$*t*Bu]$^+$, 841.3 (calc'd: $C_{35}H_{44}BrN_6O_6$ [M+H]$^+$, 841.1)

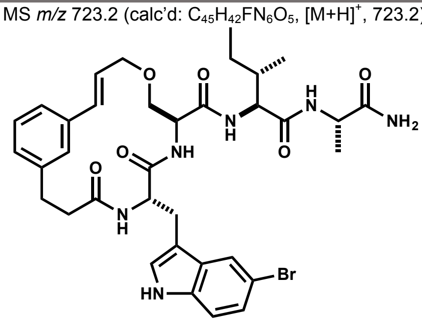


MS *m/z* 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



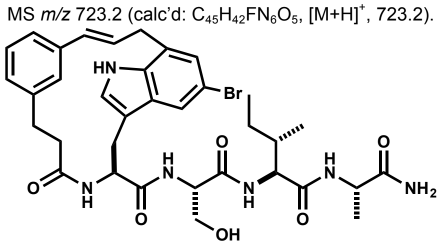
16a

MS *m/z* 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



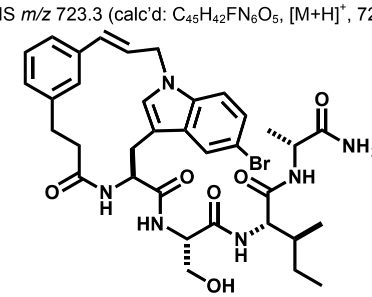
16b

MS *m/z* 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



16d

MS *m/z* 723.3 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



16e

Analytical HPLC Method
Column: Waters Sunfire™ C$_{18}$, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	30
2.5	30
24	86
29	30

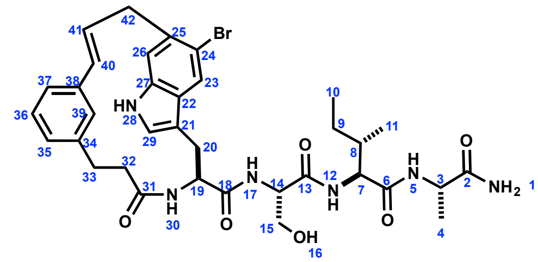
Preparative HPLC Method
Column: Waters Sunfire™ C$_{18}$, 19x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B ACN + 0.1% TFA
Flow rate: 18.0 mL/min

Time	%B
0	45
2	45
12	50
13	50
15	100

Semi-Prep HPLC Method
Column: Waters XSelect™ C$_{18}$, 10x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 6.00 mL/min

Time	%B
0	45
1	45
4	50
10	54
12	45

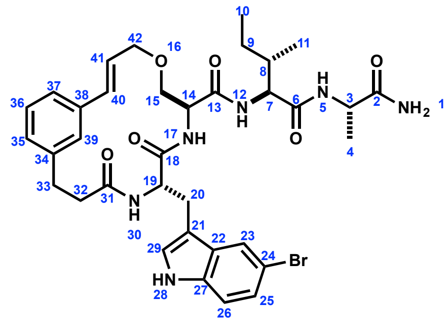
*Macrocyclic Product **16a***



(600 MHz, DMSO-d_6, 298K)

	13C	1H	key correlation
1	-	7.12 ppm (br s) (1H) ; 6.99 ppm (br s) (1H)	HMBC 1 -> 2 / TOCSY 1 -> 1'
2	173.9 ppm	-	HMBC 3 -> 2
3	47.7 ppm	4.21-4.18 ppm (m) (1H)	COSY 5 -> 3
4	17.9 ppm	1.22 ppm (d) J=7.2 Hz (3H)	COSY 3 -> 4
5	-	7.90 ppm (d) J=7.5 Hz (1H)	HMBC 5 -> 6
6	170.1 ppm	-	HMBC 7 -> 6
7	56.9 ppm	4.23-4.21 ppm (m) (1H)	COSY/HMBC 12 -> 7
8	36.4 ppm	1.83-1.78 ppm (m) (1H)	COSY 7 -> 8
9	23.8 ppm	1.46-1.42 ppm (m) (1H) ; 1.18-1.11 ppm (m) (1H)	COSY 8 -> 9
10	11.1 ppm	0.83 ppm (t) J=7.4 Hz (3H)	COSY 9 -> 10
11	15.0 ppm	0.87 ppm (d) J=6.8 Hz (3H)	COSY 8 -> 11
12	-	7.74 ppm (d) J=8.1 Hz (1H)	HMBC 12 -> 13
13	170.5 ppm	-	HMBC 14 -> 13
14	54.6 ppm	4.42 ppm (q) J=6.5 Hz (1H)	COSY 17 -> 14
15	61.3 ppm	3.69 ppm (dd) J=10.3, 5.9 Hz (1H) ; 3.62 ppm (dd) J=10.5, 6.4 Hz (1H)	COSY 14 -> 15
16	-	Not Observed	-
17	-	8.51 ppm (d) J=7.6 Hz (1H)	HMBC 17 -> 18
18	172.7 ppm	-	HMBC 19 -> 18
19	52.6 ppm	4.76 ppm (ddd) J=12.8, 6.2, 4.6 Hz (1H)	COSY 30 -> 19
20	27.7 ppm	3.32 ppm (dd) J=14.3, 3.8 Hz (1H) ; 2.81 ppm (t) J=13.7 Hz (1H)	COSY 19 -> 20
21	109.1 ppm	-	HMBC 20, 28, 29 -> 21
22	126.6 ppm	-	HMBC 26, 29 -> 22
23	122.6 ppm	8.19 ppm (s) (1H)	HMBC 23 -> 21
24	116.2 ppm	-	HMBC 23, 26 -> 24
25	131.6 ppm	-	HMBC 23, 42 -> 25
26	115.4 ppm	7.33 ppm (s) (1H)	TOCSY 23 -> 26
27	136.5 ppm	-	HMBC 23, 28, 29 -> 27
28	-	10.93 ppm (d) J=1.7 Hz (1H)	
29	126.5 ppm	7.23 ppm (d) J= 1.7 Hz (1H)	COSY/TOCSY 28 -> 29
30	-	7.52 ppm (d) J=6.4 Hz (1H)	HMBC 30 -> 31
31	171.7 ppm	-	HMBC 32, 33 -> 31
32	31.3 ppm	2.25 ppm (ddd) J=16.9, 5.6, 1.9 Hz (1H) ; 2.15 ppm (ddd) J=16.9, 12.7, 1.8 Hz (1H)	COSY/TOCSY 33 -> 32
33	25.9 ppm	3.02 ppm (dd) J=16.5, 12.6 Hz (1H) ; 2.45 ppm (dd) J=16.4, 5.6 Hz (1H)	HMBC 33 -> 35, 39
34	141.0 ppm	-	HMBC 32, 33, 36 -> 34
35	126.4 ppm	6.83 ppm (d) J=7.4 Hz (1H)	TOCSY 37 -> 35
36	127.6 ppm	7.05 ppm (t) J=7.7 Hz (1H)	COSY/TOCSY 37 -> 36
37	119.2 ppm	7.17 ppm (d) J=7.7 Hz (1H)	HMBC 37 -> 40, 41 (slight)
38	135.2 ppm	-	HMBC 36 -> 38
39	128.0 ppm	5.52 ppm (s)	TOCSY 37 -> 39
40	132.1 ppm	3.70 ppm (d) J=16.0 Hz (1H)	
41	127.9 ppm	6.30 ppm (dt) J=16.2, 3.9 Hz (1H)	
42	37.7 ppm	3.79 ppm (ddd) J= 17.0, 4.6, 1.5 Hz (1H) ; 3.43 ppm (dt) J=16.7, 2.3 Hz (1H)	COSY 41 -> 42

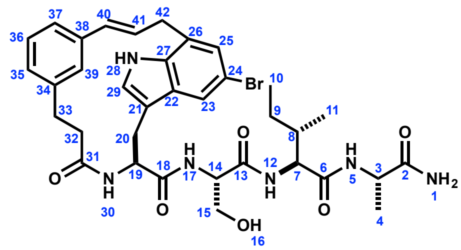
*Macrocyclic Product **16b***



(600 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	-	7.20 ppm (br s) (1H) ; 6.96 ppm (br s) (1H)	TOCSY 1 -> 1'
2	173.9 ppm	-	HMBC 3 -> 2
3	47.8 ppm	4.22 ppm (pentet) J=7.1 Hz (1H)	COSY 3 -> 4
4	18.2 ppm	1.21 ppm (d) J=7.2 Hz (3H)	
5	-	7.90 ppm (d) J=7.5 Hz (1H)	COSY 5 -> 3 ; slight HMBC 5 -> 3
6	170.2 ppm	-	HMBC 5 -> 6
7	56.8 ppm	4.22 ppm (dd) J=8.6, 6.6 Hz (1H)	TOCSY 7 -> 11
8	36.8 ppm	1.77-1.73 ppm (m) (1H)	COSY 7 -> 8
9	23.8 ppm	1.46-1.42 ppm (m) (1H) ; 1.13 -1.08 ppm (m) (1H)	COSY 8 -> 9
10	11.2 ppm	0.82 ppm (dd) J=7.5, 7.5 Hz (3H)	COSY 9 -> 10
11	15.2 ppm	0.85 ppm (d) J=7.0 Hz (3H)	COSY 8 -> 11
12	-	7.70 ppm (d) J=8.7 Hz	COSY 12 -> 7 ; slight HMBC 12 -> 7
13	169.1 ppm	-	HMBC 12 -> 13
14	52.5 ppm	4.60 ppm (ddd) J=7.5, 7.5, 3.6 Hz (1H)	COSY 14 -> 15
15	68.7 ppm	3.75 ppm (dd) J=11.3, 7.1 Hz (1H) ; 3.66 ppm (dd) J=11.1, 3.4 Hz (1H)	HMBC 15 -> 42
16	-	-	-
17	-	8.82 ppm (d) J=7.9 Hz (1H)	COSY 17 -> 14
18	172.0 ppm	-	HMBC 17 -> 18
19	51.8 ppm	5.05 ppm (ddd) J=9.6, 9.6, 9.6 Hz (1H)	COSY 30 -> 19
20	29.1 ppm	2.97 ppm (dd) J=14.6, 4.3 Hz (1H) ; 2.80 ppm (dd) J=14.2, 9.9 Hz (1H)	HMBC 20 -> 21 ; COSY/TOCSY 19 -> 20
21	110.2 ppm	-	HMBC 28, 29 -> 21
22	129.3 ppm	-	HMBC 26, 29 -> 22
23	121.0 ppm	7.85 ppm (d) J=1.9 Hz	HMBC 23 -> 27
24	110.9 ppm	-	HMBC 23, 26 -> 24
25	123.2 ppm	7.14 ppm (dd) J=8.4, 1.7 Hz (1H)	HMBC 25 -> 27 / 23 -> 25
26	113.1 ppm	7.27 ppm (d) J=8.7 Hz (1H)	TOCSY 23 -> 26
27	134.8 ppm	-	HMBC 29 -> 27
28	-	10.91 ppm (d) J=2.1 Hz (1H)	Indole
29	125.3 ppm	7.17 ppm (d) J=2.5 Hz (1H)	COSY/TOCSY 28 -> 29
30	-	8.16 ppm (d) J=9.4 Hz	HMBC 30 -> 31
31	171.2 ppm	-	HMBC 33 -> 31
32	36.6 ppm	2.41-2.36 ppm (m) (1H) ; 2.07 ppm (ddd) J=13.9, 7.1, 3.2 Hz (1H)	COSY/TOCSY 33 -> 32
33	30.2 ppm	3.02-2.97 ppm (m) (1H) ; 2.63-2.60 ppm (m) (1H)	HMBC 35, 39 -> 33
34	141.6 ppm	-	HMBC 33 -> 34
35	127.8 ppm	7.01 ppm (d) J=7.9 Hz (1H)	TOCSY 39 -> 35
36	123.2 ppm	7.16 ppm (dd) J=7.3, 7.3 Hz (1H)	HMBC 36 -> 38
37	125.3 ppm	7.02 ppm (d) J=8.1 Hz (1H)	HMBC 37 -> 40
38	135.9 ppm	-	HMBC 41 -> 38
39	123.9 ppm	7.26 ppm (br s) (1H)	HMBC 40 -> 39
40	131.3 ppm	6.47 ppm (d) J=15.8 (1H)	
41	127.3 ppm	6.04 ppm (ddd) J=15.9, 7.0, 5.6 Hz (1H)	
42	69.6 ppm	4.31 ppm (ddd) J=14.0, 5.1, 1.3 Hz (1H) ; 3.99 ppm (dd) J=14.1, 7.0 Hz (1H)	COSY 41 -> 42 ; HMBC 15 -> 42 / 42 -> 15

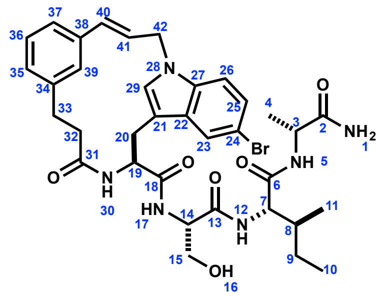
*Macrocyclic Product **16c***



(600 MHz, DMSO-d$_6$, 298K)

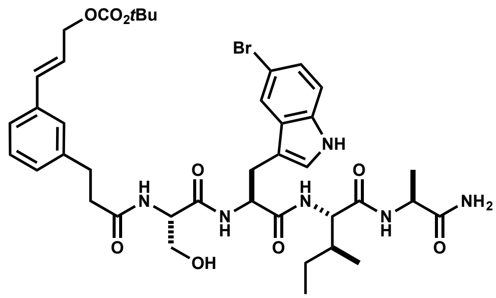
	13C	1H	key correlation
1	-	7.14 ppm (br s) (1H) ; 6.97 ppm (br s) (1H)	TOCSY 1 -> 1'
2	174.1 ppm	-	HMBC 3 -> 2
3	47.6 ppm	4.21 ppm (dd) J=7.3, 7.3 Hz (1H)	COSY 3 -> 4
4	18.0 ppm	1.21 ppm (d) J=7.1 Hz (3H)	
5	-	7.93 ppm (d) J=7.6 Hz (3H)	COSY 5 -> 3
6	170.2 ppm	-	HMBC 5 -> 6
7	56.6 ppm	4.23 ppm (dd) J=8.3, 6.1 Hz (1H)	TOCSY 7 -> 8
8	36.4 ppm	1.83-1.79 ppm (m) (1H)	COSY 8 -> 11 / TOCSY 8 -> 10
9	23.6 ppm	1.48-1.44 ppm (m) (1H)	COSY 9 -> 10
10	11.2 ppm	0.84 ppm (dd) J=7.4, 7.4 Hz (3H)	
11	15.1 ppm	0.88 ppm (d) J=6.8 Hz (3H)	
12	-	7.74 ppm (d) J=8.3 Hz (1H)	COSY/TOCSY 12 -> 7
13	170.3 ppm	-	HMBC 12 -> 13
14	54.7 ppm	4.42 ppm (dd) 13.5, 6.1 Hz (1H)	HMBC 14 -> 13
15	61.1 ppm	3.71-3.64 ppm (m) (2H)	COSY 14 -> 15
16	-	Not Observed	-
17	-	8.51 ppm (d) J=7.5 Hz (1H)	HMBC 17 -> 14
18	172.7 ppm	-	HMBC 17 -> 18
19	53.1 ppm	4.51 ppm (ddd) J=12.4, 8.5, 3.4 Hz	COSY 30 -> 19
20	27.1 ppm	3.23 ppm (dd) J=14.0, 3.0 Hz (1H) ; 2.78 ppm (dd) J=13.4 Hz (1H)	COSY 19 -> 20
21	109.7 ppm	-	HMBC 20, 28, 29 -> 21
22	128.0 ppm	-	HMBC 29 -> 22
23	119.8 ppm	7.95 ppm (d) 1.4 Hz (1H)	COSY/TOCSY 23 -> 25
24	110.0 ppm	-	HMBC 23 -> 24
25	123.9 ppm	7.09 ppm	HMBC 42 -> 25
26	126.9 ppm	-	HMBC 42 -> 26
27	134.6 ppm	-	HMBC 23, 25, 29 -> 27
28	-	10.56 ppm (d) J=1.9 Hz (1H)	
29	126.6 ppm	7.27 ppm (d) J=2.5 Hz (1H)	COSY/TOCSY 28 -> 29
30	-	8.12 ppm (d) J=8.3 Hz	HMBC 30 -> 31
31	172.0 ppm	-	HMBC 32, 33 -> 31
32	34.5 ppm	2.40-2.36 ppm (m) (1H) ; 2.15 ppm (dd) J=14.9, 11.9 Hz (1H)	COSY/TOCSY 33 -> 32
33	27.2 ppm	3.06 ppm (dd) J=13.5, 12.2 Hz (1H) ; 2.36-2.33 (m) (1H)	HMBC 35 -> 33
34	142.6 ppm	-	HMBC 36 -> 34
35	126.5 ppm	6.85 ppm (d) J=7.7 Hz (1H)	TOCSY 35 -> 39
36	128.1 ppm	7.08 ppm (dd) J=7.4, 7.4 Hz (1H)	COSY/TOCSY 35, 37 -> 36
37	120.4 ppm	7.19 ppm (d) J=7.7 Hz (1H)	HMBC 40 -> 37 / TOCSY 37 -> 39
38	137.3 ppm	-	HMBC 36 -> 38
39	127.7 ppm	5.69 ppm (s) (1H)	HMBC 39 -> 40
40	132.3 ppm	4.68 ppm (d) J=16.4 Hz (1H)	
41	126.6 ppm	6.11 ppm (ddd) J=16.3, 5.8, 2.9 Hz (1H)	
42	33.3 ppm	3.89 ppm (dd) J=17.3, 5.9 Hz (1H) ; 3.52-3.49 ppm (m) (1H)	COSY 41 -> 42

Macrocyclic Product ***16d***

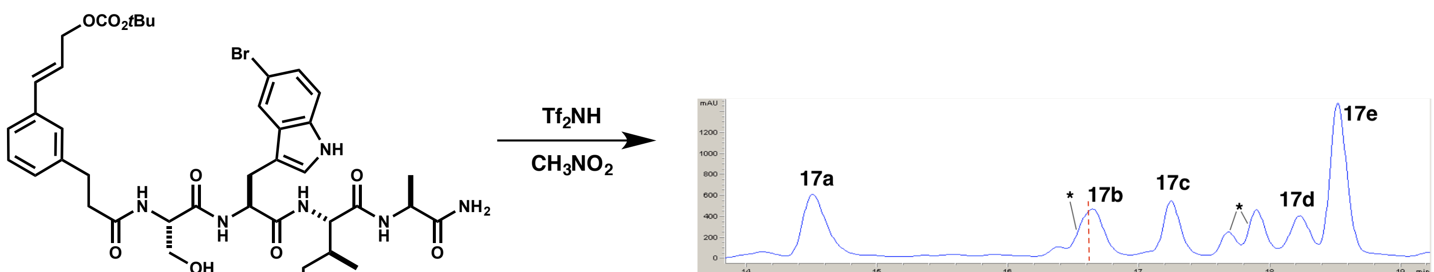


(600 MHz, DMSO-d_6, 298K)

	13C	1H	key correlation
1	-	7.11 ppm (br s) (1H) ; 6.97 ppm (br s) (1H)	HMBC 1' (slight) -> 3
2	174.1 ppm	-	HMBC 3 -> 2
3	47.9 ppm	4.21-4.18 ppm (m) (1H)	HMBC 5 -> 3
4	18.2 ppm	1.20 ppm (d) 7.1 Hz (3H)	COSY 3 -> 4
5	-	7.89 ppm (d) J=6.2 Hz (1H)	HMBC 5 ->
6	170.3 ppm	-	HMBC 7 -> 6
7	57.2 ppm	4.23-4.20 ppm (m) (1H)	HMBC 12 -> 7
8	36.8 ppm	1.82-1.78 ppm (m) (1H)	COSY 7 -> 8
9	24.0 ppm	1.46-1.42 ppm (m) (1H) ; 1.18-1.13 ppm (m) (1H)	COSY 8 -> 9
10	11.4 ppm	0.83 ppm (dd) J=7.4, 7.4 Hz (3H)	COSY 9 -> 10
11	15.4 ppm	0.87 ppm (d) J=7.2 Hz (3H)	COSY 8 -> 11
12	-	7.73 ppm (d) 8.3 Hz (1H)	HMBC 12 -> 13
13	170.3 ppm	-	HMBC 14 -> 13
14	54.9 ppm	4.39 ppm (ddd) J=6.5, 6.5, 6.5 Hz (1H)	COSY 17 -> 14
15	61.3 ppm	3.69 ppm (dd) J= 10.6, 5.9 Hz (1H) ; 3.62 ppm (dd) J=10.6, 5.9 Hz (1H)	COSY 14 -> 15
16	-	Not Observed	-
17	-	8.34 ppm (d) J=7.5 Hz (1H)	HMBC 17 -> 18
18	172.6 ppm	-	HMBC 19 -> 18
19	53.6 ppm	4.53 ppm (ddd)	COSY 19 -> 20
20	26.9 ppm	3.11 ppm (dd) J=14.9, 1.7 Hz (1H) ; 2.88 ppm (dd) J=14.8, 12.5 Hz (1H)	HMBC 20 -> 29
21	112.1 ppm	-	HMBC 20, 29 -> 21
22	130.3 ppm	-	HMBC 26, 29 -> 22
23	121.3 ppm	7.90 ppm (d) J=1.8 Hz (1H)	COSY/TOCSY 23 -> 25
24	111.9 ppm	-	HMBC 23, 25 (slight), 26 -> 24
25	123.7 ppm	7.25 ppm (d) J=8.7, 1.8 Hz (1H)	
26	112.6 ppm	7.57 ppm (d) J=8.7 Hz (1H)	TOCSY 23 -> 26 ; COSY 25 -> 26
27	136.3 ppm	-	HMBC 23, 25, 29 -> 27
28	-	-	-
29	129.5 ppm	7.37 ppm (s)	HMBV 42 -> 29
30	-	8.38 ppm (d) J=7.6 Hz (1H)	HMBC 30 -> 31 ; COSY 30 -> 19
31	172.5 ppm	-	HMBC 32, 33 -> 31
32	32.8 ppm	2.66 ppm (dd) J=14.0, 14.0 Hz (1H) ; 2.40-2.36 ppm (1H)	HMBC 32 -> 34
33	27.0 ppm	2.66-2.62 ppm (m) (1H) ; 3.09 ppm (dd) J=15.8, 12.9 Hz (1H)	HMBC 33' -> 34,35,39
34	141.7 ppm	-	HMBC 36 -> 34
35	127.4 ppm	6.99 ppm (d) J=8.1 Hz (1H)	COSY 36 -> 35
36	128.0 ppm	7.13 ppm (dd) J=7.6 Hz (1H)	COSY 36 -> 37
37	123.8 ppm	6.98 ppm (d) J=7.1 Hz (1H)	HMBC 37 -> 40
38	137.8 ppm	-	HMBC 41 -> 38
39	125.1 ppm	6.69 ppm (s) (1H)	HMBC 39 -> 40
40	132.2 ppm	6.50 ppm (d) J=15.6 Hz (1H)	
41	128.1 ppm	5.99 ppm (ddd) J=15.5, 7.7, 6.5 Hz (1H)	
42	45.4 ppm	4.80-4.78 ppm (m) (2H)	COSY 41 -> 42

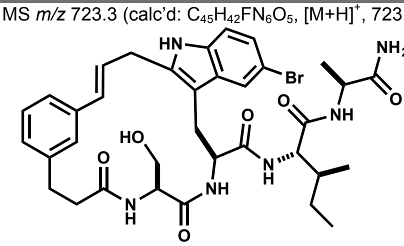


Acyclic Cinnamyl Carbonate 13: Synthesized according to Procedure A with 0.350 mmol starting template. Purified via SiO_2 chromatography using a gradient from 1% to 10% methanol in chloroform. Beige Solid. 80% yield. ^{1}H-NMR (500 MHz, DMSO-d_6) δ 11.06 (s, 1H), 8.09 (d, *J* = 7.7 Hz, 1H), 7.94 (d, *J* = 7.6 Hz, 1H), 7.88 (d, *J* = 7.7 Hz, 1H), 7.85 (d, *J* = 8.6 Hz, 1H), 7.74 (d, *J* = 1.3 Hz, 1H), 7.29-7.18 (m, 5H), 7.15 (dd, *J* = 8.5, 1.5 Hz, 1H), 6.97 (br s, 2H), 6.63 (d, *J* = 16.0 Hz, 1H), 6.33 (ddd, *J* = 15.9, 6.2, 6.2 Hz, 1H), 5.03 (dd, *J* = 5.5, 5.5 Hz, 1H), 4.66 (d, *J* = 6.1 Hz, 1H), 4.52 (ddd, *J* = 8.0, 8.0, 4.8 Hz, 1H), 4.32 (dd, *J* = 13.6, 6.4 Hz, 1H), 4.19 (pentet, *J* = 7.5 Hz, 1H), 4.15 (dd, *J* = 8.0, 8.0 Hz, 1H), 3.64-3.58 (m, 1H), 3.48 (dd, *J* = 5.8, 5.8 Hz, 1H), 3.14-3.11 (m, 2H), 2.95 (dd, *J* = 14.7, 8.8 Hz, 1H), 2.77-2.74 (m, 2H), 2.46-2.40 (m, 2H), 1.73-1.68 (m, 1H), 1.43 (s, 9H), 1.39-1.35 (m, 1H), 1.20 (d, *J* = 7.1 Hz, 3H), 1.07-1.01 (m, 1H), 0.79 (d, *J* = 7.1 Hz, 3H), 0.79 (dd, *J* = 7.5, 7.5 Hz, 3H). (126 MHz, DMSO-d_6) δ174.0, 171.6, 171.2, 170.4, 170.3, 152.8, 141.8, 135.8, 134.7, 133.4, 129.2, 128.6, 128.0, 126.4, 125.5, 124.2, 123.3, 123.3, 120.7, 113.3, 111.0, 109.8, 81.5, 66.9, 61.8, 57.0, 55.0, 53.5, 48.1, 36.6, 30.9, 27.4, 24.2, 18.2, 18.1, 16.7, 15.2, 11.1. MS *m/z* [M-OCO_2tBu]$^+$, 841.3 (calc'd: $C_{35}H_{44}BrN_6O_6$ [M+H]$^+$, 841.1)



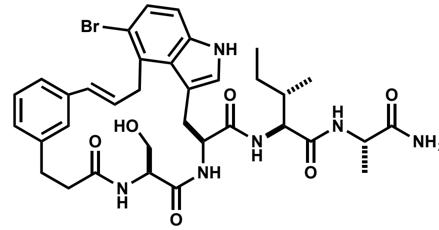
*Unidentified isomeric products

MS *m/z* 723.3 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



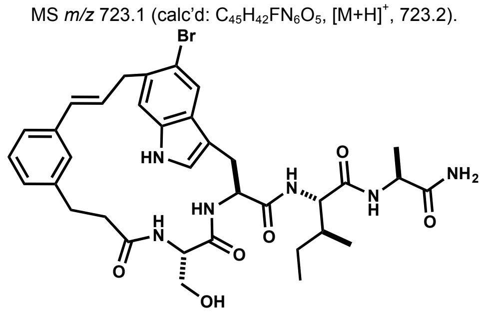
17a

MS *m/z* 723.1 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



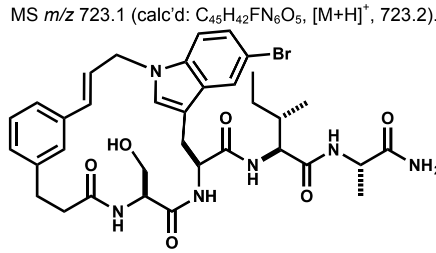
17b

MS *m/z* 723.1 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



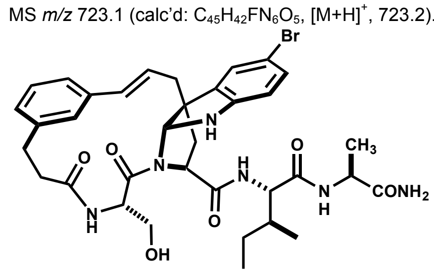
17c

MS *m/z* 723.1 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



17d

MS *m/z* 723.1 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



17e

Analytical HPLC Method
Column: Waters Sunfire™
C_{18}, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	30
2.5	30
24	86
29	30

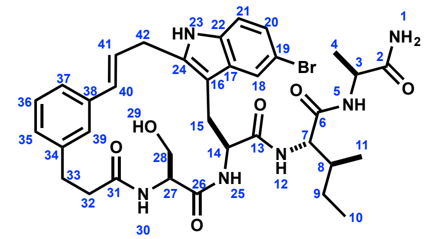
Preparative HPLC Method
Column: Waters Sunfire™
C_{18}, 19x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent A: ACN + 0.1% TFA
Flow rate: 18.0 mL/min

Time	%B
0	35
4	45
18	57
18.5	35

Semi-Prep HPLC Method
Column: Waters XSelect™
C_{18}, 10x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 6.00 mL/min

Time	%B
0	45
1	45
9	49

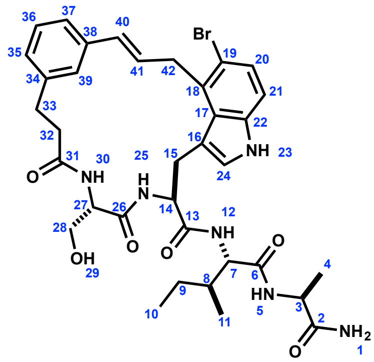
*Macrocyclic Product **17a***



(600 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	-	6.91 ppm (br s) (1H) ; 7.17 ppm (br s) (1H)	HMBC 1 -> 2 / TOCSY 1 -> 1'
2	173.7 ppm	-	HMBC 3 -> 2
3	47.9 ppm	4.19 ppm (p) J=7.1 Hz (1H)	COSY 5 -> 3
4	17.9 ppm	1.22 ppm (d) J=7.1 Hz (3H)	COSY 3 -> 4
5	-	7.97 ppm (d) J=7.1 Hz (3H)	HMBC 5 -> 6
6	170.4 ppm	-	HMBC 7 -> 6
7	56.9 ppm	4.27 ppm (t) J=8.1 Hz (1H)	COSY 12 -> 7
8	36.3 ppm	1.79-1.75 ppm (m) (1H)	COSY 7 -> 8
9	24.1 ppm	1.50-1.46 ppm (m) (1H) ; 1.18-1.11 ppm (m) (1H)	COSY 8 -> 9
10	10.7 ppm	0.86 ppm (t) J=7.4 Hz (3H)	COSY 9 -> 10
11	15.0 ppm	0.88 ppm (d) J=6.8 Hz (3H)	COSY 8 -> 11
12	-	7.81 ppm (d) J=8.9 Hz (1H)	HMBC 12 -> 13
13	171.0 ppm	-	HMBC 14 -> 13
14	25.8 ppm	4.50-4.47 ppm (m) (1H)	COSY 25 -> 14
15	25.9 ppm	3.29 ppm (dd) J=14.7 & 2.4 Hz (1H) ; 2.91 ppm (dd) J=14.7 & 10.3 Hz (1H)	COSY 14 -> 15
16	107.1 ppm	-	HMBC 18 & 23 -> 16
17	129.7 ppm	-	HMBC 21 & 23 -> 17
18	119.7 ppm	7.65 ppm (d) J=1.3 Hz (1H)	COSY 18->20 / TOCSY 18 -> 21
19	110.6 ppm	-	HMBC 18, 20 (slight), 21 -> 19
20	122.8 ppm	7.12 ppm (dd) J=8.5 & 1.4 Hz (1H)	HMBC 18 -> 20
21	112.5 ppm	7.20 ppm (d) J=8.7 Hz (1H)	HMBC 21 -> 17
22	134.2 ppm	-	HMBC 18, 20, 23 -> 22
23	-	10.92 ppm (br s)	
24	136.1 ppm	-	HMBC 15, 22, & 42 -> 24
25	-	8.83 ppm (d) J=8.6 Hz (1H)	HMBC 25 -> 26
26	170.6 ppm	-	HMBC 27 -> 26
27	54.6 ppm	4.56-4.52 ppm (m) (1H)	COSY 30 -> 27
28	62.2 ppm	3.63 ppm (dd) J=9.5 & 5.5 Hz (1H); 3.43 (t) J=9.3 Hz (1H)	COSY/TOCSY 27 -> 28
29	-	Not Observed	-
30	-	8.01 ppm (d) J=7.2 Hz (1H)	HMBC 30 -> 31
31	172.0 ppm	-	HMBC 32 -> 31
32	34.3 ppm	2.59 ppm (ddd) J=14.2, 11.8, & 2.8 Hz (1H) ; 2.21 ppm (ddd) J= 14.4, 6.7, & 2.9 Hz (1H)	COSY/TOCSY 33 -> 32
33	29.3 ppm	3.07 ppm (ddd) J=14.6, 11.7, & 2.0 Hz (1H) ; 2.64 ppm (ddd) J= 14.9, 7.0, & 1.5 Hz (1H)	HMBC 33 -> 34, 35, & 39
34	141.2 ppm	-	HMBC 33 & 32' -> 34
35	127.5 ppm	6.99 ppm (d) J=7.6 Hz (1H)	HMBC 37 & 39 -> 35
36	128.1 ppm	7.18 ppm (t) J=7.2 Hz (1H)	COSY 36 -> 35
37	124.5 ppm	7.06 ppm (d) J=8.0 Hz (1H)	HMBC 40->37
38	136.5 ppm	-	HMBC 41 & 42 -> 38
39	122.5 ppm	7.08 ppm (s) (1H)	HMBC 40 -> 39 ; 39 ->37
40	130.6 ppm	6.57 ppm (d) J=15.7 Hz (1H)	
41	127.9 ppm	5.98 ppm (ddd) J=15.7, 8.4, 5.9 Hz (1H)	
42	29.1 ppm	3.75 (dd) J=14.4 & 5.4 Hz (1H) ; 3.50 (dd) J=14.5 & 8.7 Hz (1H)	COSY/TOCSY 40 & 41 -> 42

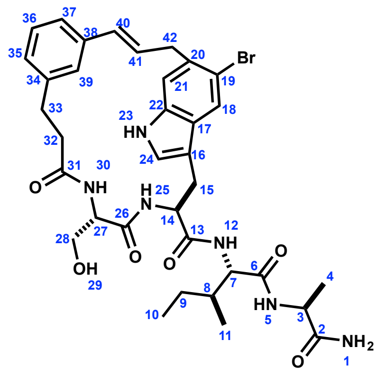
*Macrocyclic Product **17b***



(600 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	-	7.61 ppm (br s) (1H) ; 6.95 ppm (br s) (1H)	TOCSY 1 -> 1'
2	174.1 ppm	-	HMBC 3 -> 2
3	47.9 ppm	4.16 ppm (pentet) J=7.2 Hz (1H)	COSY 3 -> 4 / HMBC 1->3
4	18.1 ppm	1.16 ppm (d) J=7.1 Hz (3H)	
5	-	7.84 ppm (d) J=8.5 Hz (1H)	COSY 5 -> 3
6	170.1 ppm	-	HMBC 7 -> 6
7	56.9 ppm	4.16 ppm (dd) J=8.3, 7.4 Hz (1H)	TOCSY 7 -> 8,11
8	36.3 ppm	1.75-1.71 ppm (m) (1H)	COSY 7 -> 8
9	24.1 ppm	1.41-1.37 ppm (m) (1H) ; 1.09-1.02 ppm (m) (1H)	COSY 8 -> 9
10	11.0 ppm	0.78 ppm (dd) J=7.4, 7.4 Hz (3H)	COSY 9 -> 10
11	15.2 ppm	0.80 ppm (d) J= 6.8 Hz (3H)	COSY 8 -> 11
12	-	7.80 ppm (d) J=7.4 Hz (1H)	COSY 12 -> 7
13	170.4 ppm	-	HMBC 12 -> 13
14	53.0 ppm	4.64-4.58 ppm (m) (1H)	
15	29.4 ppm	3.36-3.34 ppm (m) (1H) ; 3.12 ppm (dd) J=15.0, 2.7 Hz (1H)	COSY 14 -> 15
16	110.8 ppm	-	HMBC 24 -> 16
17	127.4 ppm	-	HMBC 21,24 -> 17
18	129.9 ppm	-	HMBC 20 -> 18
19	115.1 ppm	-	HMBC 21 -> 19
20	124.5 ppm	7.26 ppm (d) J=8.7 Hz (1H)	
21	111.7 ppm	7.19 ppm (d) J=8.6 Hz (1H)	COSY 20 -> 21
22	135.6 ppm	-	HMBC 20,24 -> 22
23	-	11.03 ppm (s) (1H)	Indole
24	124.3 ppm	7.06 ppm (d) J=1.5 Hz (1H)	COSY 23 -> 24
25	-	8.26 ppm (br s) (1H)	COSY 25 -> 14
26	170.7 ppm	-	HMBC 25 -> 26
27	54.0 ppm	4.58 ppm (ddd) J=8.6, 6.5, 6.5 (1H)	COSY 27 -> 28
28	62.1 ppm	3.50-3.41 ppm (m) (2H)	COSY 29 -> 28
29	-	4.80 ppm (dd) J=5.4, 5.4 Hz (1H)	affected by water suppression
30	-	7.84 ppm (d) J=8.5 Hz (1H)	COSY 27 -> 30
31	170.9 ppm	-	HMBC 32, 33 -> 31
32	35.9 ppm	2.48-2.46 ppm (m) (1H) ; 2.32 ppm (ddd) J=14.5, 6.7, 2.5 Hz (1H)	COSY/TOCSY 33 -> 32
33	30.0 ppm	3.03 ppm (dd) J=12.4, 12.4 Hz (1H) ; 2.65-2.61 ppm (m) (1H)	HMBC 33 -> 35,39
34	141.7 ppm	-	HMBC 33,36 -> 34
35	127.5 ppm	6.98 ppm (d) J=7.4 Hz (1H)	HMBC 37 -> 35
36	128.2 ppm	7.11 ppm (dd) J=7.5, 7.5 Hz (1H)	COSY 36 -> 35,37 ; TOCSY 36 -> 37
37	123.0 ppm	7.06 ppm (d) J=7.7 Hz (1H)	HMBC 37 -> 40
38	136.9 ppm	-	HMBC 36 -> 38
39	125.6 ppm	7.03 ppm (br s) (1H)	HMBC 35,37 -> 39
40	130.6 ppm	6.14 ppm (d) J=15.6 Hz (1H)	HMBC 37 -> 40
41	not observed	6.36 ppm (ddd) J=16.0, 5.5, 5.5 Hz (1H)	
42	35.7 ppm	4.05-4.00 ppm (m) (2H)	COSY/TOCSY 40, 41 -> 42

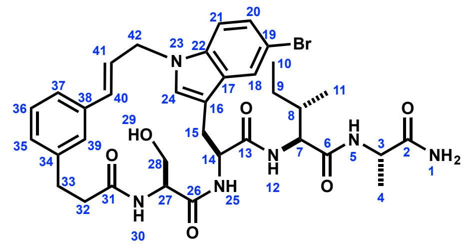
*Macrocyclic Product **17c***



(600 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	-	7.23 ppm (br s) (1H) ; 6.95 ppm (br s) (1H)	TOCSY 1 -> 1'
2	174.0 ppm	-	HMBC 3 -> 2
3	48.0 ppm	4.21 ppm (p) J=7.0 Hz (1H)	COSY 5 -> 3
4	18.2 ppm	1.21 ppm (d) J=7.0 Hz (3H)	
5	-	8.02 ppm (d) J=7.2 Hz (1H)	HMBC 5 -> 6
6	170.6 ppm	-	HMBC 7 -> 6
7	56.7 ppm	4.25 ppm (dd) J=8.6, 8.6 Hz (1H)	TOCSY 7 -> 8
8	36.5 ppm	1.80-1.75 ppm (m) (1H)	COSY/TOCSY 8 -> 11
9	24.2 ppm	1.53-1.49 ppm (m) (1H)	COSY/TOCSY 9 -> 10
10	10.9 ppm	0.86 ppm (dd) J= 7.6, 7.6 Hz	
11	15.1 ppm	0.88 ppm (d) J=6.8 Hz (1H)	
12	-	7.85 ppm (d) J=8.9 Hz (1H)	COSY 12 -> 7
13	170.8 ppm	-	HMBC 12 -> 13
14	57.5 ppm	4.47 ppm (ddd) J=12.0, 9.7, 2.2 Hz (1H)	COSY 25 -> 14
15	26.6 ppm	3.31-3.28 ppm (m) (1H) ; 2.88 ppm (dd) J=14.3, 12.2 Hz (1H)	COSY 14 -> 15
16	111.9 ppm	-	HMBC 15, 24 -> 16
17	128.6 ppm	-	HMBC 21,24 -> 17
18	122.3 ppm	8.23 ppm (s) (1H)	
19	114.6 ppm	-	HMBC 18, 21 -> 19
20	128.7 ppm		HMBC 18,42 -> 20
21	113.3 ppm	7.23 ppm (s) (1H)	
22	135.2 ppm	-	HMBC 18,24 -> 22
23	-	10.93 ppm (d) J=1.7 Hz (1H)	Indole
24	124.2 ppm	7.29 ppm (d) J=1.7 Hz (1H)	COSY 23 -> 24 ; HMBC 15 -> 24
25	-	8.68 ppm (d) J=9.6 Hz (1H)	HMBC 25 -> 26
26	169.4 ppm	-	HMBC 27 -> 26
27	54.8 ppm	4.18 ppm (dd) J=11.9, 6.3 Hz (1H)	COSY/TOCSY 30 -> 27
28	62.1 ppm	3.75-3.72 ppm (m) (1H) ; 3.47-3.44 ppm (m) (1H)	COSY 27 -> 28 ; TOCSY 30 -> 28
29	-	5.36 ppm (dd) J=5.3, 5.3 Hz (1H)	COSY 29 -> 28 ; TOCSY 29 -> 27
30		7.36 ppm (d) J=6.2 Hz (1H)	HMBC 30 -> 31
31	171.2 ppm	-	HMBC 32,33' -> 31
32	33.1 ppm	2.51-2.48 ppm (m) (1H) ; 2.29 ppm (dd) J=15.5, 6.7 Hz (1H)	COSY/TOCSY 33 -> 32
33	27.2 ppm	3.10 ppm (dd) J=15.2, 12.2 Hz (1H) ; 2.50-2.54 ppm (m) (1H)	HMBC 35 -> 33
34	141.8 ppm	-	HMBC 32,33(slight),36 -> 34
35	127.0 ppm	6.91 ppm (d) J=7.6 Hz (1H)	HMBC 32(slight),33 -> 35
36	128.2 ppm	7.13 ppm (dd) J= 7.6, 7.6 Hz (1H)	TOCSY 36 -> 39
37	121.2 ppm	7.28 ppm (d) J=7.2 Hz (1H)	COSY 37 -> 39
38	136.7 ppm	-	HMBC 36,40(slight) -> 38
39	125.8 ppm	6.38 ppm (s) (1H)	HMBC 39 -> 40
40	129.6 ppm	5.43 ppm (d) J=15.9 Hz (1H)	
41	128.6 ppm	6.46 ppm (ddd) J=16.0, 4.8, 4.8 Hz (1H)	
42	38.1 ppm	3.76-3.72 ppm (m) (1H) ; 3.52-3.48 ppm (m) (1H)	COSY/TOCSY 40, 41 -> 42 ; HMBC 21 -> 42

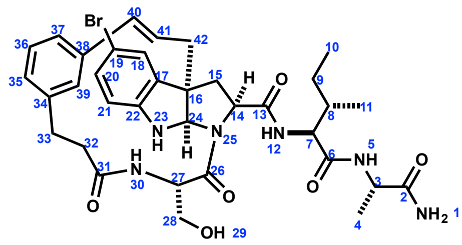
*Macrocyclic Product **17d***



(600 MHz, DMSO-d$_6$, 298K)

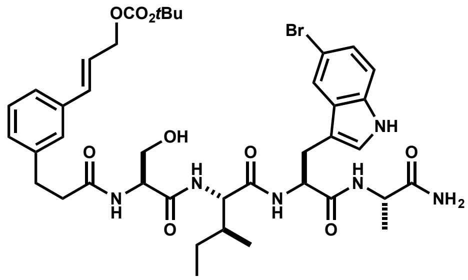
	13C	1H	key correlation
1	-	7.22 ppm (br s) (1H) ; 6.95 ppm (br s) (1H)	TOCSY 1 -> 1'
2	173.3 ppm	-	HMBC 3 -> 2
3	47.7 ppm	4.20 ppm (p) J=7.2 Hz (1H)	COSY 3 -> 4
4	18.0 ppm	1.23 ppm (d) J=7.2 Hz (3H)	
5	-	7.97 ppm (d) J=6.5 Hz (1H)	HMBC 5 -> 6
6	170.1 ppm	-	HMBC 7 -> 6
7	56.7 ppm	4.25 ppm (t) J=8.5 Hz (1H)	COSY 12 -> 7
8	22.2 ppm	1.79-1.76 ppm (m) (1H)	COSY 7 -> 8
9	24.0 ppm	1.48 -1.44 ppm (m) (1H) ; 1.15-1.10 ppm (m) (1H)	COSY 8 -> 9
10	10.7 ppm	0.84 ppm (t) J=7.5 Hz (3H)	COSY 9 -> 10
11	15.0 ppm	0.87 ppm (d) J=6.8 Hz (3H)	COSY 8 -> 11
12	-	7.97 ppm (d) J=9.4 Hz (1H)	HMBC 12 -> 13
13	171.2 ppm	-	HMBC 14 -> 13
14	53.1 ppm	4.62 ppm (ddd) J=12.2, 8.7, & 2.5 Hz (1H)	COSY/TOCSY 14 -> 15
15	26.4 ppm	3.25 ppm (dd) J=14.9, 1.7 Hz (1H) ; 2.86 ppm (dd) J=14.7, 12.5 Hz (1H)	HMBC 15 -> 16
16	110.4 ppm	-	HMBC 15, 24 -> 16
17	128.8 ppm	-	HMBC 21, 24 -> 17
18	120.5 ppm	7.79 ppm (d) J=1.9 Hz (1H)	HMBC 18 -> 20, 22
19	111.3 ppm	-	HMBC 18 -> 19
20	123.2 ppm	7.23 ppm (dd) J=8.5 Hz, 1.9 Hz (1H)	HMBC 18 -> 20
21	111.5 ppm	7.45 ppm (d) J=8.9 Hz (1H)	HMBC 21 -> 17
22	134.4 ppm	-	HMBC 18, 24, 42 -> 22
23	-	-	-
24	127.9 ppm	7.29 ppm (s) (1H)	HMBC 42 -> 24
25	-	8.56 ppm (d) J=8.5 Hz (1H)	COSY 25 -> 14
26	170.0 ppm	-	HMBC 25 -> 26
27	53.5 ppm	4.48 ppm (ddd) J=7.7, 7.7, 5.4 Hz (1H)	HMBC 27 -> 26
28	62.5 ppm	3.54 ppm (dd)	
29	-	Not Observed	-
30	-	7.78 ppm (d) J=7.9 Hz (1H)	COSY 30 -> 27
31	171.0 ppm	-	HMBC 30 -> 31
32	34.7 ppm	2.60-2.54 ppm (m) (1H) ; 2.23 ppm (ddd) J=15.2, 7.5, 2.6 Hz (1H)	HMBC 32 -> 31, 34
33	29.0 ppm	3.04 ppm (ddd) J=14.4, 11.6, 2.2 Hz (1H) ; 2.60-2.54 (m) (1H)	COSY/TOCSY 32 -> 33
34	141.5 ppm	-	HMBC 32 -> 34
35	128.1 ppm	7.18-7.17 ppm (m) (1H)	COSY 35 -> 36 / TOCSY 35 -> 39
36	128.0 ppm	7.01-6.99 ppm (m)	COSY/TOCSY 35, 37 -> 36
37	123.6 ppm	7.18-7.17 ppm (m) (1H)	HMBC 37 -> 40 / TOCSY 37 -> 39
38	135.6 ppm	-	HMBC 41 -> 38
39	123.9 ppm	6.85 ppm (s) (1H)	HMBC 39 -> 35, 37, 40
40	130.4 ppm	6.10 ppm (d) J=16.1 Hz (1H)	
41	125.1 ppm	6.19 ppm (dt) J=15.9, 5.4 Hz (1H)	
42	46.3 ppm	4.92 ppm (d) J=5.1 Hz (2H)	COSY/TOCSY 40, 41 -> 42

*Macrocyclic Product **17e***

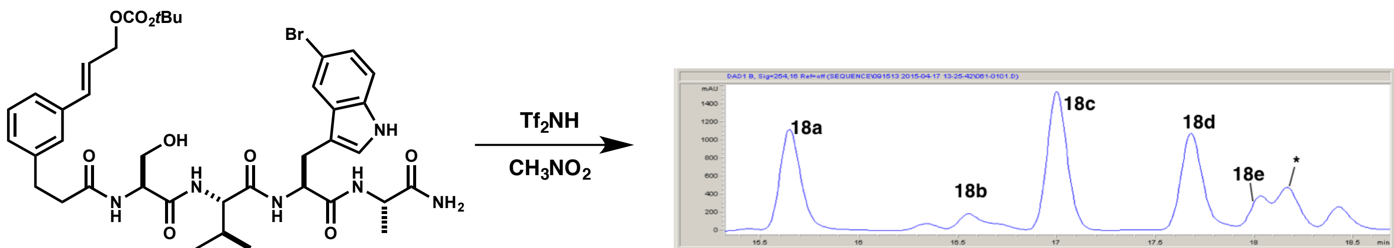


(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	-	7.04 ppm (br s) (1H) ; 6.93 ppm (br s) (1H)	HMBC 1 & 1' -> 2
2	173.8 ppm	-	HMBC 3 -> 2
3	48.0 ppm	4.10 ppm (p) J=7.3 Hz (1H)	COSY 5 -> 3
4	17.8 ppm	1.17 ppm (d) J=7.2 Hz (3H)	COSY/TOCSY 3 -> 4
5	-	7.69 ppm (d) J=7.5 Hz (1H)	HMBC 5 -> 6
6	170.3 ppm	-	HMBC 7 -> 6
7	56.5 ppm	4.01 ppm (dd) J=8.8, 5.7 Hz (1H)	COSY 12 -> 7
8	37.0 ppm	1.60-1.54 ppm (m) (1H)	COSY/TOCSY 7->8
9	23.5 ppm	1.18-1.15 ppm (m) (1H) ; 0.87-0.82 ppm (m) (1H)	COSY/TOCSY 8 -> 9
10	11.5 ppm	0.69 ppm (t) J=7.3 Hz (3H)	COSY/TOCSY 9 -> 10
11	15.1 ppm	0.57 ppm (d) J=6.8 Hz (3H)	COSY 7 -> 11
12	-	7.34 ppm (d) J=8.9 Hz (1H)	HMBC 12 -> 13
13	170.8 ppm	-	HMBC 14 -> 13
14	61.2 ppm	4.49 ppm (dd) J=10.4, 5.1 Hz (1H)	COSY/TOCSY 14 -> 15 / HMBC 14 -> 24
15	40.3 ppm	2.60 ppm (dd) J=13.8, 10.5 Hz (1H) ; 2.08 ppm (dd) J=13.8, 5.1 Hz (1H)	HMBC 42 -> 15 / HMBC 15' -> 24
16	57.6 ppm	-	HMBC 14, 15, 18, 42 -> 16
17	137.6 ppm	-	HMBC 21 -> 17
18	124.9 ppm	7.13 ppm (d) J=2.1 Hz (1H)	HMBC 18 -> 16 / TOCSY 18 -> 20, 21
19	109.5 ppm	-	HMBC 18, 20 (slight), 21 -> 19
20	130.3 ppm	7.16 ppm (dd) J= 8.2, 2.2 Hz (1H)	HMBC 18 -> 20
21	111.0 ppm	6.50 ppm (d) J= 8.3 Hz (1H)	COSY TOCSY 20 -> 21
22	146.6 ppm	-	HMBC 18, 20 -> 22
23	-	Not Observed	-
24	81.4 ppm	6.08 ppm (s) (1H)	Aminal (distinctive)
25	-	-	-
26	171.0 ppm	-	HMBC 27 -> 26
27	51.1 ppm	5.08 ppm (dt) 8.4, 5.9 Hz (1H)	COSY 30 -> 27
28	62.9 ppm	3.64-3.61 ppm (m) (1H)	COSY 27 -> 28
29	-	Not Observed	-
30	-	7.63 ppm (d) J= 8.2 Hz (1H)	HMBC 30 -> 31
31	171.8 ppm	-	HMBC 32, 33 -> 31
32	37.6 ppm	2.42 ppm (dt) J=12.4, 3.1 Hz (1H) ; 2.24 ppm (ddd) 12.8, 5.4, 4.0 Hz (1H)	COSY/TOCSY 33 -> 32
33	31.1 ppm	2.95-2.90 ppm (m) (1H) ; 2.69-2.65 ppm (m) (1H)	HMBC 33 -> 34
34	140.6 ppm	-	HMBC 36 -> 34
35	127.3 ppm	7.02 ppm (d) J=6.9 Hz (1H)	COSY/TOCSY 36 -> 35 / HMBC 35 -> 34/37
36	128.6 ppm	7.18 ppm (t) J=7.3 Hz (1H)	HMBC 36 -> 34, 38
37	123.9 ppm	7.11 ppm (d) J=7.7 Hz (1H)	COSY 36->37 / HMBC 37 -> 40
38	137.1 ppm	-	HMBC 41 -> 38
39	125.6 ppm	7.10 ppm (br s) (1H)	HMBC 39 -> 40
40	133.4 ppm	6.60 ppm (d) J=15.7 Hz (1H)	
41	125.4 ppm	6.07 ppm (dt) J=15.7, 7.8 Hz (1H)	
42	39.6 ppm	2.88 ppm (dd) J=12.9, 8.1 Hz (1H) ; 2.51-2.47 ppm (m) (1H)	COSY 41 -> 42 / TOCSY 40 -> 42

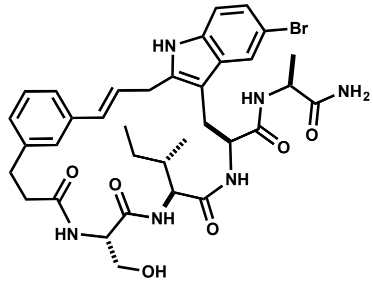


Acyclic Cinnamyl Carbonate 14: Synthesized according to Procedure A. Purified via trituration with 3x5 mL methanol. Beige Solid. ^{1}H-NMR (DMSO-d_6, 500 MHz): δ11.00 (d, J = 2.5 Hz, 1H), 7.96 (d, J = 7.8 Hz, 1H), 7.89 (d, J = 8.3 Hz, 1H), 7.84 (d, J = 7.5 Hz, 1H), 7.83 (d, J = 7.9 Hz, 1H), 7.74 (d, J = 1.8 Hz, 1H), 7.26- 7.30 (m, 1H), 7.26 (br. s, 1H), 7.23-7.24 (m, 1H), 7.21 (t, J = 7.6 Hz, 1H), 7.17 (d, J = 2.3 Hz, 1H), 7.14 (d, J = 2 Hz, 1H), 7.13 (d, J = 2 Hz, 1H), 7.1 (br. d, J = 7.5 Hz, 1H), 7.07 (br. s, 1H), 6.99 (br. s, 1H), 6.61 (d, J = 15.9 Hz, 1H), 6.32 (dt, J = 15.6, 6.2 Hz, 1H), 4.65 (dd, J = 6.3, 6.2 Hz, 2H), 4.5 (ddd, J = 9.2, 8.2, 5.0 Hz, 1H), 4.41 (apt q, J = 6.7 Hz, 1H), 4.15 (dddd, J = 7.2, 7.2, 7.2, 7.2 Hz, 1H), 4.07 (dd, J = 7.8, 6.2 Hz, 1H), 3.56 (dd, J = 10.4, 6.0 Hz, 1H), 3.51 (dd, J = 10.4, 6.3 Hz, 1H), 3.11 (dd, J = 14.9, 4.8 Hz, 1H), 2.85 (dd, J = 14.7, 9.4 Hz, 1H), 2.78 (app t, J 543 = 7.9 Hz, 2H), 2.43-2.49 (m, 3H), 1.60-2.49 (m, 1H), 1.41 (s, 9H), 1.19 (d, J = 7 Hz, 3H), 1.08- 1.16 (m, 1H), 0.90-1.00 (m, 1H), 0.65 (d, J = 6.7 Hz, 3H). ^{13}C-NMR (DMSO-d_6, 126 MHz): δ174.4, 172.1, 171.3, 171.3, 171.2, 153.3, 142.2, 136.3, 135.2, 133.9, 129.5, 129.1, 128.5, 126.9, 126.0, 124.7, 123.8, 121.2, 113.7, 111.5, 110.3, 82.0, 67.4, 62.2, 58.0, 55.0, 53.6, 48.7, 37.1, 36.7, 31.4, 27.85, 27.78, 27.6, 24.3, 18.6, 15.7, 11.7. MS m/z 841.4 (calc'd: $C_{40}H_{53}BrN_6O_9$, $[M+H]^+$, 841.1).



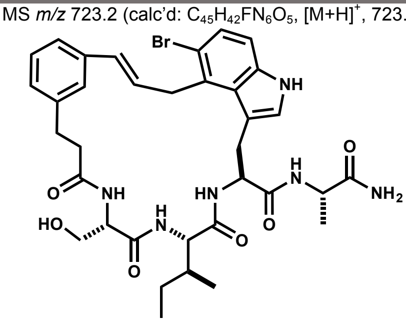
*Unidentified isomeric product

MS m/z 723.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 723.2).



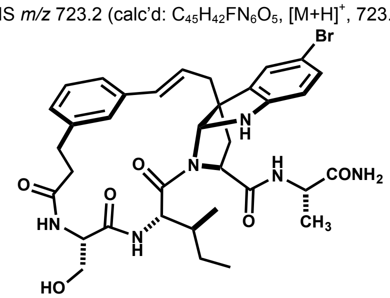
18a

MS m/z 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 723.2).



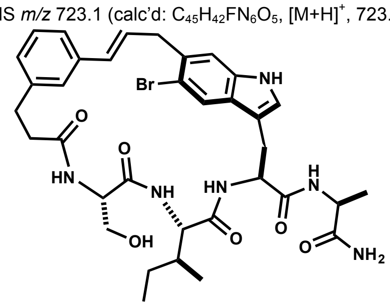
18b

MS m/z 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 723.2).



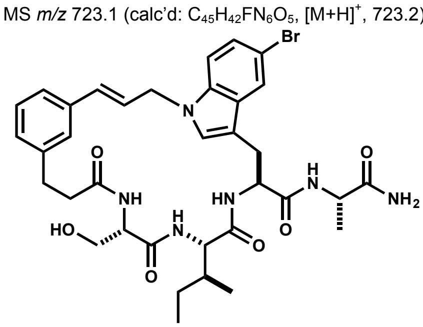
18c

MS m/z 723.1 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 723.2).



18d

MS m/z 723.1 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 723.2).



18e

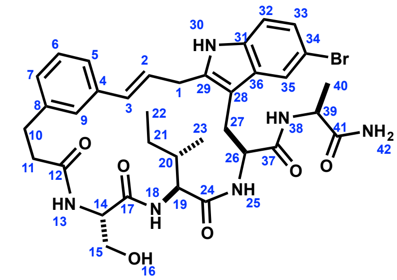
Analytical HPLC Method
Column: Waters Sunfire™ C_{18}, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	30
2.5	30
24	86
29	30

Preparative HPLC Method
Column: Waters Sunfire™ C_{18}, 19x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent A: ACN + 0.1% TFA
Flow rate: 18.0 mL/min

Time	%B
0	35
4	45
18	57
18.5	35

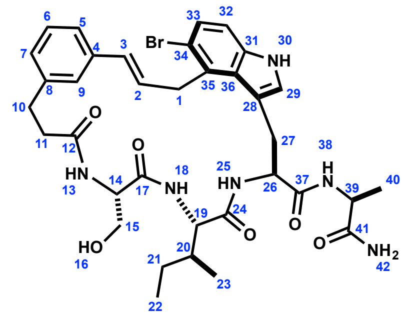
*Macrocyclic Product **18a***



(500 MHz, DMSO-d_6, 298K)

	13C	1H	key correlation
1	29.6	3.62-3.68 (m, 1H), 3.70-3.76 (m, 1H)	HMBC 1->29, 28
2	126.7	6.53-6.60 (m, 1H) overlap	COSY 2->1
3	131.4	6.53-6.60 (m, 1H) overlap	
4	136.8	-	
5	123.6	7.16-7.20 (m, 1H) overlap	
6	127.9	7.17-7.21 (m, 1H) overlap	
7	127.4	7.02-7.06 (m, 1H)	HMBC 7->5
8	141.4	-	
9	125.3	7.31 (br s, 1H) overlap	HMBC 9->3,5
10	29.9	2.68-2.75 (m, 1H), 3.02-3.10 (m, 1H) overlap	HMBC 10->7,8,9
11	35.1	2.41 (ddd, J = 14.9, 9.2, 2.2 Hz, 1H), 2.58-2.65 (m, 1H)	
12	171.5	-	
13	-	8.11 (d, J = 8.4 Hz, 1H)	
14	55.3	4.25 (ddd, J = 8.4, 5.5, 5.5 Hz, 1H)	
15	61.4	3.46-3.54 (m, 2H)	
16	-	not observed	
17	169.4	-	
18	-	7.29-7.32 (m, 1H) overlap	HMBC 18->17
19	56.6	4.06 (dd, J = 8.0, 6.6 Hz, 1H)	
20	37.3	1.60-1.69 (m, 1H)	
21	23.4	0.88-0.98 (m, 1H), 1.22-1.33 (m, 1H)	
22	10.9	0.71 (t, J = 7.4 Hz, 3H)	
23	15.0	0.68 (d, J = 6.7 Hz, 3H)	
24	170.3	-	
25	-	8.25 (d, J = 8.8 Hz, 1H)	
26	53.6	4.60 (ddd, J = 9.0, 8.8, 5.9 Hz, 1H)	HMBC 26->28
27	26.1	2.91 (dd, J = 14.4, 9.4 Hz, 1H), 3.04-3.10 (m, 1H) overlap	HMBC 27->28,29,36
28	105.9	-	
29	136.6	-	
30	-	10.94 (s, 1H)	
31	133.6	-	
32	112.3	7.17 (d, J = 8.6 Hz, 1H)	HMBC 32->36
33	122.4	7.06 (dd, J = 8.6, 1.9 Hz, 1H)	HMBC 33->31,34, TOCSY 33->32,35
34	110.6	-	
35	120.1	7.70 (d, J = 1.9 Hz, 1H)	HMBC 35->28,31,33,34
36	130.1	-	
37	170.6	-	
38	-	7.84 (d, J = 7.5 Hz, 1H)	
39	18.3	1.20 (d, J = 7.1 Hz, 3H)	
40	47.9	4.17 (dq, J = 7.1, 7.1 Hz, 1H)	
41	173.5	-	
42	-	7.00 (br s, 1H), 7.20 (br s, 1H)	TOCSY 42->42', HMBC 42->41

*Macrocyclic Product **18b***

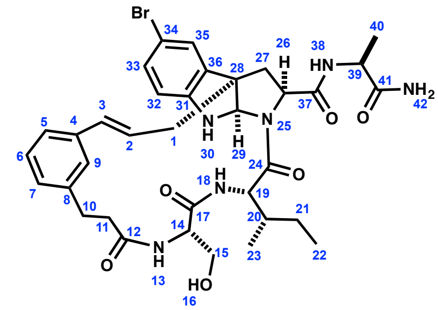


(500 MHz, DMSO-d$_6$, 298K)

*Note: This isolated compound was contaminated O-tert-butoxycarbonyl(cinnamyl alcohol 3-propionic acid)

	13C	1H	key correlation
1	34.9	3.93-3.99 (m, 1H), 4.23-4.28 (m, 1H) overlap	COSY 1→1', HMBC 1→34,35,36
2	128.4	6.45 (ddd, *J* = 16.06, 6.0, 5.5 Hz, 1H)	HMBC 2→4
3	129.8	6.20 (br d, *J* = 16.0 Hz, 1H)	HMBC 3→4
4	137	-	
5	122.9	7.00 (br d, *J* = 7.5 Hz, 1H)	HMBC 5→3
6	127.9	7.10 (dd, *J* = 7.5, 7.5 Hz, 1H)	HMBC 6→4,8, TOCSY 6→5,7,9
7	127.3	6.99 (br d, *J* = 7.5 Hz, 1H) overlap	
8	141.3	-	
9	124.6	7.22 (br s, 1H)	HMBC 9→3
10	28.9	2.68-2.74 (m, 1H) overlap, 3.01-3.05 (m, 1H) overlap	HMBC 10→7,8,9,12
11	34.3	2.48-2.53 (m, 1H) obscured, 2.66-2.72 (m, 1H) overlap	HMBC 11→9,12
12	171.5	-	
13	-	8.10 (d, *J* = 8.3 Hz, 1H)	HMBC 13→12, COSY 13→14
14	56.1	4.23-4.27 (m, 1H) overlap	HMBC 14→15,16
15	62	3.50-3.55 (m, 1H), 3.60 (ddd, *J* = 11.0, 5.7, 5.7 Hz, 1H)	HMBC 15→16
16	169.3	-	
17	-	4.90 (dd, *J* = 5.7, 5.7 Hz, 1H)	HMBC 17→14,15
18	-	7.34 (d, *J* = 8.3 Hz, 1H)	HMBC 18→16, COSY 18→19
19	56.3	4.30 (dd, *J* = 8.3, 7.4 Hz, 1H)	COSY 19→20, HMBC 19→24
20	37.1	1.65-1.72 (m, 1H)	COSY 20→21,23
21	23.9	0.98-1.06 (m, 1H), 1.39-1.48 (m, 1H)	
22	10.9	0.79 (t, *J* = 7.3 Hz, 3H)	COSY 22→21
23	14.8	0.80 (d, *J* = 6.6 Hz, 3H)	
24	170.2	-	
25	-	8.34 (d, *J* = 7.3 Hz, 1H)	HMBC 25→24, COSY 25→26
26	54.3	4.61 (ddd, *J* = 7.8, 7.8, 7.3 Hz, 1H)	HMBC 26→28, COSY 26→27
27	29.1	3.09-3.14 (m, 2H)	HMBC 27→28
28	109.6	-	
29	126.1	7.06 (d, *J* = 2.5 Hz, 1H)	HMBC 29→28,31,36
30	-	11.06 (d, *J* = 2.5 Hz, 1H)	
31	135.6	-	
32	111.8	7.18 (d, *J* = 8.6 Hz, 1H)	HMBC 32→31,34,36
33	124.6	7.26 (d, *J* = 8.6 Hz, 1H)	HMBC 33→31
34	114.9	-	
35	129.7	-	
36	126.6	-	
37	169.5	-	
38	-	7.78 (d, *J* = 7.6 Hz, 1H)	HMBC 38→37
39	18.1	4.16 (dq, *J* = 7.6, 7.0 Hz, 1H)	
40	47.8	1.14 (d, *J* = 7.0 Hz, 1H)	
41	173.3	-	
42	-	6.90 (br s, 1H), 6.91 (br s, 1H)	HMBC 42→41

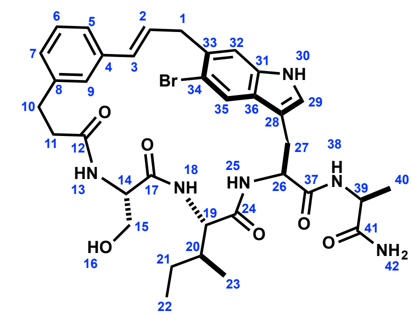
*Macrocyclic Product **18c***



(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	39.5	2.51-2.55 (m, 2H)	HMBC 1->28,29 ; NOESY 1->29
2	124.1	6.18 (ddd, J = 15.7, 8.2, 7.0 Hz, 1H)	COSY 2->1, HMBC 2->4
3	134.3	6.57 (d, J = 15.7 Hz, 1H)	
4	136.8	-	
5	124.4	7.03 (br d, J = 7.6 Hz, 1H)	HMBC 5->3, TOCSY 5->6,7,9
6	128.0	7.16 (dd, J = 7.6, 7.6 Hz, 1H)	HMBC 6->4,8
7	127.5	7.01 (br d, J = 7.6 Hz, 1H)	
8	141.4	-	
9	124.5	7.38 (br s, 1H)	HMBC 9->3
10	29.8	2.75 (apt dd, J = 14.0, 9.8 Hz, 1H), 3.02 (apt dd, J = 14.0, 10.7 Hz, 1H)	HMBC 10->7,9,12
11	35.8	2.31-2.36 (m, 1H) overlap, 2.47-2.54 (m, 1H) overlap	
12	171.3	-	
13	-	7.98 (d, J = 7.9 Hz, 1H)	HMBC 13->12
14	54.7	4.35 (ddd, J = 7.9, 7.0, 5.4 Hz, 1H)	COSY 14->13
15	61.5	3.54 (dd, J = 10.7, 7.0 Hz, 1H), 3.60 (dd, J = 10.7, 5.4 Hz, 1H)	COSY 15->14
16	-	not observed	
17	170.2	-	
18	-	8.03 (d, J = 6.1 Hz, 1H)	HMBC 18->17
19	55.6	4.23 (dd, J = 9.1, 6.1 Hz, 1H)	HMBC 19->24
20	36.4	1.71-1.77 (m, 1H) overlap	COSY 20->19
21	24.1	1.18-1.25 (m, 1H), 1.66-1.73 (m, 1H) overlap	
22	11.0	0.89 (t, J = 7.5 Hz, 3H)	COSY 22->21
23	14.8	0.99 (d, J = 6.8 Hz, 3H)	COSY 23->20
24	172.3	-	
25	-	-	
26	60.3	4.42 (dd, J = 8.7, 6.3 Hz, 1H)	COSY 26->27, HMBC 26->24 NOESY 26->29
27	38.3	2.09 (dd, J = 13.0, 6.3 Hz, 1H), 2.32-2.37 (m, 1H) overlap	HMBC 27->26,28,29,37
28	56.8	-	
29	80.8	6.35 (s, 1H)	HMBC 29->1,24,27,31,36
30	-	not observed	
31	147.8	-	
32	111.1	6.50 (d, J = 8.3 Hz, 1H)	HMBC 32->34,36
33	130.6	7.14 (dd, J = 8.3, 2.1 Hz, 1H)	HMBC 33->31
34	108.8	-	
35	124.9	7.31 (d, J = 2.1 Hz, 1H)	HMBC 35->31
36	136.7	-	
37	169.4	-	
38	-	7.33 (d, J = 7.1 Hz, 1H)	
39	47.4	3.98 (dq, J = 7.1, 6.8 Hz, 1H)	HMBC 39->41
40	17.9	0.83 (d, J = 6.8 Hz, 3H)	COSY 40->39, HMBC 40->41
41	173.3	-	
42	-	6.90 (br s, 1H), 7.38 (br s, 1H)	HMBC 42->41, TOCSY 42->42'

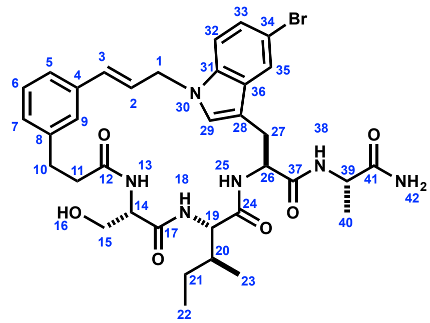
*Macrocyclic Product **18d***



(500 MHz, DMSO-d_6, 340K)

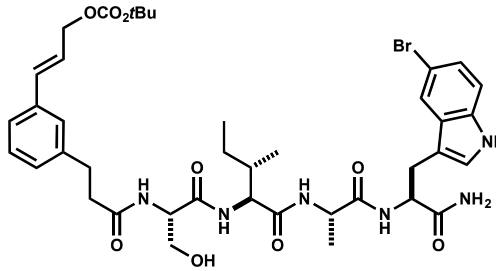
	13C	1H	key correlation
1	38.0	3.68 (apt d, J = 4.1 Hz, 2H)	
2	129.0	6.38 (ddd, J = 16.0, 5.9, 5.9 Hz, 1H)	HMBC 2->4
3	129.4	6.16 (br d, J = 16.0 Hz, 1H)	HMBC 3->5,9
4	136.8	-	
5	123.1	7.18 (d, J = 8.0 Hz, 1H) overlap	
6	127.6	7.16 (dd, J = 8.0, 8.0 Hz, 1H) overlap	HMBC 6->4,9
7	126.8	6.97 (br d, J = 8.0 Hz, 1H)	
8	140.6	-	
9	124.4	7.03 (br s, 1H)	HMBC 9->3,5,7,10
10	29.3	2.78 (ddd, J = 14.8, 7.8, 3.5 Hz, 1H), 2.83-2.89 (m, 1H) overlap	
11	34.7	2.32 (ddd, J = 14.9, 7.8, 3.5 Hz, 1H), 2.50-2.56 (m, 1H)	HMBC 11->12
12	171.4	-	
13	-	7.46 (d, J = 7.4 Hz, 1H)	
14	53.9	4.12 (apt dd, J = 11.9, 6.0 Hz, 1H)	
15	61.6	3.03 (dd, J = 10.8, 6.0 Hz, 1H), 2.84-2.89 (m, 1H) overlap	
16	-	not observed	
17	not observed	-	
18	-	7.38-7.42 (m, 1H) overlap	
19	56.8	4.03 (dd, J = 7.9, 6.4 Hz, 1H)	HMBC 19->24
20	35.8	1.72-1.80 (m, 1H)	
21	23.4	0.99-1.08 (m, 1H), 1.30-1.38 (m, 1H)	
22	10.5	0.80 (t, J = 7.4 Hz, 3H)	
23	14.8	0.84 (d, J = 6.8 Hz, 3H)	
24	170.0	-	
25	-	7.38-7.42 (m, 1H) overlap	
26	52.9	4.62 (apt dd, J = 14.4, 7.5 Hz, 1H)	HMBC 26->37
27	26.5	3.08-3.12 (m, 1H) obscured	HMBC 27->28,37
28	108.8	-	
29	125.0	7.15 (br s, 1H) overlap	HMBC 29->28,31,36
30	-	10.69 (br s, 1H)	HMBC 30->31
31	135.4	-	
32	113.0	7.32 (s, 1H)	HMBC 32->1,36
33	130.0	-	
34	113.6	-	
35	121.7	7.83 (s, 1H)	HMBC 35->28,31,33,34
36	127.4	-	
37	170.3	-	
38	-	7.60 (br s, 1H)	
39	47.6	4.29 (qd, J = 7.1, 7.0 Hz, 1H)	HMBC 39->37,41
40	17.6	1.23 (d, J = 7.1 Hz, 3H)	HMBC 40->41
41	173.4	-	
42	-	6.82 (br s, 1H), 7.06 (br s, 1H)	

Macrocyclic Product ***18e***

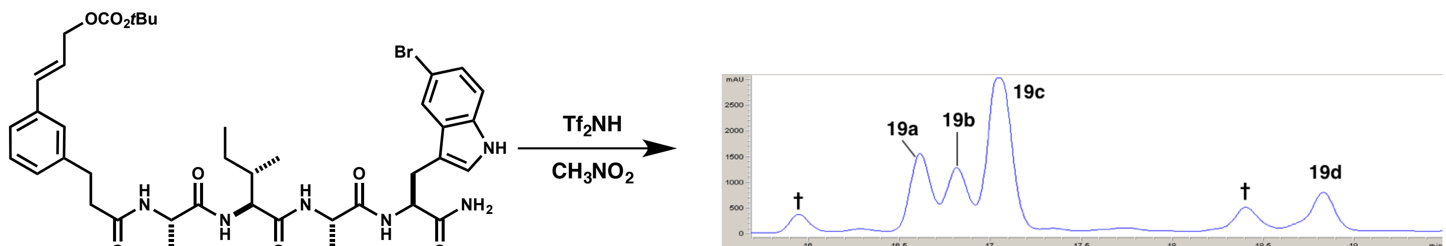


(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	47.5	4.83-4.90 (m, 2H)	HMBC 1->2,3,29,31
2	124.7	6.59-6.67 (m, 1H) overlap	HMBC 2->4
3	132.6	6.59-6.67 (m, 1H) overlap	
4	136.2	-	
5	124.5	7.16-7.20 (m, 1H) overlap	HMBC 5->3
6	127.9	7.20 (dd, J = 7.5, 7.3 Hz, 1H) overlap	HMBC 6->4,8
7	128.1	7.04 (ddd, J = 7.3, 1.5, 1.5 Hz, 1H)	
8	141.5	-	
9	124.7	7.41 (br s, 1H)	HMBC 9->3, TOCSY 9->5,6,7
10	29.6	2.70-2.76 (m, 1H), 2.94-3.00 (m, 1H) overlap	HMBC 10->7,8,9
11	34.7	2.42 (ddd, J = 14.5, 8.5, 2.7 Hz, 1H), 2.46-2.51 (m, 1H) obscured	HMBC 11->8
12	171.4	-	
13	-	7.87 (d, J = 7.4 Hz, 1H)	
14	54.5	4.22-4.27 (m, 1H) overlap	HMBC 14->17
15	61.4	3.34-3.40 (m, 2H) obscured	HMBC 15->17
16	-	not observed	
17	170.0	-	HMBC 18->17
18	-	7.70 (d, J = 8.0 Hz, 1H)	HMBC 19->24
19	57.1	4.01 (dd, J = 8.0, 7.1 Hz, 1H)	
20	35.9	1.60-1.68 (m, 1H)	
21	23.6	0.95-1.02 (m, 1H), 1.27-1.34 (m, 1H)	
22	10.7	0.73 (dd, J = 7.6, 7.6 Hz, 3H) overlap	
23	15.0	0.72 (d, J = 7.1 Hz, 3H) overlap	
24	171.2	-	
25	-	7.98 (d, J = 8.2 Hz, 1H)	HMBC 25->24,27
26	52.2	4.56 (ddd, J = 8.6, 8.2, 4.8 Hz, 1H)	HMBC 26->37, COSY 26->25,27
27	26.5	2.99-3.94 (m, 2H) overlap	
28	109.2	-	
29	128.6	7.35 (s, 1H)	HMBC 29->1,27,31,36
30	-	-	
31	134.5	-	
32	111.4	7.44 (d, J = 8.7 Hz, 1H)	HMBC 32->34,36
33	123.1	7.21 (dd, J = 8.7, 1.9 Hz, 1H)	HMBC 33->31,35
34	111.1	-	
35	120.9	7.78 (d, J = 1.9 Hz, 1H)	HMBC 35->31,33
36	129.3	-	
37	170.9	-	
38	-	7.94 (d, J = 7.3 Hz, 1H)	HMBC 38->37
39	47.8	4.21 (dq, J = 7.3, 7.2 Hz, 1H)	HMBC 39->41
40	18.1	1.17 (d, J = 7.2 Hz, 1H)	HMBC 40->39
41	173.8	-	
42	-	7.02 (br s, 1H), 7.32 (br s, 1H)	

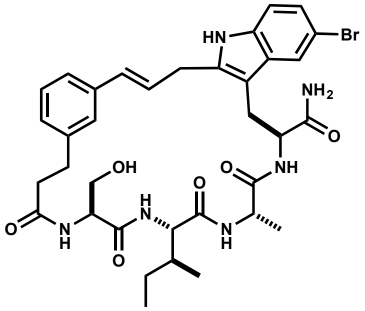


Acyclic Cinnamyl Carbonate 15: Synthesized according to Procedure A. Purified via trituration with 3x5 mL methanol. Beige solid. ^{1}H-NMR (DMSO-d_6, 500 MHz): δ10.99 (d, *J* = 1.7 Hz, 1H), 7.99 (d, *J* = 6.7 Hz, 1H), 7.98 (d, *J* = 7.5 Hz, 1H), 7.76 (d, *J* = 8.2 Hz, 1H), 7.75 (d, *J* = 8.3 Hz, 1H), 7.74 (d, *J* = 1.7 Hz, 1H), 7.32 (br s, 1H), 7.28 (br s, 1H), 7.26 (d, *J* = 8.5 Hz, 1H), 7.25 (d, *J* = 7.2 Hz, 1H), 7.21 (dd, *J* = 7.5, 7.5 Hz, 1H), 7.17 (d, *J* = 1.9 Hz, 1H), 7.12 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.10 (d, *J* = 7.3 Hz, 1H), 7.03 (br s, 1H), 6.61 (d, *J* = 16.0 Hz, 1H), 6.31 (ddd, *J* = 15.9, 6.2, 6.2 Hz, 1H), 5.00 (dd, *J* = 5.3, 5.3 Hz, 1H), 4.64 (d, *J* = 6.0 Hz, 1H), 4.39-4.34 (m, 1H), 4.22 (pentet, *J* = 7.1 Hz, 1H), 4.16 (dd, *J* = 7.9, 6.7 Hz, 1H), 3.55-3.46 (m, 2H), 3.04 (dd, *J* = 14.6, 5.7 Hz, 1H), 2.91 (dd, *J* = 14.7, 7.8 Hz, 1H), 2.78 (dd, *J* = 7.8, 7.8 Hz, 1H), 2.45 (dd, *J* = 8.6, 7.3 Hz, 1H), 1.74-1.69 (m, 1H), 1.40 (s, 9H), 1.38-1.33 (m, 1H), 1.14 (d, *J* = 7.0 Hz, 3H), 1.07-7.01 (m, 1H), 0.77 (d, *J* = 6.9 Hz, 3H), 0.76 (dd, *J* = 7.9, 7.9 Hz, 3H).). ^{13}C-NMR (DMSO-d_6, 126 MHz): δ172.9, 171.8, 171.6, 170.6, 170.4, 152.8, 141.7, 135.9, 134.7, 133.5, 129.2, 128.6, 128.0, 126.4, 125.4, 124.2, 123.3, 123.3, 120.8, 113.3, 111.0, 109.9, 81.5, 66.9, 61.7, 57.0, 54.7, 53.6, 53.2, 48.4, 36.6, 31.0, 27.4, 25.2, 24.1, 18.1, 17.8, 15.3, 11.3. MS *m/z*, 841.3 (calc'd: $C_{35}H_{44}BrN_6O_6$ [M+H]$^+$, 841.1)



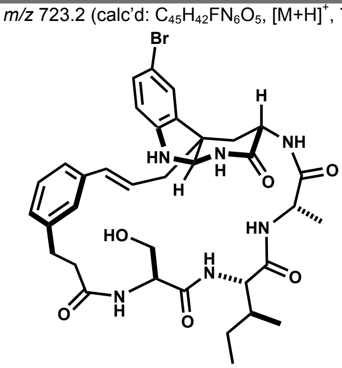
†Non-isomeric products

MS *m/z* 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



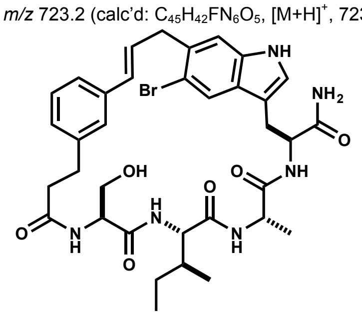
19a

MS *m/z* 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



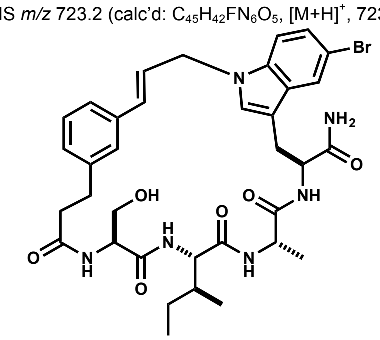
19b

MS *m/z* 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



19c

MS *m/z* 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, [M+H]$^+$, 723.2).



19d

Analytical HPLC Method
Column: Waters Sunfire™ C_{18}, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	30
2.5	30
24	86
29	30

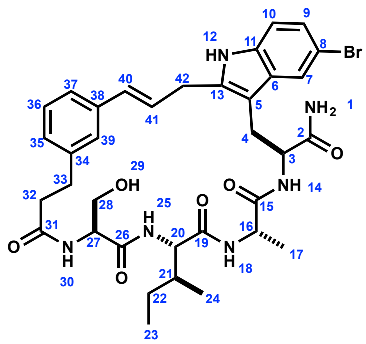
Preparative HPLC Method
Column: Waters Sunfire™ C_{18}, 19x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent A: ACN + 0.1% TFA
Flow rate: 18.0 mL/min

Time	%B
0	45
2	45
12	50
13	50

Semi-Prep HPLC Method
Column: Waters XSelect™ C_{18}, 10x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 6.00 mL/min
For re-purification of *19b*

Time	%B
0	38
1	38
20	43
21	38

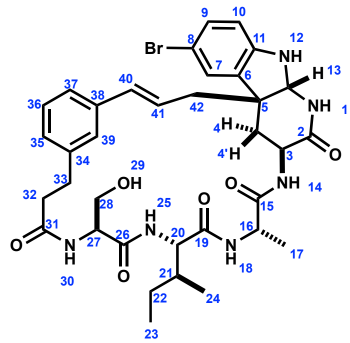
*Macrocyclic Isomer **19a***



(600 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	-	7.29 ppm (br s) (1H) ; 6.98 ppm (br s)	TOCSY 1 -> 1'
2	173.0 ppm	-	HMBC 1(slight),3 -> 2
3	53.4 ppm	4.40 ppm (ddd) J=7.5, 7.5, 7.5 Hz (1H)	COSY/TOCSY 14 -> 3
4	26.9 ppm	3.05 ppm (dd) J=14.7, 7.5 Hz (1H) ; 2.92 ppm (dd) J=14.5, 6.8 Hz (1H)	COSY/TOCSY 3 -> 4
5	105.8 ppm	-	HMBC 4,7,12,42(slight) -> 5
6	130.0 ppm	-	HMBC 10,12 -> 6
7	120.0 ppm	7.71 ppm (d) J=1.7 Hz (1H)	HMBC 7 -> 5,11
8	110.8 ppm	-	HMBC 7,10 -> 8
9	122.3 ppm	7.08 ppm (dd) J=8.5, 1.9 Hz (1H)	HMBC 7 -> 9 / 9 -> 7
10	112.3 ppm	7.19 ppm (d) J=8.2 Hz (1H)	COSY 9 -> 10 ; HMBC 10 -> 6,8
11	133.7 ppm	-	HMBC 7,9,12 -> 11
12	-	10.94 ppm (s)	indole
13	136.7 ppm	-	HMBC 4,12,41,42 -> 13
14	-	7.69 ppm (d) J=8.3 Hz	HMBC 14 -> 15
15	171.8 ppm	-	HMBC 16 -> 15
16	47.5 ppm	4.39-4.33 ppm (m) (1H)	COSY/TOCSY 18 -> 16
17	17.4 ppm	1.22 ppm (d) J=7.1 Hz (1H)	COSY/TOCSY 16 -> 17
18	-	8.01 ppm (d) J=7.7 Hz (1H)	HMBC 18 -> 19
19	169.9 ppm	-	HMBC 20 -> 19
20	56.6 ppm	4.14 ppm (d) J=8.0, 5.8 Hz (1H)	COSY/TOCSY 25 -> 20
21	36.8 ppm	1.70-1.66 ppm (m) (1H)	TOCSY 20 -> 21
22	23.7 ppm	1.33-1.28 ppm (m) (1H) ; 1.05-0.98 ppm (m) (1H)	TOCSY 21 -> 22
23	11.1 ppm	0.70 ppm (dd) J=7.4 Hz (3H)	COSY/TOCSY 22 -> 23
24	15.0 ppm	0.76 ppm (d) J=6.8 Hz (3H)	COSY/TOCSY 21 -> 24
25	-	7.49 ppm (d) J=7.9 Hz (1H)	HMBC 25 -> 26
26	170.2 ppm	-	HMBC 27 -> 26
27	55.0 ppm	4.36-4.32 ppm (m) (1H)	COSY/TOCSY 30 -> 27
28	61.3 ppm	3.50-3.45 ppm (m) (2H)	COSY/TOCSY 27 -> 28
29	-	not observed	-
30	-	8.15 ppm (d) J=8.0 Hz (1H)	HMBC 30 -> 31
31	172.0 ppm	-	HMBC 32, 33 -> 31
32	34.6 ppm	2.67 ppm (ddd) J=14.9, 8.4, 6.6 Hz (1H) ; 2.40 ppm (ddd) J=14.5, 6.4, 6.4 Hz (1H)	HMBC 32 -> 34 ; COSY 33 -> 32
33	30.0 ppm	2.89-2.80 ppm (m) (2H)	HMBC 33 -> 34
34	141.4 ppm	-	HMBC 32,33,36 -> 34
35	126.9 ppm	7.03 ppm (d) J=7.4 Hz (1H)	COSY/TOCSY 36 -> 35
36	128.2 ppm	7.20 ppm (dd) J=7.8, 7.8 Hz (1H)	COSY/TOCSY 36 -> 37
37	123.9 ppm	7.16 ppm (d) J=7.6 Hz (1H)	HMBC 37 -> 40 / 40 -> 37
38	136.6 ppm	-	HMBC 36,41 -> 38
39	125.0 ppm	7.37 ppm (s) (1H)	TOCSY 39 -> 35,36,37
40	130.7 ppm	6.54 ppm (d) J=15.7 Hz (1H)	
41	126.6 ppm	6.37 ppm (ddd) J=15.8, 6.8, 6.8 Hz (1H)	
42	29.3 ppm	3.76 ppm (dd) J=16.0, 6.1 Hz (1H) ; 3.62-3.58 ppm (m) (1H)	COSY/HMBC 41 -> 42

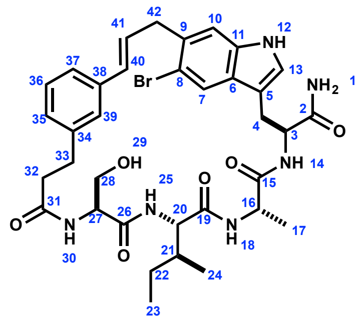
Macrocyclic Product ***19b***



(600 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	-	7.99 ppm (d) J=3.0 Hz (1H)	COSY 12 -> 13 ; HMBC 13 -> 1; HMBC 1 -> 3,5
2	170.4 ppm	-	HMBC 3,13 -> 2
3	46.0 ppm	4.11 ppm (ddd) J=12.9, 8.2, 4.6 Hz (1H)	COSY/TOCSY 14 -> 3 ; HMBC 3 -> 2
4	35.2 ppm	2.09 ppm (dd) J=13.1, 4.4 Hz (1H) ; 1.99 ppm (dd) J=13.1, 13.1 Hz (1H)	COSY/TOCSY 3 -> 4
5	47.7 ppm	-	HMBC 4,7,12,42 -> 5
6	134.6 ppm	-	HMBC 4,10,42 -> 6
7	125.8 ppm	7.16 ppm (s) (1H)	HMBC 7 -> 5
8	108.3 ppm	-	HMBC 7,10 -> 8
9	130.8 ppm	7.19-7.16 ppm (m) (1H)	COSY 9 -> 10 ; HMBC 9 -> 7
10	110.5 ppm	6.56 ppm (d) J=8.0 Hz (1H)	HMBC 10 -> 6,8
11	148.6 ppm	-	HMBC 7,12,13 -> 11
12	-	not observed	
13	73.2 ppm	4.93 ppm (d) J=3.2 Hz (1H)	HMBC 13 -> 2,4 / 13 -> 4,42 ; NOESY 13 -> 4'
14	-	7.19-7.16 ppm (m) (1H)	HMBC 14 -> 15
15	171.0 ppm	-	HMBC 16 -> 15
16	47.5 ppm	4.29 ppm (pentet) J=7.4 Hz (1H)	COSY/TOCSY 18 -> 16
17	17.5 ppm	1.11 ppm (d) J=7.1 Hz (3H)	COSY/TOCSY 16 -> 17
18	-	7.77 ppm (d) J=8.4 Hz (1H)	HMBC 18 -> 19
19	169.7 ppm	-	HMBC 20 -> 19
20	58.2 ppm	4.05 ppm (dd) J=6.9, 4.6 Hz (1H)	COSY/TOCSY 25 -> 20
21	35.6 ppm	1.88-1.84 (m) (1H)	TOCSY 20 -> 21
22	23.8 ppm	1.32-1.27 ppm (m) (1H) ; 1.23-1.17 ppm (m) (1H)	TOCSY 21 -> 22
23	11.7 ppm	0.81 ppm (dd) J=7.4, 7.4 Hz (3H)	COSY/TOCSY 22 -> 23
24	15.4 ppm	0.84 ppm (d) J=7.0 Hz (3H)	COSY/TOCSY 21 -> 24
25	-	8.19 ppm (d) J=6.7 Hz (1H)	HMBC 25 -> 26
26	171.5 ppm	-	HMBC 27 -> 26
27	53.5 ppm	4.54 ppm (ddd) J=7.8, 7.8, 6.0 Hz (1H)	COSY 27 -> 28
28	61.7 ppm	3.43 ppm (dd) J=9.3, 5.6 Hz (1H) ; 3.14 ppm (dd) J=9.2, 9.2 Hz (1H)	COSY/TOCSY 27 -> 28
29	-	not observed	-
30	-	7.91 ppm (d) J=7.4 Hz (1H)	COSY 30 -> 27
31	171.7 ppm	-	HMBC 30 -> 31
32	34.5 ppm	2.81-2.76 ppm (m) (1H) ; 2.37-2.33 ppm (m) (1H)	COSY 33 -> 32
33	30.1 ppm	2.94-2.89 ppm (m) (1H) ; 2.85-2.81 ppm (m) (1H)	HMBC 35,39 -> 33
34	140.9 ppm	-	HMBC 36 -> 34
35	128.2 ppm	7.04 ppm (d) J=7.4 Hz (1H)	HMBC 35 -> 33
36	128.4 ppm	7.21 ppm (dd) J= 7.6 Hz (1H)	HMBC 36 -> 38
37	124.0 ppm	7.18-7.16 ppm (m) (1H)	TOCSY 35,39 -> 37 ; COSY 36 -> 37
38	136.6 ppm	-	HMBC 41 -> 38
39	125.1 ppm	7.32 ppm (br s) (1H)	TOCSY 39 -> 35
40	134.5 ppm	6.42 ppm (d) J=15.8 Hz	
41	124.1 ppm	6.36 ppm (ddd) J=15.4, 7.2, 7.2 Hz (1H)	
42	43.2 ppm	2.46-2.44 ppm (m) (2H)	COSY 41 -> 42

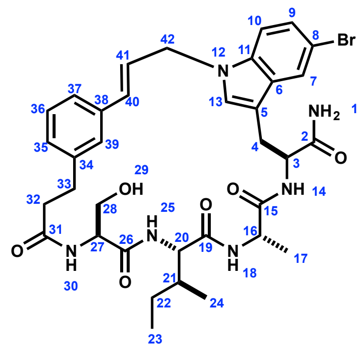
Macrocyclic Product ***19c***



(600 MHz, DMSO-d$_6$, 298K)

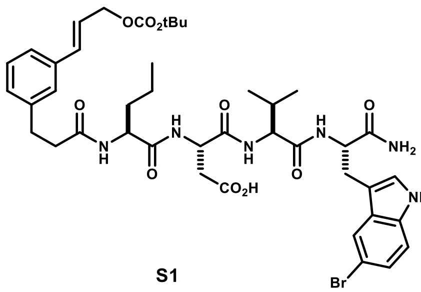
	13C	1H	key correlation
1	-	7.55 ppm (br s) (1H) ; 7.09 ppm (br s) (1H)	TOCSY 1 -> 1'
2	173.6 ppm	-	HMBC 1 -> 2
3	53.2 ppm	4.41 ppm (ddd) J=11.1, 7.9, 3.3 Hz (1H)	COSY 14 -> 3
4	27.2 ppm	3.07 ppm (dd) J=14.6, 3.1 Hz (1H) ; 2.87 ppm (dd) J=14.6, 11.1 Hz (1H)	COSY/TOCSY 3 -> 4
5	109.5 ppm	-	HMBC 4,7,12,13 -> 5
6	127.6 ppm	-	HMBC 10,12,13 -> 6
7	121.7 ppm	7.96 ppm (s) (1H)	HMBC 7 -> 5,8,9,11
8	113.8 ppm	-	HMBC 7,10 -> 8
9	130.5 ppm	-	HMBC 7,42 -> 9
10	113.2	7.35 ppm (s) (1H)	HMBC 42 -> 10 / 10 -> 42
11	135.5 ppm	-	HMBC 7,12,13 -> 11
12	-	10.82 ppm (d) J=1.9 Hz (1H)	indole
13	125.1 ppm	7.17 ppm (d) J=2.2 Hz (1H)	COSY 12 -> 13
14	-	7.72 ppm (d) J= 8.0 Hz (1H)	HMBC 14 -> 15
15	171.6 ppm	-	HMBC 16 -> 15
16	47.5 ppm	4.15 ppm (pentet) J=7.0 Hz (3H)	COSY/TOCSY 18 -> 16
17	17.8 ppm	1.17 ppm (d) J=7.0 Hz (1H)	COSY/TOCSY 16 -> 17
18	-	7.60 ppm (d) J=7.0 Hz (1H)	HMBC 18 -> 19
19	169.4 ppm	-	HMBC 20 -> 19
20	56.3 ppm	4.05 ppm (dd) J=8.4, 5.7 Hz (1H)	COSY/TOCSY 25 -> 20
21	36.6 ppm	1.64-1.59 ppm (m) (1H)	TOCSY 20 -> 21
22	23.5 ppm	1.22-1.17 ppm (m) (1H) ; 0.94-0.89 ppm (m) (1H)	COSY/TOCSY 21 -> 22
23	10.9 ppm	0.67 ppm (dd) J=7.4, 7.4 Hz (3H)	COSY/TOCSY 22 -> 23
24	14.9 ppm	0.67 ppm (d) J=6.8 Hz (3H)	COSY/TOCSY 21 -> 24
25	-	7.41 ppm (d) J=8.5 Hz (1H)	HMBC 25 -> 26
26	169.5 ppm	-	HMBC 27 -> 26
27	54.1 ppm	4.24 ppm (ddd) J=8.3, 6.1, 6.0 Hz (1H)	COSY 27 -> 28
28	61.2 ppm	3.44-3.37 ppm (m) (1H)	COSY/TOCSY 27 -> 28
29	-	4.83 ppm (dd) J=5.3, 5.3 Hz (1H)	COSY/TOCSY 29 -> 27,28
30	-	8.03 ppm (d) J=8.3 Hz (1H)	HMBC 30 -> 31
31	171.2 ppm	-	HMBC 32,33 -> 31
32	35.7 ppm	2.52-2.48 ppm (m) (1H) ; 2.38-2.33 ppm (m) (1H)	COSY 33 -> 32
33	30.0 ppm	2.76-2.72 ppm (m) (2H)	HMBC 39 -> 33
34	141.2 ppm	-	HMBC 32,33,35,36 -> 34
35	128.1 ppm	7.19 ppm (d) J=4.8 Hz (1H)	HMBC 36 -> 35
36	126.7 ppm	7.04-7.01 ppm (m) (1H)	COSY 37 -> 36 ; TOCSY 39 -> 36
37	123.4 ppm	7.19 ppm (d) J=3.7 Hz (1H)	HMBC 39,40 -> 37
38	136.9 ppm	-	HMBC 36,37,40,41 -> 38
39	125.0 ppm	7.09 ppm (s) (1H)	HMBC 40 -> 39
40	129.9 ppm	6.26 ppm (d) J=15.9 Hz (1H)	
41	129.0 ppm	6.37 ppm (ddd) J=15.8, 6.2, 6.2 Hz (1H)	
42	38.3 ppm	3.72 ppm (dd) J=16.1, 6.2 Hz (1H) ; 3.65 ppm (dd) J=16.0, 5.8 Hz (1H)	COSY 41 -> 42

*Macrocyclic Product **19d***



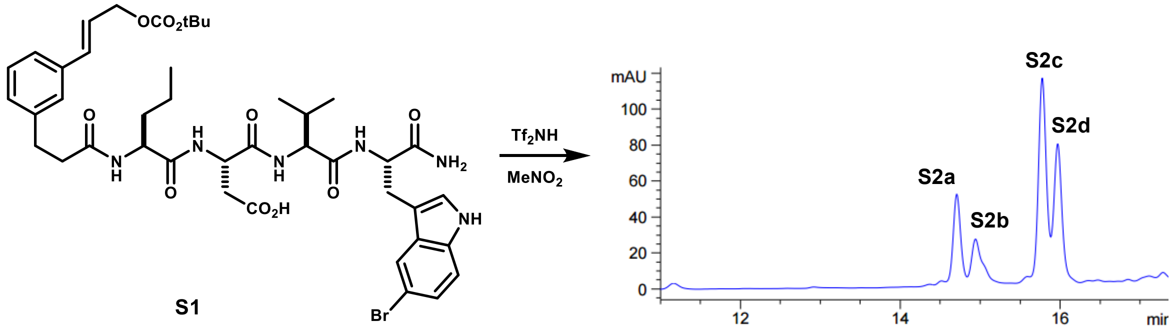
(600 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	-	7.41 ppm (br s) (1H) ; 7.08 ppm (br s) (1H)	TOCSY 1 -> 1'
2	173.2 ppm	-	HMBC 1 -> 2
3	52.7 ppm	4.38 ppm (ddd) J=10.3, 7.5, 2.9 Hz (1H)	COSY 14 -> 3
4	26.8 ppm	3.09 ppm (dd) J=15.0, 2.9 Hz (1H) ; 3.00-2.95 ppm (m) (1H)	COSY/TOCSY 3 -> 4
5	109.7 ppm	-	HMBC 4,7,13 -> 5
6	129.3 ppm	-	HMBC 10,13 -> 6
7	120.7 ppm	7.82 ppm (d) J=1.7 Hz (1H)	
8	111.1 ppm	-	HMBC 7,10 -> 8
9	123.2	7.24-7.21 ppm (m) (1H)	HMBC 7 -> 10 ; COSY 7 -> 9
10	111.5 ppm	7.45 ppm (d) J=8.6 Hz (1H)	COSY 10 -> 9 ; TOCSY 7 -> 10
11	134.4 ppm	-	HMBC 7,13, 42 -> 11
12	-	-	-
13	128.0 ppm	7.28 ppm (s) (1H)	HMBC 42 -> 13
14	-	7.76 ppm (d) J=7.7 Hz (1H)	HMBC 14 -> 15
15	172.0 ppm	-	HMBC 16 -> 15
16	17.3 ppm	1.21 ppm (d) J=7.2 Hz (3H)	COSY/TOCSY 17 -> 16
17	47.6 ppm	4.29 ppm (pentet) J=7.3 Hz (1H)	COSY/TOCSY 18 -> 17
18	-	8.00 ppm (d) J=7.4 Hz (1H)	HMBC 18 -> 19
19	170.3 ppm	-	HMBC 20 -> 19
20	56.7 ppm	4.13 ppm (dd) J=7.5, 6.4 Hz (1H)	COSY/TOCSY 25 -> 20
21	36.5 ppm	1.71-1.67 ppm (m) (1H)	COSY 20 -> 21
22	23.9 ppm	1.38-1.34 ppm (m) (1H) ; 1.09-1.04 ppm (m) (1H)	COSY/TOCSY 21 -> 22
23	10.9 ppm	0.75 ppm (dd) J=7.4 Hz (3H)	COSY/TOCSY 22 -> 23
24	14.9 ppm	0.79 ppm (d) J=6.8 Hz (3H)	COSY/TOCSY 21 -> 24
25	-	7.46 ppm (d) J=7.8 Hz (1H)	HMBC 25 -> 26
26	170.2 ppm	-	HMBC 27 -> 26
27	55.4 ppm	4.23 ppm (ddd) J=7.5, 5.9, 5.9 Hz (1H)	COSY 27 -> 28
28	61.0 ppm	3.57-3.50 ppm (m) (2H)	COSY/TOCSY 27 -> 28
29	-	not observed	-
30	-	8.13 ppm (d) J=7.6 Hz (1H)	HMBC 30 -> 31
31	172.3 ppm	-	HMBC 32,33 -> 31
32	35.5 ppm	2.53-2.50 ppm (m) (1H) ; 2.47-2.44 ppm (m) (1H)	COSY 33 -> 32
33	30.1 ppm	2.87-2.84 ppm (m) (1H) ; 2.82-2.77 ppm (m) (1H)	HMBC 33 -> 35,39
34	141.5 ppm	-	HMBC 32,33,36 -> 34
35	127.5 ppm	7.09 ppm (d) J=7.1 Hz (1H)	HMBC 37 -> 35
36	128.2 ppm	7.24-7.21 ppm (m) (1H)	COSY 37 -> 36, 37
37	124.4 ppm	7.18 ppm (d) J=7.5 Hz (1H)	HMBC 40 -> 37
38	135.9 ppm	-	HMBC 36,41 -> 38
39	124.8 ppm	7.33 ppm (s) (1H)	HMBC 40 -> 39
40	132.3 ppm	6.66 ppm (d) J=15.8 Hz (1H)	
41	124.5 ppm	6.44 ppm (ddd) J=15.8, 6.3, 6.3 Hz (1H)	
42	47.1 ppm	4.93 ppm (dd) J=15.9, 6.0 Hz (1H) ; 4.83 ppm (dd) J=15.9, 6.2 Hz (1H)	COSY 41 -> 42

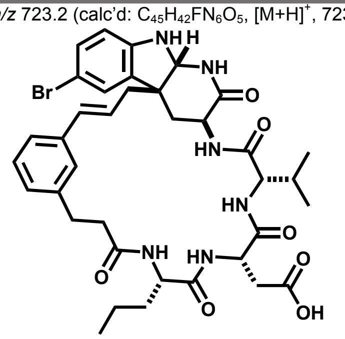


S1

Acyclic Cinnamyl Carbonate S1: Synthesized according to General Procedure A. The reaction was filtered, concentrated, and the residue partitioned between EtOAc and H_2O. The resulting solids were collected by filtration to give **S1** (546mg, 62%) as an off-white solid. ^1H NMR (DMSO-d_6, 600 MHz): δ 0.69 (d, J = 6.4 Hz, 3H), 0.72 (d, J = 6.4 Hz, 3H), 0.77 (t , J = 7.3 Hz, 3H), 1.35-1.47 (m, 2H), 1.43 (s, 9H), 1.47-1.58 (m, 1H), 1.89-1.99 (m, 1H), 2.37-2.46 (m, 1H), 2.46-2.55 (m, 1H), 2.68 (dd, J = 16.4, 5.7 Hz, 1H), 2.75-2.85 (m , 2H), 2.88-2.97 (m, 2H), 3.08 (dd, J = 14.3, 4.6 Hz, 1H), 3.43 (br s, 1H), 4.04-4.10 (m, 1H), 4.22-4.29 (m, 1H), 4.39-4.49 (m, 1H), 4.53-4.59 (m, 1H), 4.67 (d, J = 6.0 Hz, 2H), 6.33 (dt, J = 15.6, 6.0 Hz, 1H), 6.63 (d, J = 15.6 Hz, 1H), 7.04 (s, 1H), 7.11 (d, J = 6.8 Hz, 1H), 7.15 (d, J = 8.1 Hz, 1H), 7.19 (s, 1H), 7.21-7.35 (m, 5H), 7.53 (br d, J = 8.3 Hz, 1H), 7.77 (br s, 1H), 7.92-7.98 (m, 1H), 8.01 (d, J = 7.7 Hz, 1H), 8.35 (d, J = 7.2 Hz, 1H), 11.02 (s, 1H), 11.92 (br s, 1H). ^{13}C NMR ($CDCl_3$, 150 MHz): δ 173.2, 172.3, 172.1,171.5 170.7, 170.4, 162.3, 152.8, 141.7, 135.9, 134.7, 133.5, 129.2, 128.6, 128.1, 126.4, 125.4, 124.2, 123.3, 120.7, 113.3, 111.0, 110.1, 81.5, 66.9, 58.0, 53.3, 52.2, 49.7, 36.6, 35.8, 34.3, 30.9, 30.3, 27.4, 19.0, 18.4, 18.0, 17.5, 13.6. MS m/z 883.2/885.2 (calc'd: $C_{42}H_{56}BrN_6O_{10}$ $[M+H]^+$, 883.3).

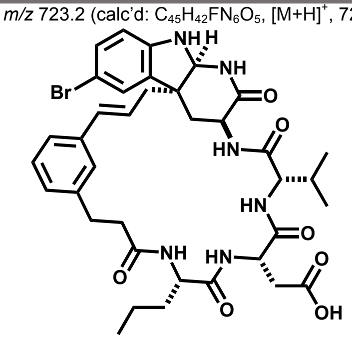


MS m/z 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 723.2).



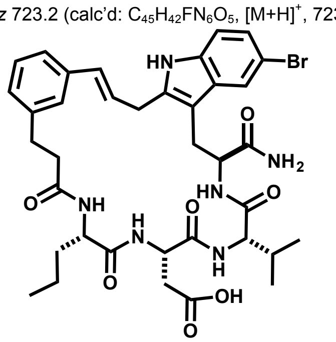
S2a

MS m/z 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 723.2).



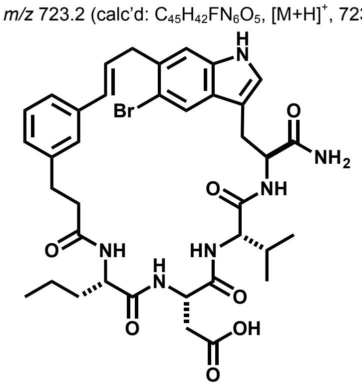
S2b

MS m/z 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 723.2).



S2c

MS m/z 723.2 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 723.2).



S2d

Analytical HPLC Method
Column: Waters X-Select™ PFP, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	10
3	10
23	70
25	10
30	10

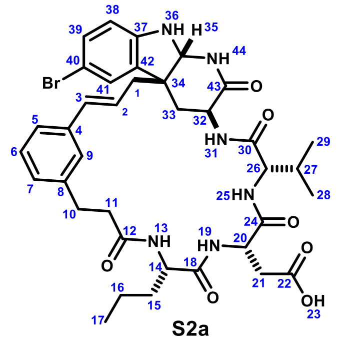
Prep HPLC Method A
Column: Waters X-Select™ PFP, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 18.00 mL/min

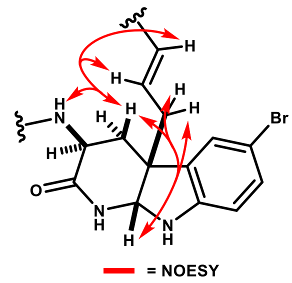
Time	%B
0	40
3	40
23	85

Prep HPLC Method B
Column: Waters X-Select™ PFP, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% HCO_2H
Solvent B: ACN + 0.1% HCO_2H
Flow rate: 18.00 mL/min

Time	%B
0	40
3	40
23	85

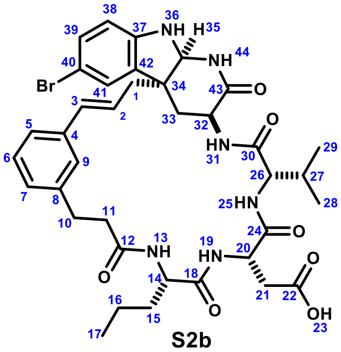
*Macrocyclic Product **S2a** (600 MHz, DMSO-d$_6$, 298K)*



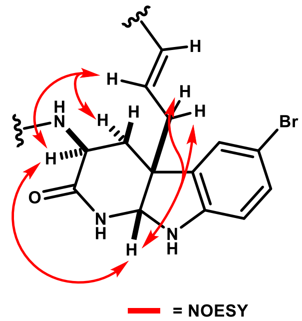


	13C	1H	key correlations
1	41.6	2.38 (dd, *J* = 13.4, 6.6 Hz, 1H), 2.46 (dd, *J* = 13.4, 5.7 Hz, 1H)	HMBC 1→35 NOESY 1→35
2	124.2	6.24-6.28 (m, 1H)	HMBC 2→4 NOESY 2→33
3	134.2	6.24-6.28 (m, 1H)	HMBC 3→1 NOESY 3→33
4	136.7	-	
5	124.1	7.04 (br d, *J* = 7.6 Hz, 1H)	HMBC 5→3
6	127.7	7.15 (dd, *J* = 7.6, 7.6 Hz, 1H)	HMBC 6→4,8
7	128	7.00 (br d, *J* = 7.6 Hz, 1H)	
8	140.1	-	
9	125.5	7.50-7.53 (m, 1H)	HMBC 9→3
10	30.4	2.83-2.88 (m, 2H)	HMBC 10→8,12
11	34.2	2.33-2.38 (m, 1H), 2.90-2.94 (m, 1H)	HMBC 11→8,12
12	170.4	-	
13	-	7.70 (br d, *J* = 8.6 Hz, 1H)	HMBC 13→12
14	50.6	4.26-4.31 (m, 1H)	
15	35.6	1.10-1.17 (m, 1H), 1.50-1.56 (m, 1H)	
16	17.5	0.51-0.57 (m, 1H), 0.63-0.69 (m, 1H)	
17	13.5	0.61-0.65 (m, 3H)	
18	172.1	-	
19	-	8.42 (br d, *J* = 5.7 Hz, 1H)	HMBC 19→18
20	52	4.25 (ddd, *J* = 8.2, 5.7, 5.7 Hz, 1H)	COSY 20→21
21	35.9	2.54-2.59 (m, 2H)	HMBC 21→22
22	171.4	-	
23	-	not detected	
24	170.6	-	
25	-	6.97 (br d, *J* = 8.0 Hz, 1H)	
26	56.9	4.08 (dd, *J* = 8.0, 6.4 Hz, 1H)	TOCSY 26→25,27,28,29 HMBC 26→30
27	30.7	1.89-1.96 (m, 1H)	
28	18.6	0.78 (d, *J* = 6.7 Hz, 3H)	
29	17.9	0.79 (d, *J* = 6.7 Hz, 3H)	
30	169.5	-	
31	-	7.92 (br d, *J* = 7.3 Hz, 1H)	HMBC 31→30 TOCSY 31→32,33 NOESY 31→33
32	46.1	4.00 (ddd, *J* = 12.5, 7.3, 5.0 Hz, 1H)	HMBC 32→30,43
33	33.3	*pro*-S 1.87 (dd, *J* = 13.1, 12.5 Hz, 1H)	
33'		*pro*-R 2.33 (dd, *J* = 13.1, 4.7 Hz, 1H)	
34	47.4	-	
35	74.9	4.82 (d, *J* = 2.3 H, 1H)	HMBC 35→1,34,37,43 COSY 35→36,44
36	-	6.31 (br s, 1H)	HMBC 36→34,37,42
37	148.3	-	
38	110.8	6.62 (d, *J* = 8.3 Hz, 1H)	HMBC 38→40,42
39	130.6	7.18 (dd, *J* = 8.3, 2.0 Hz, 1H)	HMBC 39→37,40,41
40	108	-	
41	126.3	7.05 (d, *J* = 2.0 Hz, 1H)	HMBC 41→34,37,40
42	132.4	-	
43	169.3	-	
44	-	7.96 (d, *J* = 2.3 Hz, 1H)	HMBC 44→32,34

Macrocyclic Product ***S2b*** *(600 MHz, DMSO-d_6, 298K)*

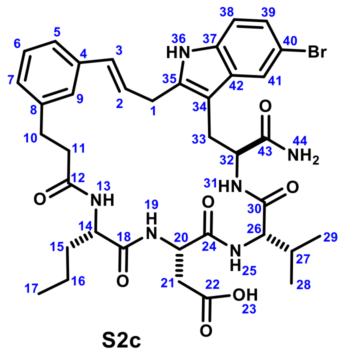


S2b



	13C	1H	key correlations
1	40.7	2.42 (dd, *J* = 13.7, 7.0 Hz, 1H), 2.47-2.52 (m, 1H)	HMBC 1→34 NOESY 1→35,32
2	124.7	6.65 (ddd, *J* = 15.6, 7.6, 7.0 Hz, 1H)	HMBC 2→4 NOESY 2→33'
3	133.2	6.40 (d, *J* = 15.6 Hz, 1H)	HMBC 3→1
4	136.6	-	
5	125.1	6.98 (br d, *J* = 7.6 Hz, 1H)	HMBC 5→3
6	127.9	7.17 (dd, *J* = 7.6, 7.5 Hz, 1H)	HMBC 6→4,8
7	127.5	7.05 (br d, *J* = 7.5 Hz, 1H)	
8	141.3	-	
9	123.9	7.81 (br s, 1H)	HMBC 9→3
10	29.9	2.84-2.91 (m, 1H)	HMBC 10→8,12
11	34.9	2.53-2.60 (m, 1H), 2.64-2.70 (m, 1H)	HMBC 11→8,12
12	171.5	-	
13	-	8.08 (br d, *J* = 7.7, 5.5 Hz, 1H)	TOCSY 13→14,15,16,17
14	52.2	4.16 (ddd, *J* = 8.9, 7.7, 5.5 Hz, 1H)	
15	33.9	1.44-1.52 (m, 1H), 1.62-1.68 (m, 1H)	
16	18.3	1.04-1.13 (m, 2H)	
17	13.4	0.77 (t, *J* = 7.3 Hz, 3H)	
18	171.9	-	
19	-	8.31 (br d, *J* = 6.9 Hz, 1H)	TOCSY 19→20,21
20	51.3	4.27-4.32 (m, 1H)	HMBC 20→22
21	35.3	2.68-2.79 (m, 1H)	HMBC 21→22
22	171.6	-	
23	-	not detected	
24	169.7	-	
25	-	7.12 (d, *J* = 7.1 Hz, 1H)	TOCSY 25→26,27,28,29 HMBC 25→24
26	56.6	4.27-4.32 (m, 1H)	
27	31.5	1.97-2.05 (m, 1H)	HMBC 27→26,30
28	17.6	0.85 (d, *J* = 6.8 Hz, 3H)	
29	18.8	0.87 (d, *J* = 6.8 Hz, 3H)	
30	169.7	-	
31	-	8.23 (br d, *J* = 7.3 Hz, 1H)	TOCSY 31→32,33 HMBC 31→30 NOESY 31→33
32	47.1	4.41 (ddd, *J* = 13.2, 7.3, 4.5 Hz, 1H)	HMBC 32→43 NOESY 32→35
33	35	*pro*-S 1.69 (dd, *J* = 13.2, 13.2 Hz, 1H)	HMBC 33→43
33'		*pro*-R 2.65 (dd, *J* = 13.2, 4.5 Hz, 1H)	
34	48.5	-	
35	72.4	4.83 (br s, 1H)	HMBC 35→1 NOESY 35→1,2,3
36	-	6.21 (br s, 1H)	HMBC 36→34,42
37	147	-	
38	110.7	6.56 (d, *J* = 8.1 Hz, 1H)	HMBC 38→40,42
39	130.3	7.12 (dd, *J* = 8.1, 1.5 Hz, 1H)	
40	108.5	-	
41	126.4	7.25 (d, *J* = 1.5 Hz, 1H)	
42	138.1	-	
43	169.8	-	
44	-	7.60 (s, 1H)	HMBC 44→32,34

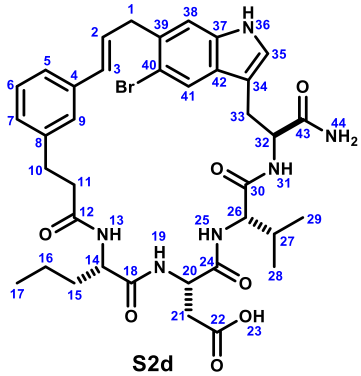
Macrocyclic Product ***S2c*** *(600 MHz, DMSO-d_6, 298K)*



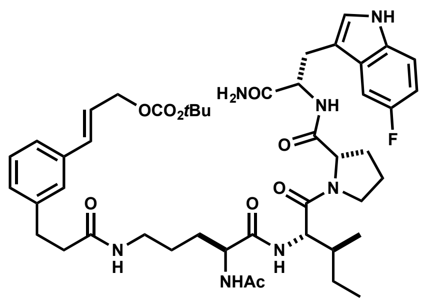
S2c

	13C	**1H**	**key correlations**
1	29.6	3.69 (dd, J = 16.5, 6.7 Hz, 1H), 3.78 (dd, J = 16.5, 7.0 Hz, 1H)	HMBC 1→2,3,34,35
2	125.5	6.47 (ddd, J = 15.6, 7.0, 6.7 Hz, 1H)	COSY 2→1 HMBC 2→4
3	131.8	6.68 (br d, J = 15.6 Hz, 1H)	HMBC 3→4
4	137	-	
5	123.5	7.25 (br d, J = 7.7 Hz, 1H)	HMBC 5→3 TOCSY 5→6,7,9
6	127.9	7.21 (dd, J = 7.7, 7.5 Hz, 1H)	HMBC 6→4,8
7	127.2	7.04 (br d, J = 7.5 Hz, 1H)	
8	141.3	-	
9	125.8	7.40 (br s, 1H)	HMBC 9→3
10	29.9	2.79 (ddd, J = 14.6, 8.6, 4.4 Hz, 1H), 2.93-2.99 (m, 1H)	HMBC 10→7,9,12
11	35.2	2.45 (ddd, J = 14.6, 8.3, 4.4 Hz, 1H), 2.51-2.56 (m, 1H)	HMBC 11→12
12	171.9	-	
13	-	8.08 (br d, J = 7.7 Hz, 1H)	HMBC 13→12 TOCSY 13→14,15,16,17
14	52.4	4.19 (ddd, J = 8.9, 7.7, 5.3 Hz, 1H)	COSY 14→15 HMBC 14→18
15	33.6	1.44-1.51 (m, 1H), 1.55-1.63 (m, 1H)	COSY 15→16 HMBC 15→18
16	18.5	1.14-1.30 (m, 2H)	
17	13.3	0.79 (dd, J = 7.3, 7.3 Hz, 3H)	
18	172.4	-	
19	-	8.10 (br d, J = 6.5 Hz, 1H)	HMBC 19→18
20	49.6	4.54 (ddd, J = 7.3, 6.5, 6.3 Hz, 1H)	HMBC 20→22,24
21	35.3	2.54 (dd, J = 16.8, 7.3 Hz, 1H), 2.71 (dd, J = 16.8, 6.3 Hz, 1H)	HMBC 21→22
22	171.9	-	
23	-	12.33 (br s, 1H)	
24	170.4	-	
25	-	7.63 (br d, J = 8.3 Hz, 1H)	HMBC 25→24
26	57.5	4.14 (dd, J = 8.3, 5.7 Hz, 1H)	HMBC 26→30
27	29.4	2.00-2.17 (m, 1H)	
28	16.7	0.61 (d, J = 6.8 Hz, 3H)	
29	18.8	0.75 (d, J = 6.8 Hz, 3H)	TOCSY 29→25,26,27,28
30	170	-	
31	-	7.67-7.70 (m, 1H)	HMBC 31→30
32	53.1	4.40 (ddd, J = 8.1, 8.0, 6.0 Hz, 1H)	
33	26.6	2.98 (dd, J = 14.4, 8.0 Hz, 1H), 3.07 (dd, J = 14.4, 6.0 Hz, 1H)	HMBC 33→34
34	105.5	-	
35	136.8	-	
36	-	10.88 (s, 1H)	HMBC 36→34
37	133.8	-	
38	112.2	7.20 (d, J = 8.4 Hz, 1H)	HMBC 38→40,42
39	122.2	7.08 (dd, J = 8.4, 1.8 Hz, 1H)	HMBC 39→37,40
40	110.8	-	
41	120.2	7.69 (d, J = 1.8 Hz, 1H)	HMBC 41→34,37,40
42	129.8	-	
43	172.7	-	
44	-	7.10 (br s, 1H), 7.19 (br s, 1H)	HMBC 44,44'→43

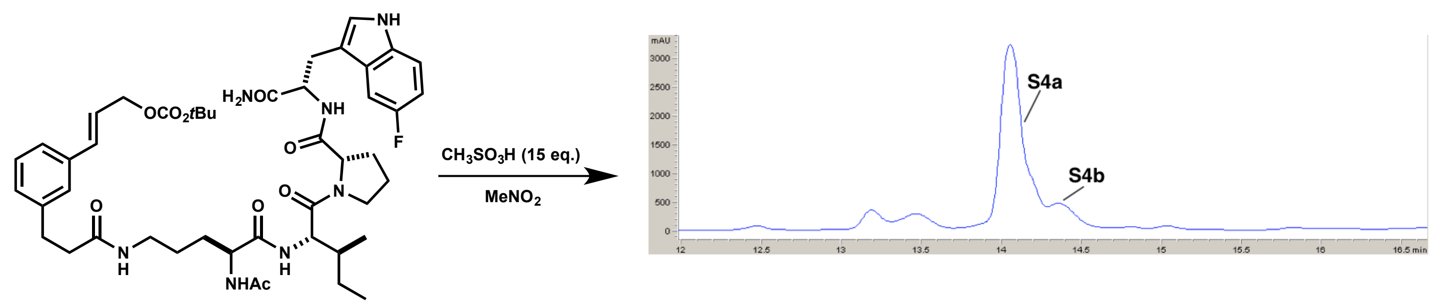
*Macrocyclic Product **S2d***   *(500 MHz, DMSO-d$_6$, 298K)*



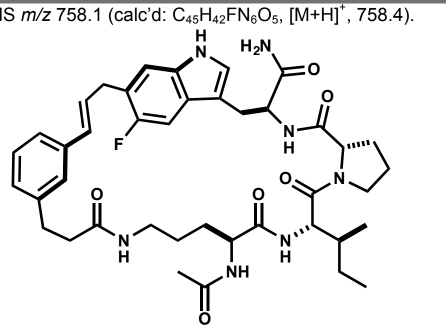
	13C	1H	key correlations
1	39	3.62 (dd, J = 15.5, 5.4 Hz, 1H), 3.72 (dd, J = 15.5, 6.5 Hz, 1H)	HMBC 1→2,3,38,39
2	129.2	6.39 (ddd, J = 15.8, 6.5, 5.4 Hz, 1H)	COSY 1→2 HMBC 2→4
3	130.3	6.24 (br d, J = 15.8 Hz, 1H)	
4	137.3	-	
5	128.3	7.15-7.19 (m, 1H)	HMBC 5→3
6	123.9	7.15-7.19 (m, 1H)	HMBC 6→4,8
7	127.5	6.96-7.01 (m, 1H)	
8	141.3	-	
9	125.1	7.13 (br s, 1H)	HMBC 9→3
10	30.4	2.68-2.75 (m, 1H), 2.77-2.85 (m, 1H)	HMBC 10→8,12
11	35.5	2.34 (ddd, J = 13.9, 5.9, 5.9 Hz, 1H), 2.53-2.61 (m, 1H)	HMBC 11→8,12
12	171.6	-	
13	-	7.90 (br d, J = 7.7 Hz, 1H)	TOCSY 13→14,15,16,17
14	52.1	4.08 (ddd, J = 7.8, 7.7, 5.0 Hz, 1H)	HMBC 14→18
15	34	1.16-1.26 (m, 1H), 1.27-1.36 (m, 1H)	HMBC 15→18
16	17.9	0.77-0.90 (m, 2H)	
17	13.3	0.46 (t, J = 7.3 Hz, 3H)	
18	172	-	
19	-	8.23 (br d, J = 7.4 Hz, 1H)	HMBC 19→18 TOCSY 19→20,21
20	50.5	4.42 (ddd, J = 8.4, 7.4, 5.0 Hz, 1H)	HMBC 20→24
21	35.9	2.55 (dd, J = 16.7, 8.4 Hz, 1H), 2.67 (dd, J = 16.7, 5.0 Hz, 1H)	HMBC 21→22
22	171.9	-	
23	-	12.15 (br s, 1H)	
24	170.5	-	
25	-	6.99-7.04 (m, 1H)	
26	57.6	4.04 (dd, J = 7.6, 5.4 Hz, 1H)	HMBC 26→30
27	30.8	1.89-1.90 (m, 1H)	
28	17.4	0.68 (d, J = 6.8 Hz, 3H)	HMBC 28→25,26,27,29
29	18.9	0.72 (d, J = 6.8 Hz, 3H)	
30	170	-	
31	-	7.84 (br d, J = 8.7 Hz, 1H)	HMBC 31→30 TOCSY 31→32,33
32	53.3	4.46 (ddd, J = 11.8, 8.7, 2.5 Hz, 1H)	
33	27.6	2.83 (dd, J = 14.3, 11.8 Hz, 1H), 3.12 (dd, J = 14.3, 2.5 Hz, 1H)	HMBC 33→34
34	109.9	-	
35	125.4	7.16-7.18 (m, 1H)	HMBC 35→37
36	-	10.76 (d, J = 1.4 Hz, 1H)	
37	135.7	-	
38	130.5	7.36 (s, 1H)	HMBC 38→1,40,42
39	113.5	-	
40	114.1	-	
41	121.9	7.96 (s, 1H)	HMBC 41→1,34,39,40
42	127.8	-	
43	173.6	-	
44	-	7.11 (br s, 1H), 7.51 (br s, 1H)	HMBC 44,44'→43



Acyclic Cinnamyl Carbonate S3: Synthesized according to Procedure A. Purified via SiO_2 chromatography using a gradient from 1% to 10% methanol in chloroform. Beige solid. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 10.93 (d, J = 2.5 Hz, 1H), 7.97 (d, J = 8.3 Hz, 1H), 7.89 (d, J = 8.4 Hz, 1H), 7.83 (t, J = 5.6 Hz, 1H), 7.28-7.37 (m, 5H), 7.22-7.27 (m, 3H), 7.11 (br. d, J = 7.3 Hz, 1H), 7.04 (br. s, 1H), 6.89 (ddd, J = 9.1, 9.1, 2.5 Hz, 1H), 6.64 (d, J = 16 Hz, 1H), 6.34 (dt, J = 16.0, 6.2 Hz, 1H), 4.68 (dd, J = 6.2, 1.1 Hz, 2H), 4.38 (ddd, J = 7.0, 7.0, 7.0 Hz, 1H), 4.26- 4.35 (m, 3H), 3.74 (ddd, J = 9.6, 6.6, 6.6 Hz, 1H), 3.53 (ddd, J = 9.6, 5.7, 6.0 Hz, 1H), 2.96-3.10 (m, 4H), 2.8 (app t, J = 7.8 Hz, 2H), 2.37 (app t, J = 7.8 Hz, 2H), 1.93-2.03 (m, 1H), 1.70-1.91 (m, 6H), 1.47-1.60 (m, 2H), 1.44 (s, 9H), 1.27-2.39 (m, 2H), 1.00-1.08 (m, 1H), 0.84 (d, J = 6.8 Hz, 3H), 0.81 (t, J = 7.4 Hz, 1H). ^{13}C NMR (DMSO-d_6, 126 MHz): δ 173.0, 171.7, 171.2, 171.1, 170.2, 169.2, 155.7, 152.8, 141.8, 135.8, 133.4, 132.7, 128.6, 128.0, 127.7, 127.7, 126.4, 125.8, 124.2, 123.3, 112.1, 112.0, 110.32, 110.28, 108.9, 108.7, 103.3, 103.1, 81.5, 67.0, 66.9, 59.5, 54.5, 53.3, 51.9, 47.2, 38.1, 36.9, 36.1, 31.0, 29.6, 28.9, 27.3, 25.8, 24.4, 24.1, 22.5, 14.9, 10.8. MS m/z 876.5 (calc'd: $C_{46}H_{62}N_7O_9$, $[M+H]^+$, 876.5)

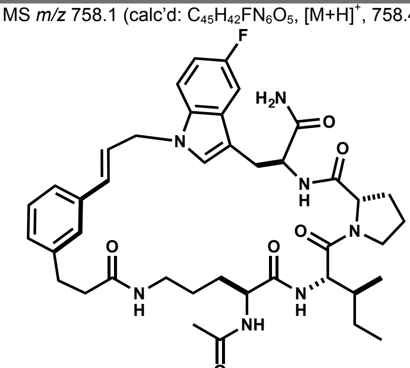


MS m/z 758.1 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 758.4).



S4a

MS m/z 758.1 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 758.4).



S4b

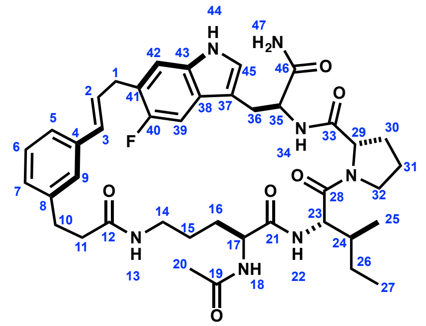
Analytical HPLC Method
Column: Waters XBridge™ C_{18}, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	30
2	30
30	60

Preparative HPLC method A:
Column: Waters XBridge™ C_{18}, 19x250mm, 5μm.
Solvent A: H_2O + 0.1%v TFA
Solvent B: ACN + 0.1%v TFA
Flow rate: 18.00 ml/min

Time	%B
0	30
2	30
30	100

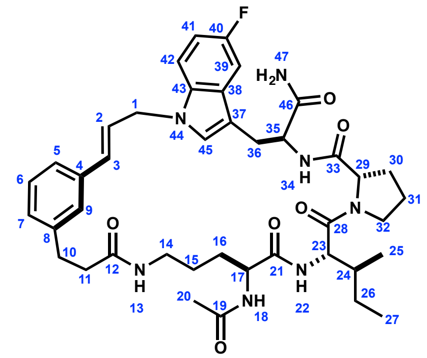
*Macrocyclic Product **S4a***



(500 MHz, DMSO-d$_6$, 298K)

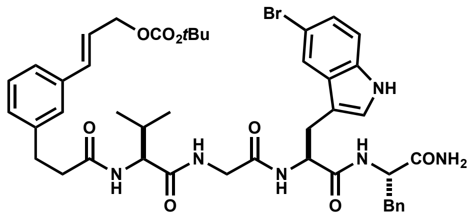
	13C	1H	key correlation
1	31.9	3.58 (br d, J = 5.8 Hz, 2H)	HMBC 1→2,3,41,42
2	129.3	6.38 (dt, J = 15.8, 5.8 Hz, 1H)	TOCSY 2→1 HMBC 2→4,41
3	129.6	6.32 (d, J = 15.8 Hz, 1H)	HMBC 3→5,9
4	136.9	-	
5	123.7	7.15-7.19 (m, 1H) overlap	
6	128.4	7.16-7.21 (m, 1H) overlap	HMBC 6→4,8
7	127.1	6.99-7.04 (m, 1H) overlap	
8	141.5	-	
9	124.8	7.15 (br s, 1H) overlap	
10	30.9	2.71 (t, J = 7.8 Hz, 2H)	HMBC 10→8,12
11	36.9	2.27 (br t, J = 7.8 Hz, 2H)	HMBC 11→8,12
12	171.1	-	
13	-	7.81 (br t, J = 6.0 Hz, 1H)	HMBC 13→12 COSY 13→14,14'
14	37.5	2.77-2.86 (m, 1H), 3.04-3.11 (m, 1H)	COSY 14→15
15	25.6	1.23-1.32 (m, 2H) overlap	COSY 15→16
16	29.6	1.26-1.33 (m, 1H) overlap, 1.48-1.55 (m, 1H) overlap	
17	51.1	4.24-4.30 (m, 1H)	TOCSY 17→14,15,16,18 HMBC 17→21
18	-	7.83 (d, J = 8.4 Hz, 1H)	HMBC 18→19
19	168.8	-	
20	22.1	1.79 (s, 3H)	HMBC 20→19
21	171.5	-	
22	-	7.93 (d, J = 7.7 Hz, 1H)	HMBC 22→21
23	54.7	4.19 (dd, J = 8.8, 7.7 Hz, 1H)	HMBC 23→21,28 TOCSY 23→24,25,26,27
24	35.6	1.63-1.71 (m, 1H) overlap	
25	14.6	0.87 (d, J = 6.8 Hz, 3H)	
26	23.8	0.98-1.09 (m, 1H), 1.45-1.54 (m, 1H) overlap	
27	10.3	0.78 (dd, J = 7.4, 7.4 Hz, 3H)	
28	126.7	-	
29	59.3	4.13 (dd, J = 8.3, 5.0 Hz, 1H)	COSY 29→30,30' TOCSY 29→30,31,32 HMBC 29→33
30	28.8	1.63-1.70 (m, 1H) overlap	
31	24.0	1.66-1.78 (m, 1H) overlap, 1.92-2.01 (m, 1H) overlap	
32	46.8	3.43-3.50 (m, 1H), 3.67-3.74 (m, 1H)	
33	171.2	-	
34	-	7.51 (br d, J = 7.7 Hz, 1H) overlap	HMBC 34→33
35	53.2	4.48 (ddd, J = 9.7, 7.7, 3.8 Hz, 1H)	HMBC 35→37,46
36	27.2	2.93-3.05 (m, 2H) overlap	HMBC 36→37
37	110.2	-	
38	126.1	-	
39	103.5	7.49 (d, JHF = 11.0 Hz, 1H)	HMBC 39→40
40	155.2 (d, J ≈ 240 Hz)	-	
41	119.9	-	
42	112.4	7.22 (d, JHF = 6.4 Hz, 1H)	HMBC 42→40
43	132.6	-	
44	-	10.84 (d, J = 1.9 Hz, 1H)	HBMC 44→37,38,43
45	124.2	7.16-7.18 (m, 1H) overlap	
46	173.6	-	
47	-	7.02 (br s, 1H) overlap, 7.38 (br s, 1H)	HMBC 47→46 TOCSY 47→47'

*Macrocyclic Product **S4b***

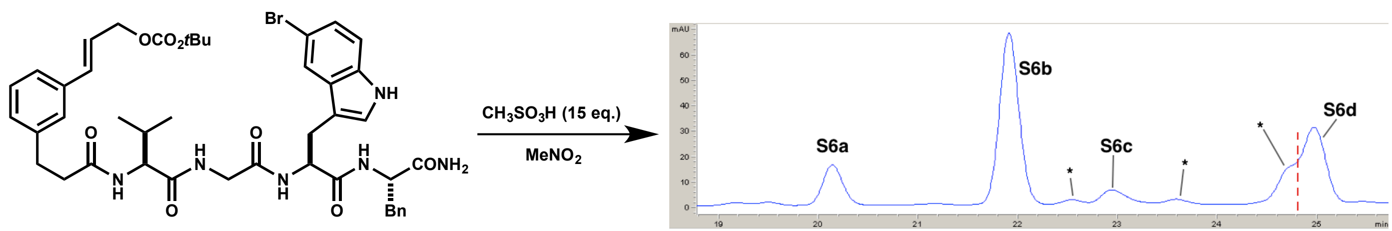


(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	47	4.49 (dd, *J* = 16.3, 5.6 Hz, 1H), 4.96 (dd, *J* = 16.6, 5.6, Hz, 1H)	HMBC 1→43,45
2	125.6	6.33 (ddd, *J* = 15.8, 5.6, 5.6 Hz, 1H)	TOCSY 2→3,1
3	131.4	6.48 (br d, *J* = 15.8 Hz, 1H)	HMBC 3→4
4	136.1	-	
5	124.3	7.16-7.19 (m, 1H) overlap	
6	128.3	7.14-7.19 (m, 1H) overlap	HMBC 6→4,8
7	127.6	7.03-7.07 (m, 1H)	
8	141.7	-	
9	125.8	7.15 (br s, 1H)	
10	30.6	2.72 (t, *J* = 7.5 Hz, 2H)	HMBC 10→8,12
11	36.7	2.23-2.32 (m, 2H)	HMBC 11→8,12
12	171.2	-	
13	-	7.69 (br t, *J* = 5.7 Hz, 1H)	HMBC 13→12 COSY 13→14
14	37.3	2.82-2.90 (m, 1H), 2.97-3.03 (m, 1H)	COSY 14→15
15	25.4	1.15-1.24 (m, 2H)	COSY 15→16,16' TOCSY 15→16,17
16	29	1.27-1.34 (m, 1H), 1.39-1.46 (m, 1H)	HMBC 16→17 COSY 16→17
17	51.6	4.22-4.28 (m, 1H)	HMBC 17→21
18	-	7.89 (d, *J* = 8.0 Hz, 1H)	
19	169.1	-	
20	22	1.79 (s, 3H)	
21	171.7	-	
22	-	7.99 (d, *J* = 8.6 Hz, 1H)	HMBC 22→21
23	54.4	4.25-4.30 (m, 1H)	
24	35.6	1.67-1.74 (m, 1H)	
25	15.1	0.81 (d, *J* = 6.7 Hz, 3H)	
26	23.5	0.95-1.03 (m, 1H), 1.43-1.51 (m, 1H)	
27	10.6	0.76 (t, *J* = 7.4 Hz, 3H)	
28	170.3	-	
29	59.7	4.16 (dd, *J* = 8.2, 5.0 Hz, 1H)	TOCSY 29→30,31,32 HMBC 29→33
30	28.7	1.57-1.65 (m, 1H), 1.84-1.89 (m, 1H)	
31	23.8	1.59-1.65 (m, 1H), 1.68-1.75 (m, 1H)	
32	46.8	3.45-3.51 (m, 1H), 3.55-3.61 (m, 1H)	
33	171.6	-	
34	-	7.79 (d, *J* = 8.1 Hz, 1H)	HMBC 34→33
35	52.6	4.36-4.42 (m, 1H)	COSY 35→36 HMBC 35→46
36	26.5	2.96-3.03 (m, 1H), 3.08-3.14 (m, 1H)	HMBC 36→35,37
37	110.4	-	
38	128.1	-	
39	103.4	7.37 (dd, *J*HF = 10.1, *J*HH = 2.4 Hz, 1H)	HMBC 39→40,43
40	156.9 (d, *J*≈230 Hz)	-	
41	108.9	6.93 (ddd, *J*HF = 9.4 Hz, *J*HH = 8.9, 2.4 Hz, 1H)	HMBC 41→43
42	110.6	7.44 (dd, *J*HH = 8.9 Hz, *J*HF = 4.5 Hz, 1H)	HMBC 42→38,40
43	132.5	-	
44	-	-	
45	128.7	7.35 (br s, 1H)	HMBC 45→37,38,43
46	173.3	-	
47	-	7.11 (br s, 1H), 7.27 (br s, 1H)	TOCSY 47→47'

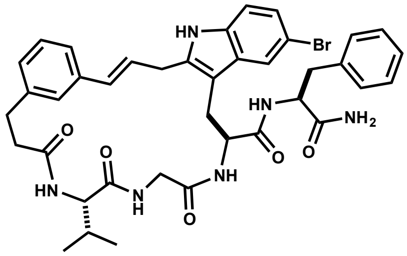


Acyclic Cinnamyl Carbonate S5: Synthesized according to Procedure A. Purified via SiO_2 chromatography using a gradient from 1% to 10% methanol in chloroform. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 0.77 (d, J = 6.8 Hz, 3H), 0.78 (d, J = 6.8 Hz, 3H), 1.43 (s, 9H), 1.86-1.96 (m, 1H), 2.41 (ddd, J = 14.5, 8.7, 5.9 Hz, 1H), 2.55 (dd, J = 14.5, 8.3 Hz, 1H), 2.72-2.88 (m, 4H), 3.03 (apt dt, J = 14.1, 4.9 Hz, 2H), 3.55 (dd, J = 16.6, 5.4 Hz, 1H), 3.74 (dd, J = 16.6, 6.0 Hz, 1H), 4.11 (dd, J = 8.2, 6.9 Hz, 1H), 4.42 (ddd, J = 8.6, 8.6, 5.1 Hz, 1H), 4.47 (ddd, J = 9.0, 8.1, 4.8 Hz, 1H), 4.66 (dd, J = 6.2, 1.1 Hz, 2H), 6.31 (dt, J = 15.9, 6.2 Hz, 1H), 6.61 (br d, J = 15.9 Hz, 1H), 7.05-7.11 (m, 2H), 7.13-7.30 (m, 12H), 7.74 (d, J = 1.9 Hz, 1H), 7.88 (d, J = 8.3 Hz, 1H), 8.02 (d, J = 8.0 Hz, 1H), 8.08 (d, J = 8.3 Hz, 1H), 8.16 (t, J = 5.7 Hz, 1H), 11.01 (d, J = 1.9 Hz, 1H). ^{13}C NMR (DMSO-d_6, 126 MHz): δ 173.2, 172.2, 172.1, 171.5, 169.2, 153.3, 142.1, 138.4, 136.3, 135.2, 133.9, 129.7, 129.6, 129.0, 128.5 (2), 126.8, 126.7, 125.9, 124.7, 123.74, 123.70, 121.1, 113.8, 111.5, 110.4, 82.0, 67.4, 58.5, 54.5, 53.9, 42.4, 37.8, 36.9, 31.4, 30.6, 27.8, 19.6,18.6



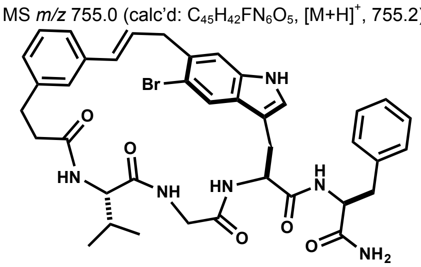
*Unidentified isomeric products

MS m/z 755.0 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 755.2).



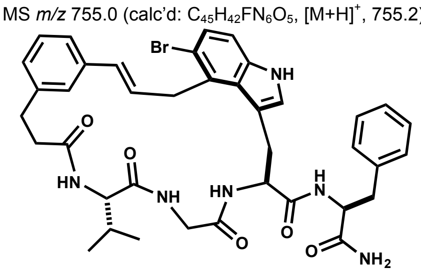
S6a

MS m/z 755.0 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 755.2).



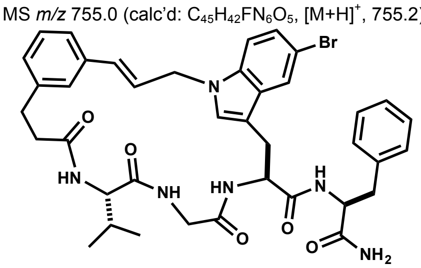
S6b

MS m/z 755.0 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 755.2).



S6c

MS m/z 755.0 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 755.2).



S6d

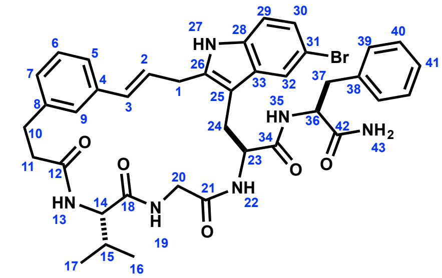
Analytical HPLC Method
Column: Waters Sunfire™ C_{18}, 4.6x250 mm, 5 μm
Solvent A: H_2O + 0.1% TFA
Solvent B: ACN + 0.1% TFA
Flow rate: 1.00 mL/min

Time	%B
0	30
2	30
30	60

Preparative HPLC method A:
Column: Waters XBridge™ C_{18}, 19x250mm, 5μm.
Solvent A: H_2O + 0.1%v TFA
Solvent B: ACN + 0.1%v TFA
Flow rate: 18.00 ml/min

Time	%B
0	30
2	30
30	100

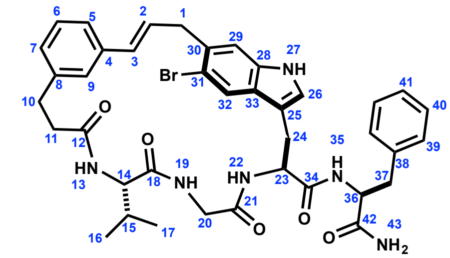
*Macrocyclic Product **S6a***



(500 MHz, DMSO-d_6, 298K)

	13C	1H	key correlation
1	28.3	3.53 (dd, *J* = 17.2, 5.9 Hz, 1H), 3.81 (dd, *J* = 17.2, 5.9 Hz, 1H)	HMBC 1→25,26
2	127.6	6.56 (ddd, *J* = 16.0, 5.9, 5.9 Hz, 1H)	HMBC 2→4, COSY 2→1
3	130.2	6.37 (br d, *J* = 16.0 Hz, 1H)	
4	136.9	-	
5	123	7.26 (d, *J* = 7.9 Hz, 1H) overlap	HMBC 5→9,3
6	127.97	7.18 (dd, *J* = 7.9, 7.9 Hz, 1H) overlap	HMBC 6→4,8
7	127.95	6.99 (d, *J* = 7.9 Hz, 1H) overlap	
8	140.7	-	
9	124.9	7.20 (s, 1H) overlap	
10	29.8	2.73-2.78 (m, 1H) overlap, 2.89-2.96 (m, 1H) overlap	HMBC 10→9
11	34	2.34-2.40 (m, 1H), 2.72-2.79 (m, 1H) overlap	TOCSY 11→11',10
12	171.3	-	
13	-	7.78 (d, *J* = 9.1 Hz, 1H)	TOCSY 13→14,15,16,17, HMBC 13→12
14	58	3.92 (dd, *J* = 9.1, 6.0 Hz, 1H)	HMBC 14→18
15	29.1	1.92-1.98 (m, 1H)	
16	17.6	0.76 (d, *J* = 3.7 Hz, 3H)	
17	19	0.77 (d, *J* = 3.7 Hz, 3H)	
18	170.8	-	
19	-	6.97 (dd, *J* = 8.0, 2.9 Hz, 1H) overlap	
20	41.1	3.12 (dd, *J* = 16.7, 2.9 Hz, 1H), 3.76 (dd, *J* = 16.7, 8.0 Hz, 1H)	TOCSY 20→19, HMBC 20→18,21
21	167.6	-	
22	-	8.04-8.08 (m, 1H) overlap	HMBC 22→21
23	53.1	4.51-4.56 (m, 1H)	HMBC 23→34
24	26.7	2.72-2.79 (m, 1H) overlap, 2.96-3.00 (m, 1H) obscured	HMBC 24→25,26,33,34
25	106.6	-	
26	136.4	-	
27	-	11.00 (s, 1H)	
28	134.1	-	
29	112.3	7.21 (d, *J* = 8.5 Hz, 1H) overlap	HMBC 29→31
30	122.5	7.12 (dd, *J* = 8.5, 1.6 Hz, 1H)	HMBC 30→28,31
31	110.7	-	
32	120	7.73 (d, *J* = 1.6 Hz, 1H)	HMBC 32→25,28,30,31
33	129.8	-	
34	171.1	-	
35	-	8.04-8.08 (m, 1H) overlap	HMBC 35→34
36	53.5	4.49 (ddd, *J* = 9.0, 8.0, 5.0 Hz, 1H)	HMBC 36→34,38,42
37	37.3	2.86 (dd, *J* = 13.7, 9.0 Hz, 1H), 3.05 (dd, *J* = 13.7, 5.0 Hz, 1H)	HMBC 37→38,39
38	137.5	-	
39	129	7.22 (d, *J* = 7.7 Hz, 2H) overlap	
40	127.9	7.26 (dd, *J* = 7.7, 7.7 Hz, 2H) overlap	HMBC 40→38
41	126	7.15-7.18 (m, 1H) overlap	
42	172.4	-	
43	-	7.15 (br s, 1H), 7.46 (br s, 1H)	HMBC 43→42, TOCSY 43→43'

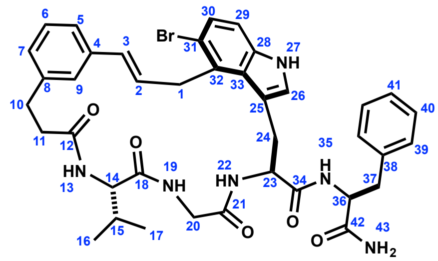
*Macrocyclic Product **S6b***



(600 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	38.3	3.61-3.67 (m, 1H) overlap, 3.69-3.75 (m, 1H) overlap	HMBC 1→3
2	129	6.40 (ddd, *J* = 16.0, 5.6, 5.6 Hz, 1H)	HMBC 2→4, COSY 2→1
3	129.1	6.08 (br d, *J* = 16.0 Hz, 1H)	HMBC 3→4,5,9
4	137	-	
5	122.6	7.21 (br d, *J* = 8.0 Hz, 1H)	HMBC 5→3,7,9
6	127.6	7.17 (dd, *J* = 8.0, 8.0 Hz, 1H) overlap	HMBC 6→8,4
7	126.7	6.99 (d, *J* = 8.0 Hz, 1H) overlap	
8	140.9	-	
9	124.5	6.99 (br s, 1H) overlap	HMBC 9→3,5,7
10	29.1	2.68-2.74 (m, 1H), 2.86-2.92 (m, 1H) overlap	HMBC 10→8
11	34.3	2.42-2.48 (m, 1H), 2.53-2.59 (m, 1H)	HMBC 11→8
12	170.9	-	
13	-	7.39 (d, *J* = 8.7 Hz, 1H)	HMBC 13→12
14	57.8	3.92 (dd, *J* = 8.7, 5.9 Hz, 1H)	COSY 14→13, HMBC 14→18
15	29.5	1.83-1.90 (m, 1H)	COSY 15→14
16	17	0.68 (d, *J* = 6.7 Hz, 3H) overlap	COSY 16→15
17	18.6	0.69 (d, *J* = 6.6 Hz, 3H) overlap	
18	170.3	-	
19	-	7.26-7.31 (m, 1H) overlap	
20	40.8	3.09-3.17 (m, 1H) overlap, 3.66-3.72 (m, 1H) overlap	HMBC 20→18,21
21	167.4	-	
22	-	7.75-7.86 (m, 1H) overlap	
23	54.8	4.50-4.56 (m, 1H) overlap	COSY 23→22
24	27.6	2.76-2.82 (m, 1H),3.04-3.10 (m, 1H) overlap	
25	109.9	-	
26	124.4	7.13 (d, *J* = 2.1 Hz, 1H)	HMBC 26→25,28,33, COSY 26→27
27	-	10.71 (br s, 1H)	HMBC 27→25,26,28,33
28	135.1	-	
29	113.1	7.31 (s, 1H)	HMBC 29→1
30	129.6	-	
31	113.8	-	
32	121.9	8.00 (s, 1H)	HMBC 32→25,28,30,31
33	128.1	-	
34	170.5	-	
35	-	7.84 (d, *J* = 8.1 Hz, 1H) overlap	HMBC 35→34
36	53.1	4.56 (ddd, *J* = 8.5, 8.1, 5.4 Hz, 1H) overlap	HMBC 36→38,42
37	37.2	2.91 (dd, *J* = 13.9, 8.5 Hz, 1H) overlap, 3.10 (dd, *J* = 13.9, 5.4 Hz, 1H) overlap	HMBC 37→38,39
38	137.4	-	
39	128.6	7.25-7.28 (m, 2H) overlap	HMBC 39→41
40	127.5	7.25-7.28 (m, 2H) overlap	HMBC 40→38
41	125.5	7.16-7.20 (m, 1H) overlap	
42	172.1	-	
43	-	Not observed	

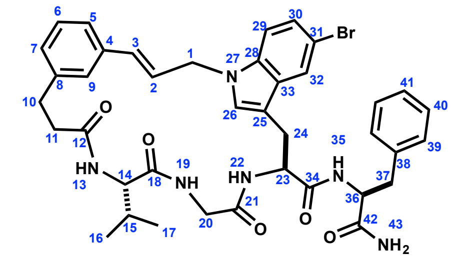
*Macrocyclic Product **S6c***



(500 MHz, DMSO-d_6, 298K)

	13C	1H	key correlation
1	34.9	3.93 (br dd, *J* = 16.6, 4.1 Hz, 1H), 4.03 (dd, *J* = 16.6, 6.3 Hz, 1H)	HMBC 1→31,33
2	128.2	6.42 (ddd, *J* = 16.0, 6.3, 4.1 Hz, 1H)	COSY 2→1, HMBC 2→4,5
3	129.8	6.13 (br d, *J* = 16.0 Hz, 1H)	
4	136.8	-	
5	123.1	6.99 (dd, *J* = 7.9 Hz, 1H) overlap	
6	127.9	7.14-7.19 (m, 1H) overlap	HMBC 6→8,4
7	127.3	7.09 (d, *J* = 7.9 Hz, 1H) overlap	HMBC 7→10
8	141.3	-	
9	124.62	7.28 (br s, 1H)	HMBC 9→10,5,7
10	29.3	2.68-2.75 (m, 1H) overlap, 2.96-3.03 (m, 1H) overlap	HMBC 10→7,8,9
11	34.3	2.40-2.47 (m, 1H), 2.69-2.77 (m, 1H) overlap	HMBC 11→8
12	171.7	-	
13	-	7.95 (d, *J* = 8.6 Hz, 1H)	TOCSY 13→14,15,16,17, HMBC 13→12
14	57.5	4.24 (dd, *J* = 8.6, 5.6 Hz, 1H)	HMBC 14→18
15	30.1	2.03-2.11 (m, 1H)	HMBC 15→18
16	17.3	0.78 (d, *J* = 6.9 Hz, 3H)	
17	19.1	0.82 (d, *J* = 6.9 Hz, 3H)	
18	171	-	
19	-	7.82-7.86 (m, 1H) overlap	HMBC 19→18
20	42.3	3.58 (dd, *J* = 16.4, 4.8 Hz, 1H), 3.78 (dd, *J* = 16.4, 6.1 Hz, 1H)	COSY 20→19, HMBC 20→18
21	168.2	-	
22	-	8.11 (d, *J* = 8.1 Hz, 1H)	HMBC 22→21
23	54	4.59 (ddd, *J* = 8.4, 8.1, 5.9 Hz, 1H)	HMBC 23→24
24	29.3	2.96-3.03 (m, 1H) overlap, 3.15 (dd, *J* = 14.7, 8.4 Hz, 1H)	HMBC 24→25,34
25	110.1	-	
26	125.5	6.90 (d, *J* = 2.2 Hz, 1H)	HMBC 26→25,33
27	-	11.05 (d, *J* = 2.2 Hz, 1H)	HMBC 27→25,26,28,33
28	135.6	-	
29	111.6	7.17 (d, *J* = 8.5 Hz, 1H) overlap	HMBC 29→31
30	124.55	7.25 (d, *J* = 8.5 Hz, 1H) overlap	
31	114.7	-	
32	129.5	-	
33	126.3	-	
34	170	-	
35	-	7.85 (d, *J* = 8.3 Hz, 1H)	HMBC 35→34
36	53.6	4.39 (ddd, *J* = 8.5, 8.3, 5.3 Hz, 1H)	HMBC 36→34,42
37	37.2	2.74-2.81 (m, 1H) overlap, 2.96-3.02 (m, 1H) overlap	HMBC 37→38,39,42
38	137.4	-	
39	128.9	7.18 (d, *J* = 7.7 Hz, 2H) overlap	HMBC 39→37
40	127.8	7.20-7.25 (m, 2H) overlap	
41	126	7.14-7.19 (m, 1H) overlap	
42	172	-	
43	-	6.99 (br s, 1H) overlap, 7.10 (br s, 1H) overlap	TOCSY 43→43', HMBC 43→42

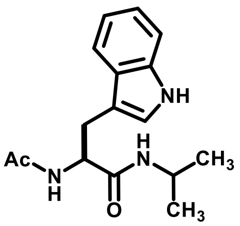
*Macrocyclic Product **S6d***



(500 MHz, DMSO-d_6, 298K)

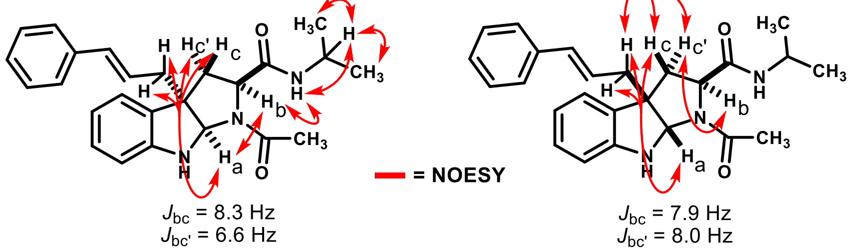
	13C	1H	key correlation
1	46.8	4.85 (dd, *J* = 16.7, 6.1 Hz, 1H), 4.91 (dd, *J* = 16.7, 5.2 Hz, 1H)	HMBC 1→2,3,28.
2	125.6	6.51 (ddd, *J* = 15.9, 6.1, 5.2 Hz, 1H)	COSY 2→3,1 HMBC 2→4
3	131.5	6.34 (d, *J* = 15.9 Hz, 1H)	
4	135.8	-	
5	123.5	7.20-7.23 (m, 1H)	HMBC 5→3
6	127.7	7.16-7.20 (m, 1H)	HMBC 6→4,8
7	128.1	7.03 (br d, *J* = 7.3 Hz, 1H)	
8	140.6	-	
9	125.8	7.24 (br s, 1H)	HMBC 9→5,7
10	30.1	2.72-2.79 (m, 1H), 2.85-2.93 (m, 1H)	
11	34.7	2.37 (ddd, *J* = 14.4, 7.8, 3.7 Hz, 1H), 2.65-2.72 (m, 1H)	HMBC 11→8,12 TOCSY 11→11',10,1'
12	171.3	-	
13	-	7.77 (d, *J* = 8.6 Hz, 1H)	HMBC 13→12 TOCSY 13→14,15,16,17
14	57.9	3.89 (dd, *J* = 8.6, 6.2 Hz, 1H)	HMBC 14→18
15	29.1	1.90-1.99 (m, 1H)	
16	17.7	0.77 (d, *J* = 6.9 Hz, 3H)	
17	18.7	0.78 (d, *J* = 6.8 Hz, 3H)	
18	170.6	-	
19	-	7.20-7.23 (m, 1H)	HMBC 19→18
20	40.9	3.14 (dd, *J* = 16.8, 4.4 Hz, 1H), 3.63 (dd, *J* = 16.8, 7.0 Hz, 1H)	HMBC 20→18,21 TOCSY 20→19
21	167.6	-	
22	-	8.00 (d, *J* = 9.1 Hz, 1H)	HMBC 22→21 TOCSY 22→23,24
23	51.9	4.56 (ddd, *J* = 11.4, 9.1, 3.1 Hz, 1H)	HMBC 23→25
24	27.2	2.66-2.73 (m, 1H), 3.04-3.10 (m, 1H)	HMBC 24→25,26,33
25	109.8	-	
26	128	7.23 (br s, 1H)	HMBC 26→25
27	-	-	
28	134.3	-	
29	111.7	7.42 (d, *J* = 8.8 Hz, 1H)	HMBC 29→31,33
30	123.1	7.19-7.24 (m, 1H)	HMBC 30→31
31	110.8	-	
32	121.1	7.84 (d, *J* = 1.9 Hz, 1H)	HMBC 32→28,31
33	129.1	-	
34	170.9	-	
35	-	8.21 (d, *J* = 8.1 Hz, 1H)	HMBC 35→34 TOCSY 35→36,37
36	53.4	4.49 (ddd, *J* = 8.9, 8.1, 4.9 Hz, 1H)	HMBC 36→38,42
37	37.1	2.85 (dd, *J* = 13.8, 8.9 Hz, 1H), 3.04 (dd, *J* = 13.8, 4.9 Hz, 1H)	HMBC 37→38
38	137.5	-	
39	128.9	7.22-7.25 (m, 2H)	TOCSY 39→41
40	127.5	7.24-7.28 (m, 2H)	HMBC 40→38
41	126	7.15-7.19 (m, 1H)	HMBC 41→39
42	172.4	-	
43	-	7.11 (br s, 1H), 7.43 (br s, 1H)	TOCSY 43→43' HMBC 43→42

***D*.2.** Synthesis of pyrroloindoline isomerization model system **21a&b**

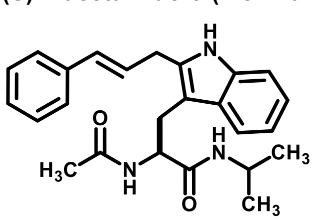


***N*-Acetyl-L-tryptophan isopropyl amide (S7):** Boc-L-Tryptophan (1.52 g, 5 mmol) was dissolved in DMF and cooled in an ice bath, then treated with HBTU (2.08 g, 5.5 mmol). The mixture was stirred cold for 10 min, then iPr_2NH (1.05 mL, 6 mmol) was added. The mixture was stirred at rt for 30 min, then concentrated, re-dissolved in EtOAc and washed successively with $NaHCO_3$, 1M HCl, brine, dried over Na_2SO_4 and concentrated. The resulting residue was treated with 4N HCl in dioxane for 30 min, then concentrated and re-dissolved in DMF. The mixture was rendered basic by the addition of iPr_2EtN, cooled to 0 ºC and treated with Ac_2O (708 µL, 7.5 mmol). The mixture was stirred at rt for 30 min, then concentrated, re-dissolved in EtOAc and washed successively with $NaHCO_3$, 1N HCl, brine, dried over Na_2SO_4 and concentrated. The residue was triturated with hexanes:$CHCl_3$ (1:1) and the resulting solid was collected by filtration to give **S7** (1.26 g, 73%) as a white solid. ^{1}H NMR (DMSO-*d*$_6$, 400 MHz): δ 0.92 (d, *J* = 6.6 Hz, 3H), 1.02 (d, *J* = 6.6 Hz, 3H), 1.78 (s, 3H), 2.88 (dd, *J* = 14.5, 8.2 Hz, 1H), 3.01 (dd, *J* = 14.5, 5.8 Hz, 1H), 3.72-3.86 (m, 1H), 4.46 (ddd, *J* = 8.6, 8.2, 5.9 Hz, 1H), 6.96 (ddd, *J* = 8.0, 7.0, 1.0 Hz, 1H), 7.04 (ddd, *J* = 8.1, 7.0, 1.1 Hz, 1H), 7.30 (ddd, *J* = 8.0, 1.1,1.0 Hz, 1H), 7.58 (br d, *J* = 7.8 Hz, 1H), 7.75 (d, *J* = 7.7 Hz, 1H), 7.96 (d, *J* = 8.3 Hz, 1H), 10.75-10.79 (m, 1H). ^{13}C NMR (DMSO-*d*$_6$, 101 MHz): δ 175.7, 174.1, 141.2, 132.6, 128.7, 126.0, 123.8, 123.3, 116.4, 115.4, 58.6, 33.5, 27.8, 27.5, 27.4. MS *m/z* 288.4 (calc'd: $C_{16}H_{22}N_3O_2$ $[M+H]^+$, 288.4).

***endo*-Pyrroloindoline (21a) and *exo*-pyrroloindoline (21b):** Anhydrous DCM was vigorously sparged with argon for 15 min. To a vial was added *N*-acetyl-L-tryptophan isopropyl amide (**S7**, 345 mg, 1 mmol), cinnamyl alcohol (147 mg, 1.1 mmol) and $Pd(PPh_3)_4$ (58 mg, 0.05 mmol), and the vessel was evacuated and backfilled with argon (x3). DCM (2.5 mL) was added, the mixture was cooled in an ice bath, and Et_3B (1.0 M in hexanes, 1.2 mL) was added in one portion. The resulting suspension was stirred at 0 ºC for 9 hrs, then diluted with EtOAc and washed with sat. $NaHCO_3$ (x2), brine, dried over Na_2SO_4 and concentrated. Purification by column chromatography on SiO_2 eluted with 0→8% MeOH in $CHCl_3$ afforded **21a** (150 mg, 37%) and **21b** (146 mg, 36%). **21a:** R_f = 0.61, 6% MeOH/$CHCl_3$, ^{1}H NMR ($CDCl_3$, 500 MHz, major rotamer): δ 0.48 (d, *J* = 6.6 Hz, 3H), 0.90 (d, *J* = 6.6 Hz, 3H), 2.01 (s, 3H), 2.49 (dd, *J* = 13.8, 8.3 Hz, 1H), 2.59 (ddd, *J* = 13.8, 6.6, 1.0 Hz, 1H), 2.63-2.67 (m, 2H), 3.56-3.70 (m, 1H), 4.36-4.43 (m, 2H), 5.55 (br s, 1H), 5.99 (ddd, *J* = 15.6, 8.3, 7.0 Hz, 1H), 6.08 (d, *J* = 7.8 Hz, 1H), 6.40 (d, *J* = 15.6 Hz, 1H), 6.61 (d, *J* = 7.8 Hz, 1H), 6.77 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.04-7.12 (m, 2H), 7.17-7.22 (m, 1H), 7.23-7.29 (m, 4H). ^{13}C NMR ($CDCl_3$, 126 MHz, major rotamer): δ 171.3, 170.2, 147.9, 136.9, 134.0, 132.0, 129.0, 128.5, 127.4, 126.1, 124.4, 123.9, 120.1, 109.6, 80.7, 63.0, 55.6, 42.8, 41.1, 40.5, 22.2, 22.2, 21.3. MS *m/z* 404.2 (calc'd: $C_{25}H_{30}N_3O_2$ $[M+H]^+$, 404.2). **21b:** R_f = 0.50, 6% MeOH/$CHCl_3$, ^{1}H NMR ($CDCl_3$, 600 MHz, major rotamer): δ 1.17 (d, *J* = 6.6 Hz, 3H), 1.18 (d, *J* = 6.6 Hz, 3H), 1.92 (s, 3H), 2.37 (dd, J = 13.1, 7.9 Hz, 1H), 2.58 (br dd, J = 13.5, 8.0 Hz, 1H), 2.63 (br dd, J = 13.5, 7.3 Hz,1H), 2.72 (dd, *J* = 13.1, 8.0 Hz, 1H), 4.04-4.08 (m, 1H), 4.09-4.17 (m, 1H), 5.53 (s, 1H), 6.08 (br d, J = 8.0 Hz, 1H), 6.09 (apt dt, *J* = 15.5, 7.7 Hz, 1H), 6.38 (d, *J* = 15.5 Hz, 1H), 6.60 (d, *J* = 7.7 Hz, 1H), 6.77 (dd, *J* = 7.3, 7.3 Hz, 1H), 7.08-7.16 (m, 2H), 7.21-7.26 (m, 1H), 7.24-7.32 (m, 5H). ^{13}C NMR ($CDCl_3$, 150 MHz, major rotamer): δ 172.0, 170.9, 148.5, 137.1, 133.8, 128.8, 128.60, 128.58, 127.4, 126.2, 124.9, 123.3, 118.9, 109.7, 82.7, 62.3, 55.0, 41.8, 41.6, 40.6, 22.8, 22.53, 22.50. MS *m/z* 404.2 (calc'd: $C_{25}H_{30}N_3O_2$ $[M+H]^+$, 404.3).

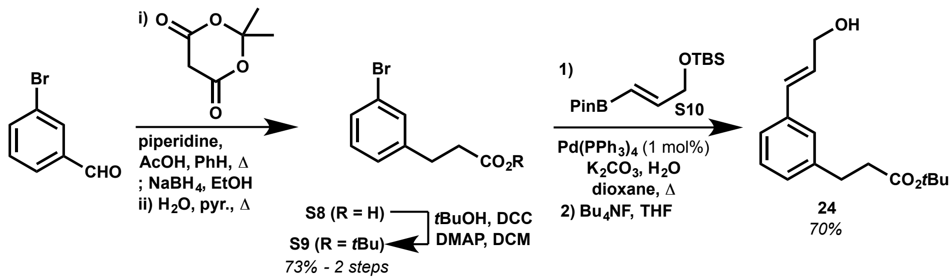


21a **21b**



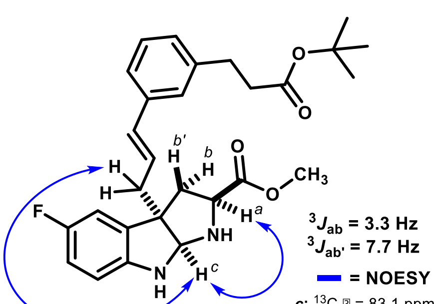
(*S*)-2-acetamido-3-(2-cinnamyl-1H-indol-3-yl)-N-isopropylpropanamide (22): *exo*-Pyrroloindoline **21b** (12.7 mg, 31.5 µmol) was dissolved in $MeNO_2$ (5.0 mL) and treated with TFA (1.3 mL). The mixture was stirred at rt for 30 min, then concentrated and dried thoroughly in vacuo. Purification by column chromatography on SiO_2 eluted with 0→2% MeOH in $CHCl_3$ afforded **22** (9.0 mg, 71%) as a light yellow film. R_f = 0.48, 6% MeOH/$CHCl_3$. ^{1}H NMR ($CDCl_3$, 500 MHz, major rotamer): δ 0.64 (d, *J* = 6.6 Hz, 3H), 0.93 (d, *J* = 6.5 Hz, 3H), 1.99 (s, 3H), 3.05 (dd, *J* = 14.1, 10.0 Hz, 1H), 3.28 (dd, *J* = 14.1, 4.8 Hz, 1H), 3.68 (dd, *J* = 16.4, 6.5 Hz, 1H), 3.75 (dd, *J* = 16.4, 6.6 Hz, 1H), 3.76-3.85 (m, 1H), 4.60 (ddd, *J* = 10.0, 7.3, 4.8 Hz, 1H), 5.07 (br d, *J* = 7.3 Hz, 1H), 6.32 (ddd, *J* = 15.7, 6.8, 6.8 Hz, 1H), 6.47 (br d, *J* = 7.3 Hz, 1H), 6.54 (d, *J* = 15.7 Hz, 1H), 7.09-7.18 (m, 2H), 7.21-7.34 (m, 4H), 7.34-7.39 (m, 2H), 7.66 (d, *J* = 7.4 Hz, 1H), 7.99 (br s, 1H). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 170.3, 170.0, 136.9, 135.5, 134.3, 132.5, 128.8, 128.6, 127.8, 126.3, 126.3, 121.9, 120.0, 118.7, 110.7, 107.3, 54.2, 41.6, 29.9, 28.2, 23.4, 22.6, 21.9. MS *m/z* 404.2 (calc'd: $C_{25}H_{30}N_3O_2$ $[M+H]^+$, 404.2).

D.3. Selective Synthesis of **9d**



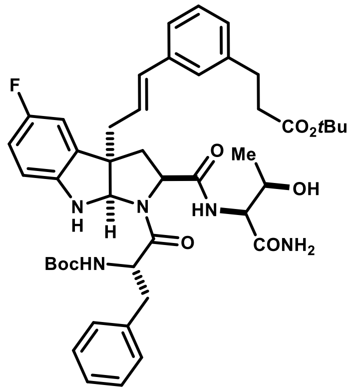
(3-bromophenyl)propanic acid *tert*-butyl ester (S8): 3-Bromobenzaldehyde (4.63 g, 25 mmol), Meldrum's acid (3.60 g, 25 mmol), piperidine (198 µL, 2 mmol), AcOH (429 µL, 7.5 mmol) were dissolved in benzene (50 mL) and heated to reflux on a Dean-Stark apparatus. After 30 min, the reaction was cooled in an ice bath and EtOH (5 mL) was added, followed by the addition of $NaBH_4$ (945 mg, 25 mmol) in portions. The mixture was stirred for 90 min, quenched by the addition of H_2O, and concentrated. To the residue was added pyridine (40 mL) and H_2O (4 mL), and the mixture was heated to reflux for 22 hours. The reaction was cooled, concentrated, diluted with 1M NaOH (75 mL), and washed with Et_2O (x2). The aqueous phase was acidified to pH <2 by the addition of conc. HCl, and extracted with DCM (x3). The combined extract was washed with brine, dried over Na_2SO_4 and concentrated to give acid **S8** (5.32 g, 93%) as a yellow crystalline solid, which was used without purification. ^1H NMR (500 MHz, $CDCl_3$): δ 2.68 (t, *J* = 7.7 Hz, 2H), 2.93 (t, *J* = 7.7 Hz, 2H), 7.10-7.21 (m, 2H), 7.31-7.42 (m, 2H), 9.33 (br s, 1H). ^{13}C NMR (126 MHz, $CDCl_3$): 178.8, 142.6, 131.5, 130.2, 129.7, 127.1, 122.7, 35.4, 30.2. MS *m/z* 227.0/229.0 (calc'd: $C_9H_8BrO_2$ $[M-H]^-$, 227.0). This material (5.32 g, 23.2 mmol) was dissolved in anhydrous DCM (75 mL) and treated with *t*-BuOH (6.61 mL, 69.6 mmol), DMAP (3.41 g, 27.9 mmol). The mixture was cooled in an ice bath, DCC (5.75 g, 27.9 mmol) was added. The mixture was refluxed overnight, and the resulting suspension was filtered through a pad of SiO_2, rinsing with DCM. The filtrate was exchanged to THF and treated with a small amount of aqueous AcOH and Norit for 30 min. The volatiles were then removed, and the residue was triturated with 1:1 hexanes:DCM and filtered through a pad of SiO_2, rinsing with the same. The filtrate was evaporated to give ester **S9** (5.22 g, 79%) as a pale yellow oil. ^1H NMR (500 MHz, $CDCl_3$): δ 1.41 (s, 9H), 2.52 (t, *J* = 7.7 Hz, 2H), 2.87 (t, *J* = 7.7 Hz, 2H), 7.10-7.17 (m, 2H), 7.32 (ddd, *J* = 6.8, 2.1, 2.1 Hz, 1H), 7.34-7.37 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$): δ 172.0, 143.2, 131.6, 130.1, 129.4, 127.2, 122.5, 80.7, 36.9, 30.8, 28.2.

(*E*)-3-(3-(3-hydroxyprop-1-en-1-yl)phenylpropionic acid *tert*-butyl ester (24): Bromide **S9** (5.22 g, 18.3 mmol), vinyl boronate **S10**[5] (6.55 g, 22.0 mmol), Na_2CO_3 (5.82 g, 54.9 mmol), and dioxane:H_2O (5:1, 48 mL) were sparged vigorously with argon for 10 min. The apparatus was opened briefly to introduce $Pd(PPh_3)_4$ (212 mg, 0.18 mmol), and sparging was continued for 5 min. The mixture was heated to reflux for 2 days, then cooled, and the volatiles were removed by rotary evaporation. The aqueous remainder was diluted, and extracted with EtOAc (x2). The combined extract was washed with brine, dried over Na_2SO_4, concentrated, reconstituted in hexanes:EtOAc (9:1), and filtered through a pad of SiO_2 rinsing with the same. The filtrate was concentrated to give 6.86 g of a red oil. This material was dissolved in THF (55 mL) and treated with Bu_4NF solution (36 mL, 36 mmol), and stirred for 30 min. The mixture was concentrated and partitioned between H_2O and EtOAc. The organic phase was washed with H_2O (x2), brine, dried over Na_2SO_4 and concentrated. Purification by column chromatography on SiO_2 eluted with 15→30% EtOAc in hexanes afforded **24** (3.37 g, 71%) as a pale yellow oil. R_f: 0.44 (7:3 hexanes : EtOAc). ^1H NMR (400 MHz, $CDCl_3$): δ 1.41 (s, 9H), 2.54 (t, *J* = 7.8 Hz, 2H), 2.90 (t, *J* = 7.8 Hz, 2H), 4.31 (br d, *J* = 5.6 Hz, 2H), 6.35 (dt, *J* = 15.9, 5.6 Hz, 1H), 6.58 (dt, *J* = 15.8, 1.4 Hz, 1H), 7.06-7.11 (m, 1H), 7.20-7.25 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ 172.4, 141.2, 136.9, 131.3, 128.8, 128.6, 127.9, 126.6, 124.5, 80.6, 63.9, 37.2, 31.2, 28.2. MS *m/z* 285.1 (calc'd: $C_{16}H_{22}NaO_3$ $[M+H]^+$, 285.3).



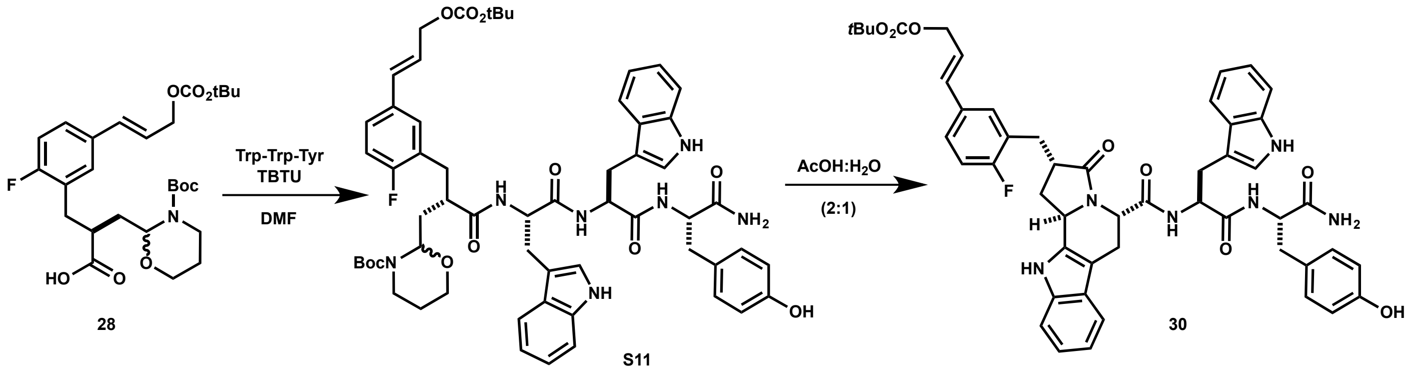
***endo*-pyrroloindoline 26:** 5-Fluoro-L-tryptophan methyl ester (59 mg, 0.25 mmol) was freshly freed from its hydrochloride, and was combined with cinnamyl alcohol **24** (72 mg, 0.28 mmol) and $Pd(PPh_3)_4$ (14 mg, 0.013 mmol). The vessel was evacuated and backfilled with argon (x3), then DCM (4.2 mL) – previously sparged with argon for 20 min – was added, and the mixture was cooled in an ice bath. Et_3B solution (300 µL, 1.0 M in hexanes) was added, and the reaction was warmed to and held at 6 ºC overnight. The reaction was quenched by addition of 5% aq. K_2CO_3 (50 mL) and extracted with DCM (x3).The combined extract was dried over K_2CO_3 and concentrated. Purification by column chromatography on SiO_2 eluted with 75→85% EtOAc in hexanes afforded **26** (96 mg, 80%) as a faintly yellow oil contaminated by ~8 mol% triphenylphosphine oxide. R_f: 0.32 (7:3 hexanes : EtOAc). ^1H NMR (400 MHz, $CDCl_3$): δ 1.40 (s, 9H), 2.39 (dd, *J* = 13.0, 7.9 Hz, 1H), 2.48 (dd, *J* = 13.0, 3.4 Hz, 1H), 2.51 (t, *J* = 8.0 Hz, 2H), 2.53 (dd, *J* = 13.5, 8.0 Hz, 1H), 2.63 (dd, *J* = 13.5, 6.6 Hz, 1H), 2.86 (t, *J* = 7.8 Hz, 2H), 3.38 (s, 1H), 3.89 (br dd, *J* = 7.3, 3.1 Hz, 2H), 4.91 (s, 1H), 6.05 (ddd, *J* = 15.4, 8.0, 7.3 Hz, 1H), 6.39 (br d, *J* = 15.7 Hz, 1H), 6.46 (dd, J_{HH} = 8.4 Hz, J_{HF} = 4.2 Hz, 1H), 6.70-6.79 (m, 2H), 7.04 (br d, *J* = 7.6 Hz, 1H), 7.11 (br s, 1H), 7.12 (br d, *J* = 7.8 Hz, 1H), 7.18 (dd, *J* = 7.8, 7.6 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$): δ 174.2, 172.3, 157.3 (d, J_{CF} = 236 Hz), 145.6, 141.2, 137.3, 133.7, 132.2 (d, J_{CF} = 9.9 Hz), 128.7, 127.5, 126.4, 125.2, 124.1, 114.7 (d, J_{CF} = 23.3 Hz), 111.0 (d, J_{CF} = 23.8 Hz), 110.2 (d, J_{CF} = 8.1 Hz), 83.2, 80.5, 59.9, 57.9 (d, J_{CF} = 1.6 Hz), 52.1, 41.9, 41.2, 37.1, 31.1, 28.2. MS *m/z* 481.2 (calc'd: $C_{28}H_{34}FN_2O_4$ $[M+H]^+$, 481.6).

Intermediate 27: Pyrroloindoline **26** (581 mg, 1.21 mmol) was dissolved in DMF, and Boc-L-phenylalanine and iPr_2EtN (505 µL, 2.9 mmol) were added. The mixture was cooled to 0 ºC, treated with HBTU (550 mg, 1.45 mmol), and allowed to warm to rt. After 40 min, the mixture was diluted with 1:1 brine : 5% aq. K_2CO_3 and extracted with EtOAc (x2). The combined extract was washed with brine, dried over Na_2SO_4 and concentrated. The residue was dissolved in THF:MeOH:H_2O (3:1:1, 12 mL) and treated with LiOH•H2O (102 mg, 2.42 mmol). The mixture was stirred for 3.5 hrs, then additional LiOH (50 mg, 1.21 mmol) was added. After 2 hrs, additional LiOH (50 mg, 1.21 mmol) was again added. The mixture was stirred for 1.5 hrs, then quenched by the addition of Et_3N•HCl (830 mg, 6.0 mmol), concentrated, and further dried in vacuo. The resulting residue was dissolved in DMF (12 mL) and treated with iPr_2EtN (843 µL, 4.84 mmol), L-threonine amide (171 mg, 1.45 mmol), and then by HBTU (550 mg, 1.45 mmol). After stirring for 1 hr, additional L-threonine amide (85 mg, 0.72 mmol) and HBTU (225 mg, 0.72 mmol) were added, and stirring continued for 2.5 hrs. The mixture was concentrated to ~4 mL by rotary evaporation, and partitioned between 5% aq. K_2CO_3 and EtOAc. The aqueous phase was extracted with EtOAc (x1), and the combined organic phase was washed sequentially with H_2O and brine, dried over Na_2SO_4 and concentrated. Purification by column chromatography on SiO_2 eluted with 0→8% MeOH in $CHCl_3$ afforded **27** (598 mg, 61%) as a white foam. An analytical sample was obtained by preparative HPLC (19x250mm C18, 40;75-100% ACN + 0.1 *v*% HCO_2H, 18 mL/min). ^1H NMR (500 MHz, DMSO-d_6, ~8:4:1 mixture of rotamers, data is of major): δ 0.66 (d, *J* = 6.1 Hz, 3H), 1.30 (s, 9H), 1.34 (s, 9H), 2.33 (dd, *J* = 13.3, 4.2 Hz, 1H), 2.49 (t, *J* = 7.8 Hz, 2H), 2.48-2.55 (m, 2H), 2.56 (dd, *J* = 13.5, 8.8 Hz, 1H), 2.66 (dd, *J* = 13.8, 6.3 Hz, 1H), 2.77 (t, *J* = 7.3 Hz, 2H), 2.88 (dd, *J* = 14.0, 11.8 Hz, 1H), 3.10 (dd, *J* = 14.0, 1.7 Hz, 1H), 3.79-3.88 (m, 2H), 4.64 (dd, *J* = 9.5, 4.5 Hz, 1H), 4.74 (ddd, *J* = 10.9, 8.1, 2.4 Hz, 1H), 4.88 (br s, 1H), 6.17 (ddd, *J* = 15.7, 7.9, 7.9 Hz, 1H), 6.22 (d, *J* = 3.8 Hz, 1H), 6.47 (d, *J* = 15.7 Hz, 1H), 6.60 (dd, J_{HH} = 8.2, J_{HF} = 4.4 Hz, 1H), 6.70-6.74 (m, 1H), 6.76 (br s, 1H), 6.78 (br dd, *J* = 8.8, 8.8 Hz, 1H), 7.03-7.09 (m, 2H), 7.11-7.27 (m, 5H), 7.26-7.36 (m, 2H), 7.44 (d, *J* = 7.9 Hz, 1H), 7.48 (apt d, *J* = 7.5 Hz, 2H). ^{13}C NMR (126 MHz, DMSO-d_6, major rotamer): δ 173.5, 171.8, 171.5, 170.5, 156.7 (d, J_{CF} = 233 Hz), 155.8, 144.9, 137.9, 136.9, 135.9, 133.6, 129.9, 128.43, 128.40 (d, J_{CF} = 20.0 Hz), 127.9, 127.2, 126.5, 126.2, 124.9, 123.6, 114.3 (d, J_{CF} = 23.2 Hz), 111.7 (d, J_{CF} = 8.0 Hz), 110.3 (d, J_{CF} = 24.0 Hz), 82.0, 79.7, 78.3, 65.5, 60.6, 57.98, 57.97, 57.5, 52.8, 36.2, 30.4, 28.1, 27.73, 27.70 (2), 19.5. ^{19}F NMR (282 MHz, DMSO-d_6, trifluoroacetate salt, mixture of rotamers): δ -73.5, -125.1 (major), -127.0 (minor). MS *m/z* 814.4 (calc'd: $C_{45}H_{57}FN_5O_8$ $[M+H]^+$, 814.4).



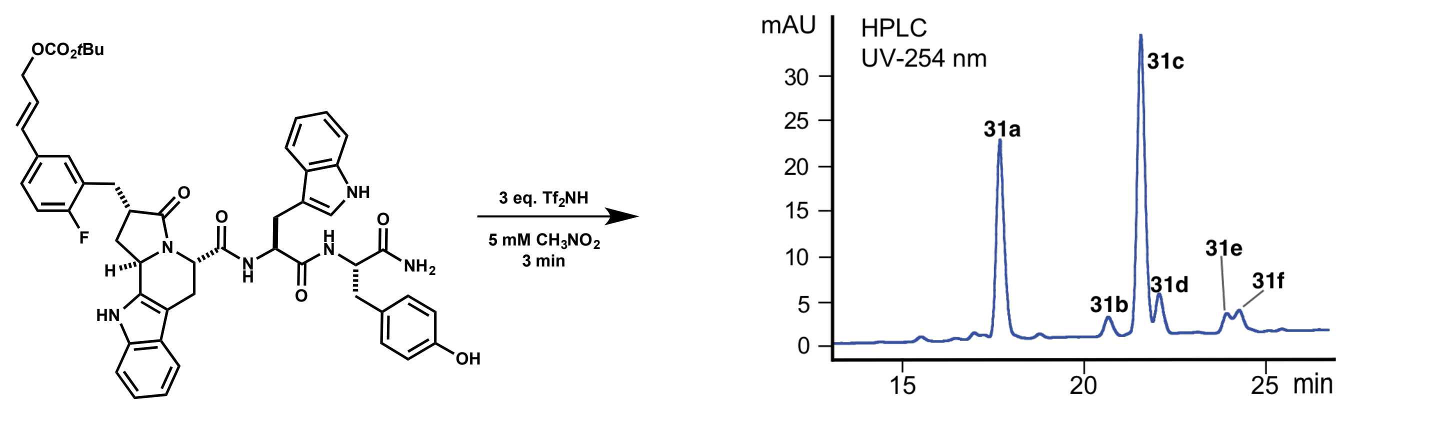
Cyclization of 27 to lactam 9d: Intermediate **27** (570 mg, 0.70 mmol) was dissolved in anhydrous DCM (7 mL) and cooled in an ice bath. Pre-cooled TFA (7 mL) was added, and the initially colorless mixture was stirred for 3.5 hours over which it turned dark pink. The mixture was then concentrated by rotary evaporation (bath 30 ºC) and further dried *in vacuo*. The resulting faintly brown residue was dissolved in DMF (10 mL) and rendered basic by the addition of iPr_2EtN (1.5 mL). This solution was added via syringe pump to a stirred solution of HBTU (1.33 g, 3.5 mmol) in DMF (130 mL) over a period of 1 hr. Stirring was continued for 20 min, and the mixture was then concentrated to ~5 mL by rotary evaporation and partitioned between 5% K_2CO_3 (aq.) and EtOAc. The aqueous phase was extracted with ethyl acetate (x2) and the combined organic phase was washed with H_2O (x1), brine, dried over Na_2SO_4 and concentrated. The resulting residue was triturated with warm MeOH and filtered to give, 169 mg of a white solid. The remaining solution was purified by column chromatography on SiO_2 eluted with 0→10% MeOH in $CHCl_3$ to give additional 112 mg. Macrocycle **9d** (combined 281 mg, 63% from **27**) obtained in this manner was spectroscopically identical to material isolated previously from acid-promoted cyclization of **7**.

***D*.4.** Reaction of trifunctional template **27** with Trp-Trp-Tyr



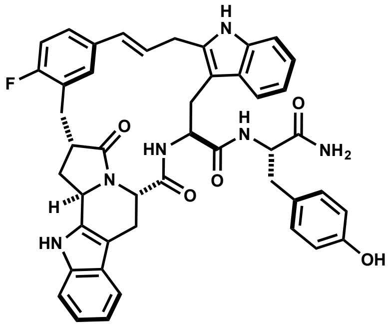
Acyclic cinnamyl carbonate (S11). Compound **S11** was prepared according to General Procedure A. The reaction was worked up by partitioning between sat. $NaHCO_3$ and EtOAc. The organic phase was then washed with sat. $NaHCO_3$, 1N HCl, H_2O, brine, dried over Na_2SO_4 and concentrated. Purification was accomplished by column chromatography on SiO_2 eluted with 0→10% MeOH in $CHCl_3$ afforded **S11** as a colorless film. A yield was not recorded. ^{1}H NMR (CD_3OD, 500 MHz, ~1:1 mixture of diastereomers): δ 1.28 (s, 9H), 1.37 (s, 9H), 1.44 (s, 9H), 1.45 (s, 9H), 1.53-1.69 (m, 2H), 1.92-2.00 (m, 1H), 2.06 (ddd, *J* = 14.0, 8.1, 5.8 Hz, 1H), 2.33-2.45 (m, 2H), 2.46-2.54 (m, 1H), 2.53-2.62 (m, 2H), 2.62-2.74 (m, 3H), 2.74-3.12 (m, 16H), 3.37-3.49 (m, 2H), 3.54-3.66 (m, 2H), 3.73-3.84 (m, 2H), 4.40 (dd, *J* = 7.8, 6.2 Hz, 1H), 4.44-4.52 (m, 3H), 4.52-4.58 (m, 2H), 4.55 (br d, *J* = 6.3 Hz, 2H), 4.60 (br d, *J* = 6.3 Hz, 2H), 5.30-5.39 (m, 1H), 5.45 (dd, *J* = 8.1, 6.4 Hz, 1H), 6.13 (dt, *J* = 15.9, 6.2 Hz, 1H), 6.17 (dt, *J* = 15.9, 6.2 Hz, 1H), 6.44 (br d, *J* = 15.9 Hz, 1H), 6.50 (br d, *J* = 15.9 Hz, 1H), 6.65 (d, *J* = 8.5 Hz, 2H), 6.70 (d, *J* = 8.4 Hz, 2H), 6.74-6.77 (m, 2H), 6.83 (s, 1H), 6.86-6.93 (m, 4H), 6.93-7.03 (m, 7H), 7.04-7.15 (m, 6H), 7.15-7.22 (m, 2H), 7.29 (apt t, *J* = 7.7 Hz, 1H), 7.32 (apt t, *J* = 7.8 Hz, 1H), 7.40-7.46 (m, 3H), 7.48 (d, *J* = 7.9 Hz, 1H). ^{13}C NMR (CD_3OD, 126 MHz, ~1:1 mixture of diastereomers): δ 177.0, 176.9, 176.5, 176.4, 176.0, 175.9, 174.38, 174.36, 174.05, 174.03, 173.4, 173.3, 163.1, 161.2, 157.2, 155.5, 154.93, 154.92, 137.92, 137.91, 137.89, 137.84, 134.03, 134.00, 133.90, 133.87, 133.75, 133.71, 131.34, 131.26, 130.8, 130.73, 130.69, 129.1, 129.0, 128.7, 128.6, 128.5, 128.0, 127.9, 127.84, 127.78, 127.3, 127.2, 124.5, 124.3, 124.1, 122.6, 122.54, 122.52, 120.0, 119.93, 119.90, 119.44, 119.38, 119.31, 119.28, 116.6, 116.5, 116.4, 116.3, 116.2, 112.4, 112.3, 110.7, 110.64, 110.57, 110.51, 83.0, 82.9, 81.7, 81.6, 68.29, 68.27, 61.0, 60.6, 56.2, 56.1, 56.0, 55.9, 55.3, 55.2, 46.0, 44.4, 44.3, 38.6, 38.4, 37.6, 32.8, 32.6, 32.1, 31.8, 30.7, 28.7, 28.61, 28.55, 28.4, 28.3, 28.2, 28.0, 26.2, 26.0. MS *m/z* 958.3 (calc'd: $C_{53}H_{61}FN_7O_9$, $[M\text{-}Boc+2H]^+$, 958.8); 940.5 (calc'd: $C_{53}H_{59}FN_7O_8$, $[M\text{-}OCO_2tBu+2H]^+$, 940.4).

Pyrrolo tetrahydro-β-carboline (30). Intermediate **S11** (147 mg, 138 μmol) was dissolved in AcOH:H_2O (2:1, 15.7 mL) and stirred at rt for 4 hr. The mixture was concentrated to give **30** (106 mg, 88%) as a colorless film. An analytical sample was obtained by preparative HPLC purification. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 1.43 (s, 9H), 1.90-2.00 (m, 2H), 2.56-2.64 (m, 2H), 2.69 (dd, *J* = 13.9, 7.9 Hz, 1H), 2.79-2.88 (m, 2H), 2.99 (dd, *J* = 14.6, 9.5 Hz, 1H), 3.05-3.13 (m, 2H), 3.20 (d, *J* = 15.6 Hz, 1H), 6.37 (dt, *J* = 15.9, 6.2 Hz, 1H), 6.65 (d, *J* = 8.5 Hz, 2H), 6.67 (br d, *J* = 16.0 Hz, 1H), 6.92-6.98 (m, H), 6.98 (d, *J* = 8.5 Hz, 2H), 6.99-7.03 (m, 1H), 7.03-7.06 (m, 2H), 7.12 (d, *J* = 2.0 Hz, 1H), 7.16 (dd, J_{HF} = 9.7, J_{HH} = 8.3 Hz, 1H), 7.21 (br d, *J* = 8.1 Hz, 1H), 7.29 (br s, 1H), 7.33 (d, *J* = 8.1 Hz, 1H), 7.36 (br d, *J* = 7.8 Hz, 1H), 7.40 (ddd, J_{HH} = 8.3, 2.0 Hz, J_{HF} = 5.0 Hz, 1H), 7.47 (dd, J_{HF} = 7.3 Hz, J_{HH} = 2.0 Hz, 1H), 7.59 (d, *J* = 7.9 Hz, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 8.15 (d, *J* = 7.8 Hz, 1H), 9.19 (br s, 1H), 10.78 (s, 1H), 10.81 (d, *J* = 1.6 Hz, 1H). MS *m/z* 765.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M\text{-}Boc+2H]^+$, 765.3).



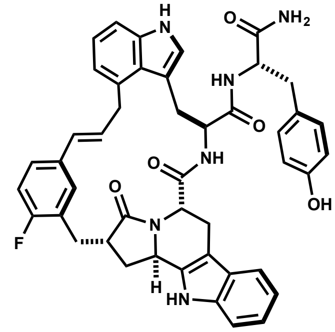
31d: This peak contained two isomeric products that were not identified

MS *m/z* 765.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 765.3).



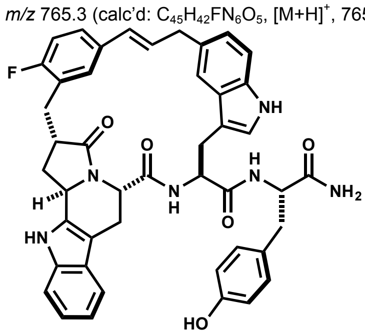
31a

MS *m/z* 765.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 765.3).



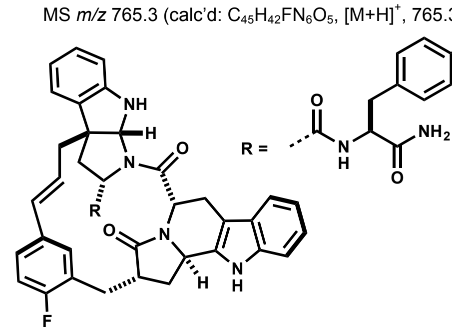
31b

MS *m/z* 765.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 765.3).



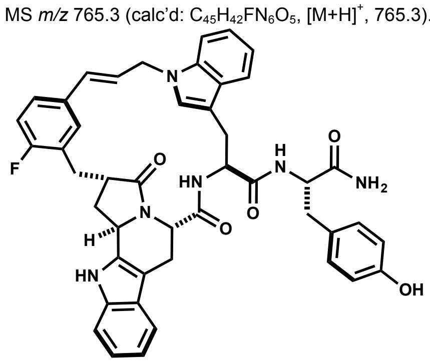
31c

MS *m/z* 765.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 765.3).



31e

MS *m/z* 765.3 (calc'd: $C_{45}H_{42}FN_6O_5$, $[M+H]^+$, 765.3).



31f

Analytical HPLC method:
Column*:* Waters XBridge™ C18, 4.6x250mm, 5µm.
Solvent A*:* H_2O + 0.1%v TFA
Solvent B*:* ACN + 0.1%v TFA
Flow rate: 1.00 ml/min

Time	%B
0	42
2	42
25	60
26	42
31	42

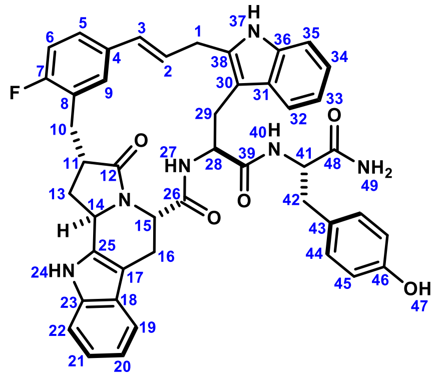
Semi-preparative HPLC method A:
Column*:* Waters XBridge™ C18, 10x250mm, 5µm.
Solvent A*:* H_2O + 0.1%v TFA
Solvent B*:* ACN + 0.1%v TFA
Flow rate: 7.00 ml/min

Time	%B
0	42
2	42
16	50
16.2	100
19	100
19.5	42

Semi-preparative HPLC method B:
Column*:* Waters XSelect™ C18, 10x250mm, 5µm.
Solvent A*:* H_2O + 0.1%v TFA
Solvent B*:* ACN + 0.1%v TFA
Flow rate: 6.00 ml/min
For re-purification of *31e*

Time	%B
0	43
1	43
31	54

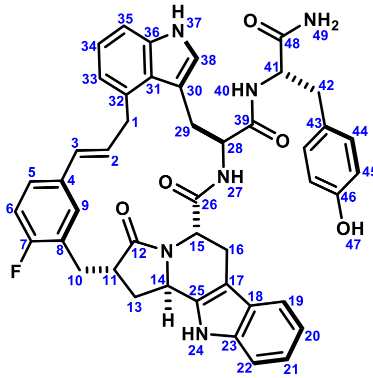
*Macrocyclic Product **31a***



(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	291	3.57 (dd, *J* = 16.8, 5.6 Hz, 1H), 3.67 (dd, *J* = 16.8, 5.1 Hz, 1H)	HMBC 1→2,3,30,38
2	126.7	6.24 (ddd, *J* = 15.8, 5.6, 5.1 Hz, 1H)	TOCSY 2→1,3 HMBC 2→4
3	128.9	5.87 (d, *J* = 15.8 Hz, 1H)	
4	133.3	-	
5	124.6	7.34-7.37 (m, 1H) overlap	HMBC 5→7 TOCSY 5→7,9
6	114.6	7.09 (dd, J_{HF} = 9.9 Hz, J_{HH} = 1.8 Hz, 1H)	HBMC 6→4,7
7	159.4 (d, *J* ≈ 240 Hz)	-	
8	123.4	-	
9	129.9	6.69 (dd, J_{HF} = 7.4 Hz, J_{HH} = 1.8 Hz, 1H)	HMBC 9→5,7
10	27.6	2.76-2.82 (m, 1H) overlap, 3.00 (dd, *J* = 13.5, 5.2 Hz, 1H)	HMBC 10→8,11,12
11	42.2	2.78-2.84 (m, 1H) overlap	
12	173.3	-	
13	29.2	1.95 (ddd, *J* = 12.7, 9.5, 9.5 Hz, 1H), 2.26 (dd, *J* = 12.7, 8.0 Hz, 1	HMBC 13→10,11,12 COSY 13→14
14	51.2	4.32 (dd, *J* = 9.5, 8.0 Hz, 1H)	HMBC 14→25
15	47.3	4.97 (d, *J* = 8.3 Hz, 1H)	HMBC 15→14,16,26 COSY 15→16
16	23.6	2.78-2.84 (m, 1H) overlap, 2.94 (d, *J* = 16.8 Hz, 1H) overlap	HMBC 16→15,17,25,26
17	102.9	-	
18	126.2	-	
19	117.8	7.33-7.36 (m, 1H) overlap	
20	118.2	6.88-6.92 (m, 1H) overlap	HMBC 20→18
21	120.2	6.95 (ddd, *J* = 7.9, 7.1, 1.0 Hz, 1H)	HMBC 21→19
22	110.4	7.17 (br d, *J* = 7.9 Hz, 1H)	HMBC 22→18
23	135.6	-	
24	-	10.81 (s, 1H)	HMBC 24→17,18,23,25
25	133.7	-	
26	169.9	-	
27	-	8.33 (d, *J* = 7.8 Hz, 1H)	
28	54.7	4.19 (ddd, *J* = 7.8, 7.8, 7.8 Hz, 1H)	HMBC 28→29,39 COSY 28→27
29	25.6	2.93-2.97 (m, 2H) overlap	HMBC 29→28,30,31,38
30	106.4	-	
31	128.8	-	
32	117.9	7.51 (d, *J* = 7.9 Hz, 1H)	HMBC 32→36
33	120.2	6.99 (ddd, *J* = 7.9, 7.0, 0.9 Hz, 1H)	
34	117.9	7.03 (ddd, *J* = 8.0, 7.0, 1.1 Hz, 1H)	HMBC 34→36
35	110.2	7.27 (d, *J* = 8.0 Hz, 1H)	
36	134.8	-	
37	-	10.89 (s, 1H)	HMBC 37→30
38	133.4	-	
39	171.3	-	
40	-	7.33-7.36 (m, 1H) overlap	HMBC 40→39
41	52.7	4.23 (ddd, *J* = 7.7, 6.2, 6.2 Hz, 1H)	HMBC 41→43 COSY 41→40
42	36.9	2.69 (dd, *J* = 13.4, 6.2 Hz, 1H), 2.77-2.81 (m, 1H) overlap	HMBC 42→43
43	126.7	-	
44	130.2	6.90 (d, *J* = 8.3 Hz, 2H)	
45	114.5	6.61 (d, *J* = 8.3 Hz, 2H)	HMBC 45→43,46
46	155.7	-	
47	-	9.14 (br s, 1H)	
48	171.8	-	
49	-	6.89 (br s, 1H) overlap, 7.29 (br s, 1H)	HMBC 49→48 TOCSY 49'→49

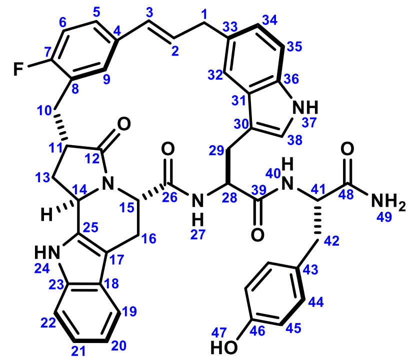
*Macrocyclic Product **31b***



(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	37	3.59-3.68 (m, 1H), 3.85-3.95 (m, 1H)	
2	130.5	6.30 (ddd, J = 15.8, 6.2, 4.9 Hz, 1H)	COSY 2→1,1' HMBC 2→4
3	128.5	5.98 (br d, J = 15.8 Hz, 1H)	
4	133.3	-	
5	125.4	7.13-7.18 (m, 1H)	HMBC 5→9
6	114.7	7.02-7.07 (m, 1H)	HMBC 6→4
7	159.7 (d, J ≈ 250 Hz)	-	
8	124.1	-	
9	129.5	6.99-7.03 (m, 1H)	HMBC 10→8,9,12
10	28.3	2.86-2.92 (m, 1H) overlap, 3.01-3.08 (m, 1H)	HMBC 11→12
11	42.2	2.87-2.94 (m, 1H) overlap	
12	173.2	-	
13	29.3	2.00 (ddd, J = 11.6, 10.1, 9.9 Hz, 1H), 2.32-2.40 (m, 1H)	COSY 13→11 TOCSY 13→10,10',11,13' HMBC 13→12
14	51.2	4.57 (apt t, J = 8.3 Hz, 1H)	HMBC 14→17 COSY 14→13
15	48.1	5.22 (d, J = 7.9 Hz, 1H)	HMBC 15→12,17,26
16	24.7	2.80-2.86 (m, 1H) overlap, 2.94 (br dd, J = 16.2, 8.1 Hz, 1H)	
17	103.1	-	
18	126.3	-	
19	110.9	7.21 (d, J = 8.1 Hz, 1H)	HMBC 19→18
20	120.7	-	
21	118.2	6.97-7.01 (m, 1H)	TOCSY 21→19,20,22
22	117.7	7.34 (d, J = 7.8 Hz, 1H)	HMBC 22→17,18,20 COSY 22→21
23	135.6	-	
24	-	10.89 (s, 1H)	HMBC 24→17,18,23,25
25	134.2	-	
26	170.1	-	
27	-	8.56 (br d, J = 5.0 Hz, 1H)	
28	52.7	4.31-4.37 (m, 1H)	HMBC 28→39 TOCSY 28→27,29
29	28.9	3.14 (dd, J = 15.5, 2.4 Hz, 1H), 3.32 (dd, J = 15.5, 10.3 Hz, 1H)	
30	111.1	-	
31	125.8	-	
32	131.2	-	
33	120.5	6.77 (d, J = 7.1 Hz, 1H)	HMBC 33→1 COSY 33→34
34	121	6.93-6.97 (m,1H) overlap	HMBC 34→32,36
35	110	7.19 (d, J = 8.2 Hz, 1H)	COSY 35→34
36	136.6	-	
37	-	10.81 (br d, J = 1.8 Hz, 1H)	HMBC 37→30,31,36,38
38	122	7.00-7.21 (m, 1H)	HMBC 38→30,36
39	170.3	-	
40	-	8.35 (br s, 1H)	
41	53.1	4.42 (ddd, J = 8.7, 7.9, 4.7 Hz, 1H)	HMBC 41→39,43,48
42	36.1	2.74 (dd, J = 14.4, 8.7 Hz, 1H), 2.84 (dd, J = 14.4, 4.7 Hz, 1H)	TOCSY 42→40,41 HMBC 42→39,43,48
43	127.8	-	
44	129.8	7.01 (d, J = 8.3 Hz, 2H)	HMBC 44→46
45	115	6.73 (d, J = 8.3 Hz, 2H)	HMBC 45→43,46
46	155.8	-	
47	-	9.28 (br s, 1H)	HMBC 47→46
48	172.8	-	
49	-	6.88 (br s, 1H), 7.31 (br s, 1H)	HMBC 49→48

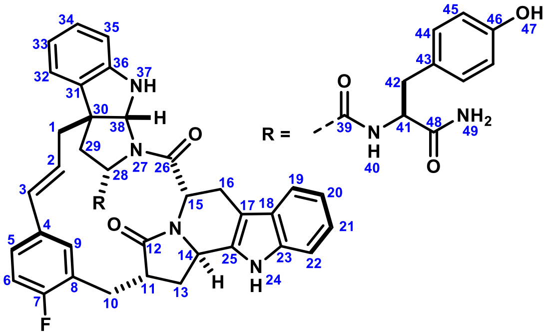
*Macrocyclic Product **31c***

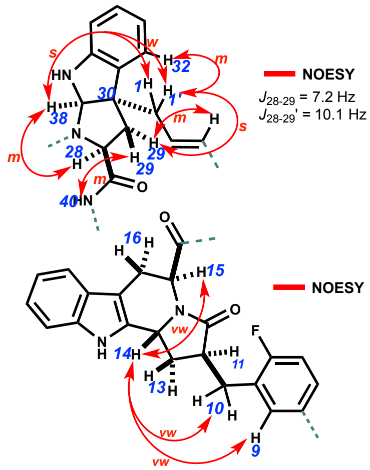


(500 MHz, DMSO-d$_6$, 298K)

	13C	1H	key correlation
1	37.5	3.54 (br dd, J = 16.6, 6.7 Hz, 1H), 3.60 (br dd, J = 16.6, 5.4 Hz,	HMBC 1→2,3,33,34
2	129.6	6.36 (ddd, J = 15.8, 6.7, 5.4 Hz, 1H)	HMBC 2→4,33 TOCSY 2→1,3
3	129.3	6.11 (br d, J = 15.8 Hz, 1H)	
4	133.6	-	
5	125.5	7.30 (ddd, JHH = 8.4, 2.0 Hz, JHF = 5.1 Hz, 1H)	TOCSY 5→6,9 HMBC 5→9
6	114.6	7.12 (dd, JHF = 10.0, JHH = 8.4 Hz, 1H)	HMBC 6→4,8
7	159.9 (d, J≈240 Hz)	-	
8	124.5	-	
9	128.8	7.00-7.03 (m, 1H) overlap	HMBC 9→3,5
10	28.4	2.92-3.00 (m, 1H) overlap	HMBC 10→8,11,12,13
11	41.3	2.81-2.87 (m, 1H)	COSY 11→13,13'
12	173.7	-	
13	29.7	2.06 (ddd, J = 12.5, 9.0, 9.0 Hz, 1H), 2.29 (ddd, J = 12.5, 7.5, 2.6 Hz, 1H)	
14	50.7	4.97 (dd, J = 9.0, 7.5 Hz, 1H)	HMBC 14→25
15	48.2	5.00 (d, J = 8.2 Hz, 1H)	HMBC 15→12,14,26
16	24.2	2.93-2.98 (m, 1H) overlap, 3.05 (br d, J = 14.8 Hz, 1H)	HMBC 16→17,25
17	103.1	-	
18	126.2	-	
19	117.4	7.34 (d, J = 7.7 Hz, 1H)	HMBC 19→23
20	118.2	6.91-6.95 (m, 1H) overlap	
21	120.4	6.97-7.00 (m, 1H) overlap	HMBC 21→23 COSY 21→22
22	110.6	7.22 (d, J = 8.0 Hz, 1H)	HMBC 22→20
23	135.8	-	
24	-	10.93 (s, 1H)	HMBC 24→17,18,23,25
25	133.5	-	
26	170.5	-	
27	-	8.28 (d, J = 8.1 Hz, 1H)	HMBC 27→26
28	52.3	4.62 (ddd, J = 8.1, 7.1, 7.1 Hz, 1H)	HMBC 28→26,29,39
29	27.9	2.88 (dd, J = 14.6, 6.8 Hz, 1H), 3.08 (dd, J = 14.6, 7.3 Hz, 1H)	HMBC 29→30
30	109	-	
31	127.2	-	
32	117.4	7.37 (br s, 1H)	TOCSY 32→34,35 HMBC 32→34
33	128.9	-	
34	122.2	6.92-6.95 (m, 1H) overlap	HMBC 34→36
35	111	7.27 (d, J = 8.2 Hz, 1H)	HMBC 35→33
36	134.9	-	
37	-	10.74 (br d, J = 1.6 Hz, 1H)	HMBC 37→30,31,36,38
38	123.9	6.98-7.00 (m, 1H) overlap	HMBC 38→36
39	170.8	-	
40	-	7.50 (br d, J = 7.6 Hz, 1H)	HMBC 40→39
41	53.3	4.17 (ddd, J = 7.6, 6.8, 6.8 Hz, 1H)	COSY 41→42 HMBC 41→39,42,43,48
42	36.3	2.46-2.56 (m, 2H) overlap	HMBC 42→41,43,48
43	127.1	-	
44	129.8	6.77 (d, J = 8.5 Hz, 2H)	HMBC 44→46
45	114.5	6.56 (d, J = 8.5 Hz, 2H)	
46	155.6	-	
47	-	9.12 (br s, 1H)	
48	172.2	-	
49	-	6.83 (br s, 1H), 7.17 (br s, 1H)	HMBC 49→48 TOCSY 49→49'

*Macrocyclic Product **31e***

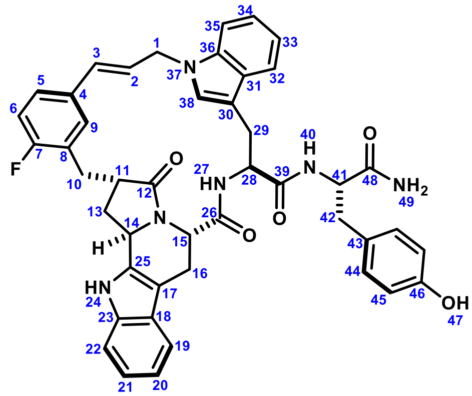




(500 MHz, DMSO-d_6, 298K)

	13C	1H	key correlation
1	38.8	2.57-2.62 (m, 1H), 2.86 (dd, J = 12.5, 10.4 Hz, 1H)	HMBC 1→30,38 ROESY 1'→29',32,38; 1→38
2	126.9	6.08-6.16 (m, 1H) overlap	HMBC 2→4,5,9
3	130	6.59 (d, J = 15.8 Hz, 1H)	TOCSY 3→1,2 HMBC 3→4 ROESY 3→29'
4	133.6	-	
5	126.6	7.06-7.10 (m, 1H) overlap	HMBC 5→2
6	114.6	7.04-7.10 (m, 1H) overlap	HMBC 6→4,8
7	159.7 (d, J ≈ 250 Hz)	-	
8	130	-	
9	128	7.45 (d, 4JHF = 7.2 Hz, 1H)	HMBC 9→4,8
10	28.7	2.93-2.99 (m, 1H) overlap, 3.02-3.08 (m, 1H) overlap	HMBC 10→11,12 TOCSY 10→11,13,13',14
11	43.2	3.00-3.06 (m, 1H) overlap	
12	173.6	-	
13	29.2	2.13-2.21 (m, 1H), 2.27-2.34 (m, 1H)	HMBC 13'→12
14	51.3	4.42 (dd, J = 8.0, 8.0 Hz, 1H)	HMBC 14→13,17,25 ROESY 14→9,10
15	45	5.66 (d, J = 7.4Hz, 1H)	HMBC 15→12,14,16,26
16	23.6	2.93-3.05 (m, 2H) overlap	HMBC 16→15,26 TOCSY 16→15,16'
17	103.2	-	
18	126.6	-	
19	118.1	7.38 (d, J = 7.6 Hz, 1H)	HMBC 19→23
20	118.5	6.95 (dd, J = 7.6, 7.0 Hz, 1H)	HMBC 20→18,22 COSY 20→19
21	120.6	6.99 (dd, J = 7.7, 7.0 Hz, 1H)	HMBC 21→19,23
22	110.7	7.22 (d, J = 7.7 Hz, 1H)	COSY 22→21 HMBC 22→17,18
23	135.7	-	
24	-	10.88 (s, 1H)	ROESY 24→22,14 HMBC 24→17,23,25
25	134.1	-	
26	169	-	
27	-	-	
28	61.8	4.10 (dd, J = 10.0, 7.1 Hz, 1H)	HMBC 28→30 TOCSY 28→29,29' ROESY 28→38
29	42	1.79 (dd, J = 13.6, 7.2 Hz, 1H), 2.45 (dd, J = 13.6, 10.2 Hz, 1H)	HMBC 29→30
30	57.3	-	
31	135.8	-	
32	122.1	7.14-7.19 (m, 1H) overlap	HMBC 32→34,36
33	119.2	6.83 (dd, J = 7.4, 7.4 Hz, 1H)	
34	128.1	7.14-7.19 (m, 1H) overlap	HMBC 34→32
35	110	6.78 (d, J = 8.1 Hz, 1H)	COSY 35→34 TOCSY 35→32,33,34
36	147.5	-	
37	-	7.33 (d, J = 4.7 Hz, 1H)	HMBC 37→31 ROESY 37→35
38	82.2	6.12 (d, J = 4.7 Hz, 1H)	
39	170	-	
40	-	7.43 (d, J = 9.0 Hz, 1H)	ROESY 40→29
41	52.1	4.10-4.16 (m, 1H)	HMBC 41→43, 48
42	38.4	2.23 (dd, J = 13.4, 8.8 Hz, 1H), 2.59 (dd, J = 13.4, 4.7 Hz, 1H) o	HMBC 42→43,48
43	126.9	-	
44	130.1	6.46 (d, J = 8.3 Hz, 2H)	HMBC 44→46
45	114.5	6.29 (d, J = 8.3 Hz, 2H)	HMBC 45→43,46
46	155.4	-	
47	-	9.01 (s, 1H)	HMBC 47→45,46
48	172.1	-	
49	-	6.68 (br s, 1H), 7.20 (br s,1 H)	HMBC 49→48 TOCSY 49→49'

*Macrocyclic Product **31f***



(500 MHz, DMSO-d_6, 298K)

	13C	1H	key correlation
1	46.1	4.72 (dd, J = 15.3, 8.0 Hz, 1H), 5.02 (dd, J = 15.3, 3.5 Hz, 1H)	HMBC 1'→38
2	124.9	6.08 (ddd, J = 15.6, 8.0, 3.5 Hz, 1H)	TOCSY 2→1,3 HMBC 2→4
3	130.8	6.40 (br d, J = 15.6 Hz, 1H)	HMBC 3→5,9
4	132.3	-	
5	127	7.25-7.29 (m, 1H) overlap	HMBC 5→9 TOCSY 5→6,9
6	120.9	7.13-7.19 (m, 1H) overlap	HMBC 6→7,8
7	160.0 (d, J≈230 Hz)	-	
8	124.3	-	
9	127.8	7.10 (br d, JHF = 6.8 Hz, 1H)	HMBC 9→10
10	26.4	2.89-2.95 (m, 1H) overlap, 2.97-3.02 (m, 1H) overlap	
11	41.4	2.76-2.81 (m, 1H) overlap	
12	174.5	-	
13	28.2	2.76-2.81 (m, 1H) overlap	HMBC 13→10,12,14 COSY 13'→11
14	50.7	4.96 (dd, J = 6.9, 6.9 Hz, 1H)	
15	48.6	5.13 (d, J = 7.7 Hz, 1H)	COSY 15→16 HMBC 15→14,26
16	24.5	2.94-3.06 (m, 2H) overlap	HMBC 16→17
17	103	-	
18	126	-	
19	117.5	7.36 (d, J = 7.7 Hz, 1H)	HMBC 19→23 COSY 19→20
20	118.3	6.93 (dd, J = 7.7, 7.0 Hz, 1H)	HMBC 20→22
21	120.4	6.99 (dd, J = 8.0, 7.0 Hz, 1H)	HMBC 21→23
22	110.5	7.22 (d, J = 8.0 Hz, 1H)	
23	135.5	-	
24	-	10.90 (s, 1H)	
25	133.4	-	
26	170.4	-	
27	-	8.53 (d, J = 8.9 Hz, 1H)	HMBC 27→26 COSY 27→28
28	52.3	4.48 (dd J = 12.5, 8.9 Hz, 1H)	COSY 28→29
29	27.5	2.85 (dd, J = 14.9, 12.5 Hz, 1H), 2.95-3.00 (m, 1H) overlap	HMBC 29→28,30,36,38
30	110.7	-	
31	127.3	-	
32	118	7.55 (d, J = 7.7 Hz, 1H)	HMBC 32→36 COSY 32→33
33	118.3	7.04-7.08 (m, 1H)	
34	120.9	7.13-7.19 (m, 1H) overlap	
35	109.5	7.50 (d, J = 8.0 Hz, 1H)	HMBC 35→31 COSY 35→34
36	135.8	-	
37	-	-	
38	124.5	7.13 (s, 1H)	HMBC 38→30,31
39	170.8	-	
40	-	7.59 (d, J = 7.4 Hz, 1H)	HMBC 40→39 COSY 40→41
41	53	4.29 (ddd, J = 7.4, 6.9, 5.9 Hz, 1H)	HMBC 41→48
42	36.6	2.67 (dd, J = 13.6, 6.2 Hz, 1H), 2.74-2.79 (m, 1H)	HMBC 42→43,44,48
43	126.6	-	
44	130	6.87 (d, J = 8.1 Hz, 2H)	HMBC 44→46
45	114.5	6.60 (d, J = 8.1 Hz, 2H)	
46	155.6	-	
47	-	9.14 (s, 1H)	HMBC 47→45,46
48	172.1	-	
49	-	7.07 (br s, 1H), 7.42 (br s, 1H)	TOCSY 49→49'

D.5. Cartesian Coordinates and Energies

endo-**S8a**

C	-3.65254	-1.89937	-1.30364
C	-5.04592	-1.87265	-1.21534
C	-5.67208	-1.11016	-0.23219
C	-4.93516	-0.35776	0.68429
H	-3.16165	-2.49622	-2.06652
H	-5.64267	-2.45273	-1.90989
H	-6.75505	-1.10162	-0.17023
H	-5.43144	0.23239	1.44747
C	-2.91538	-1.16441	-0.39134
C	-3.55309	-0.40120	0.58810
N	-2.60454	0.28788	1.37671
H	-2.86217	0.44020	2.34264
C	0.05221	1.03266	-0.33344
C	-1.31957	-0.34132	1.15404
C	-1.42475	-0.95636	-0.26333
C	-0.95287	0.22242	-1.15781
H	1.07725	0.71040	-0.55852
H	-1.83566	0.82199	-1.39512
H	-0.50429	-0.12812	-2.08958
H	-1.07029	-1.09267	1.91032
N	-0.21921	0.63291	1.07180
C	0.61154	0.89928	2.07488
O	1.52393	1.77517	1.96261
C	0.51748	0.17503	3.38150
H	1.25290	0.60148	4.06088
H	-0.47749	0.28040	3.82026
H	0.72233	-0.89108	3.25017
C	-0.57253	-2.22769	-0.44730
H	-1.00702	-3.01964	0.17237
H	-0.66708	-2.55236	-1.48915
C	0.87211	-2.01609	-0.09990
C	1.84650	-1.82457	-0.99427
C	5.90415	-0.72593	-0.21476
C	5.14560	-1.31083	0.79772
C	3.83169	-1.68920	0.55772
C	3.24980	-1.48727	-0.70109
C	4.02771	-0.91275	-1.71303
C	5.34283	-0.53116	-1.47233
H	6.93286	-0.43830	-0.02628
H	5.58694	-1.48391	1.77361
H	3.26709	-2.17653	1.34729
H	3.59649	-0.76649	-2.70004
H	5.93104	-0.08871	-2.26929
H	1.59110	-1.87991	-2.05355
H	1.10891	-1.96191	0.96268
C	0.05989	2.54748	-0.52321
O	0.75937	3.25196	0.23997
N	-0.58782	3.09381	-1.53696
H	-1.14792	2.51037	-2.13755
C	-0.52695	4.52842	-1.80230
H	0.51377	4.85242	-1.84832
H	-1.01413	4.72381	-2.75583
H	-1.03444	5.08264	-1.01033
H	1.30123	2.49564	1.15336

Electronic energy = -1206.29662
Zero-point electronic energy = -1205.838057
Enthalpy = -1205.812183
Free energy = -1205.895946
Free energy with quasiharmonic approximation = -1205.888241
Frequencies = 9.7358 18.3170 29.2224 7.7708 17.7819 29.1975
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.651967

exo-**S8b**

C	0.13178	3.07047	-1.74284
C	0.55336	4.37135	-1.46768
C	0.57434	4.83884	-0.15470
C	0.17761	4.02984	0.91015
H	0.11792	2.70129	-2.76436
H	0.86730	5.02110	-2.27656
H	0.90530	5.85195	0.04669
H	0.19246	4.40270	1.92882
C	-0.25039	2.25787	-0.68750
C	-0.22975	2.73638	0.61967
N	-0.67979	1.75229	1.53460
H	-0.14337	1.71789	2.39143
C	-2.94794	-0.20474	-0.11034
C	-0.76119	0.49343	0.82521
C	-0.79239	0.84967	-0.68533
C	-2.30489	0.84971	-1.01130
H	-3.92593	0.13862	0.24705
H	-2.50391	0.70403	-2.07566
H	-2.72819	1.82257	-0.75339
H	0.06734	-0.18206	1.06437
N	-2.02978	-0.25038	1.05573
C	-2.37207	-0.76405	2.22739
O	-3.40972	-1.49936	2.35819
C	-1.58206	-0.47944	3.46008
H	-0.51382	-0.63711	3.29006
H	-1.92377	-1.13671	4.25710
H	-1.73637	0.56368	3.74925
C	0.00213	-0.15685	-1.54282
H	-0.11608	0.13879	-2.59424
H	-0.44125	-1.15552	-1.43945
C	1.45529	-0.21252	-1.17917
C	2.04814	-1.28870	-0.65218
C	6.13462	-1.69270	0.53466
C	5.34772	-2.81718	0.30678
C	4.01748	-2.66948	-0.06907
C	3.45536	-1.39882	-0.23430
C	4.25465	-0.27476	0.01214
C	5.58261	-0.42158	0.38911
H	7.17260	-1.80530	0.82914
H	5.77035	-3.80965	0.42223
H	3.40894	-3.55155	-0.25038
H	3.83311	0.72194	-0.07763
H	6.18879	0.45849	0.57702
H	1.45651	-2.19847	-0.53317
H	2.02299	0.69872	-1.35246
C	-3.21985	-1.61320	-0.65216
O	-3.68961	-2.45801	0.13531
N	-3.02142	-1.90019	-1.92996
H	-2.62090	-1.20455	-2.53786
C	-3.34910	-3.21328	-2.47846
H	-3.21789	-3.17824	-3.55851
H	-2.69564	-3.97808	-2.05437
H	-4.38437	-3.46421	-2.24307
H	-3.69341	-1.93509	1.43346

Electronic energy = -1206.293965
Zero-point electronic energy = -1205.834231
Enthalpy = -1205.808495
Free energy = -1205.89108
Free energy with quasiharmonic approximation = -1205.884366
Frequencies = 20.3237 21.1131 27.0296 20.0715 20.6065 27.0291
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.650935

endo-**S9a**

C	0.59409	2.21537	-1.07645
C	0.33928	3.34287	-0.29516
C	-0.14201	3.23030	1.01152

C	-0.37199	1.98243	1.58888
H	-0.33696	4.12706	1.58875
H	-0.74652	1.88542	2.60194
C	0.14414	-1.24759	0.11475
C	0.44101	-0.42690	-1.09927
C	0.36448	0.96897	-0.51704
C	-0.10339	0.88550	0.79026
H	0.99257	2.31439	-2.07947
H	0.51867	4.32753	-0.71179
N	-0.24202	-0.49480	1.10221
H	-0.61064	-0.84250	1.97795
H	0.04505	-2.32360	0.14684
C	1.74465	-0.82532	-1.80664
H	1.61801	-1.82814	-2.22689
H	1.92816	-0.13551	-2.63360
C	2.95921	-0.81523	-0.87686
H	3.79430	-1.25184	-1.43822
H	3.22134	0.36237	1.38033
C	3.38691	0.64506	-0.61737
O	3.58330	1.37977	-1.57255
N	3.52598	1.01258	0.67417
C	3.83793	2.37700	1.05470
H	2.94399	2.89849	1.41081
H	4.21828	2.89119	0.17274
H	4.59950	2.38821	1.83684
N	2.63501	-1.58233	0.31259
C	3.55030	-2.26179	0.89741
C	5.00271	-2.35028	0.53559
H	5.42814	-1.35033	0.42236
H	5.54659	-2.89276	1.30715
H	5.12265	-2.87782	-0.41528
C	-0.77362	-0.65213	-2.08855
H	-0.52679	-0.07919	-2.98821
H	-0.79561	-1.71010	-2.37155
C	-2.09042	-0.22094	-1.51523
C	-2.98993	-1.07354	-1.01547
C	-6.65317	-0.09222	0.95299
C	-6.46983	-1.35001	0.38694
C	-5.27168	-1.65893	-0.24661
C	-4.24381	-0.71377	-0.33465
C	-4.43442	0.54385	0.25320
C	-5.63119	0.85259	0.88613
H	-7.58653	0.14996	1.44988
H	-7.25982	-2.09158	0.43794
H	-5.13512	-2.64021	-0.69304
H	-3.63668	1.28114	0.22914
H	-5.76712	1.83056	1.33613
H	-2.27433	0.84988	-1.47725
H	-2.80304	-2.14445	-1.11880
O	3.24607	-2.99305	1.97199
H	2.29872	-2.91197	2.13856

Electronic energy = -1206.260711
Zero-point electronic energy = -1205.801033
Enthalpy = -1205.774249
Free energy = -1205.859012
Free energy with quasiharmonic approximation = -1205.852514
Frequencies = 19.2280 22.9073 30.1530 18.9306 22.7503 29.7355
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.62461

exo-**S9b**

C	0.00459	0.55603	2.13886
C	-0.50069	1.79903	2.52378
C	-0.98593	2.71026	1.58168
C	-0.97519	2.41311	0.22050
H	-1.37843	3.66450	1.91378
H	-1.34927	3.11525	-0.51602

C	0.33276	-0.52295	-1.36394
C	0.47106	-0.98909	0.04801
C	0.02747	0.24904	0.78789
C	-0.45731	1.17882	-0.12723
H	0.37603	-0.14507	2.87923
H	-0.51969	2.06314	3.57525
N	-0.27886	0.62293	-1.42282
H	-0.53518	1.09553	-2.28064
H	0.56051	-1.09427	-2.25464
C	1.88056	-1.50084	0.37763
H	2.13356	-2.30601	-0.32108
H	1.89728	-1.92965	1.38193
C	2.92841	-0.38810	0.28204
H	2.77760	0.29429	1.12801
H	4.91294	-0.40853	-1.31057
C	4.31135	-1.01409	0.52417
O	4.50409	-1.60760	1.57292
N	5.22014	-0.85164	-0.45913
C	6.55539	-1.41253	-0.38085
H	7.30981	-0.63441	-0.52067
H	6.67333	-1.85316	0.60882
H	6.69866	-2.19059	-1.13590
N	2.76598	0.27483	-0.99732
C	2.76223	1.55356	-1.04788
C	2.94323	2.54547	0.06311
H	2.15189	2.43490	0.81083
H	2.91876	3.55786	-0.33656
H	3.90186	2.37977	0.56100
C	-0.58008	-2.15777	0.25207
H	-0.17773	-3.05159	-0.23440
H	-0.59081	-2.35073	1.32887
C	-1.95606	-1.83837	-0.25455
C	-2.89564	-1.25140	0.49360
C	-6.81855	-0.04891	-0.69695
C	-6.50151	-0.20355	0.64878
C	-5.21808	-0.58687	1.02100
C	-4.23565	-0.83355	0.05569
C	-4.56480	-0.66061	-1.29497
C	-5.84527	-0.27575	-1.66812
H	-7.81905	0.25151	-0.98948
H	-7.25418	-0.02636	1.40956
H	-4.97724	-0.71160	2.07311
H	-3.81158	-0.81784	-2.06186
H	-6.08544	-0.14651	-2.71831
H	-2.16503	-2.08667	-1.29374
H	-2.66911	-1.06103	1.54340
O	2.54828	2.14082	-2.23377
H	2.55695	1.44317	-2.90410

Electronic energy = -1206.256415
Zero-point electronic energy = -1205.797275
Enthalpy = -1205.770267
Free energy = -1205.856223
Free energy with quasiharmonic approximation = -1205.848641
Frequencies = 19.4749 23.6198 28.4571 18.2608 23.5533 28.2041
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.625014

endo-**S10a**

C	-0.61327	-2.41140	-0.28357
C	-0.38867	-3.43120	0.64432
C	0.18826	-3.16034	1.88514
C	0.55650	-1.86235	2.24211
H	0.35536	-3.96952	2.58690
H	1.00935	-1.64896	3.20386
C	0.14536	1.15725	0.33796
C	-0.32396	0.18234	-0.71414
C	-0.25845	-1.11749	0.06195

C	0.31301	-0.87504	1.30666
H	-1.04578	-2.63396	-1.25447
H	-0.65840	-4.45119	0.39456
N	0.58792	0.51701	1.38624
H	1.03799	0.96644	2.17191
H	0.40207	2.19098	0.16195
C	-1.70890	0.48451	-1.30811
H	-1.68525	1.45080	-1.82420
H	-1.90379	-0.27605	-2.07072
C	-2.88044	0.44846	-0.31333
H	-2.77420	-0.44529	0.31032
H	-4.79715	1.74151	0.69835
C	-2.80274	1.62743	0.65463
O	-1.69880	2.05514	1.03661
N	-3.95055	2.13935	1.08152
C	-4.00823	3.23866	2.03202
H	-5.05264	3.45181	2.25384
H	-3.53873	4.13189	1.61399
H	-3.49005	2.97127	2.95526
H	-4.34823	1.07337	-1.68676
N	-4.16468	0.37905	-0.97087
C	-4.90218	-0.78415	-1.19549
O	-5.77447	-0.78371	-2.03402
C	-4.58828	-1.98196	-0.32551
H	-5.31874	-2.75526	-0.55601
H	-3.58517	-2.37337	-0.52430
H	-4.64970	-1.73496	0.73865
C	0.74733	0.20492	-1.86121
H	0.41034	-0.52312	-2.60753
H	0.72101	1.19194	-2.33621
C	2.13278	-0.12116	-1.38730
C	3.09559	0.79340	-1.23707
C	7.04236	0.19290	0.28807
C	6.73108	1.33453	-0.44344
C	5.44153	1.51885	-0.92912
C	4.44578	0.56286	-0.70007
C	4.76869	-0.57561	0.05053
C	6.05628	-0.76047	0.53514
H	8.04684	0.04760	0.67104
H	7.49245	2.08270	-0.63652
H	5.20486	2.41100	-1.50274
H	4.00580	-1.31711	0.27017
H	6.29290	-1.64859	1.11197
H	2.32174	-1.16204	-1.13570
H	2.88787	1.81916	-1.54758

Electronic energy = -1206.278598
Zero-point electronic energy = -1205.818735
Enthalpy = -1205.791944
Free energy = -1205.877745
Free energy with quasiharmonic approximation = -1205.87002
Frequencies = 17.7690 20.5309 26.5737 15.961 20.3445 25.9945
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.643914

exo-**S10b**

C	0.06138	0.65067	2.04339
C	-0.43709	1.91897	2.34548
C	-0.95065	2.75677	1.35031
C	-0.97484	2.35839	0.01596
H	-1.33411	3.73422	1.61943
H	-1.36289	3.00331	-0.76394
C	0.22142	-0.71466	-1.37176
C	0.45842	-1.05450	0.06470
C	0.04494	0.24105	0.71898
C	-0.46428	1.10076	-0.24969
H	0.44942	0.00633	2.82576
H	-0.42803	2.26316	3.37377

N	-0.32967	0.44771	-1.50299
H	-0.53627	0.88455	-2.39511
H	0.40954	-1.34899	-2.23008
C	1.90339	-1.51637	0.34341
H	2.14114	-2.35601	-0.32083
H	1.97287	-1.91235	1.35862
C	2.95720	-0.41102	0.17837
H	2.78099	0.34939	0.94414
H	5.17814	0.15496	-0.97752
C	4.34891	-0.99492	0.48398
O	4.46954	-1.81950	1.37534
N	5.37111	-0.50806	-0.24494
C	6.73541	-0.94518	0.00048
H	6.86017	-2.00086	-0.25354
H	7.41158	-0.34577	-0.60831
H	6.98645	-0.81589	1.05526
H	2.81654	-0.34245	-1.93926
N	2.83565	0.24108	-1.11206
C	2.48769	1.55601	-1.35678
O	2.00916	1.85821	-2.43758
C	2.72659	2.55665	-0.25017
H	1.97039	2.46333	0.53590
H	3.71050	2.42669	0.20761
H	2.65000	3.55268	-0.68334
C	-0.54872	-2.21379	0.44722
H	-0.15780	-3.14601	0.02828
H	-0.49861	-2.30292	1.53628
C	-1.95229	-1.95432	-0.02417
C	-2.82977	-1.21312	0.65997
C	-6.71768	0.04152	-0.58080
C	-6.03290	0.72931	0.41629
C	-4.77583	0.29491	0.82073
C	-4.18031	-0.82476	0.22862
C	-4.88507	-1.51655	-0.76454
C	-6.14228	-1.08490	-1.16596
H	-7.70228	0.37314	-0.89302
H	-6.48114	1.59968	0.88374
H	-4.24366	0.83135	1.60231
H	-4.46222	-2.41365	-1.20662
H	-6.68231	-1.63521	-1.92926
H	-2.22098	-2.35323	-1.00079
H	-2.51843	-0.80980	1.62415

Electronic energy = -1206.271678
Zero-point electronic energy = -1205.812548
Enthalpy = -1205.78562
Free energy = -1205.870566
Free energy with quasiharmonic approximation = -1205.863948
Frequencies = 17.9357 23.6279 31.9068 17.6562 23.1809 30.5095
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.645547

endo-**S11a**

C	-0.50878	1.57609	-0.34865
C	-1.55569	2.21608	0.27854
C	-2.21965	1.63211	1.38185
C	-1.84693	0.39952	1.88545
H	-3.03652	2.16972	1.85133
H	-2.35254	-0.03910	2.73859
C	0.79130	-1.69865	0.61280
C	0.90649	-0.61560	-0.25342
C	-0.10546	0.31142	0.13628
C	-0.79674	-0.25527	1.23912
H	0.03938	2.04176	-1.16080
H	-1.87169	3.19411	-0.06776
N	-0.21511	-1.47541	1.50847
H	-0.44099	-2.07614	2.28368
H	1.44588	-2.55452	0.71506

C	1.96711	-0.45785	-1.30209
H	2.23313	-1.44354	-1.69935
H	1.60742	0.15509	-2.13175
C	3.25819	0.19706	-0.75830
H	4.04110	0.06857	-1.51327
H	4.44476	1.71201	0.86853
C	3.07871	1.71939	-0.62161
O	2.32861	2.32102	-1.38030
N	3.82214	2.30206	0.33720
C	3.84350	3.73726	0.54133
H	4.77611	4.17554	0.17478
H	3.73133	3.96808	1.60286
H	3.01060	4.16772	-0.01378
H	3.07501	-0.28366	1.25804
N	3.71492	-0.40961	0.48048
C	4.27544	-1.67931	0.53700
O	4.05159	-2.40138	1.49076
C	5.17753	-2.08406	-0.60584
H	4.59636	-2.29344	-1.51008
H	5.90009	-1.29959	-0.84468
H	5.70351	-2.99219	-0.31589
C	-0.63597	-2.54943	-1.46369
H	-0.19352	-1.91889	-2.22653
H	-0.14651	-3.49729	-1.27058
C	-1.86786	-2.25865	-0.93885
C	-2.47292	-1.03559	-1.23326
C	-6.06038	0.60075	0.25445
C	-5.62052	-0.63156	0.75047
C	-4.45010	-1.18809	0.27424
C	-3.69469	-0.51876	-0.71464
C	-4.14484	0.73193	-1.18741
C	-5.32418	1.28285	-0.71104
H	-6.98344	1.03021	0.62973
H	-6.20162	-1.14963	1.50492
H	-4.11639	-2.14296	0.66516
H	-3.55728	1.26039	-1.93146
H	-5.67085	2.23792	-1.08877
H	-2.32530	-2.94802	-0.23800
H	-1.96467	-0.40832	-1.96323

Electronic energy = -1206.248692
Zero-point electronic energy = -1205.791499
Enthalpy = -1205.763839
Free energy = -1205.850423
Free energy with quasiharmonic approximation = -1205.843558
Frequencies = 19.3215 28.6206 31.0126 18.1164 26.3444 30.9326
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.606497

exo-S11b

C	0.35249	1.43927	0.32179
C	1.34110	1.86757	1.17296
C	2.05123	0.95328	1.99246
C	1.77453	-0.39720	1.99265
H	2.82866	1.33210	2.64709
H	2.31904	-1.08562	2.62879
C	-0.64136	-2.05874	-0.11058
C	-0.86466	-0.71996	-0.47163
C	0.04952	0.05540	0.28033
C	0.77170	-0.83791	1.12063
H	-0.20894	2.15160	-0.27309
H	1.57826	2.92339	1.23415
N	0.28288	-2.09937	0.90506
H	0.60459	-2.93757	1.35960
H	-1.29615	-2.89334	-0.31935
C	-1.90707	-0.25059	-1.43596
H	-2.15660	-1.05260	-2.13364
H	-1.53257	0.60282	-2.01185

C	-3.21469	0.18112	-0.73533
H	-3.92329	0.46846	-1.51852
H	-4.77991	0.26877	1.22427
C	-3.85688	-1.02367	-0.02178
O	-3.63486	-2.16335	-0.41517
N	-4.67298	-0.70945	0.99992
C	-5.43535	-1.70279	1.73128
H	-5.22760	-1.63416	2.80183
H	-6.50752	-1.56789	1.56657
H	-5.14157	-2.68650	1.36636
H	-2.42539	1.16447	0.93980
N	-3.04433	1.31486	0.15026
C	-2.99579	2.63710	-0.27030
O	-2.36380	3.45401	0.37111
C	-3.77839	2.99651	-1.51329
H	-3.31085	2.57628	-2.40983
H	-4.80616	2.62672	-1.46332
H	-3.78618	4.08142	-1.60277
C	0.68405	-2.17577	-1.98058
H	0.32493	-3.18935	-2.12035
H	0.22209	-1.43039	-2.61775
C	1.95426	-1.94213	-1.44948
C	2.46743	-0.66566	-1.41065
C	5.97873	0.79728	0.44515
C	5.69100	-0.56843	0.51846
C	4.55501	-1.07203	-0.08964
C	3.68504	-0.21394	-0.79008
C	3.98570	1.15847	-0.84693
C	5.12642	1.66053	-0.23655
H	6.87303	1.18534	0.92115
H	6.36084	-1.23438	1.05095
H	4.34624	-2.13480	-0.02735
H	3.31356	1.82828	-1.37452
H	5.35123	2.71957	-0.29422
H	2.47882	-2.76410	-0.97375
H	1.88770	0.10766	-1.91207

Electronic energy = -1206.24628
Zero-point electronic energy = -1205.789749
Enthalpy = -1205.761895
Free energy = -1205.849304
Free energy with quasiharmonic approximation = -1205.841912
Frequencies = 20.2143 23.7933 32.2510 18.1639 23.1791 31.7262
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.610209

endo-**S12a**

C	-0.40653	1.22909	2.08384
C	-0.13915	2.18622	3.01285
C	0.15055	3.53157	2.61217
C	0.19361	3.93006	1.30299
H	-0.61249	0.20601	2.37915
H	-0.13768	1.94560	4.06915
H	0.35140	4.26428	3.38721
H	0.42184	4.95249	1.02659
C	-0.38883	1.59327	0.70180
C	-0.06808	2.94353	0.32482
N	-0.05979	3.04689	-1.00249
H	0.12544	3.89376	-1.51660
C	-2.28571	-0.96706	0.06393
C	-0.36785	1.77242	-1.62923
C	-0.61790	0.88060	-0.45413
C	-0.93509	-0.56614	-0.58167
H	-2.26585	-0.69658	1.12379
H	-0.13670	-1.13705	-0.09338
H	-0.94782	-0.86101	-1.63444
C	-2.12867	-3.35027	0.87623
O	-2.14029	-4.51590	0.55139

C	-1.69910	-2.87860	2.24930
H	-2.46748	-2.26612	2.73052
H	-1.51359	-3.76188	2.85777
H	-0.77260	-2.29639	2.19228
C	2.01988	0.92738	-1.75432
C	2.49908	-0.31499	-1.63968
C	5.85431	-1.66563	0.63664
C	5.68924	-0.30176	0.40191
C	4.61013	0.15431	-0.34223
C	3.67195	-0.74630	-0.86333
C	3.85618	-2.11385	-0.63003
C	4.93576	-2.57155	0.11694
H	6.70249	-2.01962	1.21289
H	6.41281	0.40727	0.79046
H	4.51130	1.21787	-0.53620
H	3.14567	-2.82743	-1.03953
H	5.06390	-3.63538	0.28607
H	1.97306	-1.11735	-2.15994
H	2.50862	1.75501	-1.24386
C	-3.40814	-0.11590	-0.55390
O	-3.15532	1.01342	-0.96725
N	-4.62816	-0.66234	-0.56340
H	-4.73159	-1.59876	-0.20177
C	-5.78117	0.06244	-1.07197
H	-5.68794	0.23119	-2.14758
H	-6.67865	-0.52308	-0.87730
H	-5.86762	1.03151	-0.57624
N	-2.55105	-2.37792	-0.02714
H	-2.68017	-2.76346	-0.95576
C	0.78314	1.26340	-2.53444
H	0.97516	2.03657	-3.28724
H	0.41920	0.38637	-3.07786
H	-1.29861	1.85617	-2.20101

Electronic energy = -1206.269077
Zero-point electronic energy = -1205.811117
Enthalpy = -1205.78361
Free energy = -1205.871697
Free energy with quasiharmonic approximation = -1205.863297
Frequencies = 15.0188 17.4051 26.4670 14.2611 16.3115 26.4469
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.635761

exo-**S12b**

C	3.87596	0.23927	-1.59853
C	5.22382	0.07271	-1.67596
C	5.91187	-0.76665	-0.73992
C	5.27787	-1.44086	0.26964
H	3.35019	0.87942	-2.29778
H	5.79795	0.57726	-2.44366
H	6.98763	-0.86939	-0.84034
H	5.82197	-2.06684	0.96662
C	3.16240	-0.44700	-0.56728
C	3.87730	-1.28179	0.36001
N	3.01900	-1.81617	1.22514
H	3.27073	-2.42034	1.99165
C	-0.17852	1.11501	-0.08441
C	1.65588	-1.39508	0.95016
C	1.83123	-0.46601	-0.21753
C	0.72058	0.20535	-0.94622
H	-0.68590	0.48405	0.65279
H	0.07489	-0.56784	-1.37827
H	1.13584	0.79002	-1.77281
C	-2.52739	1.90793	-0.64149
O	-3.26919	2.28630	-1.52294
C	-2.97654	1.62908	0.77235
H	-3.98499	2.02232	0.88617
H	-3.00794	0.55030	0.95540

H	-2.31409	2.09067	1.51071
C	-0.72560	-2.22157	0.74823
C	-1.57780	-2.28505	-0.27958
C	-5.60153	-0.87798	-0.08166
C	-5.05204	-1.54927	1.00831
C	-3.75307	-2.03963	0.94361
C	-2.98823	-1.86673	-0.21703
C	-3.56263	-1.22214	-1.31796
C	-4.85573	-0.71662	-1.24590
H	-6.61196	-0.48746	-0.02632
H	-5.63781	-1.69578	1.90959
H	-3.33567	-2.57827	1.78891
H	-2.98402	-1.08842	-2.22800
H	-5.27454	-0.18851	-2.09472
H	-1.21393	-2.62911	-1.24907
H	-1.08036	-1.85230	1.71091
C	0.71449	2.07156	0.72662
O	1.67705	1.60344	1.33423
N	0.35505	3.35985	0.72802
H	-0.43505	3.61564	0.15357
C	1.06820	4.38333	1.46992
H	0.41335	4.84549	2.21223
H	1.90843	3.90934	1.97619
H	1.44229	5.15435	0.79230
N	-1.17280	1.74979	-0.91215
H	-0.94881	1.92948	-1.88225
C	0.72018	-2.60671	0.68740
H	0.93631	-3.34737	1.46585
H	0.97566	-3.06470	-0.27331
H	1.28395	-0.81120	1.79957

Electronic energy = -1206.276962
Zero-point electronic energy = -1205.81826
Enthalpy = -1205.791216
Free energy = -1205.875586
Free energy with quasiharmonic approximation = -1205.870073
Frequencies = 21.7826 32.8790 38.6054 21.4997 32.8436 38.4241
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.63836

S13

C	3.87314	1.26551	-0.33618
C	5.12156	1.34583	0.25503
C	5.75570	0.19986	0.76947
C	5.15685	-1.04638	0.69849
H	3.39658	2.15358	-0.74101
H	5.62305	2.30551	0.32470
H	6.73369	0.29508	1.22995
H	5.64720	-1.93003	1.09471
C	3.23895	0.01723	-0.41489
C	3.89594	-1.11946	0.10322
N	3.06673	-2.19695	-0.10525
H	3.27084	-3.14765	0.14980
C	0.28332	1.43008	-0.51221
C	1.91006	-1.78036	-0.73317
C	1.97482	-0.42388	-0.94241
C	0.91510	0.45029	-1.53500
H	1.00939	2.21908	-0.28947
H	0.11614	-0.16732	-1.95600
H	1.30885	1.05992	-2.35308
C	-0.34655	1.23095	1.92826
O	-0.53980	0.48950	2.88233
C	-0.41813	2.73726	2.03738
H	-1.16075	3.14429	1.34502
H	-0.68896	2.99212	3.06057
H	0.54753	3.19180	1.79523
C	-0.43648	-2.47405	-0.26161
C	-1.58725	-2.07740	-0.81202

C	-5.11342	-0.61675	1.12264
C	-3.91804	-0.69446	1.83339
C	-2.77092	-1.19744	1.23058
C	-2.79655	-1.62177	-0.10630
C	-4.00233	-1.52706	-0.81318
C	-5.15297	-1.03957	-0.20362
H	-6.00584	-0.21973	1.59552
H	-3.86407	-0.34824	2.86025
H	-1.84879	-1.20130	1.80336
H	-4.03402	-1.83654	-1.85446
H	-6.07867	-0.98005	-0.76736
H	-1.64853	-2.04241	-1.90050
H	-0.34093	-2.55187	0.82117
C	-0.89806	2.11610	-1.21261
O	-0.70199	2.98201	-2.05311
N	-2.12339	1.65060	-0.88461
H	-2.19739	0.90784	-0.20490
C	-3.31209	2.12744	-1.55653
H	-3.43684	3.20390	-1.40946
H	-3.25431	1.94131	-2.63341
H	-4.17539	1.60333	-1.14547
N	-0.07940	0.71434	0.69120
H	0.10236	-0.27982	0.69656
C	0.81234	-2.76173	-1.05432
H	1.16954	-3.77768	-0.84577
H	0.58615	-2.72443	-2.12553

Electronic energy = -1205.914871
Zero-point electronic energy = -1205.468035
Enthalpy = -1205.441092
Free energy = -1205.525293
Free energy with quasiharmonic approximation = -1205.519686
Frequencies = 18.8574 30.2699 39.4726 16.6743 30.1767 39.3951
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.22147

endo-**S14a**

C	-0.92522	2.46514	1.20445
C	-0.71926	3.58500	0.39637
C	-0.25072	3.43226	-0.90666
C	-0.00951	2.16557	-1.44143
H	-1.28421	2.58094	2.22297
H	-0.92192	4.57666	0.78585
H	-0.08725	4.30979	-1.52419
H	0.32567	2.04334	-2.46640
C	-0.66166	1.20720	0.68994
C	-0.22526	1.06170	-0.62662
N	-0.09805	-0.29728	-0.98959
H	0.82265	-0.54579	-1.33075
C	-2.86855	-1.00670	0.81659
C	-0.53473	-1.10542	0.15562
C	-0.68520	-0.14479	1.37826
C	-2.03675	-0.51753	2.00440
H	-3.63100	-1.73387	1.10065
H	-2.52049	0.31876	2.51184
H	-1.90248	-1.33244	2.72309
H	0.18992	-1.90425	0.33315
N	-1.87180	-1.65648	-0.02511
C	-2.19194	-2.75909	-0.77935
O	-3.33206	-3.18804	-0.82629
C	-1.05173	-3.39679	-1.54713
H	-0.56011	-2.66444	-2.19344
H	-0.29821	-3.80656	-0.86646
H	-1.46238	-4.20543	-2.14937
C	0.49755	-0.27105	2.36846
H	0.30479	0.39627	3.21699
H	0.52479	-1.29660	2.75596
C	1.79616	0.08102	1.70675

C	2.68880	-0.80551	1.25828
C	6.12343	0.13698	-1.11780
C	6.11088	-1.00263	-0.31867
C	4.99151	-1.30447	0.44868
C	3.86864	-0.46958	0.44016
C	3.88540	0.66311	-0.38543
C	5.00525	0.96649	-1.15006
H	6.99561	0.37256	-1.71907
H	6.97456	-1.65964	-0.29359
H	4.98794	-2.19408	1.07300
H	3.00679	1.30074	-0.44231
H	5.00078	1.84815	-1.78344
H	2.55104	-1.86186	1.49337
H	1.95640	1.13939	1.51129
C	-3.60520	0.17458	0.16115
O	-4.36174	0.85607	0.83812
N	-3.35307	0.38819	-1.14933
H	-2.60754	-0.14327	-1.57291
C	-3.82051	1.58054	-1.81947
H	-3.05044	2.36097	-1.81780
H	-4.09752	1.35006	-2.85089
H	-4.69483	1.94900	-1.28298

Electronic energy = -1205.910736
Zero-point electronic energy = -1205.463152
Enthalpy = -1205.43725
Free energy = -1205.519795
Free energy with quasiharmonic approximation = -1205.513857
Frequencies = 21.9134 27.5511 32.7545 21.6447 26.2233 32.5417
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.212965

exo-**S14b**

C	-0.57873	3.21114	-1.62264
C	-0.46768	4.55963	-1.26976
C	-0.62483	4.94323	0.05939
C	-0.89625	4.00873	1.05986
H	-0.45243	2.90396	-2.65751
H	-0.25591	5.30425	-2.02926
H	-0.53157	5.99114	0.32761
H	-1.01974	4.31729	2.09293
C	-0.83630	2.27536	-0.63817
C	-1.00045	2.67067	0.69581
N	-1.29748	1.57550	1.49828
H	-1.03691	1.57878	2.47282
C	-2.81803	-0.84753	-0.31312
C	-0.99660	0.35576	0.76552
C	-1.02933	0.77812	-0.72937
C	-2.47176	0.39643	-1.14899
H	-3.88922	-0.87103	-0.09493
H	-2.57254	0.19990	-2.21822
H	-3.15286	1.20767	-0.88179
H	-0.03160	-0.06227	1.07331
N	-2.02675	-0.66838	0.89887
C	-2.31844	-1.14462	2.15144
O	-1.65049	-0.80599	3.11768
C	-3.48455	-2.10262	2.26311
H	-3.43801	-2.89167	1.50808
H	-4.42921	-1.56566	2.13011
H	-3.46813	-2.54212	3.25918
C	0.01961	0.06612	-1.60732
H	-0.07089	0.48040	-2.62013
H	-0.22203	-0.99857	-1.68579
C	1.42429	0.22605	-1.11229
C	2.19843	-0.79954	-0.74667
C	6.20962	-0.73781	0.75297
C	5.53353	0.46310	0.54657
C	4.23238	0.46313	0.06256

C	3.57682	-0.74029	-0.23103
C	4.26770	-1.93799	-0.01458
C	5.57055	-1.94020	0.47135
H	7.22538	-0.73395	1.13509
H	6.02292	1.40647	0.76738
H	3.72185	1.41013	-0.08037
H	3.77420	-2.88164	-0.23240
H	6.08471	-2.88278	0.63038
H	1.78670	-1.80573	-0.84159
H	1.79052	1.24876	-1.04182
C	-2.52552	-2.14425	-1.08490
O	-3.15124	-2.41038	-2.09873
N	-1.55062	-2.94063	-0.58431
H	-1.04597	-2.62533	0.22845
C	-1.16277	-4.15665	-1.26746
H	-0.27211	-4.56531	-0.78887
H	-1.96205	-4.90206	-1.22832
H	-0.94746	-3.95095	-2.31941

Electronic energy = -1205.906997
Zero-point electronic energy = -1205.459681
Enthalpy = -1205.433402
Free energy = -1205.518502
Free energy with quasiharmonic approximation = -1205.510678
Frequencies = 10.9079 20.6879 25.8092 7.4167 19.8246 25.6251
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.210815

endo-**S15a**

C	-1.03463	4.18378	6.98339
C	-1.89573	4.70232	7.96101
C	-2.41990	5.98905	7.81197
C	-2.10124	6.78301	6.70569
H	-0.62408	3.18157	7.09492
H	-2.15401	4.10553	8.83107
H	-3.09091	6.38561	8.56996
H	-2.51445	7.78205	6.59895
C	-0.72103	4.95757	5.87512
C	-1.24447	6.25423	5.73954
N	-0.73102	6.86775	4.59877
H	-1.26357	7.57666	4.10858
C	-1.35126	3.88029	2.93560
C	-0.12768	5.85865	3.74400
C	0.19464	4.66123	4.69148
C	-0.22139	3.41047	3.88555
H	-1.29789	3.33743	1.98438
H	-0.53040	2.59393	4.54290
H	0.62469	3.05410	3.28536
H	0.74394	6.27940	3.23497
N	-1.02374	5.28232	2.71740
C	-1.57092	6.12007	1.77917
O	-1.30848	7.32213	1.79051
C	-2.48531	5.51212	0.73092
H	-2.67987	6.27391	-0.02526
H	-2.04194	4.63212	0.25048
H	-3.43209	5.21227	1.19115
H	1.84948	3.79010	5.81363
H	2.33014	4.48780	4.25246
C	-2.78929	3.71312	3.50608
O	-3.47404	4.69679	3.75862
N	-3.25404	2.43271	3.68490
C	-4.57469	2.24351	4.27254
H	-4.96751	3.21720	4.56007
H	-5.25550	1.77218	3.55087
H	-4.50491	1.59678	5.15641
C	1.67031	4.61882	5.11916
H	1.94906	5.54707	5.62798
C	-2.57135	1.20674	3.30552

H	-2.32067	0.60370	4.18958
H	-3.22398	0.60231	2.66135
H	-1.65214	1.40681	2.75763

Electronic energy = -936.844819
Zero-point electronic energy = -936.484379
Enthalpy = -936.464246
Free energy = -936.530557
Free energy with quasiharmonic approximation = -936.52905
Frequencies = 40.5034 59.6520 83.5486 40.1877 59.4894 83.4812
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -937.0819949

exo-**S15b**

C	3.03630	3.57444	-0.88876
C	4.01553	3.29890	-1.85403
C	4.49228	1.99511	-2.01088
C	4.01201	0.94383	-1.22182
H	2.66512	4.58968	-0.76318
H	4.40428	4.09933	-2.47660
H	5.25455	1.78896	-2.75794
H	4.38772	-0.06749	-1.35092
C	2.55899	2.54202	-0.09405
C	3.03895	1.23251	-0.26480
N	2.36992	0.35749	0.59590
H	2.82871	-0.48422	0.92688
C	2.83422	2.87691	3.01142
C	1.73228	1.13896	1.63762
C	1.48694	2.53834	0.99245
C	1.75260	3.53952	2.13238
H	3.82456	3.15176	2.63391
H	0.83270	3.70539	2.70493
H	2.08988	4.51413	1.76931
H	0.82476	0.63369	1.98245
N	2.60224	1.43534	2.81521
C	3.31002	0.41023	3.39527
O	3.10762	-0.75866	3.06230
C	4.32914	0.76022	4.46526
H	4.92788	1.64084	4.21411
H	4.97952	-0.10572	4.59782
H	3.82744	0.96736	5.41730
H	-0.03454	3.65644	-0.09427
H	-0.69216	2.59050	1.16393
C	2.84112	3.44466	4.44906
O	3.74649	4.22650	4.73004
N	1.85932	3.11725	5.35268
C	1.88571	3.76808	6.65872
H	2.84441	4.26869	6.78127
H	1.07917	4.50990	6.74108
H	1.74820	3.01989	7.44848
C	0.08015	2.68039	0.38987
H	-0.09613	1.90778	-0.36523
C	0.65941	2.33606	5.08918
H	-0.22697	2.98348	5.01851
H	0.75854	1.76022	4.17348
H	0.49510	1.63239	5.91438

Electronic energy = -936.842958
Zero-point electronic energy = -936.482804
Enthalpy = -936.462344
Free energy = -936.53017
Free energy with quasiharmonic approximation = -936.527984
Frequencies = 41.4265 45.8845 67.3297 41.397 45.4715 67.3206
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -937.0800576

endo-**S16a**

C	4.73179	1.67609	0.38067
C	6.00386	1.38763	0.87756
C	6.21018	0.33926	1.77745

C	5.15073	-0.45521	2.21068
H	7.20921	0.14061	2.14815
H	5.30312	-1.27138	2.90771
C	1.68587	-0.22346	1.27241
C	2.19113	0.94969	0.49234
C	3.67017	0.89216	0.80064
C	3.90645	-0.14223	1.69825
H	4.58448	2.49208	-0.31948
H	6.84867	1.99068	0.56334
N	2.65346	-0.76487	1.95011
H	2.52321	-1.54112	2.58763
H	0.65184	-0.56145	1.37925
C	1.81842	0.90358	-1.00373
H	0.75864	1.14214	-1.10925
H	2.38241	1.69108	-1.51495
C	2.04502	-0.45182	-1.69640
H	1.69948	-0.34281	-2.73237
H	3.99221	0.50114	-3.07896
C	3.51418	-0.90041	-1.67880
O	3.87261	-1.82868	-0.96097
N	4.35723	-0.20023	-2.45523
C	5.78095	-0.49160	-2.46335
H	6.26555	0.13846	-3.20835
H	6.21469	-0.28924	-1.48007
H	5.95502	-1.54131	-2.70884
H	1.87903	-2.29185	-0.75493
N	1.30344	-1.50612	-1.04178
C	-0.05628	-1.59994	-1.10670
O	-0.76013	-0.72710	-1.58074
C	1.55887	2.24061	1.15731
H	1.84763	2.24892	2.21459
H	2.04963	3.09795	0.68536
C	0.07428	2.31855	0.98678
C	-0.52502	3.22398	0.20727
C	-4.67698	3.40244	-0.73641
C	-3.88135	4.53758	-0.86164
C	-2.52927	4.48038	-0.55061
C	-1.96273	3.28527	-0.09535
C	-2.77409	2.15365	0.01868
C	-4.13047	2.19604	-0.29631
H	-5.73161	3.45257	-0.99107
H	-4.31800	5.46671	-1.21213
H	-1.90918	5.36601	-0.65598
H	-2.32112	1.21313	0.32211
H	-0.52162	1.56822	1.50463
H	0.09007	3.98970	-0.26698
C	-4.98496	0.95806	-0.14539
H	-5.09606	0.73820	0.92434
H	-5.99737	1.14876	-0.51119
C	-4.41725	-0.27225	-0.88519
H	-3.32827	-0.32671	-0.79247
H	-4.64242	-0.19371	-1.94979
C	-3.47409	-1.58462	1.60331
H	-3.58613	-0.49898	1.68829
C	-0.61914	-2.91103	-0.53682
N	-4.48661	-2.14525	0.72810
H	-5.05271	-2.89779	1.09823
N	-1.89633	-2.67221	0.08861
H	-2.74088	-3.04853	-0.32063
H	0.07800	-3.29087	0.22085
C	-5.06516	-1.54571	-0.38357
O	-6.04256	-2.04589	-0.90136
C	-2.04690	-1.79451	1.09124
O	-1.10272	-1.19577	1.61535
C	-3.59764	-2.18802	3.00418
H	-2.82241	-1.78123	3.65460

H	-4.57487	-1.95076	3.42986
H	-3.48441	-3.27612	2.96644
C	-0.77283	-3.93603	-1.66051
H	-1.16697	-4.87587	-1.26687
H	-1.45367	-3.55549	-2.42693
H	0.19225	-4.13962	-2.12914

Electronic energy = -1738.935518
Zero-point electronic energy = -1738.296163
Enthalpy = -1738.259123
Free energy = -1738.364203
Free energy with quasiharmonic approximation = -1738.356846
Frequencies = 21.1605 27.6667 29.6960 19.5334 27.5609 29.6271
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1739.434113

endo-**S16a-2**

C	1.67011	1.77407	-1.21336
C	2.05380	2.48436	-2.35274
C	3.35537	2.96196	-2.50625
C	4.32862	2.72967	-1.53364
H	3.62257	3.51153	-3.40186
H	5.34653	3.08294	-1.65574
C	3.99196	0.84470	1.50200
C	2.54095	0.87035	1.11774
C	2.62312	1.55373	-0.23055
C	3.92120	2.01718	-0.42219
H	0.66349	1.38592	-1.11246
H	1.32448	2.66451	-3.13439
N	4.69030	1.60274	0.70229
H	5.68019	1.77710	0.81177
H	4.39232	0.49864	2.44394
C	1.85023	-0.49777	1.10923
H	1.94877	-0.96945	2.09342
H	0.78340	-0.33496	0.94573
C	2.32129	-1.45345	0.00982
H	2.31641	-0.91271	-0.94360
H	3.35458	-3.64425	-0.72015
C	3.74697	-1.92802	0.24012
O	4.56439	-1.20611	0.83335
N	4.07884	-3.12259	-0.24859
C	5.42547	-3.65751	-0.13543
H	5.47790	-4.59586	-0.68570
H	6.14985	-2.95400	-0.55129
H	5.68030	-3.83997	0.91129
H	1.24617	-3.12292	0.66767
N	1.40169	-2.56342	-0.16269
C	0.24116	-2.31067	-0.87186
O	0.08910	-1.31876	-1.55584
C	1.84314	1.81318	2.18217
H	1.95830	1.34837	3.16765
H	2.37338	2.77110	2.19728
C	0.39687	2.02495	1.85547
C	-0.03465	3.08053	1.15831
C	-3.93471	3.71612	-0.44659
C	-3.03038	4.76984	-0.35374
C	-1.76617	4.55960	0.18013
C	-1.39288	3.29032	0.63668
C	-2.31151	2.24262	0.52889
C	-3.58330	2.43983	-0.00503
H	-4.91925	3.88385	-0.87310
H	-3.31275	5.75694	-0.70434
H	-1.05989	5.38227	0.24631
H	-2.01921	1.24320	0.84536
H	-0.29360	1.24105	2.15712
H	0.68488	3.85999	0.90680
C	-4.54944	1.28235	-0.09313
H	-5.51225	1.61771	-0.48790

H	-4.75170	0.91307	0.92016
C	-4.03188	0.12820	-0.97754
H	-2.97948	-0.08547	-0.76504
H	-4.10231	0.40991	-2.02930
C	-3.52451	-1.73734	1.25978
H	-3.43773	-0.67447	1.50592
C	-0.80452	-3.43169	-0.80451
N	-4.54996	-1.94741	0.25588
H	-5.24725	-2.65756	0.43635
N	-2.06000	-2.89777	-0.32305
H	-2.91572	-3.07936	-0.82775
H	-0.45510	-4.20589	-0.10851
C	-4.89480	-1.10301	-0.78471
O	-5.86156	-1.34674	-1.47864
C	-2.12429	-2.16022	0.80211
O	-1.12585	-1.85708	1.45393
C	-3.87782	-2.49420	2.54150
H	-3.08840	-2.35738	3.28150
H	-4.81778	-2.11879	2.95195
H	-3.98386	-3.56540	2.34233
C	-0.99210	-4.03427	-2.19359
H	-1.29780	-3.25681	-2.89751
H	-0.05798	-4.47535	-2.54786
H	-1.74998	-4.82137	-2.16986

Electronic energy = -1738.930536
Zero-point electronic energy = -1738.290076
Enthalpy = -1738.25321
Free energy = -1738.358399
Free energy with quasiharmonic approximation = -1738.350313
Frequencies = 20.4109 22.7503 31.2793 20.1752 22.1862 31.0461
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1739.427098

exo-**S16b**

C	0.81462	0.70652	1.34595
C	0.64773	0.16980	2.62335
C	1.71850	0.09870	3.51865
C	2.98747	0.55724	3.16720
H	1.56230	-0.32726	4.50322
H	3.82005	0.50574	3.86024
C	4.05905	1.87685	0.00081
C	2.60205	1.73581	-0.32072
C	2.07544	1.15257	0.97307
C	3.11985	1.06943	1.88829
H	-0.02019	0.74978	0.65485
H	-0.31934	-0.23301	2.90615
N	4.28597	1.58180	1.24899
H	5.19049	1.66851	1.69410
H	4.80011	2.36923	-0.61090
C	2.31183	0.89208	-1.57859
H	1.23545	0.92167	-1.75840
H	2.79329	1.37286	-2.43618
C	2.69905	-0.59206	-1.58884
H	2.44321	-0.94442	-2.59552
H	3.90188	-2.79280	-1.70289
C	4.19823	-0.82502	-1.39014
O	4.96710	0.11184	-1.14657
N	4.60309	-2.09636	-1.49662
C	5.99029	-2.51288	-1.37972
H	6.09378	-3.27486	-0.60406
H	6.35186	-2.91488	-2.32898
H	6.58551	-1.64072	-1.11349
H	2.32757	-1.54257	0.27171
N	1.97268	-1.44995	-0.66919
C	0.64775	-1.74438	-0.87147
O	0.03964	-1.36864	-1.85732
C	2.08544	3.21373	-0.54906

H	2.33588	3.81023	0.33482
H	2.63603	3.62693	-1.40240
C	0.61185	3.25281	-0.79702
C	-0.28424	3.67289	0.10012
C	-4.50101	3.24809	-0.36761
C	-3.98578	4.35236	0.30781
C	-2.61439	4.49858	0.47111
C	-1.74024	3.55284	-0.07549
C	-2.27417	2.45402	-0.75299
C	-3.64903	2.27962	-0.89834
H	-5.57573	3.12939	-0.47036
H	-4.66099	5.09616	0.71771
H	-2.21691	5.35396	1.00934
H	-1.60505	1.69156	-1.14883
H	0.26204	2.88631	-1.75915
H	0.06330	4.10773	1.03748
C	-4.17184	1.00700	-1.51863
H	-5.23393	1.09551	-1.75869
H	-3.65658	0.79231	-2.45952
C	-3.95898	-0.15850	-0.55137
H	-4.50172	0.03391	0.38278
H	-2.89980	-0.22839	-0.27449
C	-3.67037	-2.59625	1.02961
H	-4.14530	-1.77621	1.57717
C	0.04194	-2.64534	0.20289
N	-4.09269	-2.60274	-0.35963
H	-4.51053	-3.45156	-0.71744
N	-1.39399	-2.61872	0.12377
H	-1.85097	-2.77753	-0.76383
H	0.29537	-2.24993	1.19254
C	-4.39788	-1.49215	-1.12324
O	-4.95194	-1.60816	-2.19832
C	-2.16090	-2.39476	1.21872
O	-1.70297	-2.10940	2.31821
C	-4.06338	-3.91331	1.70291
H	-3.73679	-3.90700	2.74346
H	-5.14756	-4.04392	1.67270
H	-3.59170	-4.76132	1.19568
C	0.58986	-4.07488	0.07251
H	0.15208	-4.70177	0.85159
H	0.33294	-4.49937	-0.90219
H	1.67749	-4.09619	0.18393

Electronic energy = -1738.924463
Zero-point electronic energy = -1738.284835
Enthalpy = -1738.247625
Free energy = -1738.353741
Free energy with quasiharmonic approximation = -1738.345321
Frequencies = 19.5015 27.7561 30.4260 19.4041 27.7026 30.4214
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1739.427349

endo-**S17a**

C	-5.26283	1.04540	-1.79876
C	-6.44796	0.35163	-2.05337
C	-6.54230	-1.00484	-1.75348
C	-5.46701	-1.69787	-1.19530
H	-7.46712	-1.53564	-1.95538
H	-5.54758	-2.75469	-0.96123
C	-2.05283	-0.47789	-0.61406
C	-2.82273	0.85322	-0.84144
C	-4.18801	0.36355	-1.25504
C	-4.29490	-0.99565	-0.95410
H	-5.18356	2.10363	-2.03016
H	-7.29648	0.86929	-2.48730
N	-3.10244	-1.48395	-0.37663
H	-2.85193	-2.41838	-0.67461
H	-1.44768	-0.75316	-1.48354

C	-2.84376	1.49560	0.57239
H	-2.72498	2.57915	0.51934
H	-3.80443	1.29556	1.05245
C	-1.69481	0.85605	1.38008
H	-0.87784	1.55340	1.57193
H	-2.85905	-1.33608	1.87185
C	-2.19662	0.38247	2.74793
O	-2.15245	1.12514	3.71454
N	-2.73882	-0.86206	2.75935
C	-3.38245	-1.39248	3.93904
H	-3.01462	-0.83632	4.80164
H	-3.13972	-2.45121	4.06182
H	-4.47224	-1.28174	3.89407
N	-1.20524	-0.22695	0.52833
C	0.04726	-0.71397	0.75994
O	0.74295	-0.28792	1.66906
C	-2.12778	1.76044	-1.88251
H	-2.25950	1.31449	-2.87482
H	-2.64207	2.72784	-1.89232
C	-0.66881	1.94358	-1.57919
C	-0.15172	2.95383	-0.87678
C	3.85811	3.01807	0.58251
C	2.83479	3.63173	1.29684
C	1.53937	3.64500	0.79591
C	1.24795	3.02537	-0.42321
C	2.29239	2.43953	-1.14390
C	3.59738	2.42683	-0.65489
H	4.86415	2.97916	0.98610
H	3.04627	4.09376	2.25564
H	0.73982	4.11183	1.36475
H	2.08640	1.99725	-2.11657
H	-0.00941	1.12683	-1.87204
H	-0.81107	3.75356	-0.53800
C	4.69445	1.75945	-1.45286
H	4.60565	2.04862	-2.50541
H	5.67178	2.09497	-1.09828
C	4.63845	0.23171	-1.36909
H	5.31831	-0.20871	-2.11053
H	3.63283	-0.12635	-1.61431
C	4.24417	-2.58835	-0.63876
H	4.69429	-2.42674	-1.62372
C	0.53892	-1.88270	-0.09205
N	4.70926	-1.57954	0.29652
H	5.08214	-1.89492	1.18162
N	1.98082	-1.87116	-0.02108
H	2.39770	-1.33323	0.72947
H	0.25554	-1.78022	-1.14400
C	5.05396	-0.28242	0.00111
O	5.66419	0.39824	0.80664
C	2.72767	-2.58936	-0.88243
O	2.25536	-3.25151	-1.79913
C	4.65235	-3.97879	-0.14881
H	4.29387	-4.73076	-0.85256
H	5.73949	-4.05312	-0.06593
H	4.20850	-4.18321	0.83132
C	-0.03799	-3.19599	0.45922
H	0.31898	-4.02696	-0.15151
H	0.29112	-3.33352	1.49236
H	-1.13127	-3.18905	0.45392

Electronic energy = -1738.545204
Zero-point electronic energy = -1737.917456
Enthalpy = -1737.881177
Free energy = -1737.985514
Free energy with quasiharmonic approximation = -1737.977293
Frequencies = 24.5910 28.6105 31.2155 24.4002 28.5094 31.1584
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1738.993872

exo-**S17b**

C	5.80838	0.49731	0.01094
C	6.94395	-0.25664	-0.29519
C	6.80938	-1.48682	-0.93403
C	5.55535	-1.99498	-1.27385
H	7.69612	-2.06590	-1.17256
H	5.45726	-2.95840	-1.76351
C	2.26425	-0.38987	-0.87592
C	3.19257	0.62284	-0.13962
C	4.56063	0.00796	-0.33293
C	4.43447	-1.23237	-0.96438
H	5.90622	1.46118	0.50334
H	7.92917	0.11813	-0.03996
N	3.09379	-1.54917	-1.17471
H	2.84279	-2.07717	-1.99675
H	1.80370	-0.00072	-1.78527
C	2.71576	0.59762	1.33659
H	3.54021	0.40297	2.02309
H	2.27932	1.56367	1.60093
C	1.63042	-0.49416	1.45890
H	2.04307	-1.40868	1.89645
H	-0.34440	1.00659	0.87370
C	0.51379	-0.03119	2.40348
O	0.52258	-0.37144	3.57558
N	-0.41037	0.78812	1.85527
C	-1.57392	1.23747	2.59369
H	-1.27696	1.49744	3.61120
H	-1.98546	2.11816	2.09650
H	-2.33185	0.44847	2.64280
N	1.20635	-0.70770	0.07582
C	0.19011	-1.50910	-0.35554
O	-0.03272	-1.66510	-1.55276
C	3.10451	2.05733	-0.73260
H	3.64277	2.73898	-0.06453
H	3.62779	2.06473	-1.69435
C	1.68342	2.49470	-0.91467
C	0.97579	3.24547	-0.06646
C	-3.26045	3.81117	-0.20765
C	-2.45820	4.68472	0.52245
C	-1.07982	4.51210	0.55648
C	-0.47945	3.46706	-0.15470
C	-1.30400	2.59934	-0.87939
C	-2.68872	2.75416	-0.91677
H	-4.33830	3.94619	-0.22042
H	-2.91293	5.50188	1.07333
H	-0.46055	5.18799	1.13910
H	-0.86529	1.75618	-1.41017
H	1.17744	2.08666	-1.78756
H	1.47606	3.70444	0.78611
C	-3.52379	1.75368	-1.67830
H	-4.54603	2.12269	-1.80131
H	-3.12383	1.61468	-2.68647
C	-3.54703	0.39308	-0.97916
H	-4.07779	0.45917	-0.02224
H	-2.52495	0.08061	-0.73001
C	-4.41498	-2.19830	0.14931
H	-5.05286	-1.46400	0.65428
C	-0.63884	-2.28921	0.66541
N	-4.33026	-1.92630	-1.27604
H	-4.77934	-2.58236	-1.90064
N	-1.97825	-2.41285	0.13636
H	-2.06826	-2.49166	-0.86878
H	-0.73080	-1.78880	1.62878
C	-4.14198	-0.69322	-1.86050
O	-4.38874	-0.52214	-3.04083

C	-3.06842	-2.12051	0.88126
O	-3.03841	-1.86408	2.07662
C	-5.00974	-3.58972	0.36936
H	-5.07584	-3.79290	1.43930
H	-6.01298	-3.65173	-0.06147
H	-4.37692	-4.35380	-0.09254
C	0.02764	-3.65544	0.87404
H	0.08955	-4.19484	-0.07497
H	1.03896	-3.53941	1.27443
H	-0.56218	-4.23997	1.58269

Electronic energy = -1738.549453
Zero-point electronic energy = -1737.922092
Enthalpy = -1737.885601
Free energy = -1737.989463
Free energy with quasiharmonic approximation = -1737.98221
Frequencies = 22.8308 28.1311 34.0100 22.6648 28.0433 33.944
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1738.991441

endo-**TS-1a**

C	-0.80996	2.31968	1.03054
C	-0.65629	3.41859	0.18236
C	-0.19357	3.26089	-1.12405
C	0.11689	1.99843	-1.63285
H	-0.07807	4.13127	-1.76027
H	0.47375	1.87105	-2.64908
C	-0.22269	-1.18370	0.01070
C	-0.47791	-0.29706	1.21175
C	-0.49662	1.06356	0.54037
C	-0.04852	0.93061	-0.77081
H	-1.18806	2.44935	2.03868
H	-0.90002	4.41034	0.54611
N	0.17675	-0.44982	-1.01401
H	0.62265	-0.81115	-1.84407
H	0.08002	-2.22039	0.08974
C	-1.75670	-0.70110	1.94874
H	-1.57748	-1.63690	2.48733
H	-2.04734	0.05872	2.67637
C	-2.88072	-0.90211	0.93258
H	-3.68946	-1.46621	1.40990
H	-3.05402	0.21182	-1.39818
C	-3.50395	0.46268	0.56296
O	-3.90873	1.17636	1.46322
N	-3.59709	0.76194	-0.75274
C	-4.06855	2.06512	-1.18912
H	-4.80486	2.42123	-0.46945
H	-4.53500	1.97502	-2.17106
H	-3.24821	2.78828	-1.23680
N	-2.28956	-1.64016	-0.17108
C	-2.98206	-2.44061	-0.89782
C	-4.45312	-2.69140	-0.78370
H	-4.99575	-1.74332	-0.77925
H	-4.79129	-3.30787	-1.61499
H	-4.66914	-3.21200	0.15402
C	0.75947	-0.41646	2.16865
H	0.53648	0.22109	3.03093
H	0.81459	-1.44777	2.53526
C	2.05272	-0.01240	1.52543
C	2.96962	-0.88626	1.10020
C	6.60417	-0.02739	-0.97902
C	6.42187	-1.25391	-0.34822
C	5.23225	-1.52188	0.31952
C	4.21050	-0.56763	0.37582
C	4.39972	0.65699	-0.27874
C	5.58829	0.92577	-0.94420
H	7.53205	0.18439	-1.49954
H	7.20657	-2.00239	-0.37377

H	5.09767	-2.47886	0.81661
H	3.60910	1.40189	-0.27877
H	5.72326	1.88049	-1.44190
H	2.20446	1.05399	1.37800
H	2.80998	-1.94631	1.30779
O	-2.40786	-3.14138	-1.87113
H	-1.45893	-2.97182	-1.89212

Electronic energy = -1206.259324
Zero-point electronic energy = -1205.800186
Enthalpy = -1205.774216
Free energy = -1205.85643
Free energy with quasiharmonic approximation = -1205.850854
Frequencies = -105.5577 15.9972 24.8230 -105.5538 15.7851 24.6182
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.624359

exo-**TS-1b**

C	0.21651	1.32591	-2.06797
C	0.89194	2.54708	-2.01779
C	1.41602	3.02888	-0.81800
C	1.27180	2.31529	0.37245
H	1.94213	3.97681	-0.80605
H	1.68325	2.68582	1.30470
C	-0.48721	-0.78346	0.87967
C	-0.49835	-0.77267	-0.63565
C	0.06792	0.60757	-0.89343
C	0.58727	1.11716	0.29287
H	-0.17476	0.94921	-3.00786
H	1.01476	3.12719	-2.92540
N	0.29935	0.18665	1.32435
H	0.63521	0.27467	2.27168
H	-0.63837	-1.67639	1.47269
C	-1.90530	-1.01601	-1.17437
H	-2.19287	-2.05593	-0.98110
H	-1.95461	-0.85649	-2.25296
C	-2.86112	-0.06712	-0.44821
H	-2.73347	0.93980	-0.86296
H	-4.47076	-1.39932	1.04564
C	-4.31325	-0.46230	-0.75471
O	-4.76898	-0.18678	-1.84883
N	-4.98019	-1.12309	0.22157
C	-6.31700	-1.65289	0.01331
H	-6.94750	-1.43611	0.87792
H	-6.73455	-1.16968	-0.86943
H	-6.29438	-2.73336	-0.15549
N	-2.45644	-0.10545	0.95024
C	-2.83379	0.81353	1.76881
C	-3.73135	1.96610	1.46395
H	-3.35581	2.52122	0.60038
H	-3.80276	2.62712	2.32604
H	-4.72724	1.59207	1.20850
C	0.50058	-1.87229	-1.15421
H	0.03684	-2.85175	-0.99776
H	0.57625	-1.72115	-2.23528
C	1.85583	-1.81316	-0.50755
C	2.83625	-1.01062	-0.93150
C	6.59849	-0.32404	0.96736
C	6.03102	0.64351	0.14347
C	4.81475	0.39648	-0.48285
C	4.14300	-0.81545	-0.28576
C	4.72941	-1.78617	0.53543
C	5.94686	-1.54091	1.15760
H	7.55269	-0.13806	1.44879
H	6.53963	1.58841	-0.01691
H	4.37292	1.15159	-1.12798
H	4.24623	-2.75031	0.66323
H	6.39660	-2.30567	1.78222

H	1.99880	-2.41181	0.39048
H	2.65236	-0.39318	-1.81096
O	-2.41369	0.78014	3.02982
H	-1.95998	-0.05495	3.19925

Electronic energy = -1206.255067
Zero-point electronic energy = -1205.796521
Enthalpy = -1205.770159
Free energy = -1205.854413
Free energy with quasiharmonic approximation = -1205.847407
Frequencies = -131.4952 20.4898 24.2649 -131.4931 20.1645 23.7061
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.621767

endo-**TS-2a**

C	-0.37530	2.37230	0.01152
C	0.65410	3.24023	0.34921
C	1.95626	3.05850	-0.14144
C	2.26894	2.00066	-0.98418
H	2.73322	3.76089	0.13977
H	3.27457	1.85350	-1.36215
C	0.01954	-0.54140	-2.16966
C	-0.86586	0.17885	-1.35028
C	-0.08447	1.29945	-0.83788
C	1.22829	1.14025	-1.31368
H	-1.37068	2.53506	0.41352
H	0.44532	4.07905	1.00371
N	1.24564	-0.00353	-2.11874
H	2.04961	-0.33976	-2.62600
H	-0.20675	-1.40553	-2.77921
C	-2.36199	0.15763	-1.58170
H	-2.64720	1.18095	-1.85001
H	-2.56908	-0.47548	-2.44601
C	-3.30493	-0.30451	-0.46171
H	-4.32102	-0.18366	-0.85181
H	-3.96230	-1.55095	1.61851
C	-3.15773	-1.81790	-0.21613
O	-2.68744	-2.54287	-1.08521
N	-3.58396	-2.24336	0.98879
C	-3.61471	-3.64578	1.35933
H	-3.10139	-3.80101	2.31117
H	-3.10938	-4.20983	0.57585
H	-4.64337	-4.00514	1.44674
H	-2.31565	0.48847	1.21942
N	-3.21830	0.47373	0.75831
C	-3.87603	1.67808	0.98006
O	-3.39126	2.48923	1.74526
C	-5.18928	1.89813	0.26795
H	-5.85950	1.04051	0.37093
H	-5.65827	2.78089	0.69890
H	-5.02721	2.07804	-0.79986
C	-0.17867	-1.41263	0.23532
H	-1.06833	-1.15104	0.79128
H	-0.27643	-2.31868	-0.35219
C	1.06548	-0.96954	0.69798
C	2.22772	-1.47800	0.17920
C	6.22027	-0.26898	0.91908
C	5.17235	0.46881	1.47061
C	3.85938	0.08655	1.24741
C	3.57609	-1.04917	0.46395
C	4.64454	-1.77741	-0.09111
C	5.95628	-1.39302	0.13792
H	7.24643	0.03273	1.10012
H	5.38496	1.34315	2.07576
H	3.05389	0.67503	1.67220
H	4.43577	-2.65769	-0.69307
H	6.77321	-1.96504	-0.28731
H	1.08964	-0.15698	1.41630

H	2.13422	-2.31268	-0.51596

Electronic energy = -1206.241391
Zero-point electronic energy = -1205.783924
Enthalpy = -1205.757261
Free energy = -1205.840946
Free energy with quasiharmonic approximation = -1205.835262
Frequencies = -163.9362 20.1600 26.8721 -163.9346 18.3189 26.6842
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.600955

exo-**TS-2b**

C	-0.88818	2.56401	-0.47963
C	-2.16825	3.04645	-0.68008
C	-3.14464	2.27200	-1.33845
C	-2.86601	0.99685	-1.79699
H	-4.13483	2.68681	-1.49203
H	-3.62072	0.39741	-2.29392
C	0.28032	-0.74121	-1.51215
C	0.61489	0.46230	-0.87860
C	-0.57300	1.27470	-0.94105
C	-1.57686	0.51367	-1.57338
H	-0.14302	3.17987	0.01408
H	-2.42440	4.04389	-0.34020
N	-1.00963	-0.70667	-1.91175
H	-1.48717	-1.46456	-2.37175
H	0.91075	-1.60053	-1.68761
C	1.97905	1.02845	-0.57098
H	1.83128	1.96593	-0.02582
H	2.44645	1.30478	-1.52557
C	3.01946	0.22518	0.24169
H	2.58655	-0.04860	1.20802
H	5.13466	-0.31781	-1.00357
C	3.38055	-1.11139	-0.43124
O	2.59725	-2.06112	-0.36811
N	4.55315	-1.14052	-1.07812
C	5.05548	-2.31937	-1.75801
H	5.98913	-2.65742	-1.30226
H	4.30715	-3.10583	-1.66586
H	5.23033	-2.10762	-2.81554
H	4.20974	1.96221	-0.06346
N	4.16275	1.09024	0.44737
C	5.05307	1.04831	1.51224
O	5.86503	1.93709	1.65291
C	4.95359	-0.14606	2.43432
H	4.03215	-0.10306	3.02450
H	4.95707	-1.09117	1.88370
H	5.80266	-0.11762	3.11483
C	0.02908	-0.95408	1.06915
H	0.85174	-1.64943	0.92424
H	0.26072	-0.00353	1.53858
C	-1.29801	-1.35183	0.97537
C	-2.30694	-0.43742	1.19411
C	-6.48833	-0.88170	0.77507
C	-5.93626	0.36194	1.07006
C	-4.56271	0.48485	1.21791
C	-3.72341	-0.63496	1.06403
C	-4.29907	-1.88644	0.76494
C	-5.66969	-2.00541	0.62592
H	-7.56269	-0.98324	0.66398
H	-6.57663	1.22850	1.18976
H	-4.12176	1.45042	1.44561
H	-3.67466	-2.76721	0.65854
H	-6.11164	-2.97095	0.40713
H	-1.51944	-2.36062	0.64357
H	-2.00028	0.56715	1.48446

Electronic energy = -1206.243785
Zero-point electronic energy = -1205.786809

Enthalpy = -1205.759795
Free energy = -1205.844962
Free energy with quasiharmonic approximation = -1205.838393
Frequencies = -46.9291 17.1590 20.5421 -46.8605 16.7567 19.7092
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.602557

endo-**TS-3a**

C	0.61426	1.60939	0.29633
C	1.68252	2.20851	-0.32260
C	2.30190	1.62084	-1.45692
C	1.85428	0.43870	-2.00578
H	3.14870	2.12472	-1.91058
H	2.32636	0.00427	-2.87967
C	-0.80006	-1.62013	-0.68873
C	-0.87286	-0.51897	0.18245
C	0.14322	0.37627	-0.22069
C	0.77325	-0.18386	-1.36873
H	0.10324	2.07201	1.13322
H	2.06354	3.15229	0.05072
N	0.13080	-1.35295	-1.66840
H	0.36361	-1.97362	-2.42570
H	-1.54050	-2.39847	-0.83233
C	-1.86326	-0.36052	1.29481
H	-2.08214	-1.33984	1.73452
H	-1.46319	0.28372	2.08019
C	-3.20081	0.25060	0.81503
H	-3.92138	0.16018	1.63477
H	-4.54878	1.66068	-0.77576
C	-3.04632	1.76270	0.57303
O	-2.23135	2.40785	1.21973
N	-3.88303	2.28380	-0.34348
C	-3.93933	3.70458	-0.62930
H	-4.85297	4.15130	-0.22749
H	-3.90039	3.87552	-1.70733
H	-3.07860	4.17613	-0.15627
H	-3.17956	-0.33626	-1.18310
N	-3.74395	-0.43472	-0.34546
C	-4.26236	-1.72188	-0.27485
O	-4.07341	-2.50449	-1.18836
C	-5.07148	-2.07083	0.95214
H	-4.42222	-2.20369	1.82403
H	-5.79838	-1.29016	1.18985
H	-5.58779	-3.01029	0.76198
C	0.50292	-2.70787	0.87758
H	0.06146	-2.29781	1.77867
H	0.08065	-3.64528	0.53284
C	1.79374	-2.33827	0.50727
C	2.37680	-1.21228	1.05068
C	6.05286	0.66414	0.12718
C	5.68628	-0.48834	-0.57300
C	4.49108	-1.12320	-0.28572
C	3.64101	-0.61635	0.71736
C	4.02209	0.55069	1.40484
C	5.22055	1.18497	1.11413
H	6.99357	1.15463	-0.09980
H	6.34210	-0.88733	-1.33879
H	4.21964	-2.01989	-0.83262
H	3.36550	0.95498	2.16915
H	5.50749	2.08000	1.65444
H	2.28927	-2.88885	-0.28541
H	1.82060	-0.71209	1.84183

Electronic energy = -1206.247976
Zero-point electronic energy = -1205.790976
Enthalpy = -1205.763991
Free energy = -1205.84912
Free energy with quasiharmonic approximation = -1205.842646

Frequencies = -183.9717 20.6420 24.0857 -183.9699 19.3061 23.9699
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.605127

exo-**TS-3b**

C	0.35249	1.43927	0.32179
C	1.34110	1.86757	1.17296
C	2.05123	0.95328	1.99246
C	1.77453	-0.39720	1.99265
H	2.82866	1.33210	2.64709
H	2.31904	-1.08562	2.62879
C	-0.64136	-2.05874	-0.11058
C	-0.86466	-0.71996	-0.47163
C	0.04952	0.05540	0.28033
C	0.77170	-0.83791	1.12063
H	-0.20894	2.15160	-0.27309
H	1.57826	2.92339	1.23415
N	0.28288	-2.09937	0.90506
H	0.60459	-2.93757	1.35960
H	-1.29615	-2.89334	-0.31935
C	-1.90707	-0.25059	-1.43596
H	-2.15660	-1.05260	-2.13364
H	-1.53257	0.60282	-2.01185
C	-3.21469	0.18112	-0.73533
H	-3.92329	0.46846	-1.51852
H	-4.77991	0.26877	1.22427
C	-3.85688	-1.02367	-0.02178
O	-3.63486	-2.16335	-0.41517
N	-4.67298	-0.70945	0.99992
C	-5.43535	-1.70279	1.73128
H	-5.22760	-1.63416	2.80183
H	-6.50752	-1.56789	1.56657
H	-5.14157	-2.68650	1.36636
H	-2.42539	1.16447	0.93980
N	-3.04433	1.31486	0.15026
C	-2.99579	2.63710	-0.27030
O	-2.36380	3.45401	0.37111
C	-3.77839	2.99651	-1.51329
H	-3.31085	2.57628	-2.40983
H	-4.80616	2.62672	-1.46332
H	-3.78618	4.08142	-1.60277
C	0.68405	-2.17577	-1.98058
H	0.32493	-3.18935	-2.12035
H	0.22209	-1.43039	-2.61775
C	1.95426	-1.94213	-1.44948
C	2.46743	-0.66566	-1.41065
C	5.97873	0.79728	0.44515
C	5.69100	-0.56843	0.51846
C	4.55501	-1.07203	-0.08964
C	3.68504	-0.21394	-0.79008
C	3.98570	1.15847	-0.84693
C	5.12642	1.66053	-0.23655
H	6.87303	1.18534	0.92115
H	6.36084	-1.23438	1.05095
H	4.34624	-2.13480	-0.02735
H	3.31356	1.82828	-1.37452
H	5.35123	2.71957	-0.29422
H	2.47882	-2.76410	-0.97375
H	1.88770	0.10766	-1.91207

Electronic energy = -1206.249609
Zero-point electronic energy = -1205.792502
Enthalpy = -1205.765618
Free energy = -1205.850501
Free energy with quasiharmonic approximation = -1205.843929
Frequencies = -184.8306 17.4524 21.0985 -184.8301 15.7357 20.9413
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.607549

endo-**TS-4a**

C	-3.38162	-1.74318	-1.42529
C	-4.61838	-2.32287	-1.22461
C	-5.13545	-2.49005	0.07520
C	-4.43757	-2.08525	1.20035
H	-6.10981	-2.95027	0.19820
H	-4.84575	-2.21785	2.19574
C	-1.17443	-0.54920	1.24548
C	-1.37440	-0.68195	-0.15221
C	-2.65621	-1.32004	-0.30173
C	-3.19339	-1.49244	0.98882
H	-2.98263	-1.62019	-2.42696
H	-5.20273	-2.65776	-2.07376
N	-2.28232	-0.99005	1.89947
H	-2.40598	-0.95463	2.89808
H	-0.44417	0.07689	1.73525
C	-0.61343	0.00002	-1.24971
H	0.29822	-0.55703	-1.50394
H	-1.23809	0.00174	-2.14621
C	-0.20193	1.45753	-0.92633
H	0.18207	1.89490	-1.85993
H	-1.52075	3.16107	-2.28089
C	-1.45696	2.22732	-0.46936
O	-1.92623	2.06608	0.64904
N	-2.01211	3.01399	-1.41347
C	-3.22511	3.77567	-1.17076
H	-3.00042	4.83093	-0.99276
H	-3.70831	3.36450	-0.28546
H	-3.89805	3.69078	-2.02603
H	1.38680	0.58204	0.11646
N	0.81801	1.41804	0.09206
C	1.35679	2.43166	0.85879
O	2.25561	2.16246	1.63692
C	0.78724	3.82034	0.71575
H	-0.15677	3.88931	1.26295
H	1.50007	4.52239	1.14523
H	0.59628	4.08448	-0.32821
C	-0.03200	-2.14229	0.69609
H	-0.45623	-2.64062	1.56333
H	-0.28460	-2.67779	-0.21632
C	1.34645	-1.69776	0.78439
C	2.21406	-1.83605	-0.24502
C	6.28370	-0.61684	-0.40214
C	5.80115	-1.58044	-1.28492
C	4.46767	-1.96080	-1.23022
C	3.60507	-1.39721	-0.27806
C	4.09806	-0.41658	0.59818
C	5.42912	-0.03093	0.52993
H	7.32425	-0.31350	-0.44961
H	6.46350	-2.02988	-2.01663
H	4.09101	-2.71086	-1.92039
H	3.44149	0.09272	1.29830
H	5.79691	0.73671	1.20171
H	1.68114	-1.25819	1.72074
H	1.86192	-2.35409	-1.13882

Electronic energy = -1206.234977
Zero-point electronic energy = -1205.777476
Enthalpy = -1205.750778
Free energy = -1205.834634
Free energy with quasiharmonic approximation = -1205.828743
Frequencies = -376.2497 24.4078 29.3241 -376.2487 24.0023 29.2599
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.595886

exo-**TS-4b**

C	-3.85106	-0.40460	1.77292
C	-5.20189	-0.67632	1.86433

C	-5.94380	-1.04412	0.72463
C	-5.36293	-1.14781	-0.52822
H	-7.00320	-1.24975	0.83250
H	-5.94345	-1.43001	-1.39912
C	-1.88989	-0.57338	-1.31773
C	-1.88938	-0.27727	0.06667
C	-3.24074	-0.49729	0.51360
C	-4.00163	-0.86084	-0.61425
H	-3.28007	-0.12424	2.65210
H	-5.70308	-0.60736	2.82270
N	-3.15917	-0.86465	-1.71286
H	-3.42904	-1.07043	-2.66088
H	-1.13263	-0.29601	-2.03478
C	-0.81710	0.37665	0.88078
H	-1.31801	1.07642	1.55985
H	-0.33520	-0.37649	1.51774
C	0.30419	1.12641	0.13404
H	0.75679	0.44779	-0.59413
H	0.56411	3.59838	0.58288
C	-0.27429	2.28757	-0.69780
O	-0.92678	2.03546	-1.70682
N	-0.02743	3.52160	-0.23139
C	-0.49245	4.72275	-0.89906
H	-1.06062	4.41896	-1.77779
H	-1.13618	5.30730	-0.23738
H	0.35246	5.34064	-1.21279
H	1.05533	1.47376	2.09424
N	1.29693	1.51288	1.11285
C	2.66369	1.63748	0.90766
O	3.40232	1.76638	1.86224
C	3.13991	1.61401	-0.52448
H	4.18139	1.92998	-0.53789
H	3.08219	0.59634	-0.92543
H	2.54523	2.27139	-1.16504
C	-1.06778	-2.21456	-0.41514
H	-1.32524	-2.40858	0.62324
H	-1.70080	-2.79144	-1.08381
C	0.35213	-2.16190	-0.70302
C	1.28721	-2.14649	0.27044
C	5.45169	-1.44031	-0.20513
C	4.83488	-1.23940	1.02533
C	3.48361	-1.52530	1.17475
C	2.72600	-1.98310	0.08841
C	3.36234	-2.19243	-1.14598
C	4.71618	-1.92712	-1.28685
H	6.50827	-1.22575	-0.32394
H	5.39761	-0.85040	1.86572
H	3.00364	-1.35540	2.13403
H	2.80561	-2.58658	-1.99029
H	5.20322	-2.10072	-2.24020
H	0.64948	-2.08692	-1.74592
H	0.95233	-2.22013	1.30546

Electronic energy = -1206.237886
Zero-point electronic energy = -1205.780815
Enthalpy = -1205.75399
Free energy = -1205.837376
Free energy with quasiharmonic approximation = -1205.83244
Frequencies = -361.4792 32.3351 35.1546 -361.4778 32.2412 35.0046
SCF (ωB97x-D/6-311+G(d,p)–SMD (nitromethane)) = -1206.602148

On the prevalence of bridged macrocyclic pyrroloindolines formed in regiodivergent alkylations of tryptophan

Tristin E. Rose,[†] Brice H. Curtin,[†] Kenneth V. Lawson,[†] Adam Simon, K. N. Houk, Patrick G. Harran
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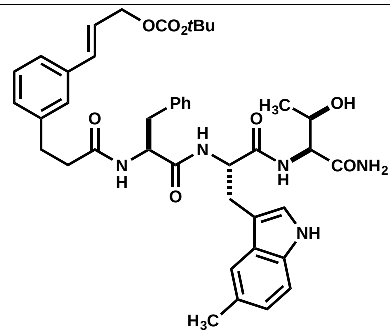
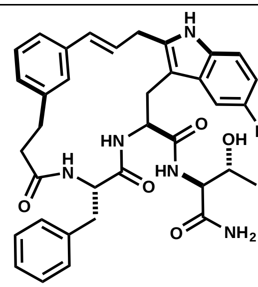
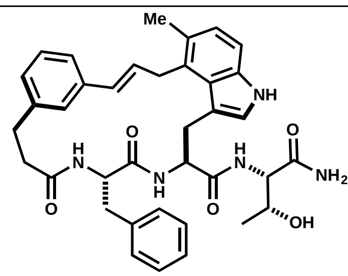
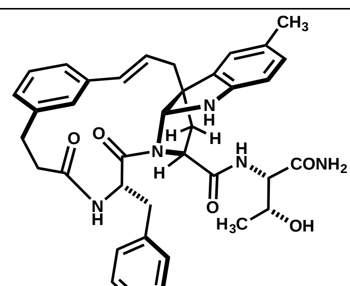
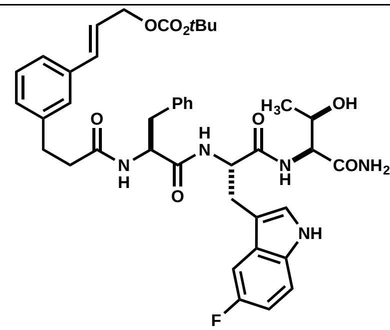
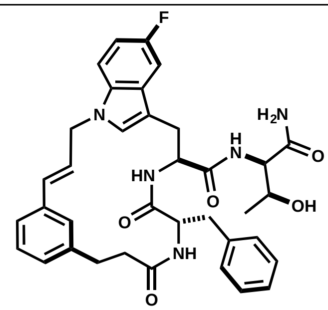
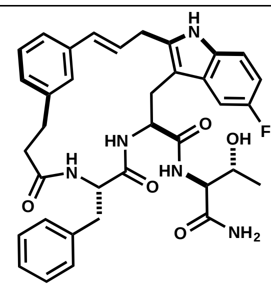
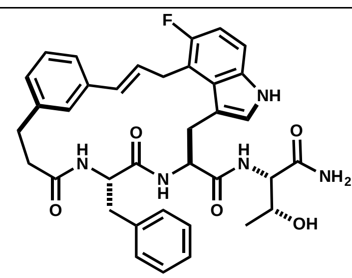
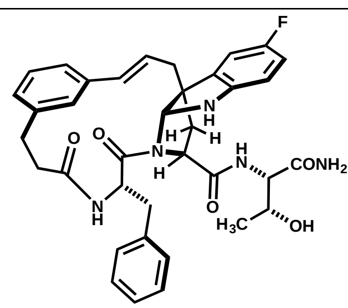
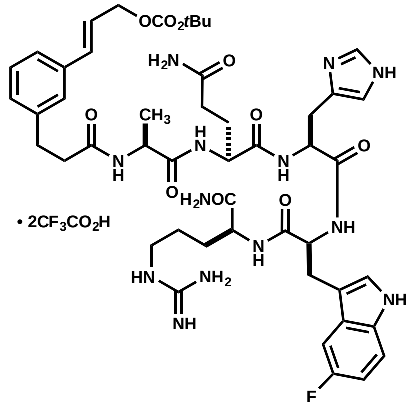
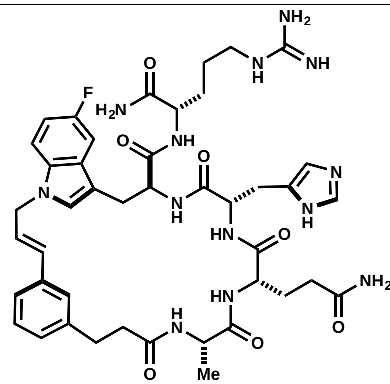
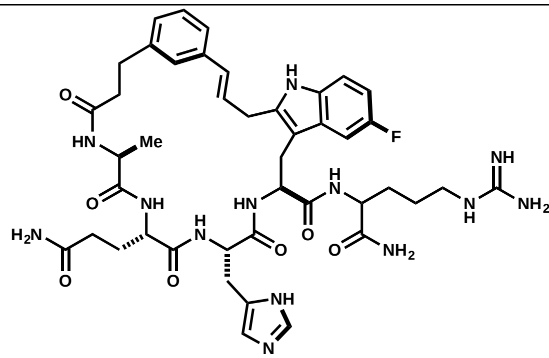
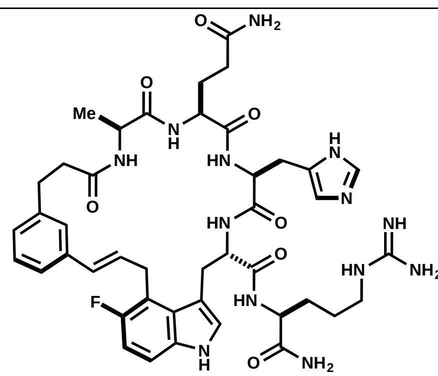
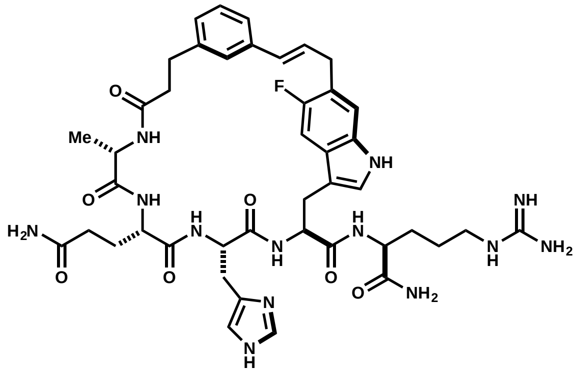
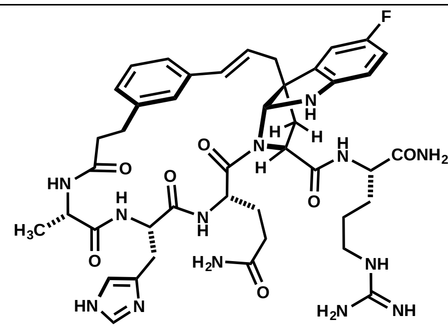
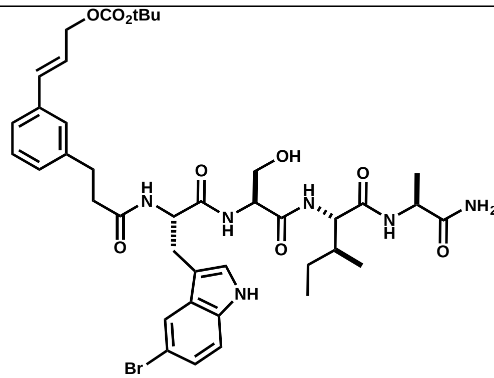
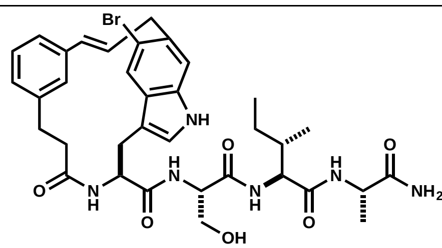
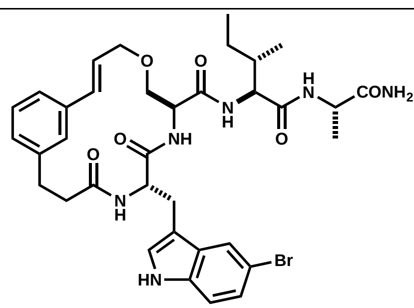
607 Charles E. Young Drive East, Los Angeles, CA 90095-1569

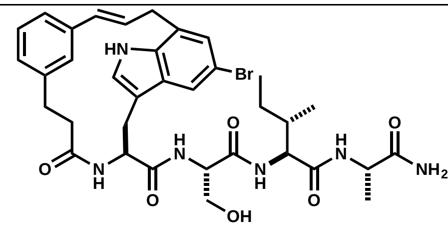
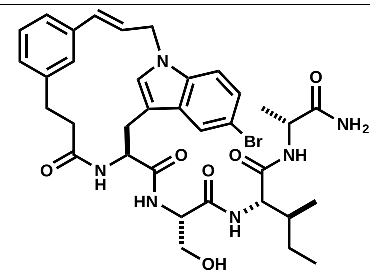
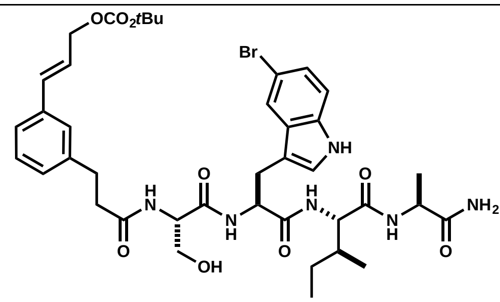
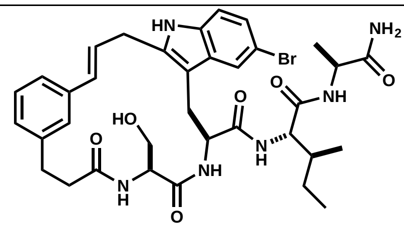
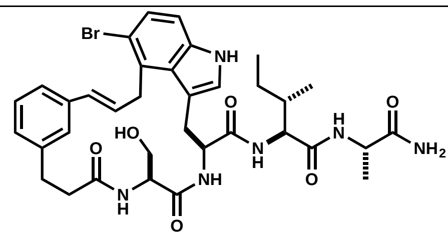
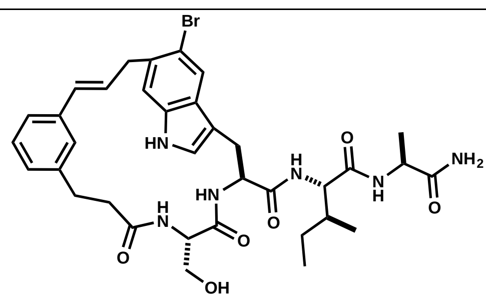
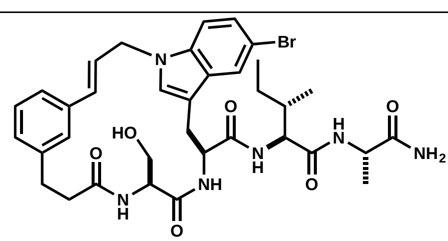
NMR Structural Data

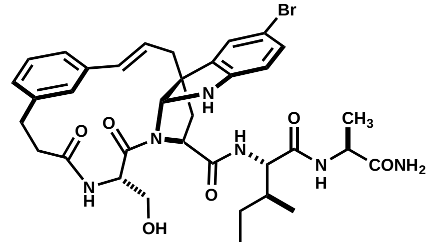
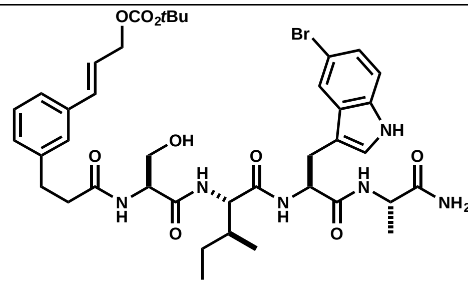
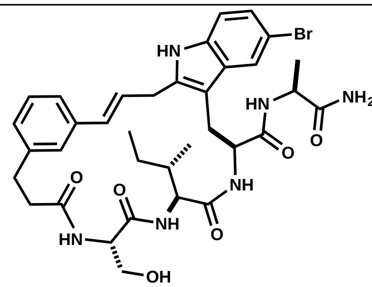
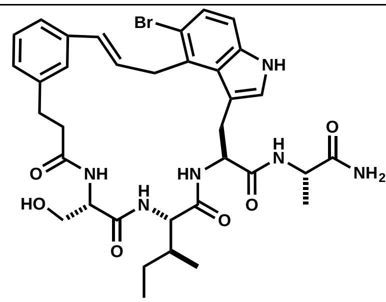
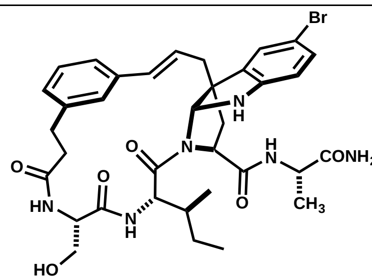
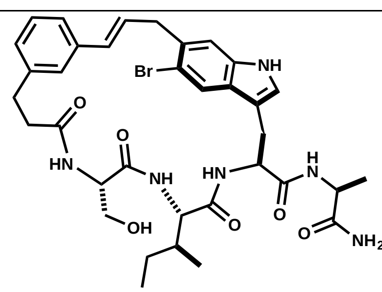
Compound No.	***Structure***	***Page #***
6		*13*
8b		*14*
8c		*17*
8d		*19*

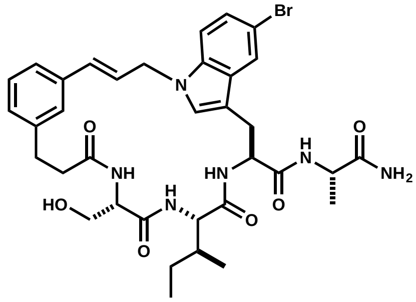
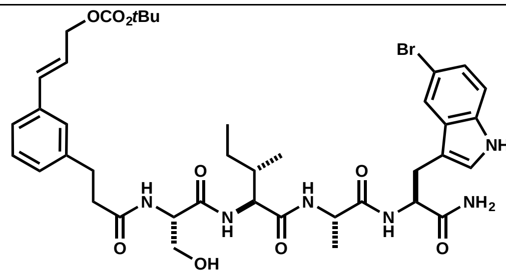
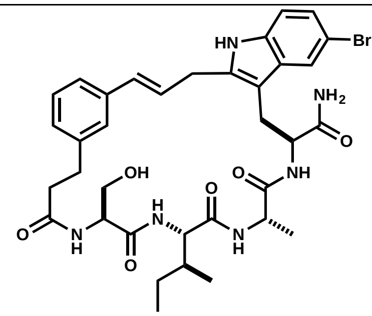
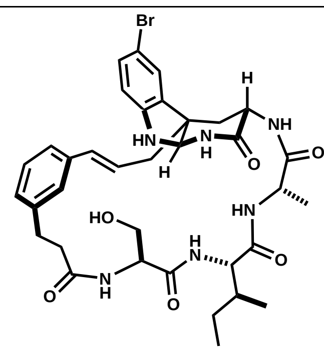
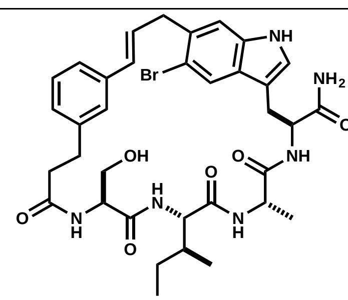
7		*22*
9a		*23*
9b		*26*
9c		*29*
9d		*32*

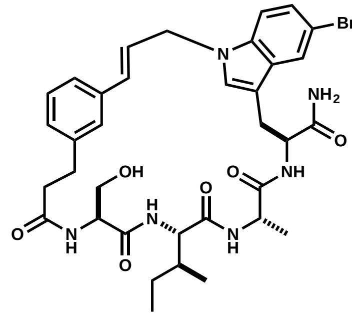
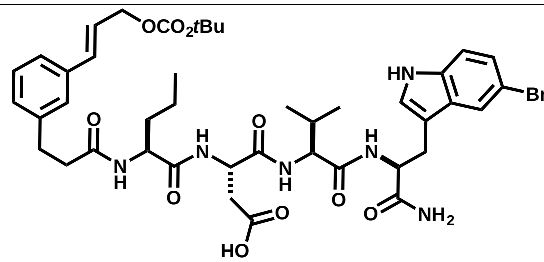
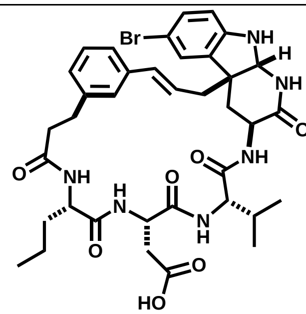
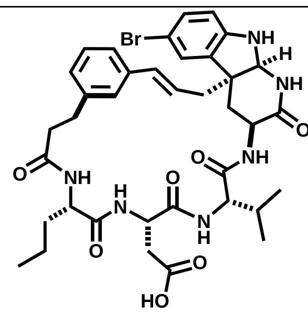
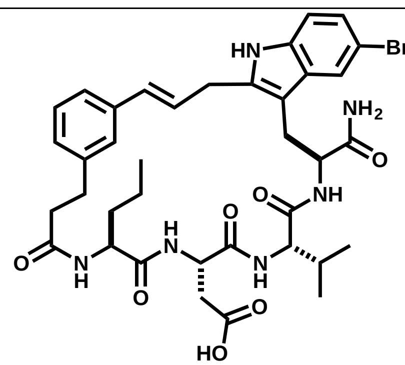
10		*35*
11a		*36*
11b		*39*
11c		*42*

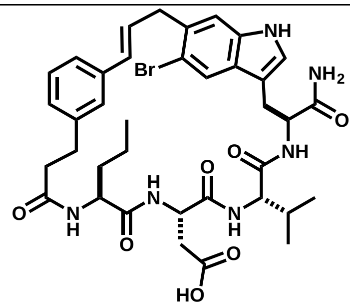
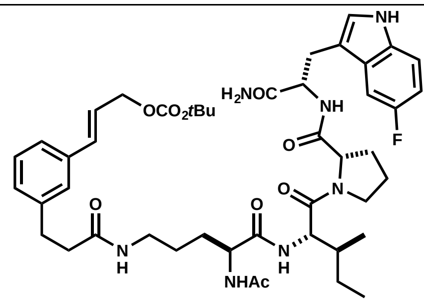
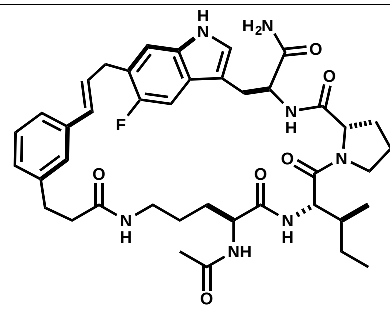
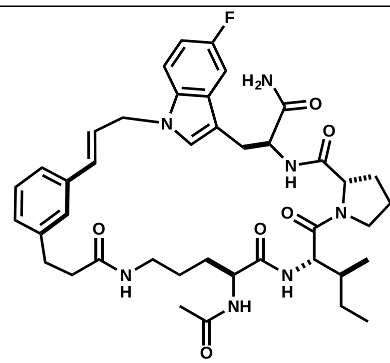
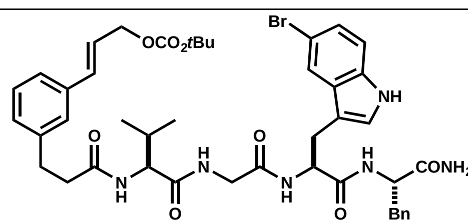
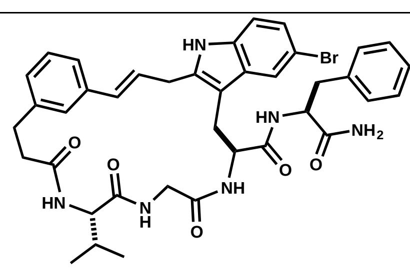
11d		*45*
11a		*48*
12		*51*
16a		*52*
16b		*55*

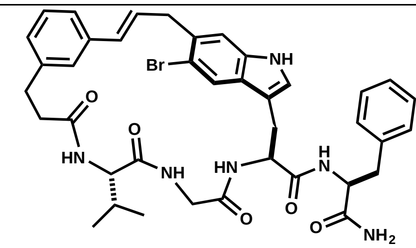
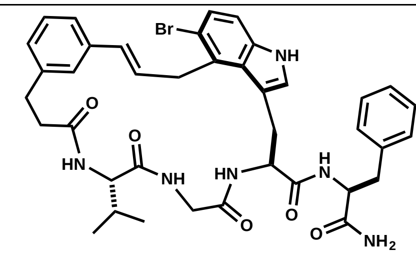
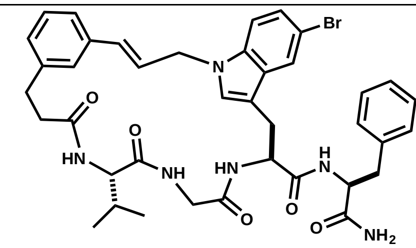
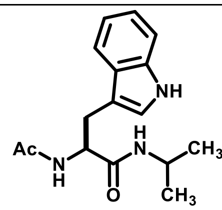
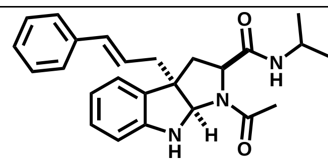
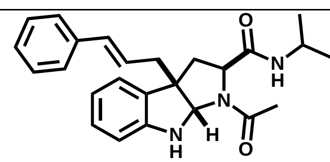
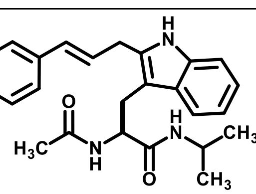
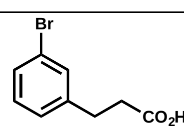
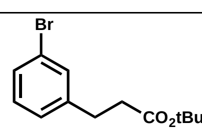
16c		*58*
16d		*61*
13		*64*
17a		*65*
17b		*68*
17c		*71*
17d		*74*

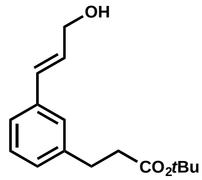
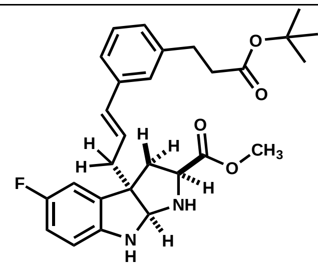
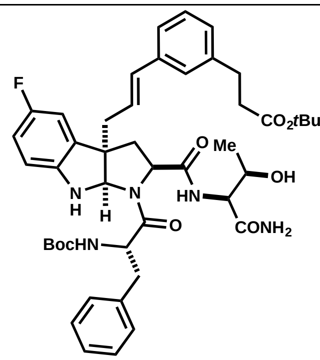
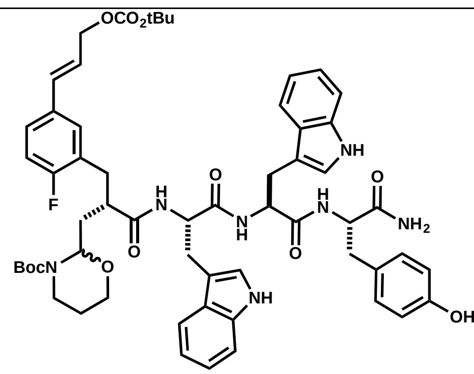
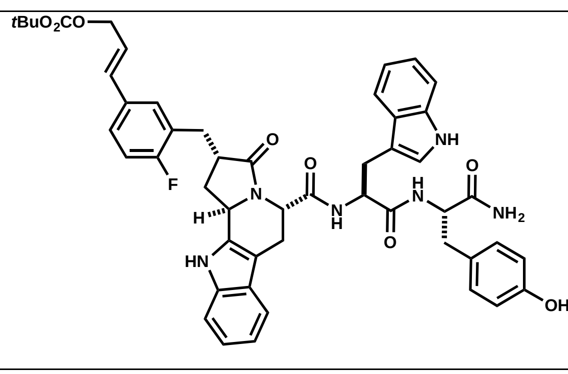
17e		*77*
14		*80*
18a		*81*
18b		*84*
18c		*87*
18d		*90*

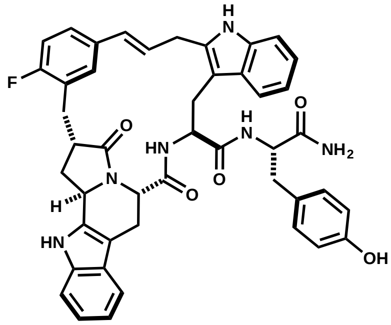
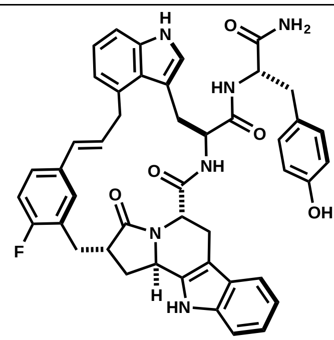
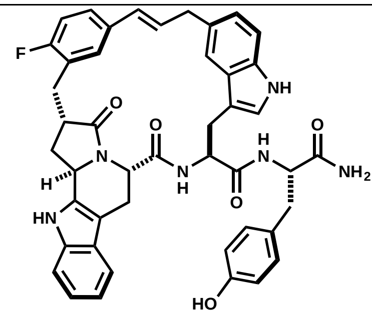
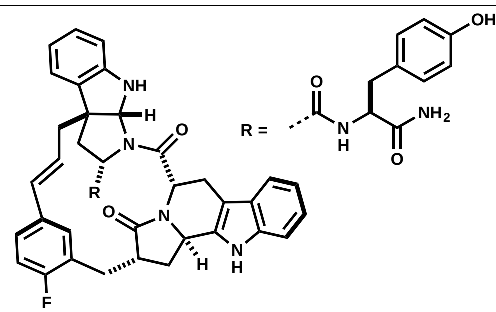
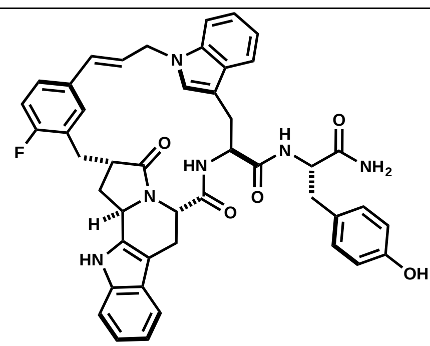
18e		*92*
15		*94*
19a		*95*
19b		*98*
19c		*101*

19d		*104*
S1		*108*
S2a		*109*
S2b		*111*
S2c		*114*

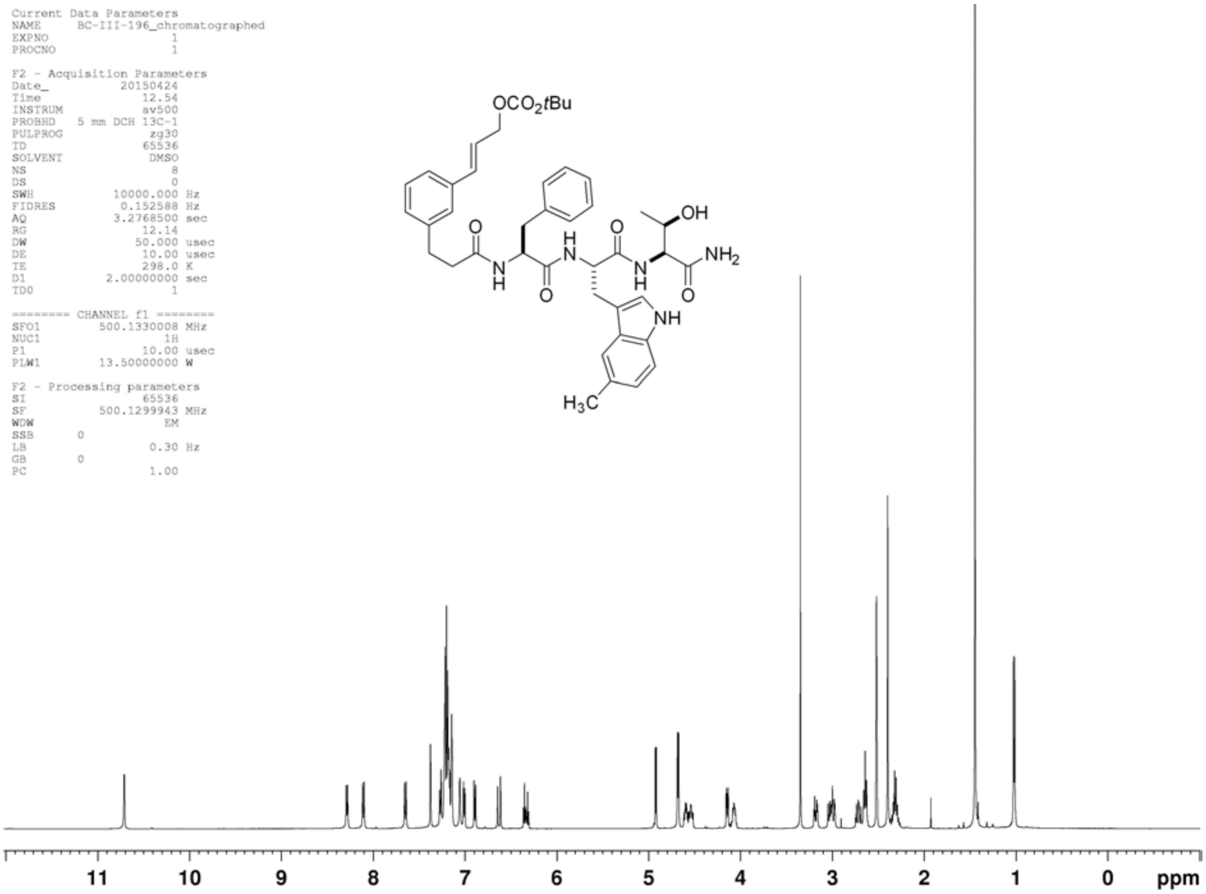
S2d		*117*
S3		*120*
S4a		*121*
S4b		*124*
S5		*127*
S6a		*128*

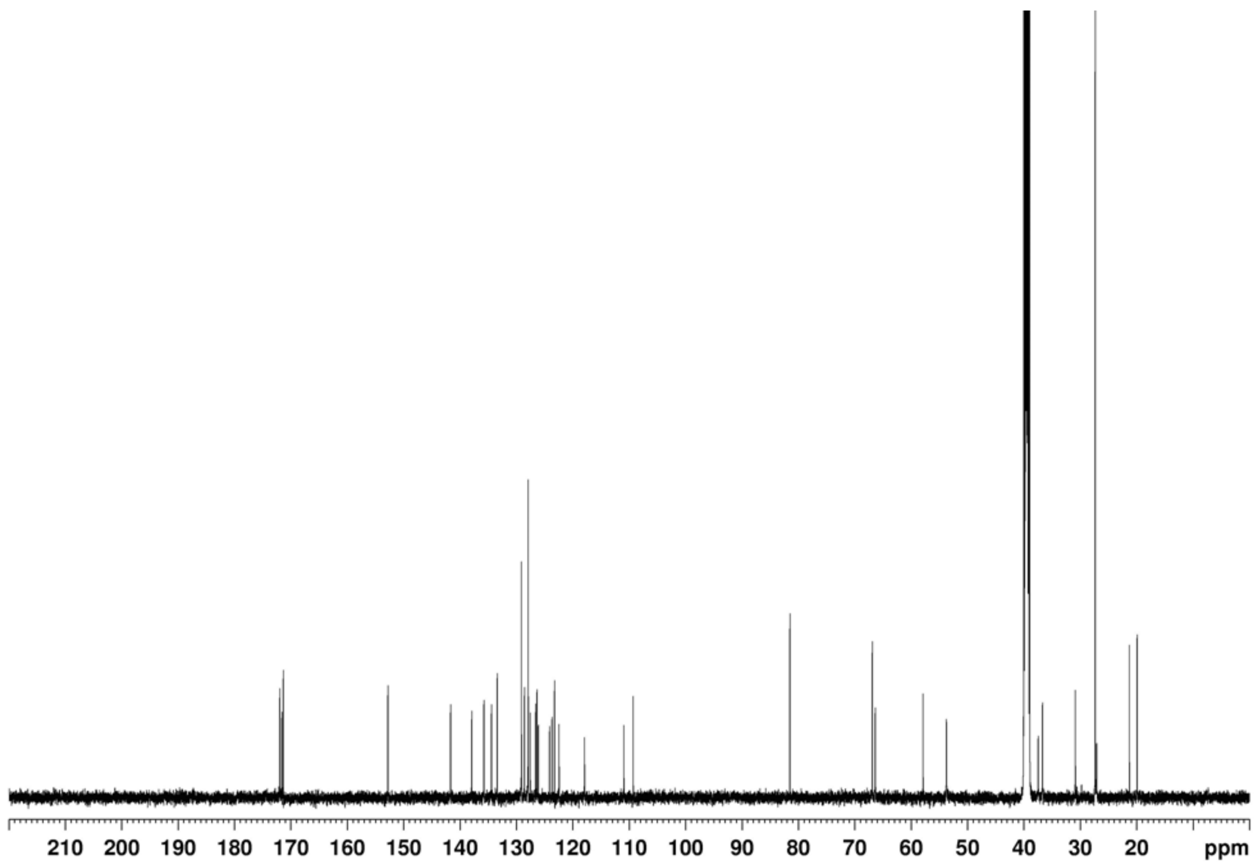
S6b		*131*
S6c		*133*
S6d		*136*
S7		*139*
21a		*140*
21b		*142*
22		*144*
S8		*145*
S9		*146*

24		*147*
26		*148*
27		*150*
S11		*151*
30		*152*

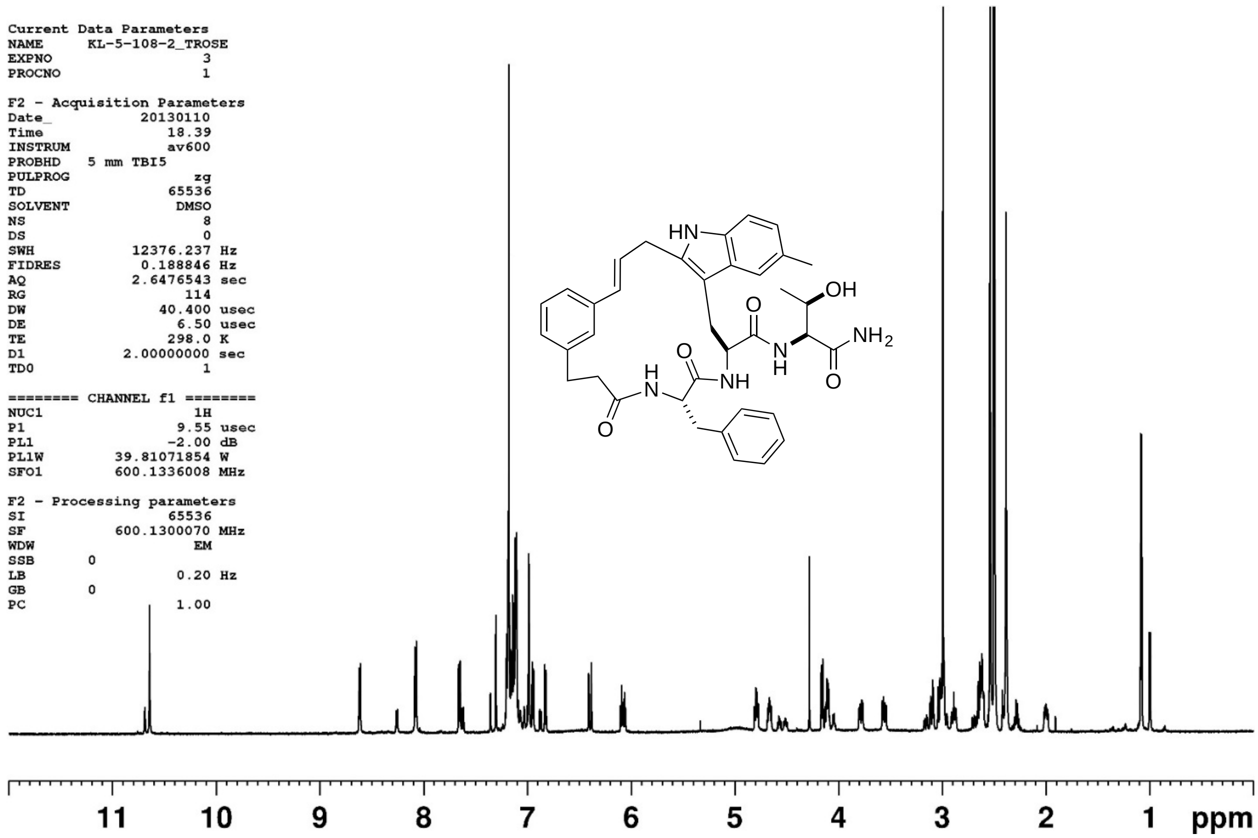
31a		*154*
31b		*157*
31c		*160*
31e		*163*
31f		*167*

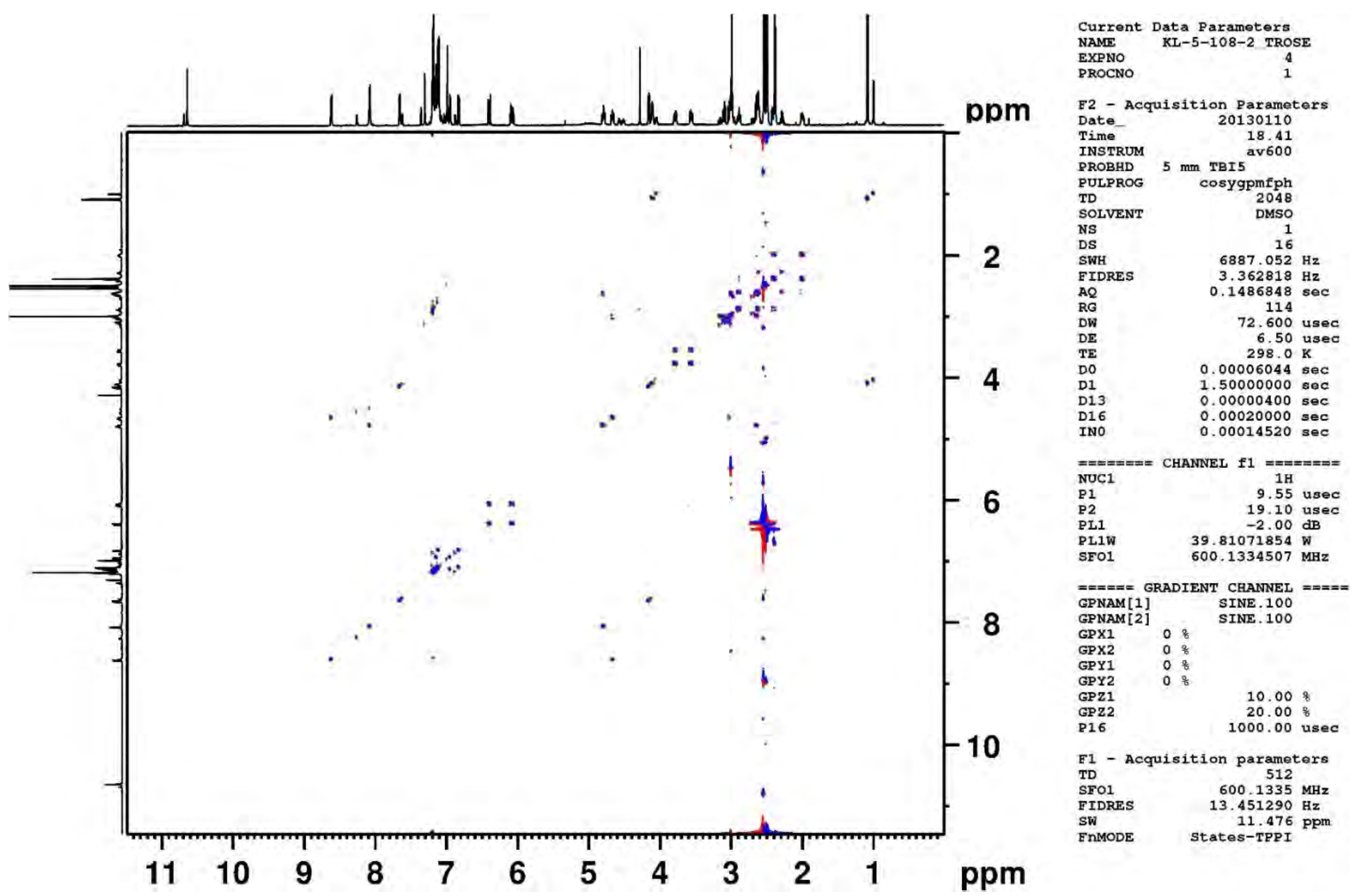
*Acyclic Precursor **6***

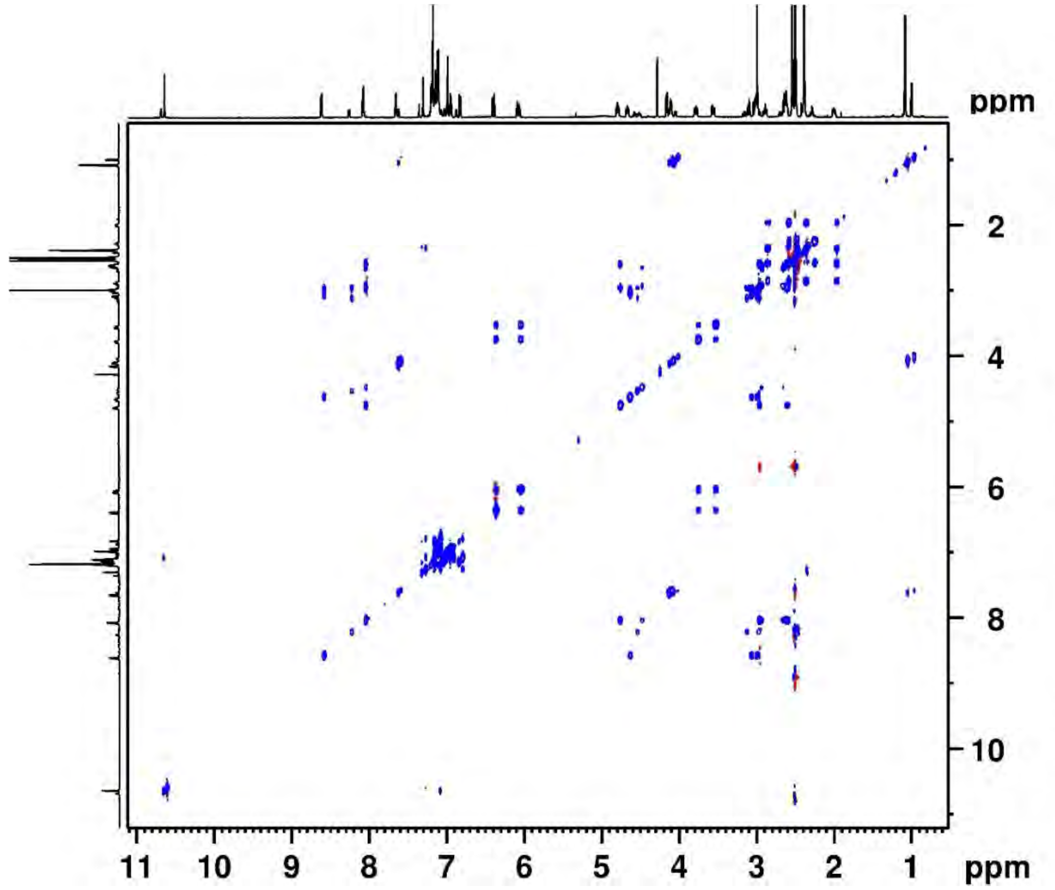




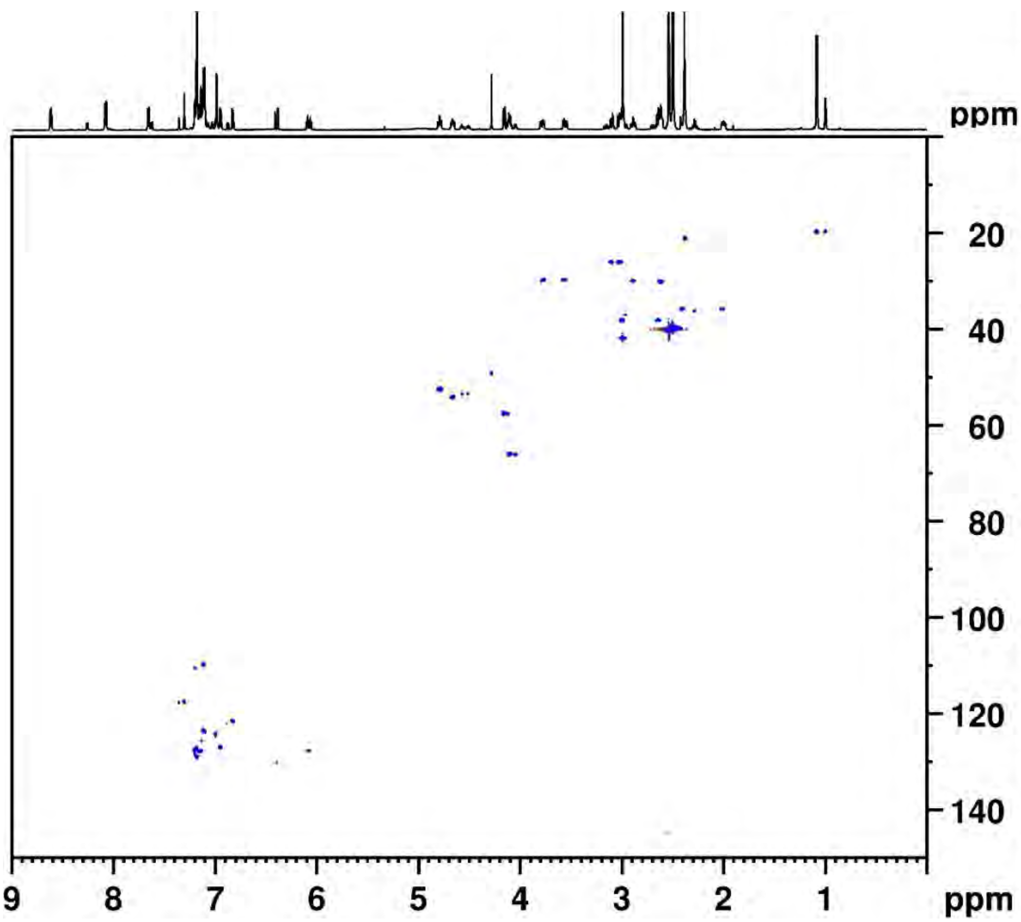
*Macrocyclic Product **8b***



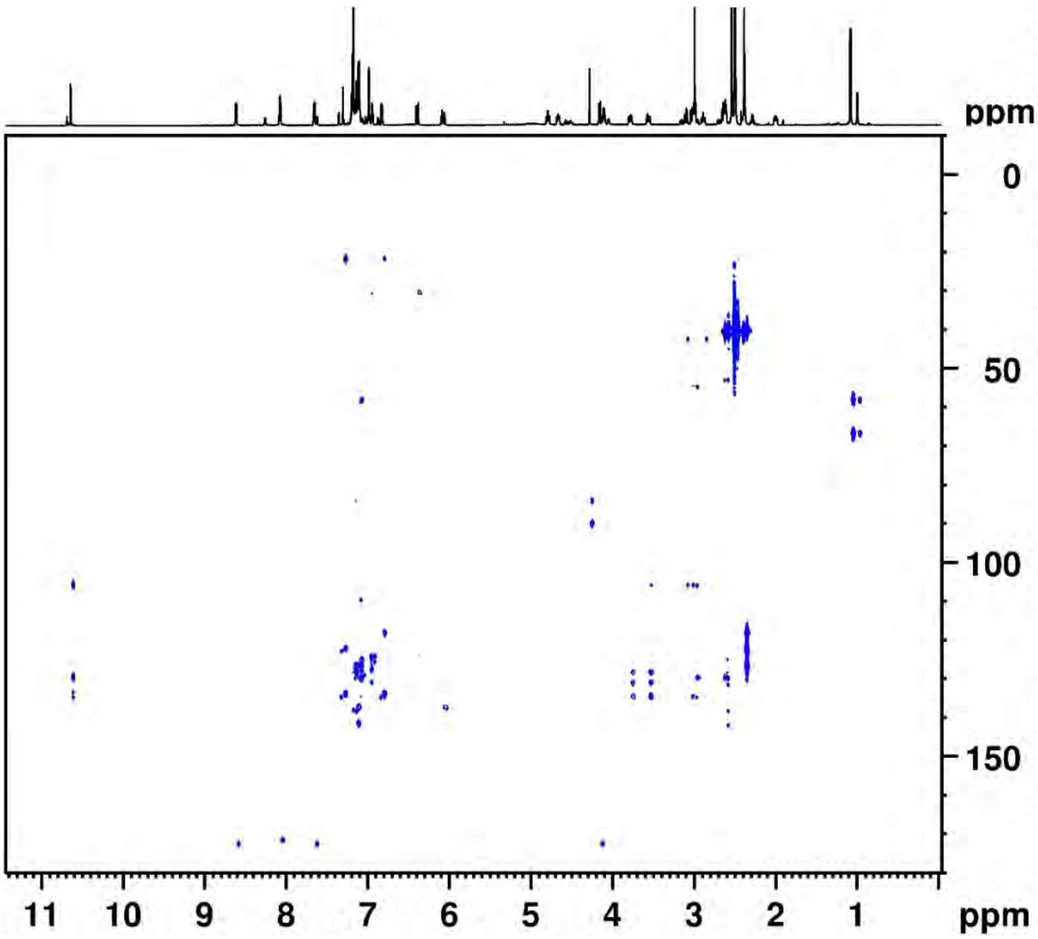





```
Current Data Parameters
NAME      KL-5-108-2_TROSE
EXPNO                    8
PROCNO                   1

F2 - Acquisition Parameters
Date_             20130111
Time                  1.25
INSTRUM              av600
PROBHD    5 mm TBI5
PULPROG        dipsi2etgpsi
TD                    2048
SOLVENT               DMSO
NS                       8
DS                      16
SWH               6887.052 Hz
FIDRES            3.362818 Hz
AQ               0.1486848 sec
RG                     362
DW                  72.600 usec
DE                    6.50 usec
TE                   298.0 K
D0              0.00000300 sec
D1              1.50000000 sec
D9              0.06000000 sec
D11             0.03000000 sec
D16             0.00020000 sec
D20             0.00001000 sec
D21             0.00001000 sec
IN0             0.00014520 sec
L1                      14

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.55 usec
P2                   19.10 usec
P6                   40.00 usec
PL1                  -2.00 dB
PL10                 10.44 dB
PL1W           39.81071854 W
PL10W           2.26986504 W
SFO1           600.1334507 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPX1        0 %
GPX2        0 %
GPY1        0 %
GPY2        0 %
GPZ1                 30.00 %
GPZ2                 30.00 %
P16                1000.00 usec
```




```
Current Data Parameters
NAME      KL-5-108-2_TROSE
EXPNO                    6
PROCNO                   1

F2 - Acquisition Parameters
Date_             20130110
Time                 18.58
INSTRUM              av600
PROBHD    5 mm TBI5
PULPROG       hsqcetgpsisp
TD                    2048
SOLVENT               DMSO
NS                      16
DS                      16
SWH               6009.615 Hz
FIDRES            2.934382 Hz
AQ               0.1703936 sec
RG                   26008
DW                  83.200 usec
DE                    6.00 usec
TE                   298.1 K
CNST2          145.0000000
D0              0.00000300 sec
D1              1.00000000 sec
D4              0.00172414 sec
D11             0.03000000 sec
D16             0.00020000 sec
D24             0.00086200 sec
IN0             0.00002070 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.55 usec
P2                   19.10 usec
P28                1000.00 usec
PL1                  -2.00 dB
PL1W           39.81071854 W
SFO1           600.1327006 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                   13C
P3                   18.50 usec
P4                   37.00 usec
P14                1000.00 usec
PCPD2                65.00 usec
PL0                 120.00 dB
PL2                  -3.00 dB
PL12                  7.91 dB
PL0W        0 W
PL2W          150.35617065 W
PL12W          12.19330025 W
SFO2           150.9133722 MHz
```



Current Data Parameters
NAME KL-5-108-2_TROSE
EXPNO 7
PROCNO 1

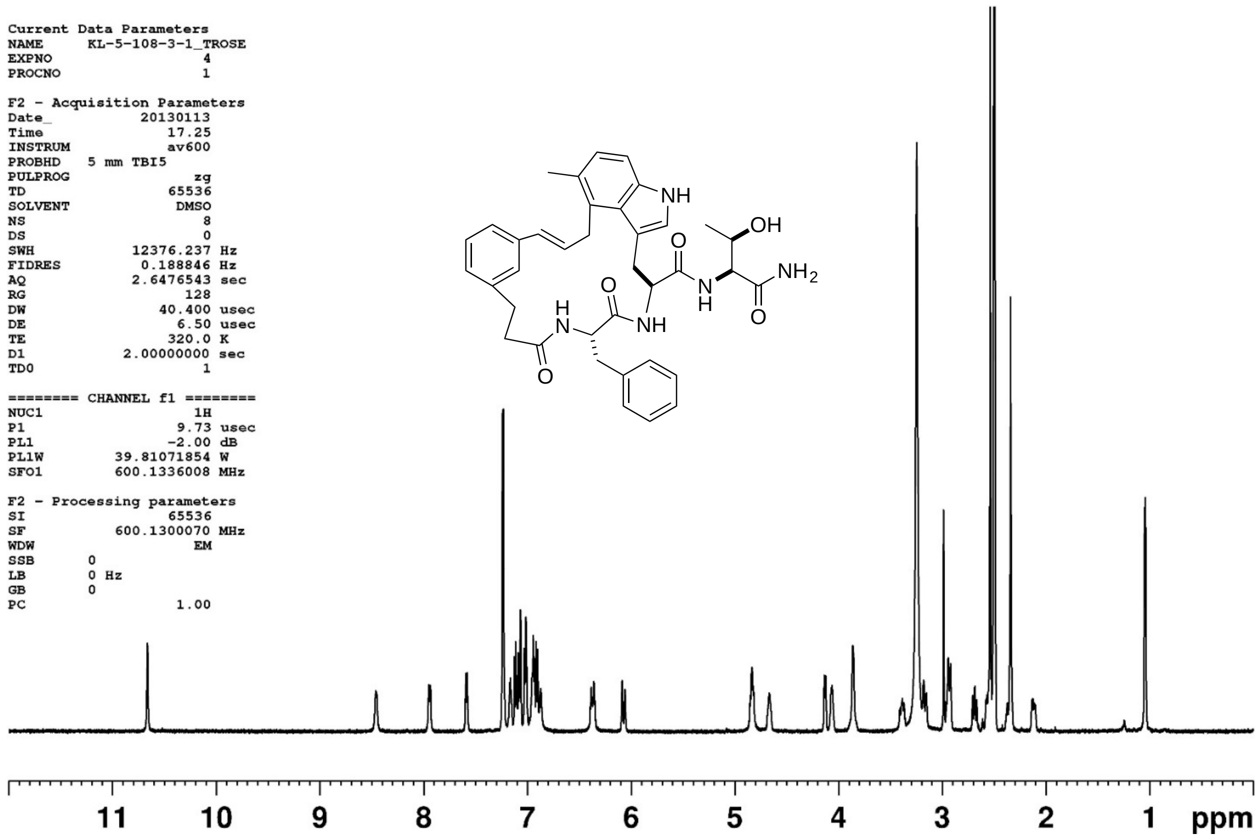
F2 - Acquisition Parameters
Date_ 20130110
Time 20.20
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 50
DS 24
SWH 6887.052 Hz
FIDRES 3.362818 Hz
AQ 0.1486848 sec
RG 26008
DW 72.600 usec
DE 6.00 usec
TE 298.0 K
CNST6 125.0000000
CNST7 165.0000000
CNST13 8.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D6 0.06250000 sec
D16 0.00020000 sec
IN0 0.00001745 sec

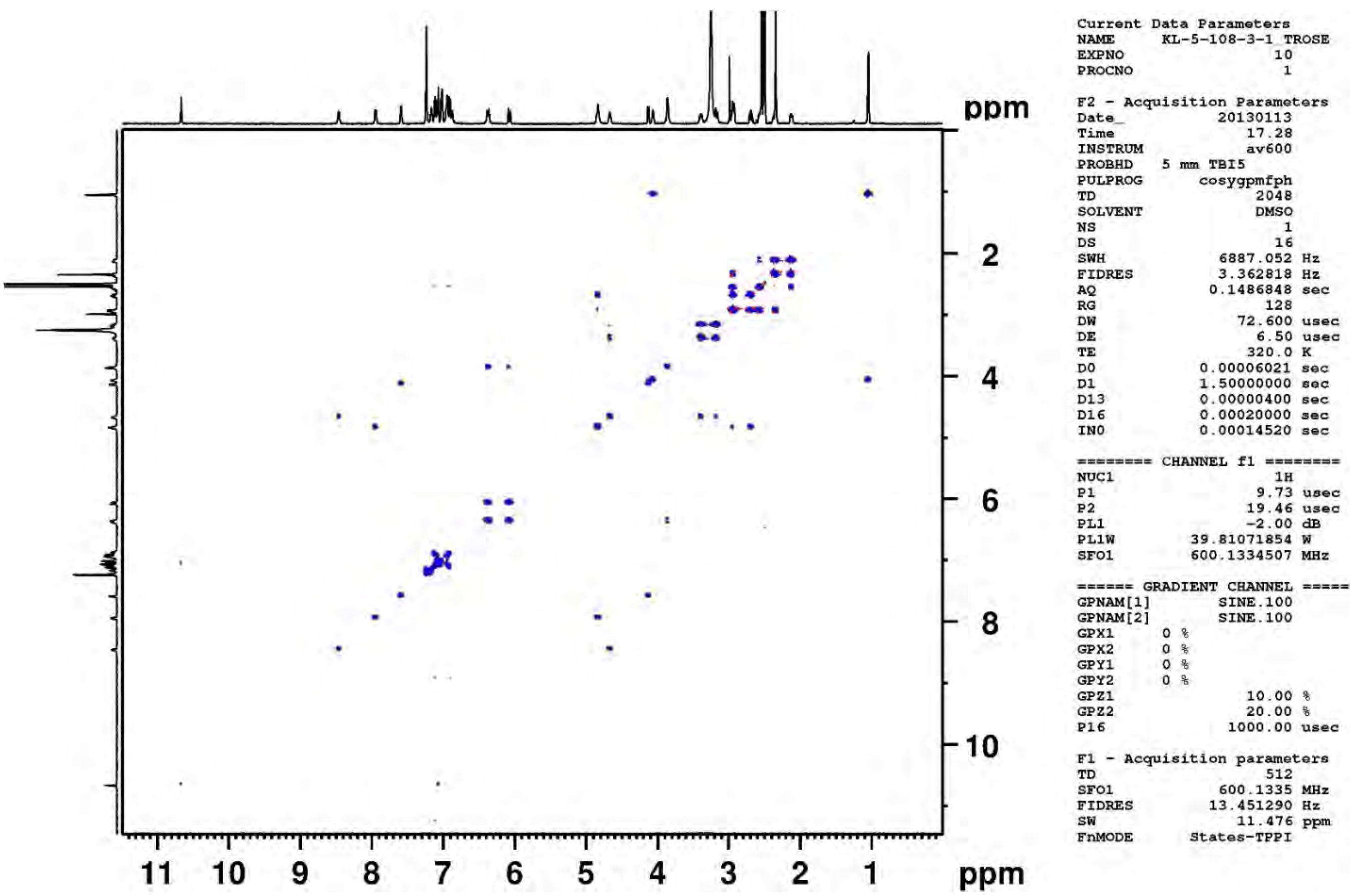
======== CHANNEL f1 ========
NUC1 1H
P1 9.55 usec
P2 19.10 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1334507 MHz

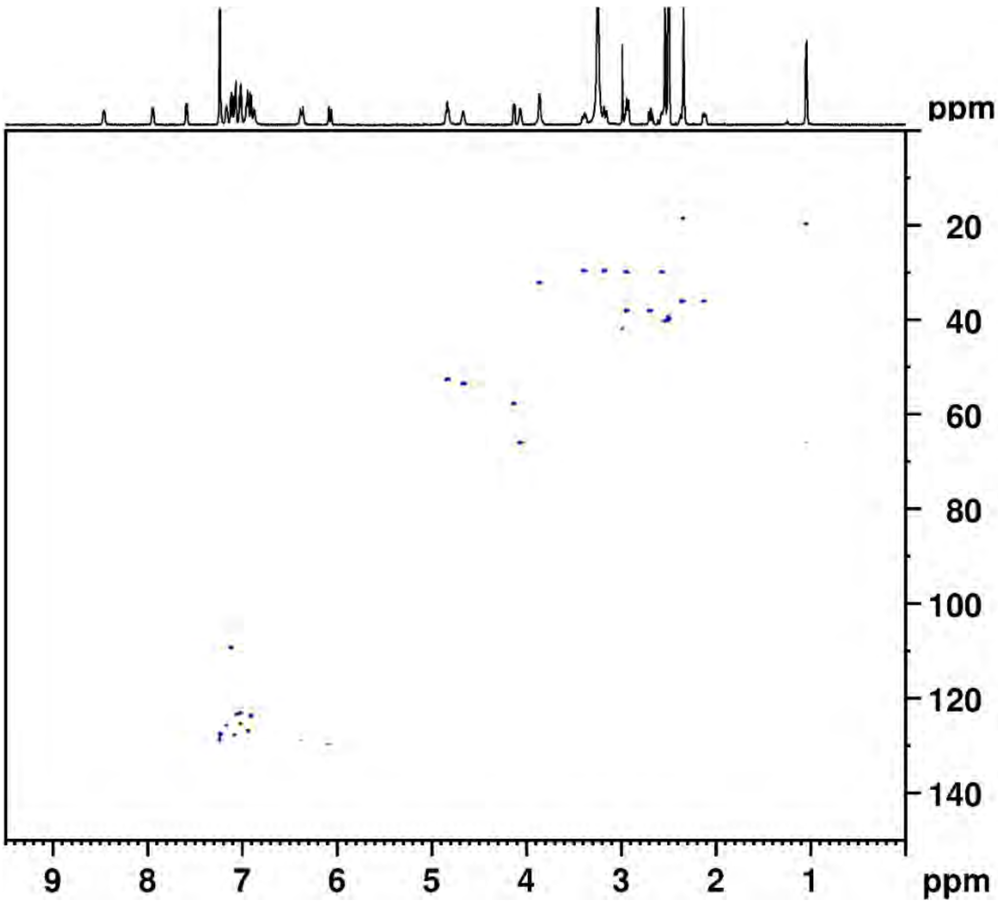
======== CHANNEL f2 ========
NUC2 13C
P3 18.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPNAM[4] SINE.100
GPNAM[5] SINE.100
GPNAM[6] SINE.100
GPX1 0 %
GPX2 0 %

*Macrocyclic Product **8c***





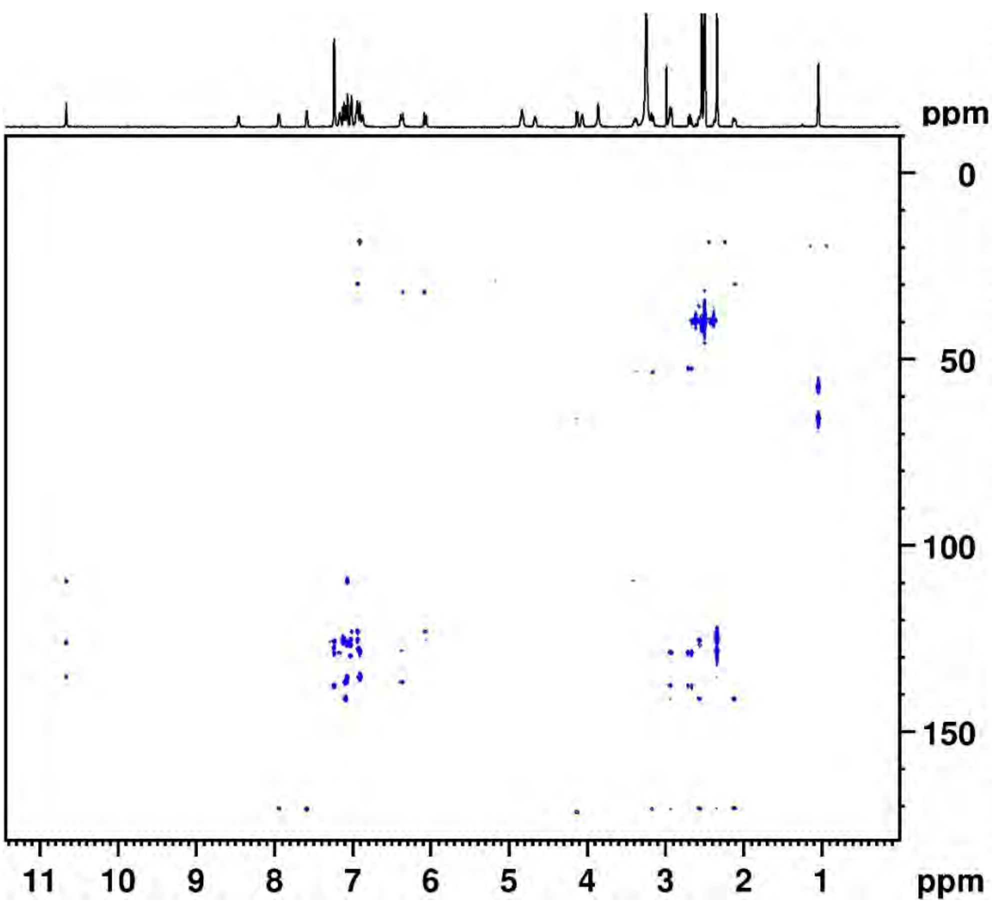


Current Data Parameters
NAME KL-5-108-3-1_TROSE
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130113
Time 17.43
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 26008
DW 83.200 usec
DE 6.00 usec
TE 320.1 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.00000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1 1H
P1 9.73 usec
P2 19.46 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1327006 MHz

======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 18.50 usec
P4 37.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.91 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 12.19330025 W
SFO2 150.9133722 MHz



Current Data Parameters
NAME KL-5-108-3-1_TROSE
EXPNO 14
PROCNO 1

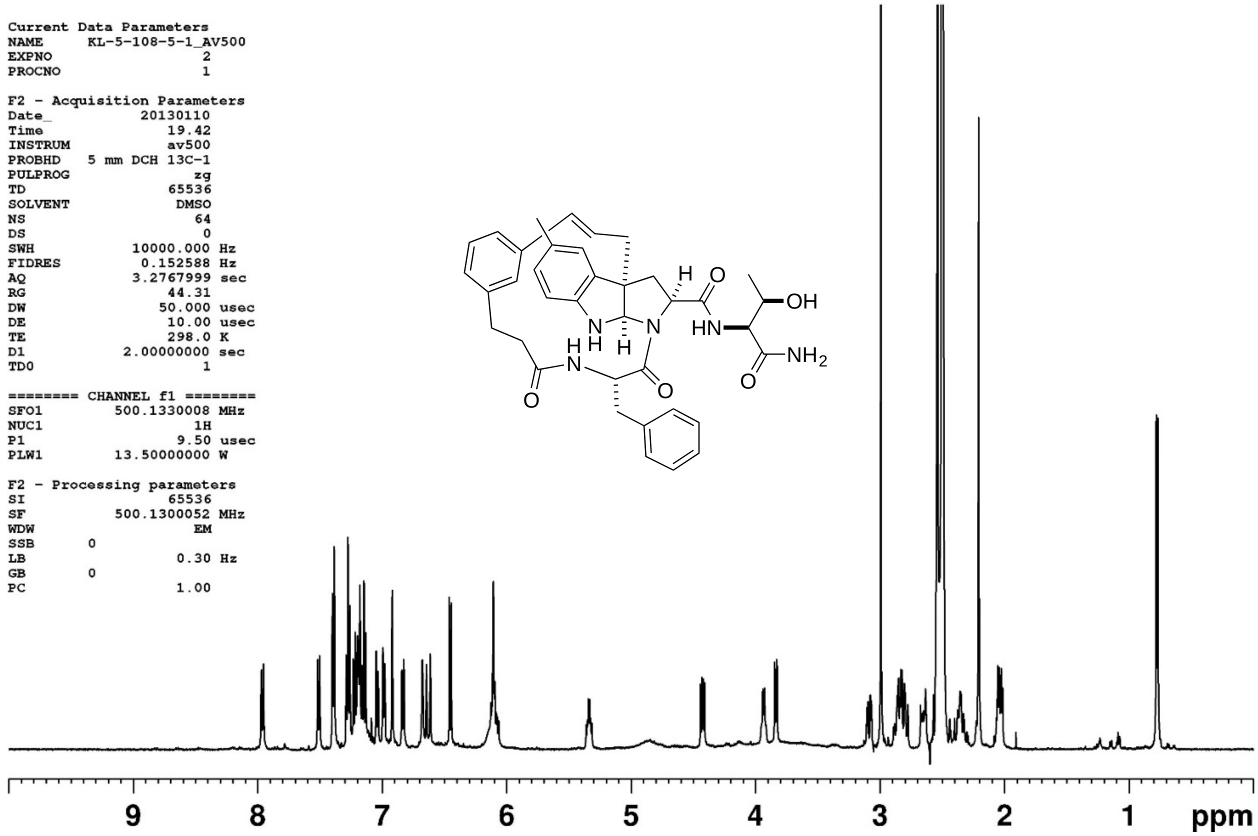
F2 - Acquisition Parameters
Date_ 20130113
Time 21.51
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 50
DS 24
SWH 6887.052 Hz
FIDRES 3.362818 Hz
AQ 0.1486848 sec
RG 26008
DW 72.600 usec
DE 6.00 usec
TE 320.0 K
CNST6 125.0000000
CNST7 165.0000000
CNST13 8.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D6 0.06250000 sec
D16 0.00020000 sec
IN0 0.00001745 sec

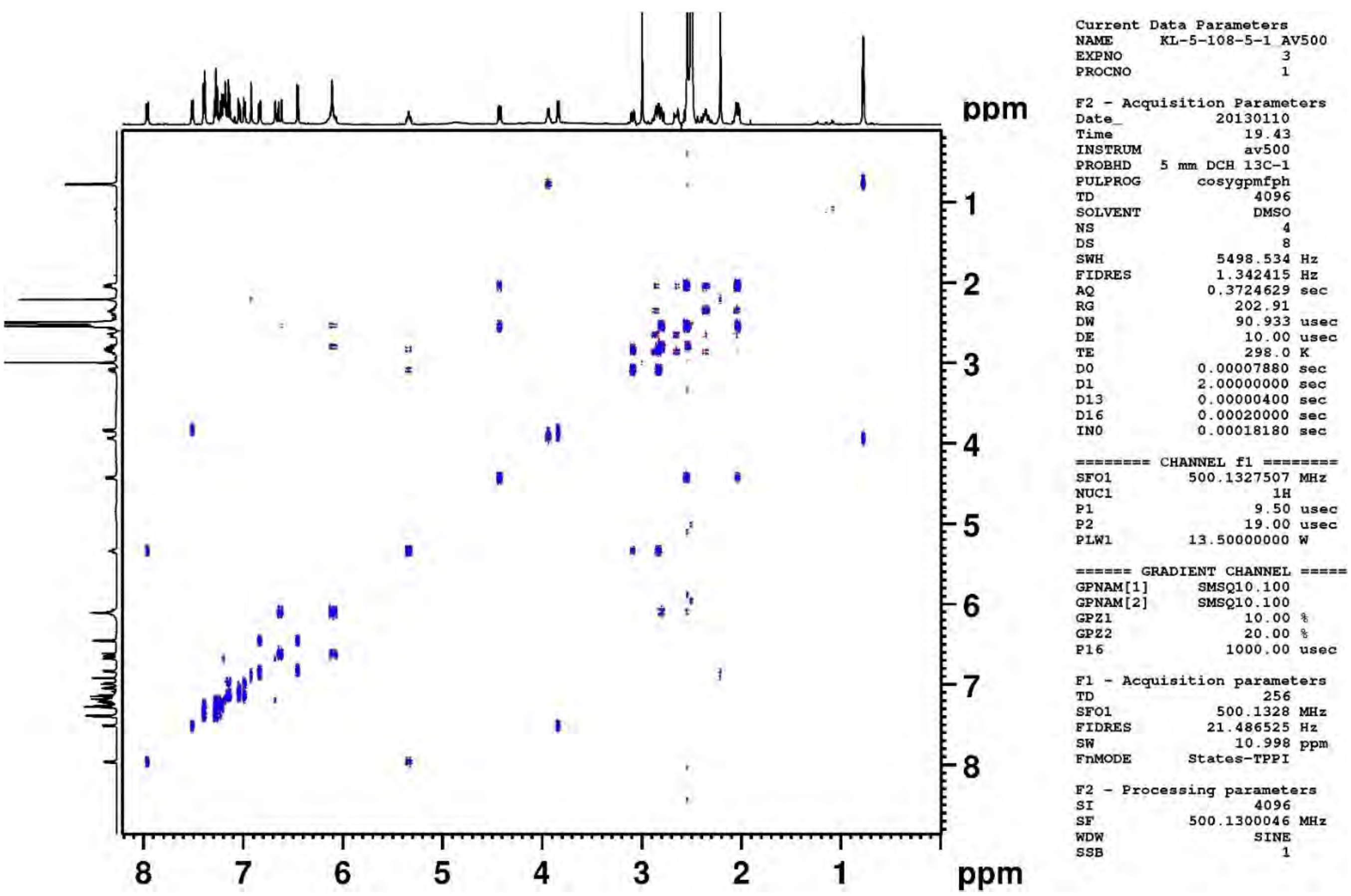
======== CHANNEL f1 ========
NUC1 1H
P1 9.73 usec
P2 19.46 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1334507 MHz

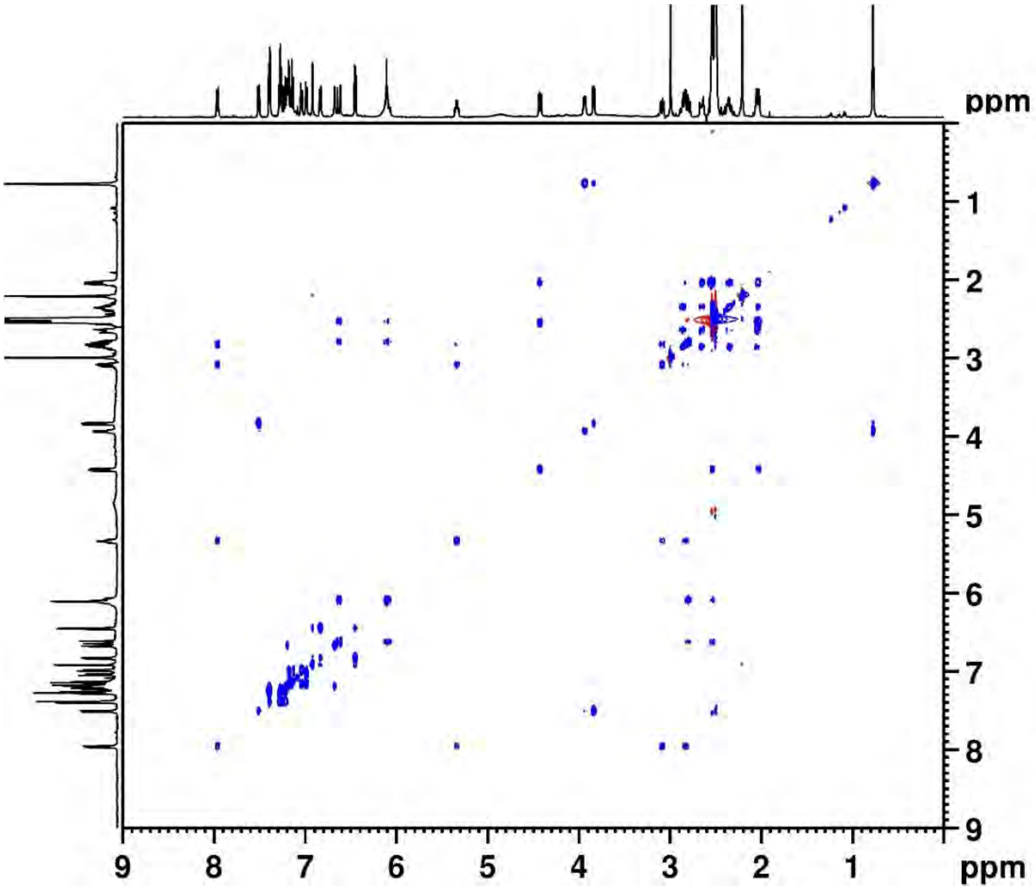
======== CHANNEL f2 ========
NUC2 13C
P3 18.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPNAM[4] SINE.100
GPNAM[5] SINE.100
GPNAM[6] SINE.100
GPX1 0 %
GPX2 0 %

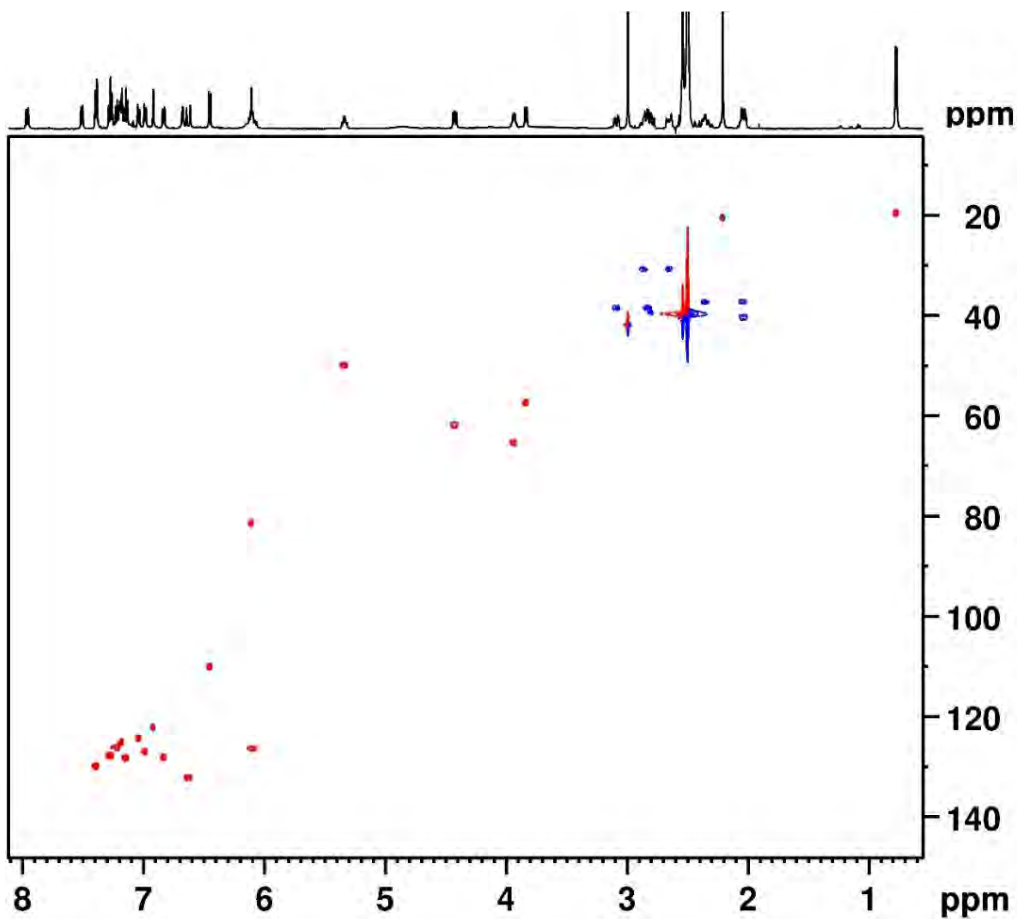
Macrocyclic Product ***8d***



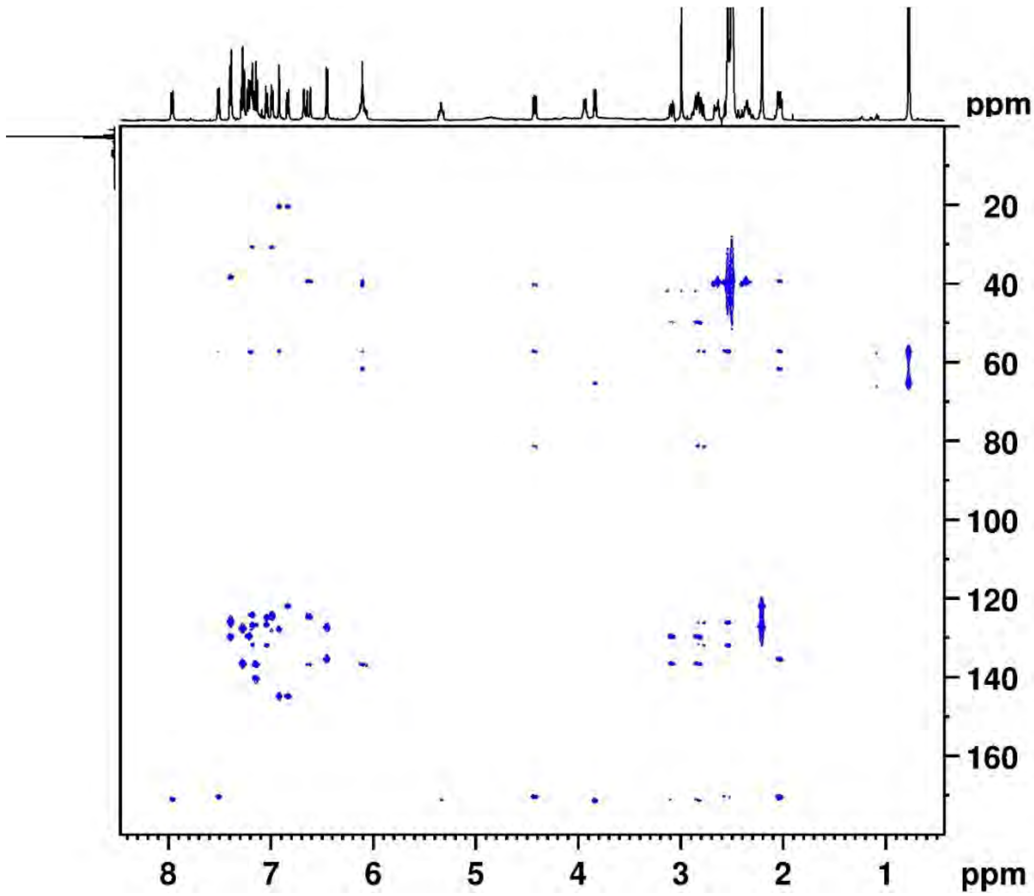





```
Current Data Parameters
NAME       KL-5-108-5-1_AV500
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20130110
Time               20.24
INSTRUM            av500
PROBHD   5 mm DCH 13C-1
PULPROG      mlevetgp.js
TD                  2048
SOLVENT             DMSO
NS                     4
DS                     8
SWH             5000.000 Hz
FIDRES          2.441406 Hz
AQ             0.2048000 sec
RG                 37.94
DW               100.000 usec
DE                 10.00 usec
TE                 298.0 K
D0            0.00000300 sec
D1            2.00000000 sec
D9            0.06000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00020000 sec
L1                    24

======== CHANNEL f1 ========
SFO1         500.1325007 MHz
NUC1                  1H
P1                  9.50 usec
P2                 19.00 usec
P5                 26.68 usec
P6                 40.00 usec
P7                 80.00 usec
P17              2500.00 usec
PLW1         13.50000000 W
PLW10         0.84375000 W

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPZ1               30.00 %
GPZ2               30.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1            500.1325 MHz
FIDRES         19.531250 Hz
```

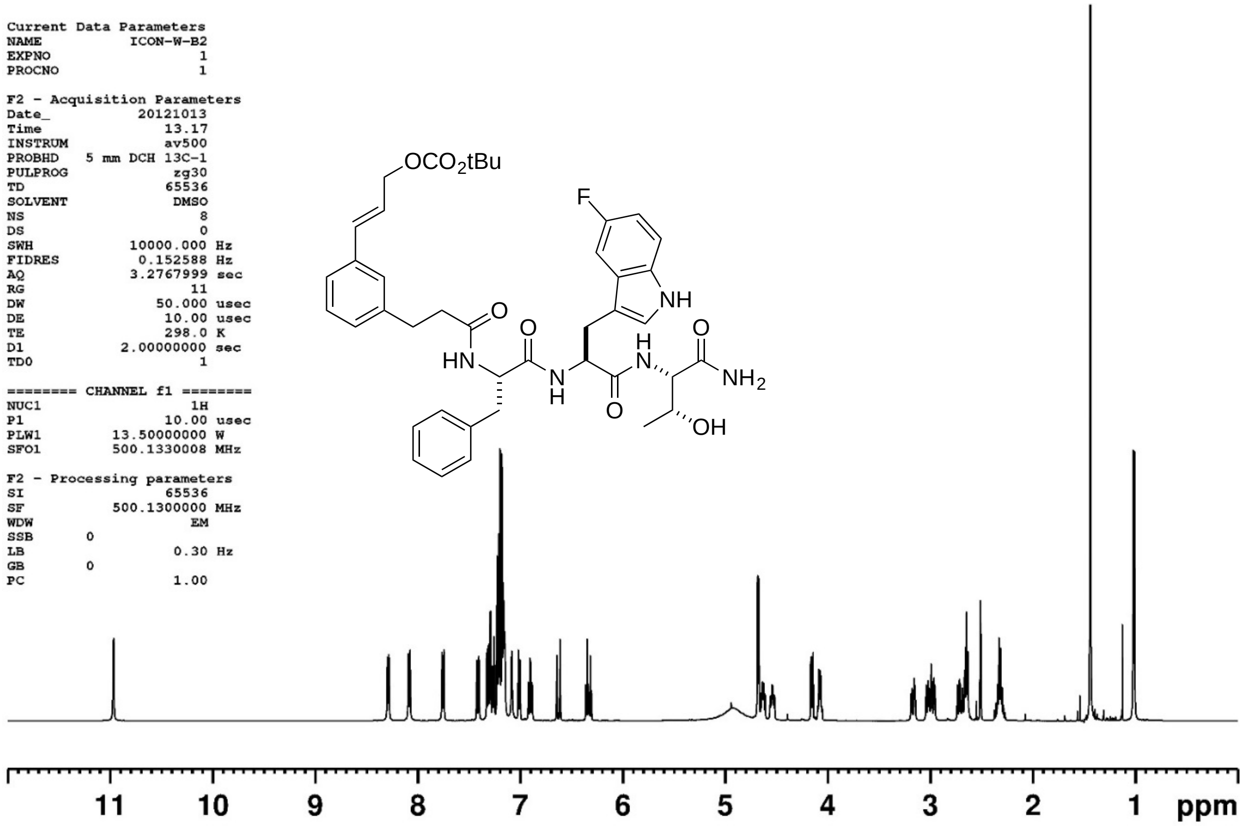


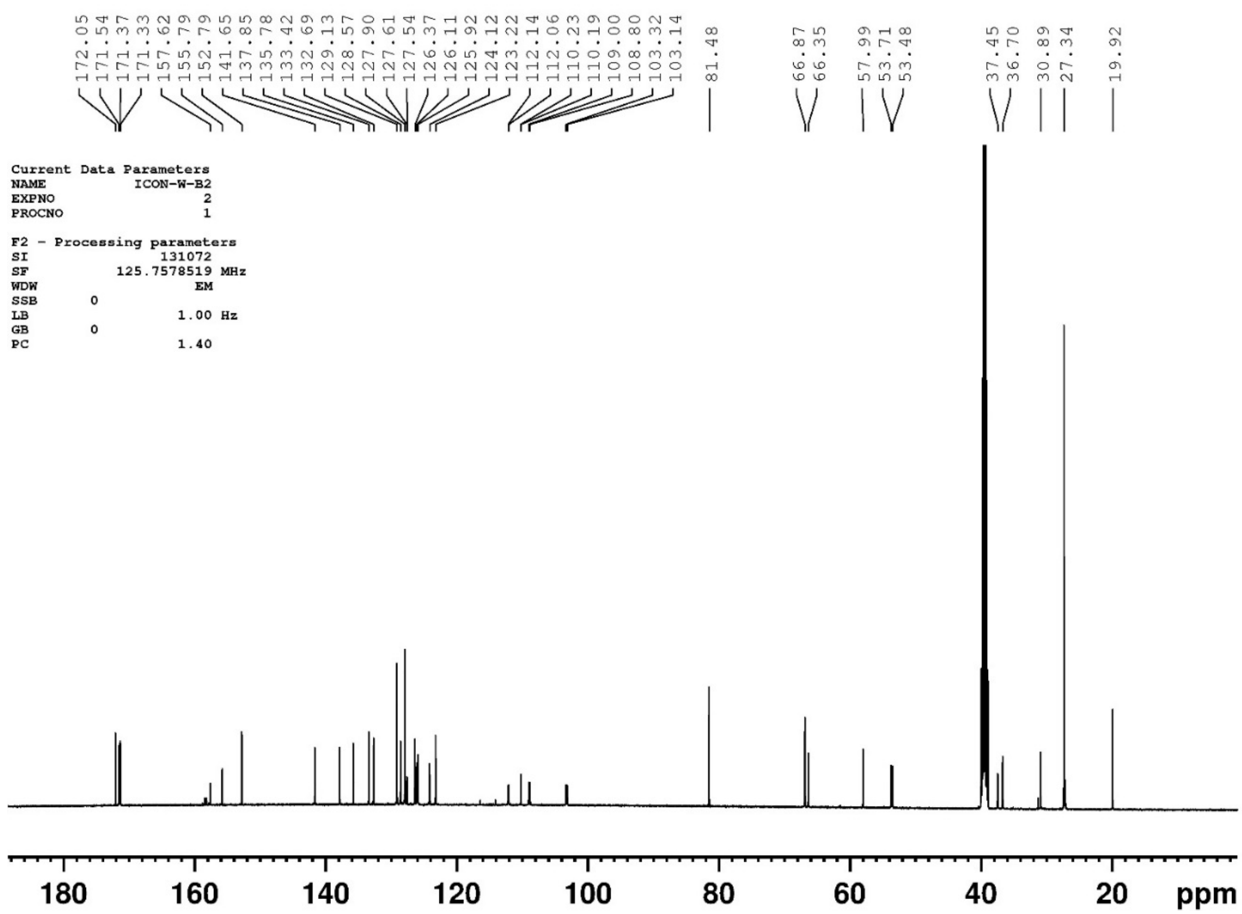

```
Current Data Parameters
NAME       KL-5-108-5-1_AV500
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_           20130110
Time               21.05
INSTRUM            av500
PROBHD   5 mm DCH 13C-1
PULPROG       hsqcedetgp
TD                  2048
SOLVENT             DMSO
NS                    16
DS                    16
SWH             5000.000 Hz
FIDRES          2.441406 Hz
AQ             0.2048000 sec
RG                202.91
DW               100.000 usec
DE                 10.00 usec
TE                 298.0 K
CNST2        145.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D4            0.00172414 sec
D11           0.03000000 sec
D13           0.00000400 sec
D16           0.00020000 sec
D21           0.00345000 sec
IN0           0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
SFO1         500.1325007 MHz
NUC1                  1H
P1                  9.50 usec
P2                 19.00 usec
P28       0 usec
PLW1         13.50000000 W

======== CHANNEL f2 ========
SFO2         125.7678496 MHz
NUC2                 13C
CPDPRG[2            garp
P3                  9.63 usec
P4                 19.26 usec
PCPD2              70.00 usec
PLW2         23.01399994 W
PLW12         0.43557000 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[2]       SMSQ10.100
GPZ1               80.00 %
```



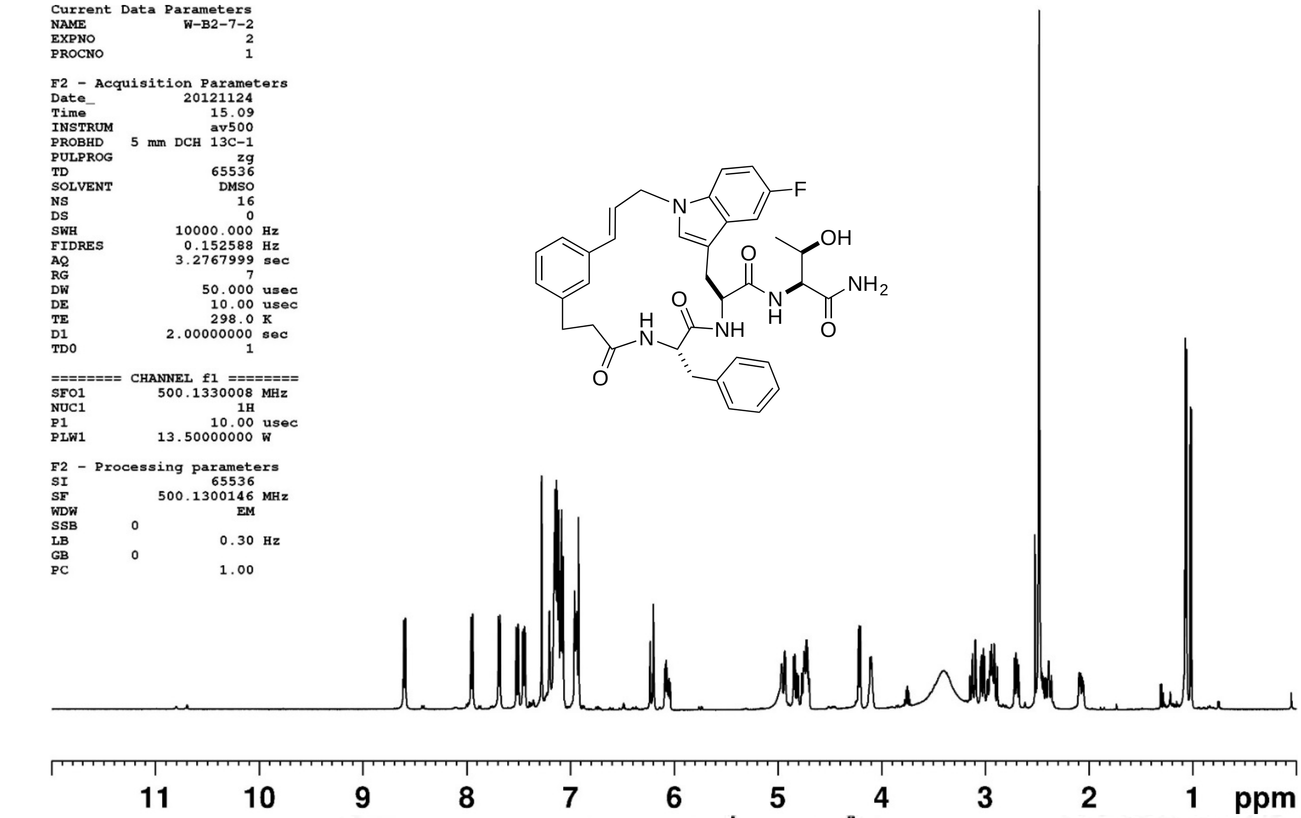

```
Current Data Parameters
NAME        KL-5-108-5-1_AV500
EXPNO                        6
PROCNO                       1

F2 - Acquisition Parameters
Date_              20130110
Time                  23.03
INSTRUM               av500
PROBHD    5 mm DCH 13C-1
PULPROG       hmbcgpl2ndqf
TD                     2048
SOLVENT                DMSO
NS                       32
DS                       16
SWH               6009.615 Hz
FIDRES            2.934382 Hz
AQ               0.1703936 sec
RG                   202.91
DW                   83.200 usec
DE                    10.00 usec
TE                    298.0 K
CNST6           120.0000000
CNST7           160.0000000
CNST13            7.0000000
D0               0.00000300 sec
D1               1.50000000 sec
D6               0.07142857 sec
D16              0.00020000 sec
IN0              0.00001990 sec

======== CHANNEL f1 ========
SFO1          500.1330008 MHz
NUC1                   1H
P1                   9.50 usec
P2                  19.00 usec
PLW1          13.50000000 W

======== CHANNEL f2 ========
SFO2          125.7703648 MHz
NUC2                  13C
P3                   9.63 usec
PLW2          23.01399994 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[2]       SMSQ10.100
GPNAM[3]       SMSQ10.100
GPNAM[4]       SMSQ10.100
GPNAM[5]       SMSQ10.100
GPNAM[6]       SMSQ10.100
GPZ1                50.00 %
GPZ2                30.00 %
GPZ3                40.10 %
```

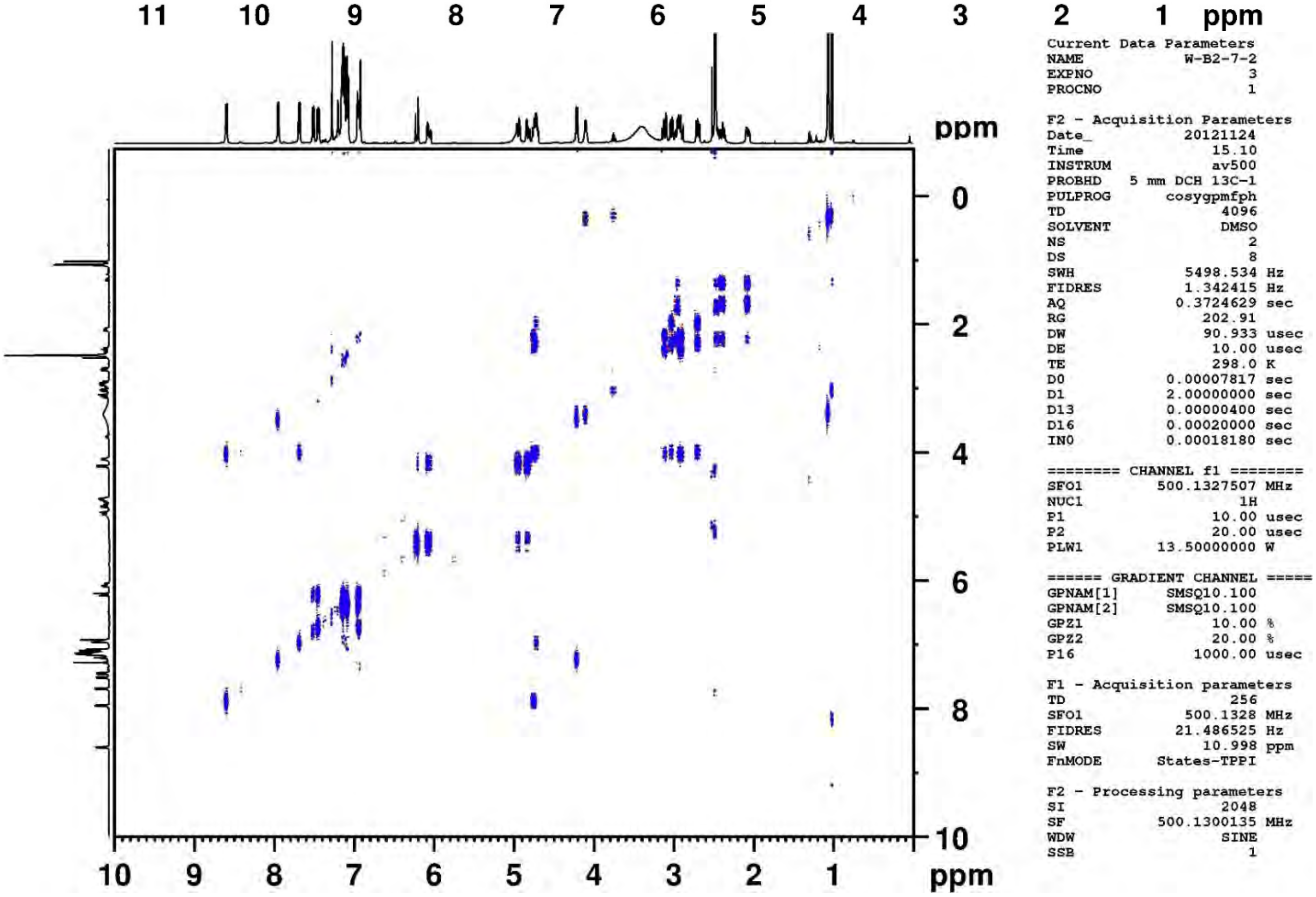
*Acyclic Precursor **7***

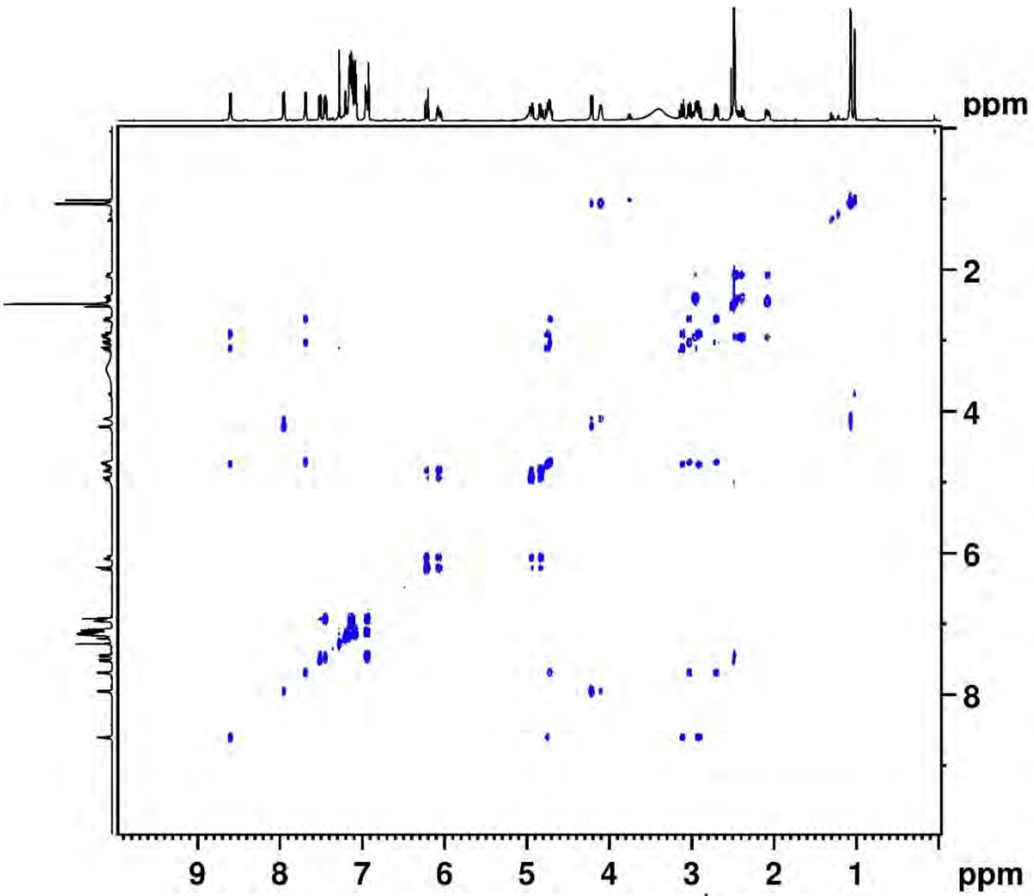




*Macrocyclic Product **9a***







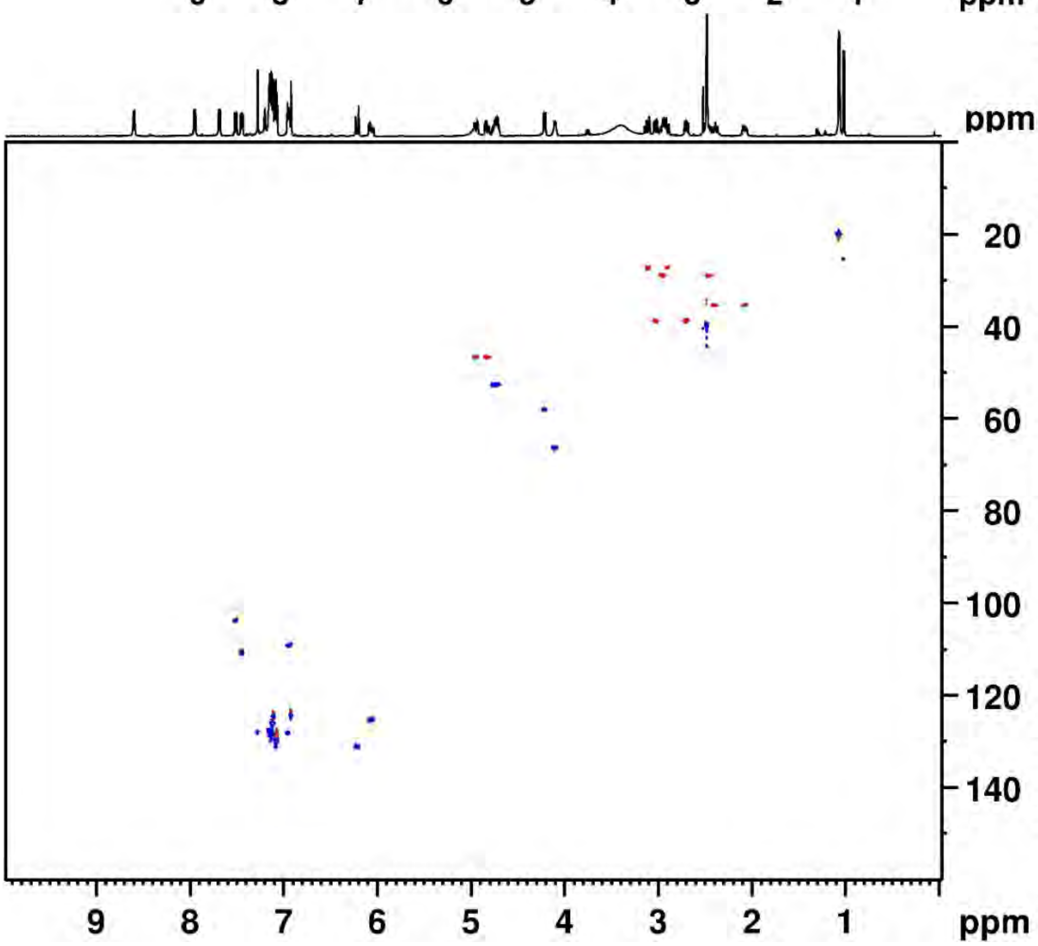
Current Data Parameters
NAME W-B2-7-2
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121124
Time 15.31
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG mlevetgp.js
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 37.94
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00020000 sec
L1 24

======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PLW1 13.50000000 W
PLW10 0.84375000 W

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.531250 Hz



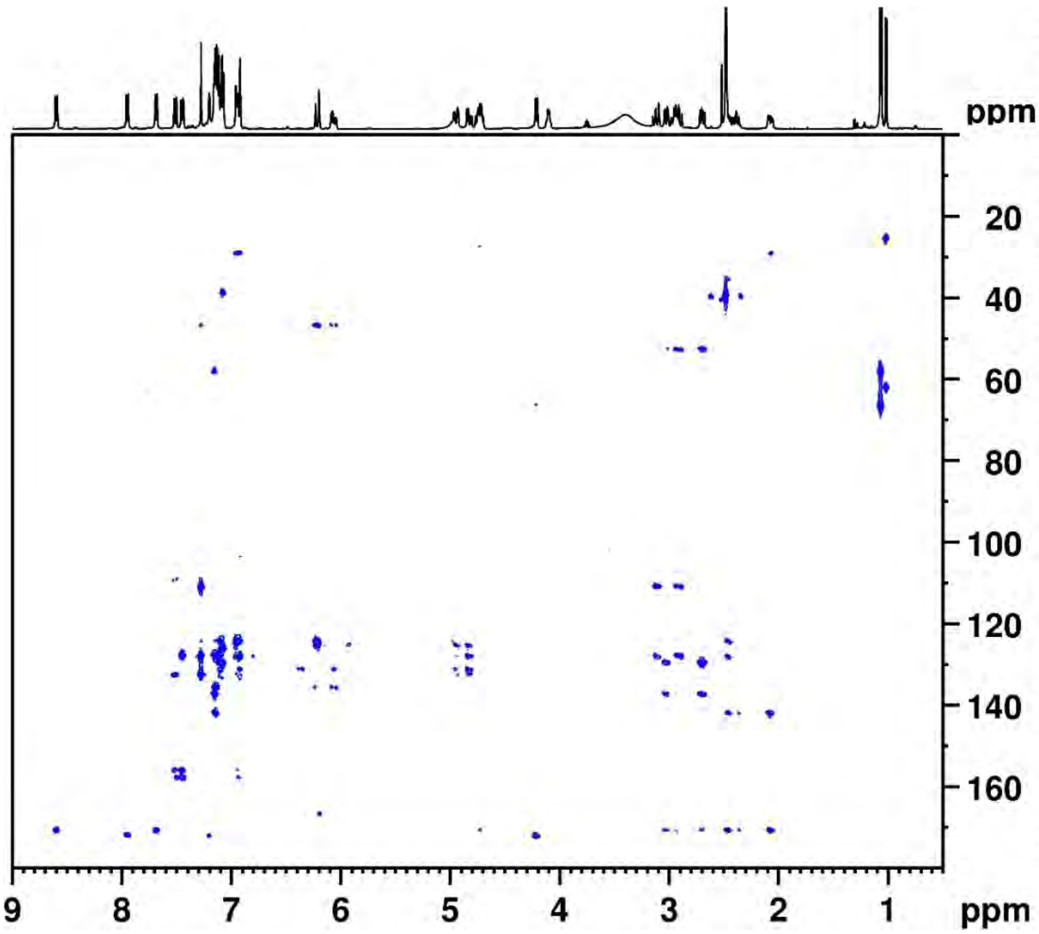
Current Data Parameters
NAME W-B2-7-2
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121124
Time 15.51
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcedetgp
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 202.91
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
IN0 0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P28 0 usec
PLW1 13.50000000 W

======== CHANNEL f2 ========
SFO2 125.7678496 MHz
NUC2 13C
CPDPRG[2 garp
P3 9.63 usec
P4 19.26 usec
PCPD2 70.00 usec
PLW2 23.01399994 W
PLW12 0.43557000 W

====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %



Current Data Parameters
NAME W-B2-7-2
EXPNO 6
PROCNO 1

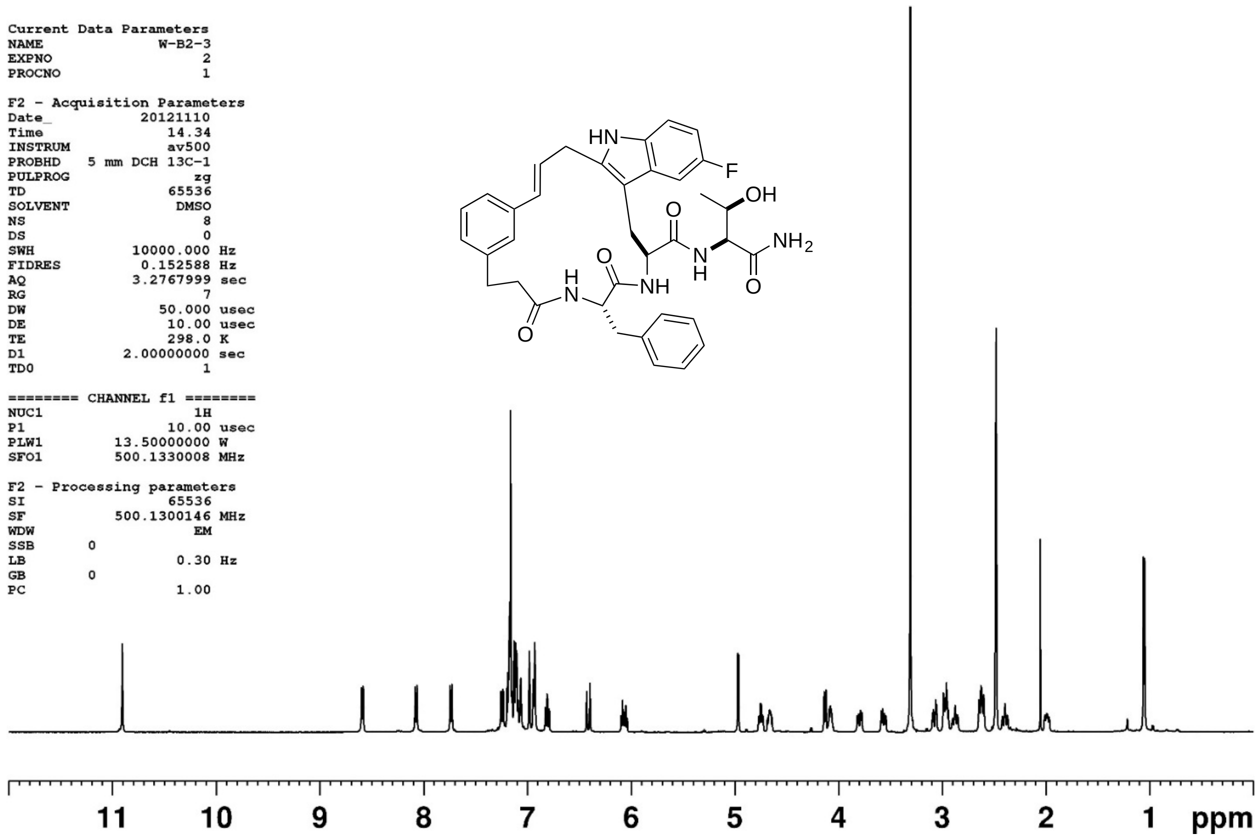
F2 - Acquisition Parameters
Date_ 20121124
Time 16.07
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 202.91
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec

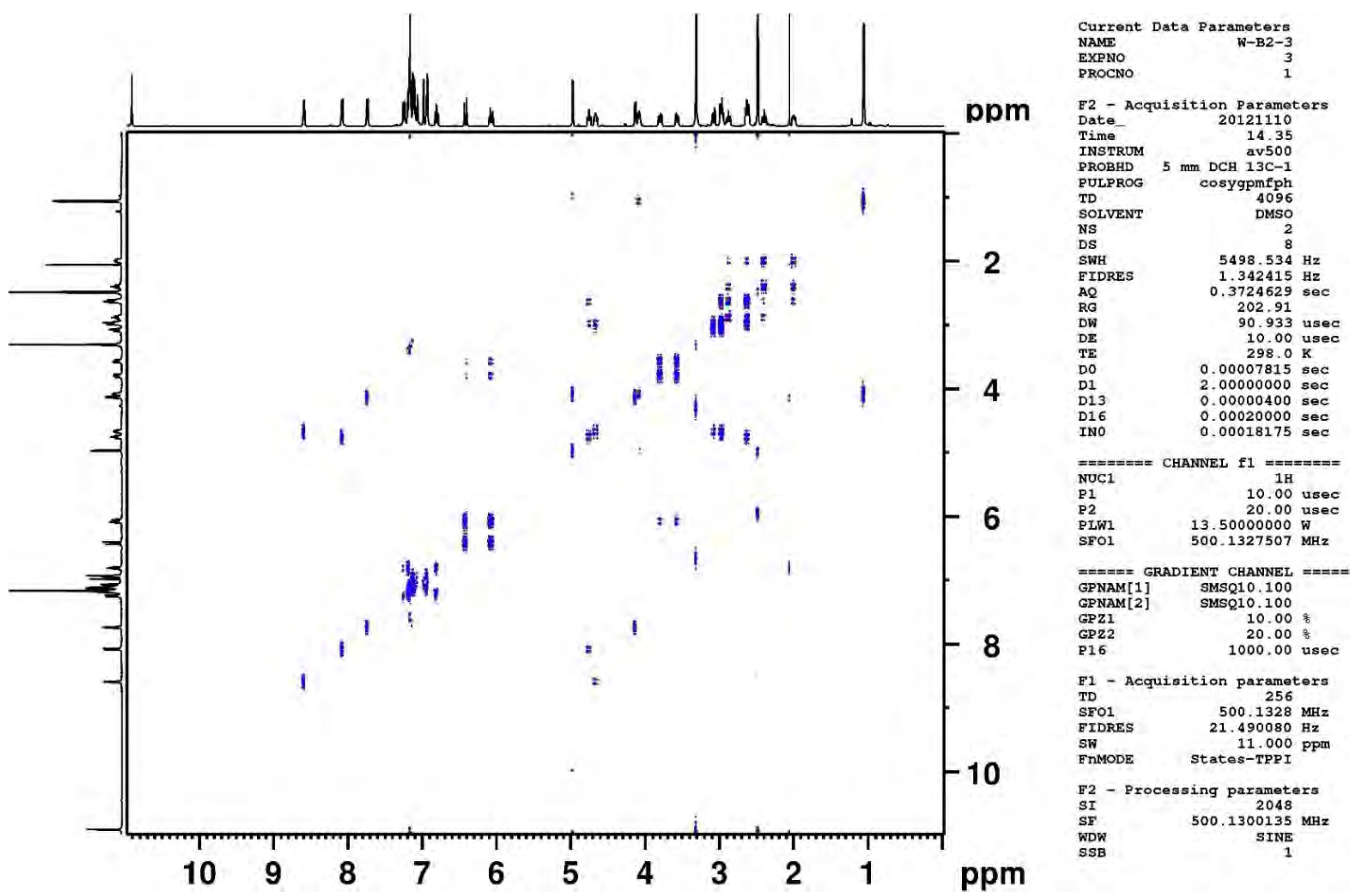
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W

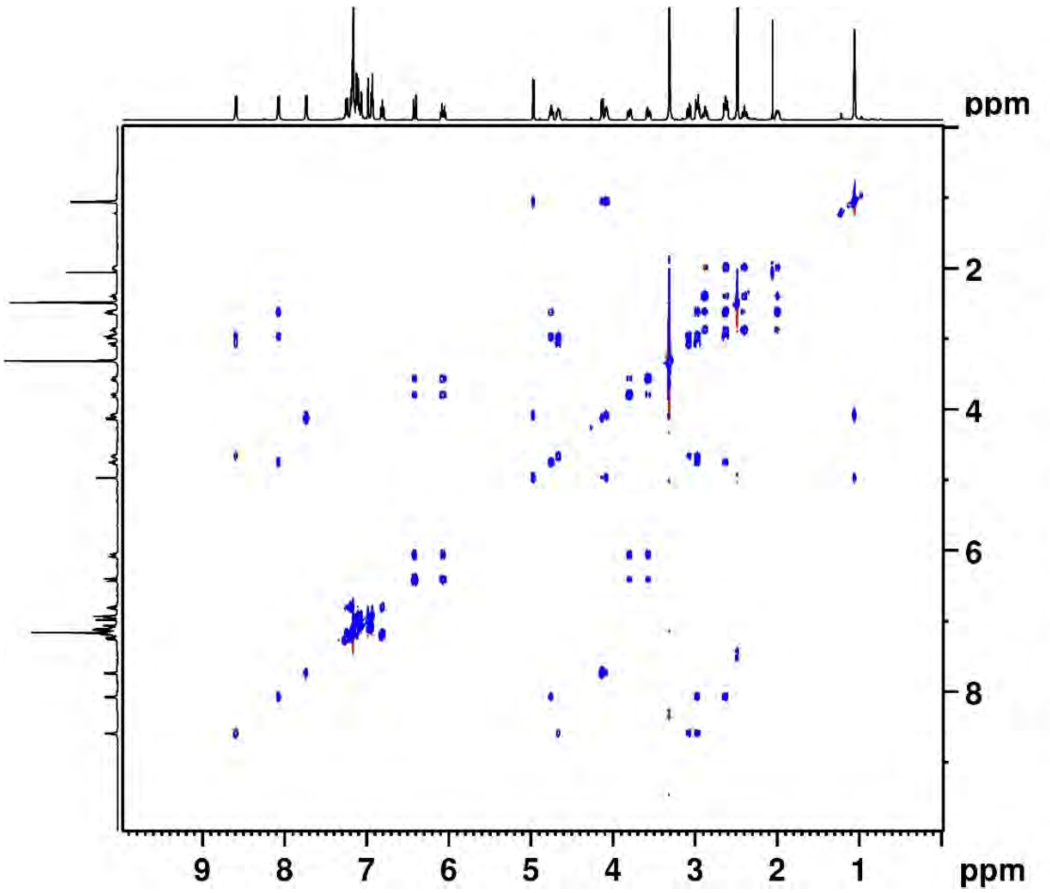
======== CHANNEL f2 ========
SFO2 125.7703648 MHz
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W

====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

*Macrocyclic Product **9b***







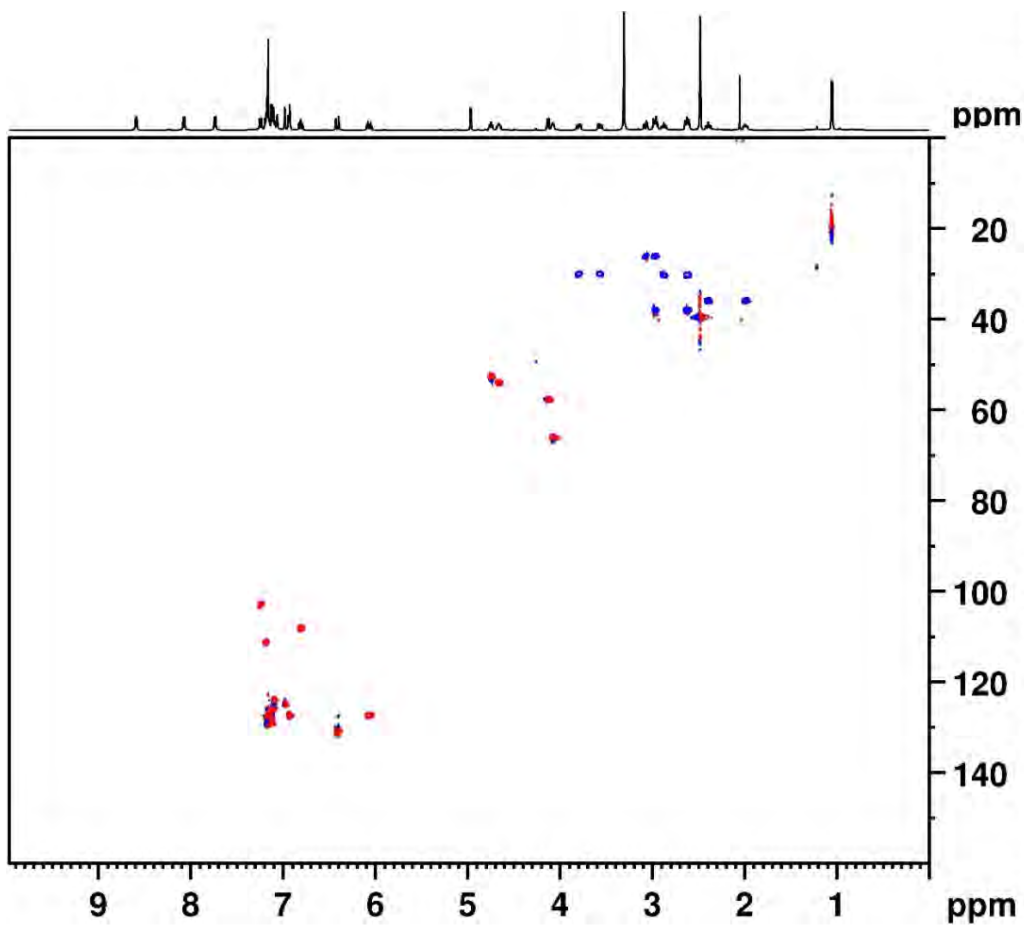
Current Data Parameters
NAME W-B2-3
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121110
Time 14.55
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG mlevetgp.js
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 37.94
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00019995 sec
L1 24

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PLW1 13.50000000 W
PLW10 0.84375000 W
SFO1 500.1325007 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.536406 Hz



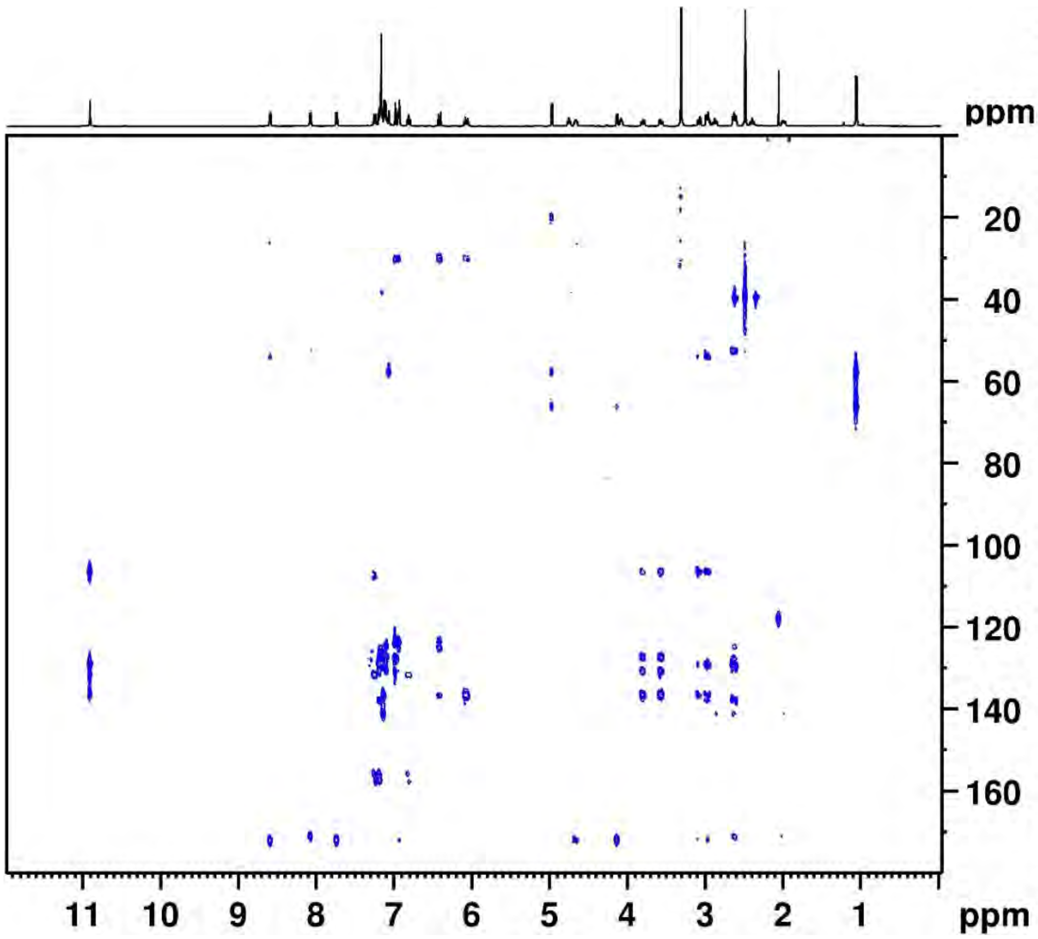
Current Data Parameters
NAME W-B2-3
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121110
Time 15.16
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcedetgp
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 202.91
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
IN0 0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P28 0 usec
PLW1 13.50000000 W
SFO1 500.1325007 MHz

======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 9.63 usec
P4 19.26 usec
PCPD2 70.00 usec
PLW2 23.01399994 W
PLW12 0.43557000 W
SFO2 125.7678496 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %



Current Data Parameters
NAME W-B2-3
EXPNO 6
PROCNO 1

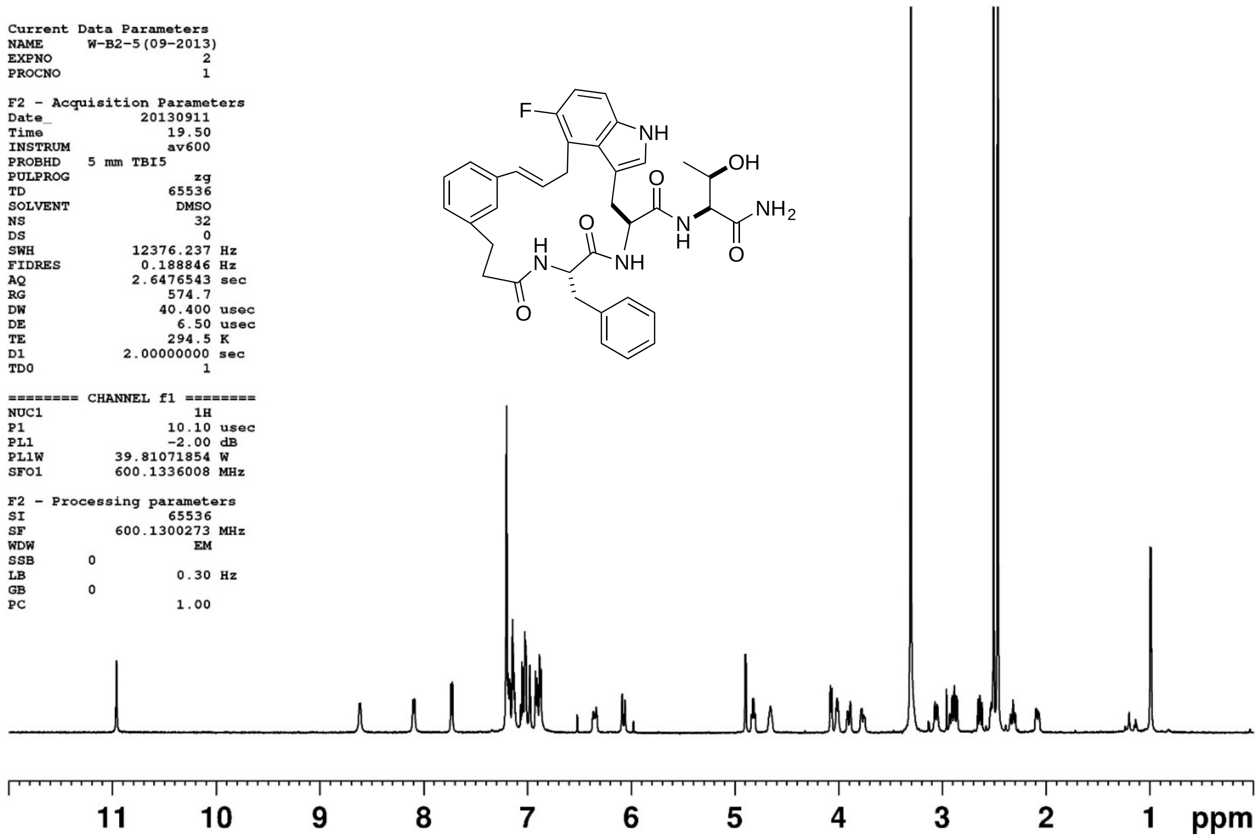
F2 - Acquisition Parameters
Date_ 20121110
Time 15.31
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 202.91
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec

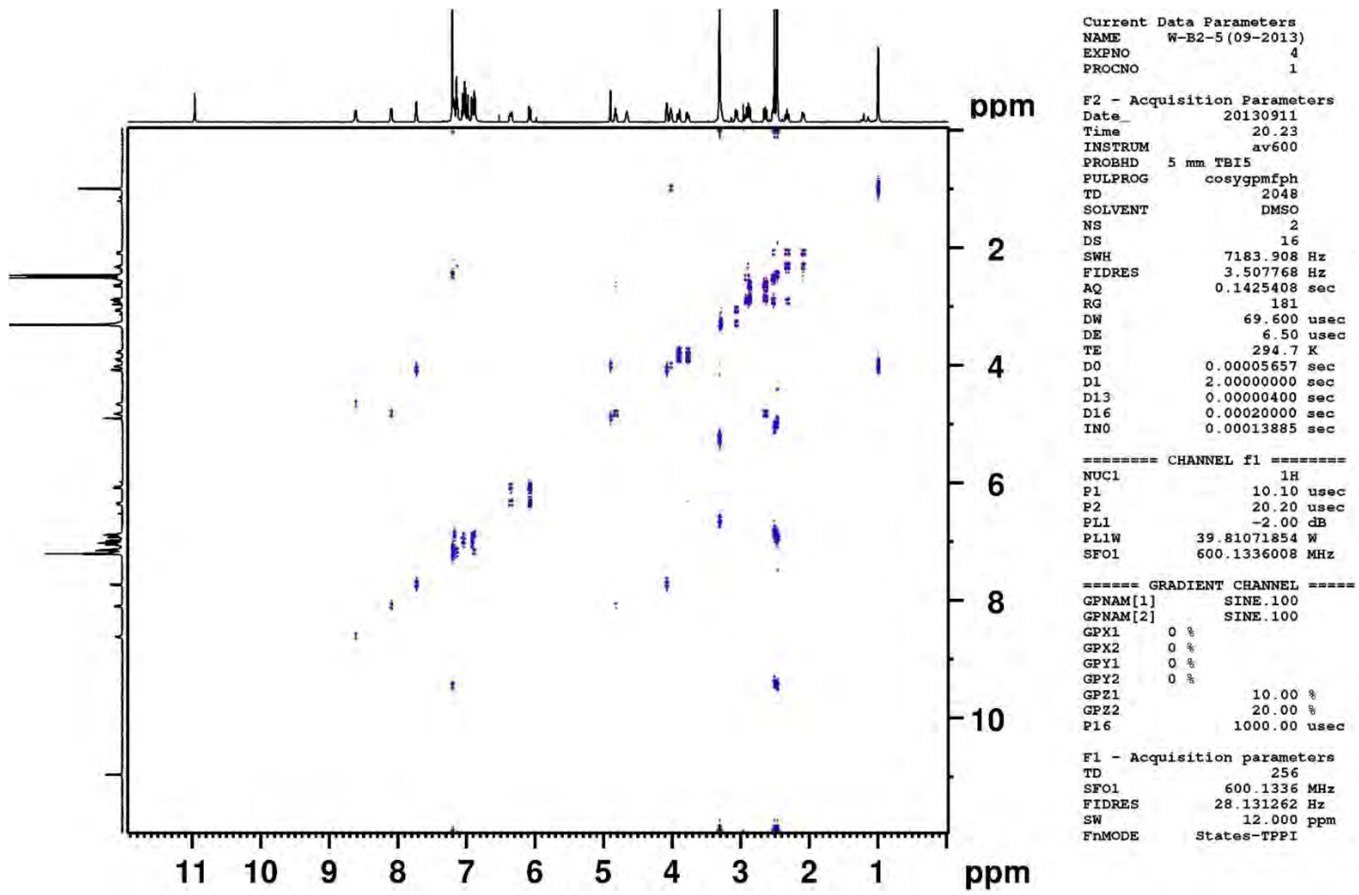
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W
SFO1 500.1330008 MHz

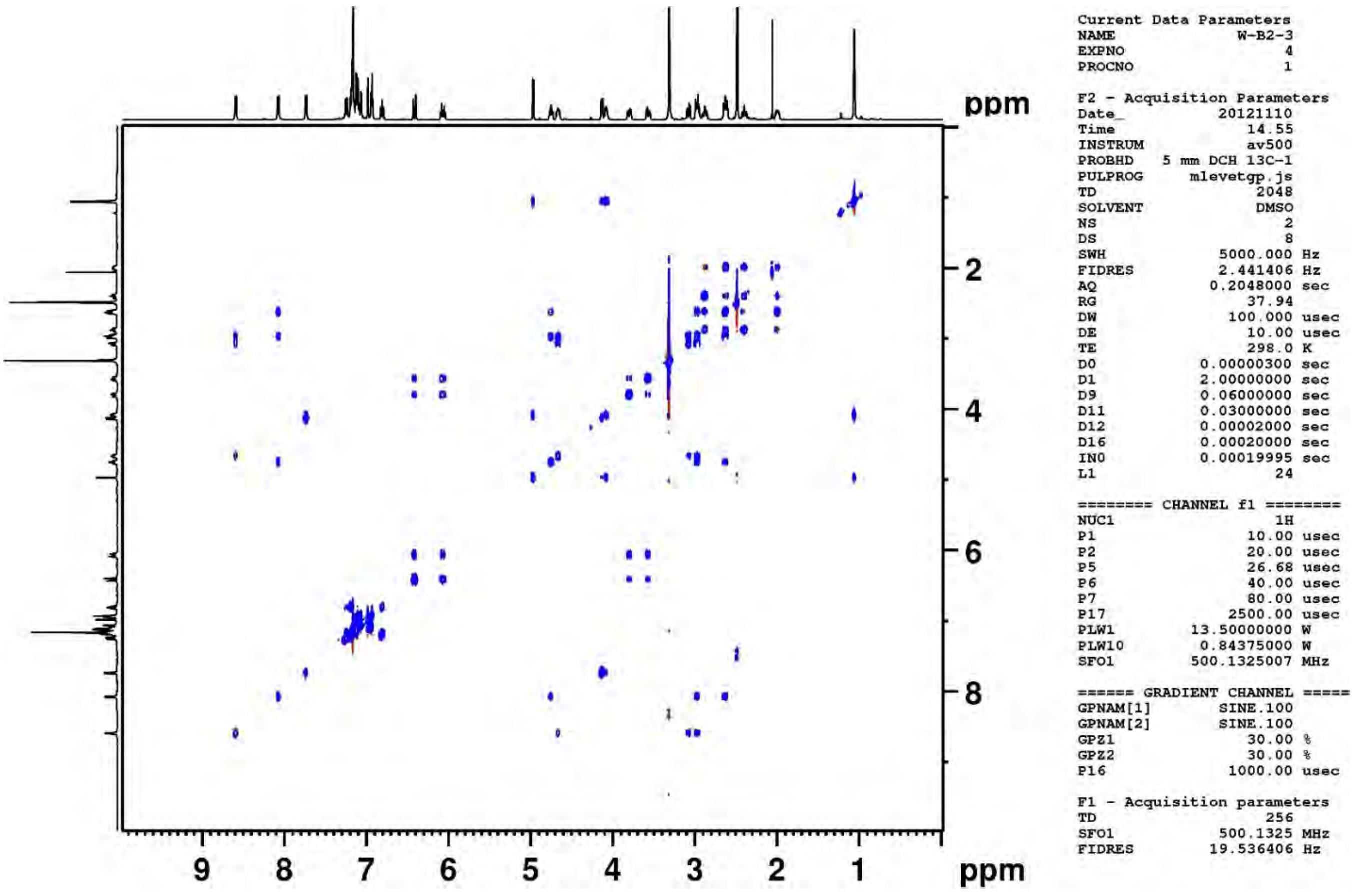
======== CHANNEL f2 ========
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
SFO2 125.7703648 MHz

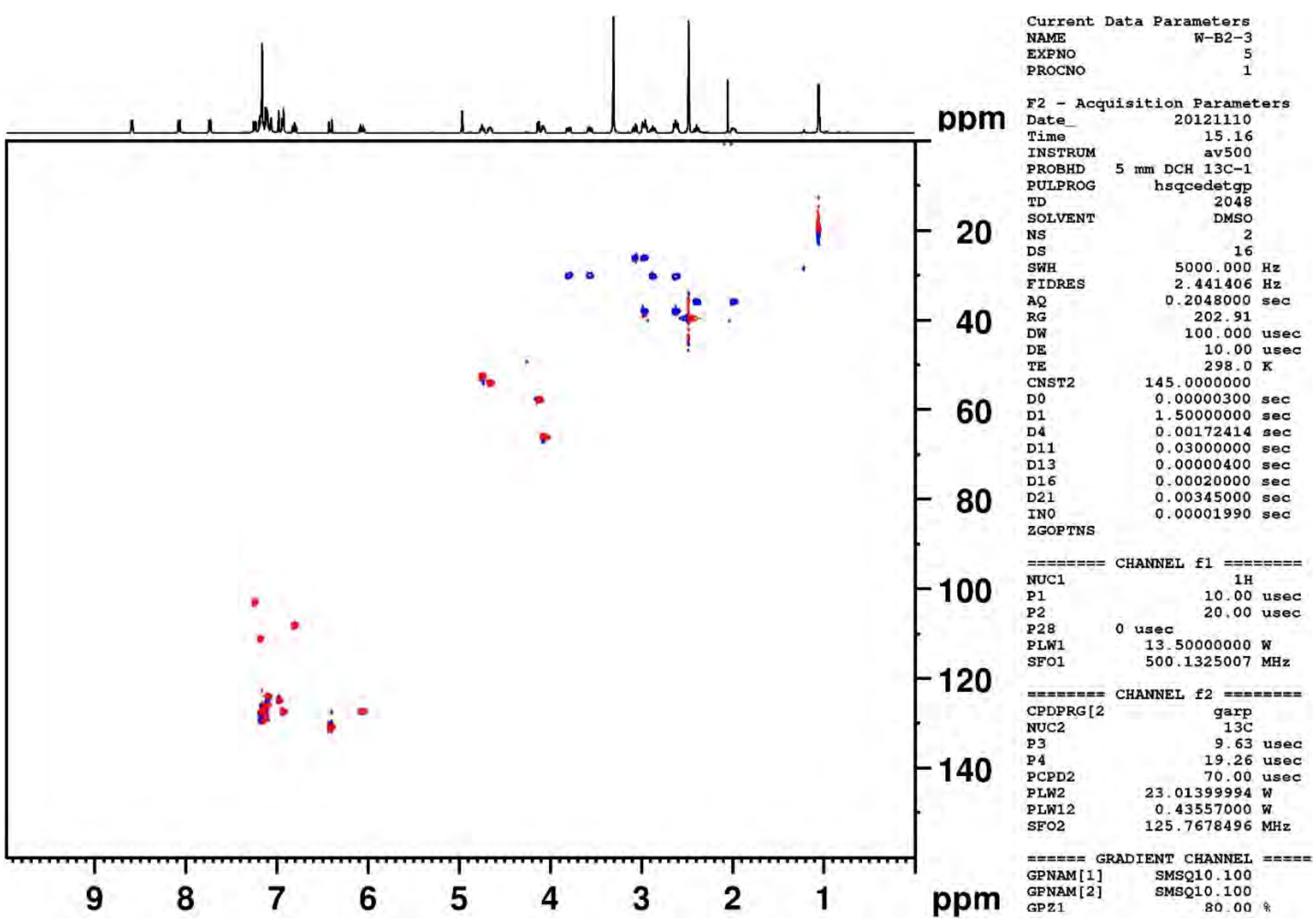
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

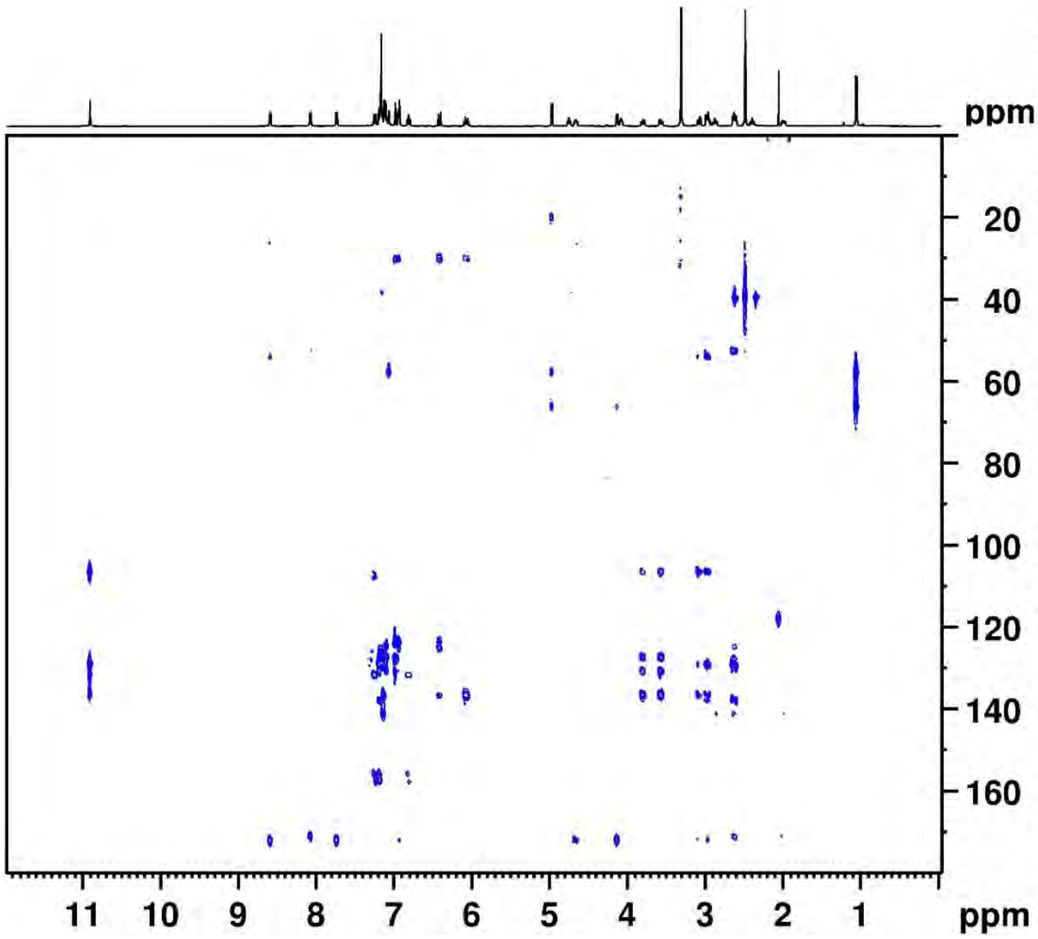
*Macrocyclic Product **9c***






ppm
2
4
6
8
9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME W-B2-3
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121110
Time 14.55
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG mlevetgp.js
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 37.94
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00019995 sec
L1 24
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PLW1 13.50000000 W
PLW10 0.84375000 W
SFO1 500.1325007 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.536406 Hz


ppm
20
40
60
80
100
120
140
9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME W-B2-3
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121110
Time 15.16
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcedetgp
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 202.91
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
IN0 0.00001990 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P28 0 usec
PLW1 13.50000000 W
SFO1 500.1325007 MHz
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 9.63 usec
P4 19.26 usec
PCPD2 70.00 usec
PLW2 23.01399994 W
PLW12 0.43557000 W
SFO2 125.7678496 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %



Current Data Parameters
NAME W-B2-3
EXPNO 6
PROCNO 1

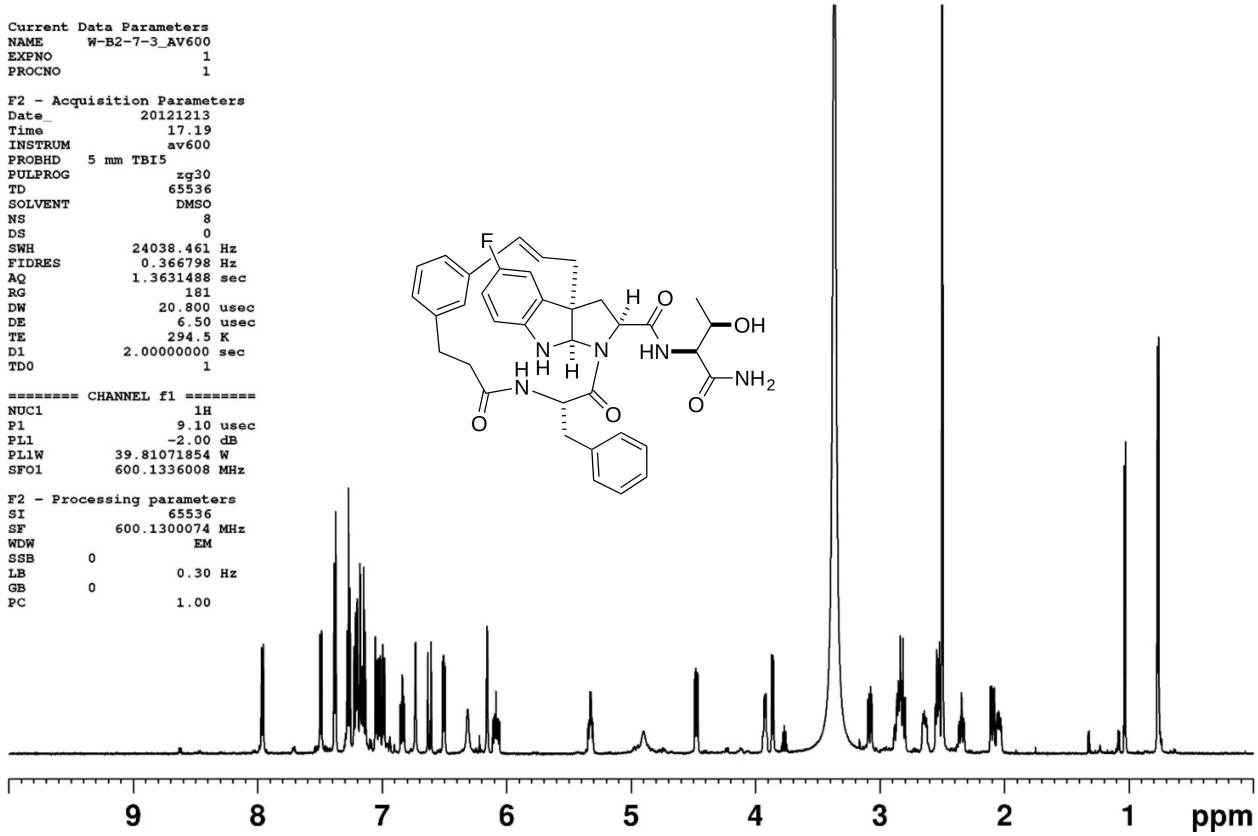
F2 - Acquisition Parameters
Date_ 20121110
Time 15.31
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 202.91
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec

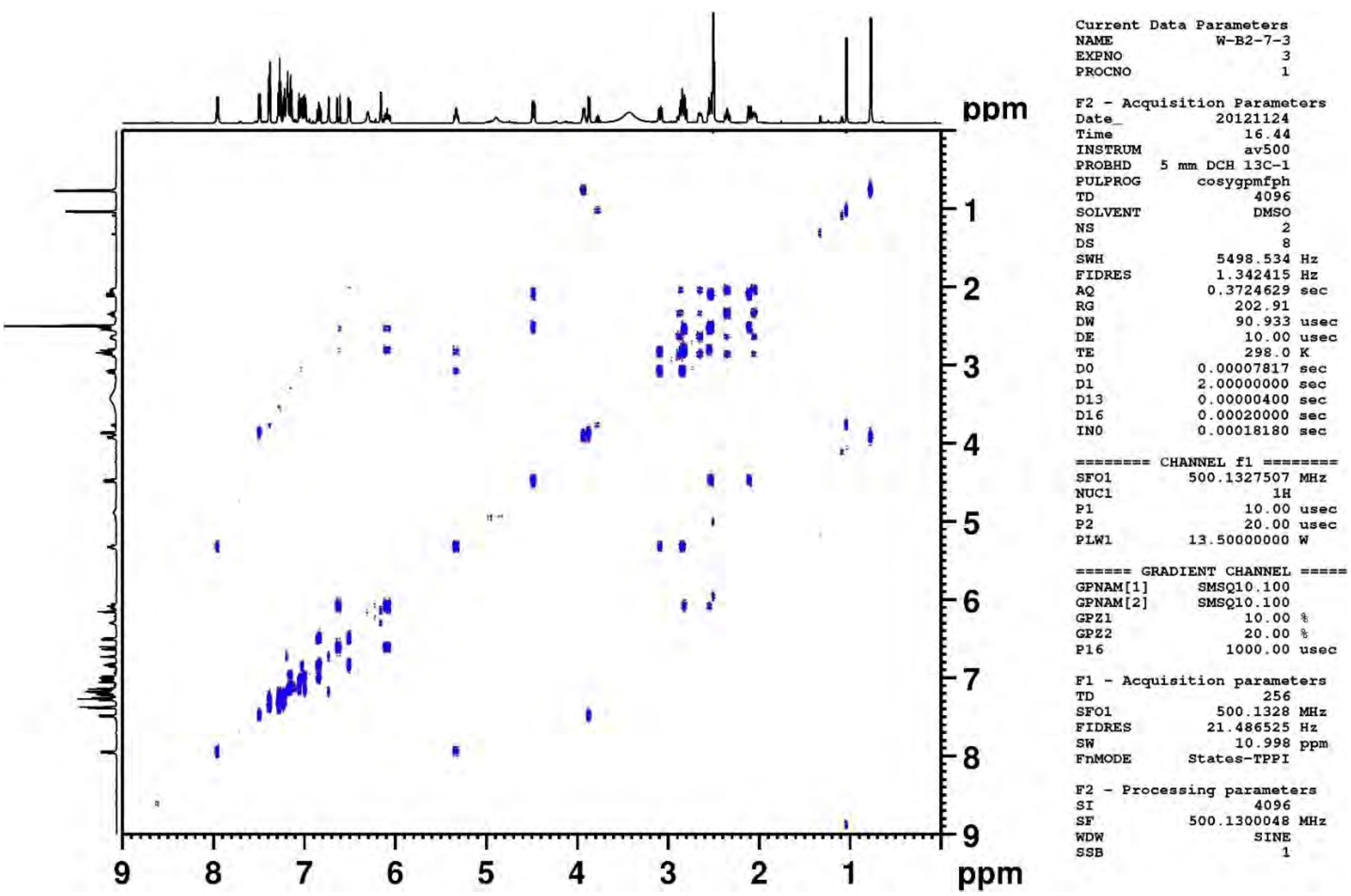
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W
SFO1 500.1330008 MHz

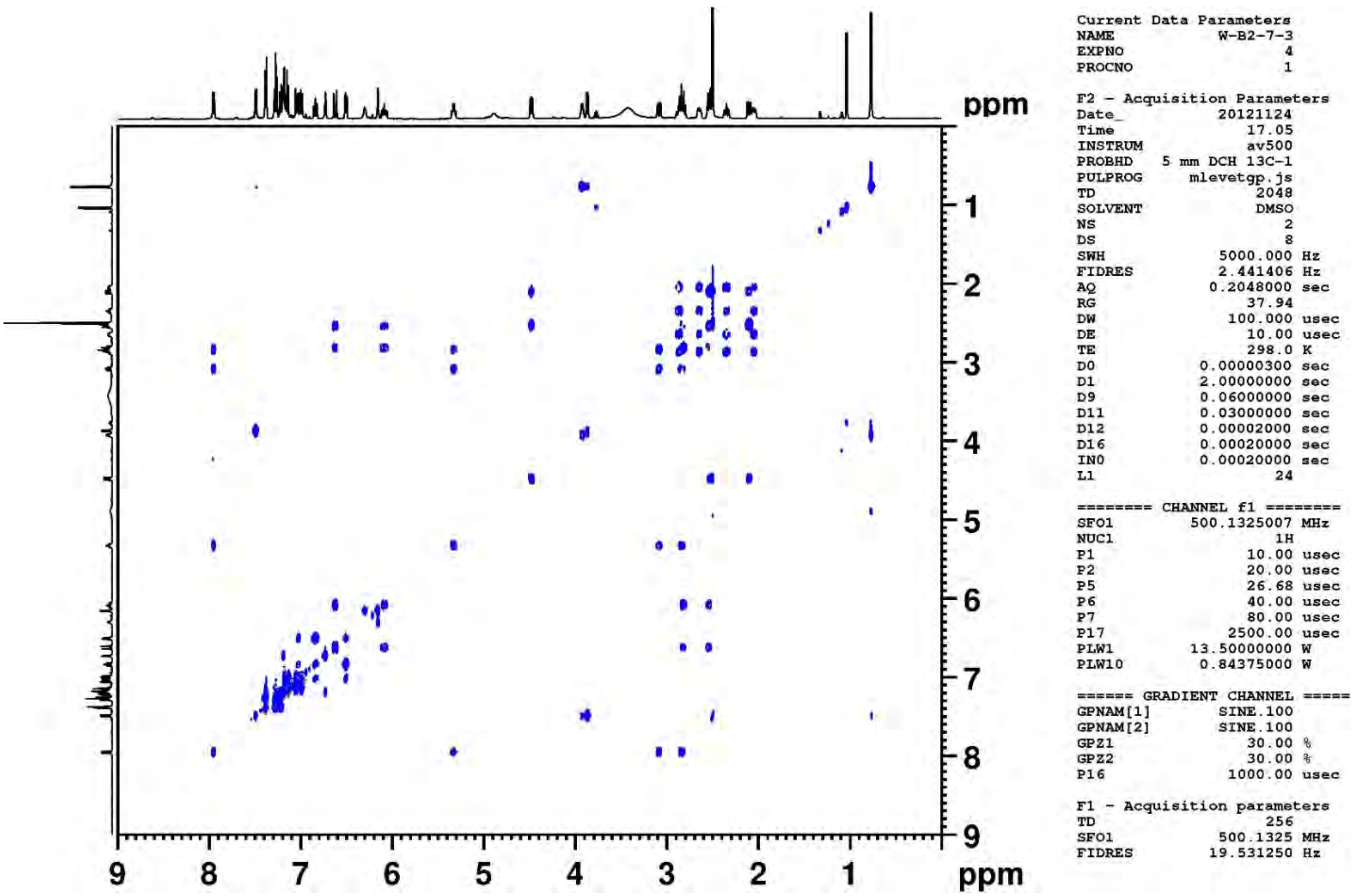
======== CHANNEL f2 ========
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
SFO2 125.7703648 MHz

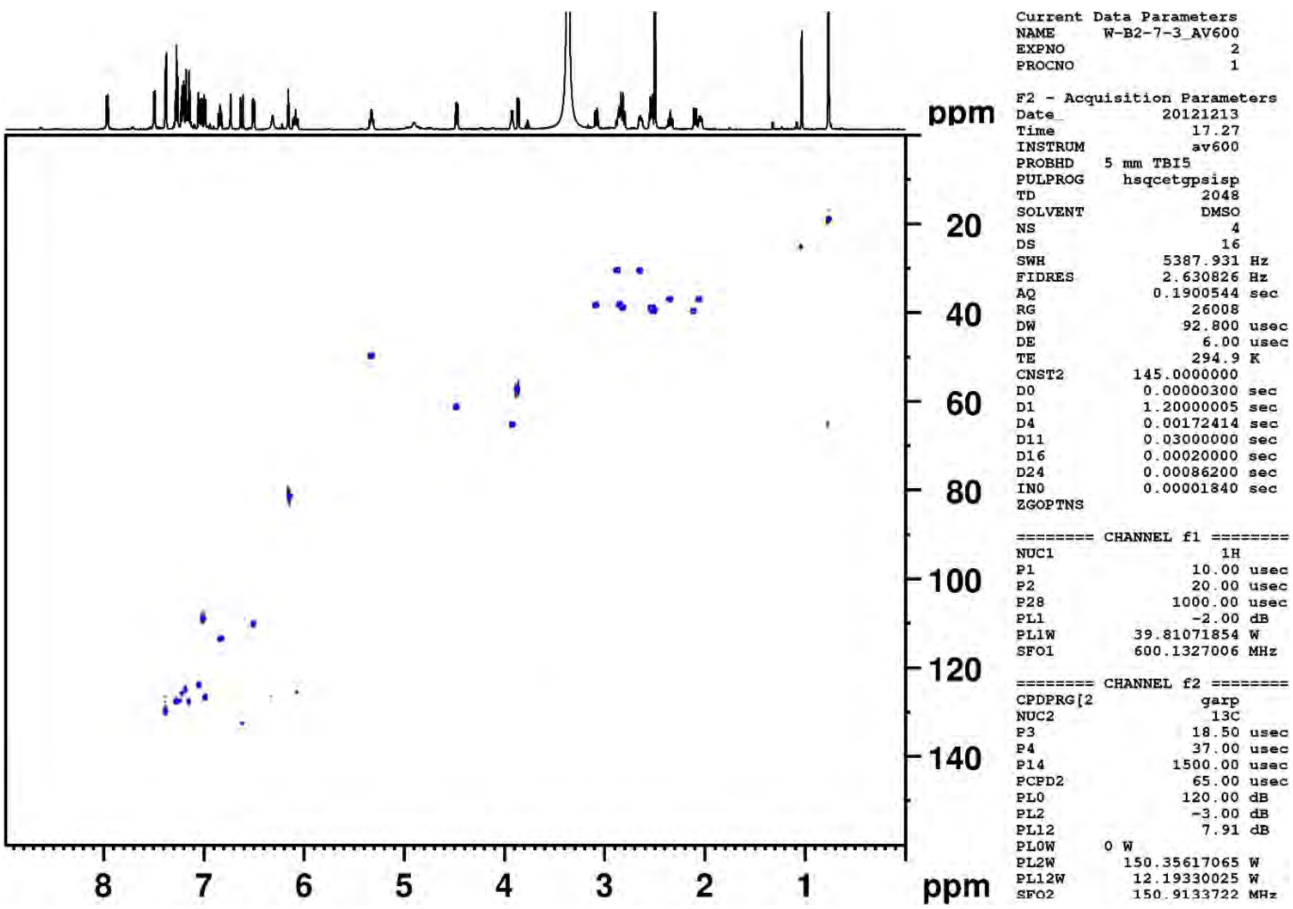
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

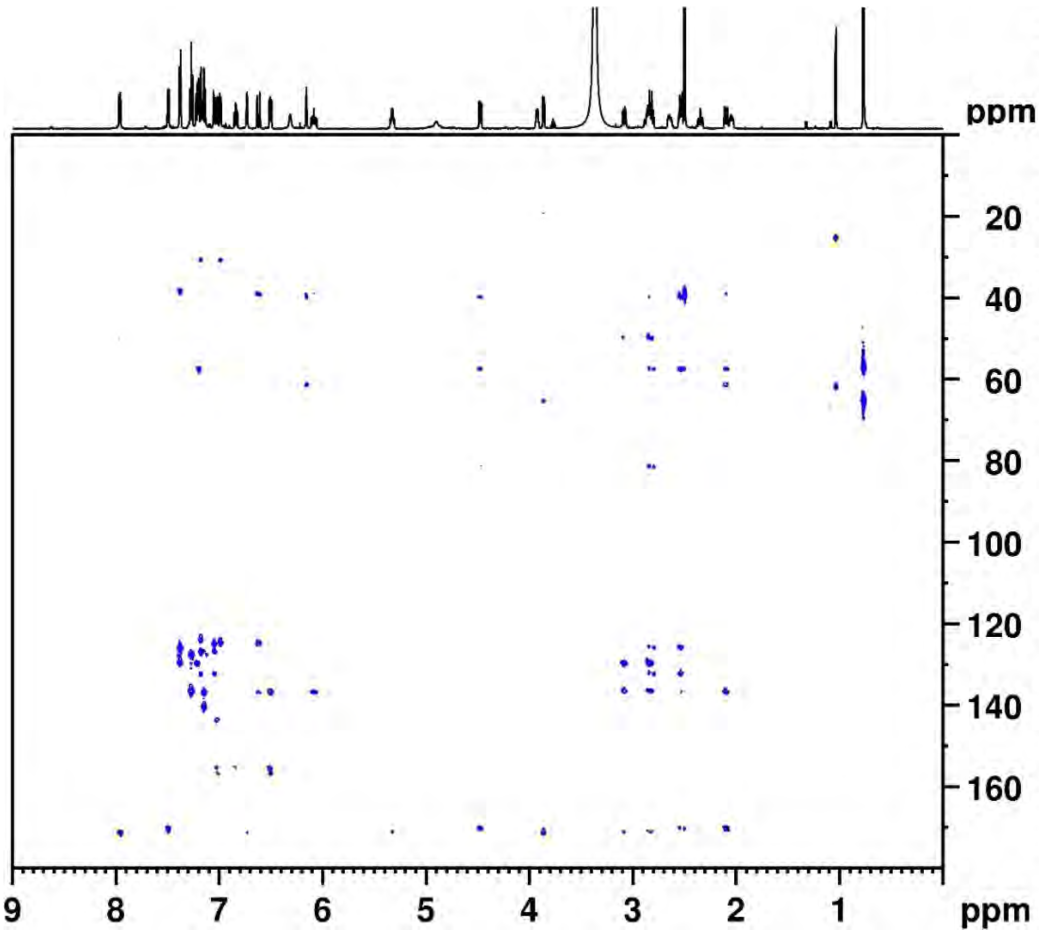
*Macrocyclic Product **9d***





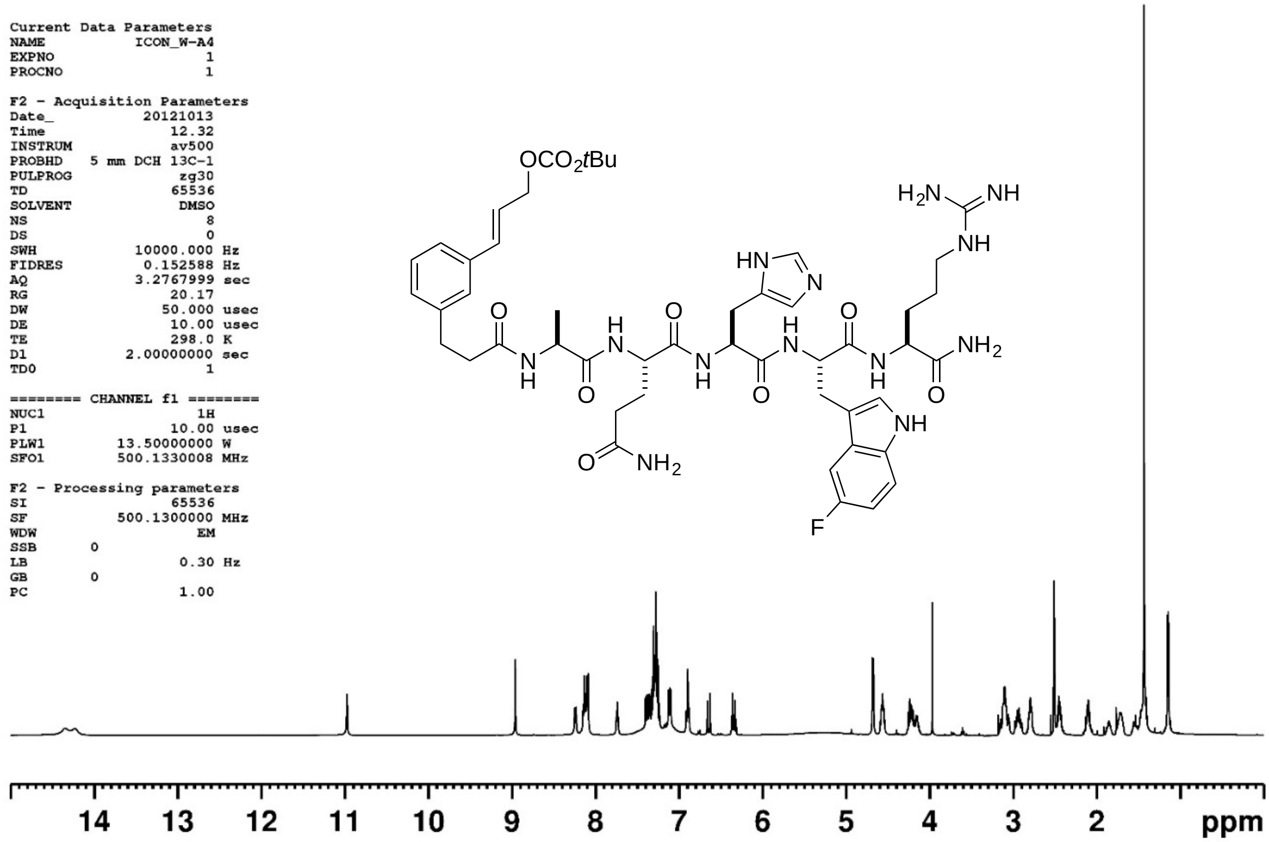

ppm
9
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME W-B2-7-3
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121124
Time 17.05
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG mlevetgp.js
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 37.94
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00020000 sec
L1 24
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PLW1 13.50000000 W
PLW10 0.84375000 W
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.531250 Hz

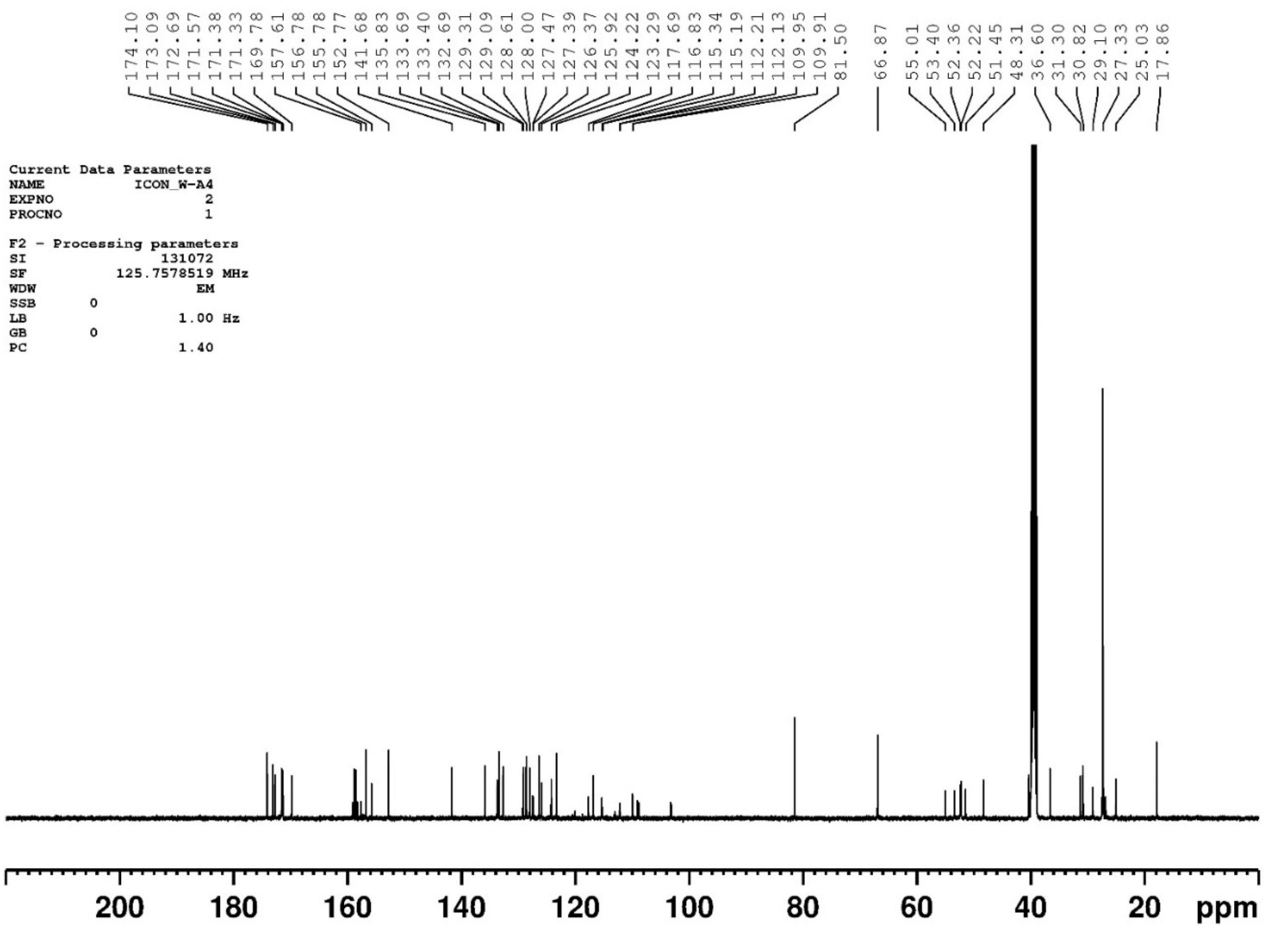

ppm
20
40
60
80
100
120
140
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME W-B2-7-3_AV600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121213
Time 17.27
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 5387.931 Hz
FIDRES 2.630826 Hz
AQ 0.1900544 sec
RG 26008
DW 92.800 usec
DE 6.00 usec
TE 294.9 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00001840 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1327006 MHz
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 18.50 usec
P4 37.00 usec
P14 1500.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.91 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 12.19330025 W
SFO2 150.9133722 MHz




```
Current Data Parameters
NAME       W-B2-7-3_AV600
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121213
Time                17.52
INSTRUM             av600
PROBHD    5 mm TBI5
PULPROG      hmbcgpl2ndqf
TD                   2048
SOLVENT              DMSO
NS                     16
DS                     16
SWH              6009.615 Hz
FIDRES           2.934382 Hz
AQ              0.1703936 sec
RG                  29193
DW                 83.200 usec
DE                   6.50 usec
TE                  297.1 K
CNST6         120.0000000
CNST7         160.0000000
CNST13          7.0000000
D0             0.00000300 sec
D1             1.20000005 sec
D6             0.07142857 sec
D16            0.00020000 sec
IN0            0.00001655 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
P2                  20.00 usec
PL1                 -2.00 dB
PL1W          39.81071854 W
SFO1        600.1330006 MHz

======== CHANNEL f2 ========
NUC2                  13C
P3                  18.50 usec
PL2                 -3.00 dB
PL2W         150.35617065 W
SFO2        150.9163903 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPNAM[3]        SINE.100
GPNAM[4]        SINE.100
GPNAM[5]        SINE.100
GPNAM[6]        SINE.100
GPX1        0 %
GPX2        0 %
```

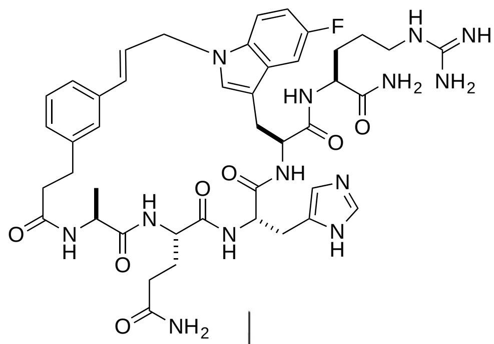
Acyclic Precursor ***10***

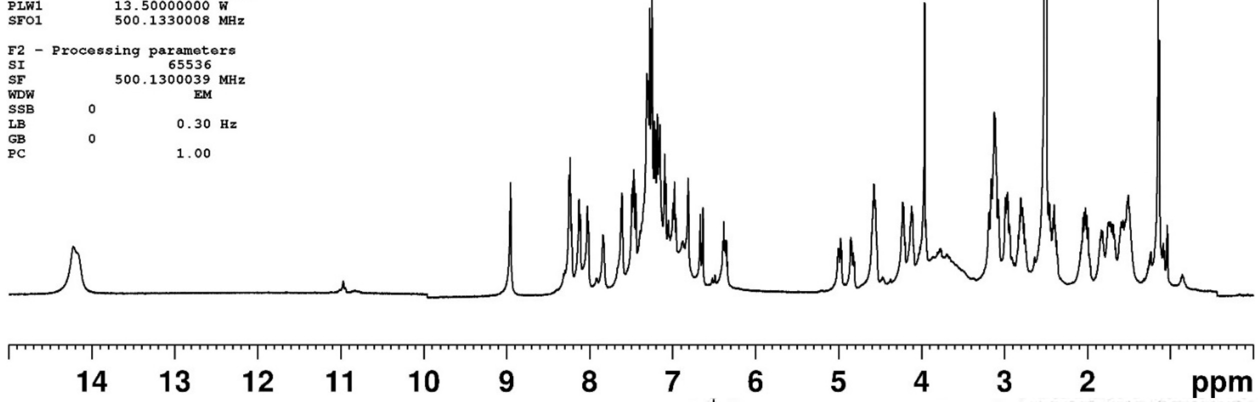




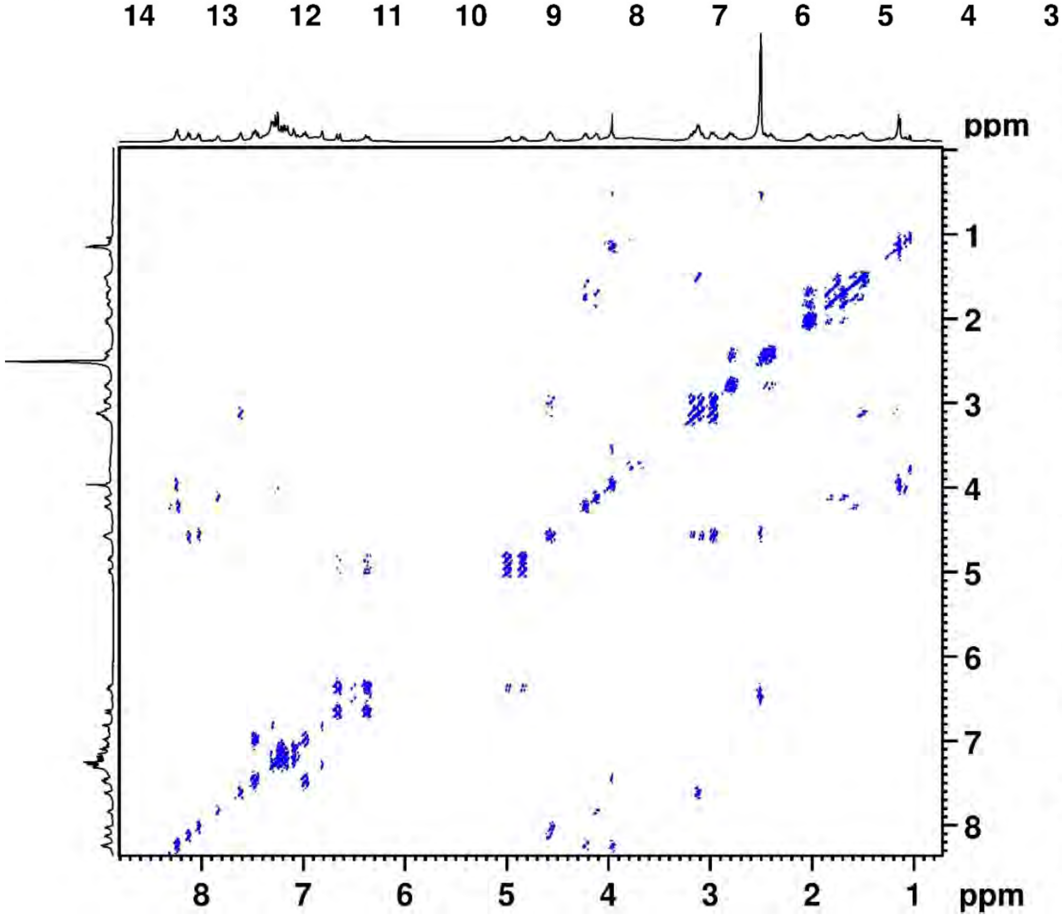
*Macrocyclic Product **11a***

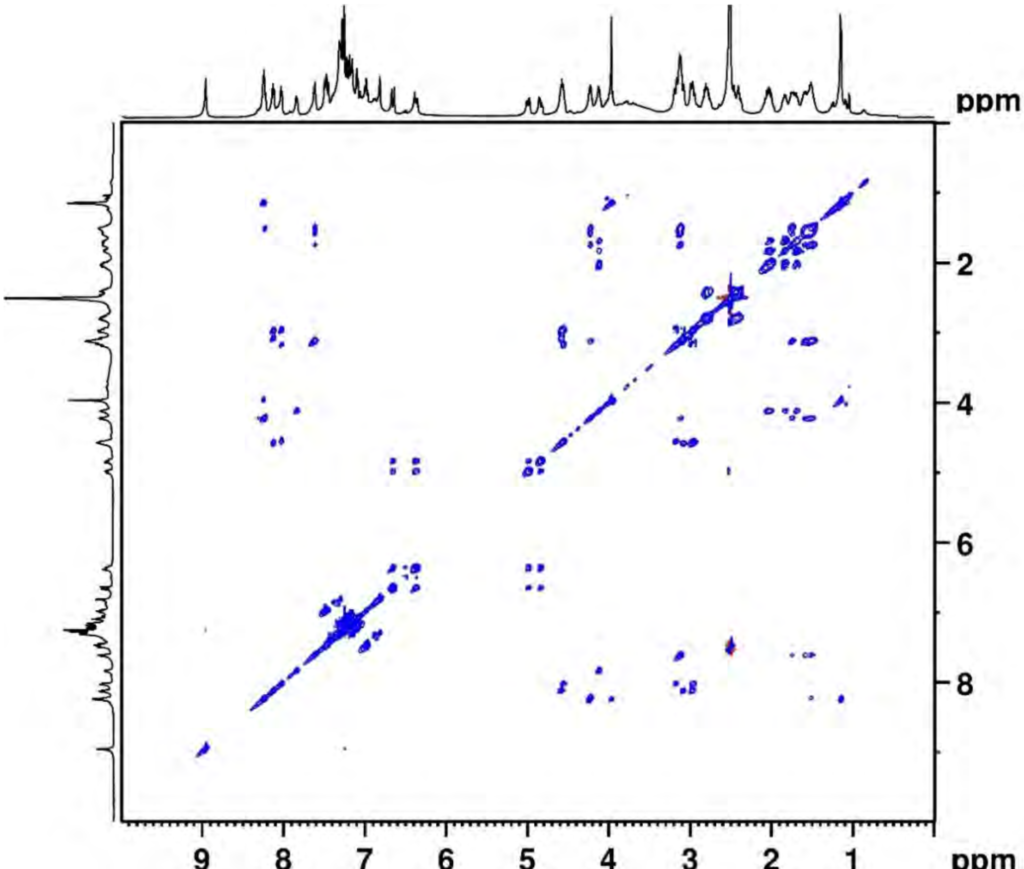
```
Current Data Parameters
NAME              W-A4-5-1
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20121027
Time                 17.23
INSTRUM              av500
PROBHD   5 mm DCH 13C-1
PULPROG                 zg
TD                   65536
SOLVENT               DMSO
NS                       8
DS                       0
SWH             10000.000 Hz
FIDRES           0.152588 Hz
AQ              3.2767999 sec
RG                       7
DW                  50.000 usec
DE                  10.00 usec
TE                  298.0 K
D1             2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  10.00 usec
PLW1          13.50000000 W
SFO1          500.1330008 MHz

F2 - Processing parameters
SI                   65536
SF            500.1300039 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```






```
Current Data Parameters
NAME              W-A4-5-1
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20121027
Time                 17.24
INSTRUM              av500
PROBHD   5 mm DCH 13C-1
PULPROG         cosygpmfph
TD                    4096
SOLVENT               DMSO
NS                       2
DS                       8
SWH              5498.534 Hz
FIDRES           1.342415 Hz
AQ              0.3724629 sec
RG                  202.91
DW                  90.933 usec
DE                  10.00 usec
TE                  298.0 K
D0             0.00008724 sec
D1             2.00000000 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00019995 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  10.00 usec
P2                  20.00 usec
PLW1          13.50000000 W
SFO1          500.1327507 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SMSQ10.100
GPNAM[2]        SMSQ10.100
GPZ1                10.00 %
GPZ2                20.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                     256
SFO1              500.1328 MHz
FIDRES           19.536423 Hz
SW                  10.000 ppm
FnMODE         States-TPPI

F2 - Processing parameters
SI                    4096
SF            500.1300044 MHz
WDW                   SINE
SSB                      1
```





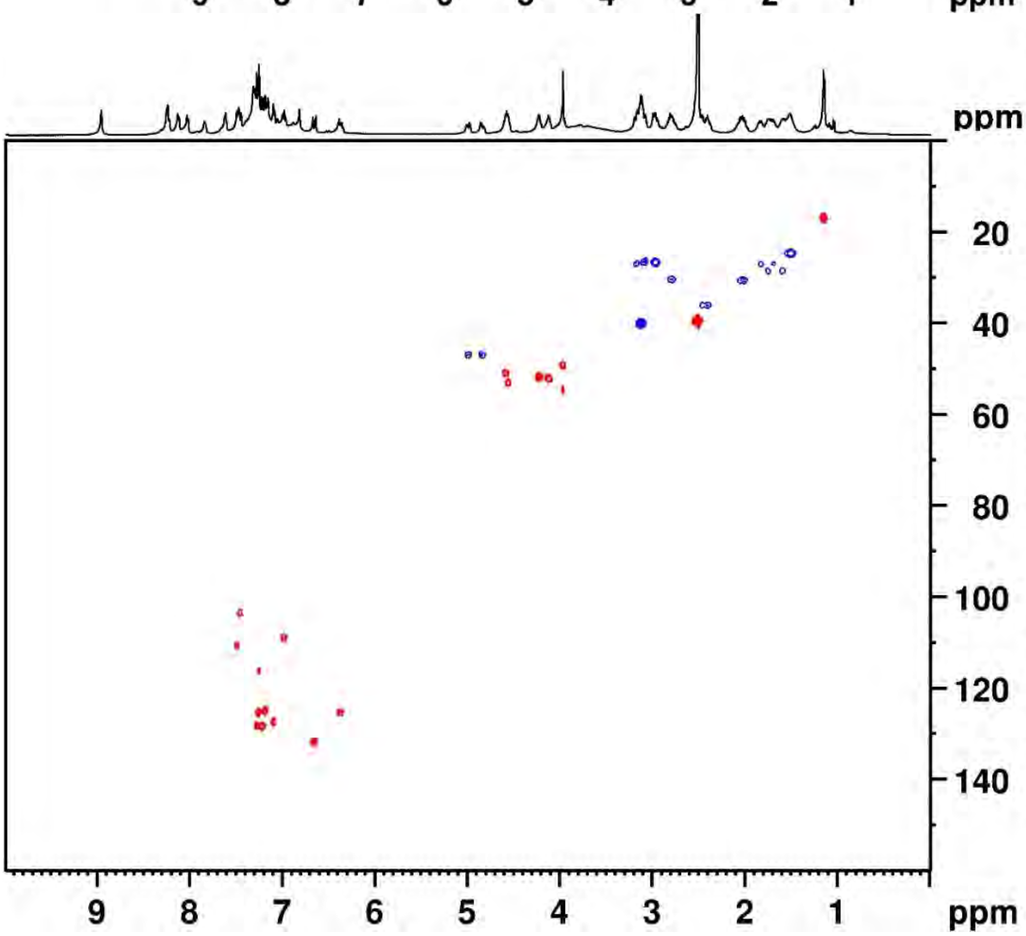
Current Data Parameters
NAME W-A4-5-1
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121027
Time 17.45
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG mlevetgp.js
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 37.94
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00019995 sec
L1 24

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PLW1 13.50000000 W
PLW10 0.84375000 W
SFO1 500.1325007 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.536406 Hz



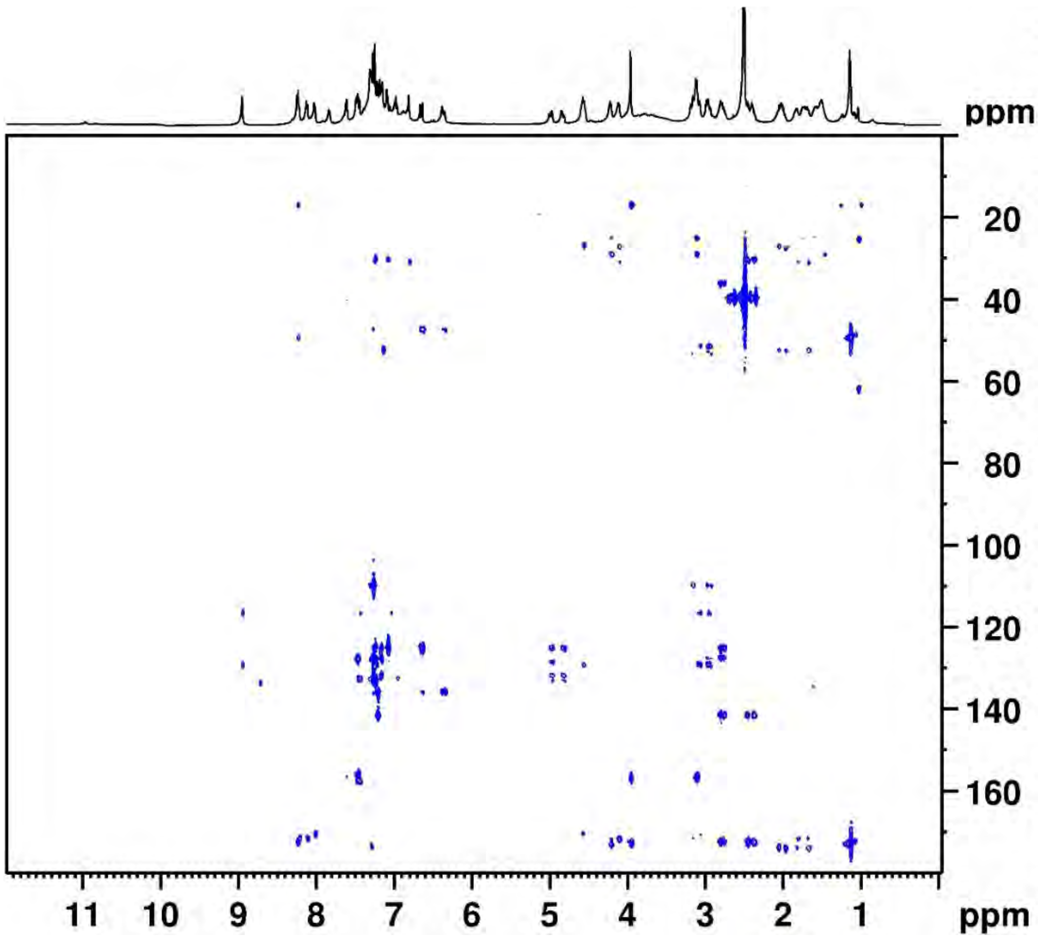
Current Data Parameters
NAME W-A4-5-1
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121027
Time 18.06
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcedetgp
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 202.91
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
IN0 0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P28 0 usec
PLW1 13.50000000 W
SFO1 500.1325007 MHz

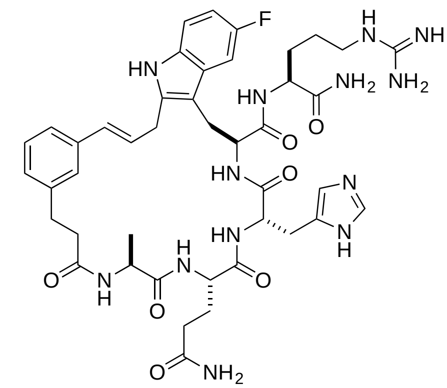
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 9.63 usec
P4 19.26 usec
PCPD2 70.00 usec
PLW2 23.01399994 W
PLW12 0.43557000 W
SFO2 125.7678496 MHz

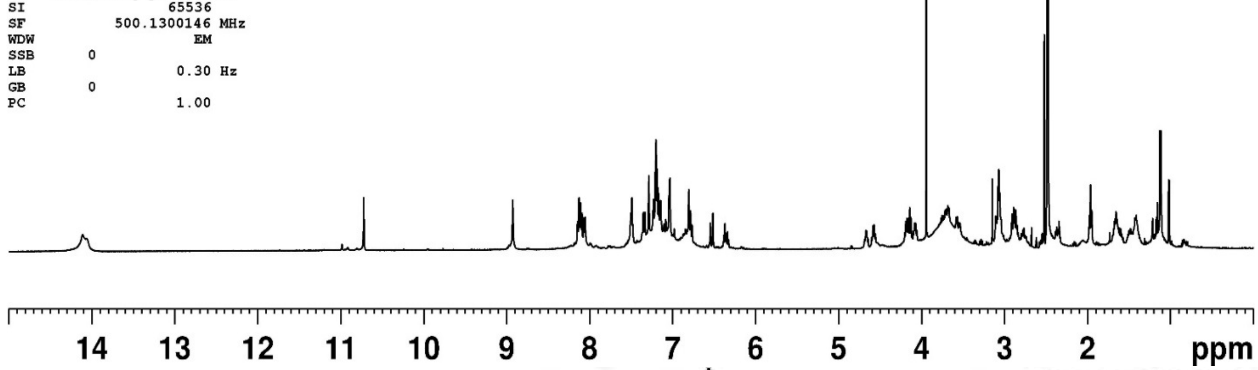
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %

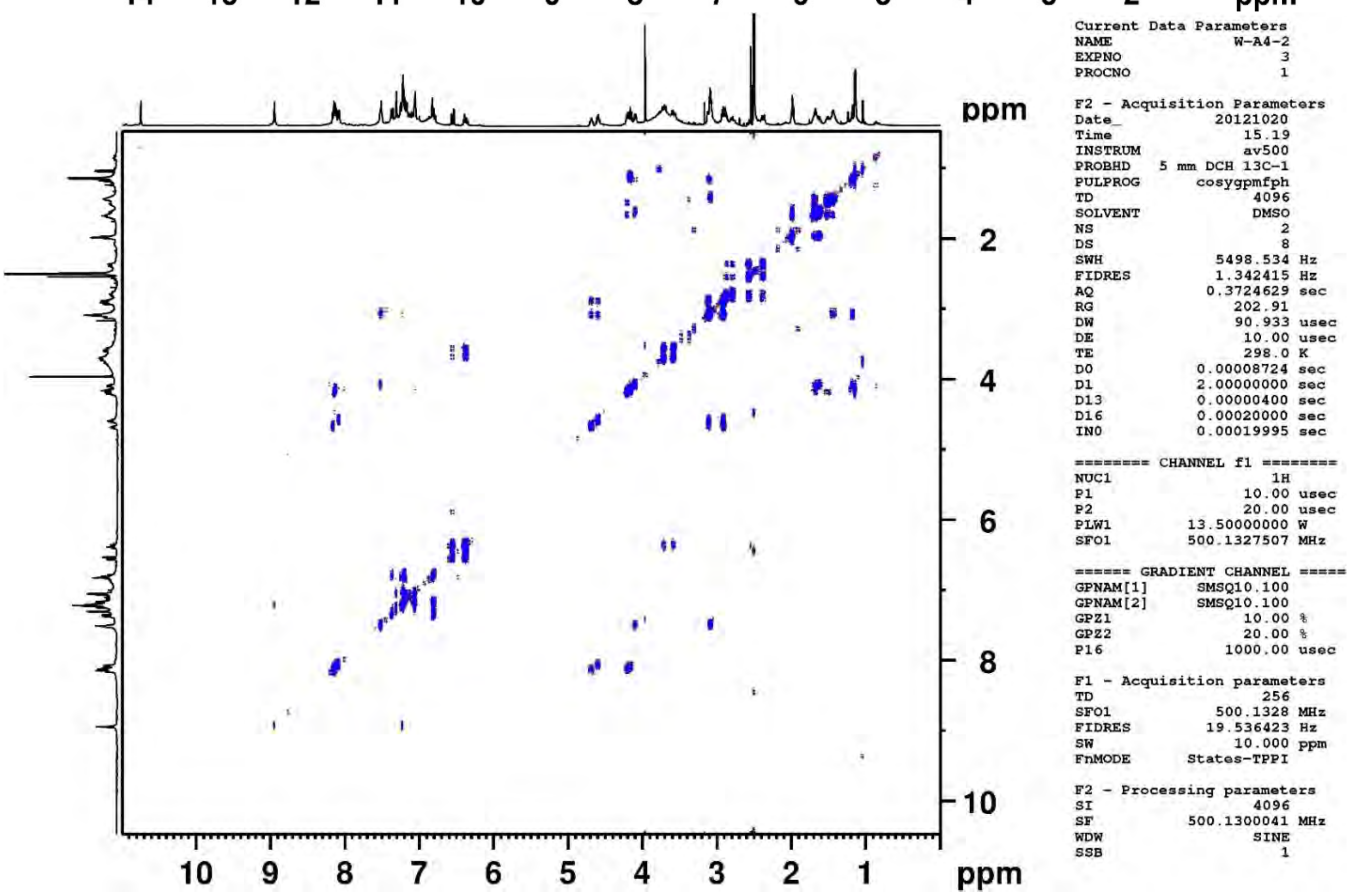


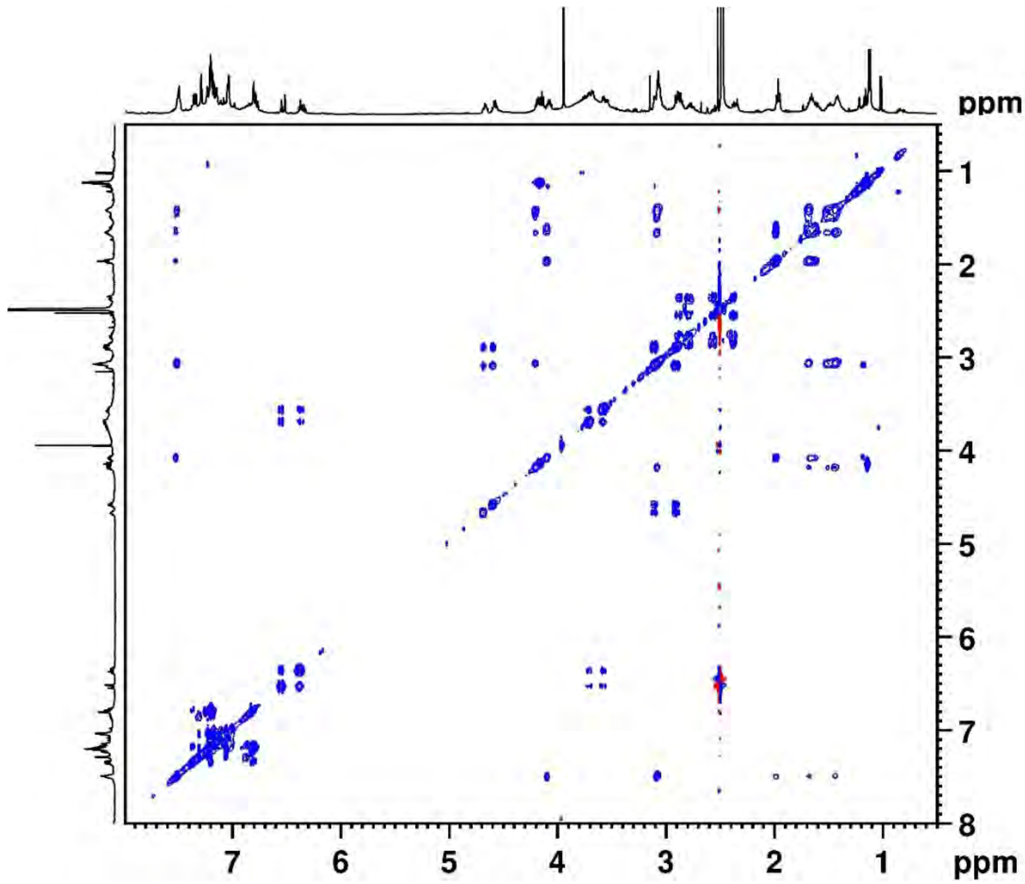

```
Current Data Parameters
NAME             W-A4-5-1
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121027
Time                18.36
INSTRUM             av500
PROBHD    5 mm DCH 13C-1
PULPROG       hmbcgpl2ndqf
TD                   2048
SOLVENT              DMSO
NS                     10
DS                     16
SWH              6009.615 Hz
FIDRES           2.934382 Hz
AQ              0.1703936 sec
RG                 202.91
DW                 83.200 usec
DE                  10.00 usec
TE                  298.0 K
CNST6         120.0000000
CNST7         160.0000000
CNST13          7.0000000
D0             0.00000300 sec
D1             1.50000000 sec
D6             0.07142857 sec
D16            0.00020000 sec
IN0            0.00001990 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
P2                  20.00 usec
PLW1          13.50000000 W
SFO1          500.1330008 MHz

======== CHANNEL f2 ========
NUC2                  13C
P3                   9.63 usec
PLW2          23.01399994 W
SFO2          125.7703648 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[2]       SMSQ10.100
GPNAM[3]       SMSQ10.100
GPNAM[4]       SMSQ10.100
GPNAM[5]       SMSQ10.100
GPNAM[6]       SMSQ10.100
GPZ1                50.00 %
GPZ2                30.00 %
GPZ3                40.10 %
GPZ4                15.00 %
```

Macrocyclic Product ***11b***

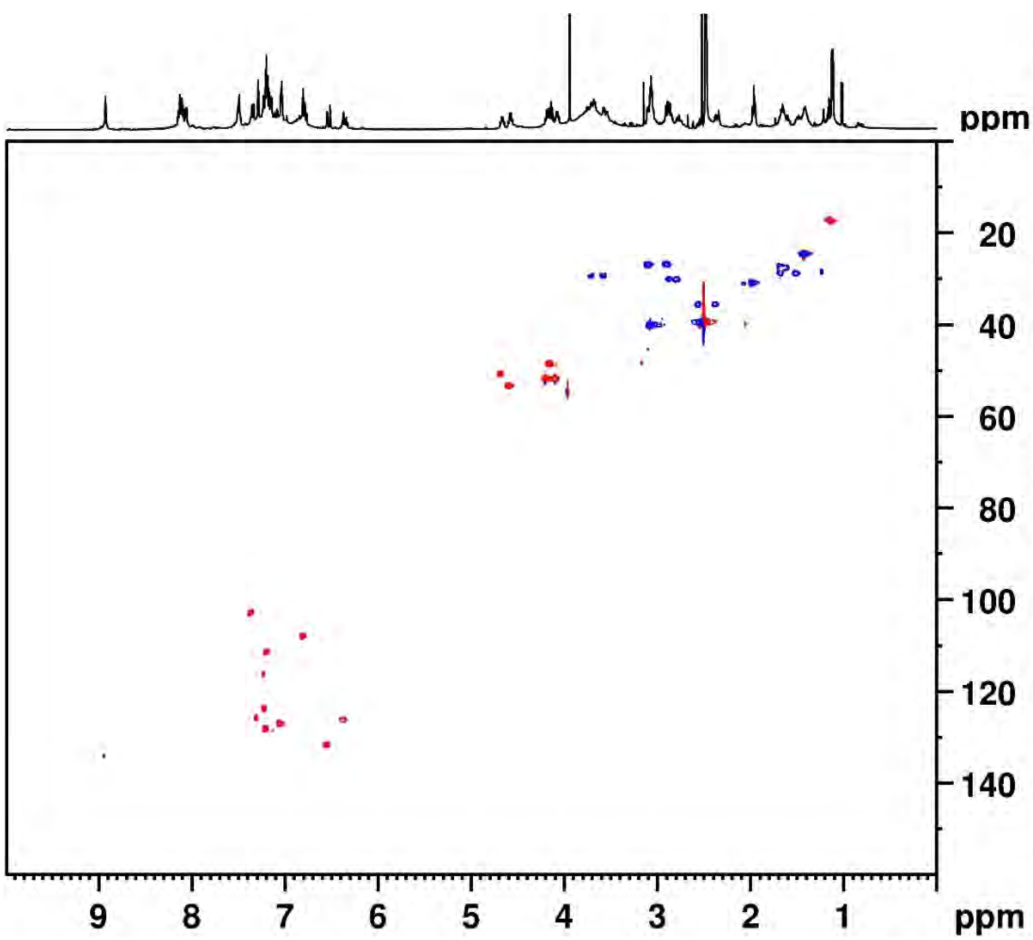




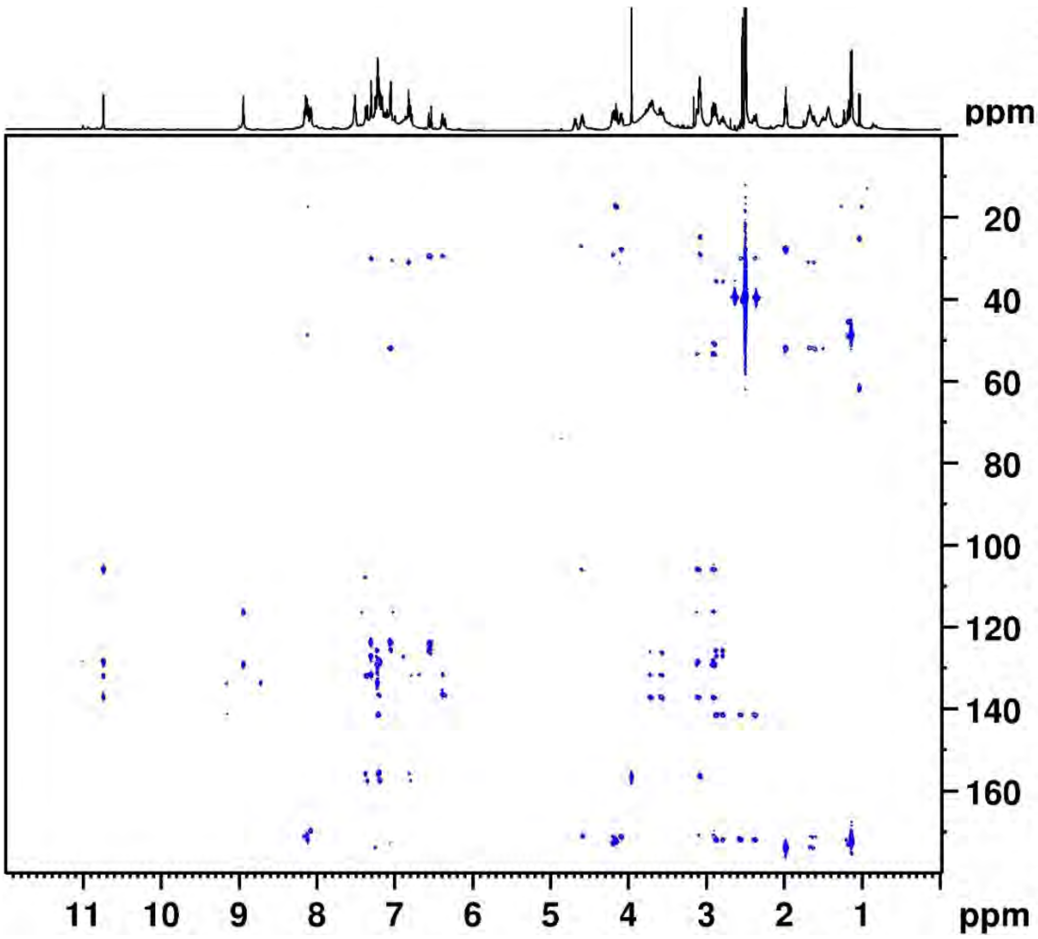





```
Current Data Parameters
NAME                W-A4-2
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20121020
Time                 15.40
INSTRUM              av500
PROBHD   5 mm DCH 13C-1
PULPROG        mlevetgp.js
TD                    2048
SOLVENT               DMSO
NS                       2
DS                       8
SWH            4000.000 Hz
FIDRES         1.953125 Hz
AQ            0.2560000 sec
RG                   37.94
DW              125.000 usec
DE               10.00 usec
TE                298.0 K
D0           0.00000300 sec
D1           2.00000000 sec
D9           0.06000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
D16          0.00020000 sec
IN0          0.00024995 sec
L1                      24

======== CHANNEL f1 ========
NUC1                    1H
P1               10.00 usec
P2               20.00 usec
P5               26.68 usec
P6               40.00 usec
P7               80.00 usec
P17            2500.00 usec
PLW1        13.50000000 W
PLW10        0.84375000 W
SFO1      500.1320005 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPZ1               30.00 %
GPZ2               30.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                     256
SFO1           500.132 MHz
FIDRES        15.629138 Hz
```



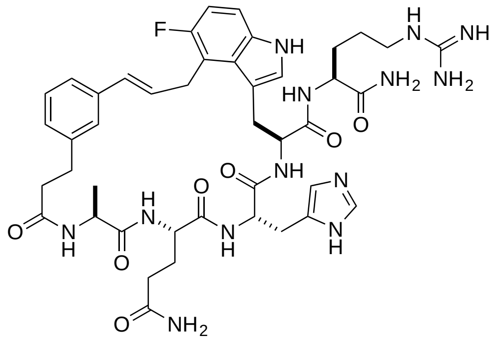

```
Current Data Parameters
NAME                W-A4-2
EXPNO                    6
PROCNO                   1

F2 - Acquisition Parameters
Date_             20121022
Time                 17.40
INSTRUM              av500
PROBHD   5 mm DCH 13C-1
PULPROG        hsqcedetgp
TD                    2048
SOLVENT               DMSO
NS                       4
DS                      16
SWH            5000.000 Hz
FIDRES         2.441406 Hz
AQ            0.2048000 sec
RG                  202.91
DW              100.000 usec
DE               10.00 usec
TE                298.0 K
CNST2          145.0000000
D0           0.00000300 sec
D1           1.50000000 sec
D4           0.00172414 sec
D11          0.03000000 sec
D13          0.00000400 sec
D16          0.00020000 sec
D21          0.00345000 sec
IN0          0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                    1H
P1               10.00 usec
P2               20.00 usec
P28        0 usec
PLW1        13.50000000 W
SFO1      500.1325007 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                   13C
P3                9.63 usec
P4               19.26 usec
PCPD2            70.00 usec
PLW2        23.01399994 W
PLW12        0.43557000 W
SFO2      125.7678496 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SMSQ10.100
GPNAM[2]        SMSQ10.100
GPZ1               80.00 %
```

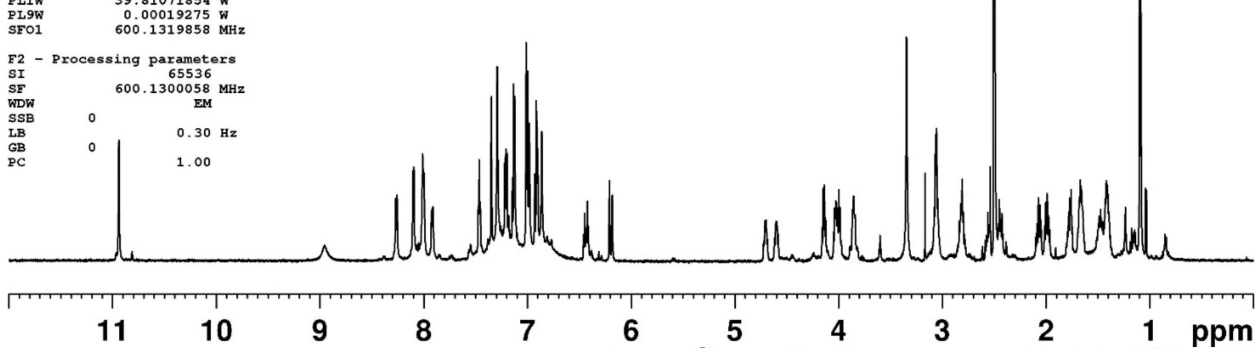


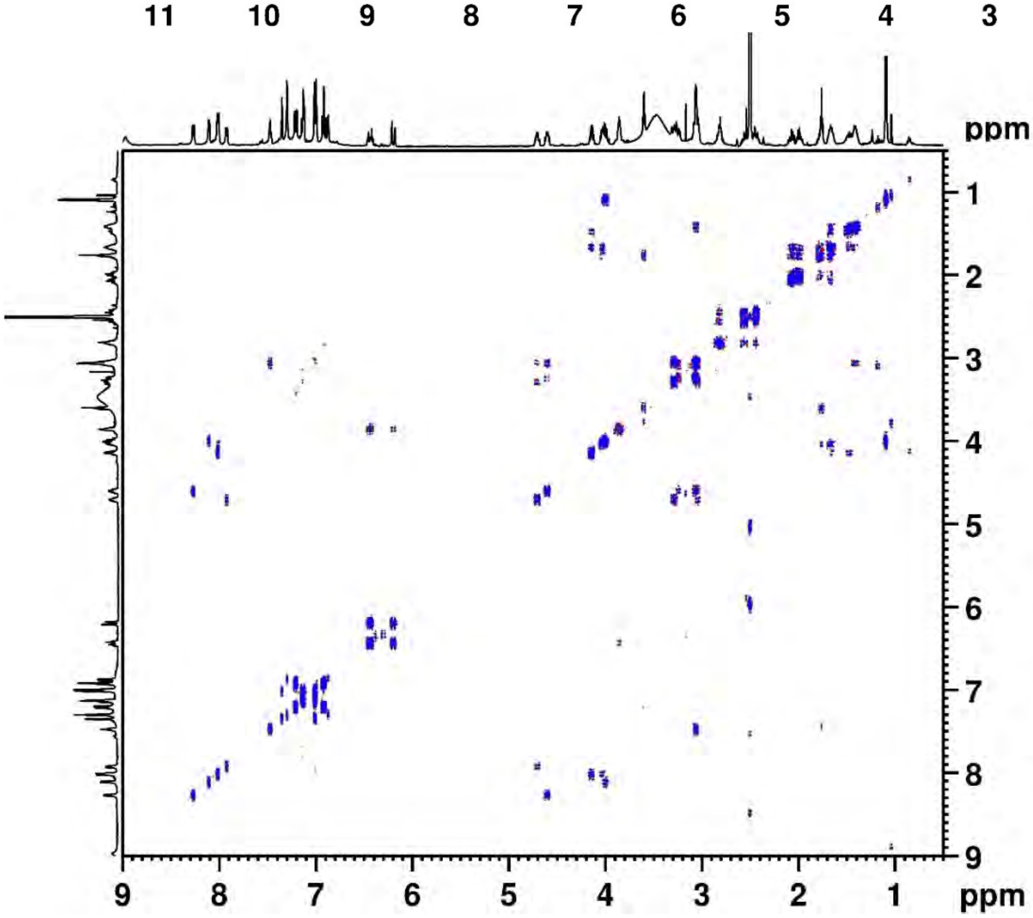

```
Current Data Parameters
NAME              W-A4-2
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121022
Time               18.10
INSTRUM            av500
PROBHD   5 mm DCH 13C-1
PULPROG      hmbcgpl2ndqf
TD                  2048
SOLVENT             DMSO
NS                     6
DS                    16
SWH             6009.615 Hz
FIDRES          2.934382 Hz
AQ             0.1703936 sec
RG                202.91
DW                83.200 usec
DE                 10.00 usec
TE                 298.0 K
CNST6        120.0000000
CNST7        160.0000000
CNST13         7.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D6            0.07142857 sec
D16           0.00020000 sec
IN0           0.00001990 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
P2                 20.00 usec
PLW1         13.50000000 W
SFO1         500.1330008 MHz

======== CHANNEL f2 ========
NUC2                 13C
P3                  9.63 usec
PLW2         23.01399994 W
SFO2         125.7703648 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1               50.00 %
GPZ2               30.00 %
GPZ3               40.10 %
GPZ4               15.00 %
```

*Macrocyclic Product **11c***

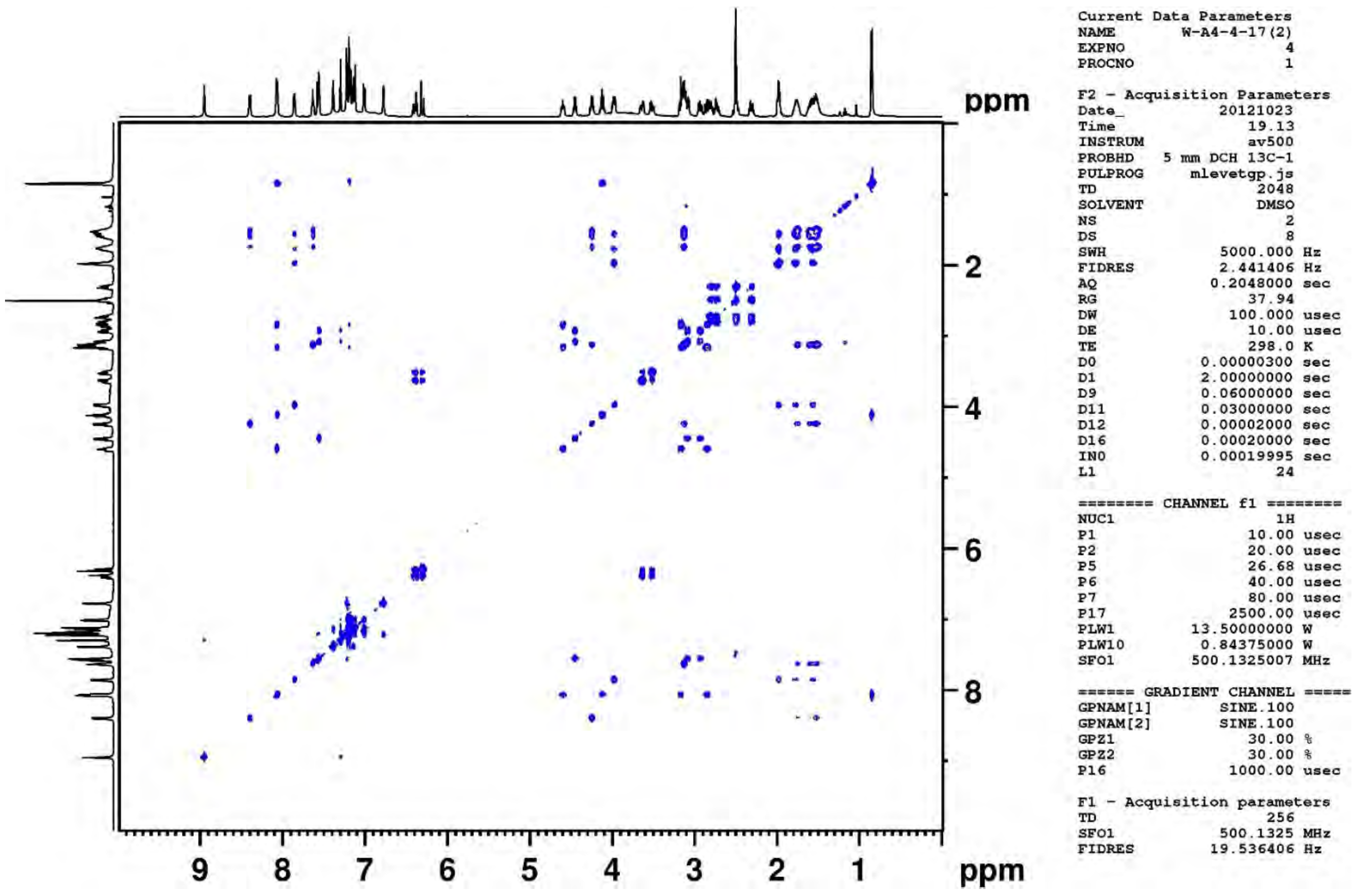
```
Current Data Parameters
NAME      W-A4-3-1(09-2013)
EXPNO                     6
PROCNO                    1

F2 - Acquisition Parameters
Date_              20130929
Time                   8.38
INSTRUM               av600
PROBHD   5 mm TBI5
PULPROG                zgpr
TD                    65536
SOLVENT                DMSO
NS                      128
DS                        0
SWH            12376.237 Hz
FIDRES          0.188846 Hz
AQ            2.6476543 sec
RG                      181
DW               40.400 usec
DE                 6.50 usec
TE                  298.0 K
D1           2.00000000 sec
D12          0.00002000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                10.20 usec
PL1               -2.00 dB
PL9               51.15 dB
PL1W         39.81071854 W
PL9W          0.00019275 W
SFO1        600.1319858 MHz

F2 - Processing parameters
SI                    65536
SF          600.1300058 MHz
WDW                      EM
SSB      0
LB                  0.30 Hz
GB       0
PC                     1.00
```

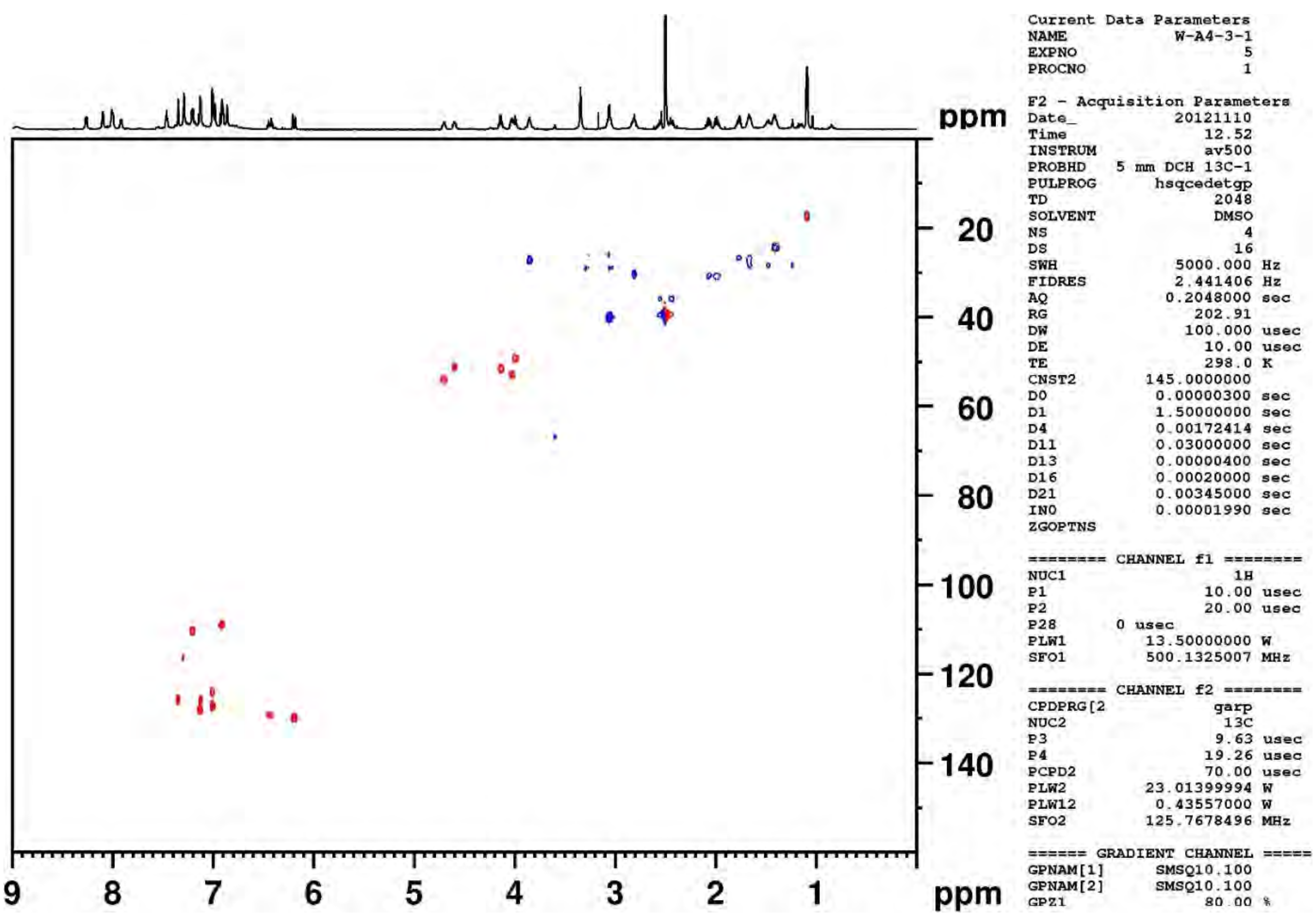


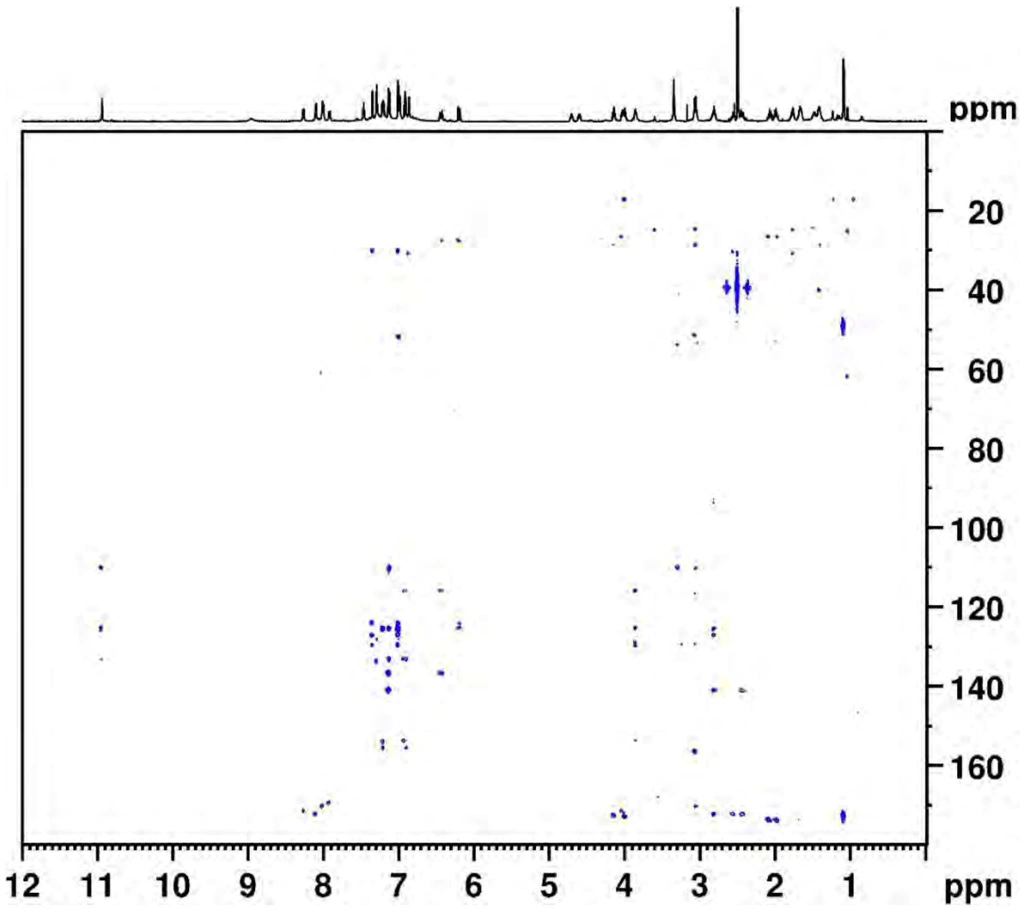





```
Current Data Parameters
NAME               W-A4-3-1
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121110
Time                  12.10
INSTRUM               av500
PROBHD   5 mm DCH 13C-1
PULPROG          cosygpmfph
TD                     4096
SOLVENT                DMSO
NS                        2
DS                        8
SWH             5498.534 Hz
FIDRES          1.342415 Hz
AQ            0.3724629 sec
RG                   202.91
DW               90.933 usec
DE                10.00 usec
TE                  298.0 K
D0           0.00007815 sec
D1           2.00000000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00018175 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                10.00 usec
P2                20.00 usec
PLW1         13.50000000 W
SFO1        500.1327507 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SMSQ10.100
GPNAM[2]        SMSQ10.100
GPZ1               10.00 %
GPZ2               20.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                      256
SFO1           500.1328 MHz
FIDRES        21.490080 Hz
SW               11.000 ppm
FnMODE         States-TPPI

F2 - Processing parameters
SI                     4096
SF          500.1300056 MHz
WDW                    SINE
SSB                       1
```


Current Data Parameters
NAME W-A4-4-17(2)
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121023
Time 19.13
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG mlevetgp.js
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 37.94
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00019995 sec
L1 24
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PLW1 13.50000000 W
PLW10 0.84375000 W
SFO1 500.1325007 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.536406 Hz
ppm


Current Data Parameters
NAME W-A4-3-1
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121110
Time 12.52
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcedetgp
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 202.91
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
IN0 0.00001990 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P28 0 usec
PLW1 13.50000000 W
SFO1 500.1325007 MHz
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 9.63 usec
P4 19.26 usec
PCPD2 70.00 usec
PLW2 23.01399994 W
PLW12 0.43557000 W
SFO2 125.7678496 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %
ppm



Current Data Parameters
NAME W-A4-3-1
EXPNO 6
PROCNO 1

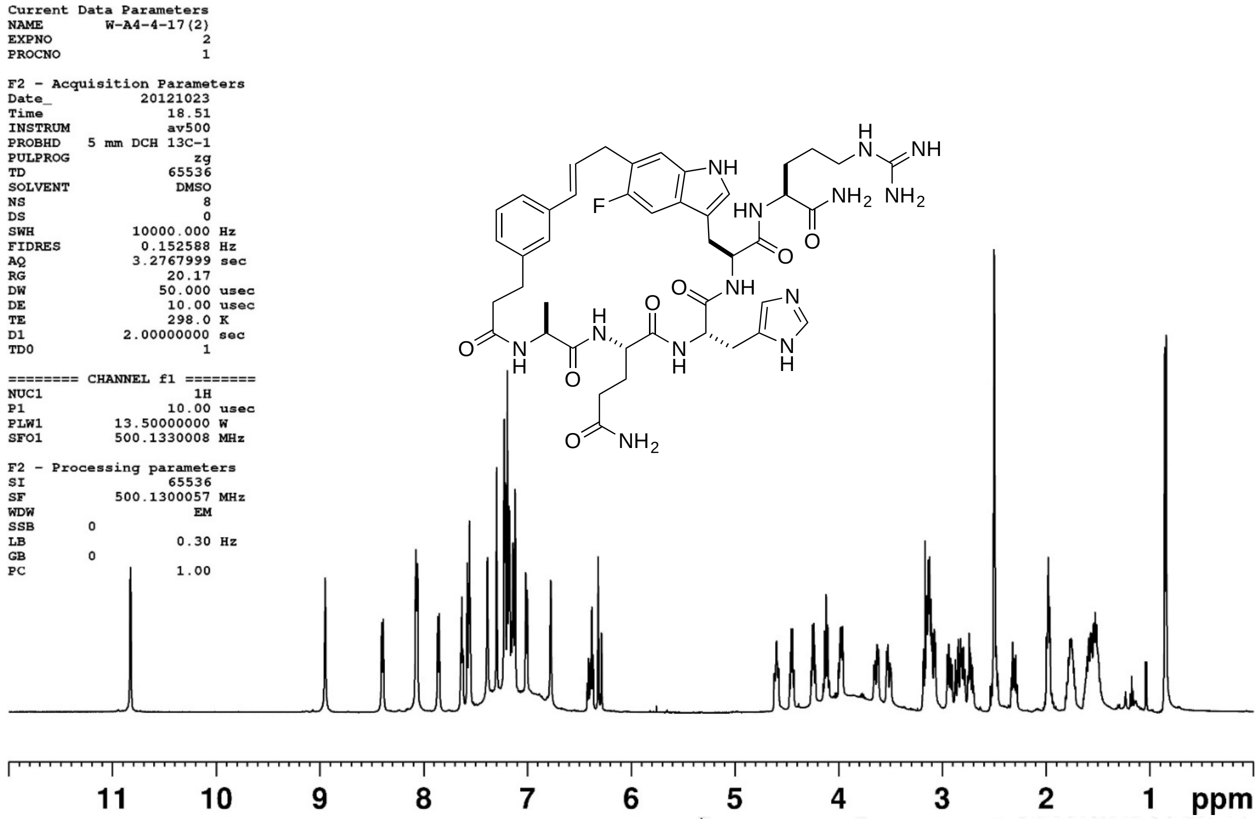
F2 - Acquisition Parameters
Date_ 20121110
Time 13.22
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 202.91
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec

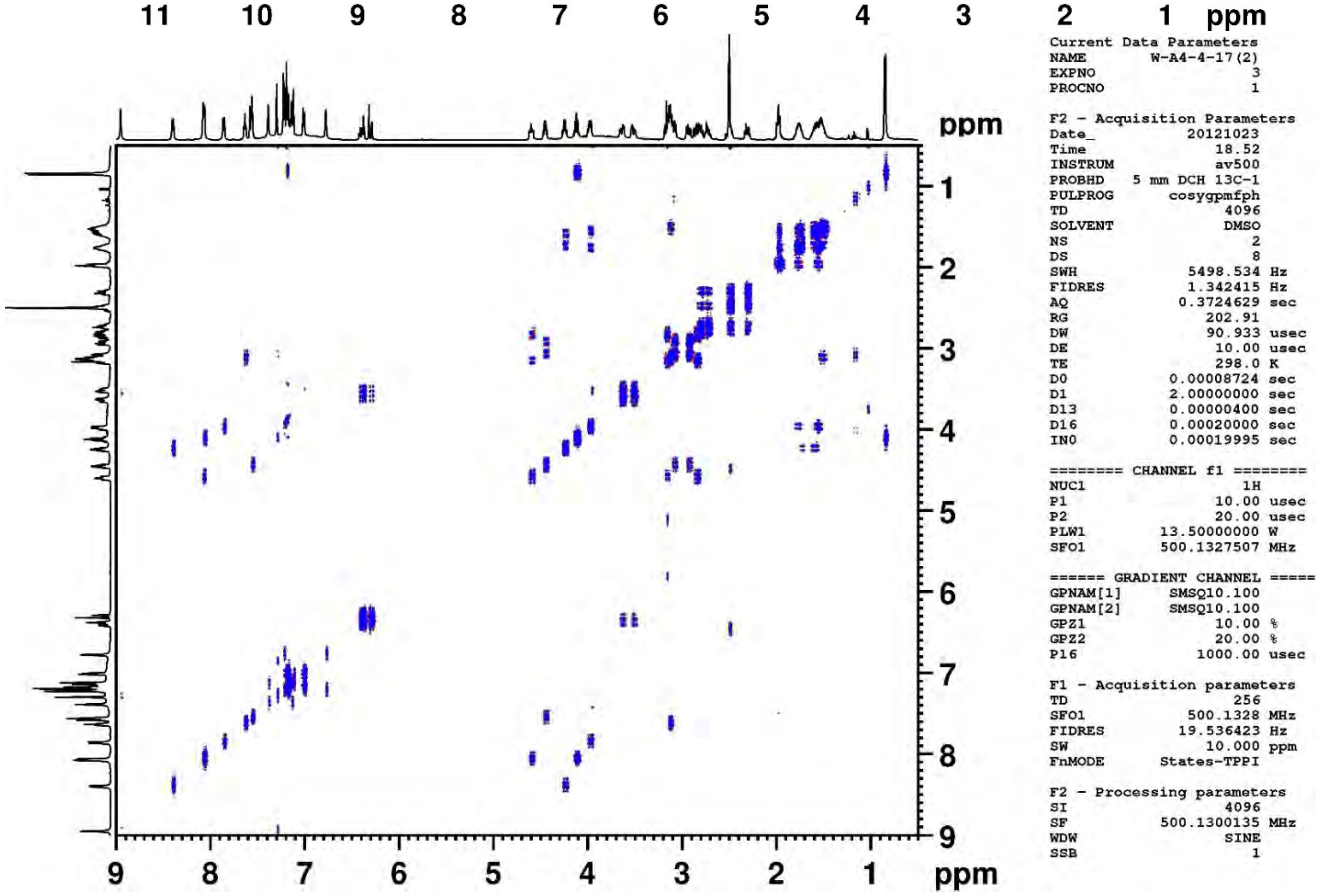
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W
SFO1 500.1330008 MHz

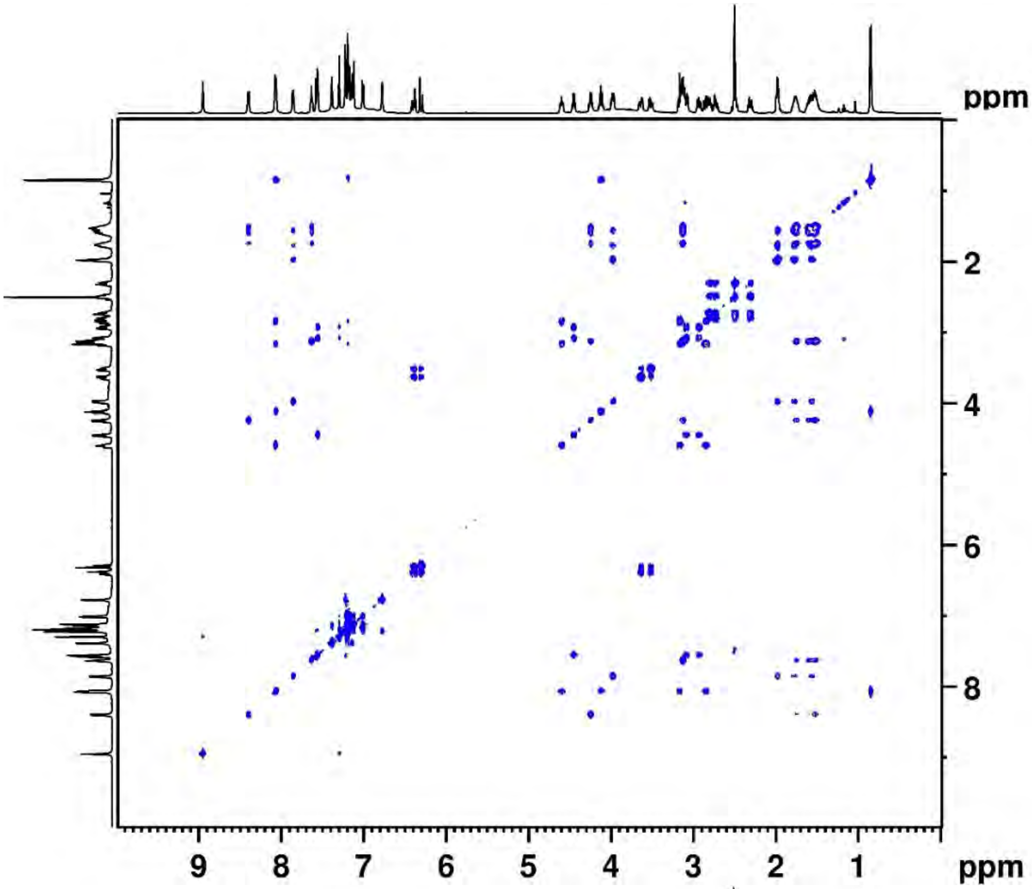
======== CHANNEL f2 ========
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
SFO2 125.7703648 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

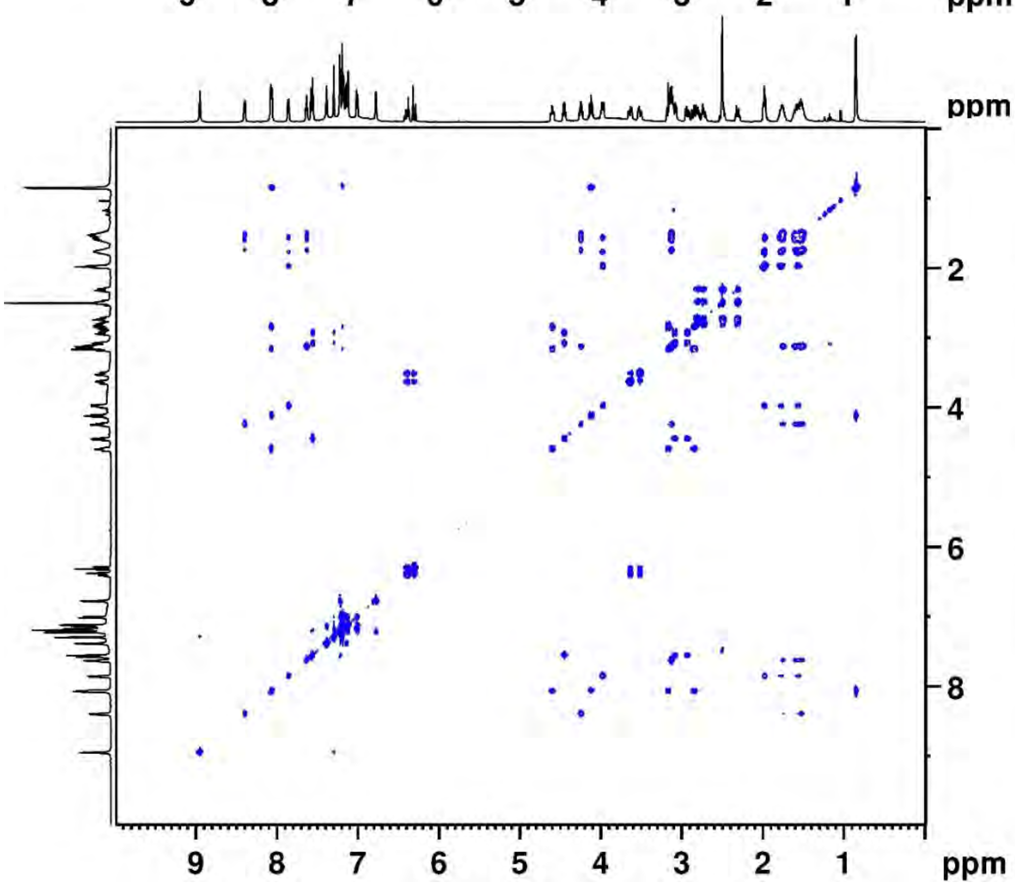
Macrocyclic Product ***11d***



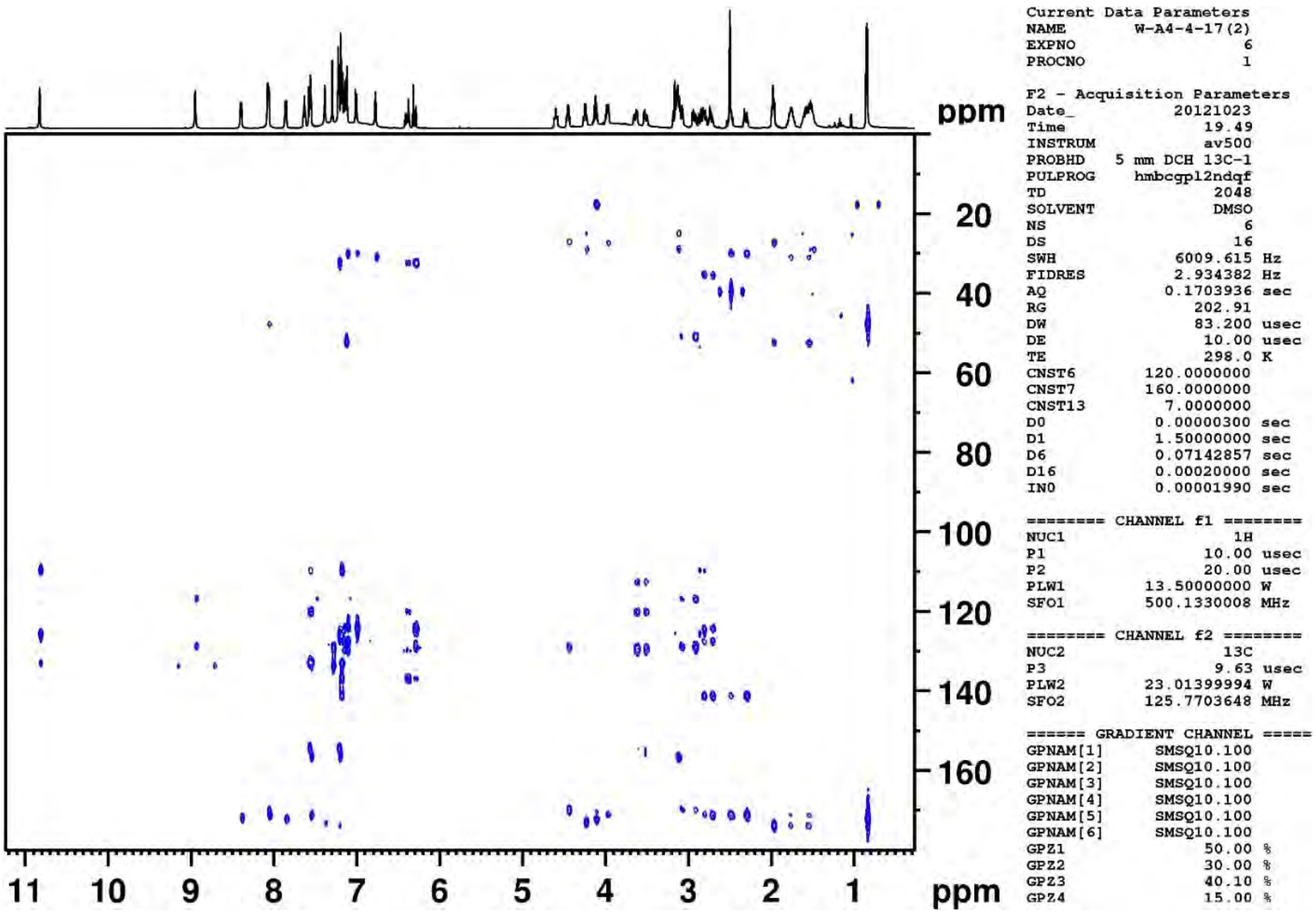





```
Current Data Parameters
NAME          W-A4-4-17(2)
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20121023
Time                 19.13
INSTRUM              av500
PROBHD   5 mm DCH 13C-1
PULPROG       mlevetgp.js
TD                    2048
SOLVENT               DMSO
NS                       2
DS                       8
SWH            5000.000 Hz
FIDRES         2.441406 Hz
AQ            0.2048000 sec
RG                   37.94
DW              100.000 usec
DE                10.00 usec
TE                298.0 K
D0           0.00000300 sec
D1           2.00000000 sec
D9           0.06000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
D16          0.00020000 sec
IN0          0.00019995 sec
L1                      24

======== CHANNEL f1 ========
NUC1                    1H
P1                10.00 usec
P2                20.00 usec
P5                26.68 usec
P6                40.00 usec
P7                80.00 usec
P17             2500.00 usec
PLW1         13.50000000 W
PLW10         0.84375000 W
SFO1       500.1325007 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPZ1                30.00 %
GPZ2                30.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                     256
SFO1          500.1325 MHz
FIDRES        19.536406 Hz
```




```
Current Data Parameters
NAME          W-A4-4-17(2)
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20121023
Time                 19.13
INSTRUM              av500
PROBHD   5 mm DCH 13C-1
PULPROG       mlevetgp.js
TD                    2048
SOLVENT               DMSO
NS                       2
DS                       8
SWH            5000.000 Hz
FIDRES         2.441406 Hz
AQ            0.2048000 sec
RG                   37.94
DW              100.000 usec
DE                10.00 usec
TE                298.0 K
D0           0.00000300 sec
D1           2.00000000 sec
D9           0.06000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
D16          0.00020000 sec
IN0          0.00019995 sec
L1                      24

======== CHANNEL f1 ========
NUC1                    1H
P1                10.00 usec
P2                20.00 usec
P5                26.68 usec
P6                40.00 usec
P7                80.00 usec
P17             2500.00 usec
PLW1         13.50000000 W
PLW10         0.84375000 W
SFO1       500.1325007 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPZ1                30.00 %
GPZ2                30.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                     256
SFO1          500.1325 MHz
FIDRES        19.536406 Hz
```


ppm
20
40
60
80
100
120
140
160
11 10 9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME W-A4-4-17(2)
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121023
Time 19.49
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 6
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 202.91
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W
SFO1 500.1330008 MHz
======== CHANNEL f2 ========
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
SFO2 125.7703648 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

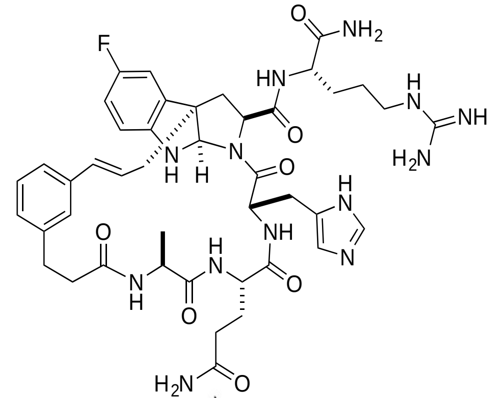
Macrocyclic Product ***11e***

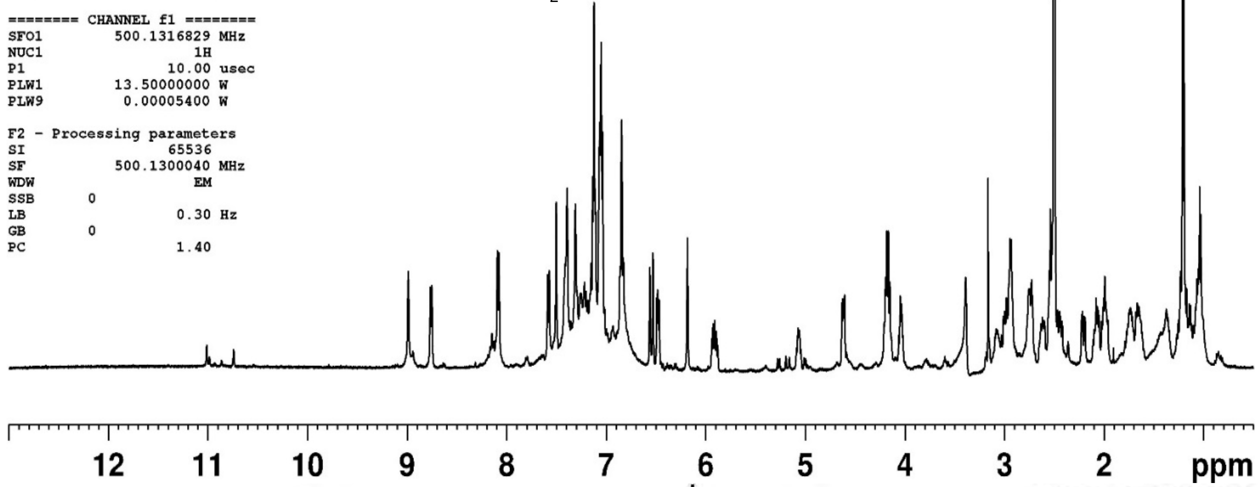
Current Data Parameters
NAME W-A4-1-1_AV500
EXPNO 8
PROCNO 1

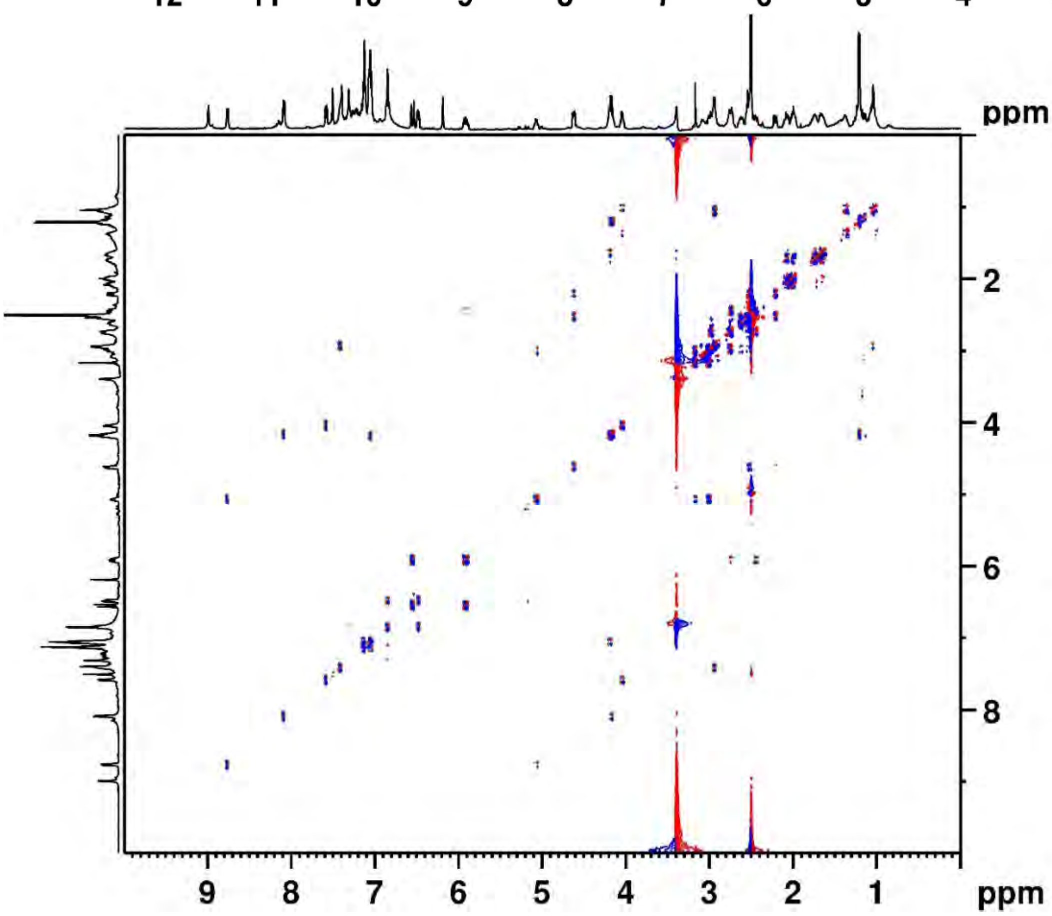
F2 - Acquisition Parameters
Date_ 20140222
Time 20.16
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpr
TD 65536
SOLVENT DMSO
NS 15
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1316829 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
PLW9 0.00005400 W

F2 - Processing parameters
SI 65536
SF 500.1300040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40







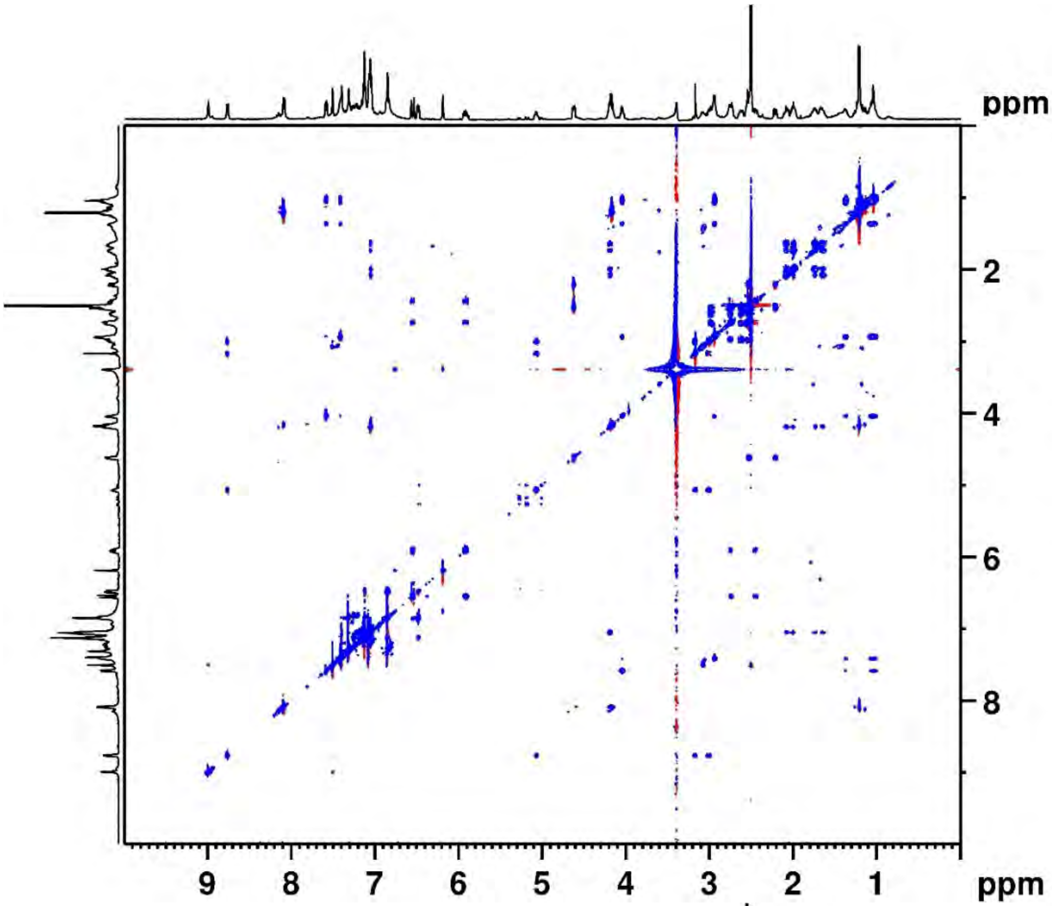
Current Data Parameters
NAME W-A4-1-1
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121217
Time 16.36
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG cosygpmfph
TD 4096
SOLVENT DMSO
NS 8
DS 4
SWH 6009.615 Hz
FIDRES 1.467191 Hz
AQ 0.3407872 sec
RG 71.8
DW 83.200 usec
DE 6.50 usec
TE 294.0 K
D0 0.00007058 sec
D1 2.00000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00016665 sec

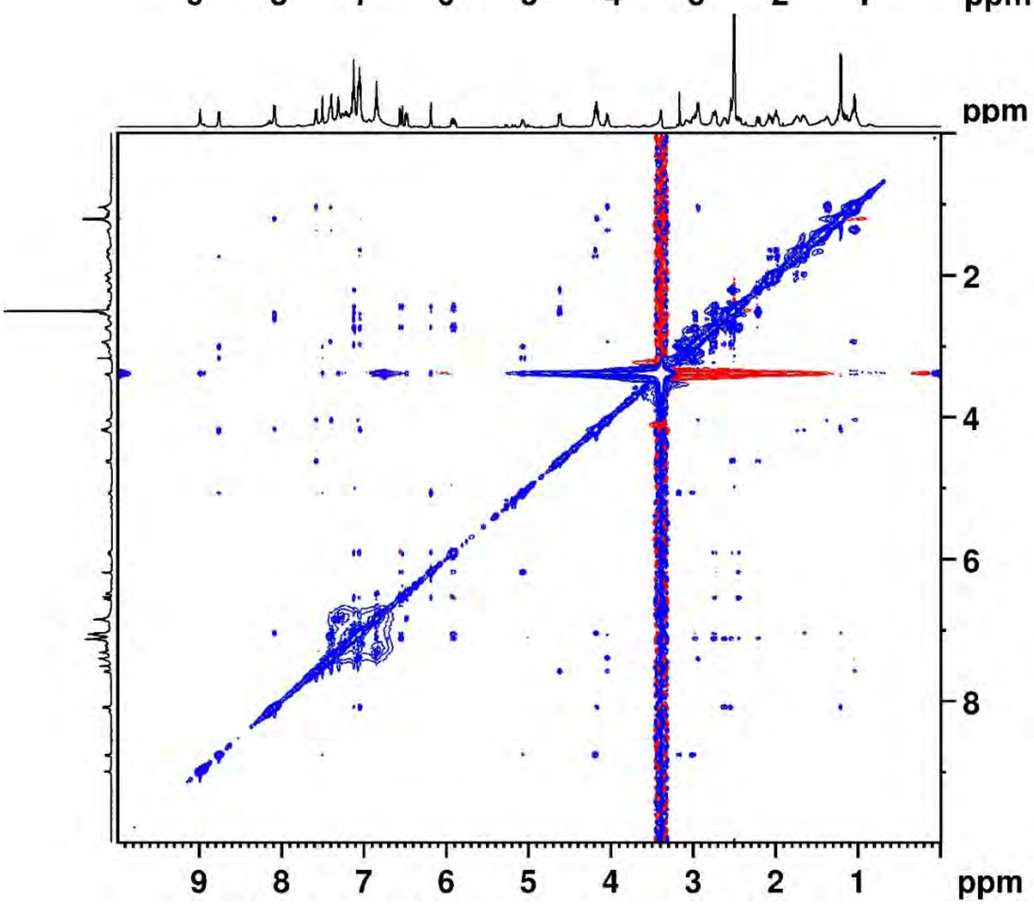
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1330006 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 10.00 %
GPZ2 20.00 %
P16 1000.00 usec

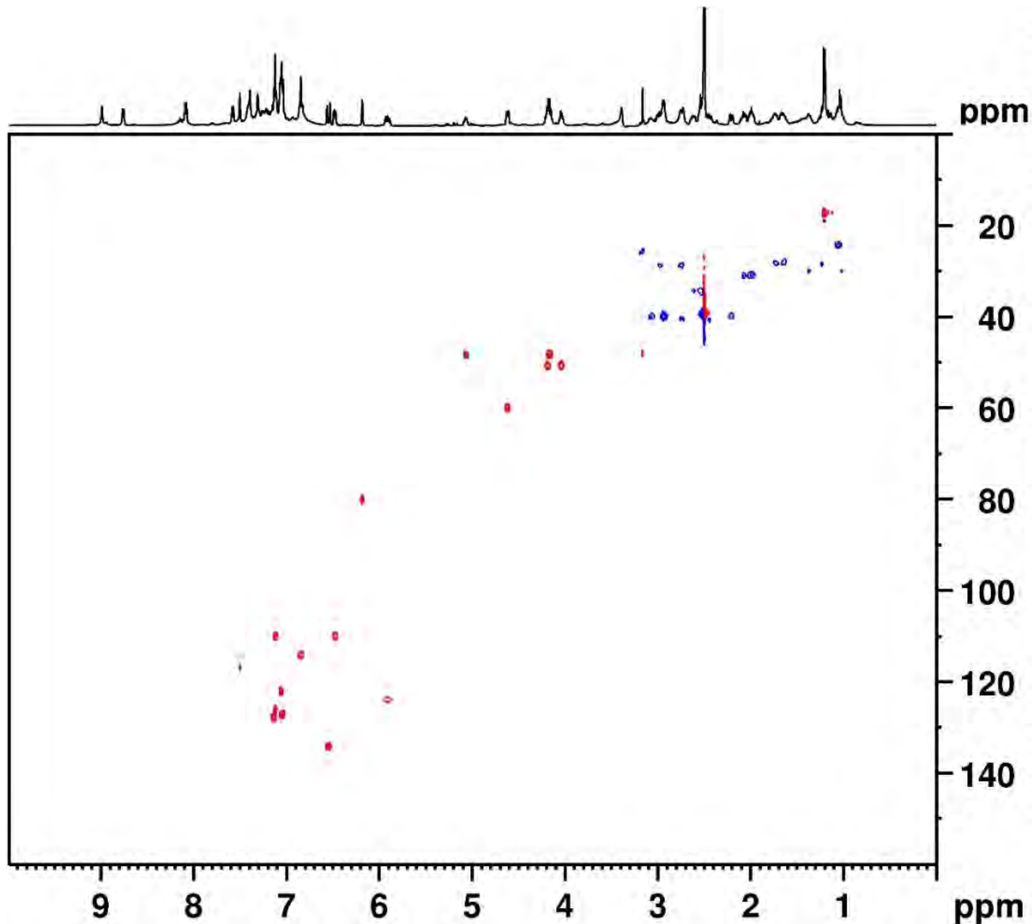
F1 - Acquisition parameters
TD 256
SFO1 600.133 MHz
FIDRES 23.442696 Hz
SW 10.000 ppm
FnMODE States-TPPI




```
Current Data Parameters
NAME              W-A4-1-1
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20121217
Time                 17.57
INSTRUM              av600
PROBHD     5 mm TBI5
PULPROG        mlevetgp.js
TD                    4096
SOLVENT               DMSO
NS                       4
DS                      16
SWH               6009.615 Hz
FIDRES            1.467191 Hz
AQ               0.3407872 sec
RG                    71.8
DW                  83.200 usec
DE                    6.50 usec
TE                   294.0 K
D0             0.00000300 sec
D1             1.20000005 sec
D9             0.06000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00016665 sec
L1                      24

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.00 usec
P2                   20.00 usec
P5                   26.68 usec
P6                   40.00 usec
P7                   80.00 usec
P17                2500.00 usec
PL1                  -2.00 dB
PL10                  9.54 dB
PL1W           39.81071854 W
PL10W           2.79254389 W
SFO1        600.1330006 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPNAM[2]         SINE.100
GPX1        0 %
GPX2        0 %
GPY1        0 %
GPY2        0 %
GPZ1                 30.00 %
GPZ2                 30.00 %
```




```
Current Data Parameters
NAME         W-A4-1-1_AV500
EXPNO                     7
PROCNO                    1

F2 - Acquisition Parameters
Date_              20140223
Time                   4.31
INSTRUM               av500
PROBHD     5 mm DCH 13C-1
PULPROG           noesygpph
TD                     2048
SOLVENT                DMSO
NS                       16
DS                        8
SWH                5000.000 Hz
FIDRES             2.441406 Hz
AQ                0.2048000 sec
RG                    26.58
DW                  100.000 usec
DE                    10.00 usec
TE                    298.0 K
D0              0.00008727 sec
D1              2.00000000 sec
D8              0.20000000 sec
D16             0.00020000 sec
IN0             0.00020000 sec

======== CHANNEL f1 ========
SFO1         500.1325007 MHz
NUC1                     1H
P1                    10.00 usec
P2                    20.00 usec
PLW1            13.50000000 W

====== GRADIENT CHANNEL =====
GPNAM[1]           SINE.100
GPZ1                  40.00 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                      256
SFO1               500.1325 MHz
FIDRES            19.531250 Hz
SW                    9.997 ppm
FnMODE          States-TPPI

F2 - Processing parameters
SI                     2048
SF             500.1300036 MHz
WDW                   QSINE
SSB                       2
LB           0 Hz
GB           0
```



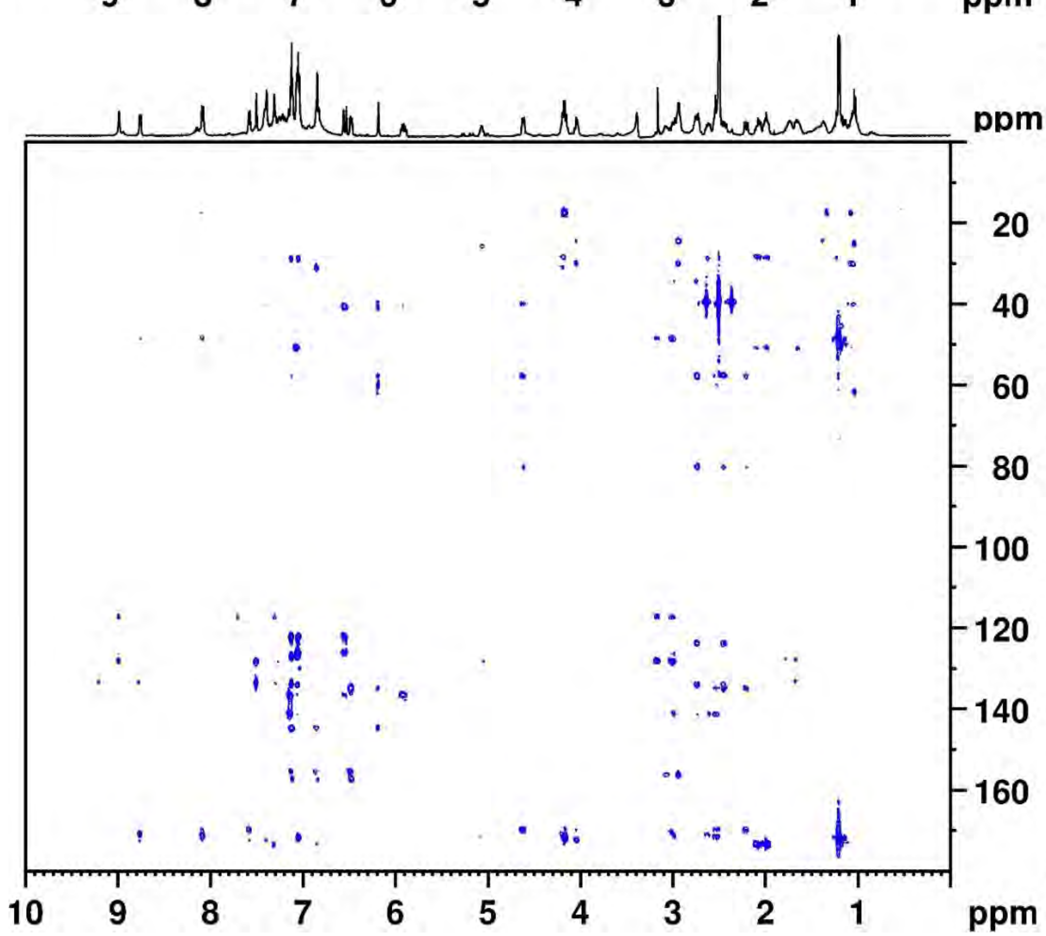
Current Data Parameters
NAME W-A4-1-1_AV500
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121219
Time 17.14
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcedetgp
TD 2048
SOLVENT DMSO
NS 6
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 202.91
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
IN0 0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P28 0 usec
PLW1 13.50000000 W

======== CHANNEL f2 ========
SFO2 125.7678496 MHz
NUC2 13C
CPDPRG[2 garp
P3 9.63 usec
P4 19.26 usec
PCPD2 70.00 usec
PLW2 23.01399994 W
PLW12 0.43557000 W

====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %



Current Data Parameters
NAME W-A4-1-1_AV500
EXPNO 6
PROCNO 1

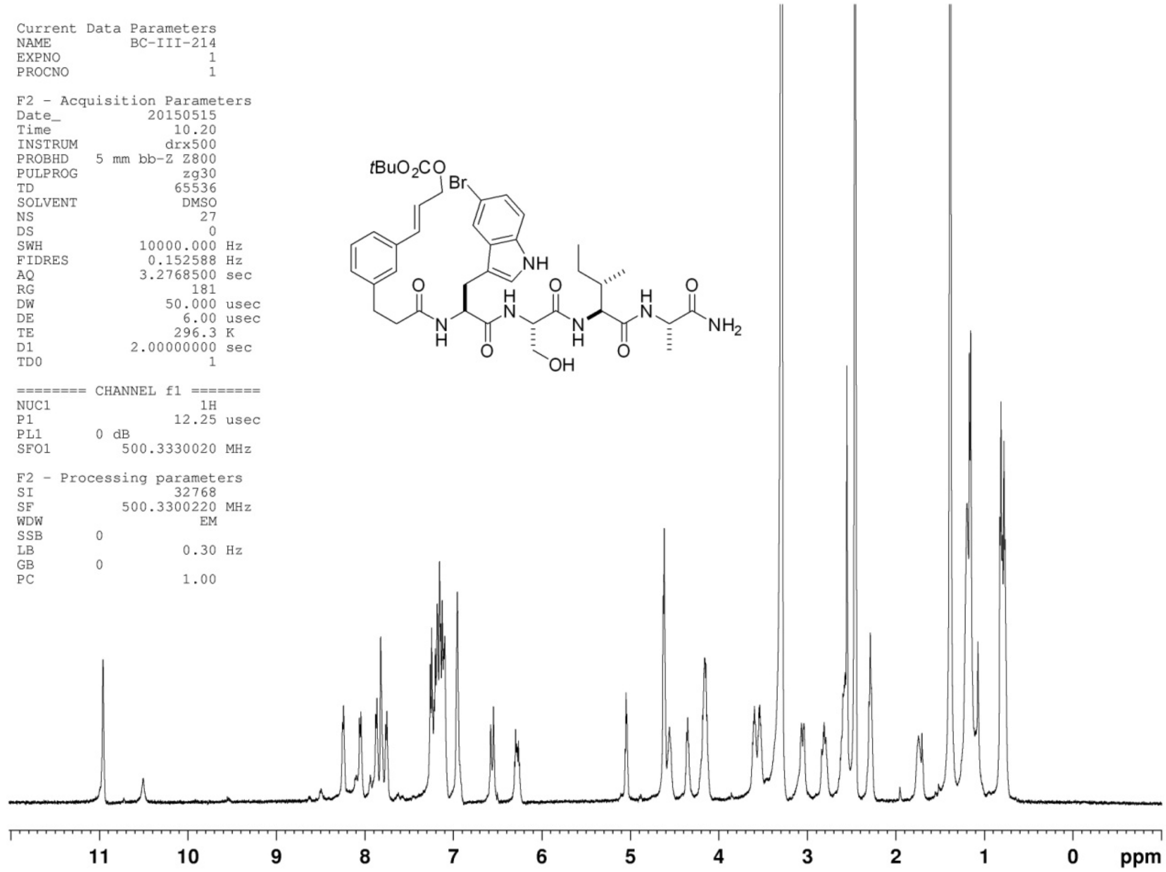
F2 - Acquisition Parameters
Date_ 20140222
Time 20.21
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 64
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 204.86
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec

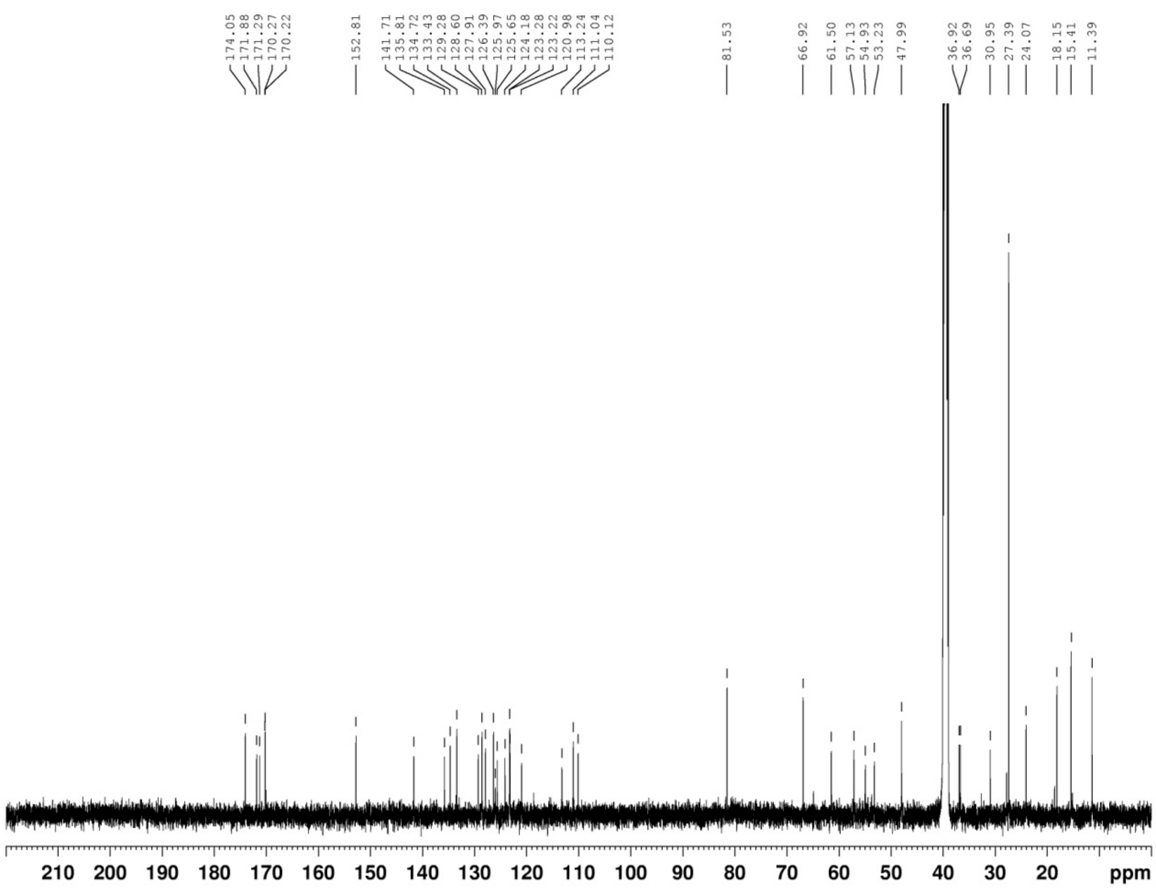
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W

======== CHANNEL f2 ========
SFO2 125.7703648 MHz
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W

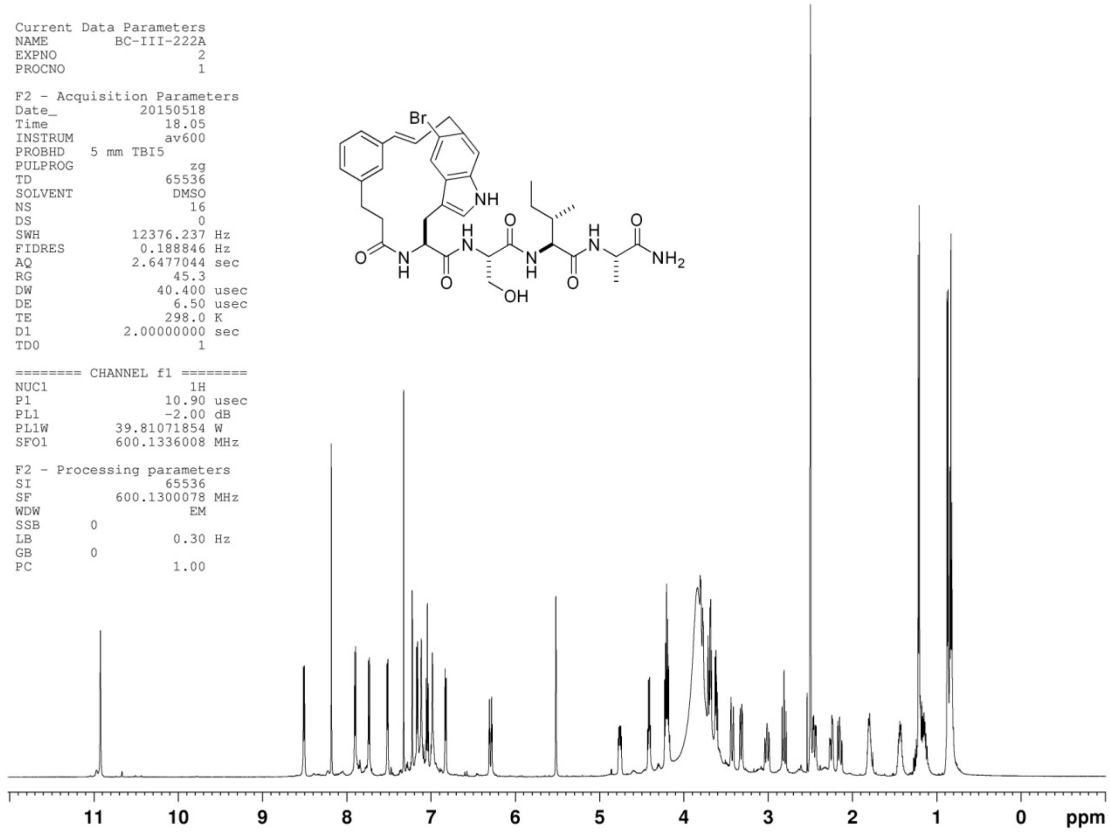
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

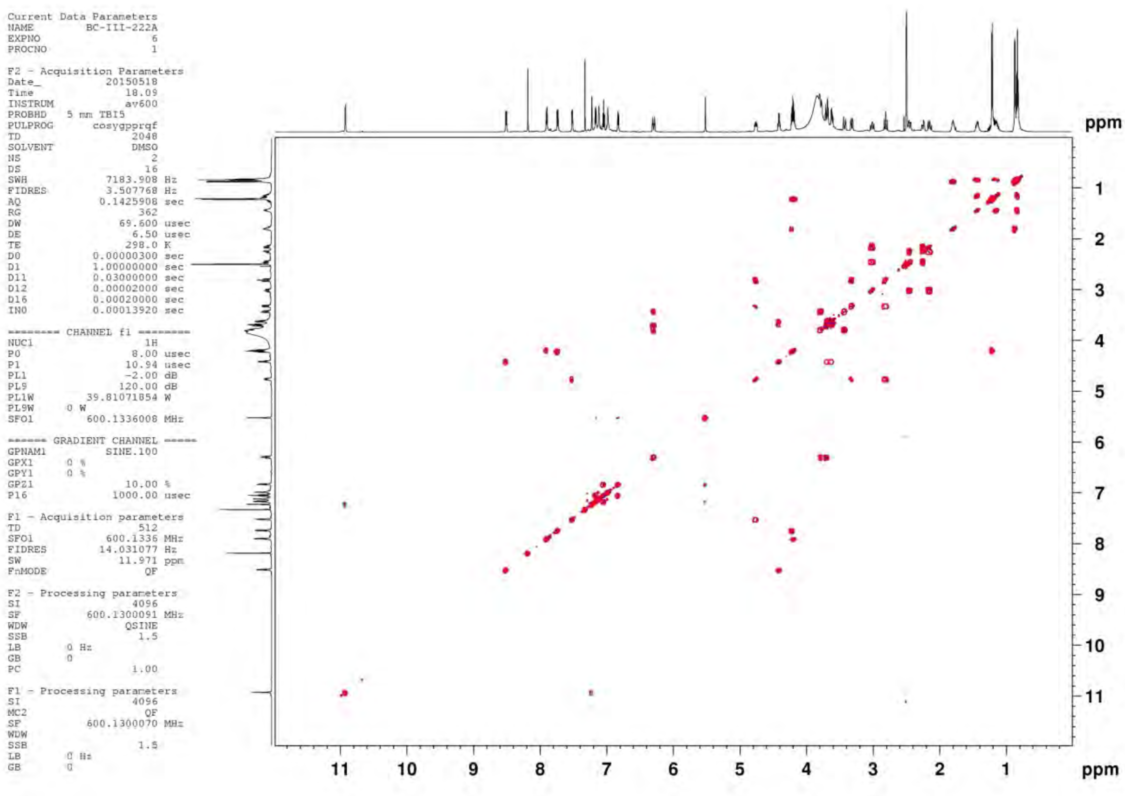
Acyclic Precursor ***12***

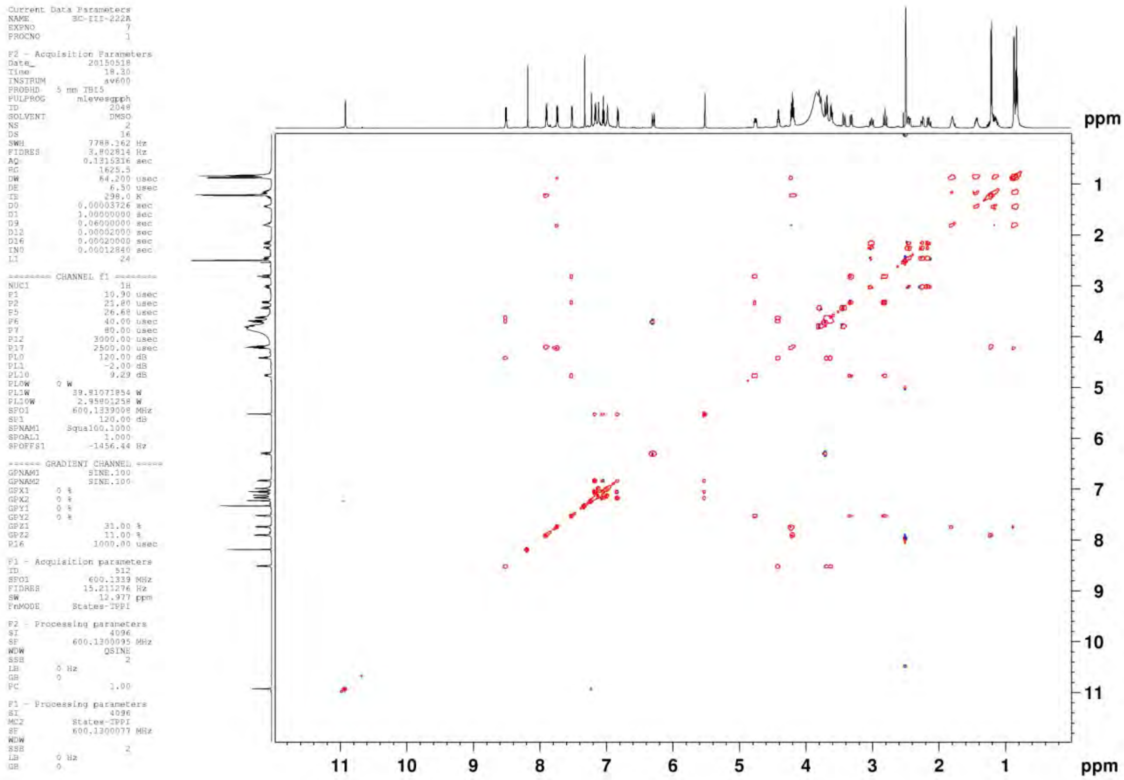


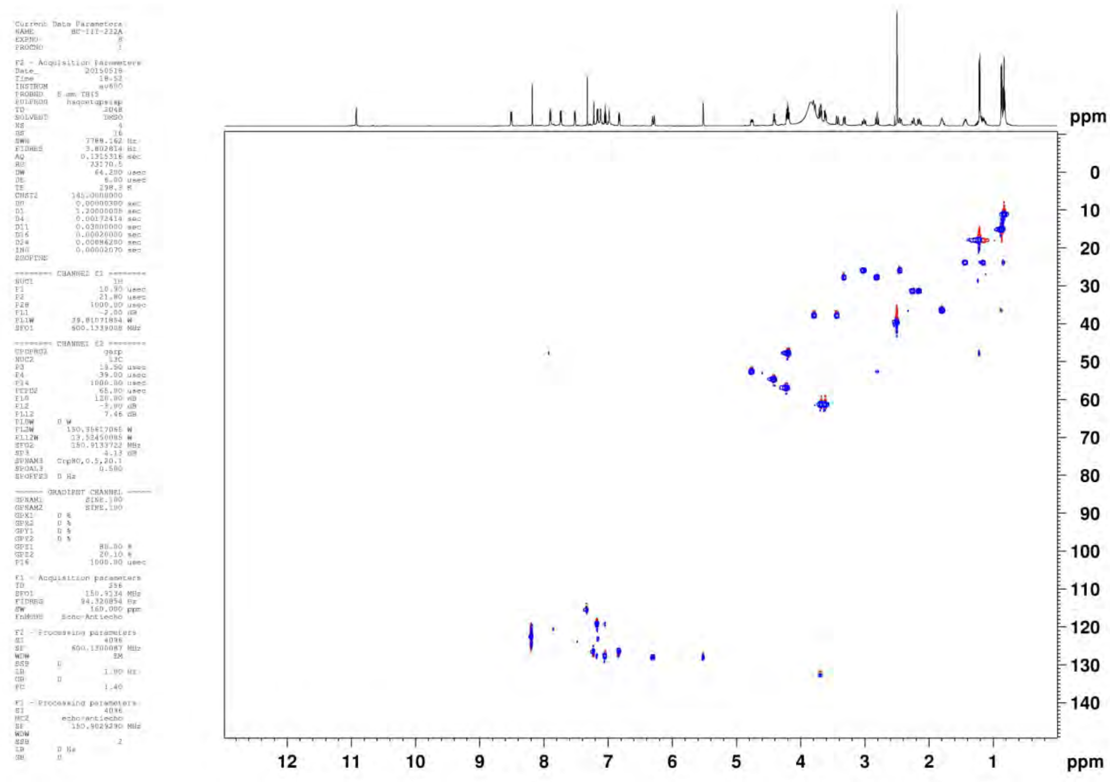


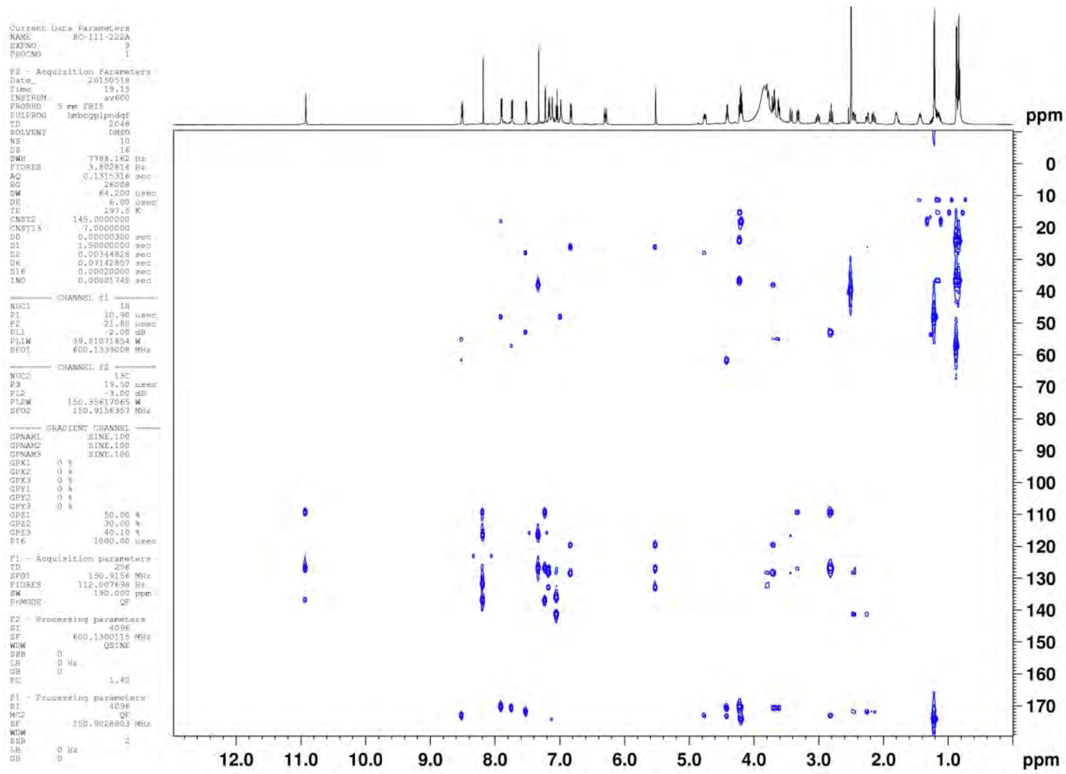
Macrocyclic Product ***16a***



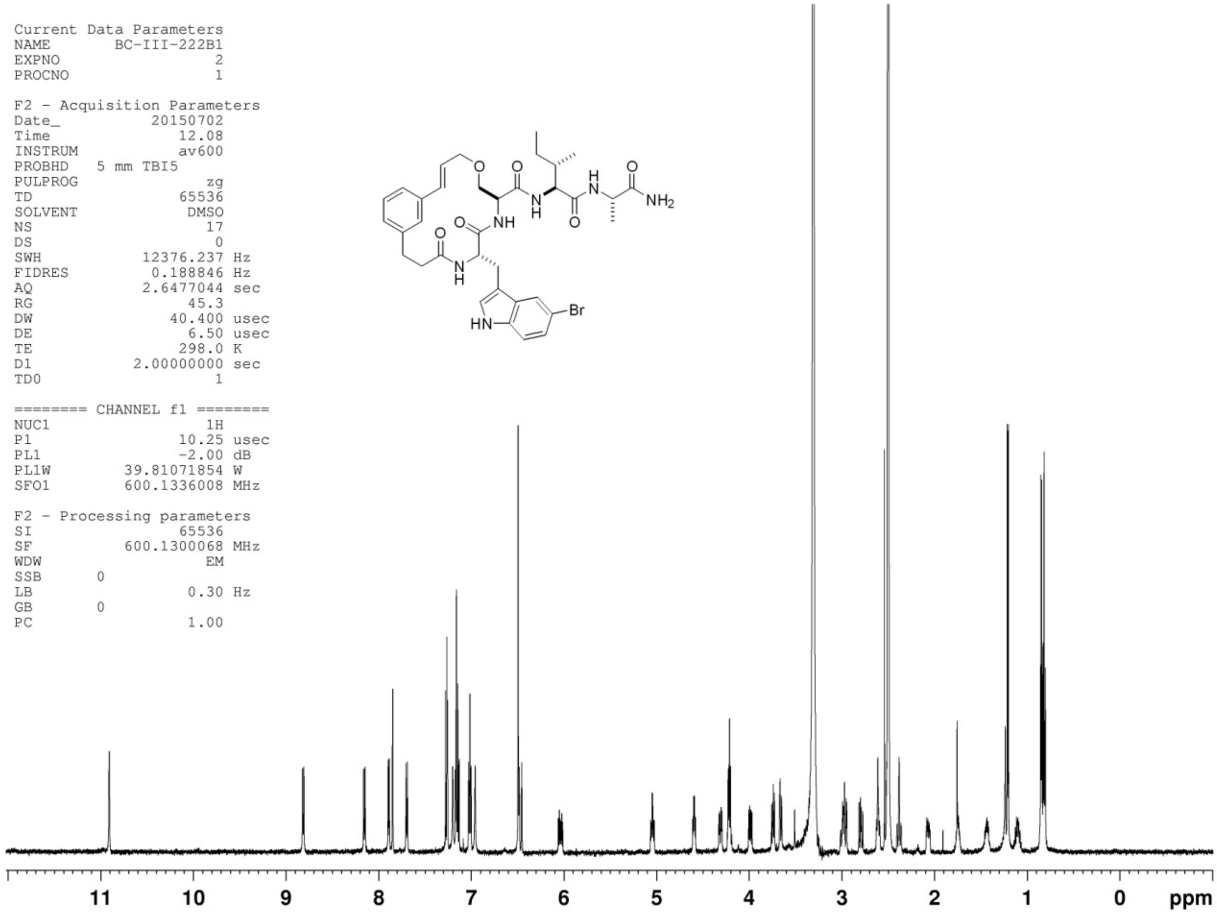


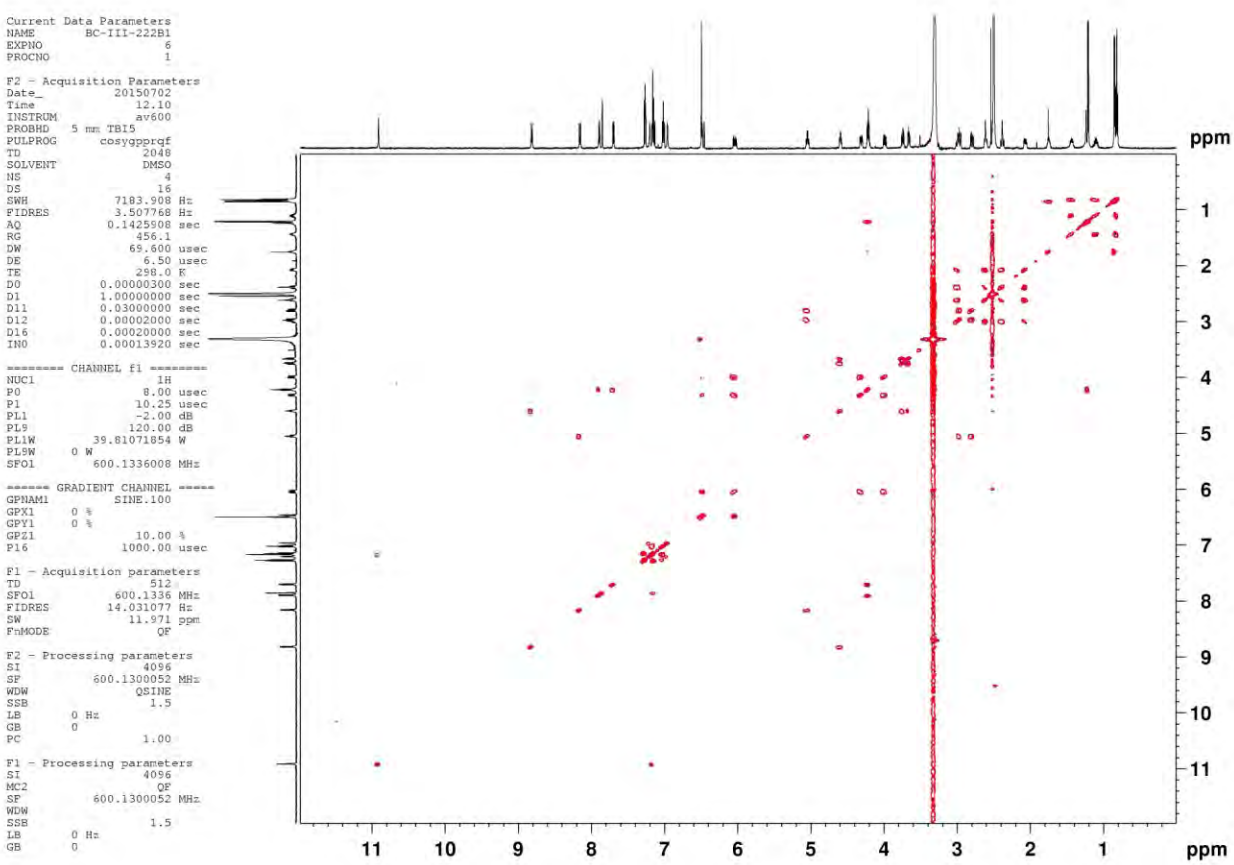

Current Data Parameters
NAME BC-III-222A
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150518
Time 18.30
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevesgpph
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 1625.5
DW 64.200 usec
DE 6.50 usec
TE 298.0 K
D0 0.00003726 sec
D1 1.00000000 sec
D9 0.06000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00012840 sec
L1 24
======== CHANNEL f1 ========
NUC1 1H
P1 10.90 usec
P2 21.80 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P12 3000.00 usec
P17 2500.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL10 9.29 dB
PL0W 0 W
PL1W 39.81071854 W
PL10W 2.95801258 W
SFO1 600.1339008 MHz
SP1 120.00 dB
SPNAM1 Squa100.1000
SPOAL1 1.000
SPOFFS1 -1456.44 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 31.00 %
GPZ2 11.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 512
SFO1 600.1339 MHz
FIDRES 15.211276 Hz
SW 12.977 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 4096
SF 600.1300095 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 4096
MC2 States-TPPI
SF 600.1300077 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
ppm

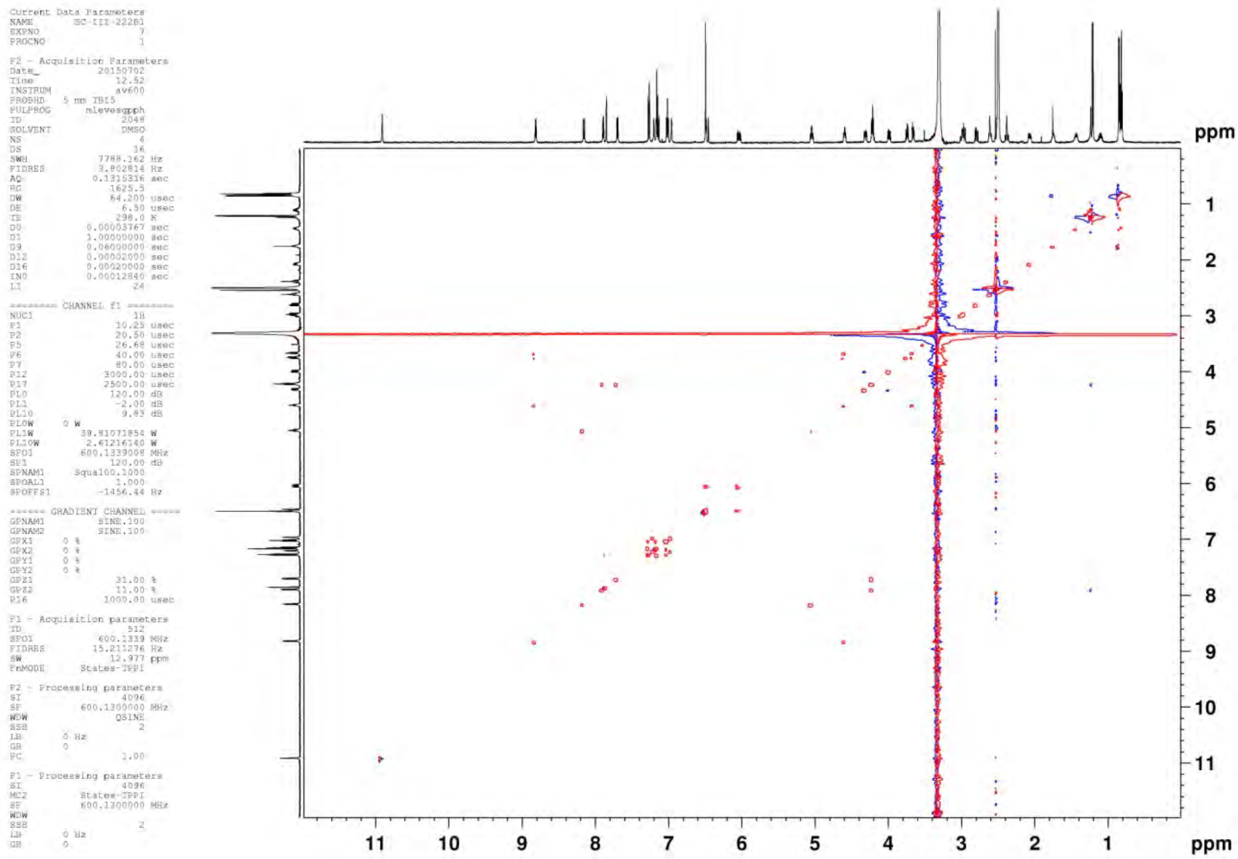

Current Data Parameters
NAME BC-III-222A
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150518
Time 18.52
INSTRUM av600
PROBHD 5 mm TBI5
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
DW 64.200 usec
DE 6.00 usec
D11 0.03000000 sec
D16 0.00020000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.90 usec
P2 21.80 usec
P28 1000.00 usec
PL1W 39.81071854 W
SFO1 600.1339008 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P4 39.00 usec
P14 1000.00 usec
PL12 7.46 dB
SFO2 150.9133722 MHz
SPNAM3 Crp80,0.5,20.1
SPOAL3 0.500
SPOFFS3 0 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ2 20.10 %
P16 1000.00 usec
F1 - Acquisition parameters
SFO1 150.9134 MHz
FIDRES 94.320854 Hz
SW 160.000 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 4096
SF 600.1300087 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 echo-antiecho
SF 150.9029290 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
ppm

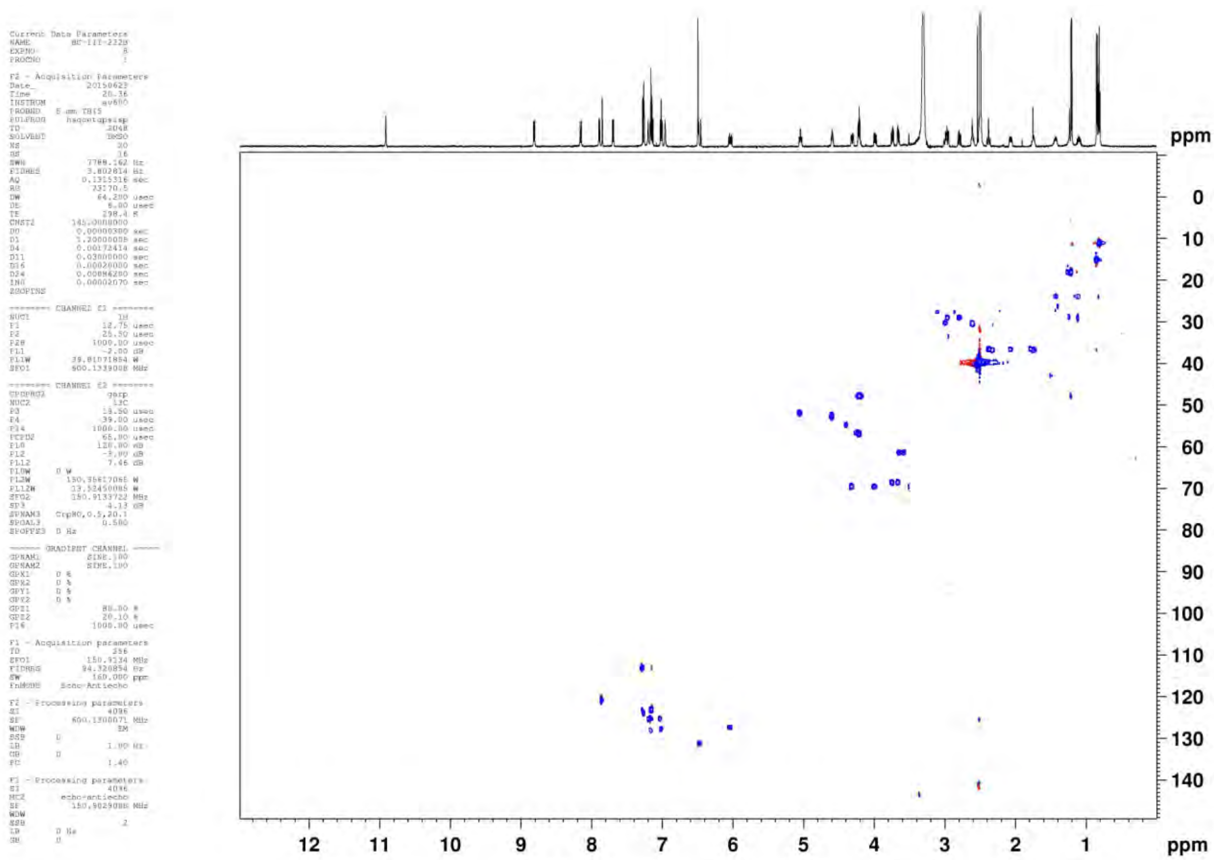

Current Data Parameters
NAME BC-III-222A
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150518
Time 19.15
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 10
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 26008
DW 64.200 usec
DE 6.00 usec
TE 297.5 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec
CHANNEL f1
NUC1 1H
P1 10.90 usec
P2 21.80 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
CHANNEL f2
NUC2 13C
P3 19.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 150.9156 MHz
FIDRES 112.007698 Hz
SW 190.000 ppm
FnMODE QF
F2 - Processing parameters
SI 4096
SF 600.1300115 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 QF
SF 150.9028803 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm

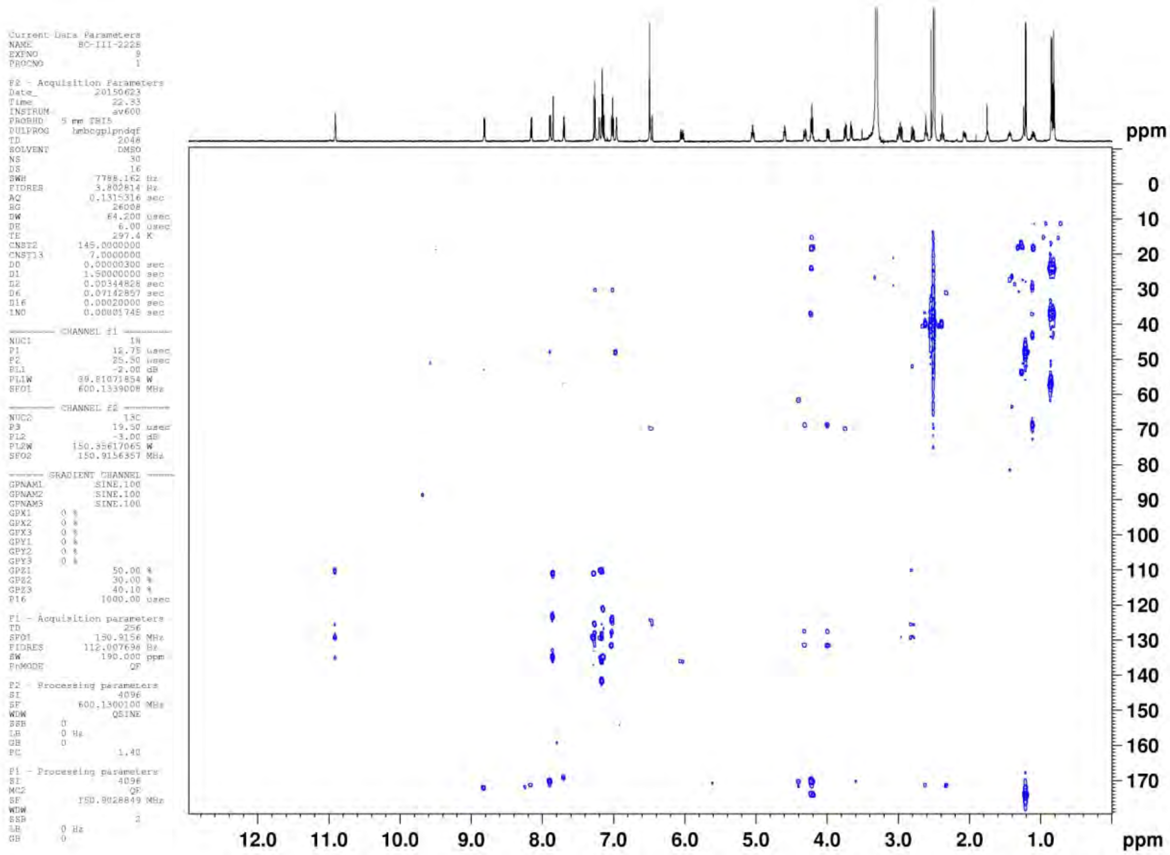
*Macrocyclic Product **16b***





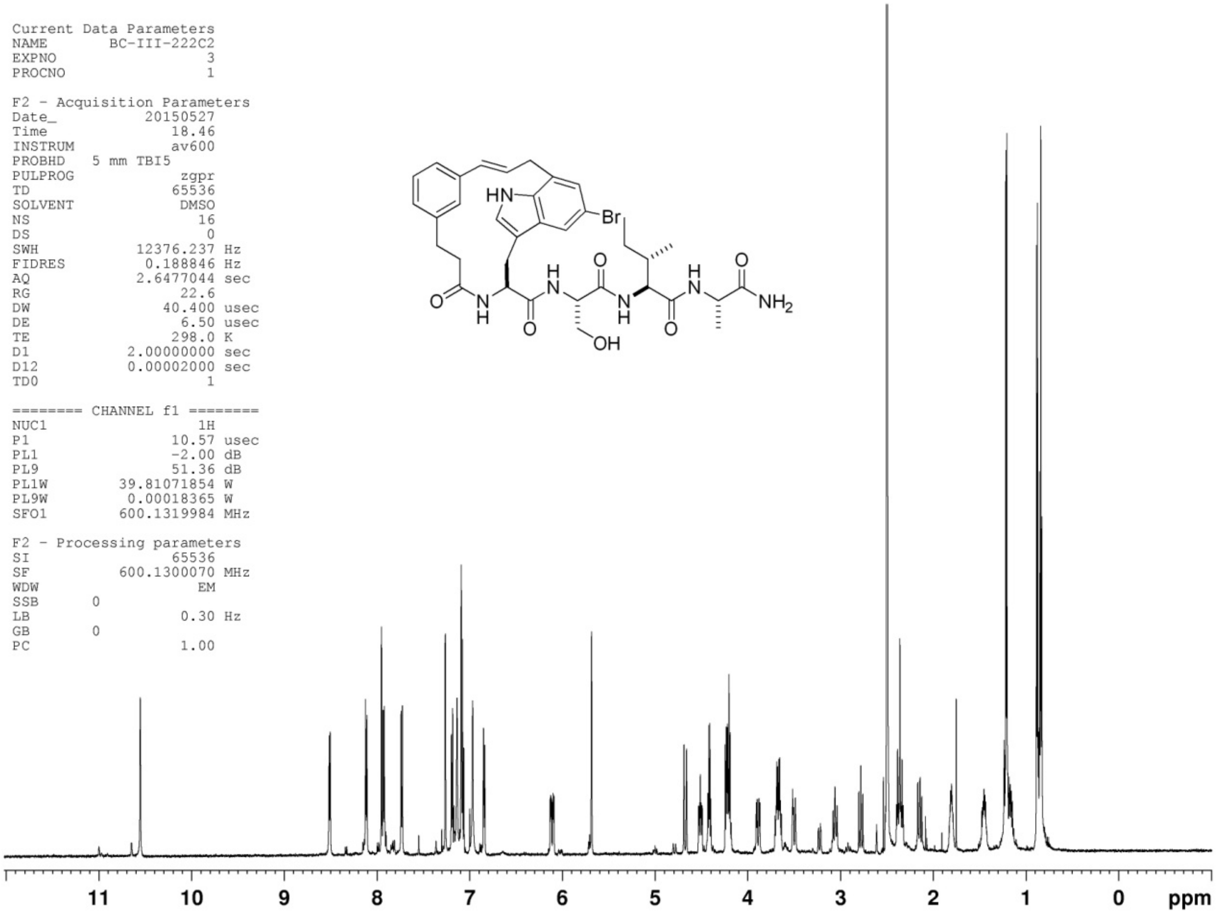

ppm
ppm

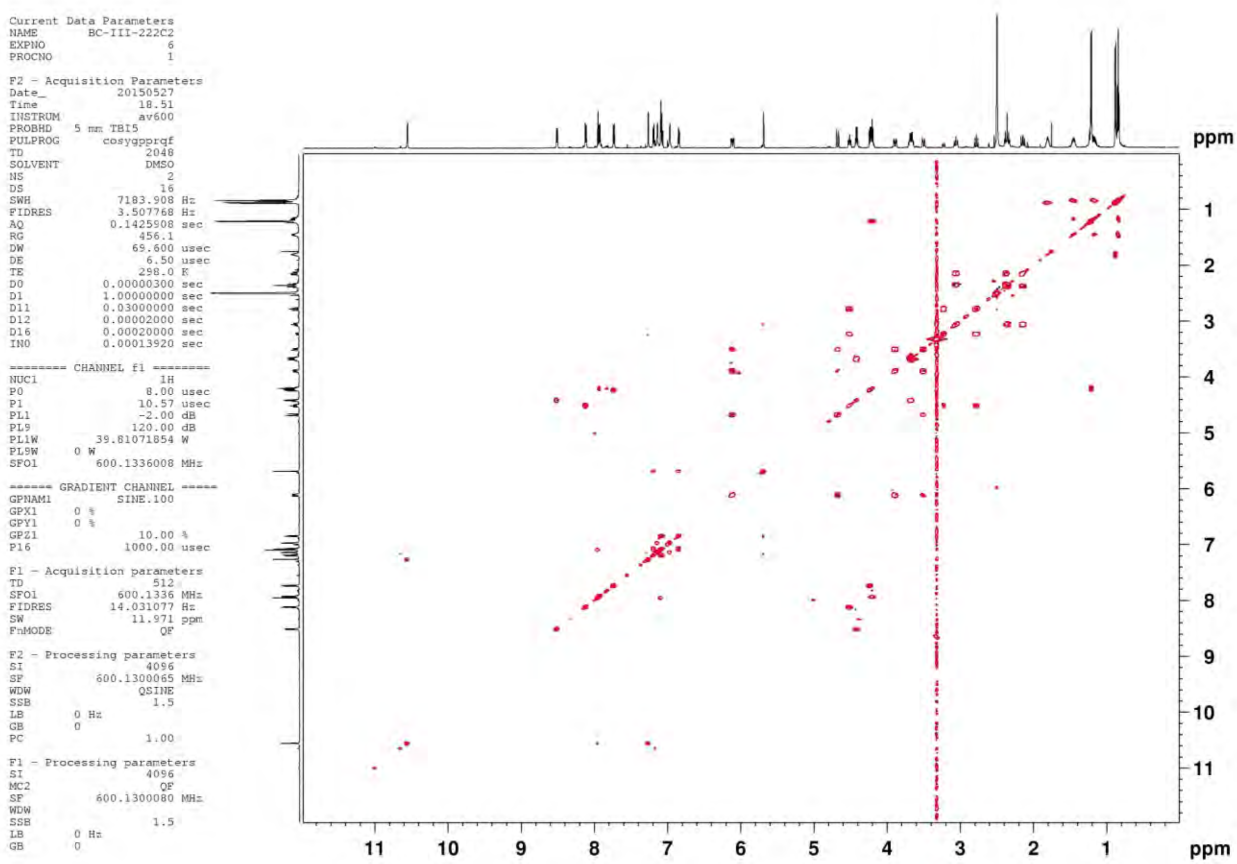

ppm
ppm

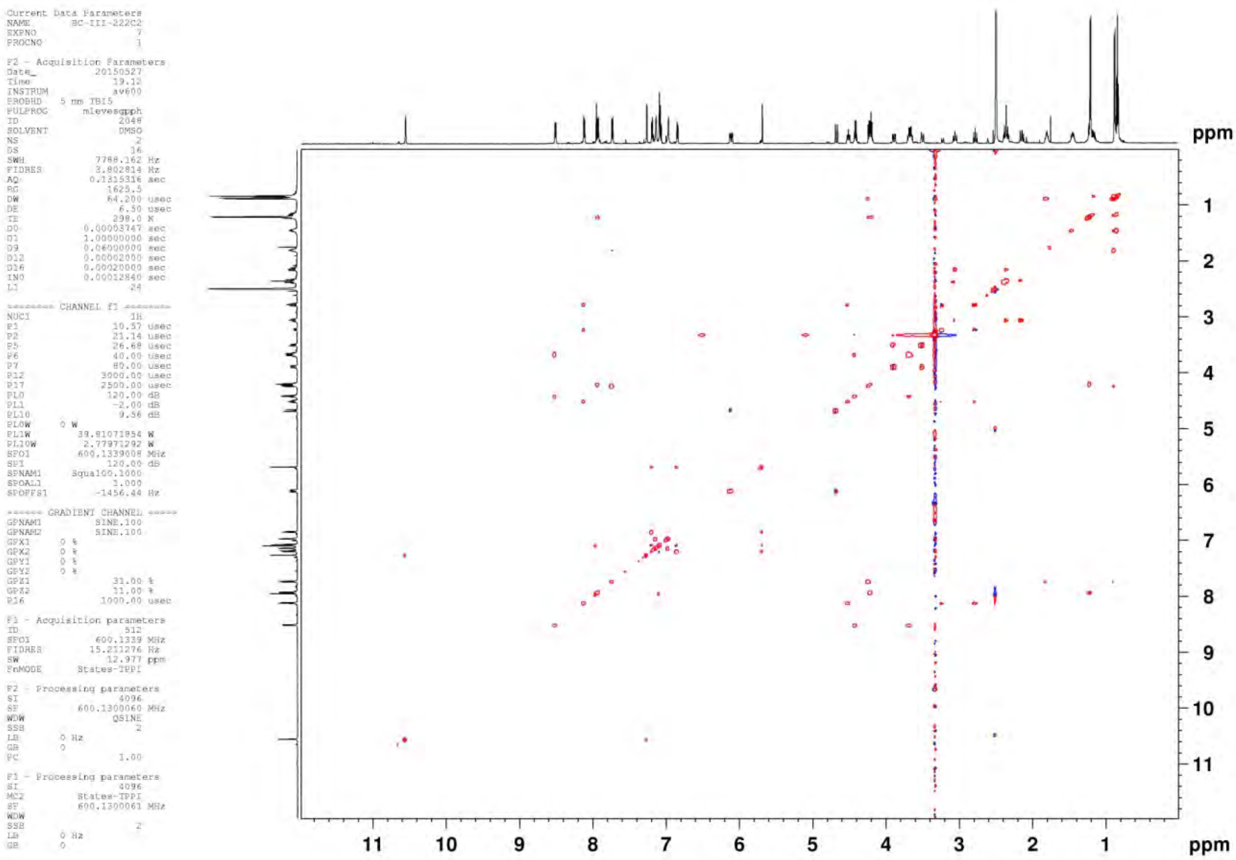


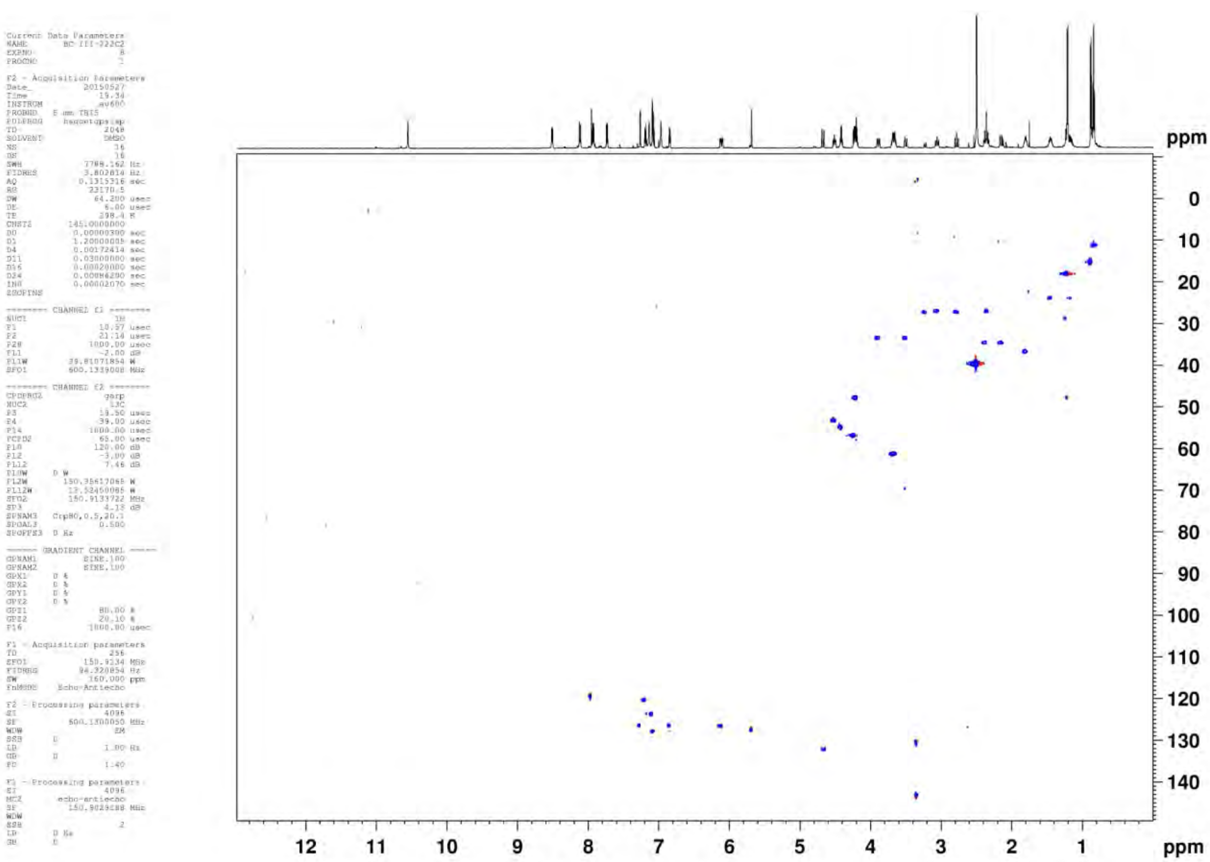
Current Data Parameters
NAME BC-III-222B
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150623
Time 22.33
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 30
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 26008
DW 64.200 usec
DE 6.00 usec
TE 297.4 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec
CHANNEL f1
NUC1 1H
P1 12.75 usec
P2 25.50 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
CHANNEL f2
NUC2 13C
P3 19.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 150.9156 MHz
FIDRES 112.007698 Hz
SW 190.000 ppm
FnMODE QF
F2 - Processing parameters
SI 4096
SF 600.1300100 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 QF
SF 150.9028849 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm

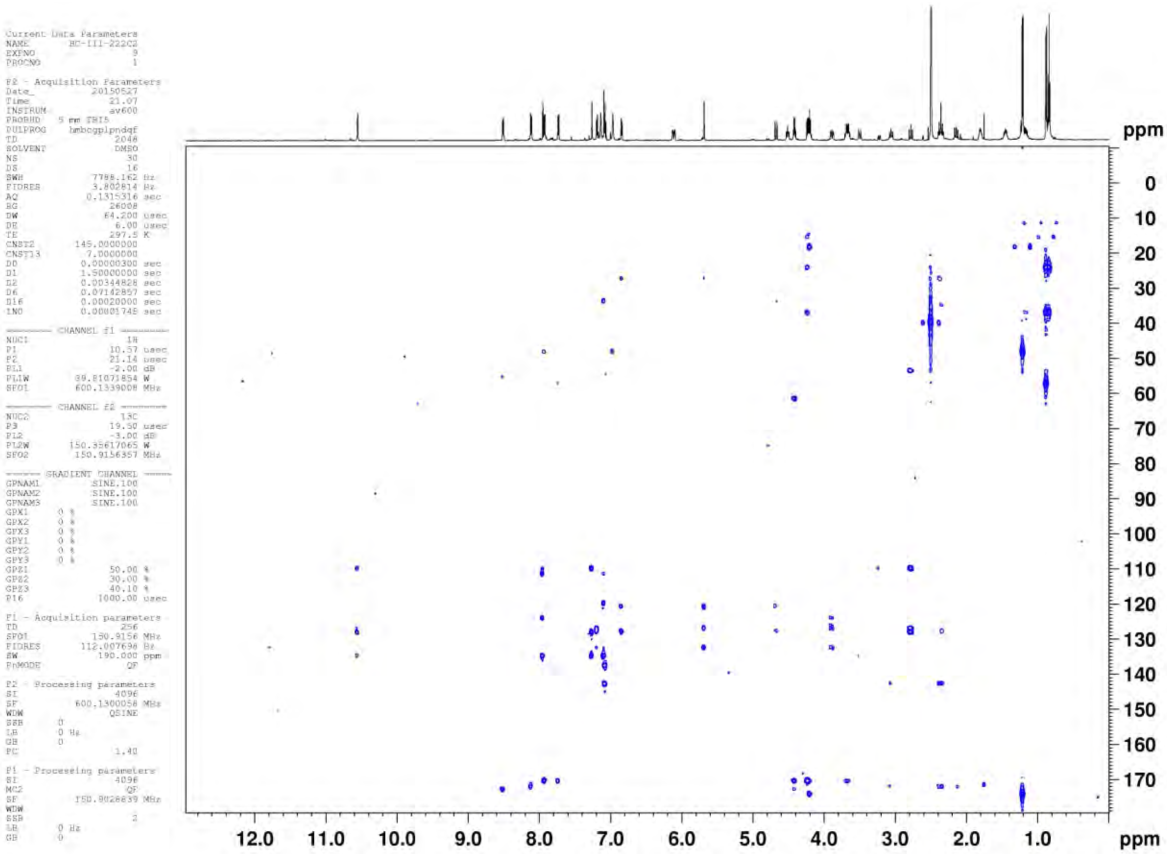
Macrocyclic Product ***16c***



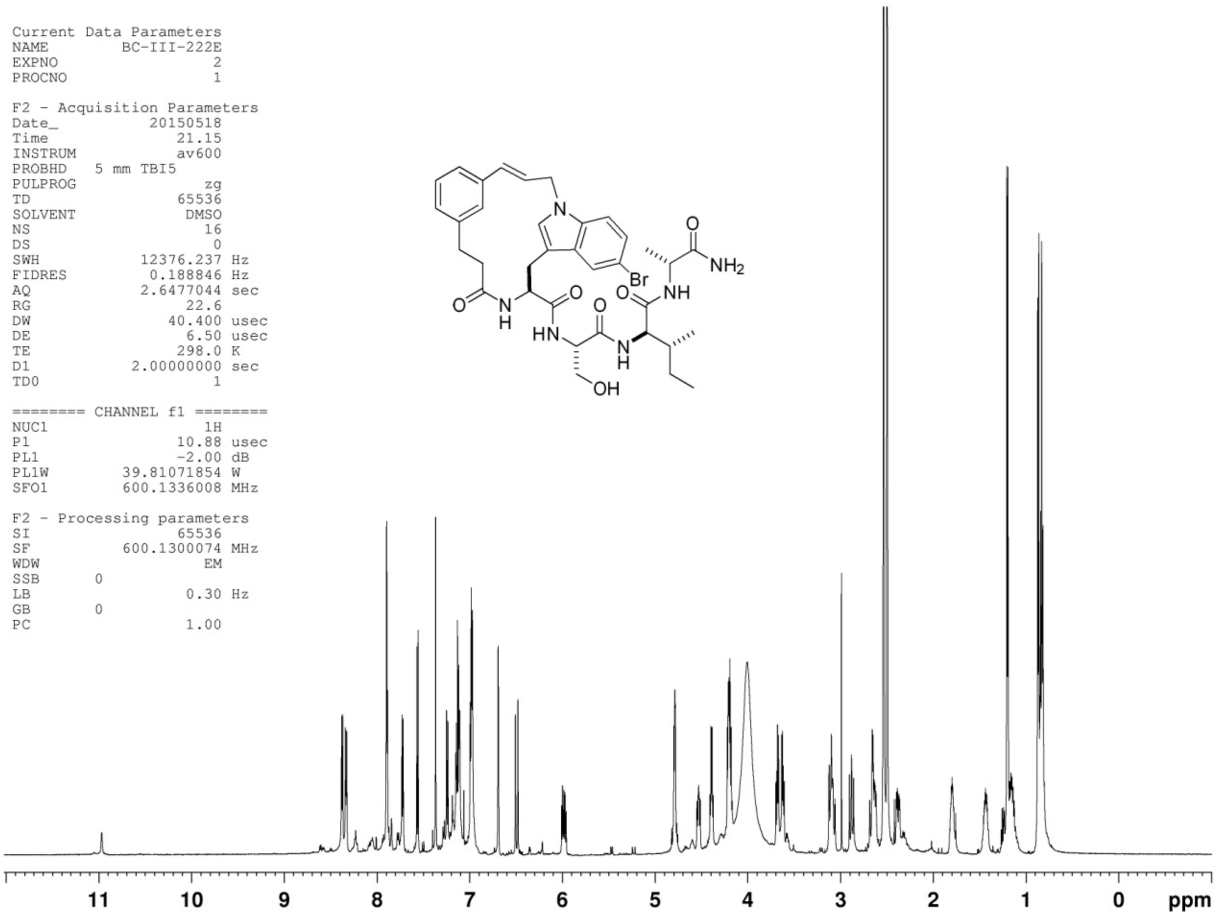


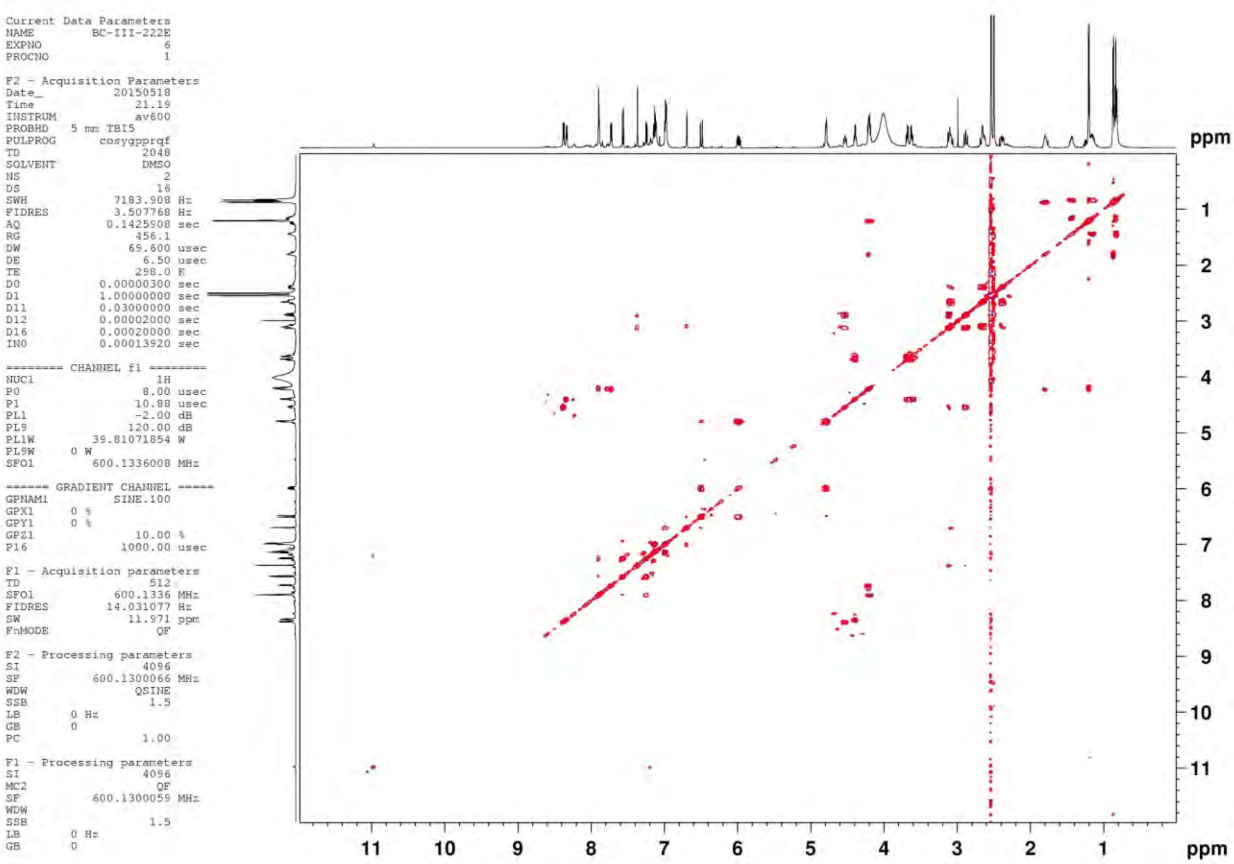

Current Data Parameters
NAME BC-III-222C2
ppm


Current Data Parameters
NAME BC-III-222C2
ppm

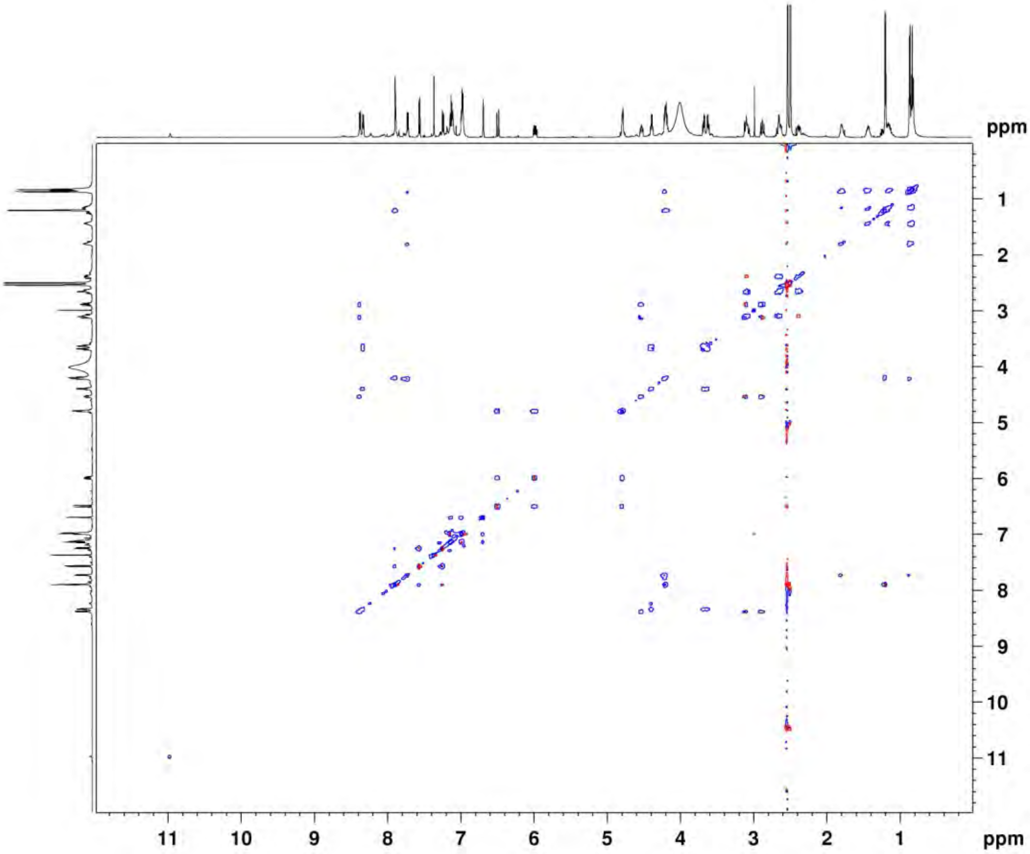

Current Data Parameters
NAME BC-III-222C2
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150527
Time 21.07
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 30
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 26008
DW 64.200 usec
DE 6.00 usec
TE 297.5 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec
CHANNEL f1
NUC1 1H
P1 10.57 usec
P2 21.14 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
CHANNEL f2
NUC2 13C
P3 19.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 150.9156 MHz
FIDRES 112.007698 Hz
SW 190.000 ppm
FnMODE QF
F2 - Processing parameters
SI 4096
SF 600.1300058 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 QF
SF 150.9028839 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0
ppm

*Macrocyclic Product **16d***

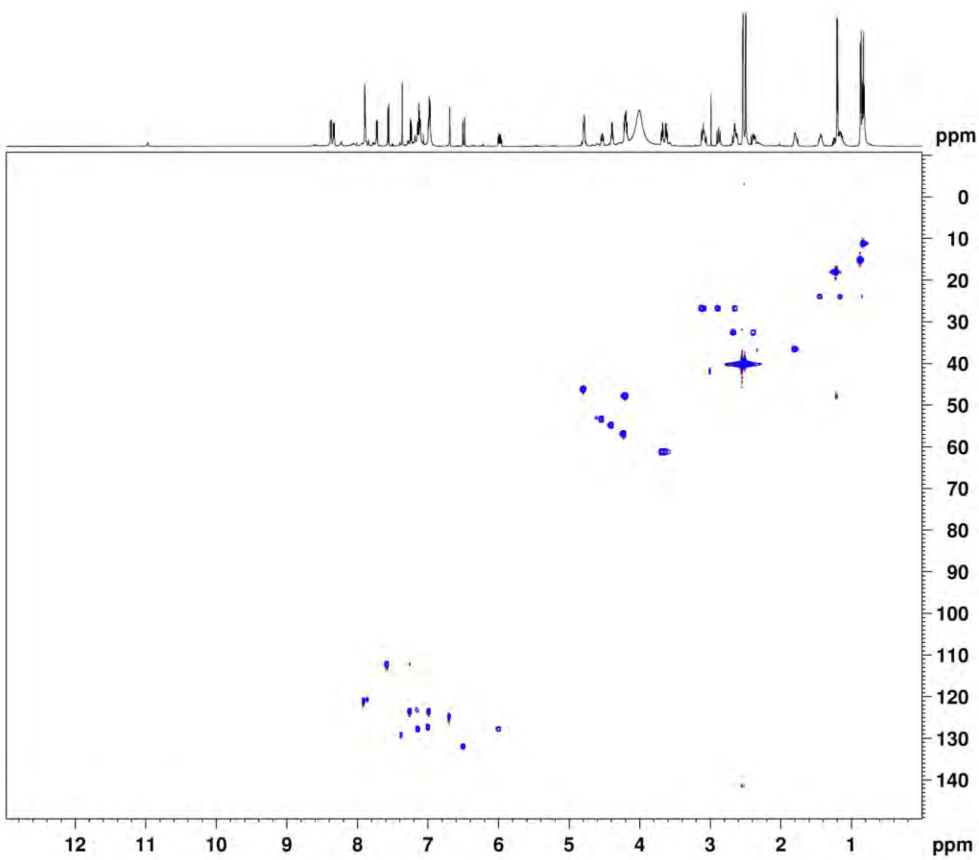


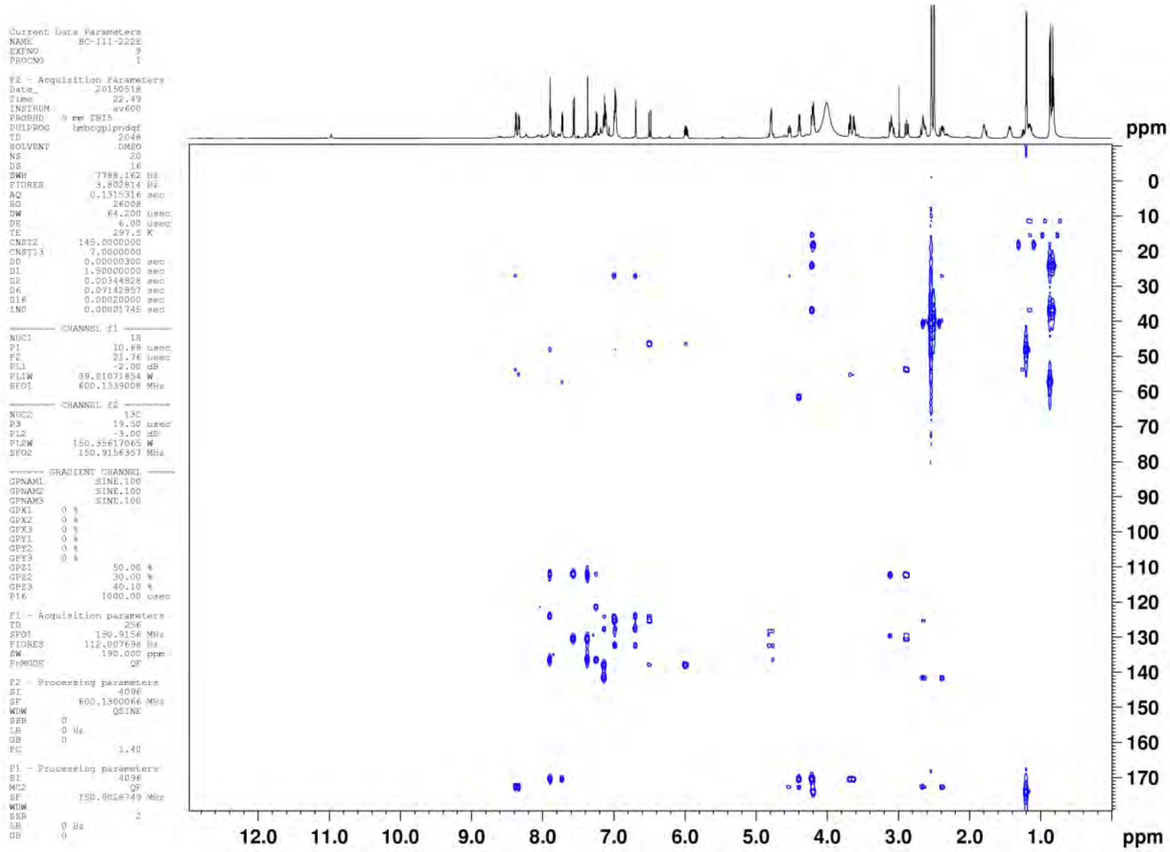


```
Current Data Parameters
NAME        BC-III-222E
EXPNO                 7
PROCNO                1

F2 - Acquisition Parameters
Date_          20150518
Time              21.40
INSTRUM           av600
PROBHD   5 mm TBI5
PULPROG      mlevesgpph
TD                 2048
SOLVENT            DMSO
NS                    2
DS                   16
SWH            7788.162 Hz
FIDRES         3.802814 Hz
AQ            0.1315316 sec
RG               1625.5
DW               64.200 usec
DE                 6.50 usec
TE                298.0 K
D0           0.00003727 sec
D1           1.00000000 sec
D9           0.06000000 sec
D12          0.00002000 sec
D16          0.00020000 sec
IN0          0.00012840 sec
L1                   24

======== CHANNEL f1 ========
NUC1                 1H
P1                10.88 usec
P2                21.76 usec
P5                26.68 usec
P6                40.00 usec
P7                80.00 usec
P12             3000.00 usec
P17             2500.00 usec
PL0              120.00 dB
PL1               -2.00 dB
PL10               9.31 dB
PL0W     0 W
PL1W        39.91071854 W
PL10W        2.94442129 W
SFO1        600.1339008 MHz
SP1              120.00 dB
SPNAM1     Squa100.1000
SPOAL1            1.000
SPOFFS1        -1456.44 Hz

====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPX1     0 %
GPX2     0 %
GPY1     0 %
GPY2     0 %
GPZ1              31.00 %
GPZ2              11.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  512
SFO1           600.1339 MHz
FIDRES        15.211276 Hz
SW               12.977 ppm
FnMODE      States-TPPI

F2 - Processing parameters
SI                 4096
SF          600.1300066 MHz
WDW               QSINE
SSB                   2
LB       0 Hz
GB       0
PC                 1.00

F1 - Processing parameters
SI                 4096
MC2         States-TPPI
SF          600.1300065 MHz
WDW
SSB                   2
LB       0 Hz
GB       0
```

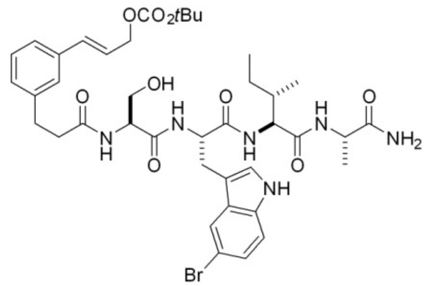


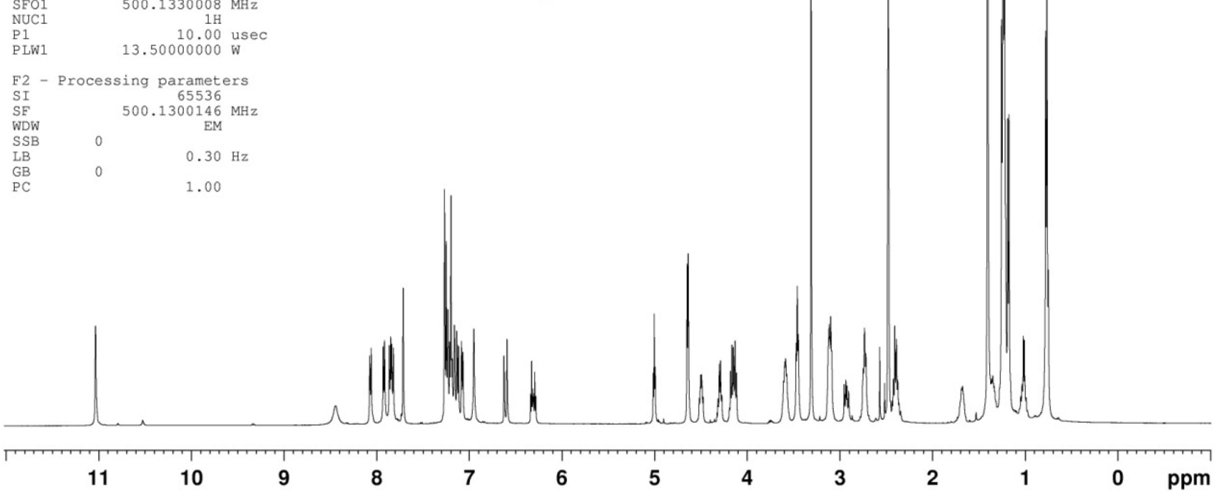

```
Current Data Parameters
NAME        BC-III-222E
EXPNO                 8
PROCNO                1

F2 - Acquisition Parameters
Date_          20150518
Time              22.02
INSTRUM           av600
PROBHD   5 mm TBI5
PULPROG     hsqcetgpsisp
TD                 2048
SOLVENT            DMSO
NS                    8
DS                   16
SWH            7788.162 Hz
FIDRES         3.802814 Hz
AQ            0.1315316 sec
RG              23170.5
DW               64.200 usec
DE                 6.00 usec
TE                298.4 K
CNST2       145.0000000
D0           0.00000300 sec
D1           1.20000005 sec
D4           0.00172414 sec
D11          0.03000000 sec
D16          0.00020000 sec
D24          0.00086200 sec
IN0          0.00002070 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                 1H
P1                10.88 usec
P2                21.76 usec
P28             1000.00 usec
PL1               -2.00 dB
PL1W        39.91071854 W
SFO1        600.1339008 MHz

======== CHANNEL f2 ========
CPDPRG2            garp
NUC2                13C
P3                13.50 usec
P4                39.00 usec
P14             1000.00 usec
PCPD2             65.00 usec
PL0              120.00 dB
PL2               -3.00 dB
PL12               7.46 dB
PL0W     0 W
PL2W       150.35617065 W
PL12W       13.52450085 W
SFO2        150.9133722 MHz
SP3                4.13 dB
SPNAM3    Crp80,0.5,20.1
SPOAL3            0.500
SPOFFS3  0 Hz

====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPX1     0 %
GPX2     0 %
GPY1     0 %
GPY2     0 %
GPZ1              80.00 %
GPZ2              20.10 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1           150.9134 MHz
FIDRES        94.320854 Hz
SW              160.000 ppm
FnMODE      Echo-Antiecho

F2 - Processing parameters
SI                 4096
SF          600.1300052 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40

F1 - Processing parameters
SI                 4096
MC2        echo-antiecho
SF          150.9028279 MHz
WDW
SSB                   2
LB       0 Hz
GB       0
```

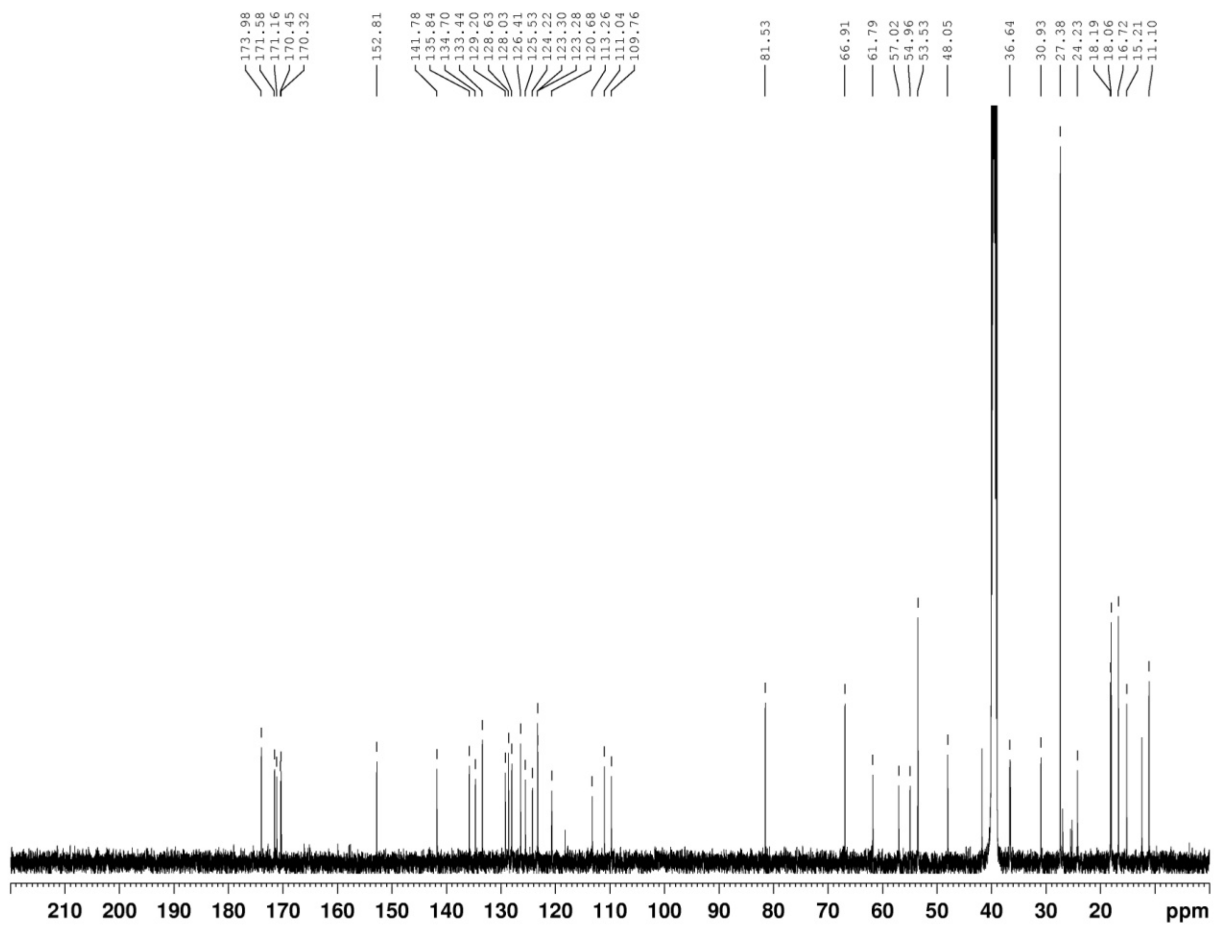



Current Data Parameters
NAME BC-III-222E
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150518
Time 22.49
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 20
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 26008
DW 64.200 usec
DE 6.00 usec
TE 297.5 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec
CHANNEL f1
NUC1 1H
P1 10.88 usec
P2 21.76 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
CHANNEL f2
NUC2 13C
P3 19.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 150.9156 MHz
FIDRES 112.007698 Hz
SW 190.000 ppm
FnMODE QF
F2 - Processing parameters
SI 4096
SF 600.1300066 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 QF
SF 150.9028749 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0
ppm

Acyclic Precursor **13**

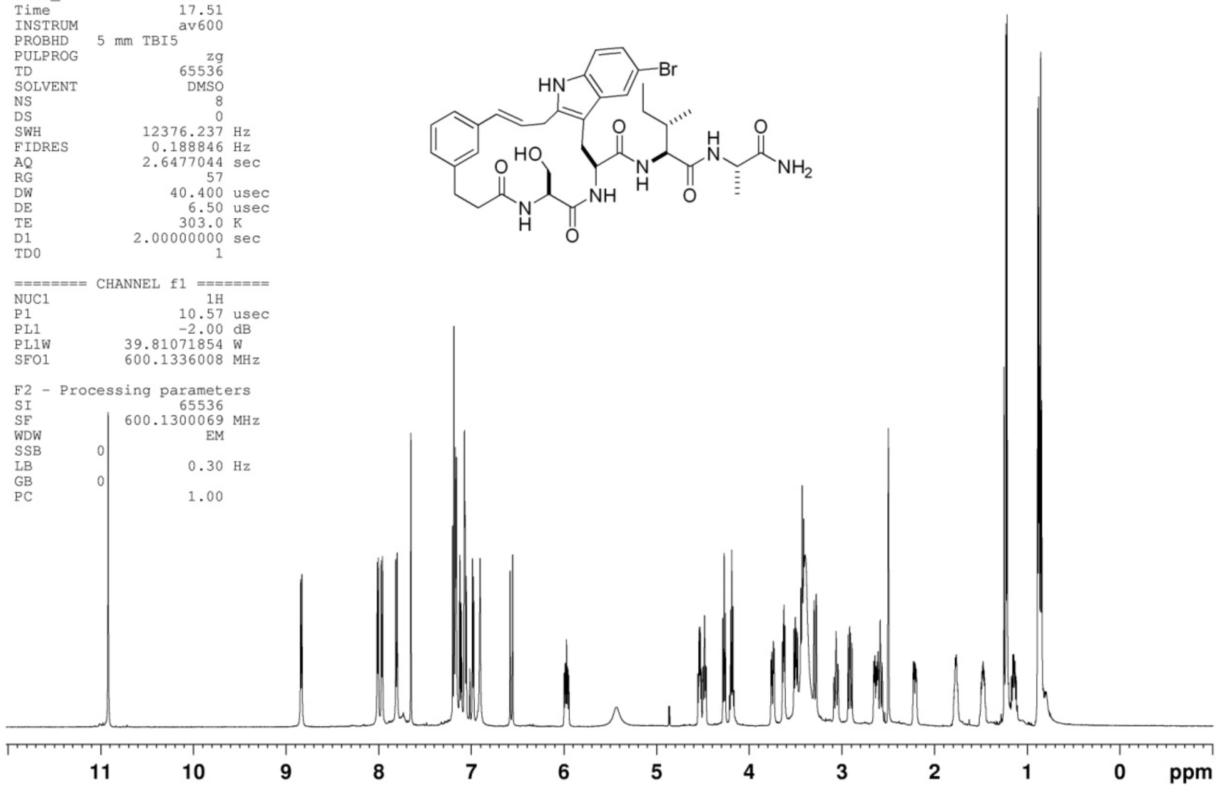




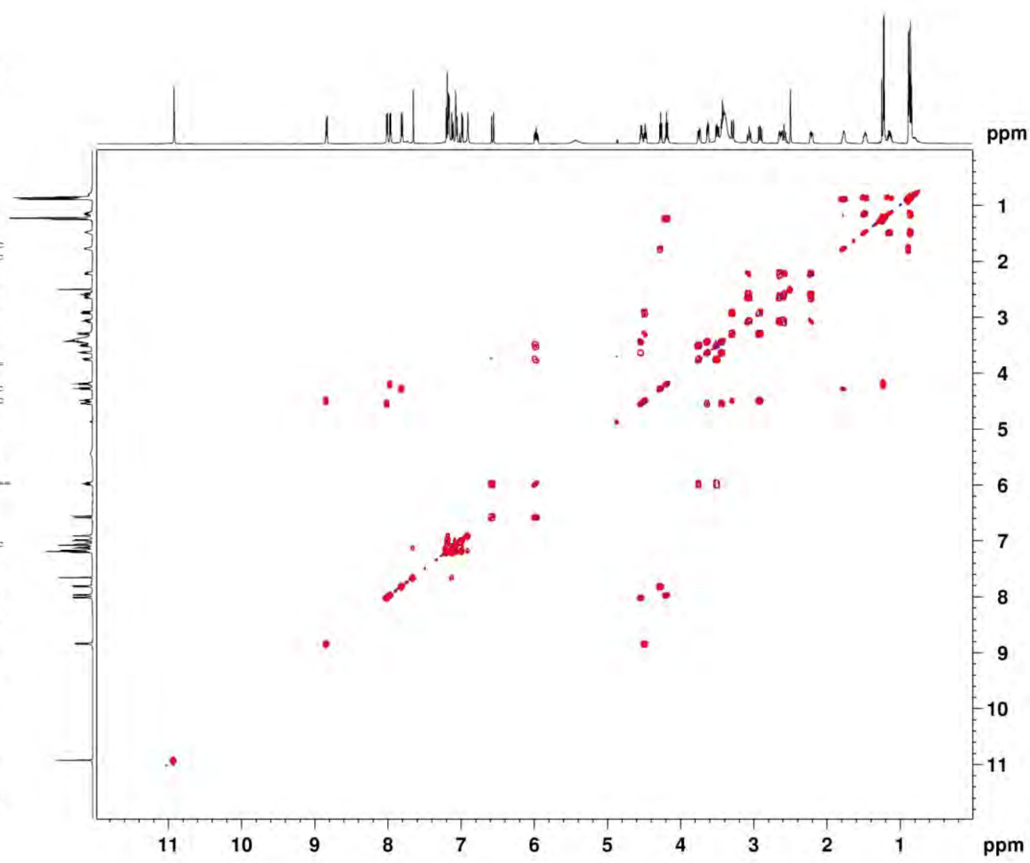


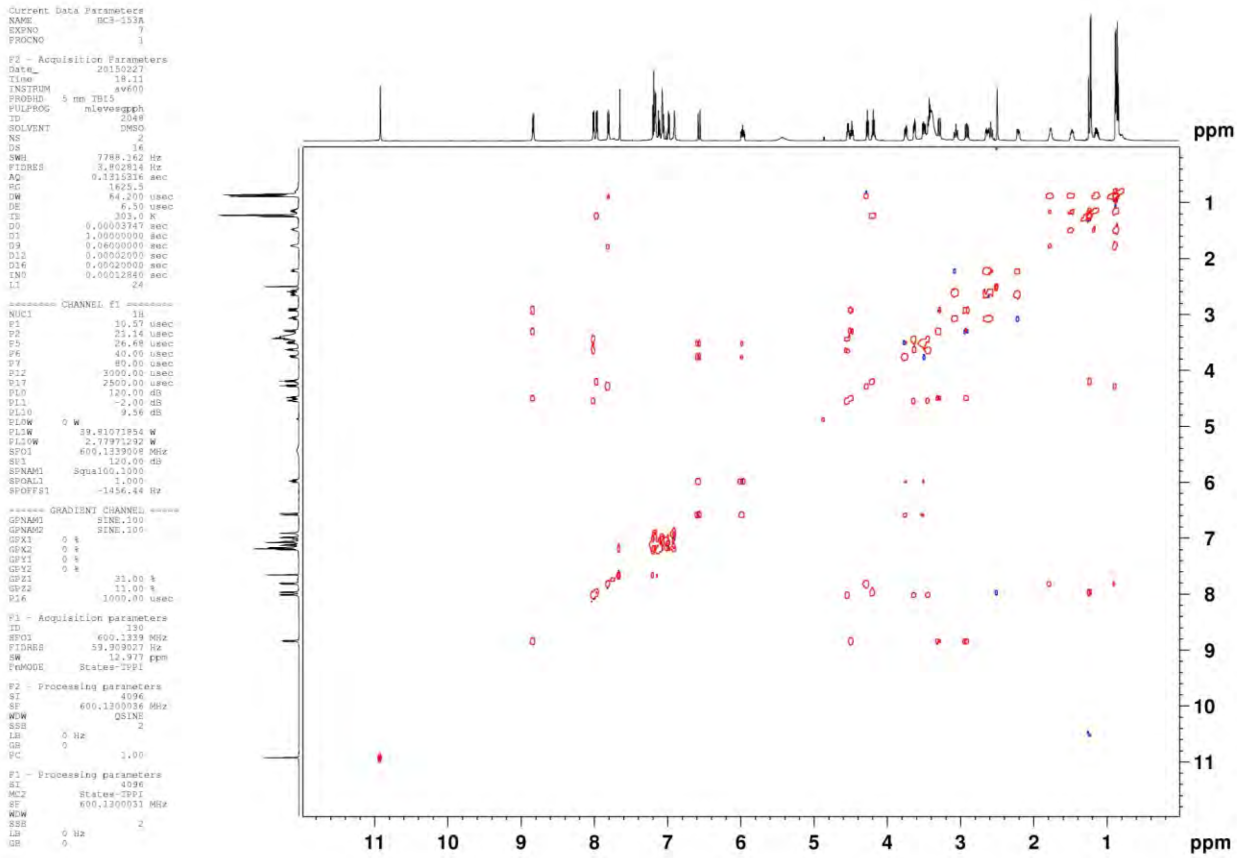
*Macrocyclic Product **17a***

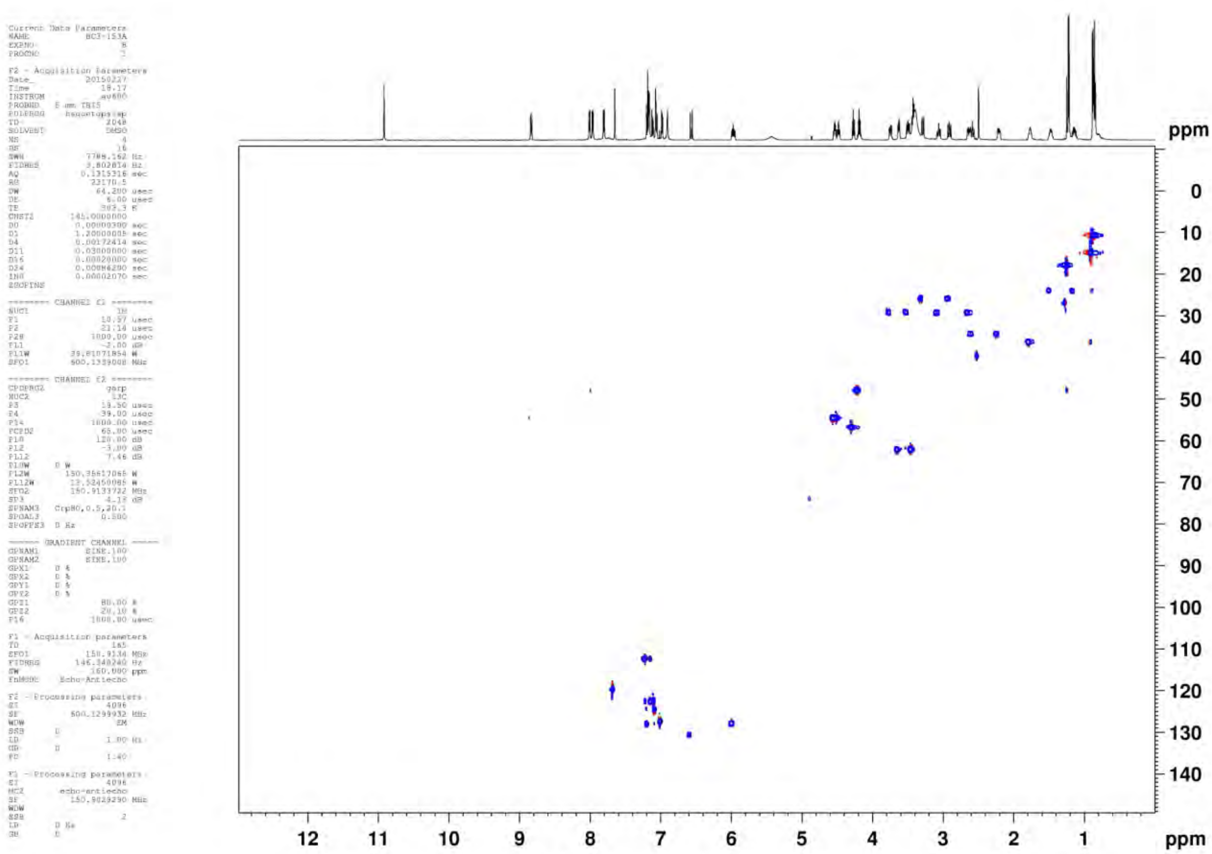
```
Current Data Parameters
NAME             BC3-153A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150227
Time                17.51
INSTRUM             av600
PROBHD   5 mm TBI5
PULPROG                zg
TD                  65536
SOLVENT              DMSO
NS                      8
DS                      0
SWH             12376.237 Hz
FIDRES           0.188846 Hz
AQ              2.6477044 sec
RG                     57
DW                 40.400 usec
DE                   6.50 usec
TE                  303.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.57 usec
PL1                 -2.00 dB
PL1W          39.81071854 W
SFO1          600.1336008 MHz

F2 - Processing parameters
SI                  65536
SF            600.1300069 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```

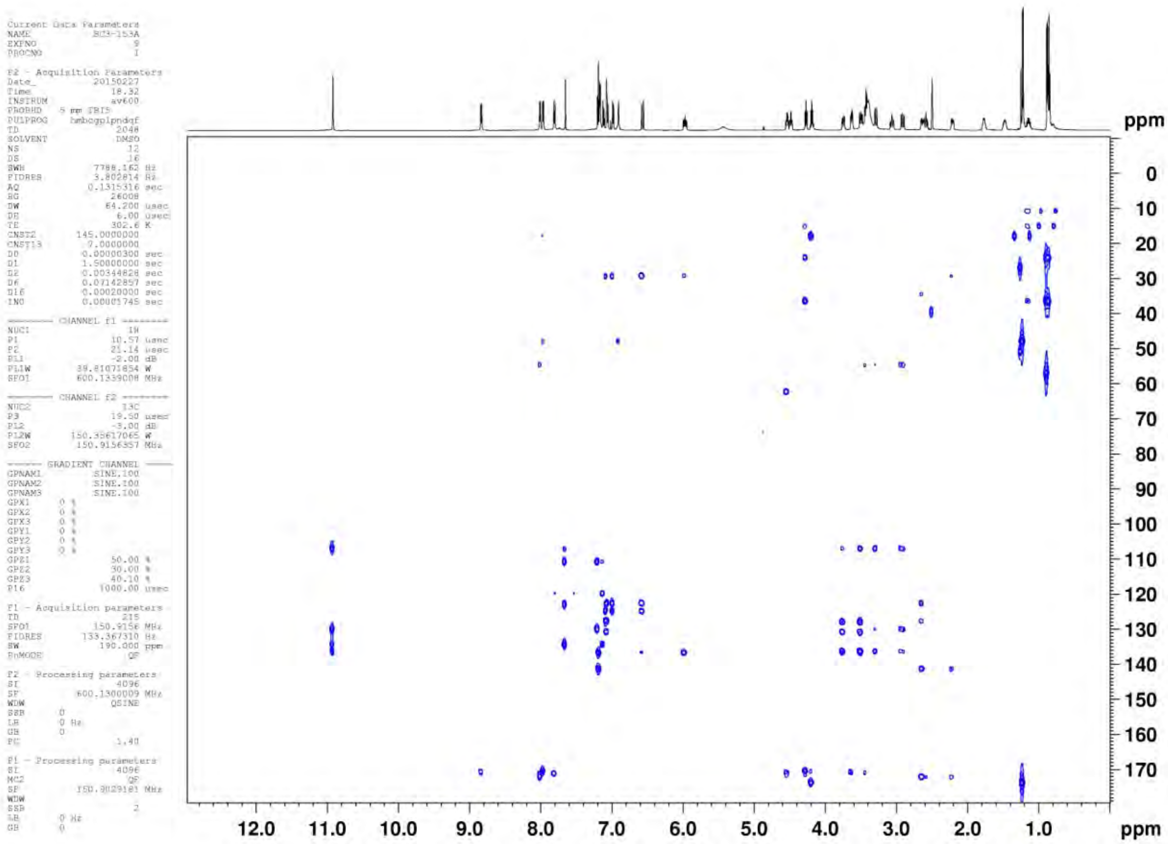



```
Current Data Parameters
NAME             BC3-153A
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150227
Time                17.54
INSTRUM             av600
PROBHD   5 mm TBI5
PULPROG        cosygpprqf
TD                   2048
SOLVENT              DMSO
NS                      2
DS                     16
SWH              7183.908 Hz
FIDRES           3.507768 Hz
AQ              0.1425908 sec
RG                    362
DW                 69.600 usec
DE                   6.50 usec
TE                  303.0 K
D0             0.00000300 sec
D1             1.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00013920 sec

======== CHANNEL f1 ========
NUC1                   1H
P0                   8.00 usec
P1                  10.57 usec
PL1                 -2.00 dB
PL9                120.00 dB
PL1W          39.81071854 W
PL9W     0 W
SFO1          600.1336008 MHz

====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPX1     0 %
GPY1     0 %
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    259
SFO1             600.1336 MHz
FIDRES          27.737112 Hz
SW                 11.971 ppm
FnMODE                 QF

F2 - Processing parameters
SI                   4096
SF            600.1300052 MHz
WDW                 QSINE
SSB                   1.5
LB       0 Hz
GB       0
PC                   1.00

F1 - Processing parameters
SI                   4096
MC2                    QF
SF            600.1300052 MHz
WDW
SSB                   1.5
LB       0 Hz
GB       0
```



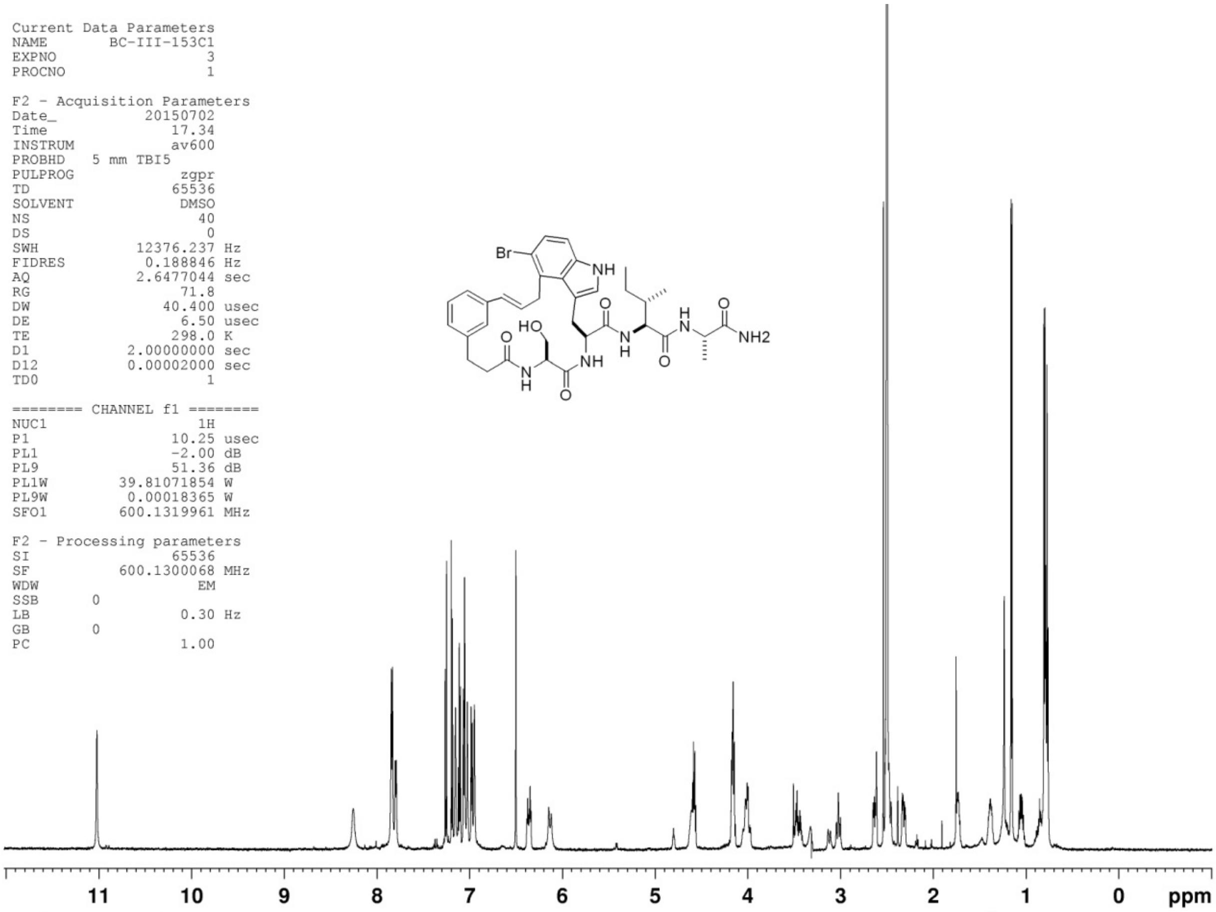

Current Data Parameters
NAME BC3-153A
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150227
Time 18.11
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevesgpph
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 1625.5
DW 64.200 usec
DE 6.50 usec
TE 303.0 K
D0 0.00003747 sec
D1 1.00000000 sec
D9 0.06000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00012840 sec
L1 24
======== CHANNEL f1 ========
NUC1 1H
P1 10.57 usec
P2 21.14 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P12 3000.00 usec
P17 2500.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL10 9.56 dB
PL0W 0 W
PL1W 39.81071854 W
PL10W 2.77971292 W
SFO1 600.1339008 MHz
SP1 120.00 dB
SPNAM1 Squa100.1000
SPOAL1 1.000
SPOFFS1 -1456.44 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 31.00 %
GPZ2 11.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 130
SFO1 600.1339 MHz
FIDRES 59.909027 Hz
SW 12.977 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 4096
SF 600.1300036 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 4096
MC2 States-TPPI
SF 600.1300031 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
1
2
3
4
5
6
7
8
9
10
11
11 10 9 8 7 6 5 4 3 2 1 ppm


Current Data Parameters
NAME BC3-153A
EXPNO 8
F2 - Acquisition Parameters
Date_ 20150227
Time 18.17
INSTRUM av600
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 23170.5
DW 64.200 usec
DE 6.00 usec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
IN0 0.00002070 sec
NUC1 1H
P1 10.57 usec
P2 21.14 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
CPDPRG2 garp
NUC2 13C
P3 19.50 usec
P4 39.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.46 dB
PL0W 0 W
PL2W 150.35617066 W
SFO2 150.9133722 MHz
SPOAL3 0.500
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 165
SFO1 150.9134 MHz
FIDRES 146.348240 Hz
SW 160.000 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 4096
SF 600.1299932 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 echo-antiecho
SF 150.9029290 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
12 11 10 9 8 7 6 5 4 3 2 1 ppm

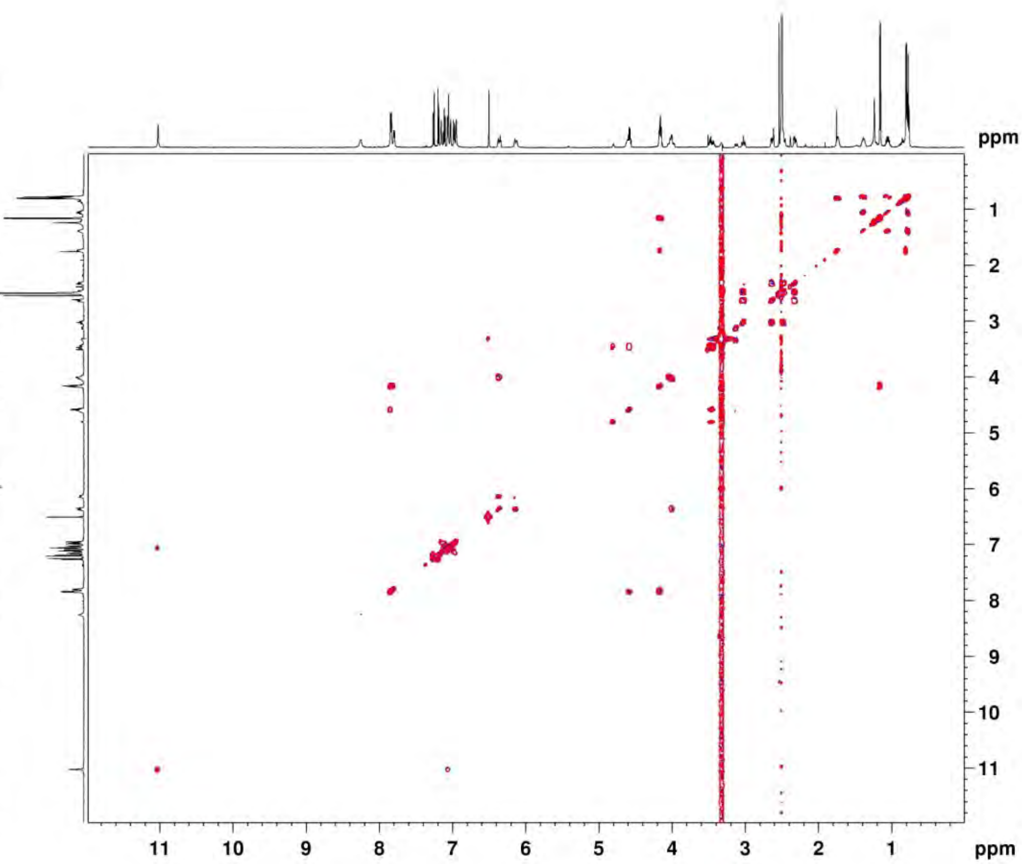


Macrocyclic Product ***17b***

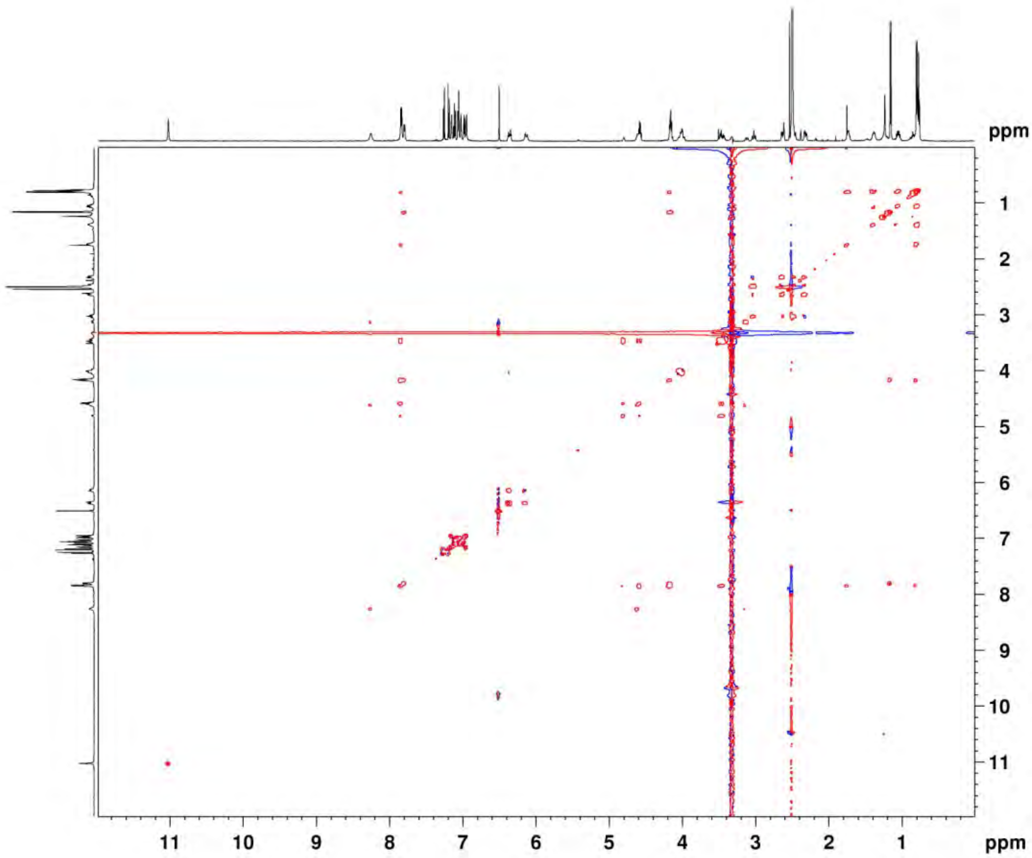
```
Current Data Parameters
NAME        BC-III-153C1
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150702
Time               17.34
INSTRUM            av600
PROBHD    5 mm TBI5
PULPROG             zgpr
TD                 65536
SOLVENT             DMSO
NS                    40
DS                     0
SWH            12376.237 Hz
FIDRES          0.188846 Hz
AQ             2.6477044 sec
RG                  71.8
DW                40.400 usec
DE                  6.50 usec
TE                 298.0 K
D1            2.00000000 sec
D12           0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.25 usec
PL1                -2.00 dB
PL9                51.36 dB
PL1W         39.81071854 W
PL9W          0.00018365 W
SFO1         600.1319961 MHz

F2 - Processing parameters
SI                 65536
SF           600.1300068 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



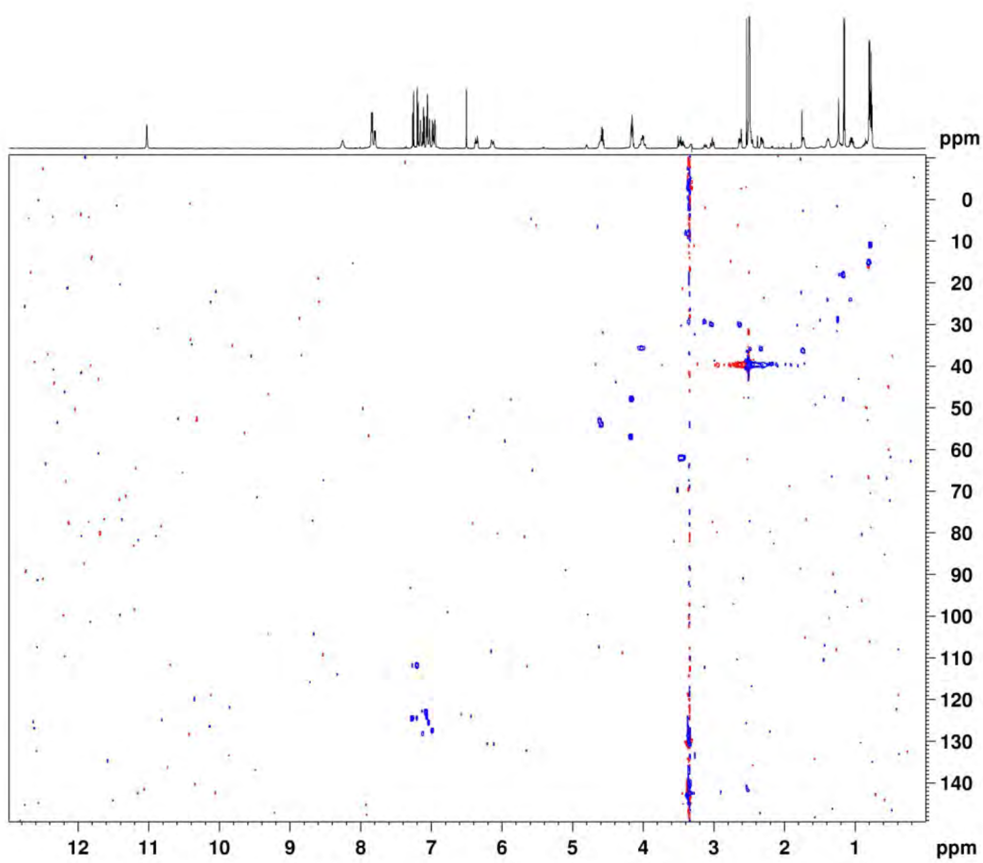

```
Current Data Parameters
NAME        BC-III-153C1
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150702
Time               17.38
INSTRUM            av600
PROBHD    5 mm TBI5
PULPROG        cosygpprqf
TD                  2048
SOLVENT             DMSO
NS                     2
DS                    16
SWH             7183.908 Hz
FIDRES          3.507768 Hz
AQ             0.1425908 sec
RG                 456.1
DW                69.600 usec
DE                  6.50 usec
TE                 298.0 K
D0            0.00000300 sec
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00013920 sec

======== CHANNEL f1 ========
NUC1                  1H
P0                  8.00 usec
P1                 10.25 usec
PL1                -2.00 dB
PL9               120.00 dB
PL1W         39.81071854 W
PL9W      0 W
SFO1         600.1336008 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPX1      0 %
GPY1      0 %
GPZ1               10.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   512
SFO1            600.1336 MHz
FIDRES         14.031077 Hz
SW                11.971 ppm
FnMODE                QF

F2 - Processing parameters
SI                  4096
SF           600.1300086 MHz
WDW                QSINE
SSB                  1.5
LB        0 Hz
GB        0
PC                  1.00

F1 - Processing parameters
SI                  4096
MC2                   QF
SF           600.1300082 MHz
WDW
SSB                  1.5
LB        0 Hz
GB        0
```

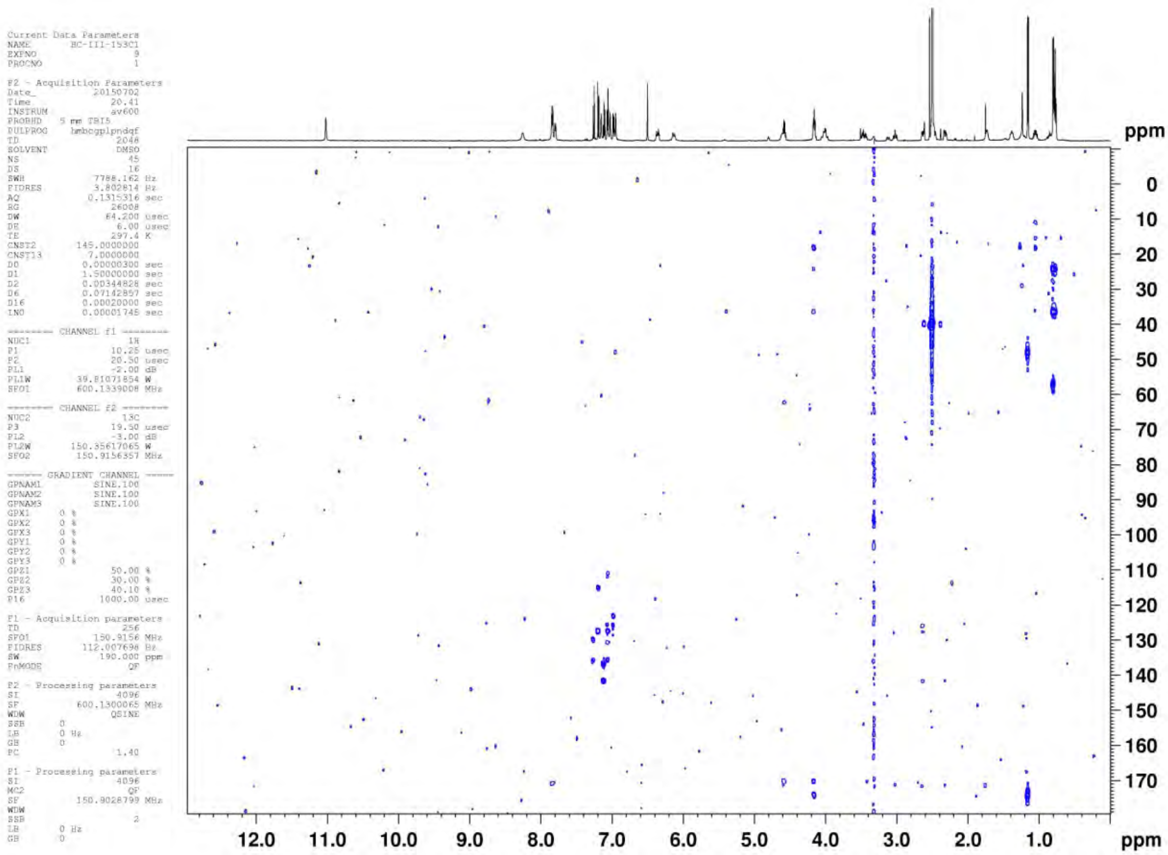


```
Current Data Parameters
NAME         BC-III-153C1
EXPNO                   7
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150702
Time                17.59
INSTRUM             av600
PROBHD   5 mm TBI5
PULPROG        mlevesgpph
TD                   2048
SOLVENT              DMSO
NS                      2
DS                     16
SWH              7788.162 Hz
FIDRES           3.802814 Hz
AQ              0.1315316 sec
RG                 1625.5
DW                 64.200 usec
DE                   6.50 usec
TE                  298.0 K
D0             0.00003767 sec
D1             1.00000000 sec
D9             0.06000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00012840 sec
L1                     24

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.25 usec
P2                  20.50 usec
P5                  26.68 usec
P6                  40.00 usec
P7                  80.00 usec
P12               3000.00 usec
P17               2500.00 usec
PL0                120.00 dB
PL1                 -2.00 dB
PL10                 9.83 dB
PL0W      0 W
PL1W          39.81071854 W
PL10W          2.61216140 W
SFO1          600.1339008 MHz
SP1                120.00 dB
SPNAM1        Squa100.1000
SPOAL1              1.000
SPOFFS1          -1456.44 Hz

====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPNAM2           SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                31.00 %
GPZ2                11.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    512
SFO1             600.1339 MHz
FIDRES          15.211276 Hz
SW                 12.977 ppm
FnMODE        States-TPPI

F2 - Processing parameters
SI                   4096
SF            600.1300057 MHz
WDW                 QSINE
SSB                     2
LB        0 Hz
GB        0
PC                   1.00

F1 - Processing parameters
SI                   4096
MC2           States-TPPI
SF            600.1300061 MHz
WDW
SSB                     2
LB        0 Hz
GB        0
```

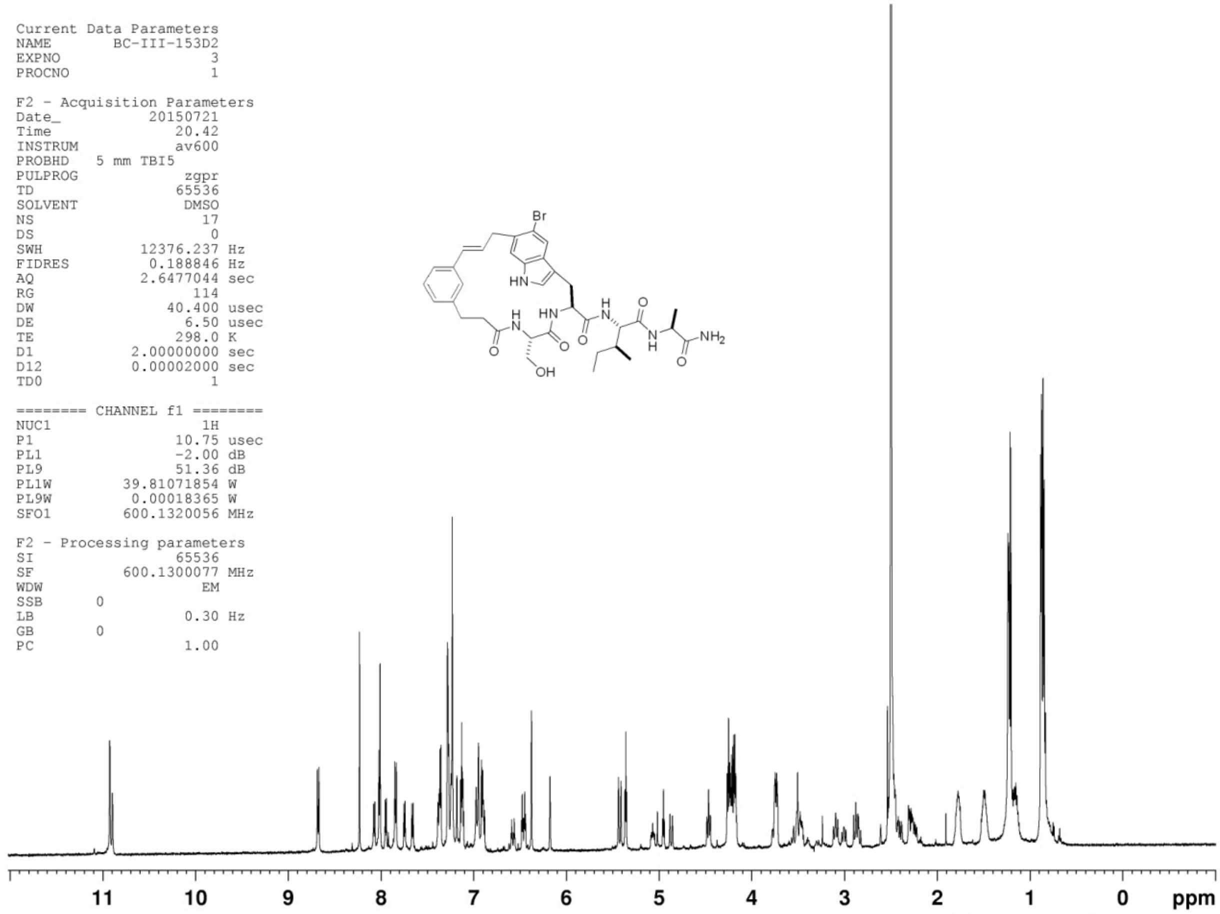


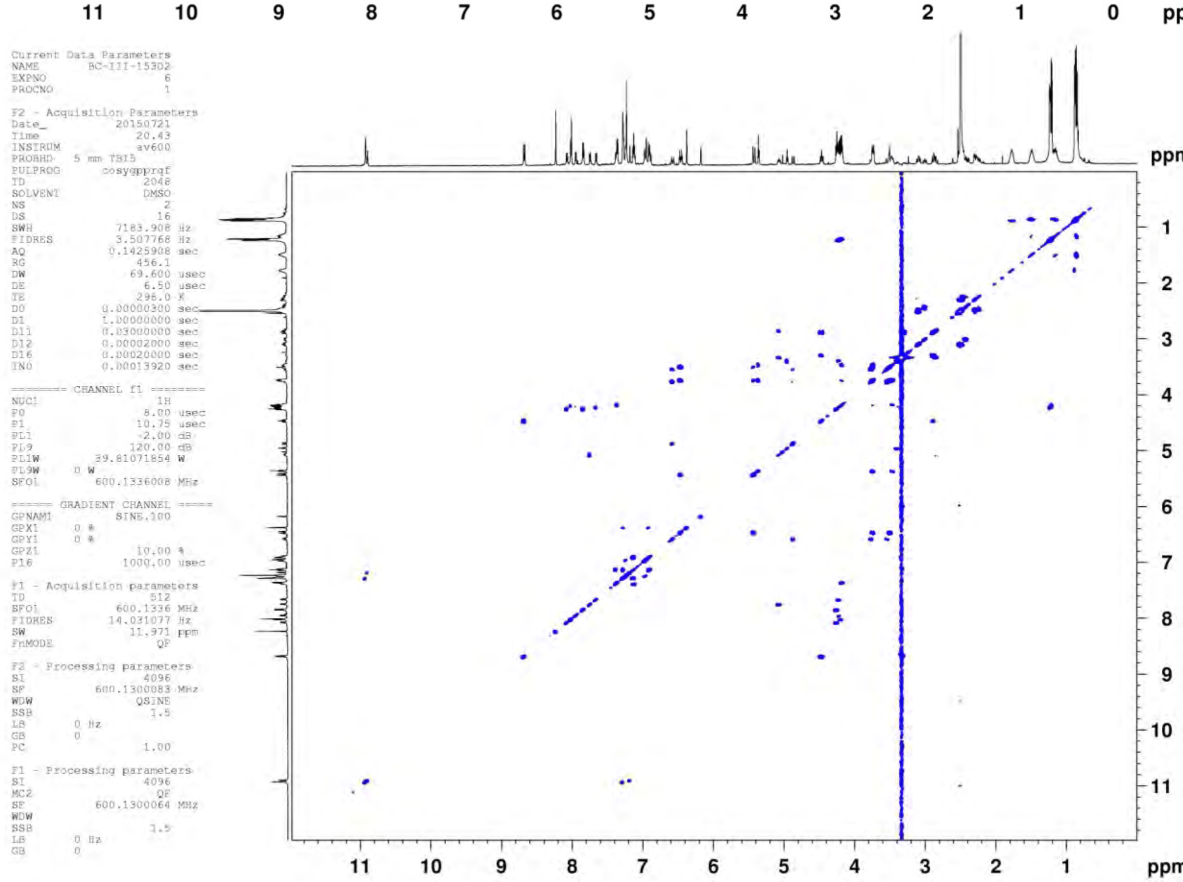

```
Current Data Parameters
NAME         BC-III-153C1
EXPNO                   8
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150702
Time                18.21
INSTRUM             av600
PROBHD   5 mm TBI5
PULPROG      hsqcetgpsisp
TD                   2048
SOLVENT              DMSO
NS                     24
DS                     16
SWH              7788.162 Hz
FIDRES           3.802814 Hz
AQ              0.1315316 sec
RG                23170.5
DW                 64.200 usec
DE                   6.00 usec
TE                  298.5 K
CNST2         145.0000000
D0             0.00000300 sec
D1             1.20000005 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D24            0.00086200 sec
IN0            0.00002070 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.25 usec
P2                  20.50 usec
P28               1000.00 usec
PL1                 -2.00 dB
PL1W          39.81071854 W
SFO1          600.1339008 MHz

======== CHANNEL f2 ========
CPDPRG2              garp
NUC2                  13C
P3                  19.50 usec
P4                  39.00 usec
P14               1000.00 usec
PCPD2               65.00 usec
PL0                120.00 dB
PL2                 -3.00 dB
PL12                 7.46 dB
PL0W      0 W
PL2W         150.35617065 W
PL12W         13.52450085 W
SFO2          150.9133722 MHz
SP3                  4.13 dB
SPNAM3    Crp80,0.5,20.1
SPOAL3              0.500
SPOFFS3   0 Hz

====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPNAM2           SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                80.00 %
GPZ2                20.10 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    256
SFO1             150.9134 MHz
FIDRES          94.320854 Hz
SW                160.000 ppm
FnMODE       Echo-Antiecho

F2 - Processing parameters
SI                   4096
SF            600.1300051 MHz
WDW                    EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40

F1 - Processing parameters
SI                   4096
MC2         echo-antiecho
SF            150.9029120 MHz
WDW
SSB                     2
LB        0 Hz
GB        0
```

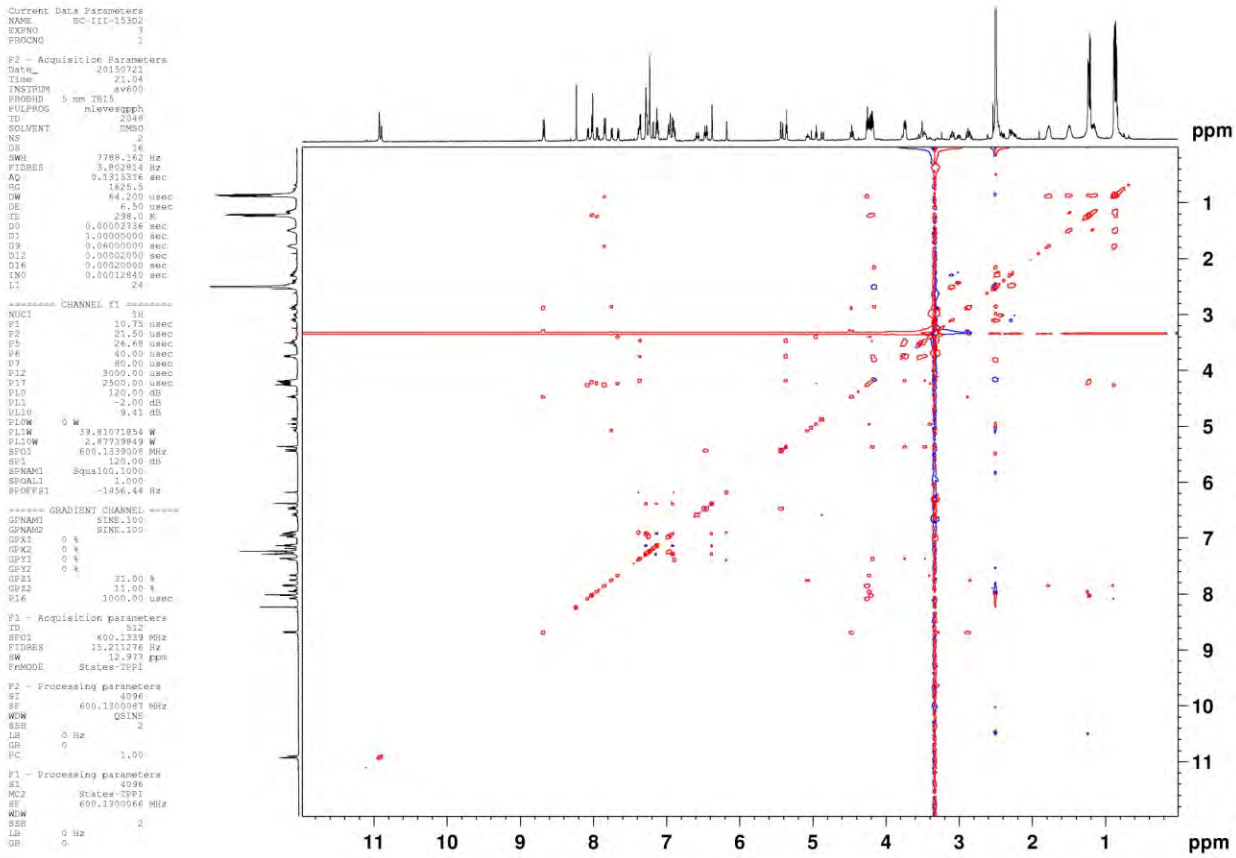


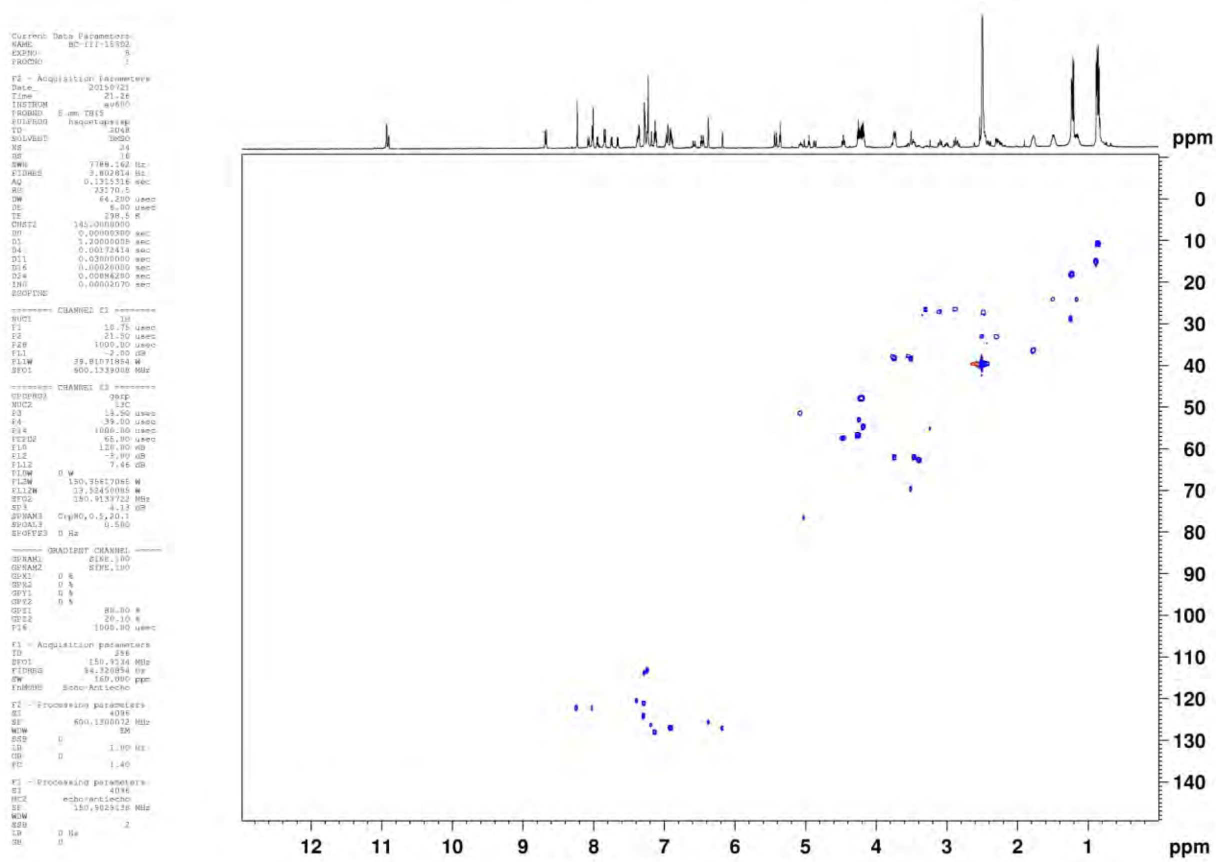


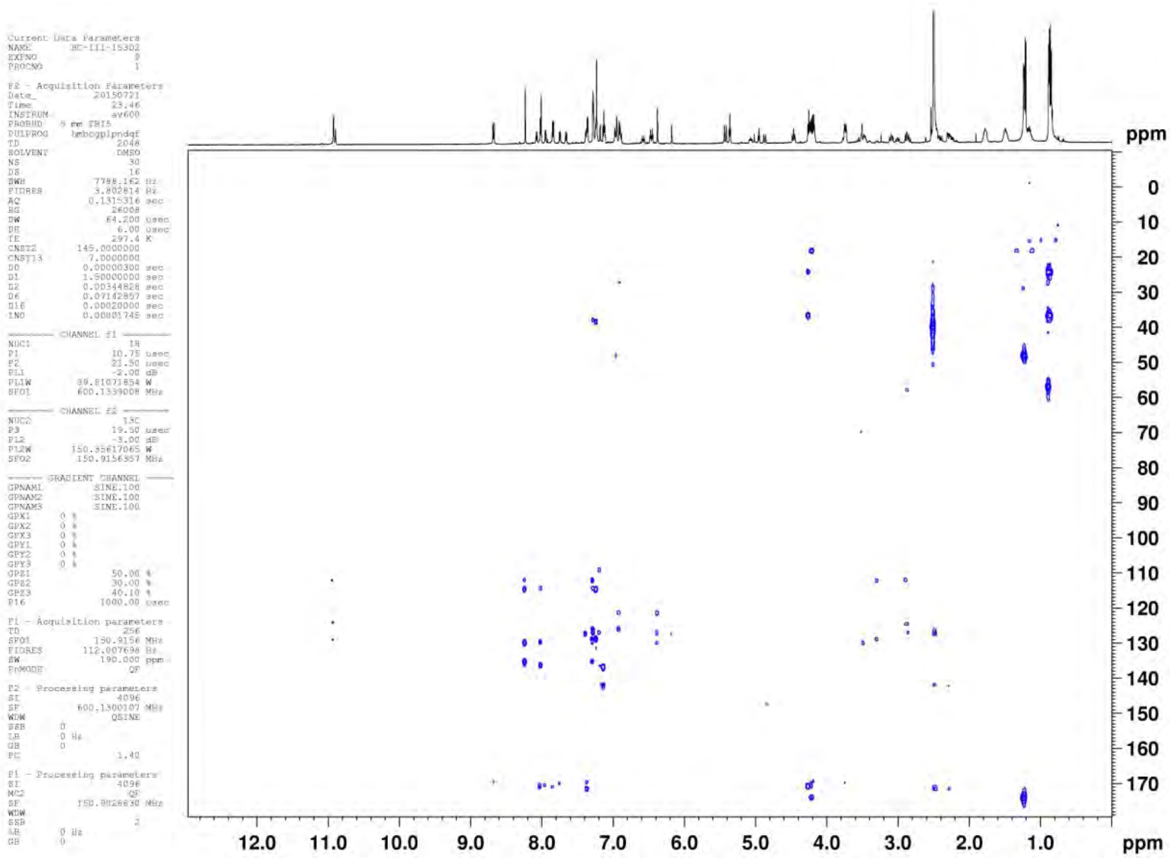
*Macrocyclic Product **17c***



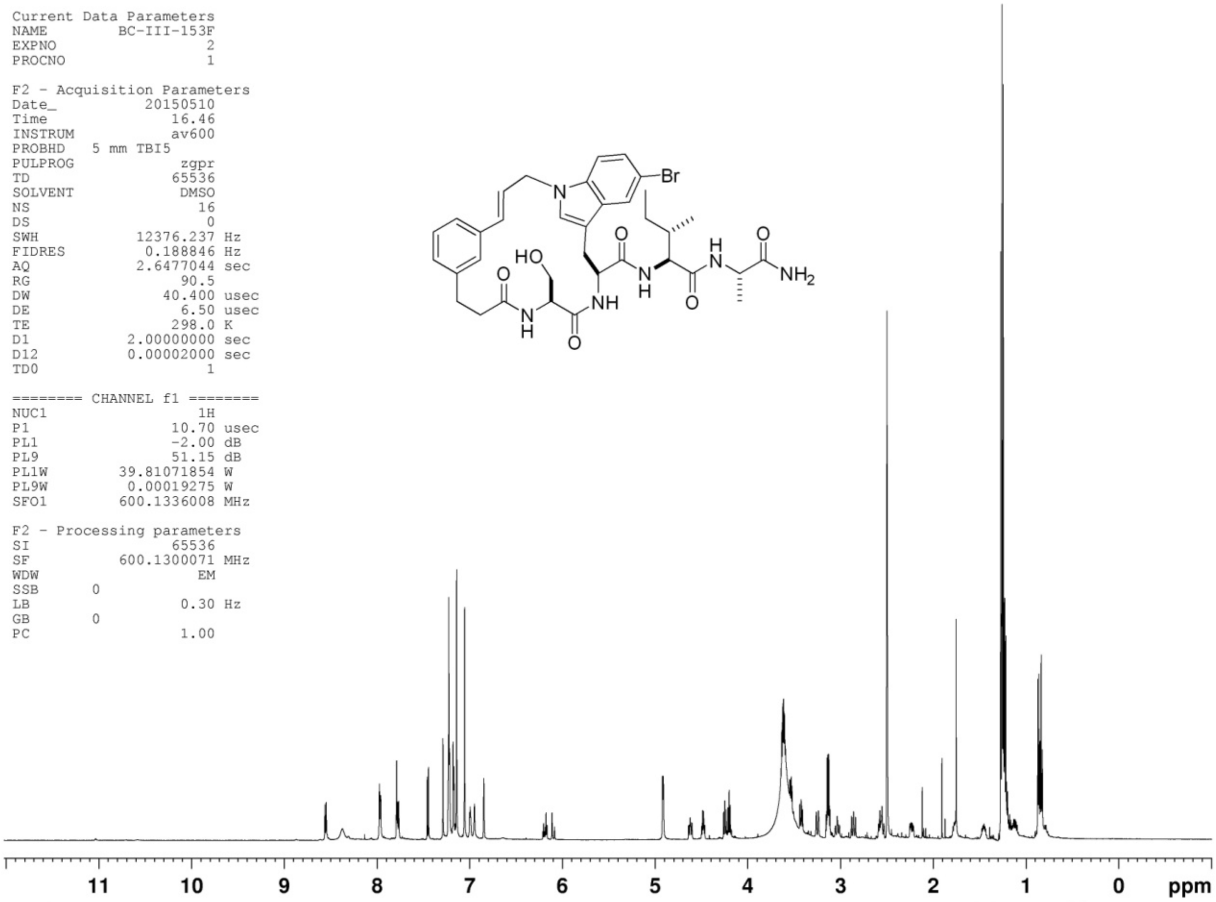


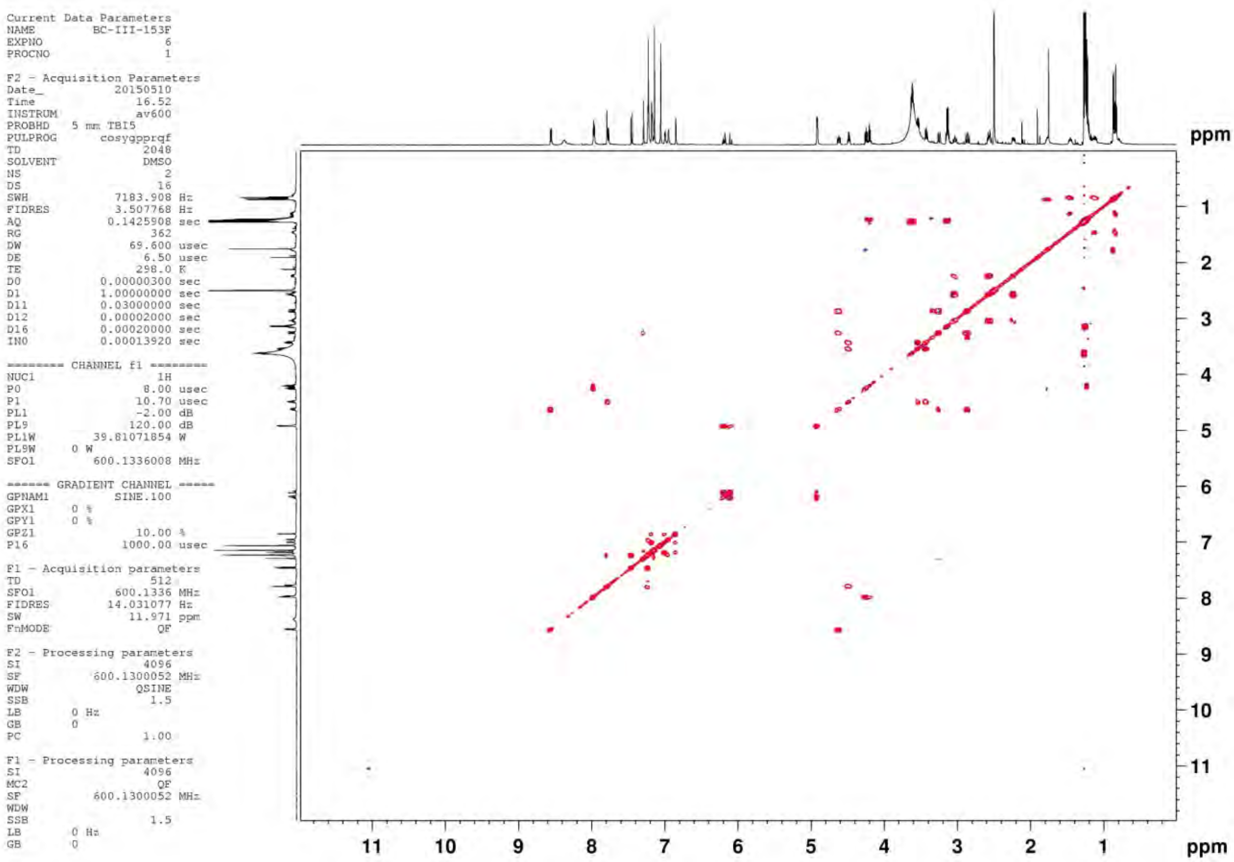

ppm
1
2
3
4
5
6
7
8
9
10
11
ppm
11 10 9 8 7 6 5 4 3 2 1

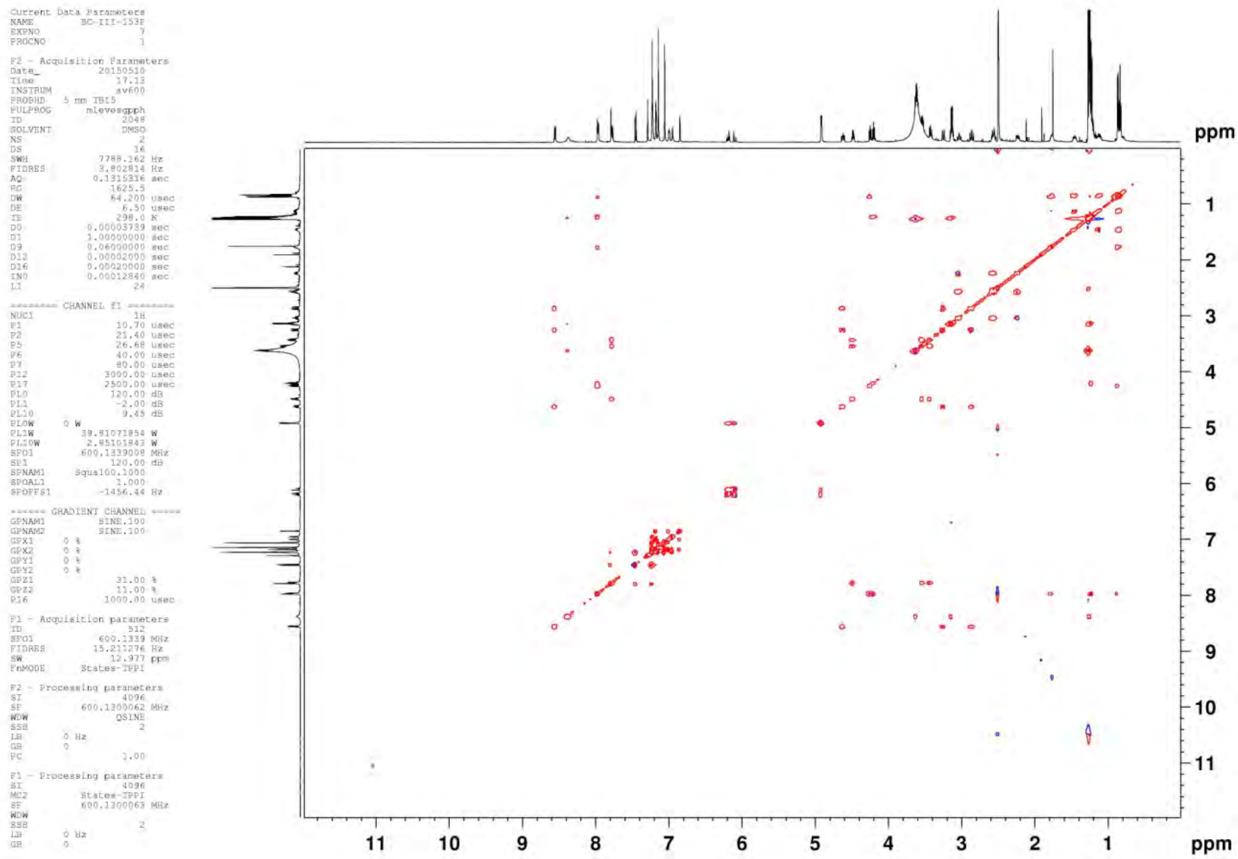

ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
ppm
12 11 10 9 8 7 6 5 4 3 2 1

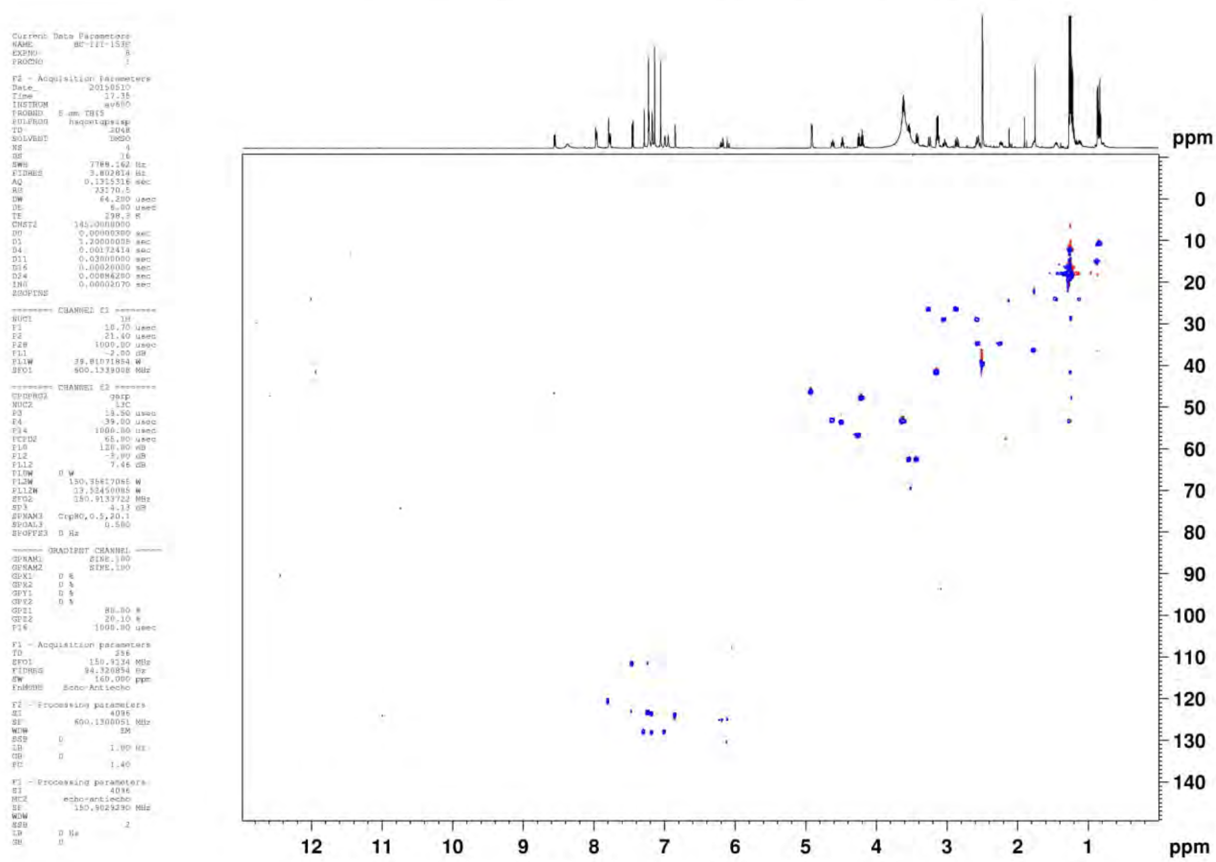


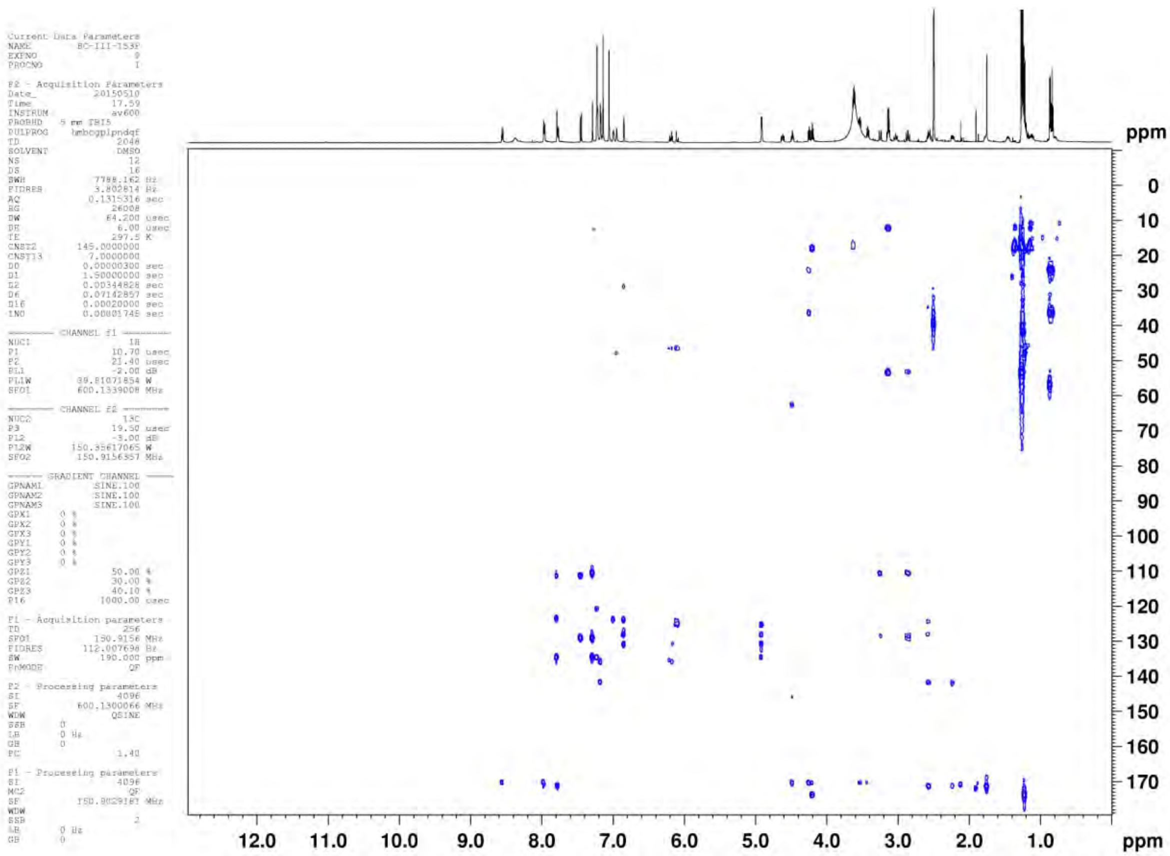
*Macrocyclic Product **17d***



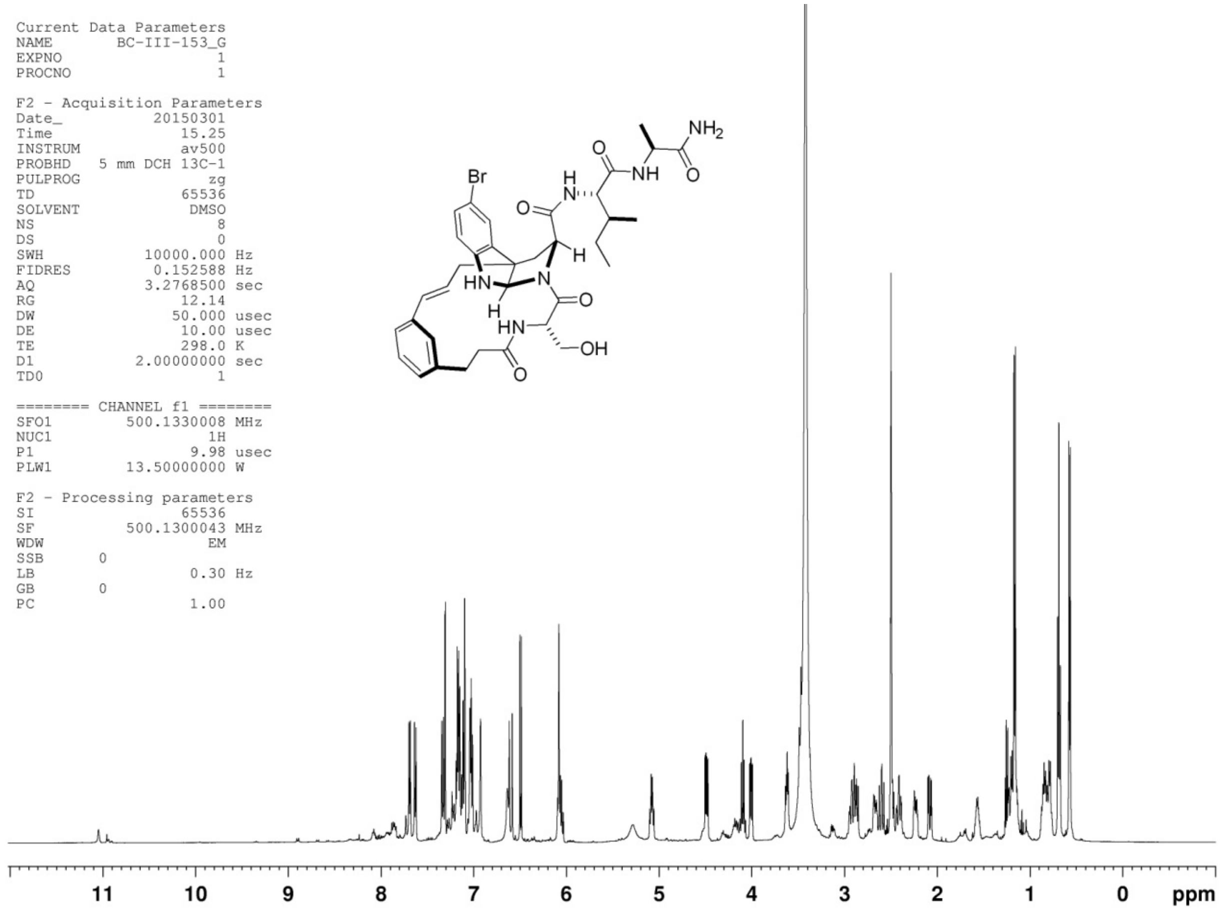


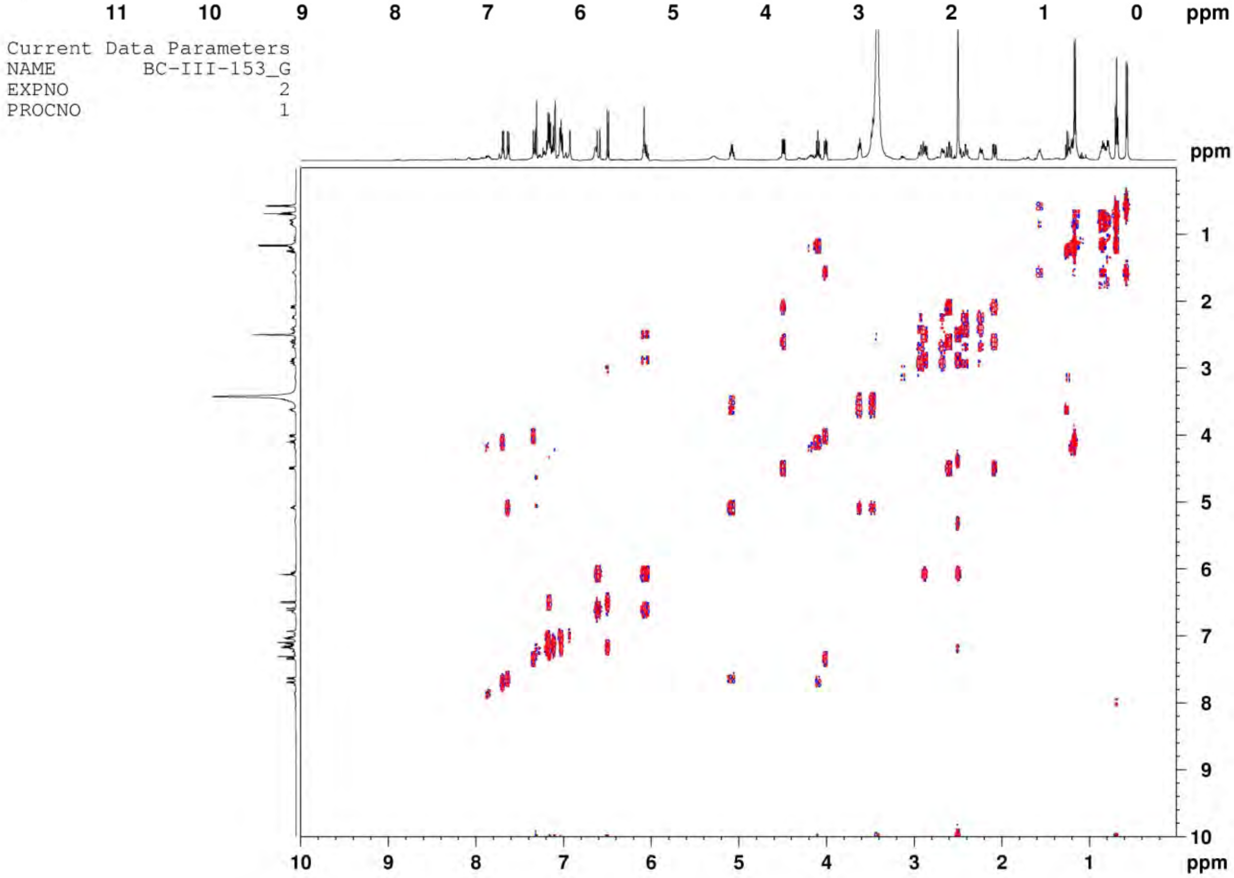

ppm
ppm
11 10 9 8 7 6 5 4 3 2 1
1 2 3 4 5 6 7 8 9 10 11


ppm
ppm
12 11 10 9 8 7 6 5 4 3 2 1
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140

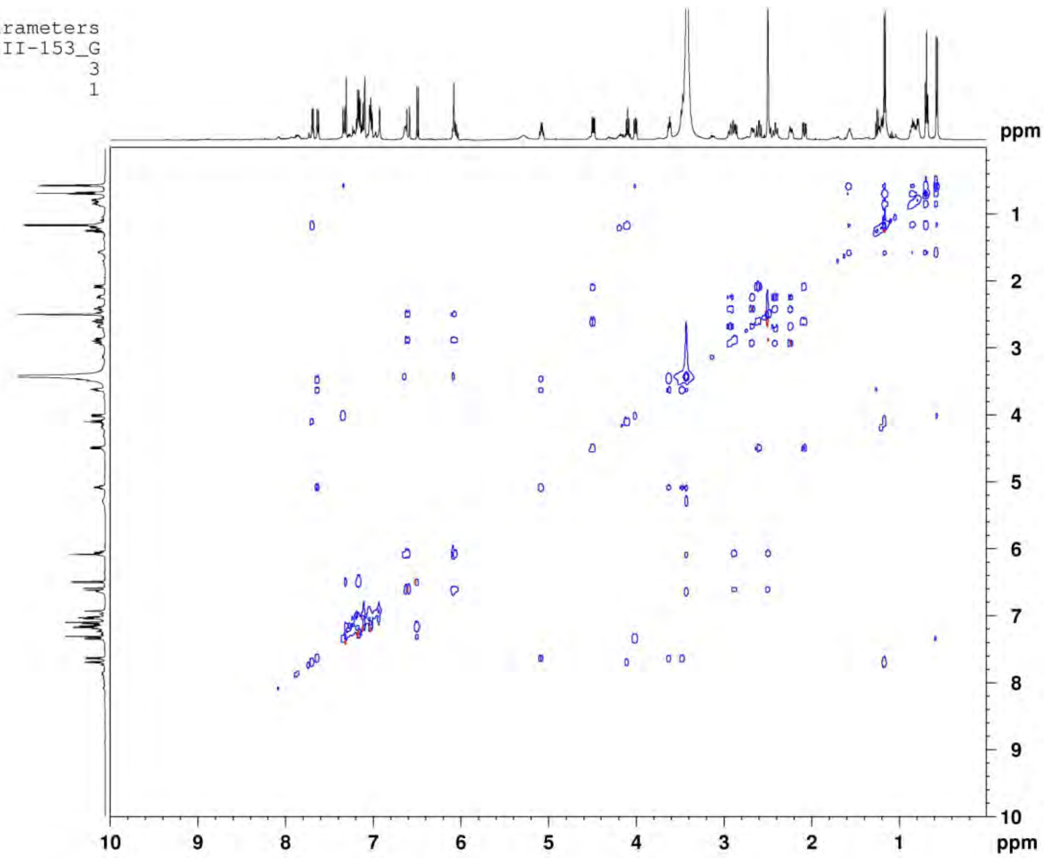

Current Data Parameters
NAME BC-III-153P
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150510
Time 17.59
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 12
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 26008
DW 64.200 usec
DE 6.00 usec
TE 297.5 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec
CHANNEL f1
NUC1 1H
P1 10.70 usec
P2 21.40 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
CHANNEL f2
NUC2 13C
P3 19.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 150.9156 MHz
FIDRES 112.007698 Hz
SW 190.000 ppm
FnMODE QF
F2 - Processing parameters
SI 4096
SF 600.1300066 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 QF
SF 150.9029187 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm

Macrocyclic Product ***17e***

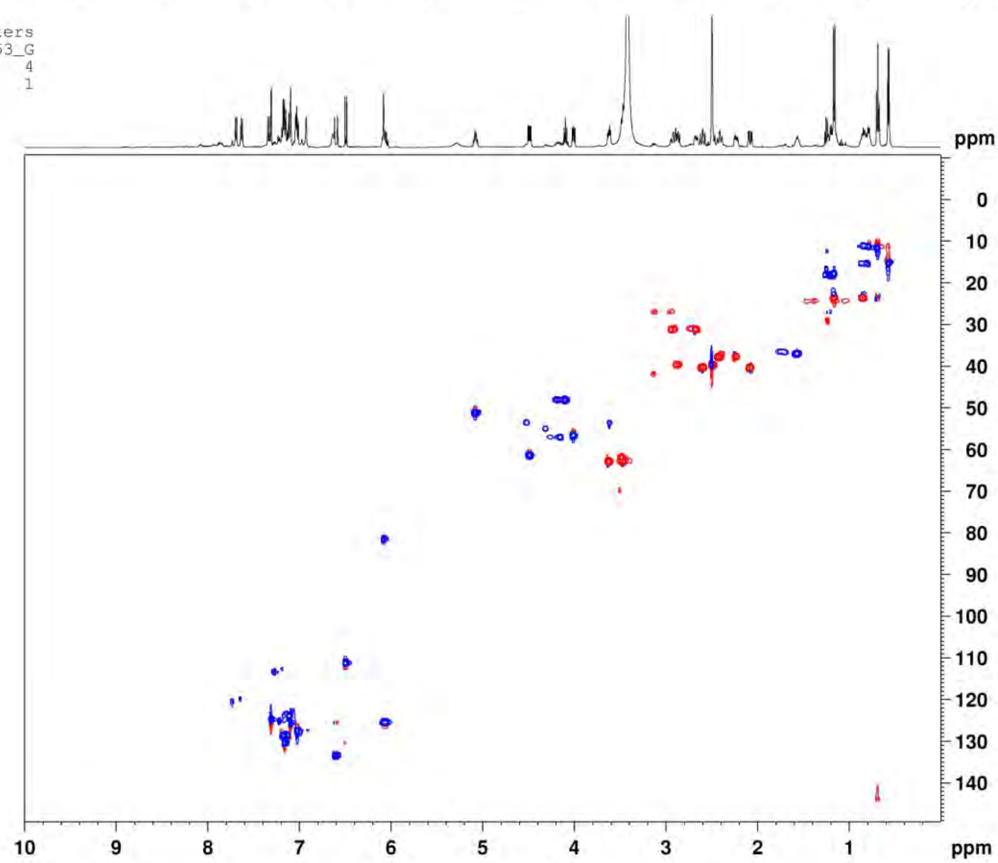




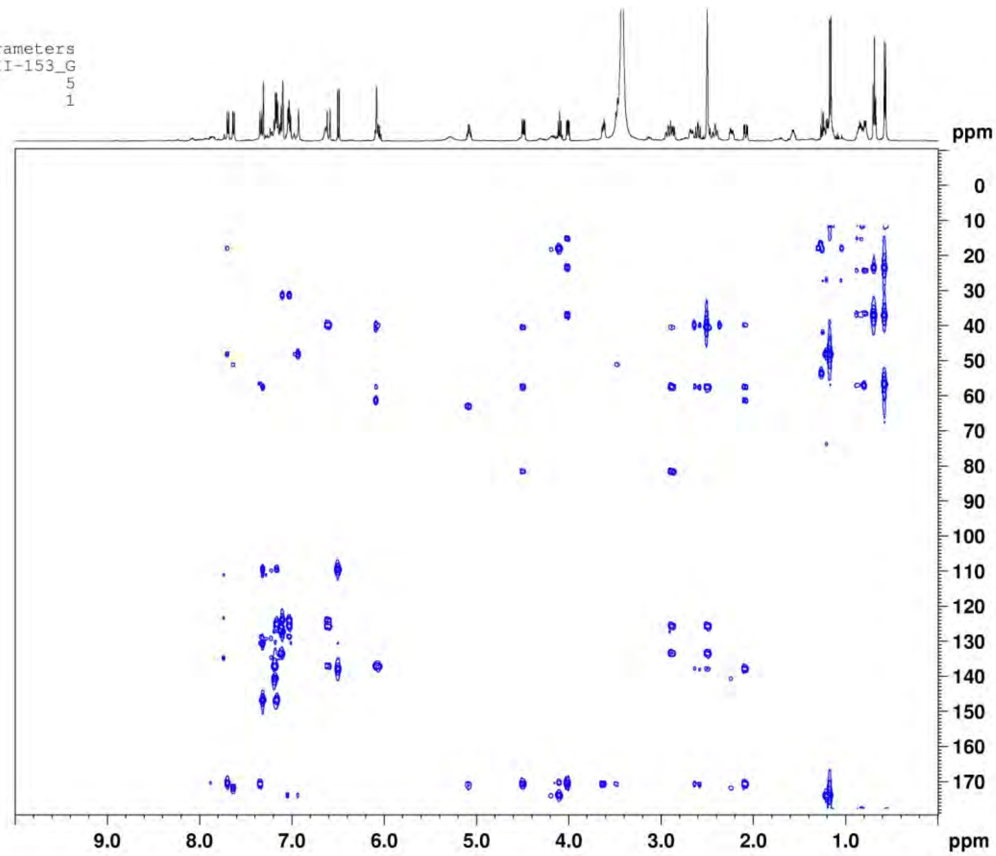
```
Current Data Parameters
NAME        BC-III-153_G
EXPNO                  3
PROCNO                 1
```




```
Current Data Parameters
NAME        BC-III-153_G
EXPNO                  4
PROCNO                 1
```

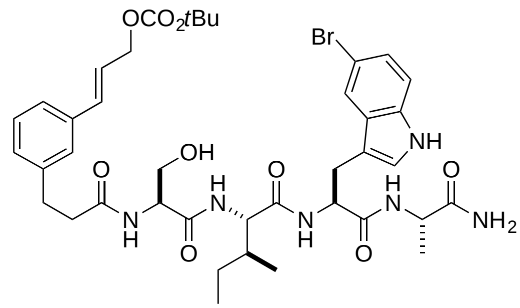


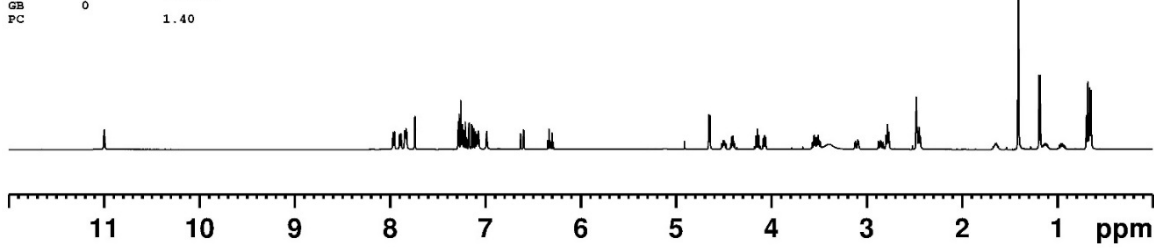
Current Data Parameters
NAME BC-III-153_G
EXPNO 5
PROCNO 1



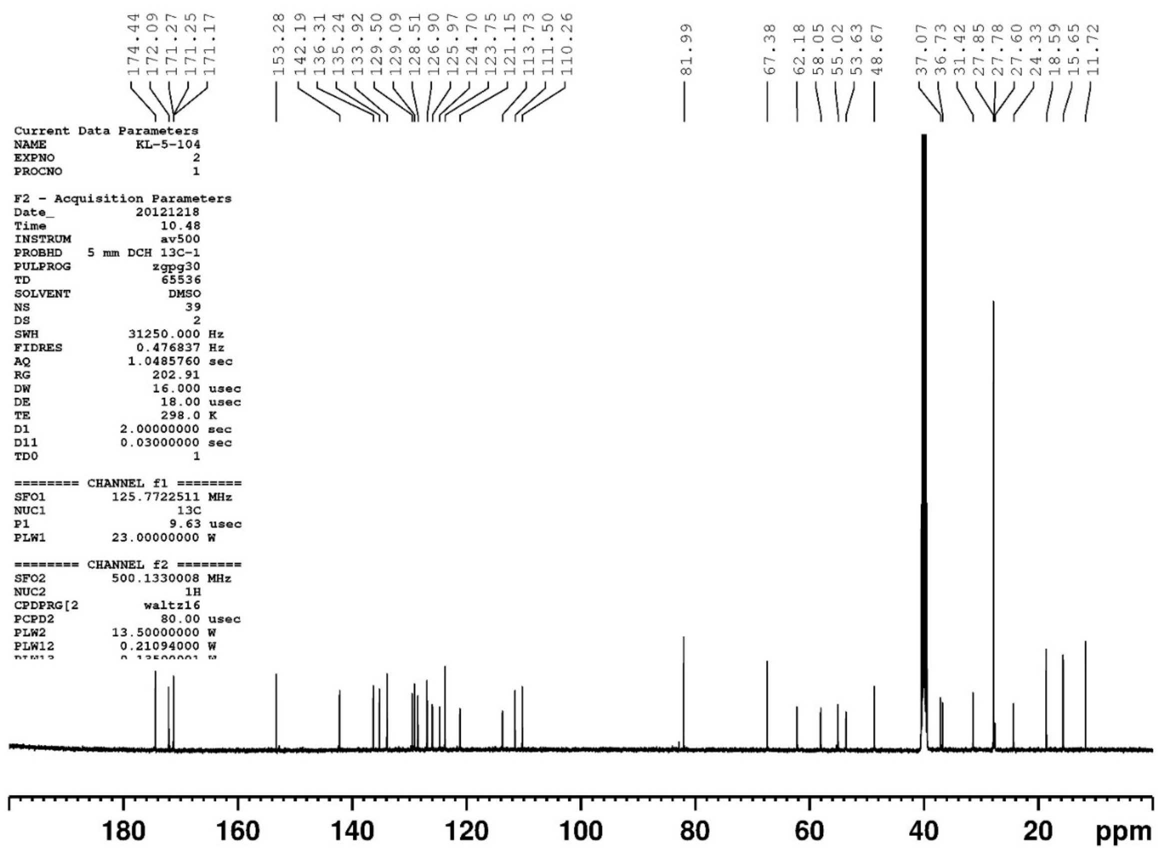
Acyclic Precursor ***14***

```
Current Data Parameters
NAME           KL-5-104
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20121218
Time              10.45
INSTRUM           av500
PROBHD   5 mm DCH 13C-1
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                    8
DS                    0
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                   11
DW               50.000 usec
DE                10.00 usec
TE                298.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330008 MHz
NUC1                 1H
P1                10.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                65536
SF          500.1300146 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.40
```

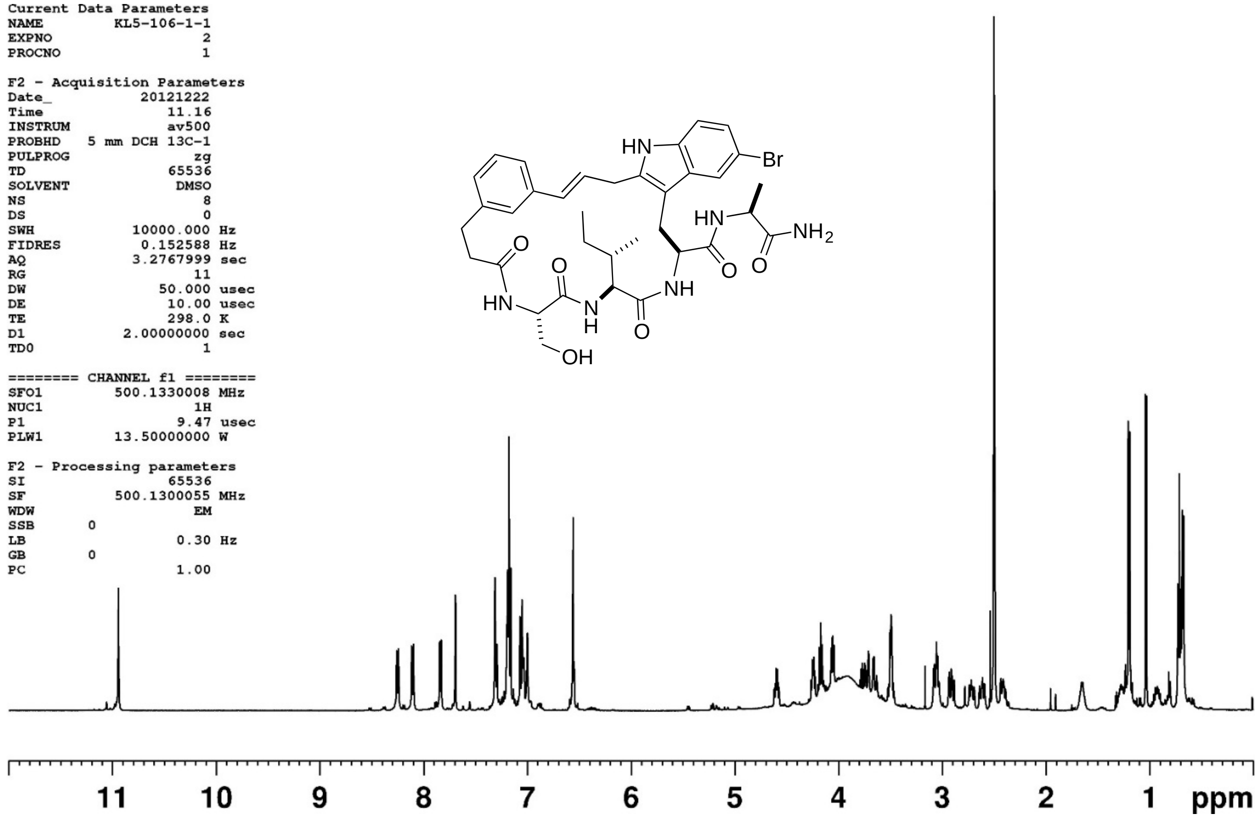


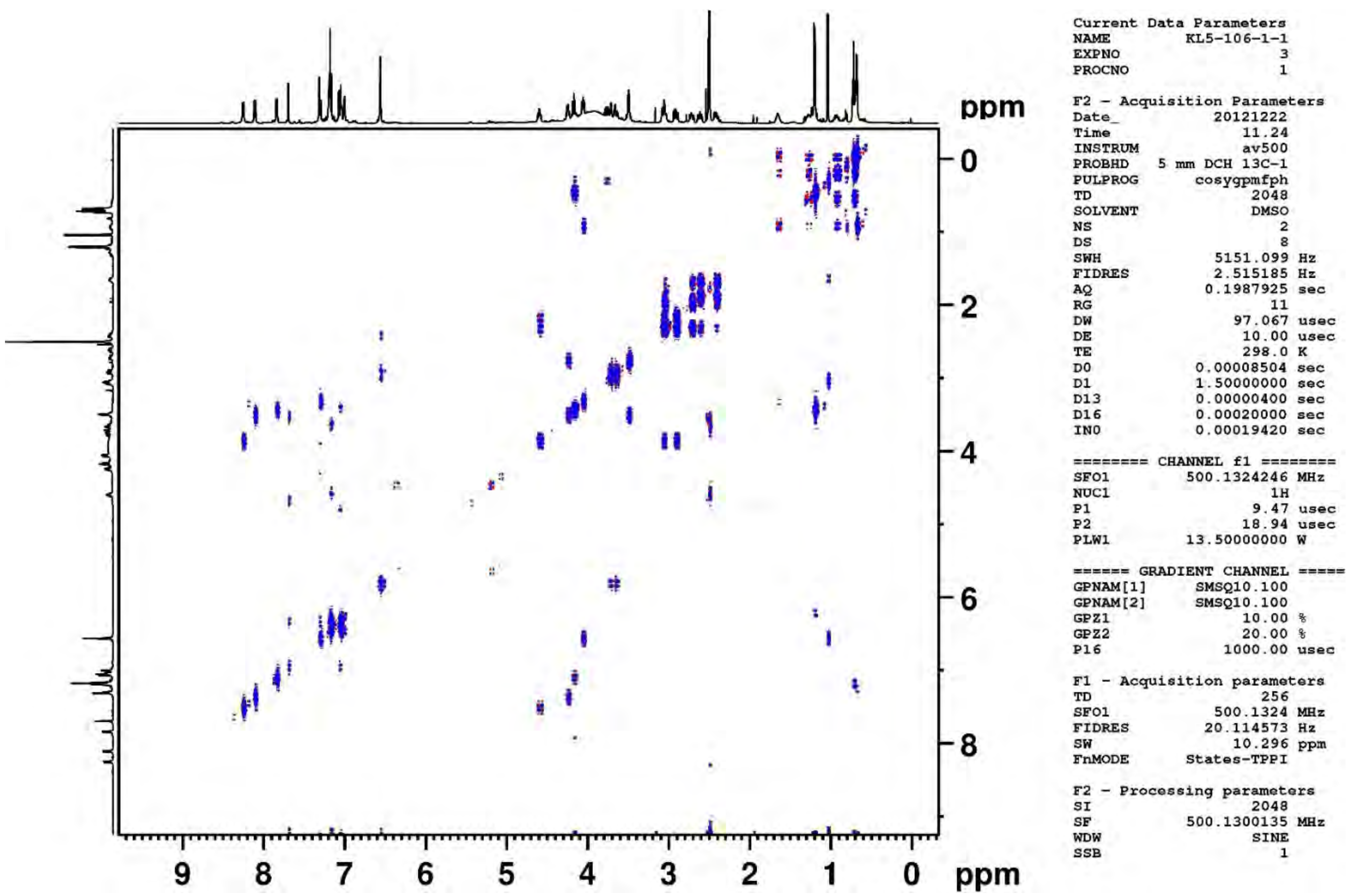


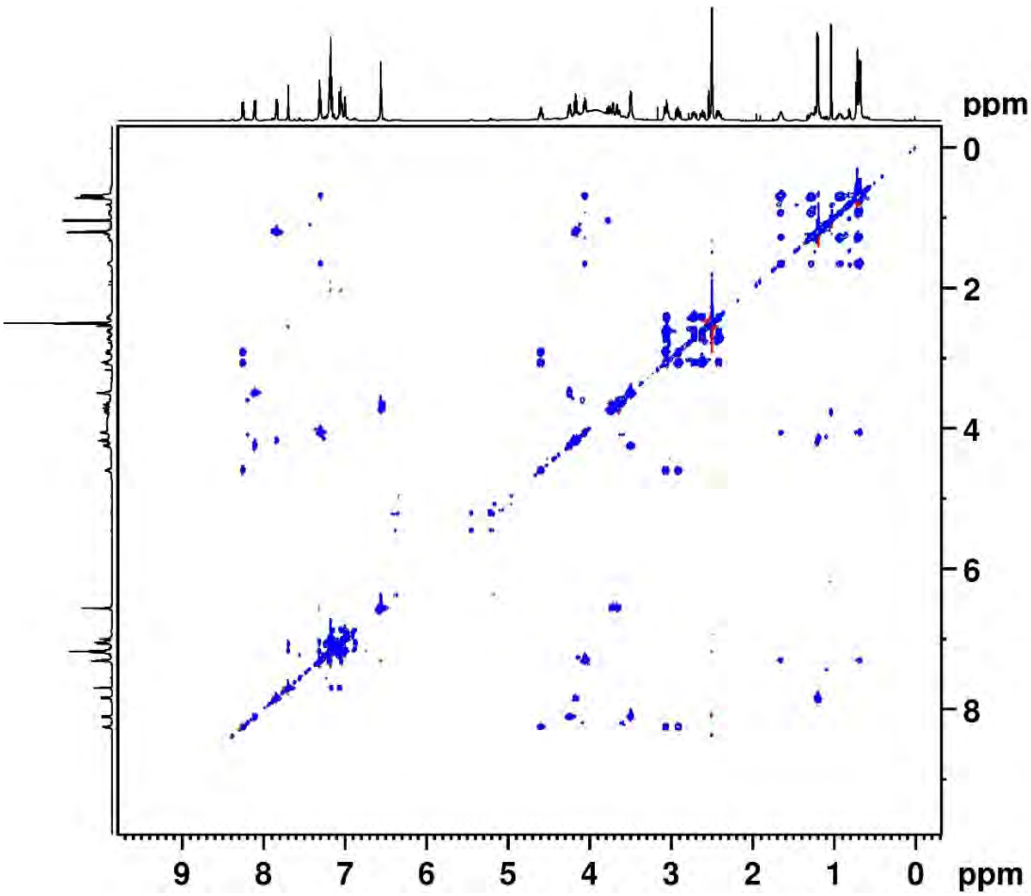

```
Current Data Parameters
NAME           KL-5-104
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20121218
Time              10.48
INSTRUM           av500
PROBHD   5 mm DCH 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                   39
DS                    2
SWH           31250.000 Hz
FIDRES         0.476837 Hz
AQ            1.0485760 sec
RG               202.91
DW               16.000 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7722511 MHz
NUC1                13C
P1                 9.63 usec
PLW1        23.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330008 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2             80.00 usec
PLW2        13.50000000 W
PLW12        0.21094000 W
```



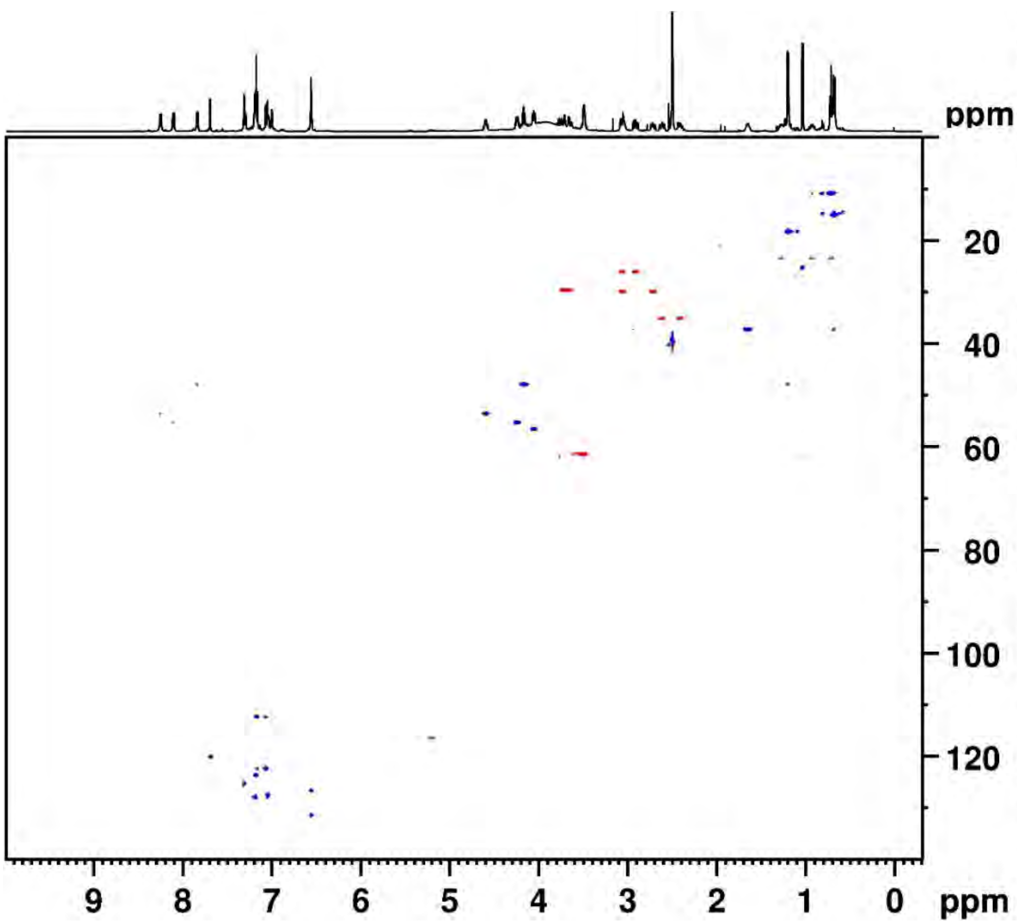
*Macrocyclic Product **18a***



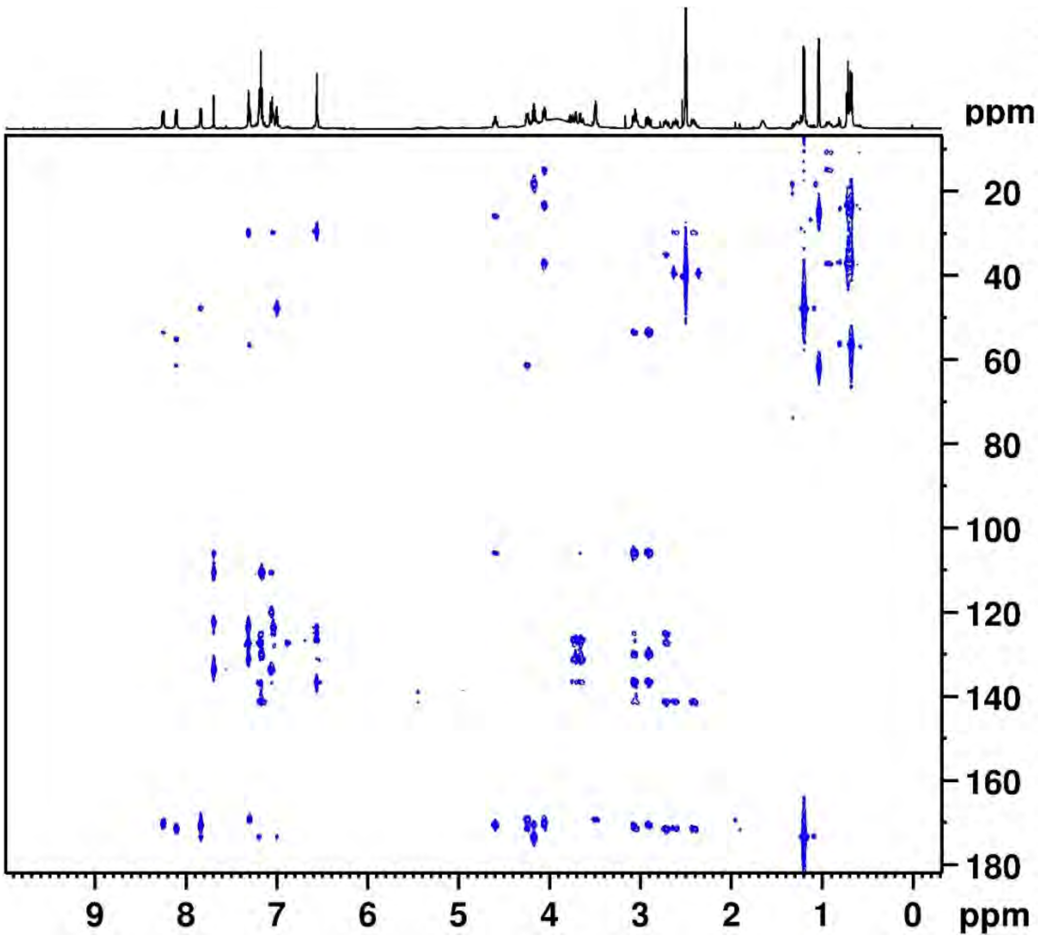





```
Current Data Parameters
NAME          KL5-106-1-1
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121222
Time                11.39
INSTRUM             av500
PROBHD   5 mm DCH 13C-1
PULPROG      mlevetgp.js
TD                   2048
SOLVENT              DMSO
NS                      2
DS                     16
SWH              5151.099 Hz
FIDRES           2.515185 Hz
AQ              0.1987925 sec
RG                 202.91
DW                 97.067 usec
DE                  10.00 usec
TE                  298.0 K
D0             0.00000300 sec
D1             1.50000000 sec
D9             0.06000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00019420 sec
L1                     24

======== CHANNEL f1 ========
SFO1         500.1324246 MHz
NUC1                   1H
P1                   9.47 usec
P2                  18.94 usec
P5                  26.68 usec
P6                  40.00 usec
P7                  80.00 usec
P17               2500.00 usec
PLW1          13.50000000 W
PLW10          0.75683290 W

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPNAM[2]         SINE.100
GPZ1                30.00 %
GPZ2                30.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    256
SFO1             500.1324 MHz
FIDRES          20.114573 Hz
```

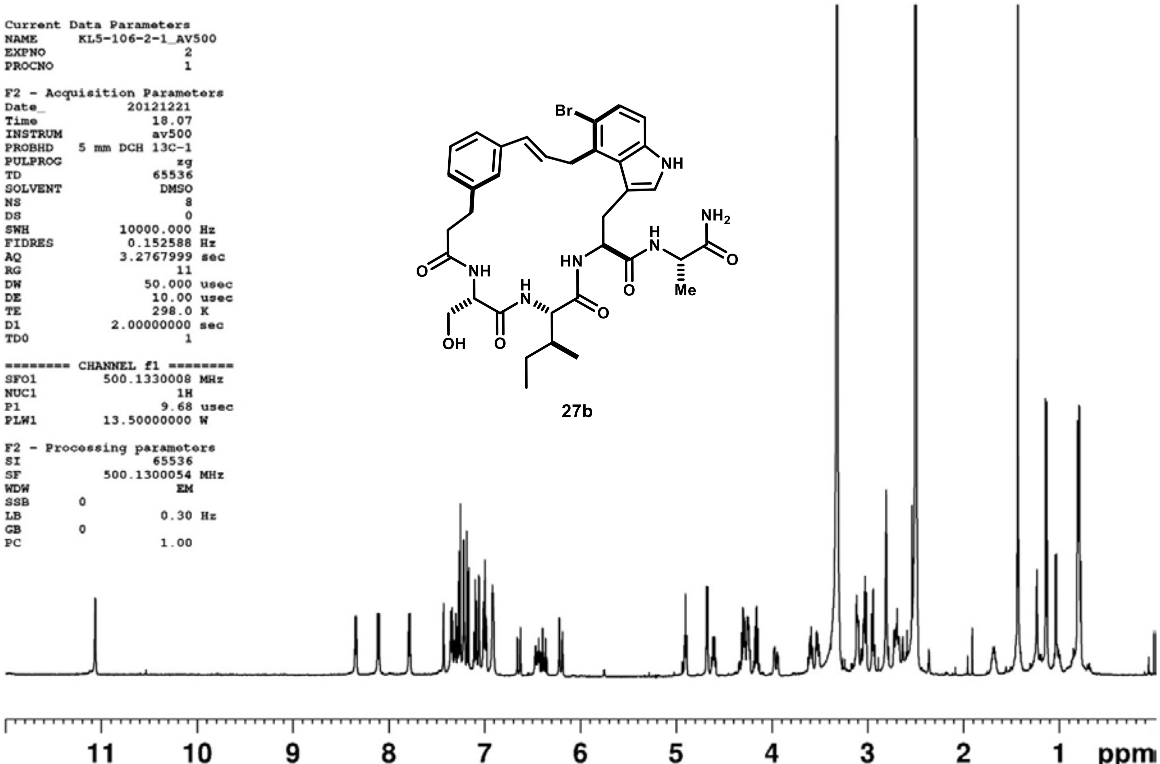


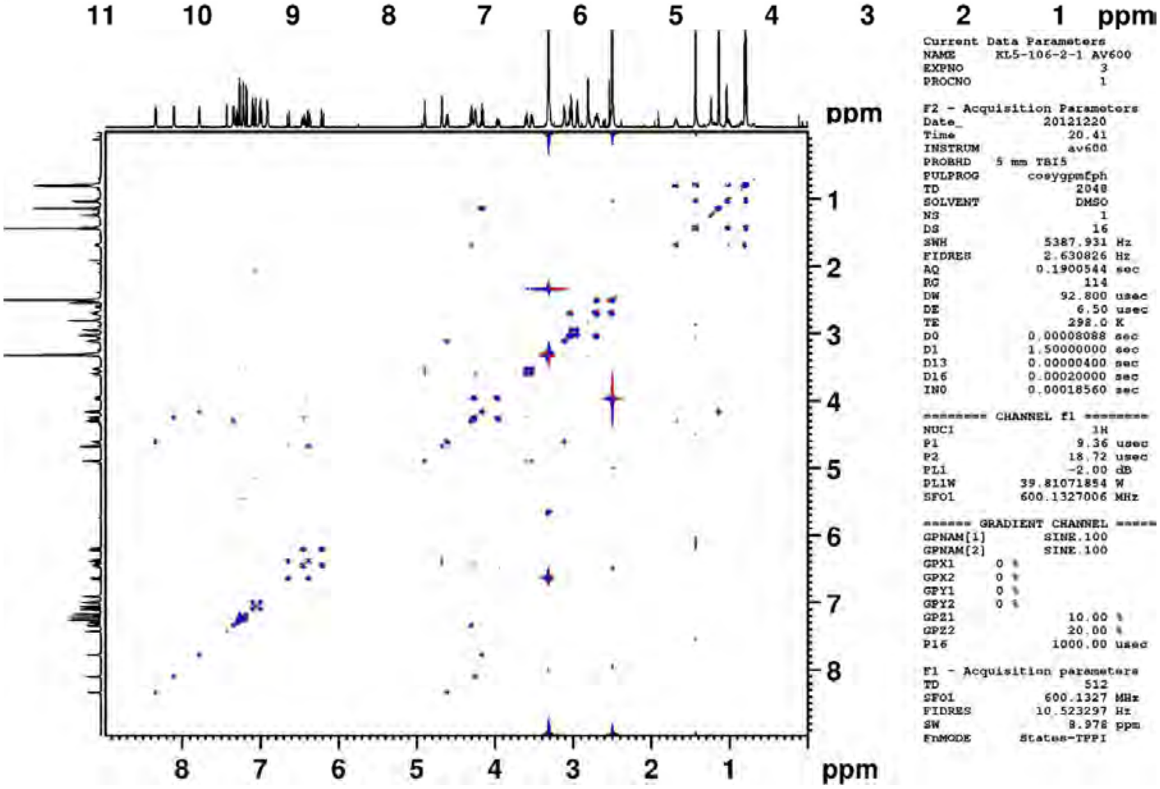

```
Current Data Parameters
NAME          KL5-106-1-1
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121222
Time                11.55
INSTRUM             av500
PROBHD   5 mm DCH 13C-1
PULPROG        hsqcetgpsi
TD                   2048
SOLVENT              DMSO
NS                      6
DS                     16
SWH              5151.099 Hz
FIDRES           2.515185 Hz
AQ              0.1987925 sec
RG                 202.91
DW                 97.067 usec
DE                  10.00 usec
TE                  298.0 K
CNST2         145.0000000
D0             0.00000300 sec
D1             1.00000000 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D24            0.00345000 sec
IN0            0.00002940 sec
ZGOPTNS

======== CHANNEL f1 ========
SFO1         500.1324246 MHz
NUC1                   1H
P1                   9.47 usec
P2                  18.94 usec
P28      0 usec
PLW1          13.50000000 W

======== CHANNEL f2 ========
SFO2         125.7675352 MHz
NUC2                  13C
CPDPRG[2             garp
P3                   9.63 usec
P4                  19.26 usec
PCPD2               70.00 usec
PLW2          23.01399994 W
PLW12          0.43557000 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[2]       SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
```

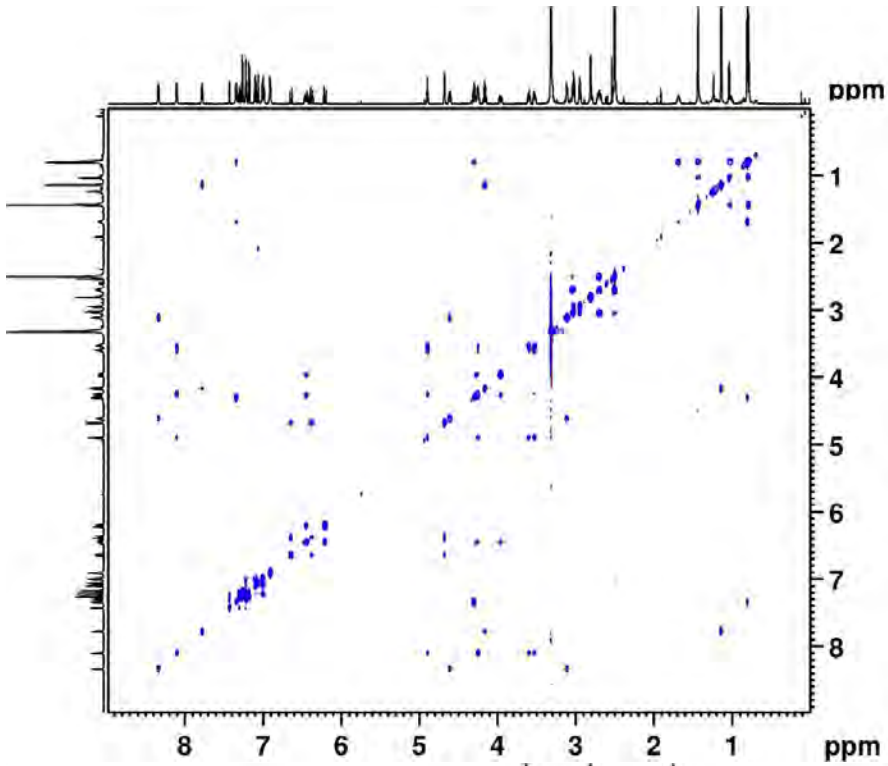



```
Current Data Parameters
NAME            KL5-106-1-1
EXPNO                     6
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121222
Time                  12.59
INSTRUM               av500
PROBHD      5 mm DCH 13C-1
PULPROG         hmbcgpl2ndqf
TD                     2048
SOLVENT                DMSO
NS                       24
DS                       16
SWH                5151.099 Hz
FIDRES             2.515185 Hz
AQ                0.1987925 sec
RG                   202.91
DW                   97.067 usec
DE                    10.00 usec
TE                    298.0 K
CNST6           120.0000000
CNST7           160.0000000
CNST13            7.0000000
D0               0.00000300 sec
D1               1.20000005 sec
D6               0.07142857 sec
D16              0.00020000 sec
IN0              0.00002270 sec

======== CHANNEL f1 ========
SFO1            500.1324246 MHz
NUC1                     1H
P1                     9.47 usec
P2                    18.94 usec
PLW1            13.50000000 W

======== CHANNEL f2 ========
SFO2            125.7697360 MHz
NUC2                    13C
P3                     9.63 usec
PLW2            23.01399994 W

====== GRADIENT CHANNEL =====
GPNAM[1]         SMSQ10.100
GPNAM[2]         SMSQ10.100
GPNAM[3]         SMSQ10.100
GPNAM[4]         SMSQ10.100
GPNAM[5]         SMSQ10.100
GPNAM[6]         SMSQ10.100
GPZ1                  50.00 %
GPZ2                  30.00 %
GPZ3                  40.10 %
GPZ4                  15.00 %
```

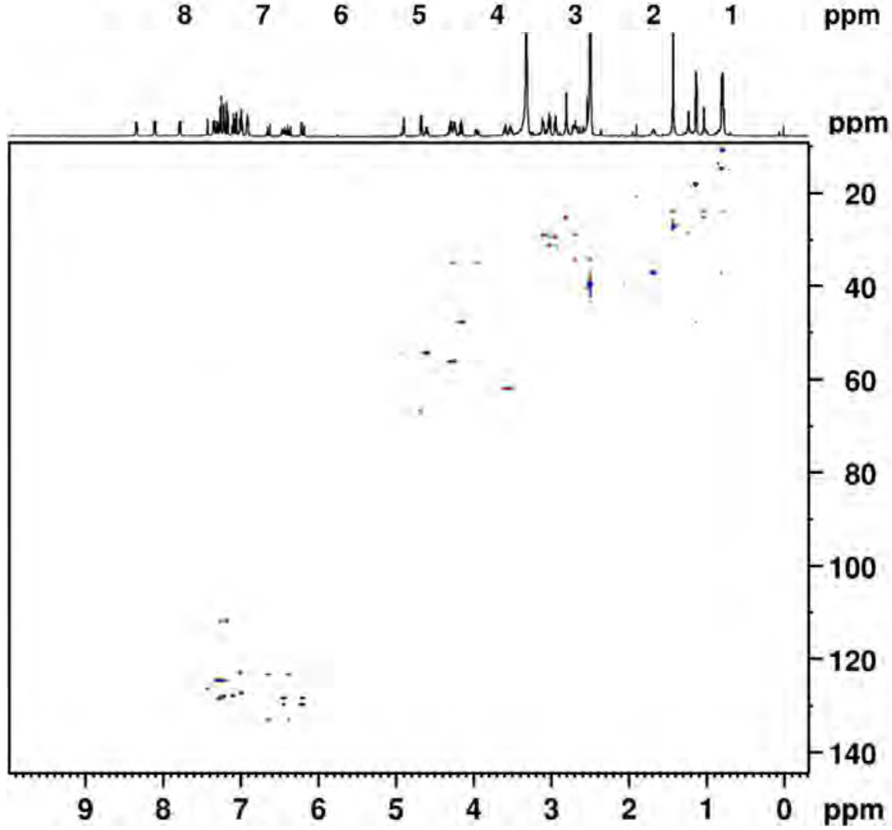
*Macrocyclic Product **18b***



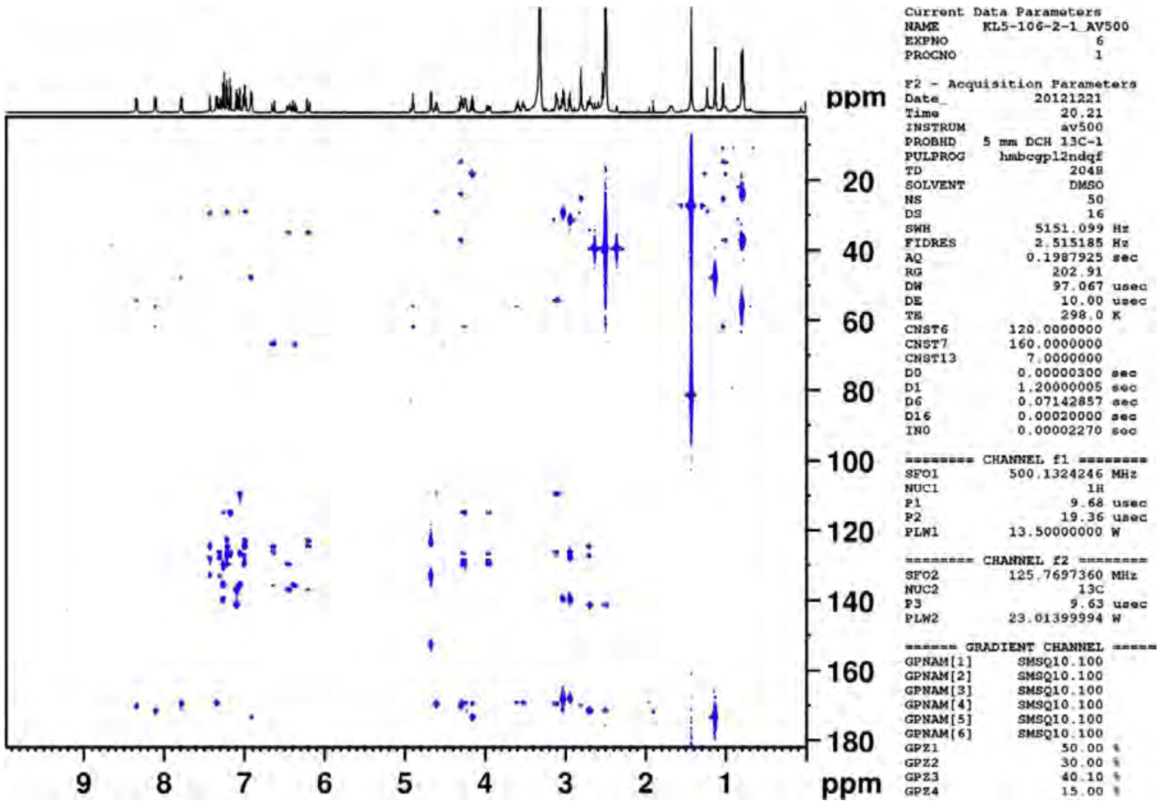




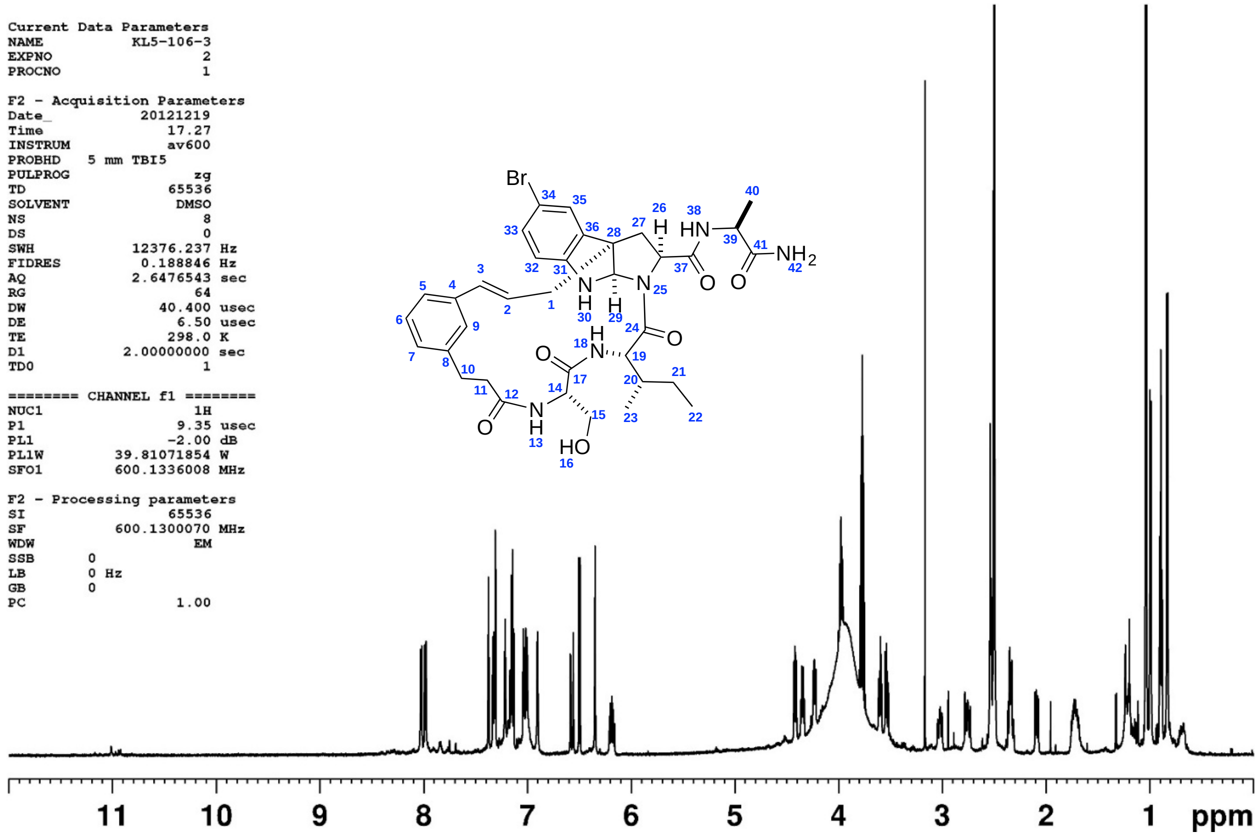

```
Current Data Parameters
NAME      KL5-106-2-1 AV600
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_              20121220
Time                  20.56
INSTRUM               av600
PROBHD    5 mm TBI5
PULPROG         dipsi2etgpsi
TD                     2048
SOLVENT                DMSO
NS                        8
DS                       16
SWH              5387.931 Hz
FIDRES           2.630826 Hz
AQ              0.1900544 sec
RG                      362
DW                   92.800 usec
DE                     6.50 usec
TE                    298.0 K
D0             0.00000300 sec
D1             1.50000000 sec
D9             0.06000000 sec
D11            0.03000000 sec
D16            0.00020000 sec
D20            0.00001000 sec
D21            0.00001000 sec
IN0            0.00018560 sec
L1                       14

======== CHANNEL f1 ========
NUC1                     1H
P1                     9.36 usec
P2                    18.72 usec
P6                    40.00 usec
PL1                   -2.00 dB
PL10                  10.62 dB
PL1W            39.81071854 W
PL10W            2.17770982 W
SFO1           600.1327006 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]           SINE.100
GPNAM[2]           SINE.100
GPX1        0 %
GPX2        0 %
GPY1        0 %
GPY2        0 %
GPZ1                  30.00 %
GPZ2                  30.00 %
P16                 1000.00 usec
```

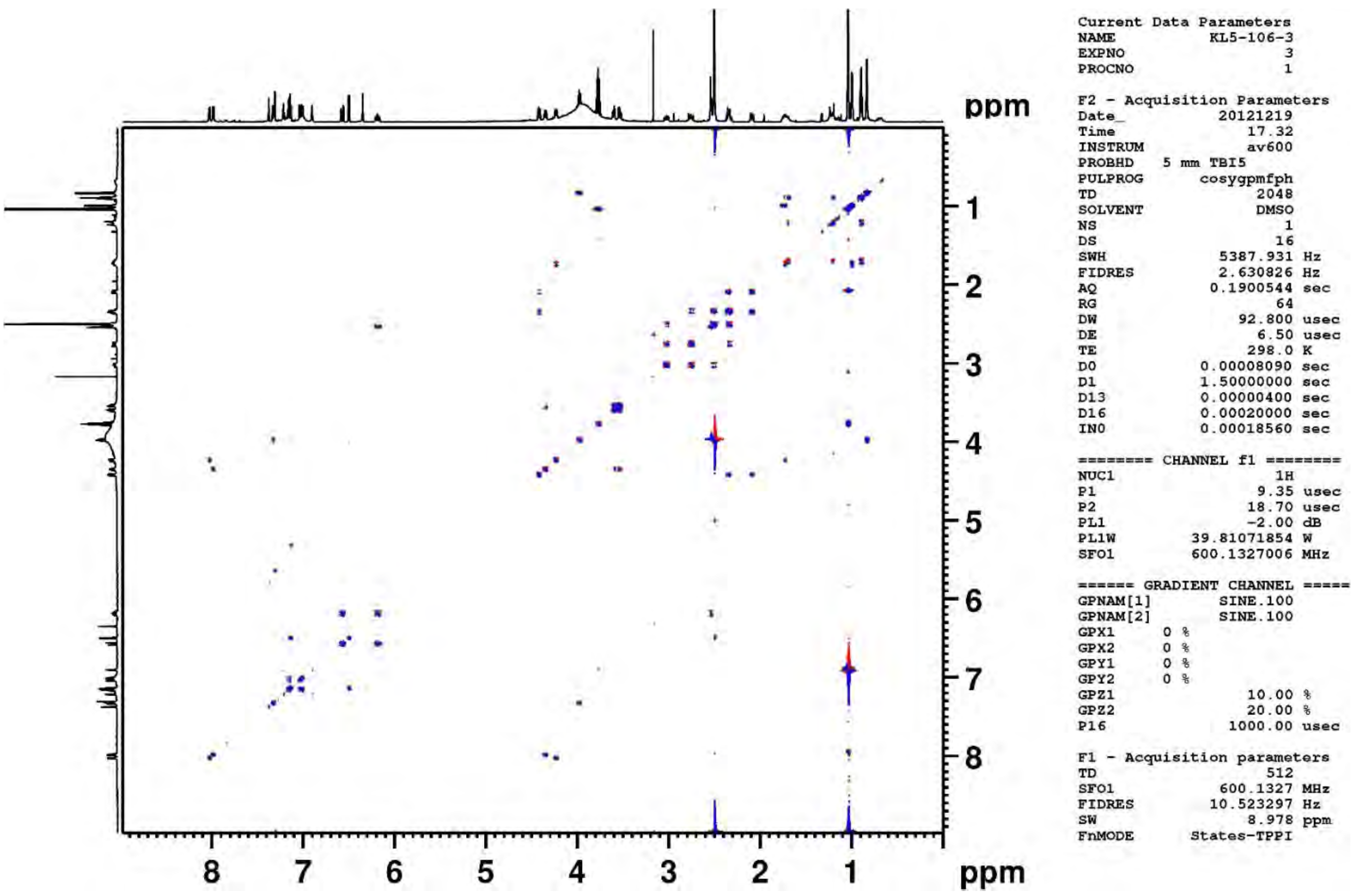


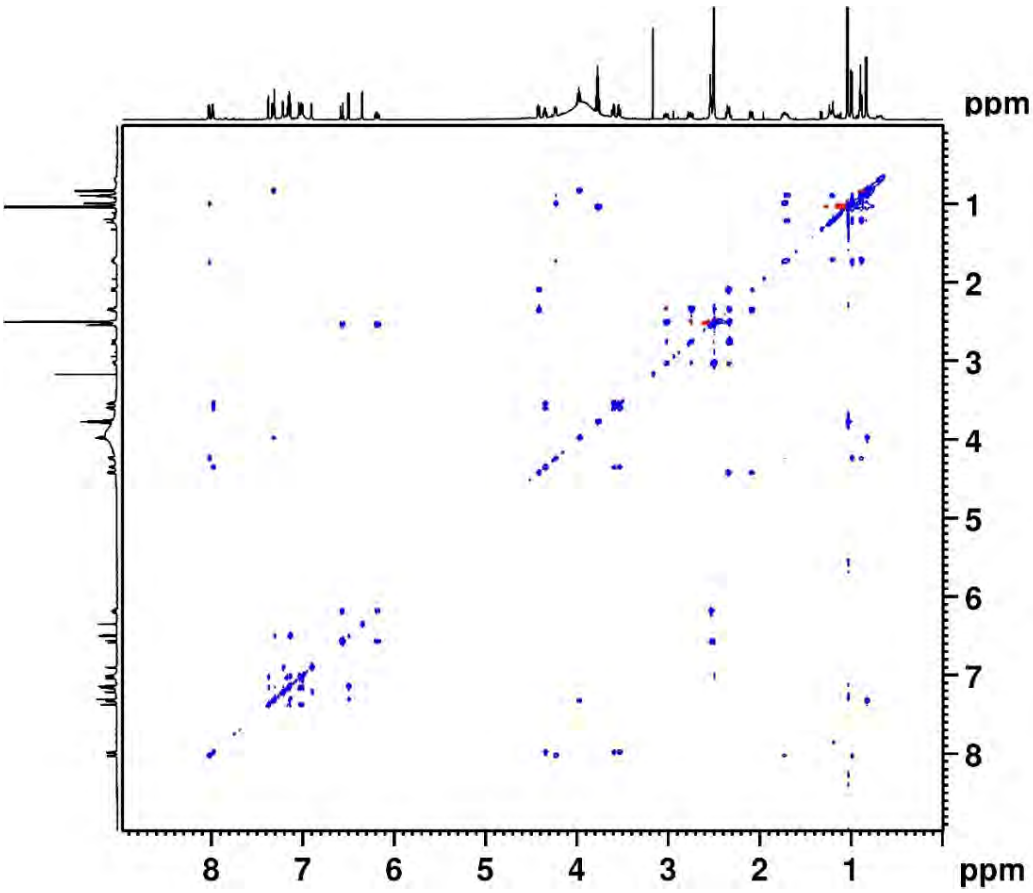

```
Current Data Parameters
NAME      KL5-106-2-1_AV500
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_              20121221
Time                  18.14
INSTRUM               av500
PROBHD    5 mm DCH 13C-1
PULPROG          hsqcetgpsi
TD                     2048
SOLVENT                DMSO
NS                       12
DS                       16
SWH              5151.099 Hz
FIDRES           2.515185 Hz
AQ              0.1987925 sec
RG                   202.91
DW                   97.067 usec
DE                    10.00 usec
TE                    298.0 K
CNST2         145.0000000
D0             0.00000300 sec
D1             1.00000000 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D24            0.00345000 sec
IN0            0.00002940 sec
ZGOPTNS

======== CHANNEL f1 ========
SFO1           500.1324246 MHz
NUC1                     1H
P1                     9.68 usec
P2                    19.36 usec
P28        0 usec
PLW1           13.50000000 W

======== CHANNEL f2 ========
SFO2           125.7675352 MHz
NUC2                    13C
CPDPRG[2                garp
P3                     9.63 usec
P4                    19.26 usec
PCPD2                 70.00 usec
PLW2           23.01399994 W
PLW12           0.43557000 W

====== GRADIENT CHANNEL =====
GPNAM[1]         SMSQ10.100
GPNAM[2]         SMSQ10.100
GPZ1                  80.00 %
GPZ2                  20.10 %
```


ppm
20
40
60
80
100
120
140
160
180
9
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME KL5-106-2-1_AV500
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121221
Time 20.21
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 50
DS 16
SWH 5151.099 Hz
FIDRES 2.515185 Hz
AQ 0.1987925 sec
RG 202.91
DW 97.067 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00002270 sec
======== CHANNEL f1 ========
SFO1 500.1324246 MHz
NUC1 1H
P1 9.68 usec
P2 19.36 usec
PLW1 13.50000000 W
======== CHANNEL f2 ========
SFO2 125.7697360 MHz
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

Macrocyclic Product ***18c***







Current Data Parameters
NAME KL5-106-3
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121219
Time 17.48
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG dipsi2etgpsi
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 5387.931 Hz
FIDRES 2.630826 Hz
AQ 0.1900544 sec
RG 362
DW 92.800 usec
DE 6.50 usec
TE 298.0 K
D0 0.00000300 sec
D1 1.50000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00001000 sec
D21 0.00001000 sec
IN0 0.00018560 sec
L1 14

======== CHANNEL f1 ========
NUC1 1H
P1 9.35 usec
P2 18.70 usec
P6 40.00 usec
PL1 -2.00 dB
PL10 10.62 dB
PL1W 39.81071854 W
PL10W 2.17770982 W
SFO1 600.1327006 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec

Current Data Parameters
NAME KL-5-106-3 (02-2014)
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140218
Time 21.13
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG noesygpph
TD 2048
SOLVENT DMSO
NS 16
DS 16
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425908 sec
RG 161.3
DW 69.600 usec
DE 6.50 usec
TE 680.6 K
D0 0.00005674 sec
D1 2.00000000 sec
D8 0.20000000 sec
D16 0.00020000 sec
IN0 0.00013920 sec

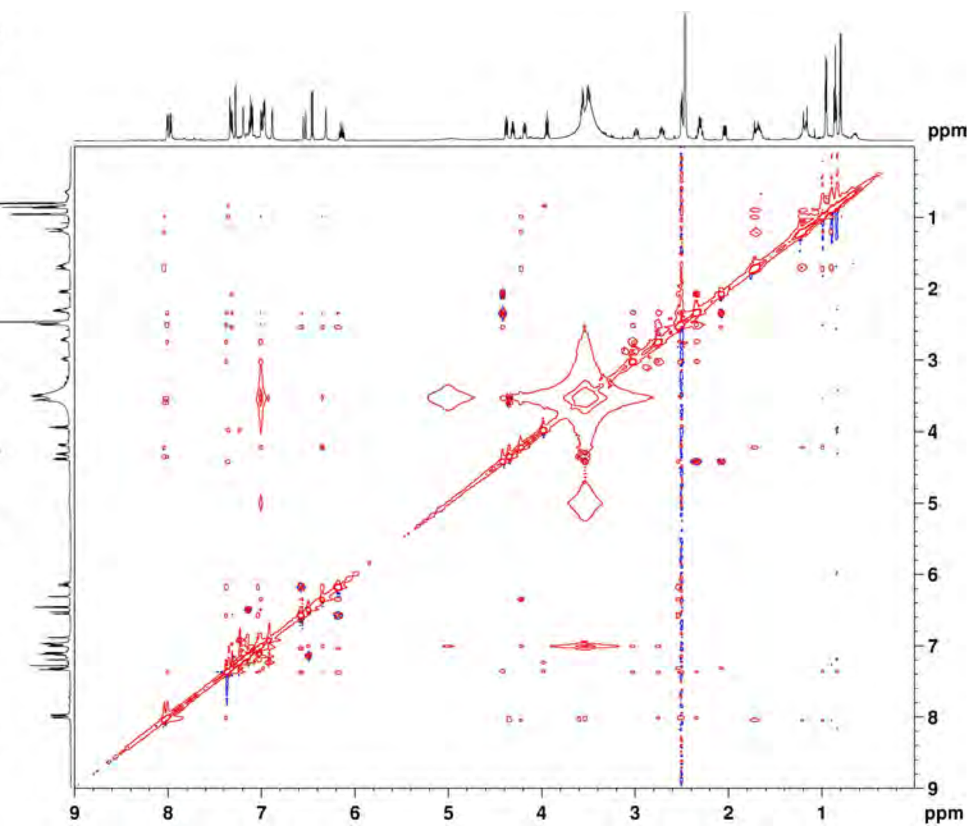
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
P2 20.20 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1336008 MHz

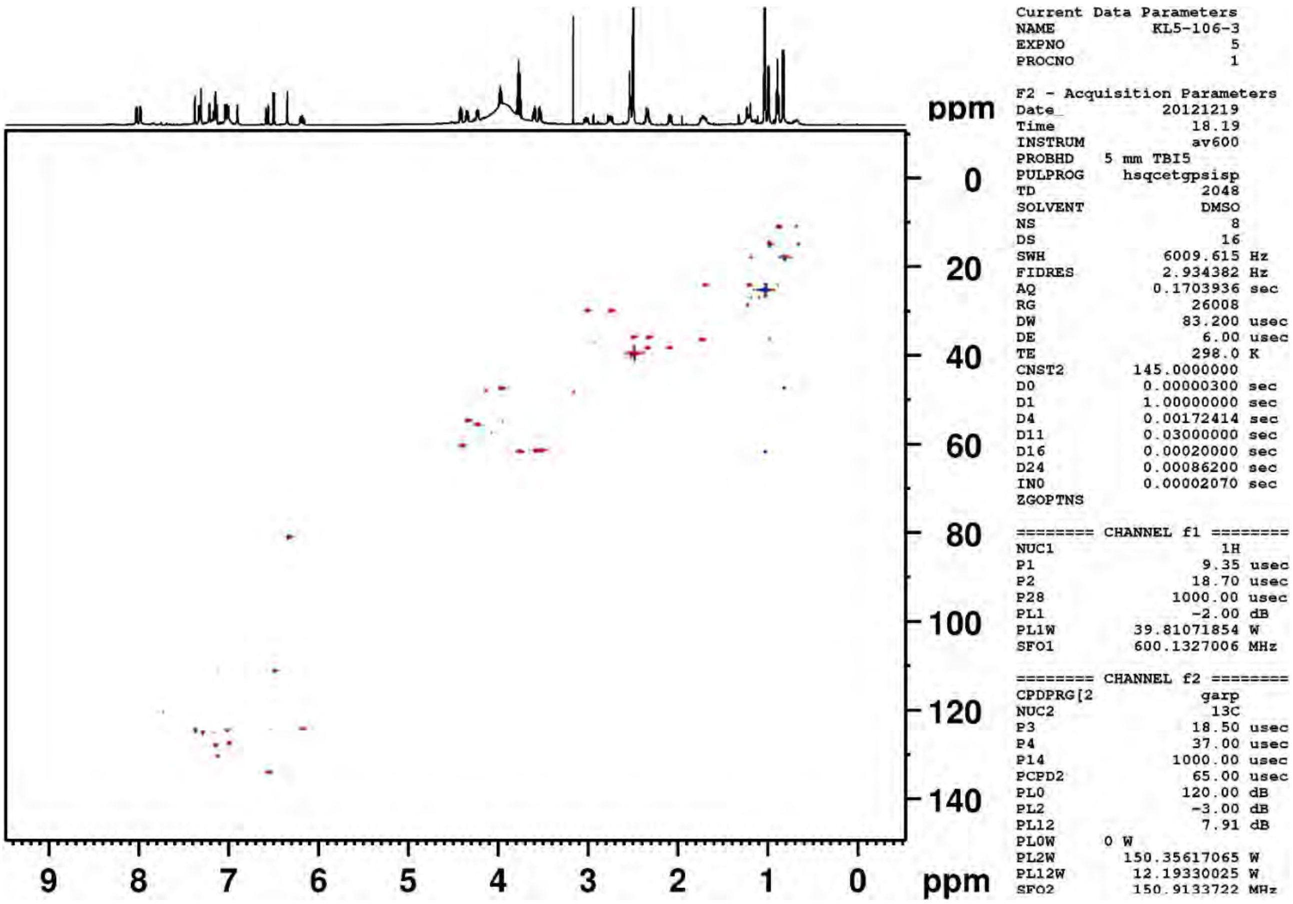
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPX1 0 %
GPY1 0 %
GPZ1 40.00 %
P16 1000.00 usec

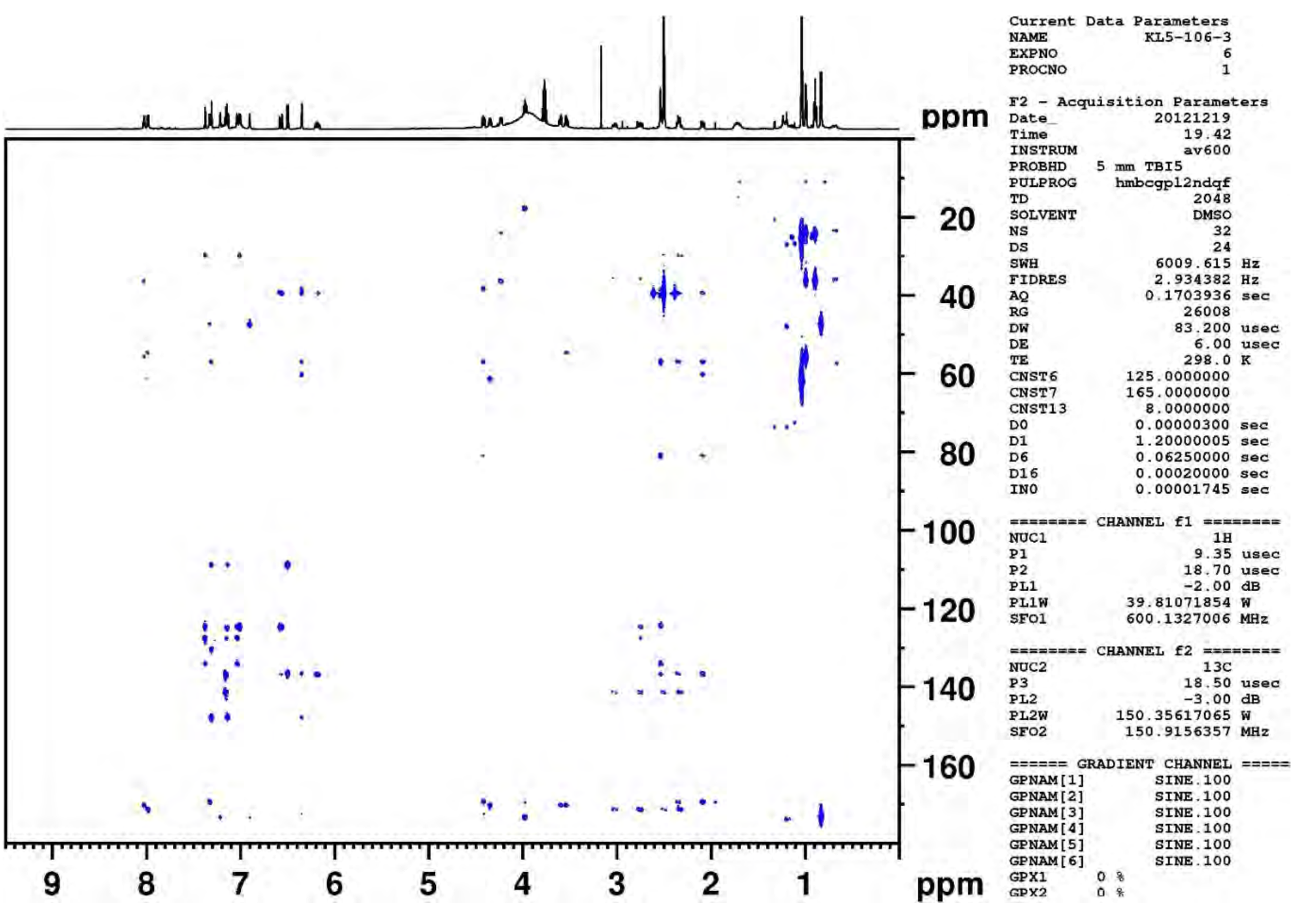
F1 - Acquisition parameters
TD 512
SFO1 600.1336 MHz
FIDRES 14.031089 Hz
SW 11.971 ppm
FnMODE States-TPPI

F2 - Processing parameters
SI 1024
SF 600.1300038 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

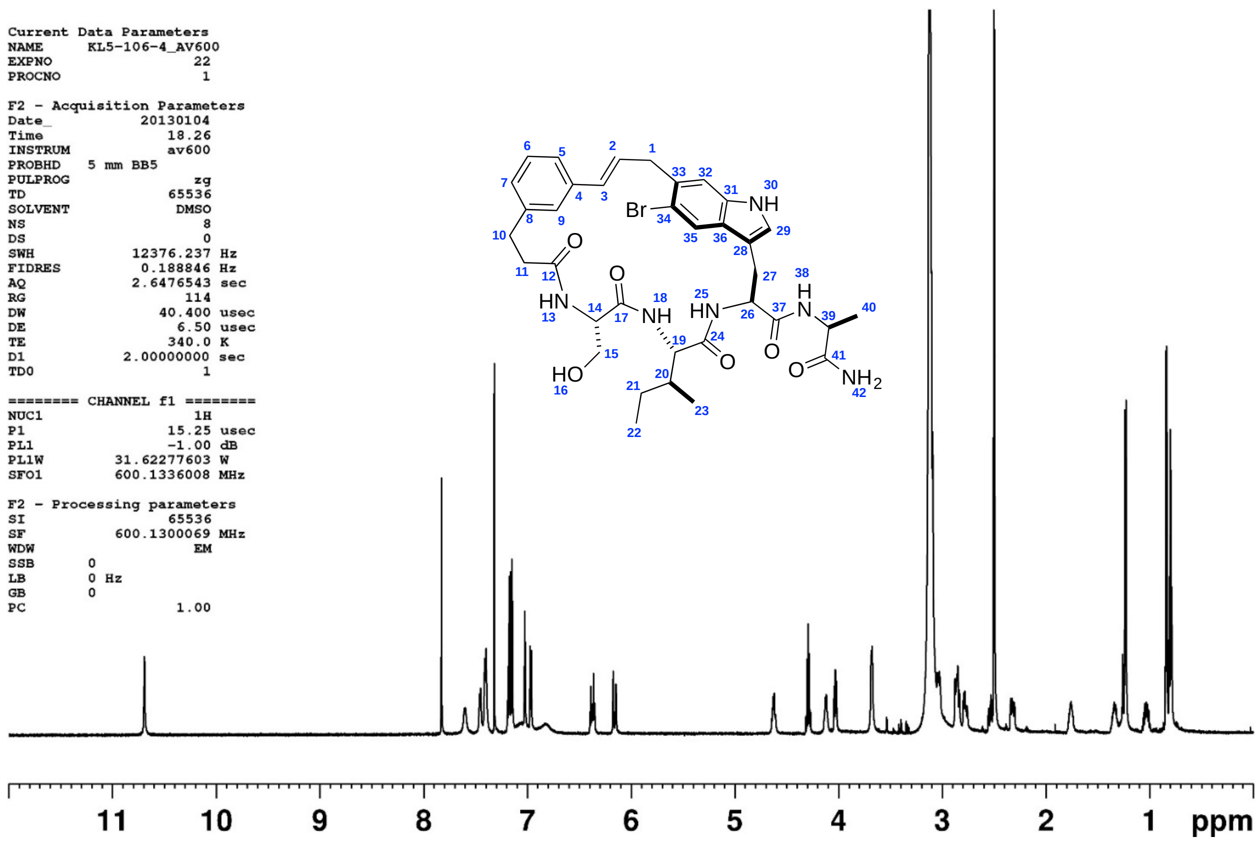
F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 600.1300037 MHz
WDW
SSB 2
LB 0 Hz
GB 0

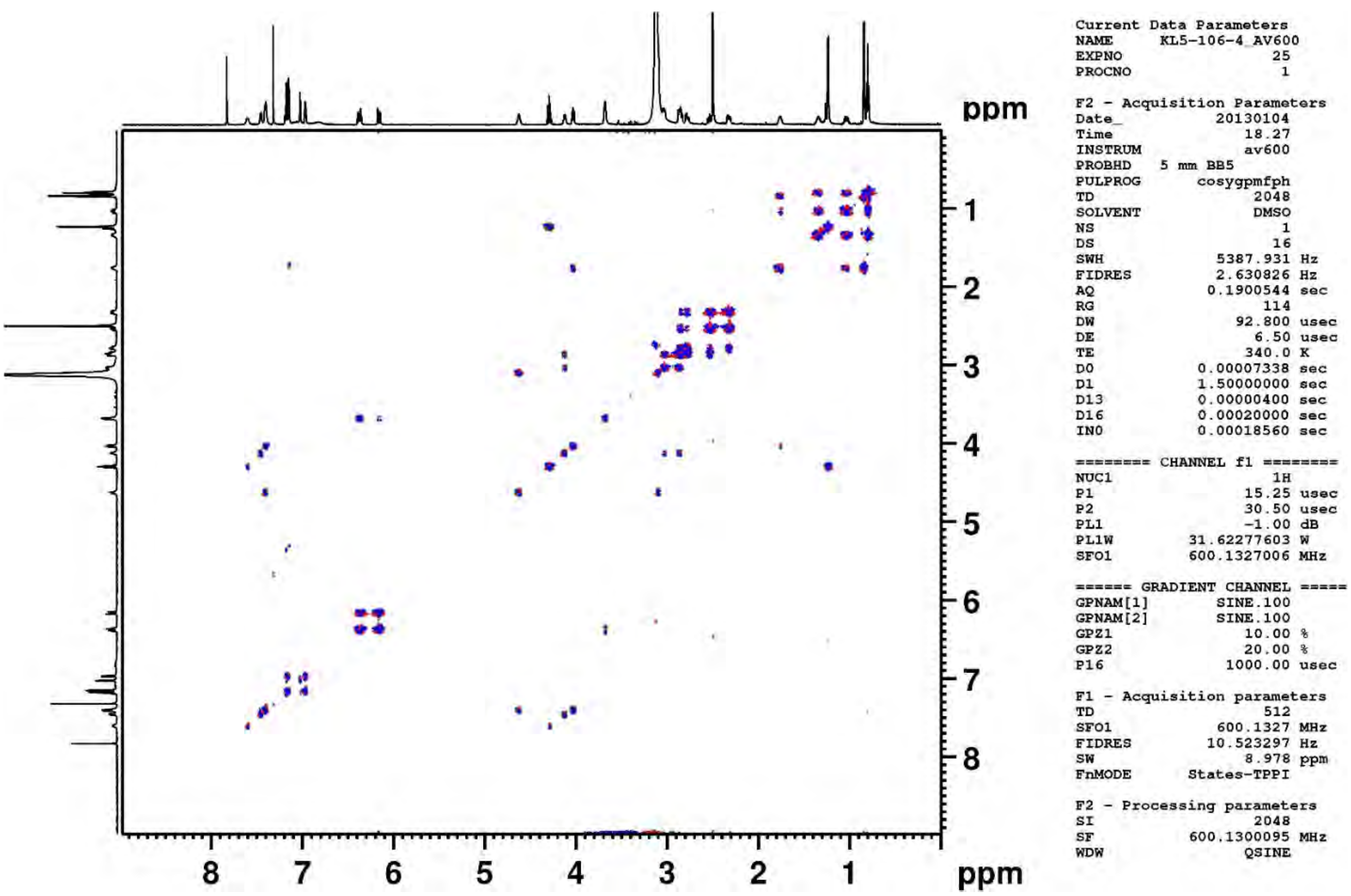


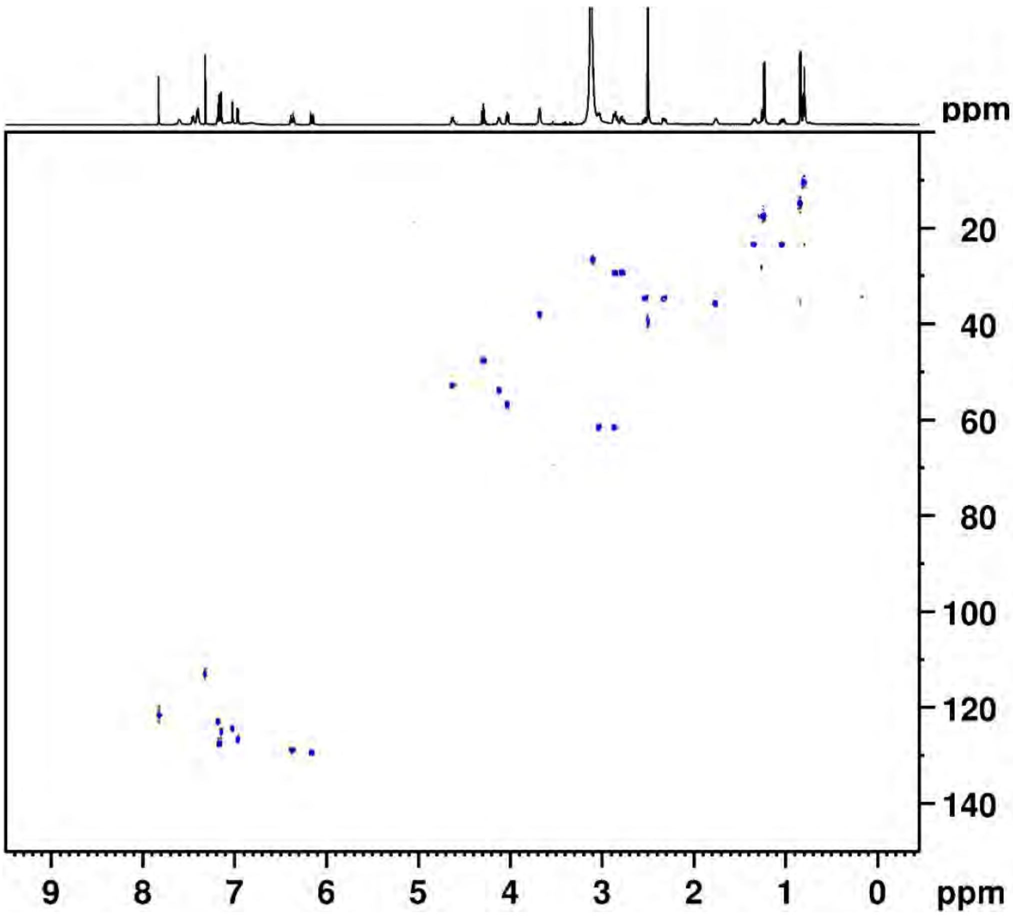

ppm
0
20
40
60
80
100
120
140
9 8 7 6 5 4 3 2 1 0 ppm
Current Data Parameters
NAME KL5-106-3
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121219
Time 18.19
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 26008
DW 83.200 usec
DE 6.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.00000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 9.35 usec
P2 18.70 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1327006 MHz
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 18.50 usec
P4 37.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.91 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 12.19330025 W
SFO2 150.9133722 MHz


ppm
20
40
60
80
100
120
140
160
9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME KL5-106-3
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121219
Time 19.42
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 32
DS 24
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 26008
DW 83.200 usec
DE 6.00 usec
TE 298.0 K
CNST6 125.0000000
CNST7 165.0000000
CNST13 8.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D6 0.06250000 sec
D16 0.00020000 sec
IN0 0.00001745 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.35 usec
P2 18.70 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1327006 MHz
======== CHANNEL f2 ========
NUC2 13C
P3 18.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPNAM[4] SINE.100
GPNAM[5] SINE.100
GPNAM[6] SINE.100
GPX1 0 %
GPX2 0 %

Macrocyclic Product ***18d***





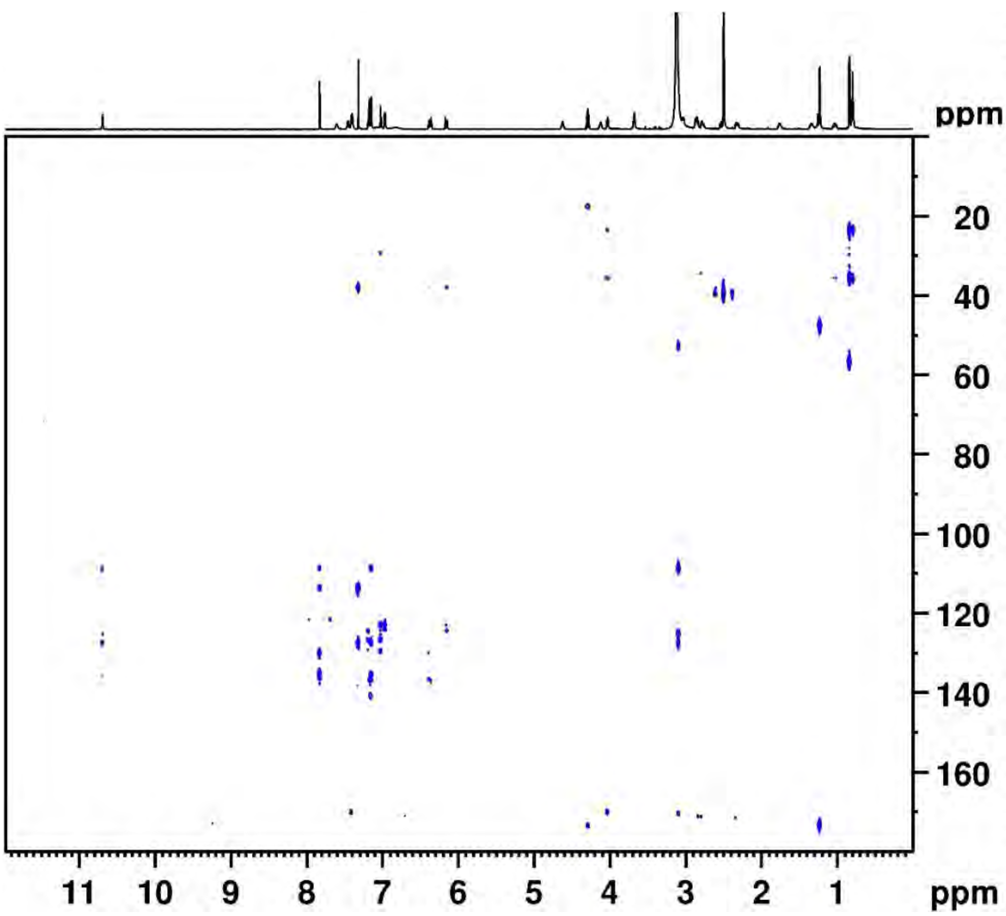


Current Data Parameters
NAME KL5-106-4_AV600
EXPNO 27
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130104
Time 18.44
INSTRUM av600
PROBHD 5 mm BB5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 6
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 26008
DW 83.200 usec
DE 6.00 usec
TE 340.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.00000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1 1H
P1 15.25 usec
P2 30.50 usec
P28 0 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1327006 MHz

======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 9.75 usec
P4 19.50 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 0 dB
PL12 16.48 dB
PL0W 0 W
PL2W 75.35659027 W
PL12W 1.69481111 W
SFO2 150.9133722 MHz



Current Data Parameters
NAME KL5-106-4_AV600
EXPNO 28
PROCNO 1

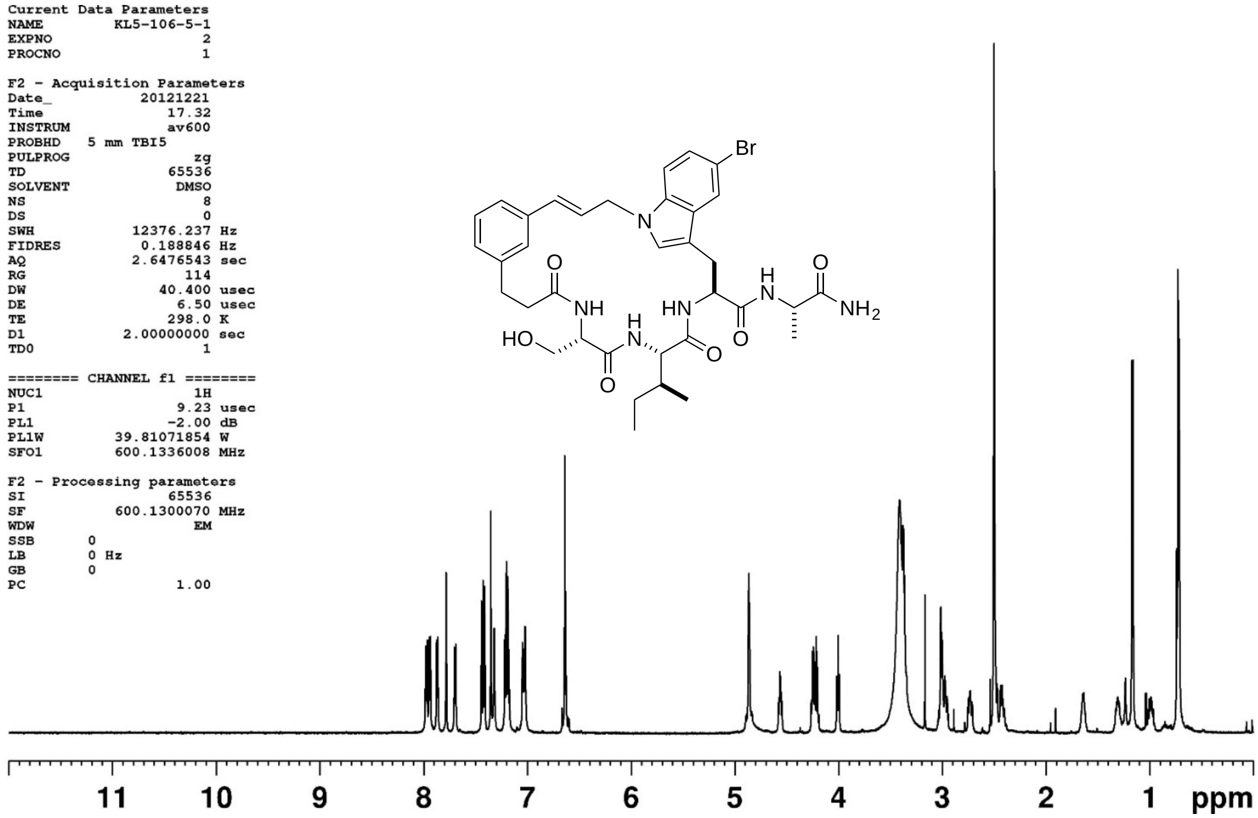
F2 - Acquisition Parameters
Date_ 20130104
Time 19.06
INSTRUM av600
PROBHD 5 mm BB5
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 32
DS 24
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425408 sec
RG 26008
DW 69.600 usec
DE 6.00 usec
TE 340.0 K
CNST6 125.0000000
CNST7 165.0000000
CNST13 8.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D6 0.06250000 sec
D16 0.00020000 sec
IN0 0.00001745 sec

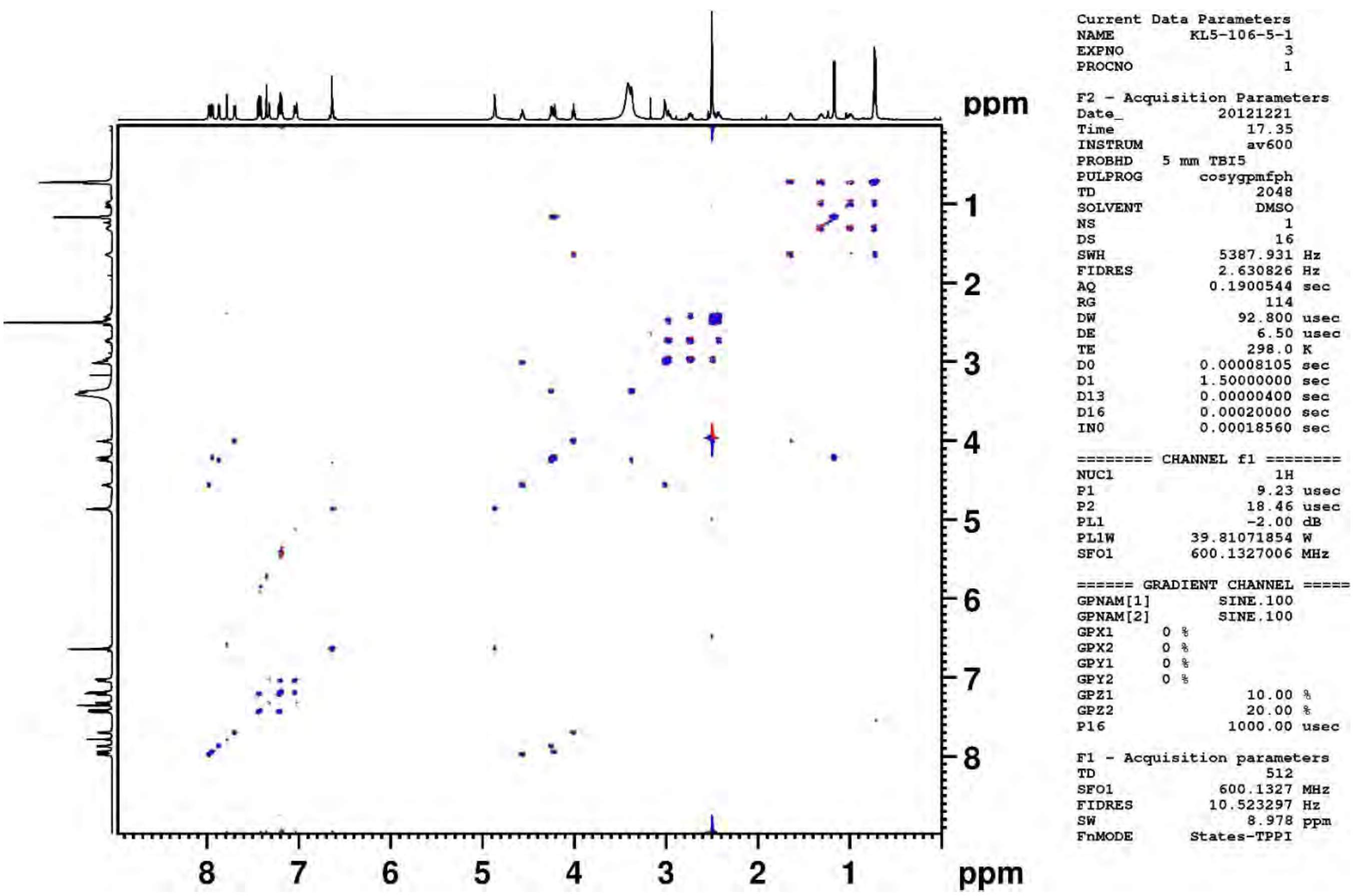
======== CHANNEL f1 ========
NUC1 1H
P1 15.25 usec
P2 30.50 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1336008 MHz

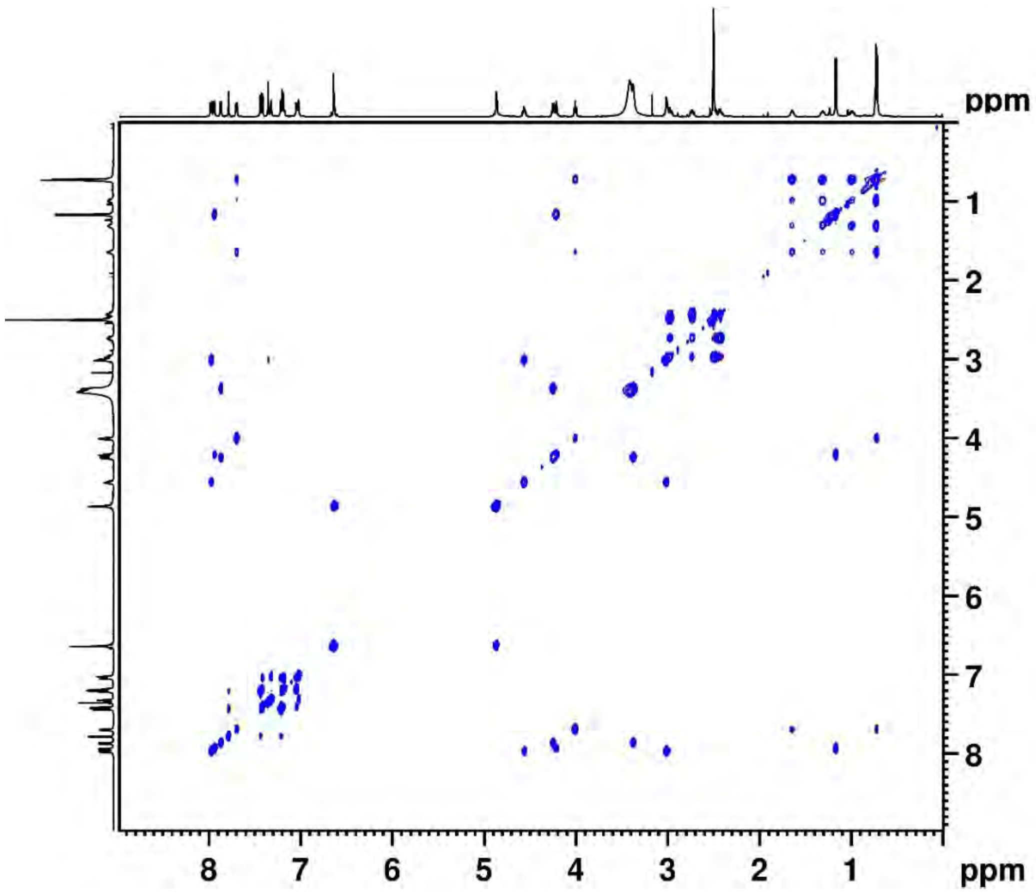
======== CHANNEL f2 ========
NUC2 13C
P3 9.75 usec
PL2 0 dB
PL2W 75.35659027 W
SFO2 150.9156357 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPNAM[4] SINE.100
GPNAM[5] SINE.100
GPNAM[6] SINE.100
GPZ1 50.00 %
GPZ2 30.00 %

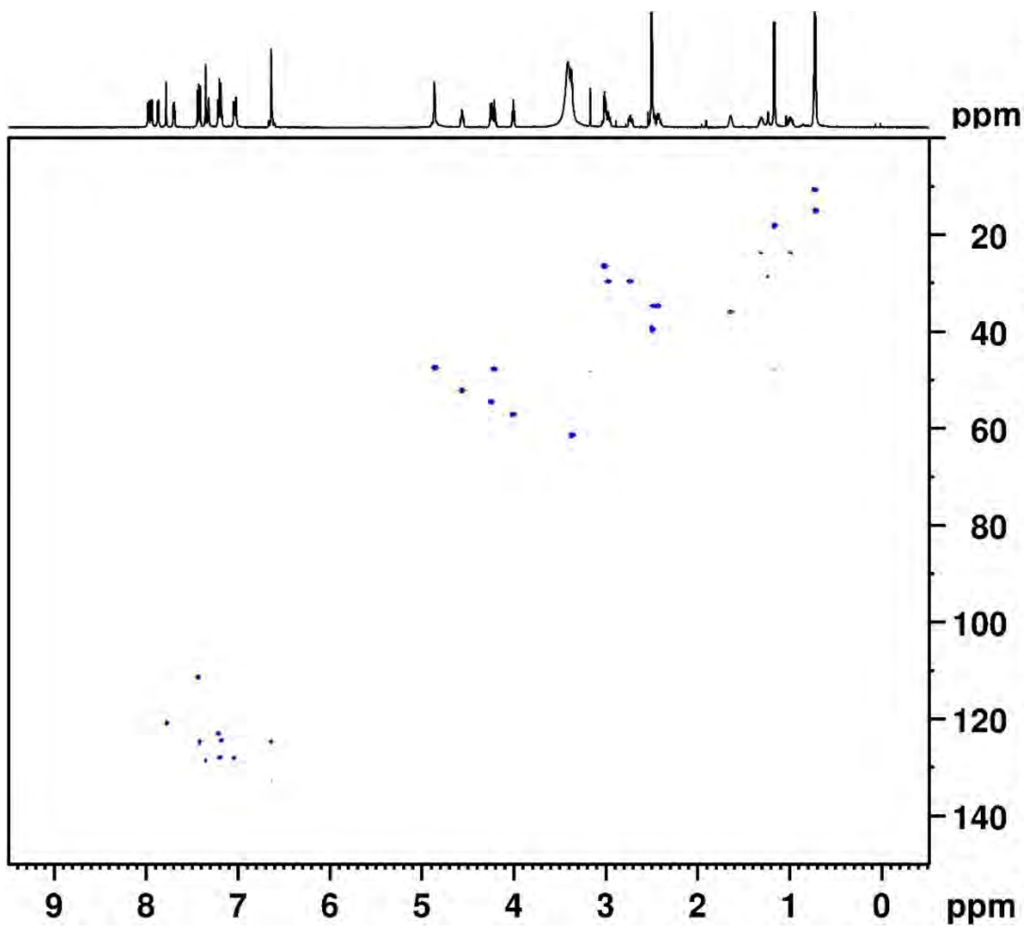
Macrocyclic Product ***18e***





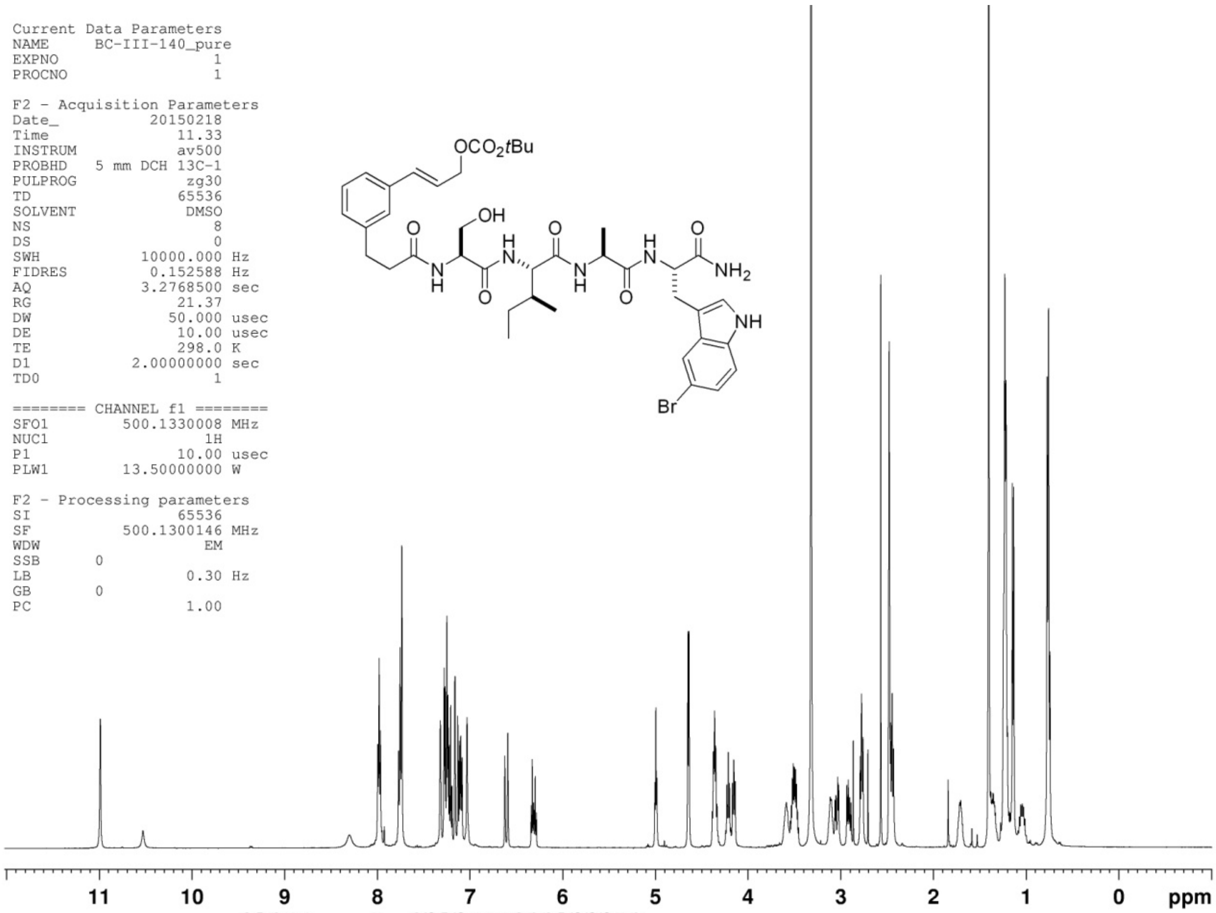


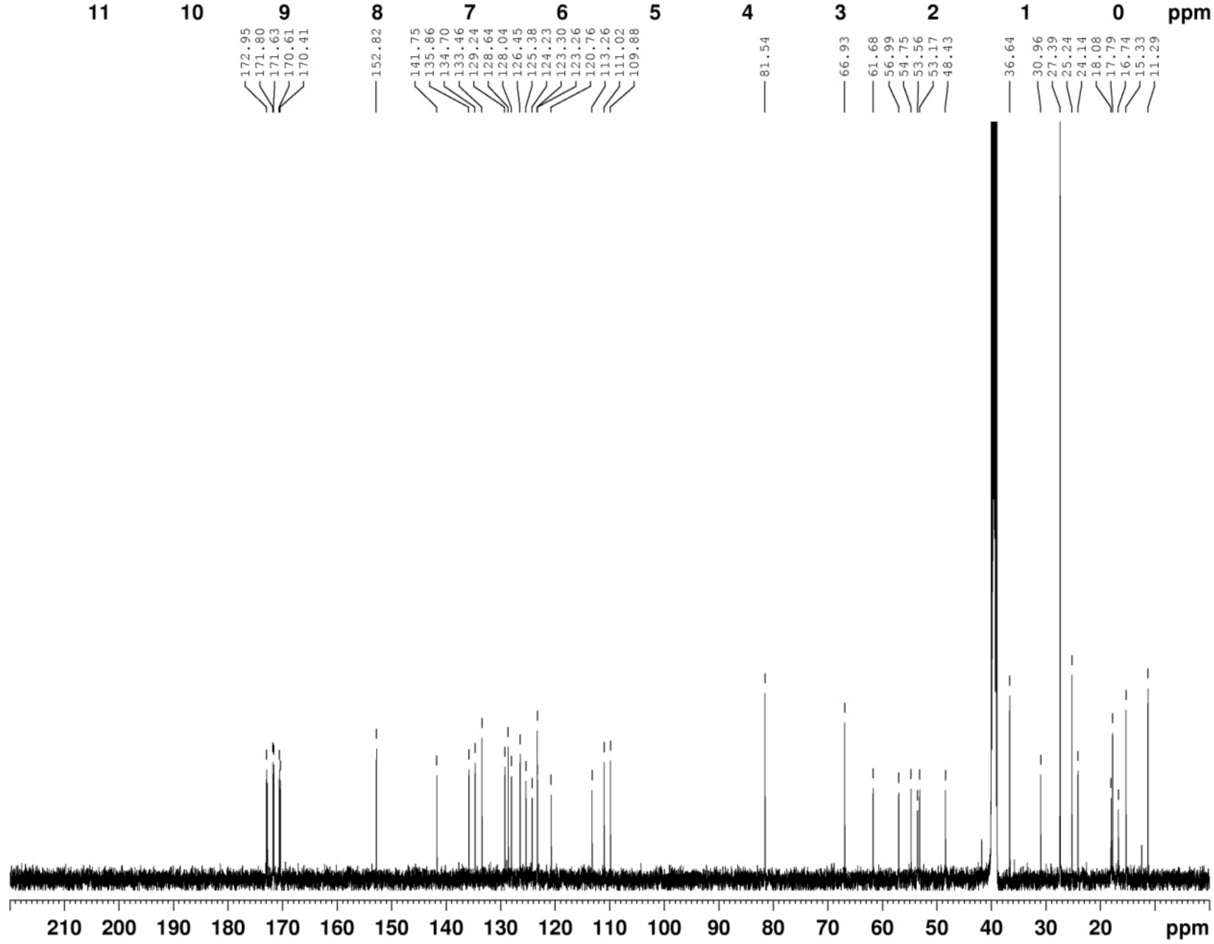

```
Current Data Parameters
NAME          KL5-106-5-1
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121221
Time                17.51
INSTRUM             av600
PROBHD   5 mm TBI5
PULPROG      dipsi2etgpsi
TD                   2048
SOLVENT              DMSO
NS                      8
DS                     16
SWH              5387.931 Hz
FIDRES           2.630826 Hz
AQ              0.1900544 sec
RG                    362
DW                 92.800 usec
DE                   6.50 usec
TE                  298.0 K
D0             0.00000300 sec
D1             1.50000000 sec
D9             0.06000000 sec
D11            0.03000000 sec
D16            0.00020000 sec
D20            0.00001000 sec
D21            0.00001000 sec
IN0            0.00018560 sec
L1                     14

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.23 usec
P2                  18.46 usec
P6                  40.00 usec
PL1                 -2.00 dB
PL10                10.74 dB
PL1W          39.81071854 W
PL10W          2.11836123 W
SFO1          600.1327006 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPNAM[2]         SINE.100
GPX1        0 %
GPX2        0 %
GPY1        0 %
GPY2        0 %
GPZ1                30.00 %
GPZ2                30.00 %
P16               1000.00 usec
```



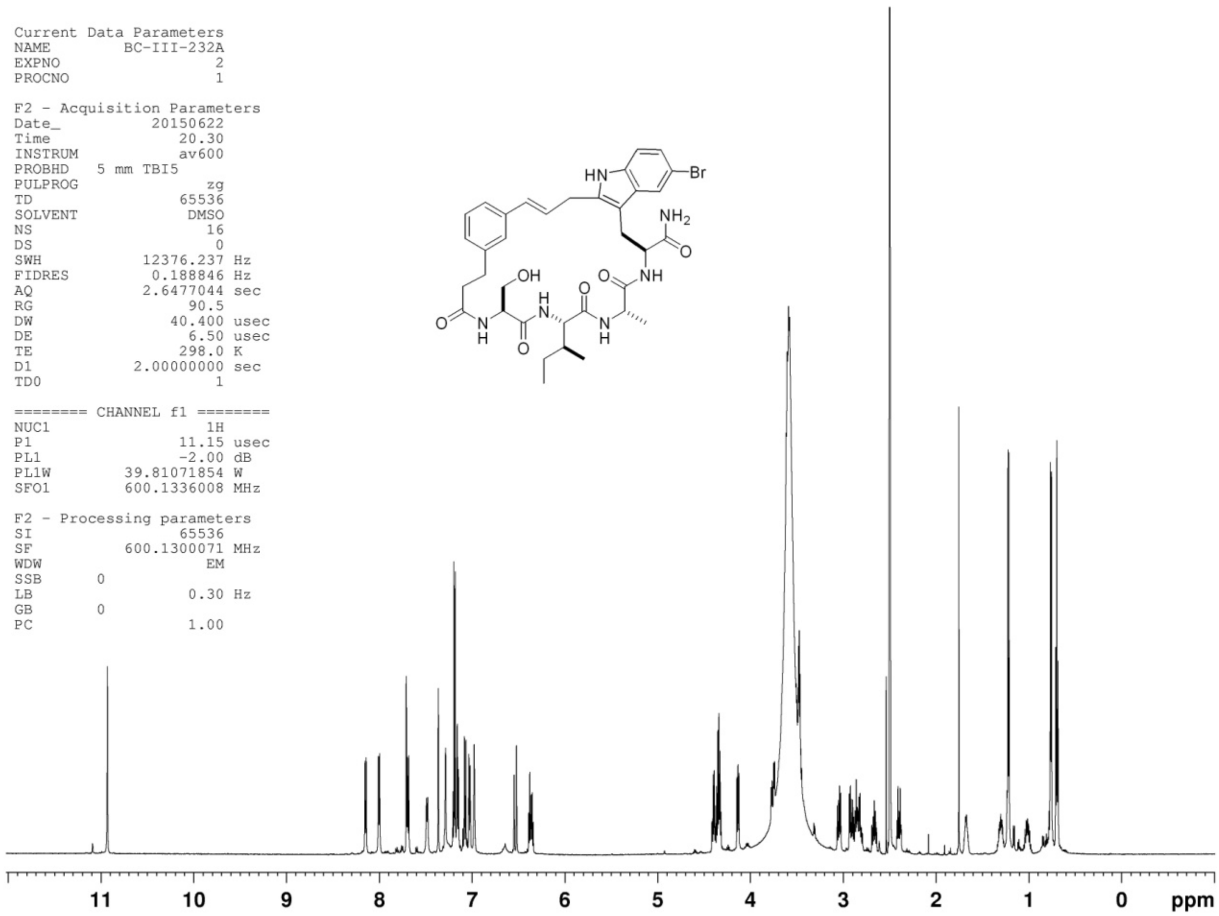

```
Current Data Parameters
NAME          KL5-106-5-1
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121221
Time                18.22
INSTRUM             av600
PROBHD   5 mm TBI5
PULPROG      hsqcetgpsisp
TD                   2048
SOLVENT              DMSO
NS                     10
DS                     16
SWH              6009.615 Hz
FIDRES           2.934382 Hz
AQ              0.1703936 sec
RG                  26008
DW                 83.200 usec
DE                   6.00 usec
TE                  298.1 K
CNST2         145.0000000
D0             0.00000300 sec
D1             1.00000000 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D24            0.00086200 sec
IN0            0.00002070 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.23 usec
P2                  18.46 usec
P28               1000.00 usec
PL1                 -2.00 dB
PL1W          39.81071854 W
SFO1          600.1327006 MHz

======== CHANNEL f2 ========
CPDPRG[2             garp
NUC2                  13C
P3                  18.50 usec
P4                  37.00 usec
P14               1000.00 usec
PCPD2               65.00 usec
PL0                120.00 dB
PL2                 -3.00 dB
PL12                 7.91 dB
PL0W        0 W
PL2W         150.35617065 W
PL12W         12.19330025 W
SFO2          150.9133722 MHz
```

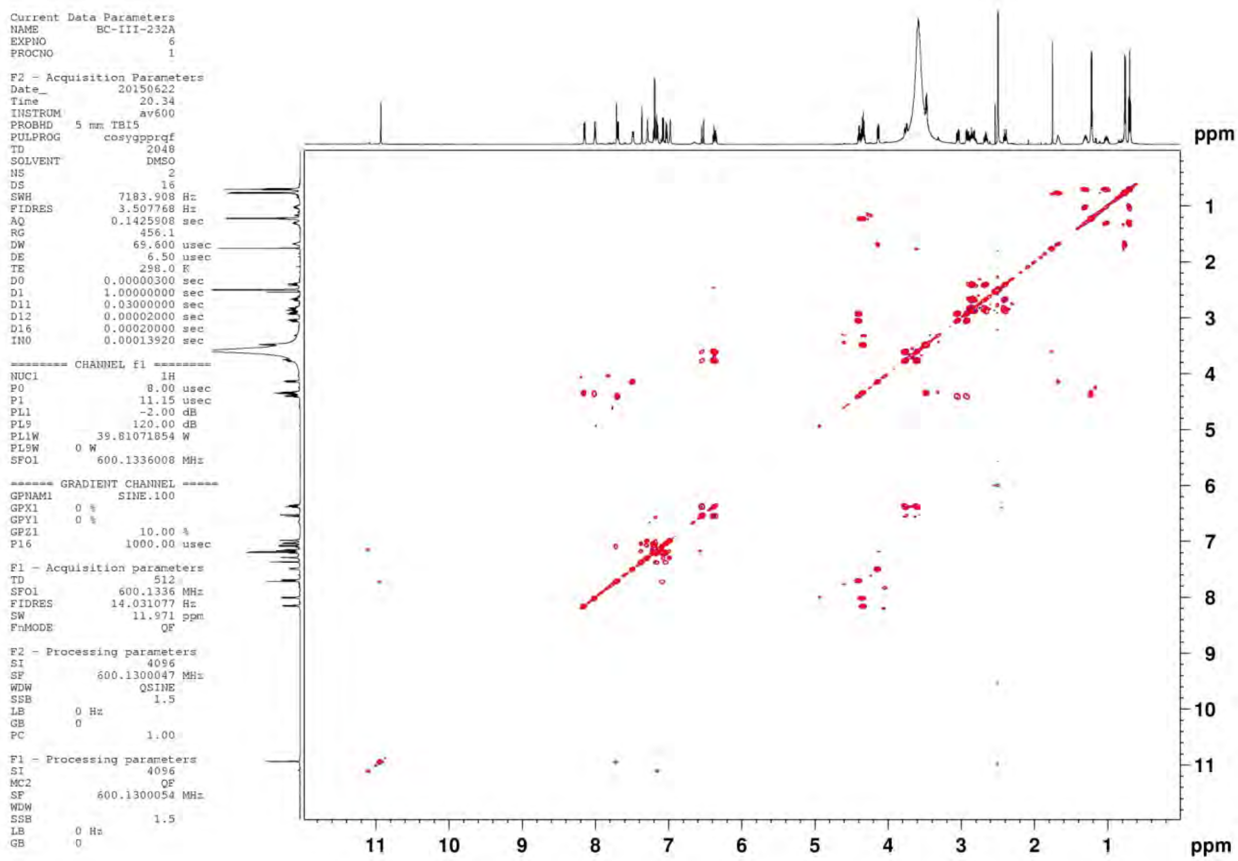
*Acyclic Precursor **15***

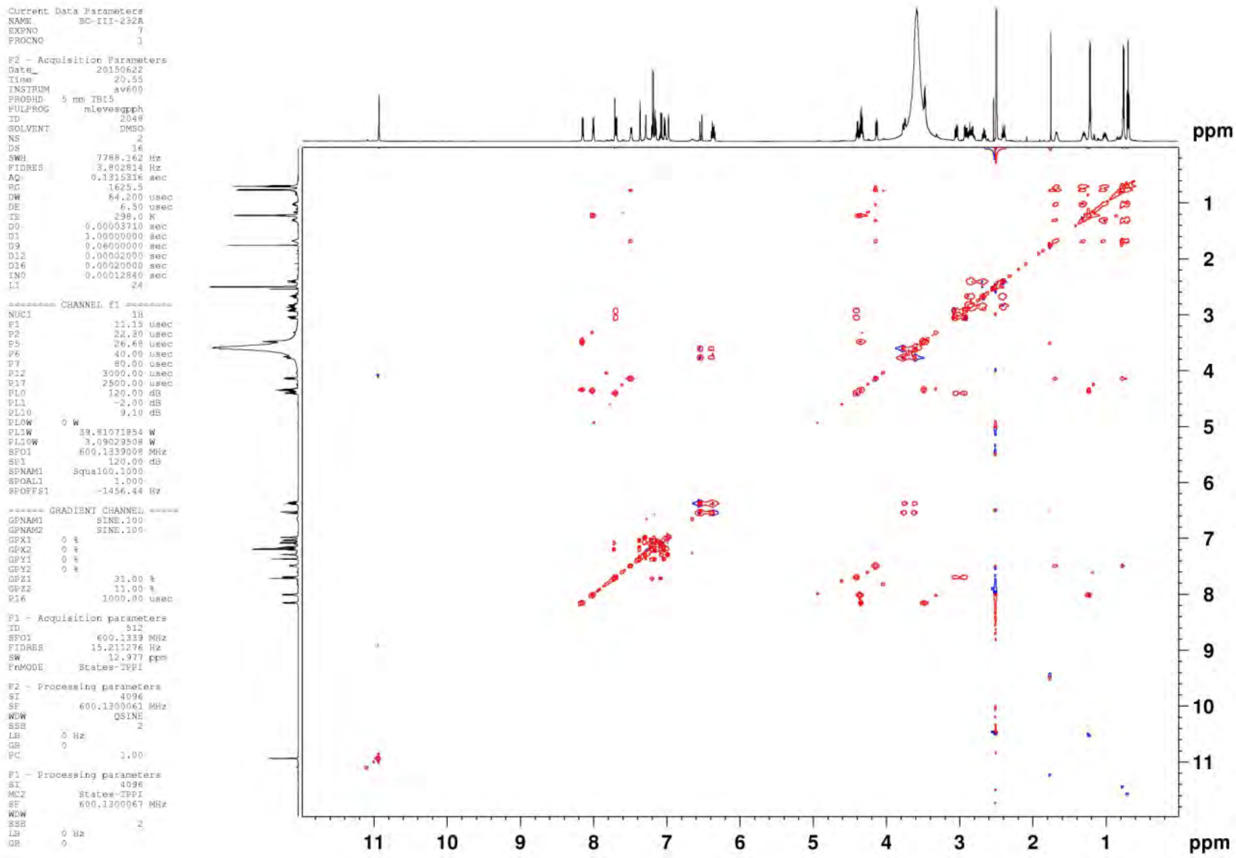


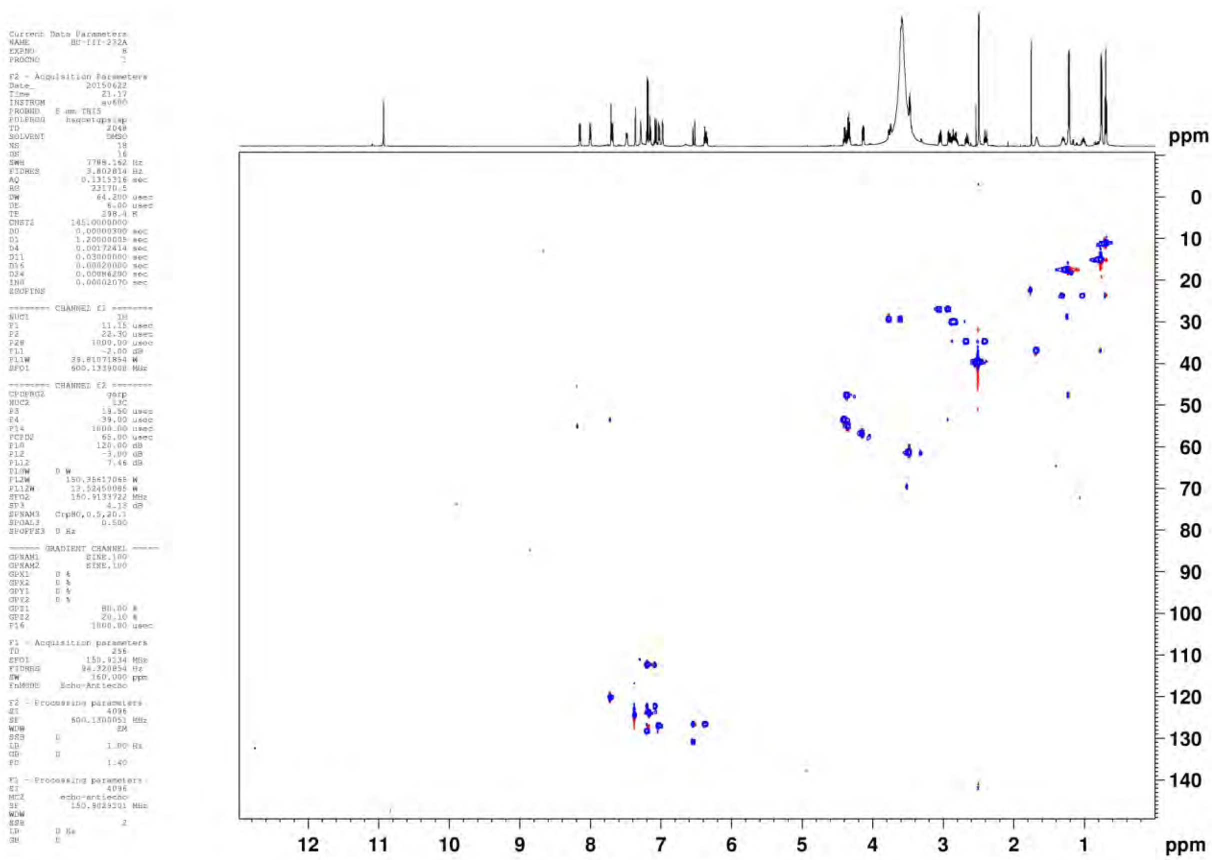


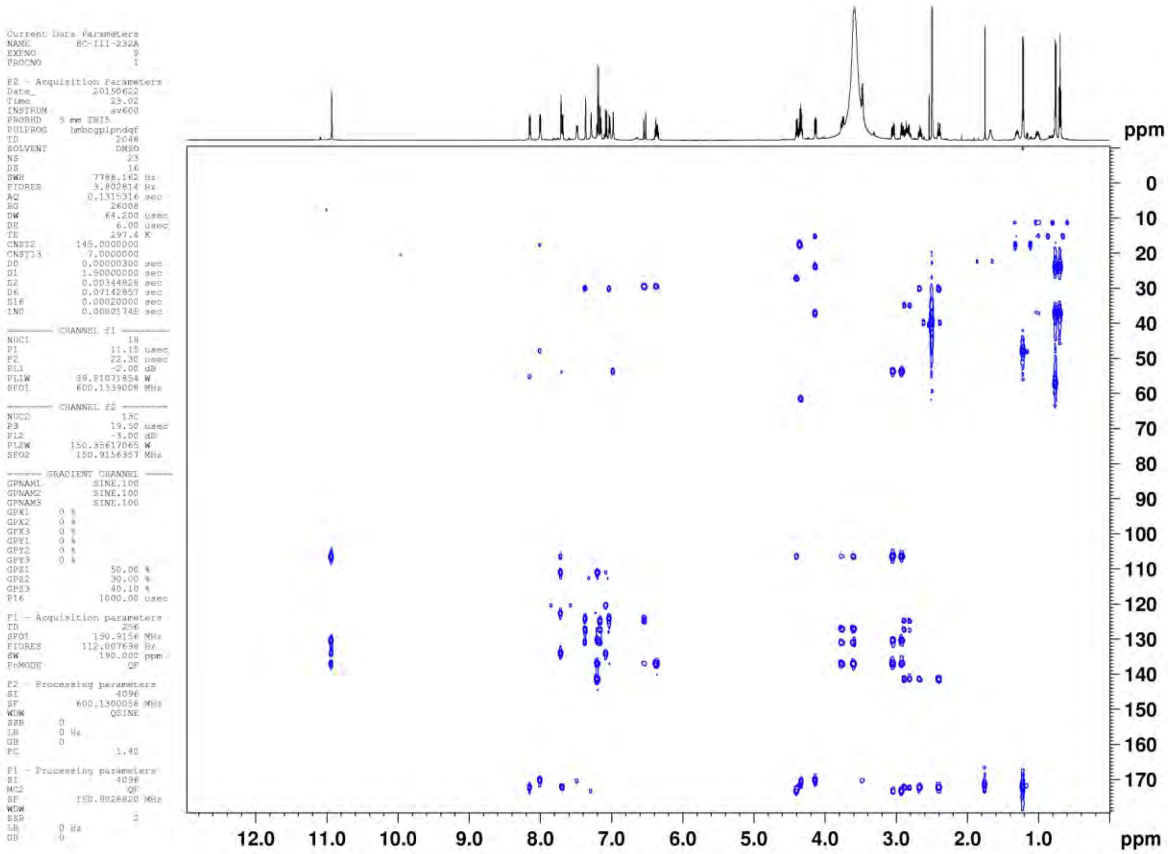
*Macrocyclic Product **19a***



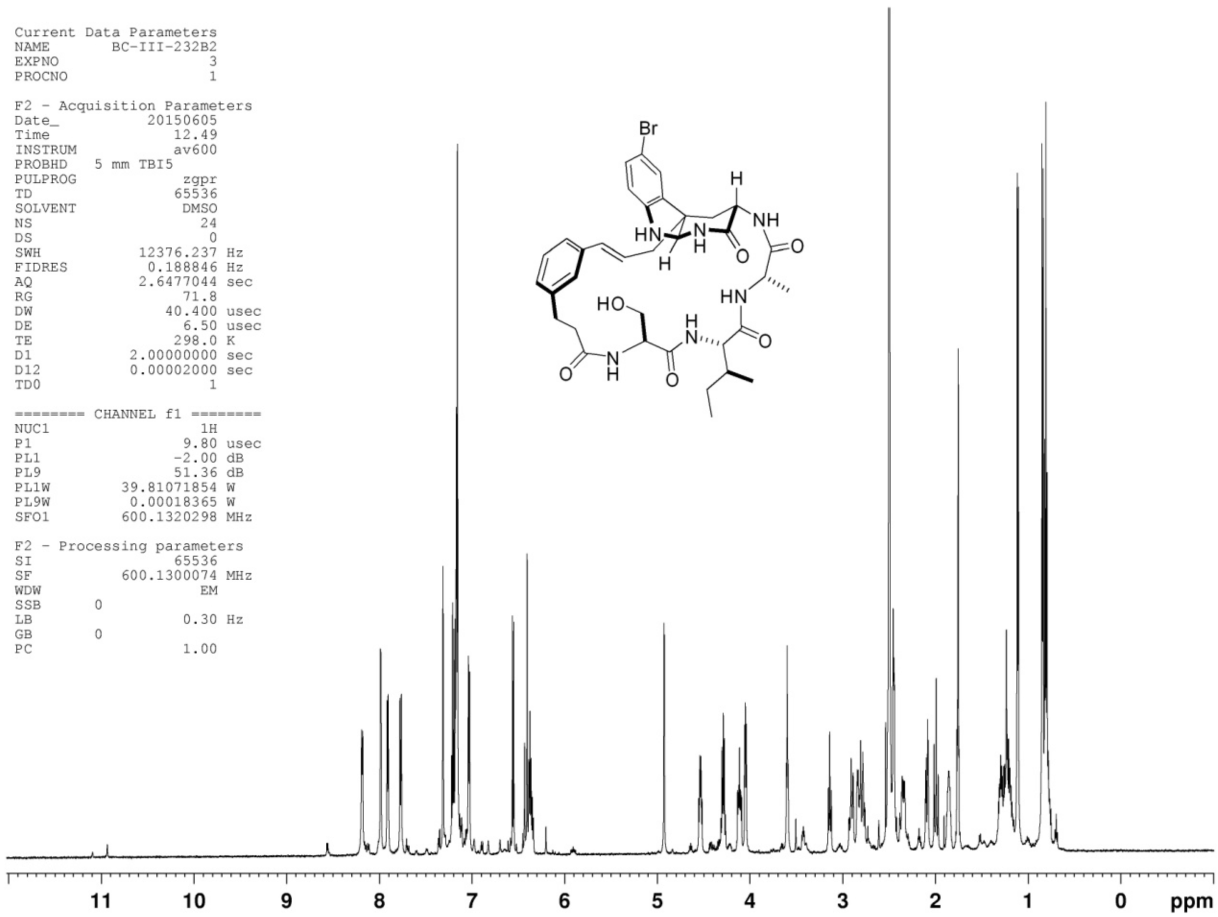


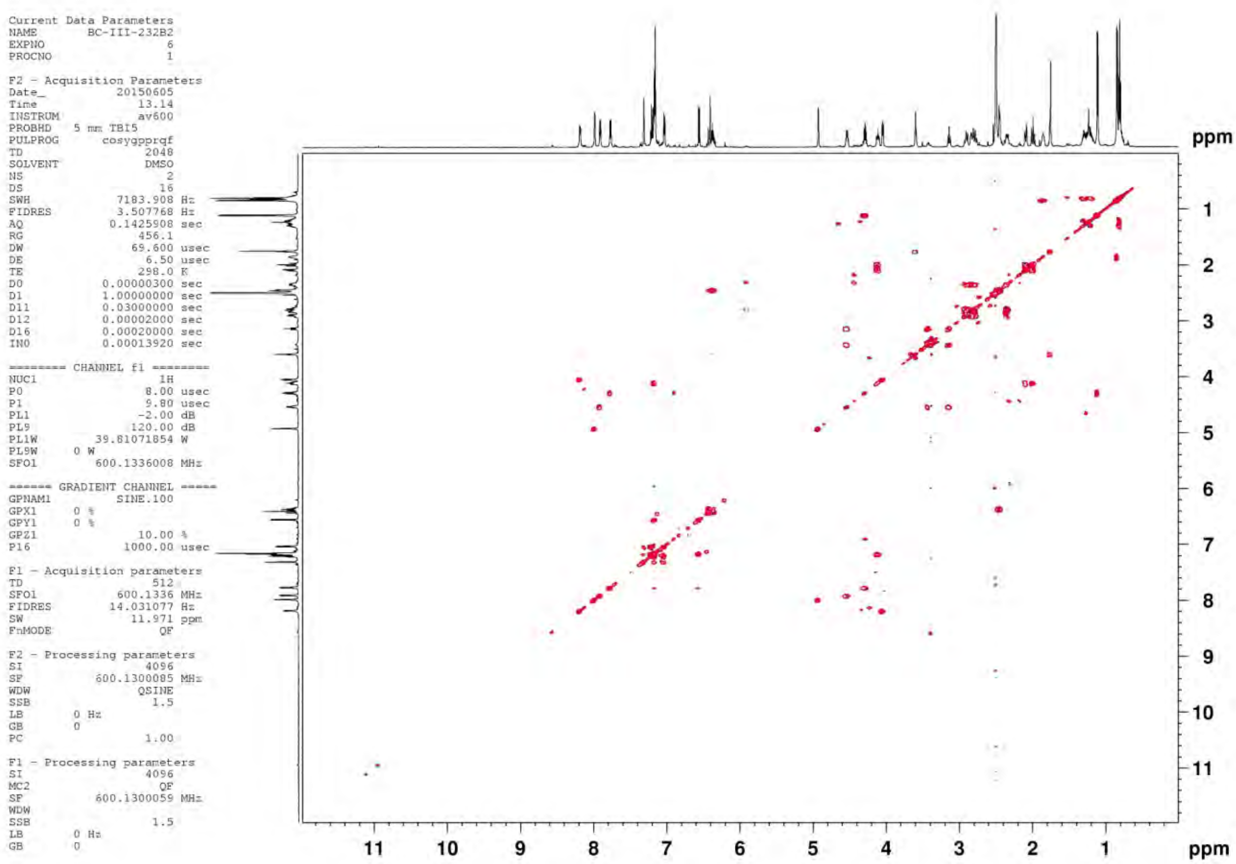


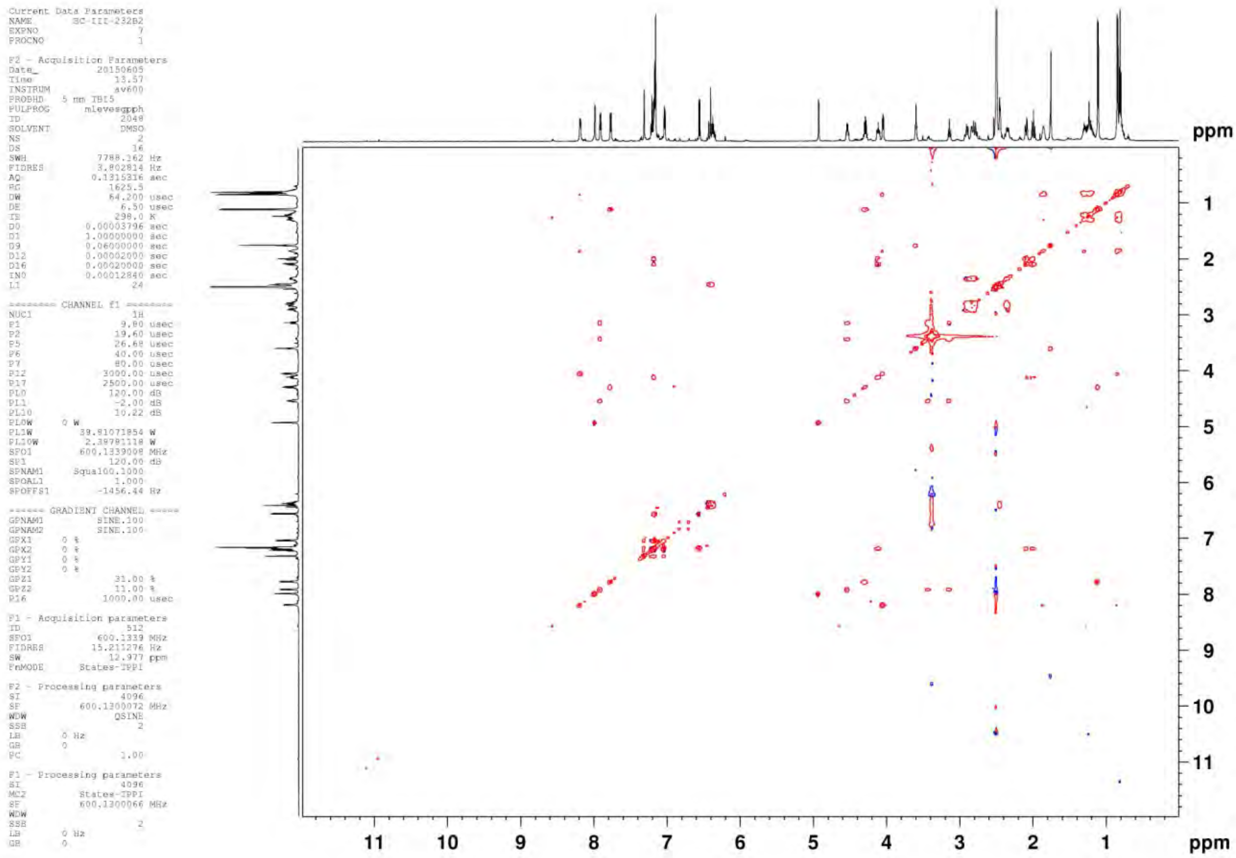


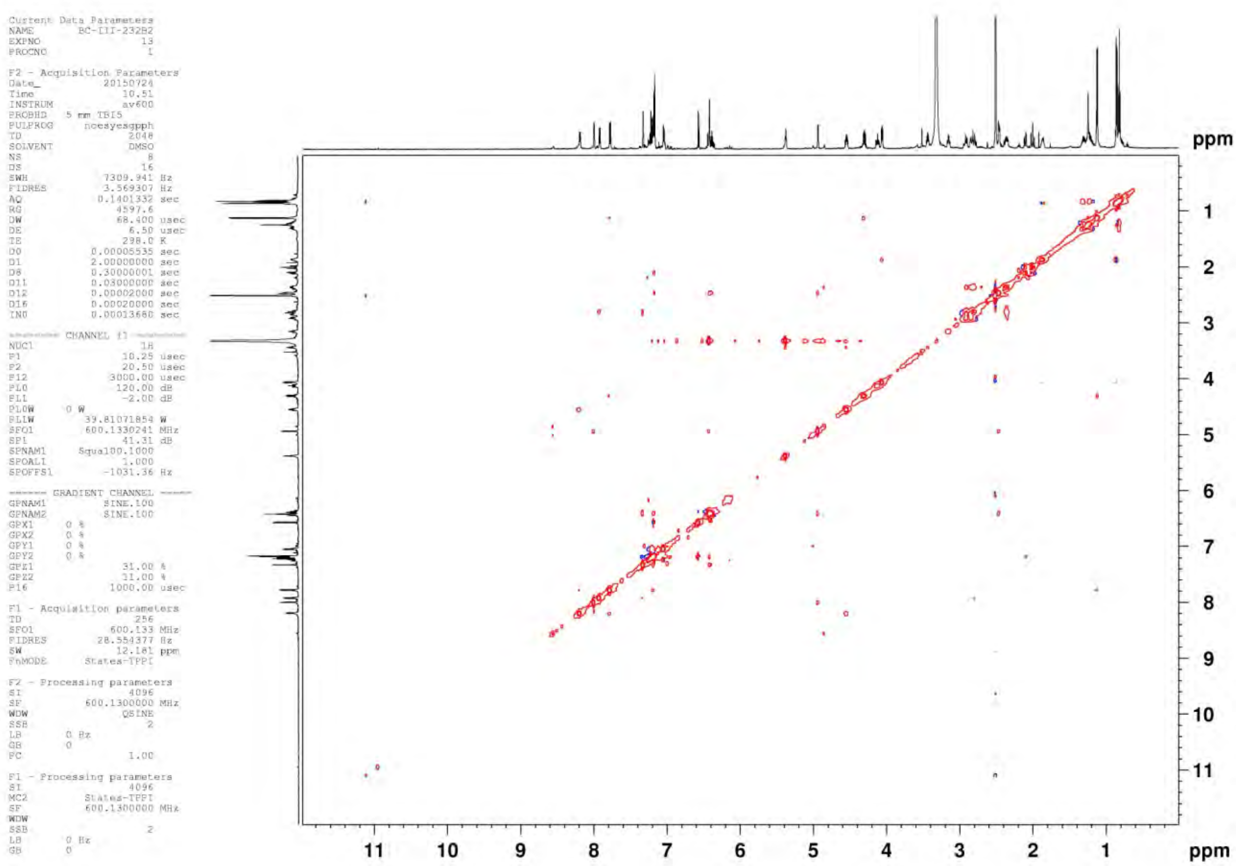

ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm

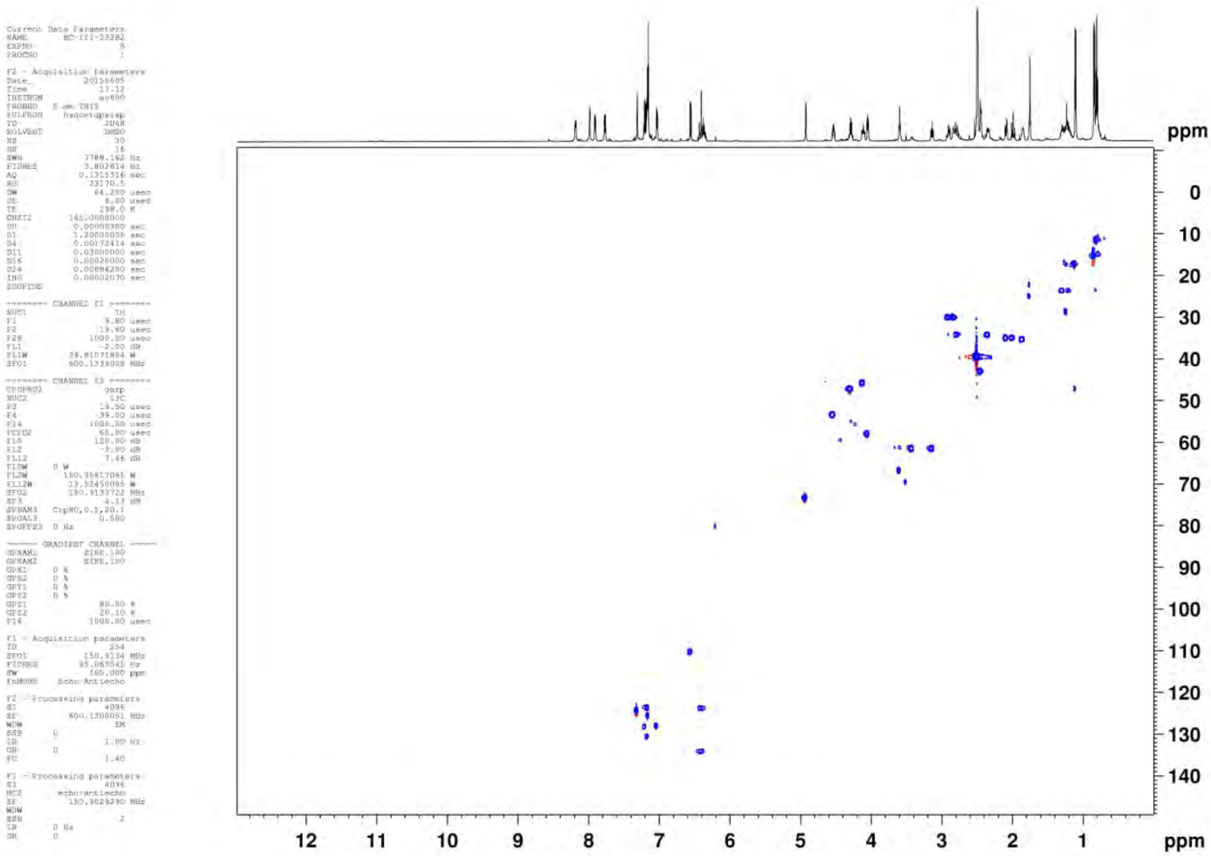
*Macrocyclic Product **19b***

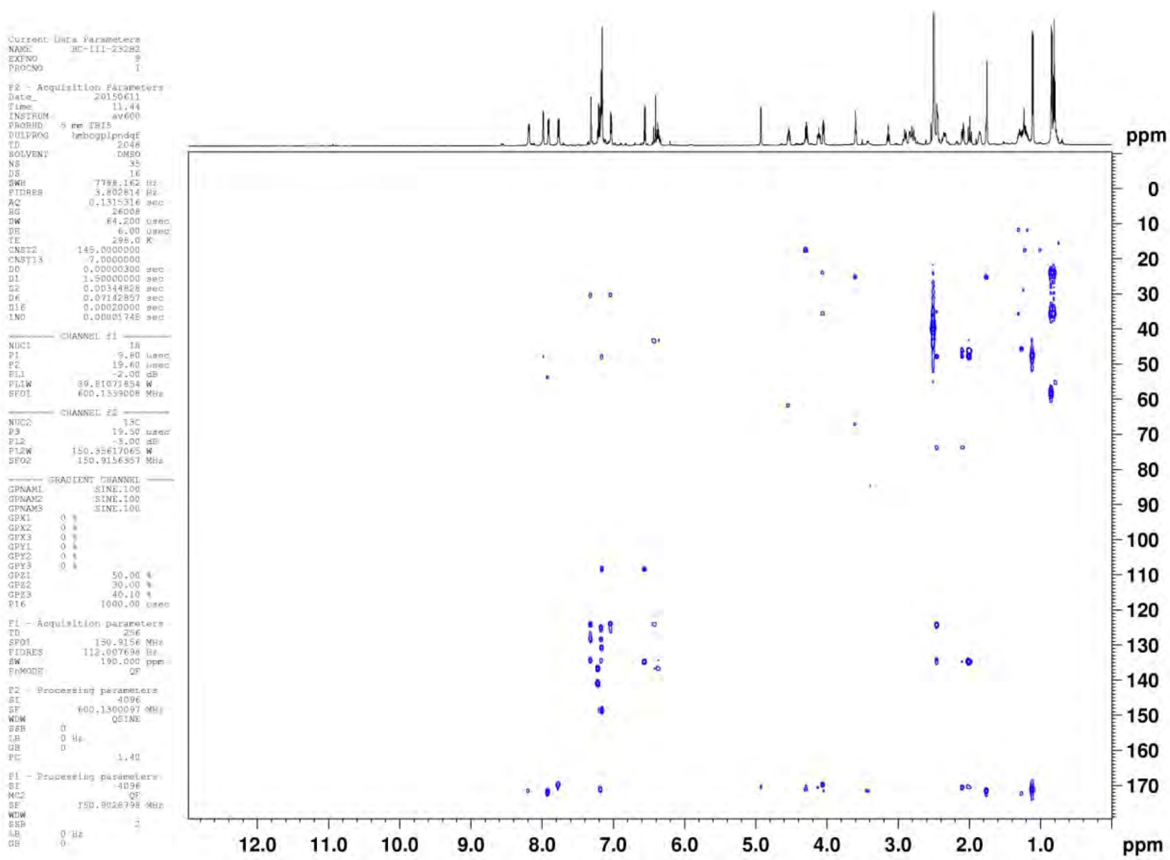




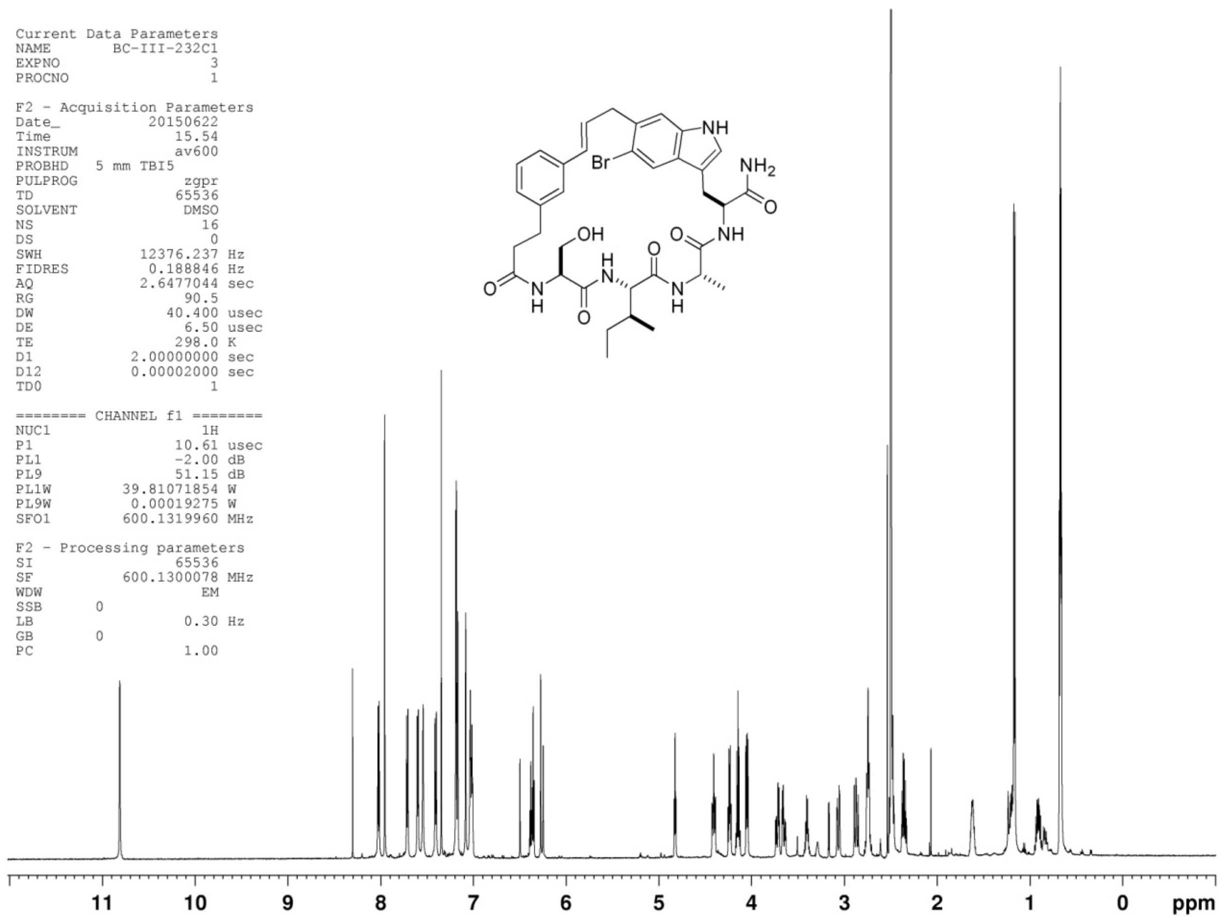

Current Data Parameters
NAME BC-III-232B2
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150605
Time 13.57
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevesgpph
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 1625.5
DW 64.200 usec
DE 6.50 usec
TE 298.0 K
D0 0.00003796 sec
D1 1.00000000 sec
D9 0.06000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00012840 sec
L1 24
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
P2 19.60 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P12 3000.00 usec
P17 2500.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL10 10.22 dB
PL0W 0 W
PL1W 39.81071854 W
PL10W 2.39781118 W
SFO1 600.1339008 MHz
SP1 120.00 dB
SPNAM1 Squa100.1000
SPOAL1 1.000
SPOFFS1 -1456.44 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 31.00 %
GPZ2 11.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 512
SFO1 600.1339 MHz
FIDRES 15.211276 Hz
SW 12.977 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 4096
SF 600.1300072 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 4096
MC2 States-TPPI
SF 600.1300066 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
1
2
3
4
5
6
7
8
9
10
11
11
10
9
8
7
6
5
4
3
2
1
ppm

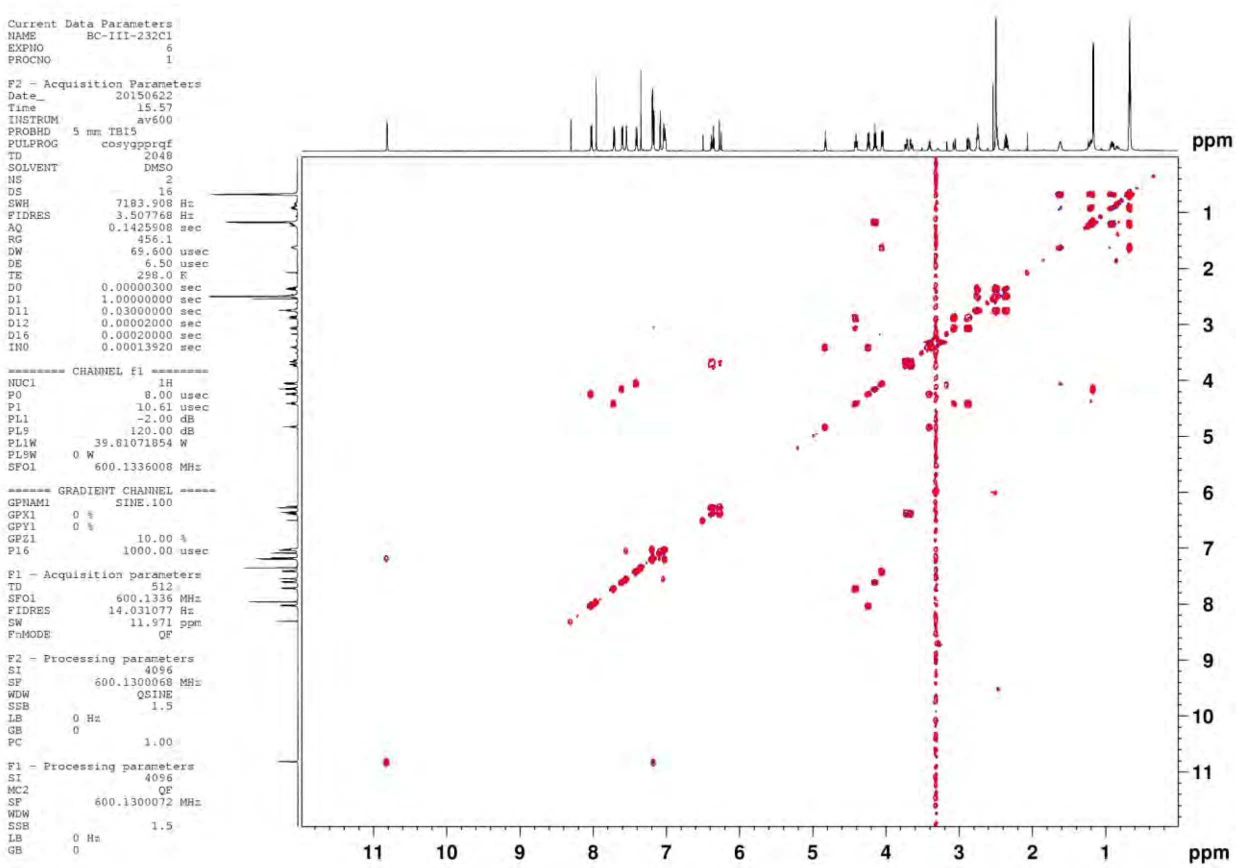

Current Data Parameters
NAME BC-III-232B2
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150724
Time 10.51
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG noesyesgpph
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 7309.941 Hz
FIDRES 3.569307 Hz
AQ 0.1401332 sec
RG 4597.6
DW 68.400 usec
DE 6.50 usec
TE 298.0 K
D0 0.00005535 sec
D1 2.00000000 sec
D8 0.30000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00013680 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
P2 20.50 usec
P12 3000.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL0W 0 W
PL1W 39.81071854 W
SFO1 600.1330241 MHz
SP1 41.31 dB
SPNAM1 Squa100.1000
SPOAL1 1.000
SPOFFS1 -1031.36 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 31.00 %
GPZ2 11.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 600.133 MHz
FIDRES 28.554377 Hz
SW 12.181 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 4096
SF 600.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 4096
MC2 States-TPPI
SF 600.1300000 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
1
2
3
4
5
6
7
8
9
10
11
11
10
9
8
7
6
5
4
3
2
1
ppm

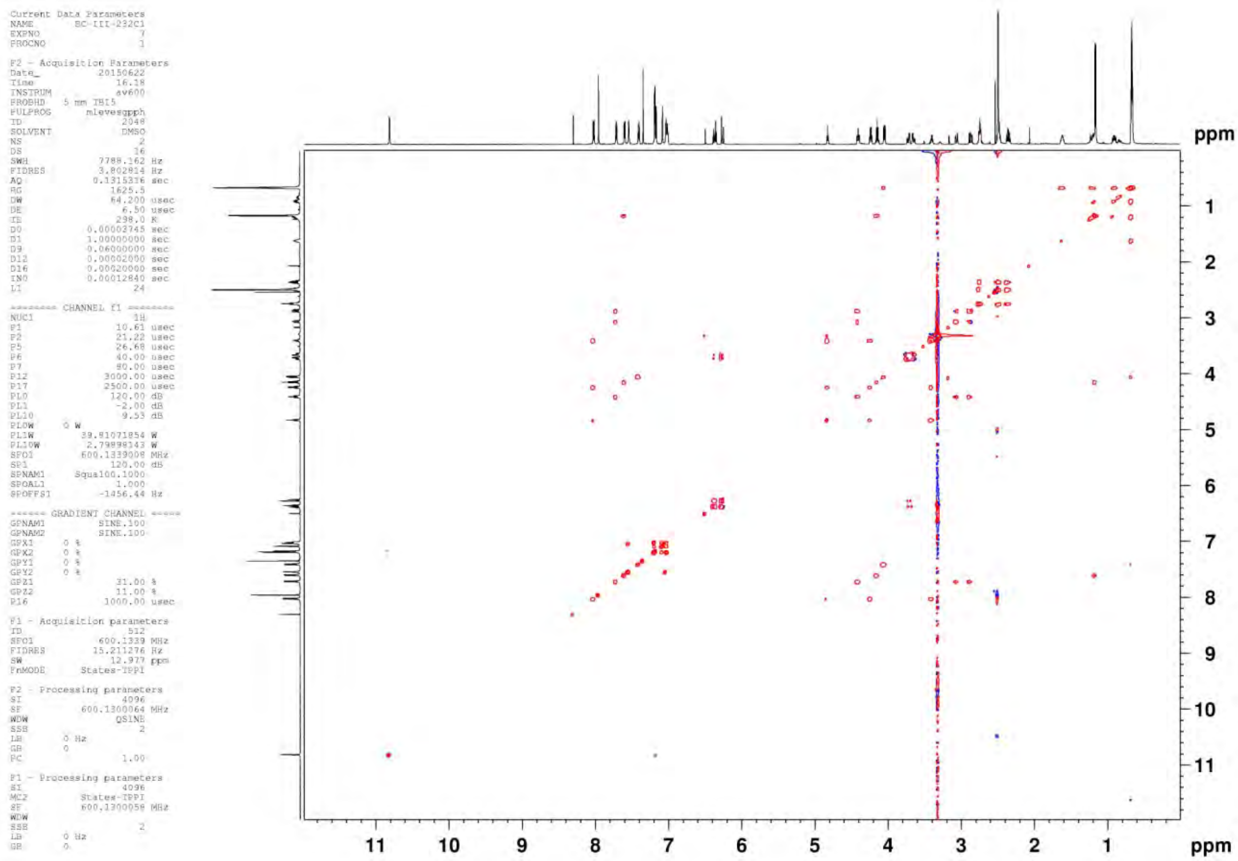

ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
12
11
10
9
8
7
6
5
4
3
2
1
ppm


ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm

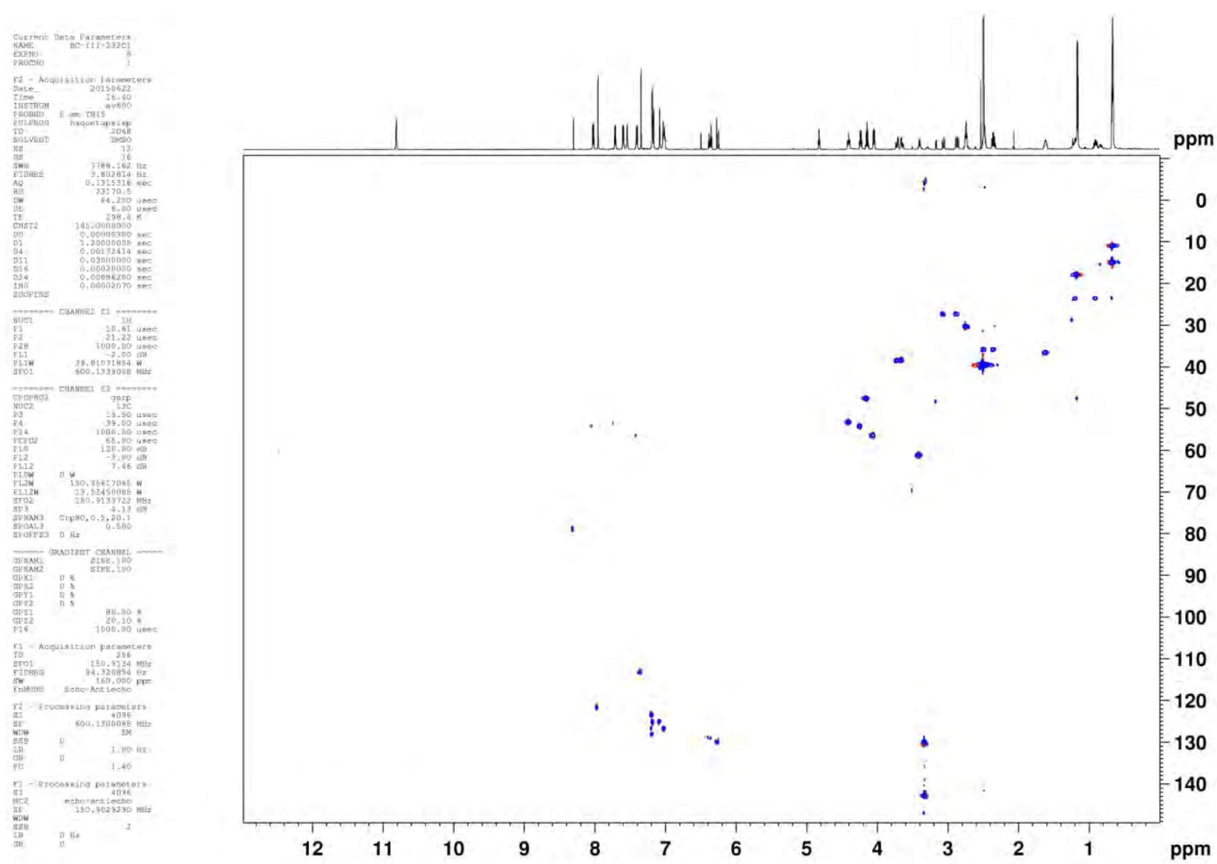
*Macrocyclic Product **19c***



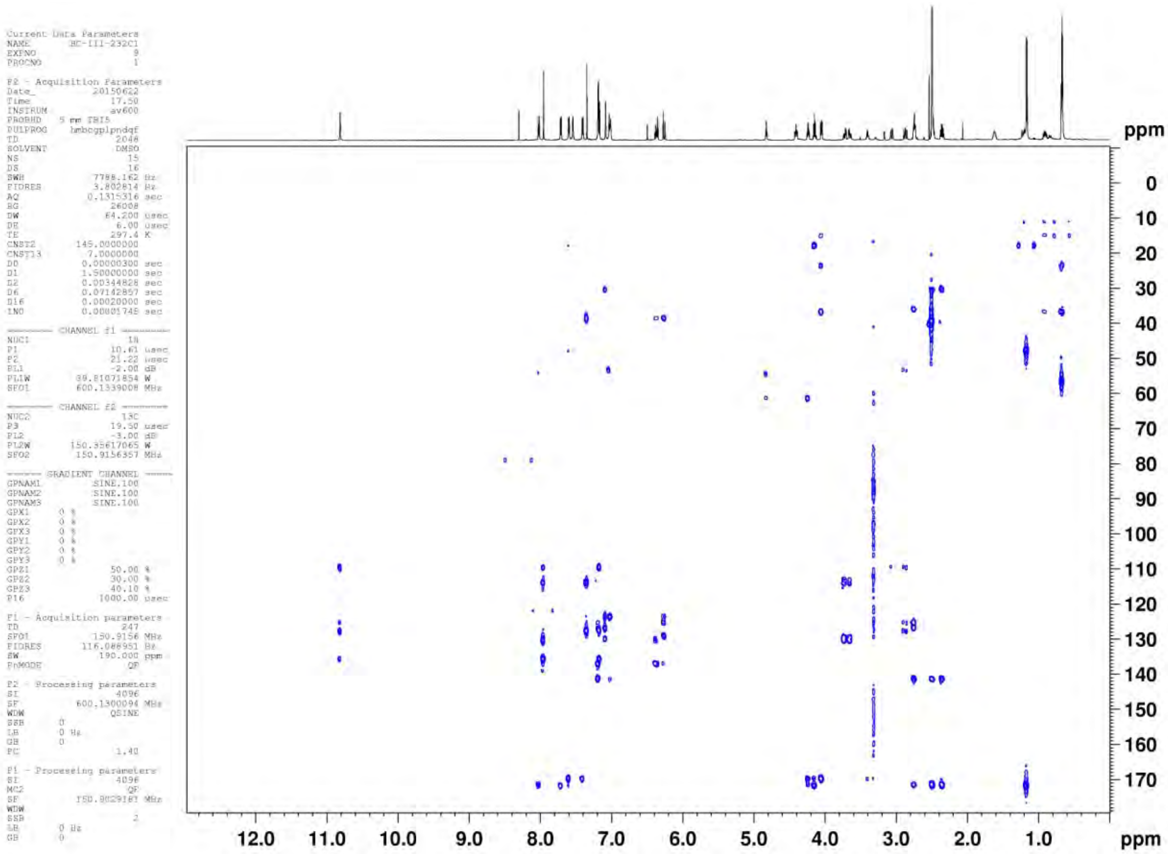




Current Data Parameters
NAME BC-III-232C1
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150622
Time 16.18
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevesgpph
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 1625.5
DW 64.200 usec
DE 6.50 usec
TE 298.0 K
D0 0.00002745 sec
D1 1.00000000 sec
D9 0.06000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00012840 sec
L1 24
======== CHANNEL f1 ========
NUC1 1H
P1 10.61 usec
P2 21.22 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P12 3000.00 usec
P17 2500.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL10 9.53 dB
PL0W 0 W
PL1W 39.81071854 W
PL10W 2.79898143 W
SFO1 600.1339008 MHz
SP1 120.00 dB
SPNAM1 Squa100.1000
SPOAL1 1.000
SPOFFS1 -1456.44 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 31.00 %
GPZ2 11.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 512
SFO1 600.1339 MHz
FIDRES 15.211276 Hz
SW 12.977 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 4096
SF 600.1300064 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 4096
MC2 States-TPPI
SF 600.1300058 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
1
2
3
4
5
6
7
8
9
10
11
11 10 9 8 7 6 5 4 3 2 1 ppm

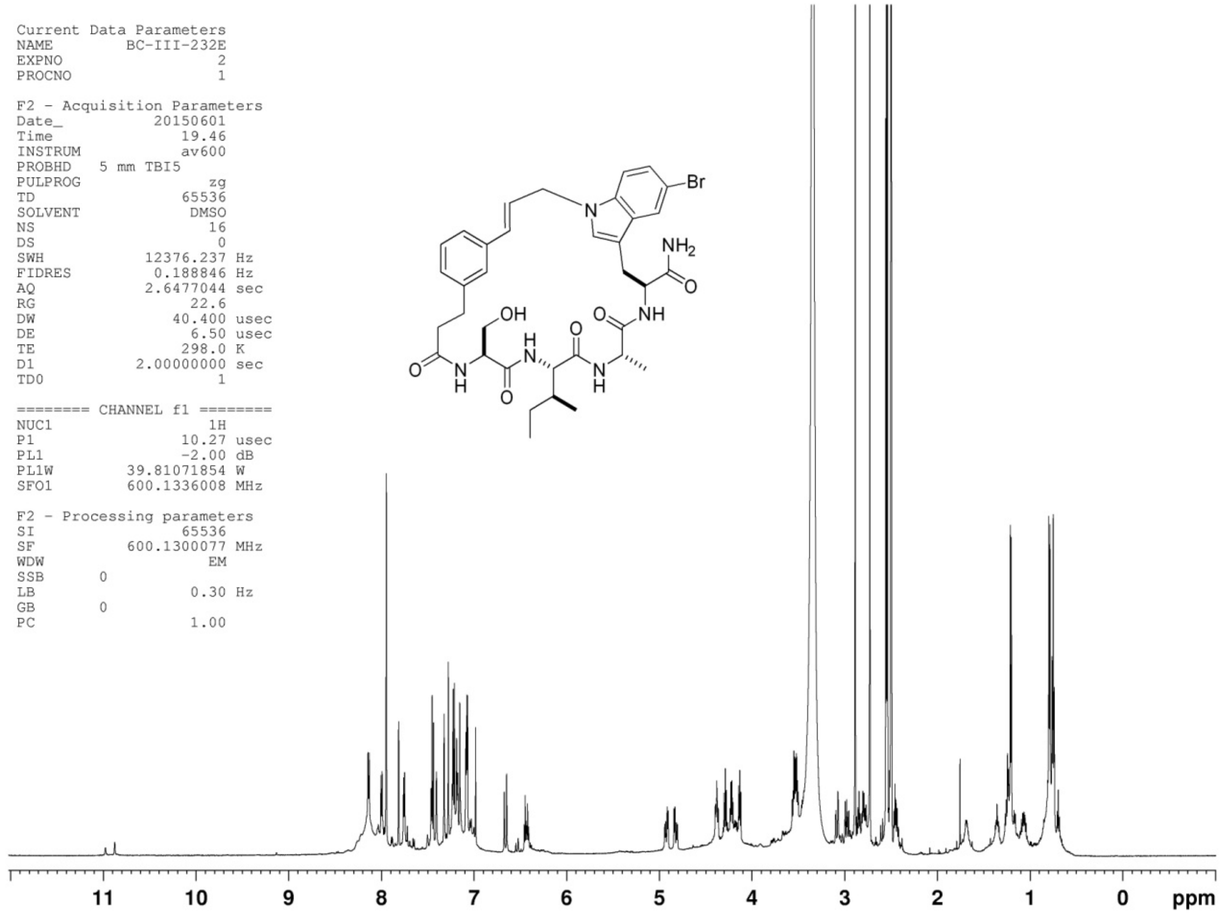


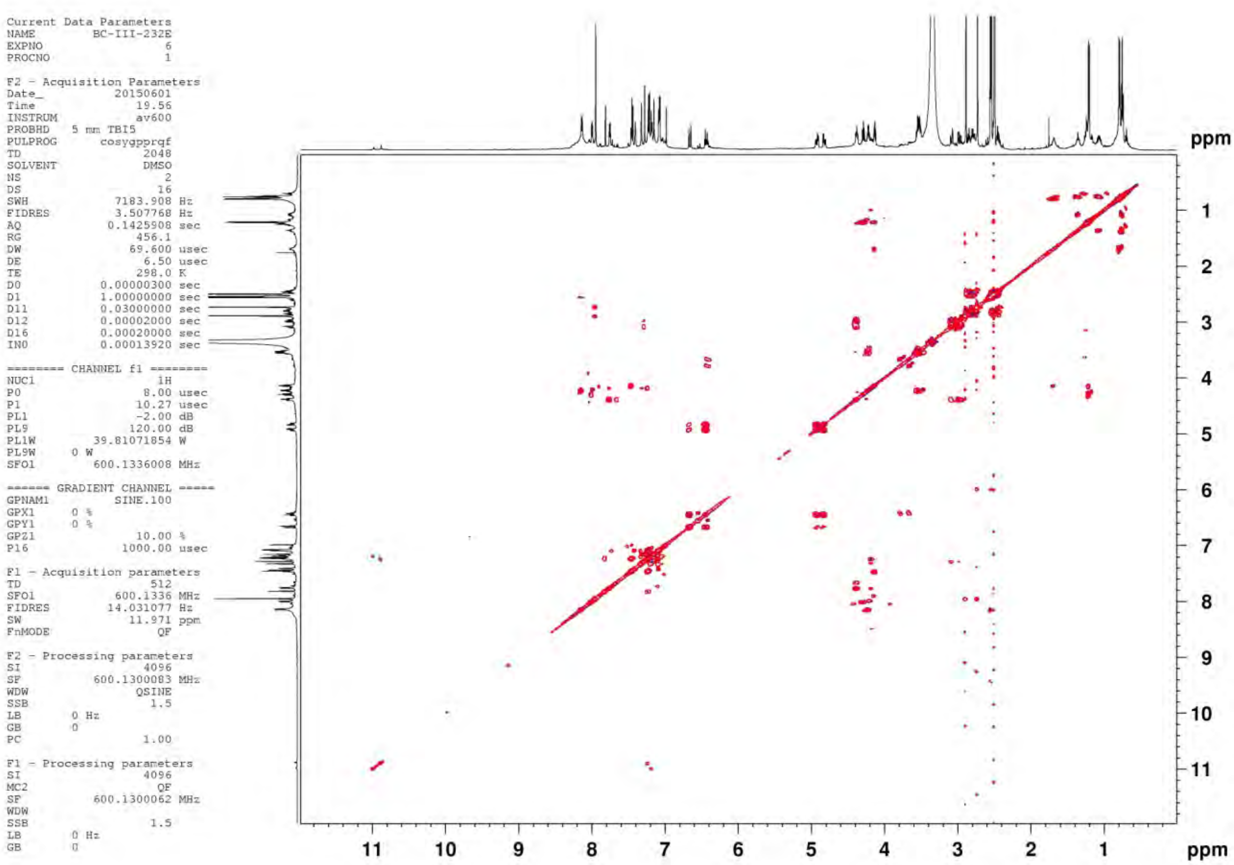
Current Data Parameters
NAME BC-III-232C1
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150622
Time 16.40
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 12
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 23170.5
DW 64.200 usec
DE 6.00 usec
TE 298.4 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 10.61 usec
P2 21.22 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 19.50 usec
P4 39.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.46 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 13.52450085 W
SFO2 150.9133722 MHz
SP3 4.13 dB
SPNAM3 Crp80,0.5,20.1
SPOAL3 0.500
SPOFFS3 0 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 150.9134 MHz
FIDRES 94.320854 Hz
SW 160.000 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 4096
SF 600.1300068 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 echo-antiecho
SF 150.9029290 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
12 11 10 9 8 7 6 5 4 3 2 1 ppm

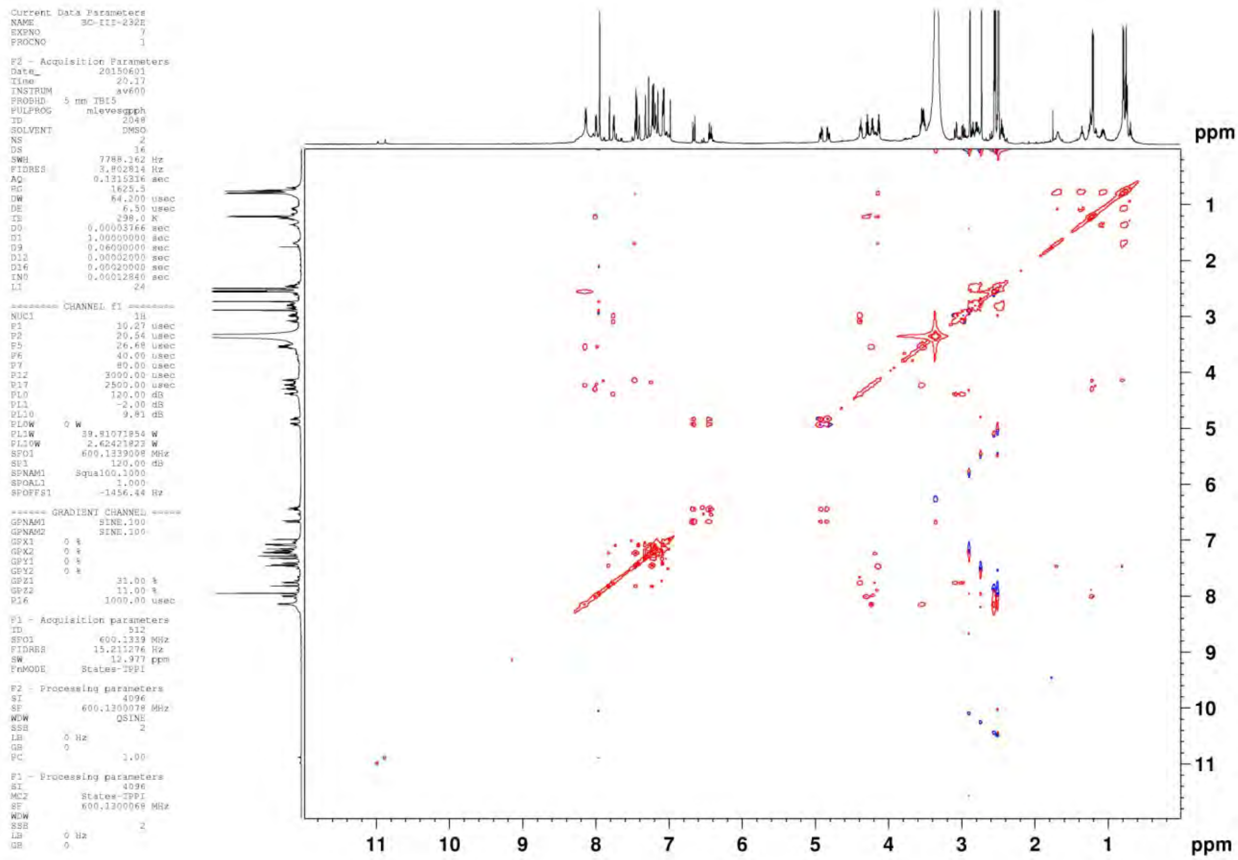


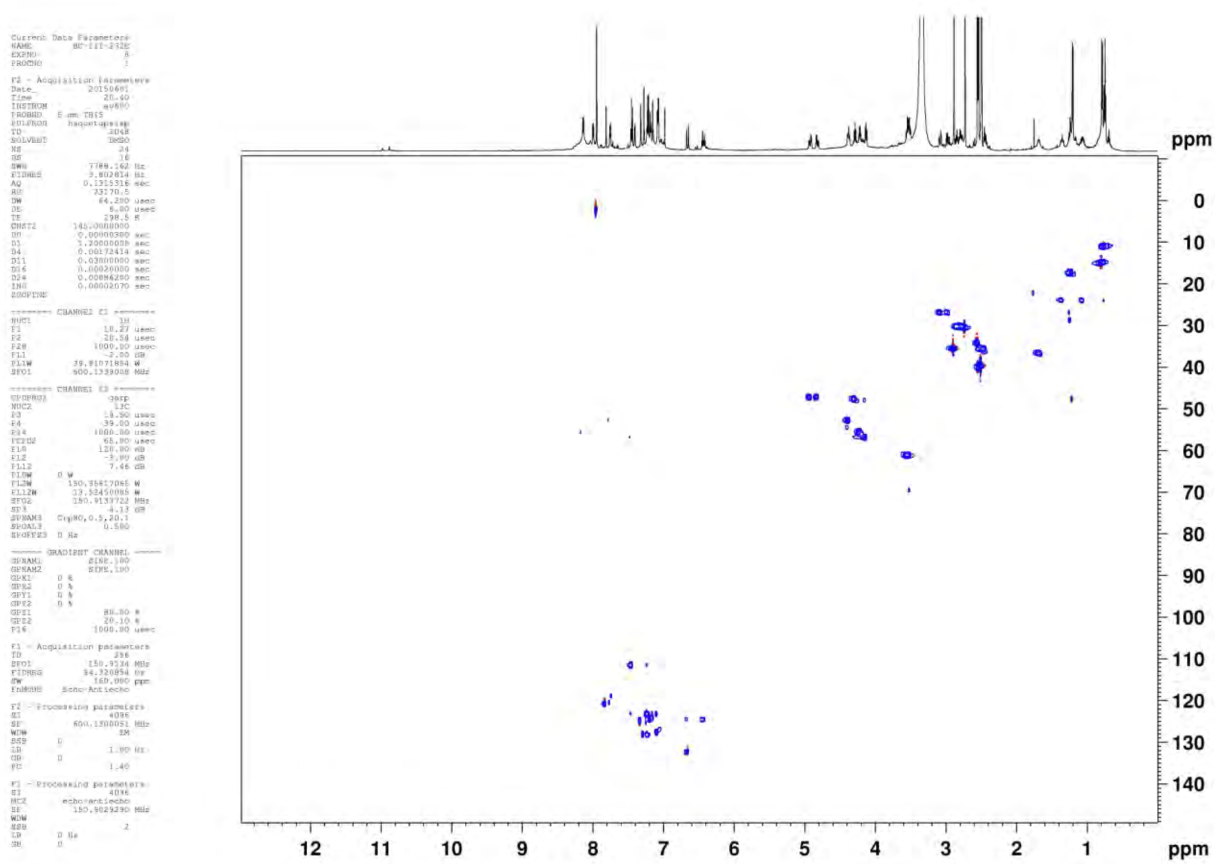
Current Data Parameters
NAME BC-III-232C1
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150622
Time 17.50
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 15
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 26008
DW 64.200 usec
DE 6.00 usec
TE 297.4 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001748 sec
CHANNEL f1
NUC1 1H
P1 10.61 usec
P2 21.22 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
CHANNEL f2
NUC2 13C
P3 19.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 247
SFO1 150.9156 MHz
FIDRES 116.088951 Hz
SW 190.000 ppm
FnMODE QF
F2 - Processing parameters
SI 4096
SF 600.1300094 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 QF
SF 150.9029187 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0
ppm

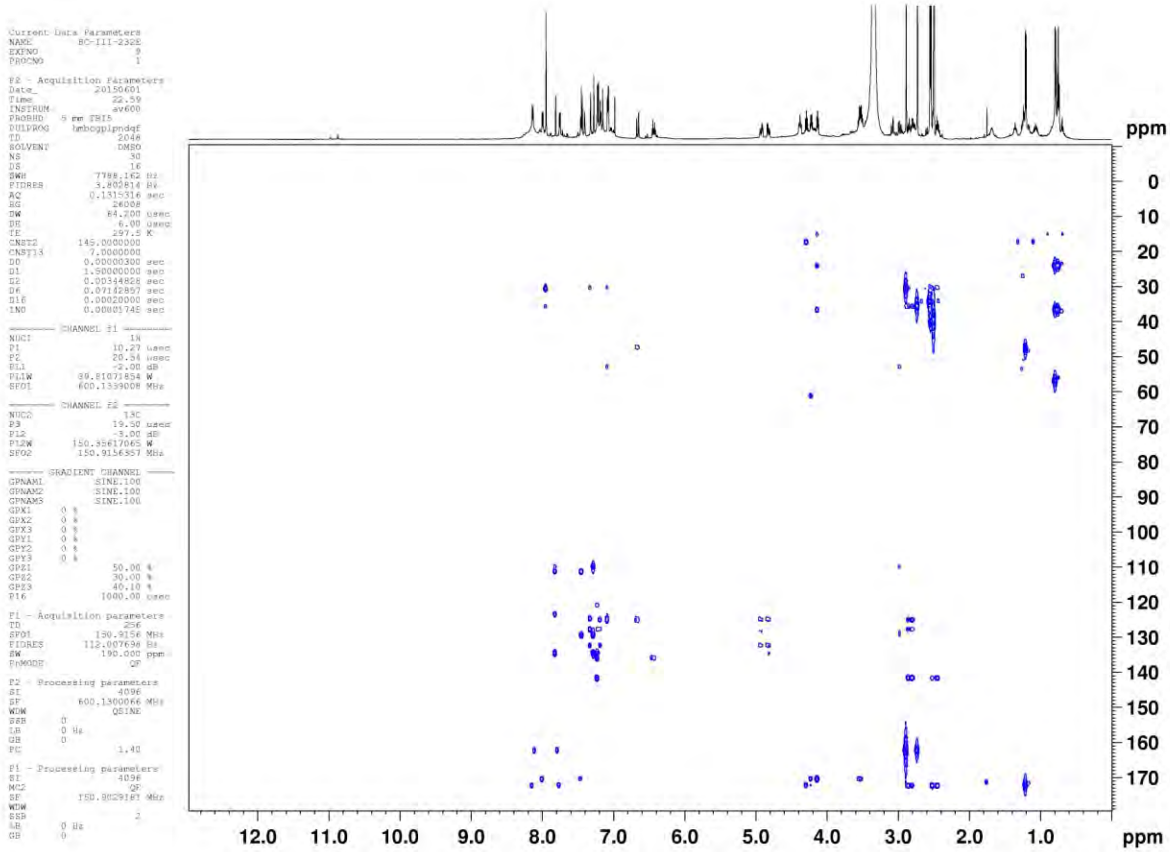
*Macrocyclic Product **19d***





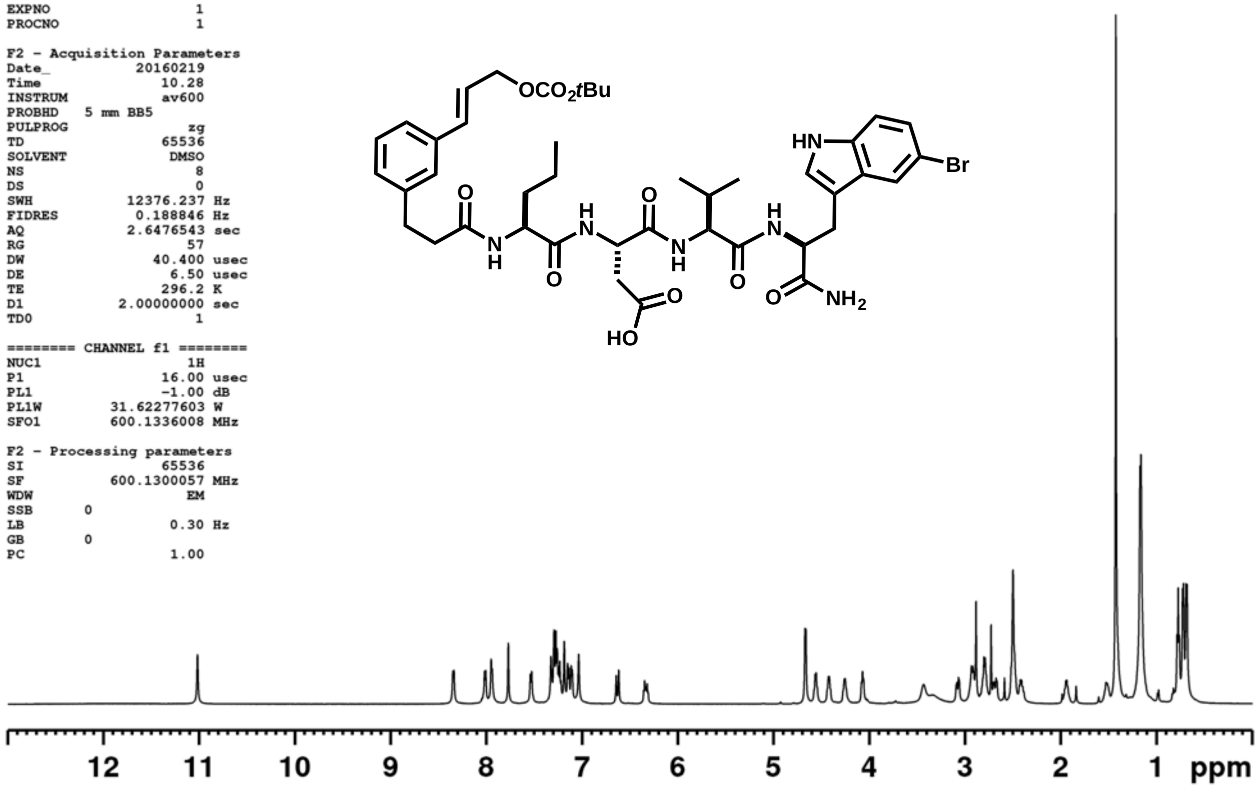

Current Data Parameters
NAME BC-III-232E
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150601
Time 20.17
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevesgpph
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 1625.5
DW 64.200 usec
DE 6.50 usec
TE 298.0 K
D0 0.00003766 sec
D1 1.00000000 sec
D9 0.06000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00012840 sec
L1 24
======== CHANNEL f1 ========
NUC1 1H
P1 10.27 usec
P2 20.54 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P12 3000.00 usec
P17 2500.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL10 9.81 dB
PL0W 0 W
PL1W 39.81071854 W
PL10W 2.62421823 W
SFO1 600.1339008 MHz
SP1 120.00 dB
SPNAM1 Squa100.1000
SPOAL1 1.000
SPOFFS1 -1456.44 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 31.00 %
GPZ2 11.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 512
SFO1 600.1339 MHz
FIDRES 15.211276 Hz
SW 12.977 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 4096
SF 600.1300078 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 4096
MC2 States-TPPI
SF 600.1300068 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
1
2
3
4
5
6
7
8
9
10
11
11
10
9
8
7
6
5
4
3
2
1
ppm

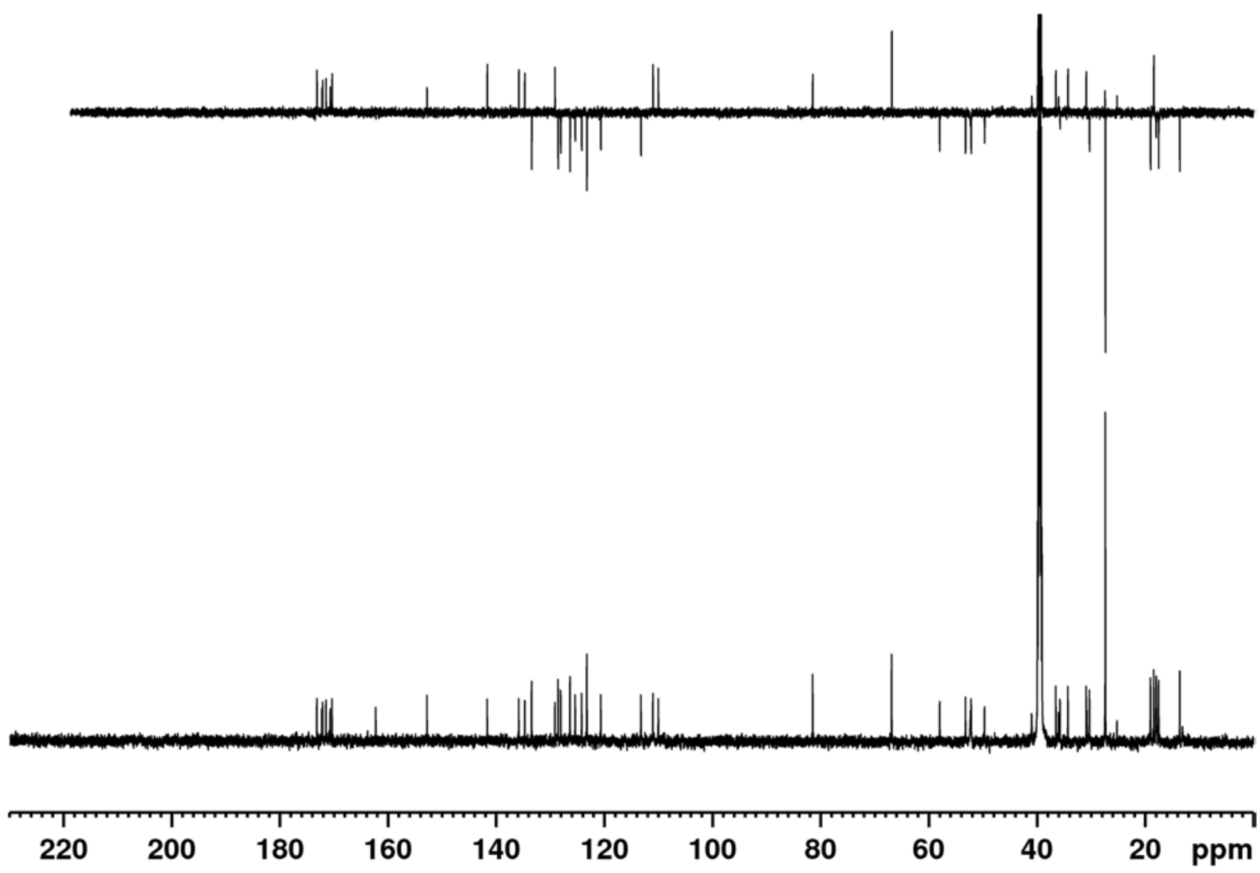

Current Data Parameters
NAME BC-III-232E
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150601
Time 20.40
INSTRUM av600
PROBHD 5 mm TBI5
TD 2048
SOLVENT DMSO
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
DW 64.200 usec
IN0 0.00002070 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.27 usec
P2 20.54 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P4 39.00 usec
PL12 7.46 dB
PL0W 0 W
SPNAM3 Crp80,0.5,20.1
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
F1 - Acquisition parameters
TD 256
SW 160.000 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 4096
WDW EM
F1 - Processing parameters
SI 4096
MC2 echo-antiecho
SF 150.9029290 MHz
WDW
SSB 2
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
12
11
10
9
8
7
6
5
4
3
2
1
ppm


Current Data Parameters
NAME BC-III-232E
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150601
Time 22.59
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 30
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1315316 sec
RG 26008
DW 64.200 usec
DE 6.00 usec
TE 297.5 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec
CHANNEL f1
NUC1 1H
P1 10.27 usec
P2 20.54 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
CHANNEL f2
NUC2 13C
P3 19.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 150.9156 MHz
FIDRES 112.007698 Hz
SW 190.000 ppm
FnMODE QF
F2 - Processing parameters
SI 4096
SF 600.1300066 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 QF
SF 150.9029181 MHz
WDW
SSB 2
LB 0 Hz
GB 0
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0
ppm

Acyclic Precursor ***S1***

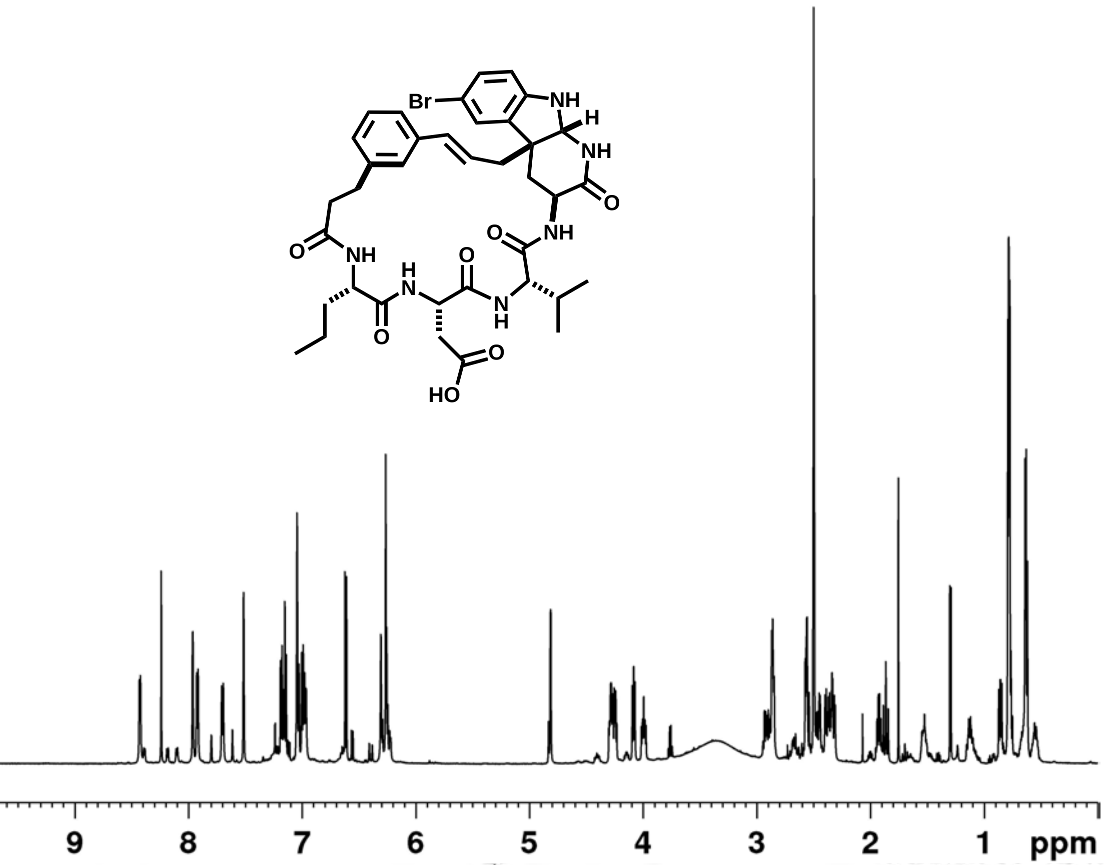
```
Current Data Parameters
NAME              TR6-164
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160219
Time                10.28
INSTRUM             av600
PROBHD   5 mm BB5
PULPROG                zg
TD                  65536
SOLVENT              DMSO
NS                      8
DS                      0
SWH             12376.237 Hz
FIDRES           0.188846 Hz
AQ              2.6476543 sec
RG                     57
DW                 40.400 usec
DE                   6.50 usec
TE                  296.2 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  16.00 usec
PL1                 -1.00 dB
PL1W          31.62277603 W
SFO1          600.1336008 MHz

F2 - Processing parameters
SI                  65536
SF            600.1300057 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```

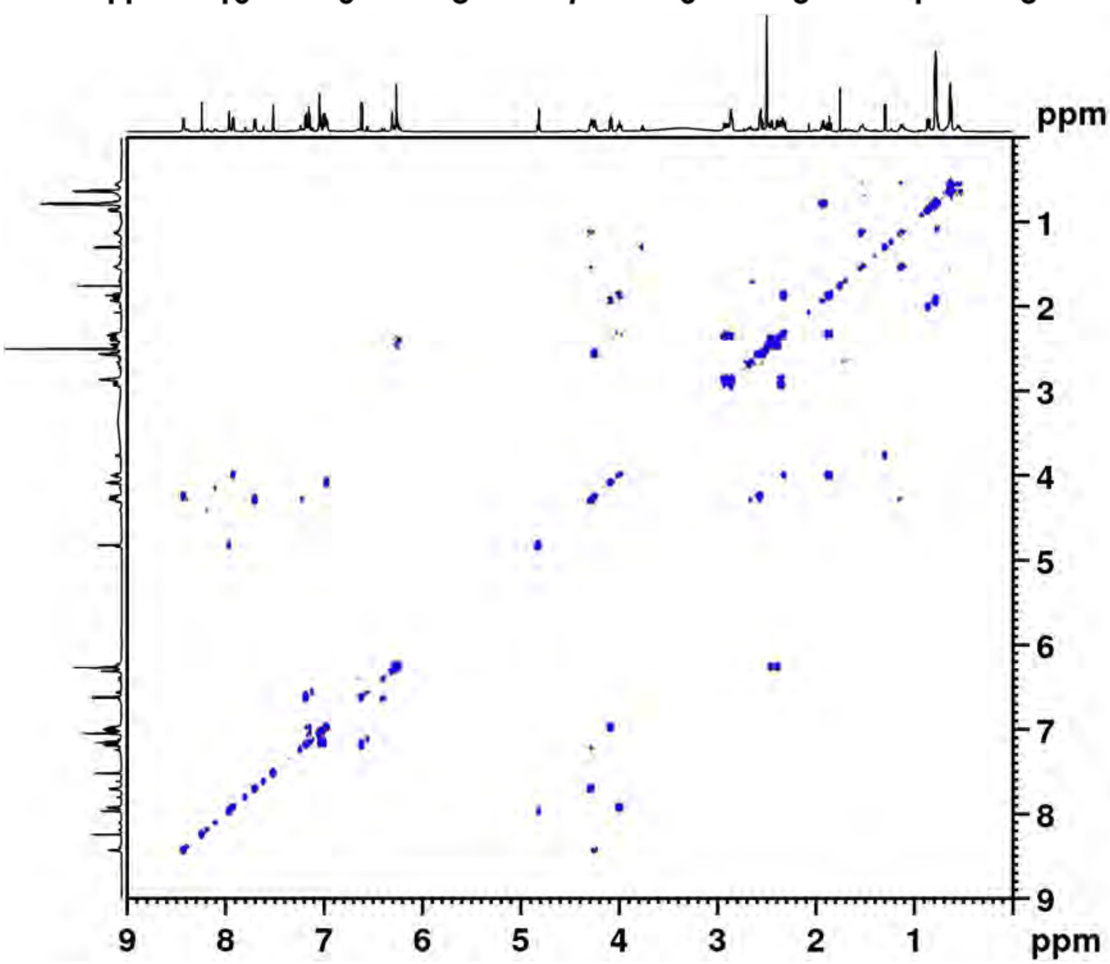




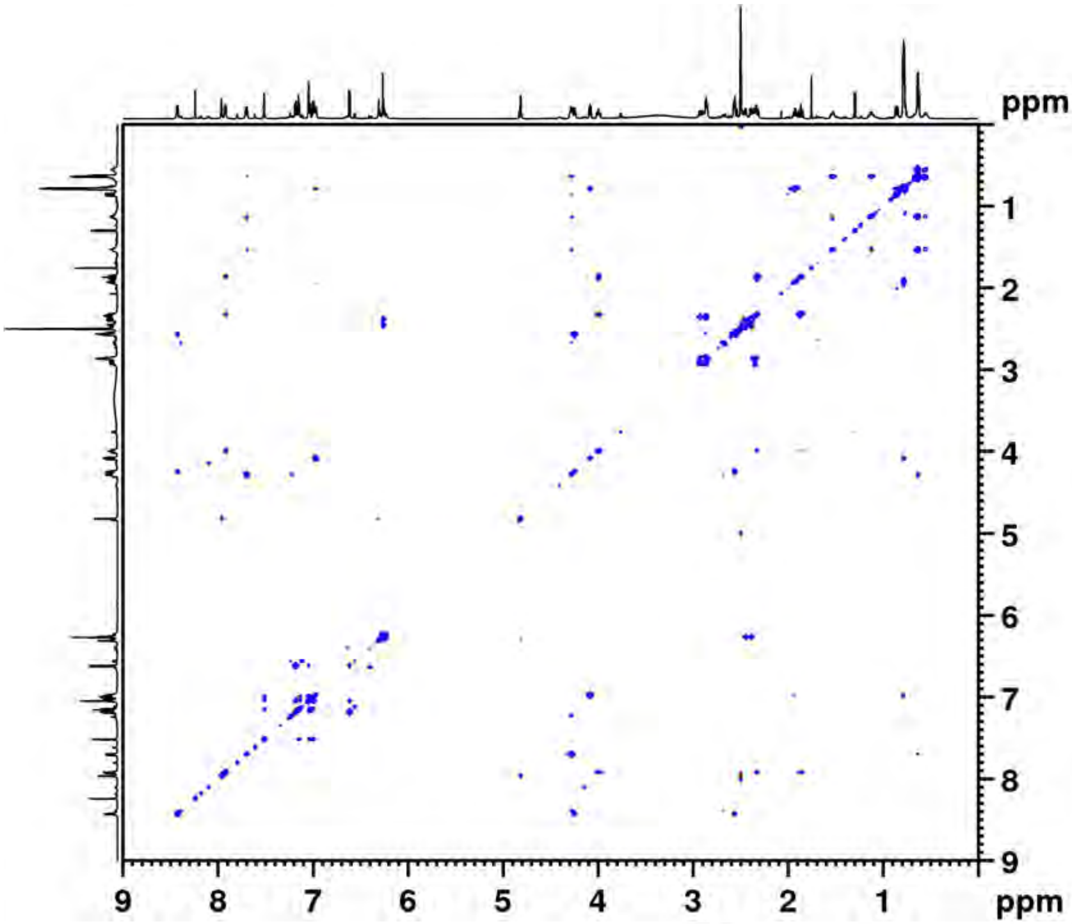
Macrocyclic Product ***S2a***

```
Current Data Parameters
NAME            TR6-186B2
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160210
Time                13.03
INSTRUM             av600
PROBHD   5 mm TBI5
PULPROG                zg
TD                  65536
SOLVENT              DMSO
NS                      8
DS                      0
SWH             12376.237 Hz
FIDRES           0.188846 Hz
AQ              2.6476543 sec
RG                   71.8
DW                 40.400 usec
DE                   6.50 usec
TE                  298.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.05 usec
PL1                 -2.00 dB
PL1W          39.81071854 W
SFO1          600.1336008 MHz

F2 - Processing parameters
SI                  65536
SF            600.1300071 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```






```
Current Data Parameters
NAME            TR6-186B2
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160210
Time                13.05
INSTRUM             av600
PROBHD   5 mm TBI5
PULPROG          cosygpprqf
TD                   2048
SOLVENT              DMSO
NS                      2
DS                     16
SWH              7183.908 Hz
FIDRES           3.507768 Hz
AQ              0.1425408 sec
RG                   35.9
DW                 69.600 usec
DE                   6.50 usec
TE                  298.0 K
D0             0.00000300 sec
D1             1.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00013920 sec

======== CHANNEL f1 ========
NUC1                   1H
P0                   8.00 usec
P1                  10.05 usec
PL1                 -2.00 dB
PL9                120.00 dB
PL1W          39.81071854 W
PL9W     0 W
SFO1          600.1336008 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPX1     0 %
GPY1     0 %
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    512
SFO1             600.1336 MHz
FIDRES          14.031077 Hz
SW                 11.971 ppm
FnMODE                 QF

F2 - Processing parameters
```

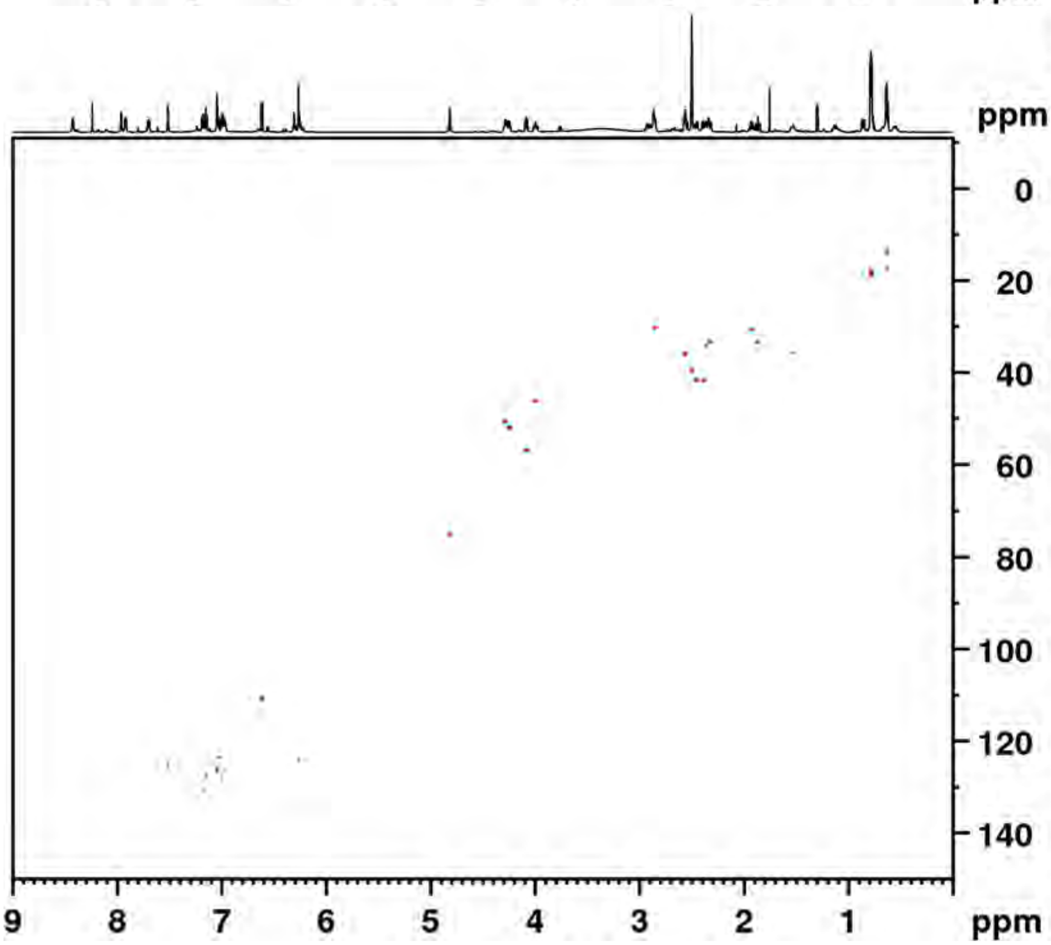


Current Data Parameters
NAME TR6-186B2
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160210
Time 13.26
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevesgpph
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1314816 sec
RG 228.1
DW 64.200 usec
DE 6.50 usec
TE 298.0 K
D0 0.00003780 sec
D1 1.00000000 sec
D9 0.06000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00012840 sec
L1 24

======== CHANNEL f1 ========
NUC1 1H
P1 10.05 usec
P2 20.10 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P12 3000.00 usec
P17 2500.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL10 10.00 dB
PL0W 0 W
PL1W 39.81071854 W
PL10W 2.51188636 W
SFO1 600.1339008 MHz
SP1 120.00 dB
SPNAM[1] Squa100.1000
SPOAL1 1.000
SPOFFS1 -1456.44 Hz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100

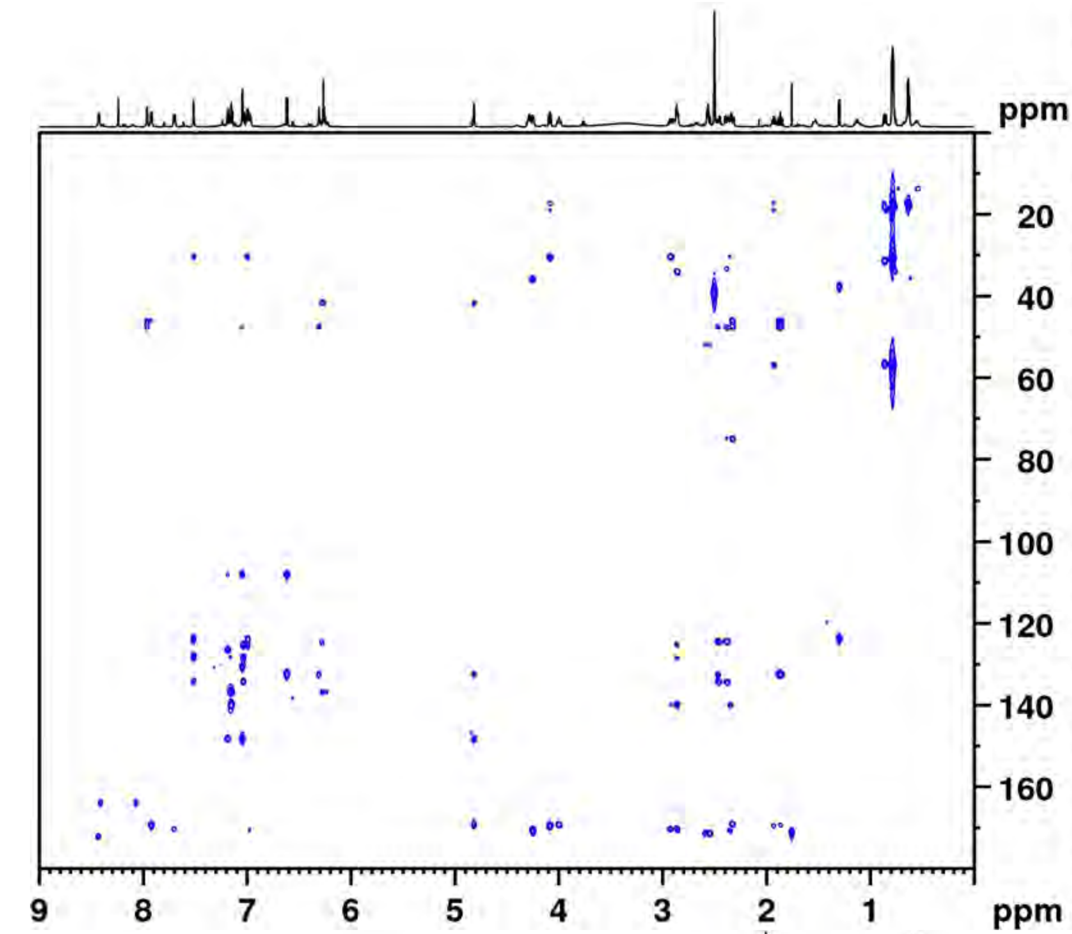


Current Data Parameters
NAME TR6-186B2
EXPNO 8
PROCNO 1

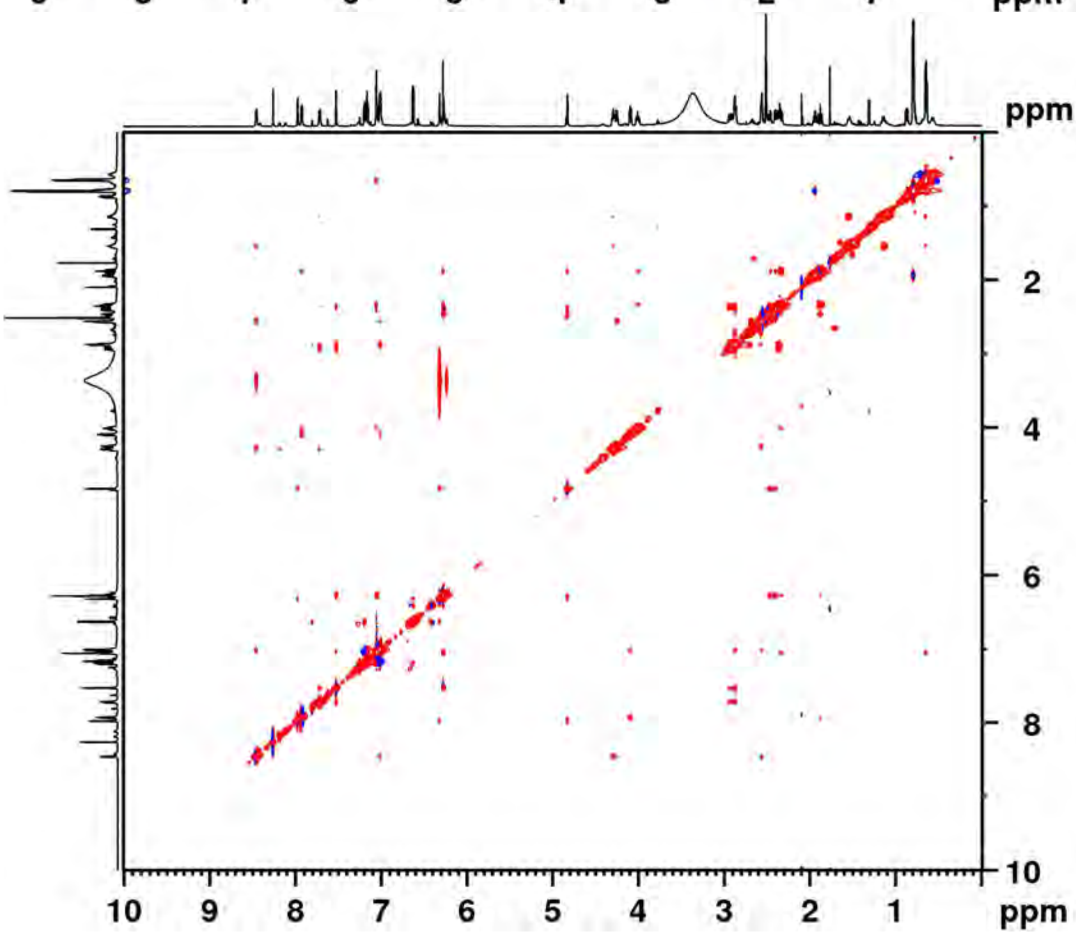
F2 - Acquisition Parameters
Date_ 20160210
Time 13.47
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 6
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1314816 sec
RG 23170.5
DW 64.200 usec
DE 6.00 usec
TE 298.3 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1 1H
P1 10.05 usec
P2 20.10 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz

======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 19.50 usec
P4 39.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.46 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 13.52450085 W
SFO2 150.9133722 MHz



Current Data Parameters	
NAME	TR6-186B2
EXPNO	9
PROCNO	1
F2 - Acquisition Parameters	
Date_	20160210
Time	14.23
INSTRUM	av600
PROBHD	5 mm TBI5
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	DMSO
NS	24
DS	16
SWH	7788.162 Hz
FIDRES	3.802814 Hz
AQ	0.1314816 sec
RG	26008
DW	64.200 usec
DE	6.00 usec
TE	297.5 K
CNST2	145.0000000
CNST13	7.0000000
D0	0.00000300 sec
D1	1.50000000 sec
D2	0.00344828 sec
D6	0.07142857 sec
D16	0.00020000 sec
IN0	0.00001745 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.57 usec
P2	21.14 usec
PL1	-2.00 dB
PL1W	39.81071854 W
SFO1	600.1339008 MHz
======== CHANNEL f2 ========	
NUC2	13C
P3	19.50 usec
PL2	-3.00 dB
PL2W	150.35617065 W
SFO2	150.9156357 MHz
====== GRADIENT CHANNEL =====	
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPNAM[3]	SINE.100
GPX1	0 %
GPX2	0 %
GPX3	0 %
GPY1	0 %
GPY2	0 %



Current Data Parameters	
NAME	TR6-186B2
EXPNO	13
PROCNO	1
F2 - Acquisition Parameters	
Date_	20160211
Time	8.54
INSTRUM	av600
PROBHD	5 mm TBI5
PULPROG	noesyesgpph
TD	2048
SOLVENT	DMSO
NS	8
DS	16
SWH	6009.615 Hz
FIDRES	2.934382 Hz
AQ	0.1703936 sec
RG	512
DW	83.200 usec
DE	6.50 usec
TE	298.0 K
D0	0.00007010 sec
D1	1.50000000 sec
D8	0.30000001 sec
D11	0.03000000 sec
D12	0.00002000 sec
D16	0.00020000 sec
IN0	0.00016665 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.38 usec
P2	20.76 usec
P12	3000.00 usec
PL0	120.00 dB
PL1	-2.00 dB
PL0W	0 W
PL1W	39.81071854 W
SFO1	600.1330006 MHz
SP1	41.20 dB
SPNAM[1]	Squa100.1000
SPOAL1	1.000
SPOFFS1	-977.55 Hz
====== GRADIENT CHANNEL =====	
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPX1	0 %
GPX2	0 %
GPY1	0 %
GPY2	0 %
GPZ1	31.00 %
GPZ2	11.00 %

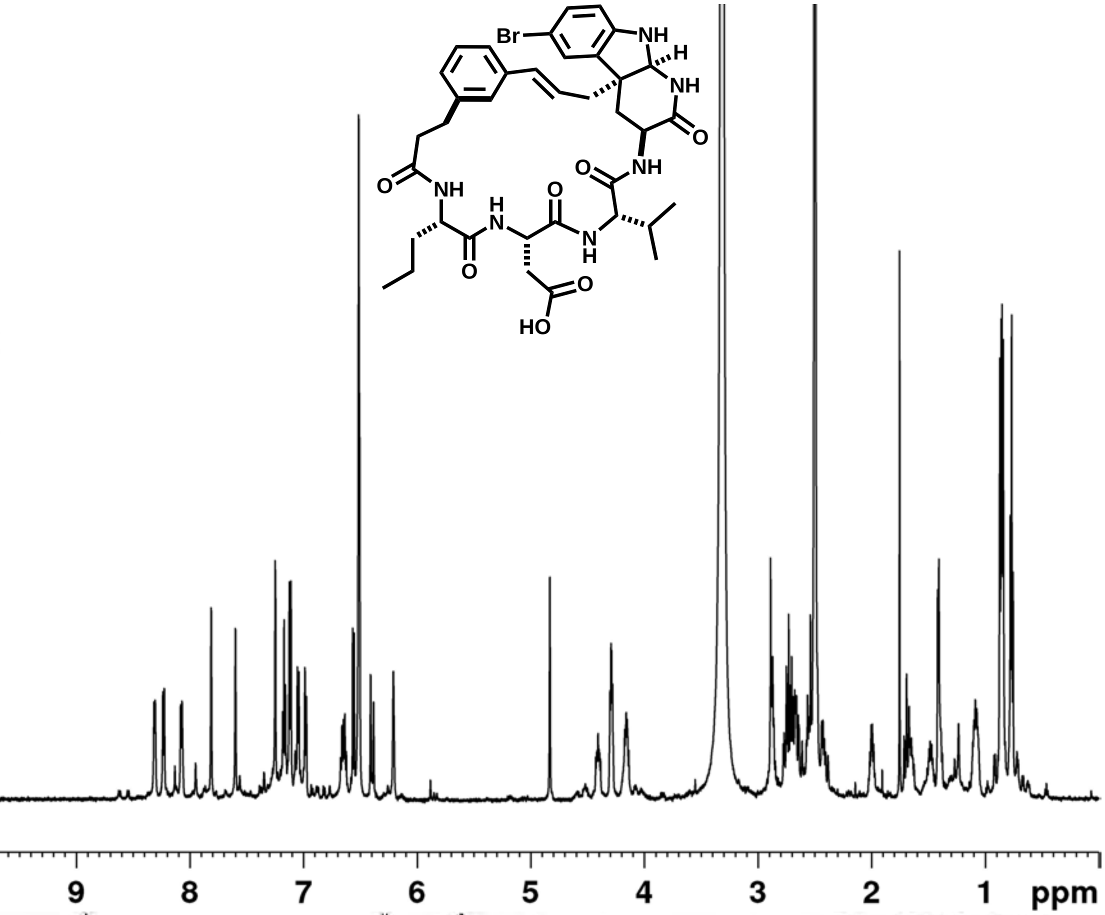
Macrocyclic Product ***S2b***

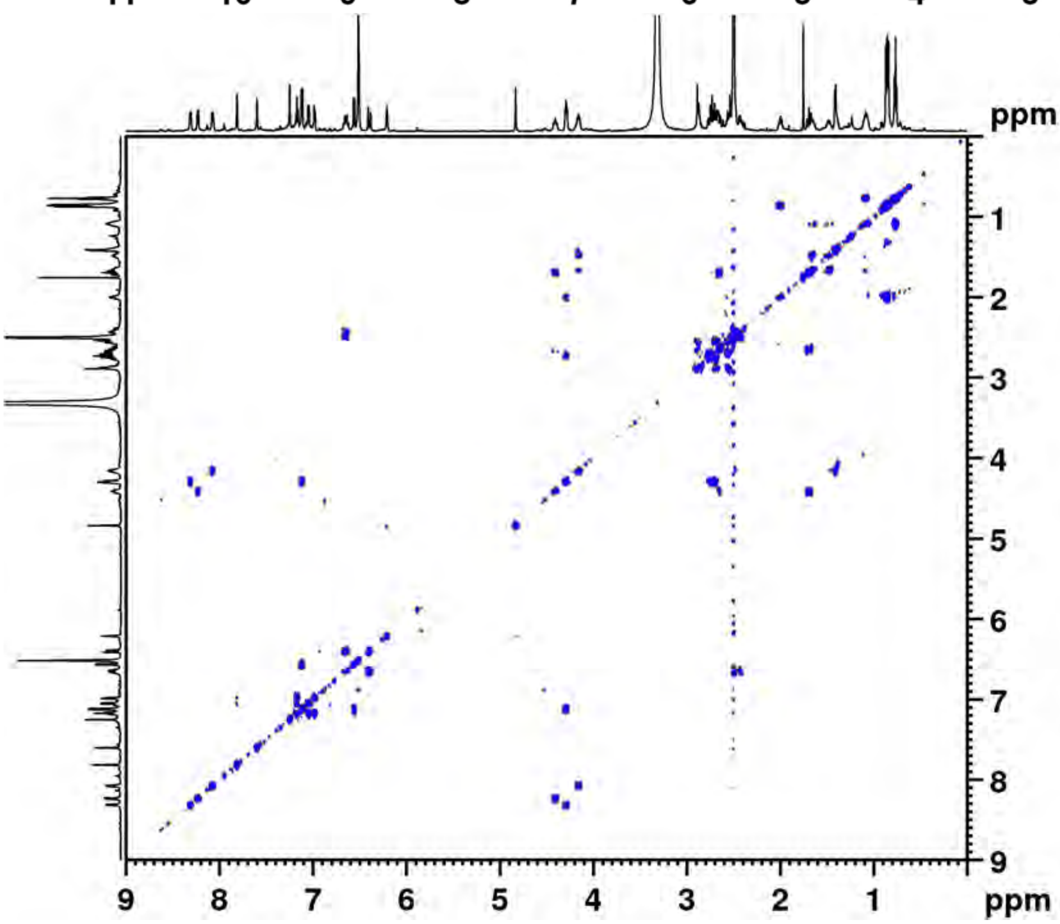
Current Data Parameters
NAME TR6-186B1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160210
Time 16.32
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG zg
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6476543 sec
RG 71.8
DW 40.400 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.32 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1336008 MHz

F2 - Processing parameters
SI 65536
SF 600.1300072 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





Current Data Parameters
NAME TR6-186B1
EXPNO 6
PROCNO 1

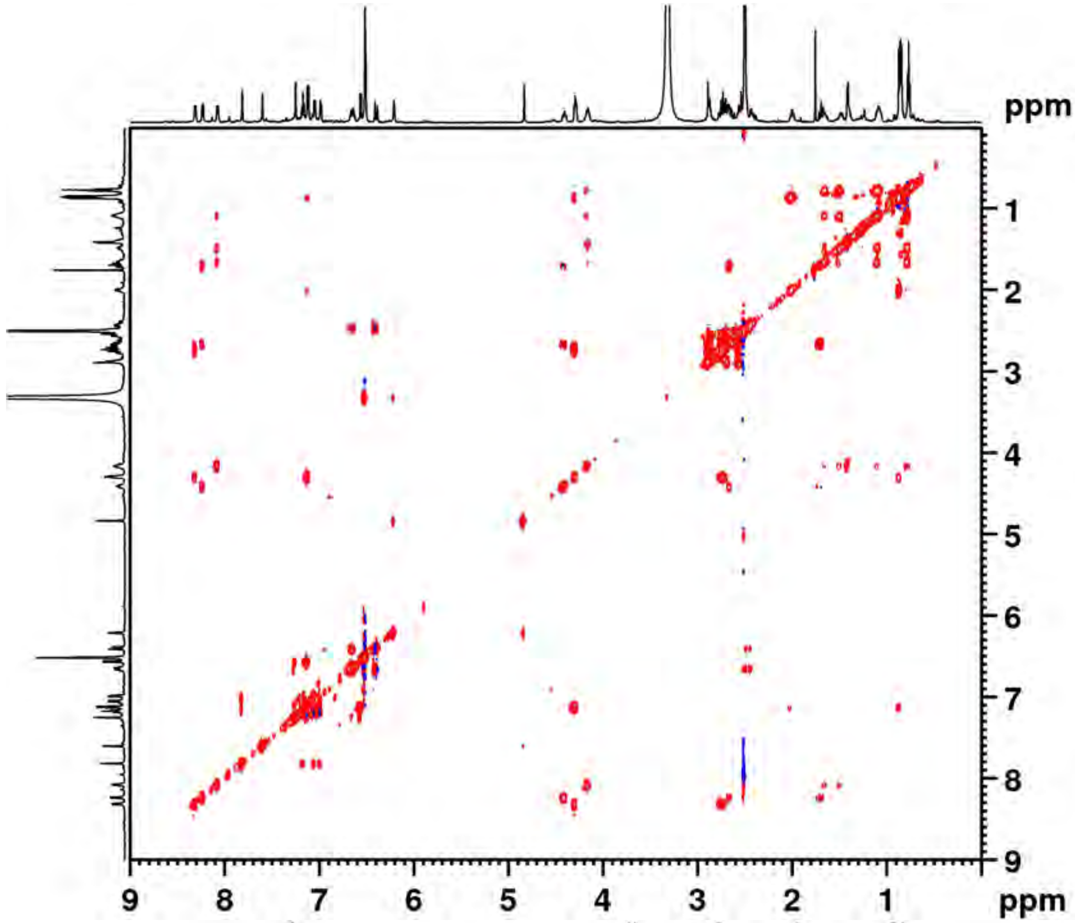
F2 - Acquisition Parameters
Date_ 20160210
Time 16.36
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG cosygpprqf
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425408 sec
RG 2048
DW 69.600 usec
DE 6.50 usec
TE 298.0 K
D0 0.00000300 sec
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00013920 sec

======== CHANNEL f1 ========
NUC1 1H
P0 8.00 usec
P1 10.32 usec
PL1 -2.00 dB
PL9 51.71 dB
PL1W 39.81071854 W
PL9W 0.00016943 W
SFO1 600.1319971 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPX1 0 %
GPY1 0 %
GPZ1 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 161
SFO1 600.132 MHz
FIDRES 44.620449 Hz
SW 11.971 ppm
FnMODE QF

F2 - Processing parameters

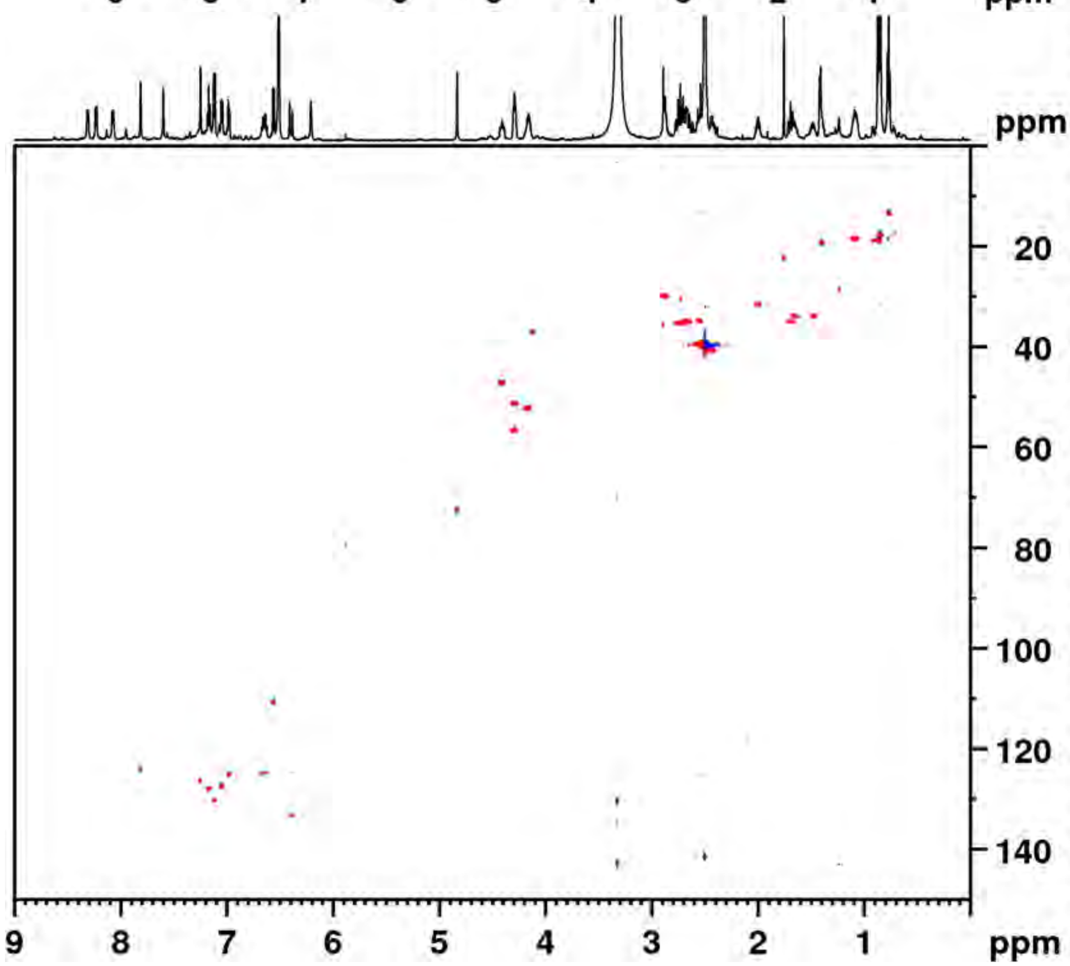


Current Data Parameters
NAME TR6-186B1
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160210
Time 16.50
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevesgpph
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1314816 sec
RG 2896.3
DW 64.200 usec
DE 6.50 usec
TE 298.0 K
D0 0.00003763 sec
D1 1.00000000 sec
D9 0.06000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00012840 sec
L1 24

======== CHANNEL f1 ========
NUC1 1H
P1 10.32 usec
P2 20.64 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P12 3000.00 usec
P17 2500.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL10 9.77 dB
PL0W 0 W
PL1W 39.81071854 W
PL10W 2.64849997 W
SFO1 600.1339008 MHz
SP1 41.24 dB
SPNAM[1] Squa100.1000
SPOAL1 1.000
SPOFFS1 -1903.74 Hz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100

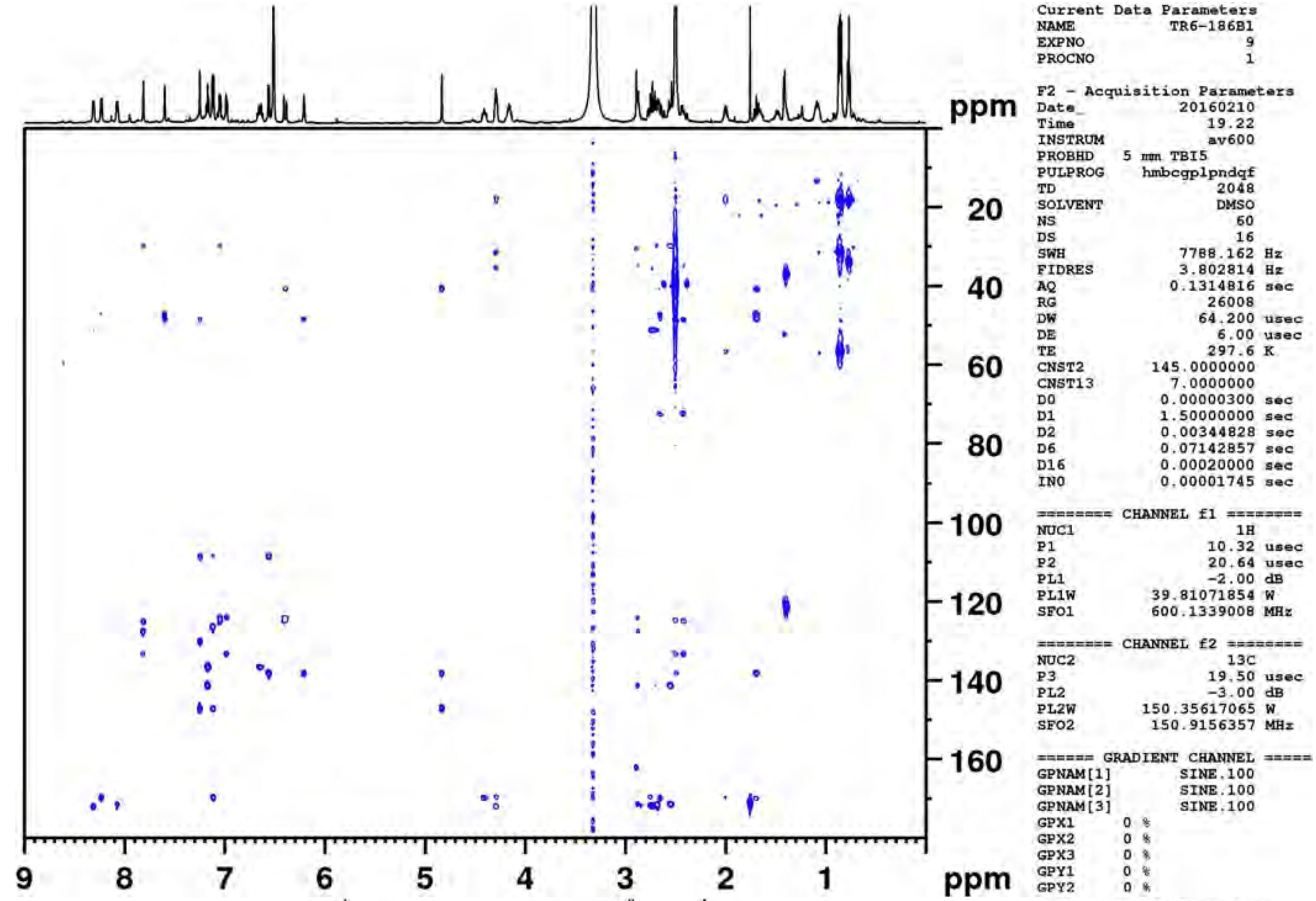


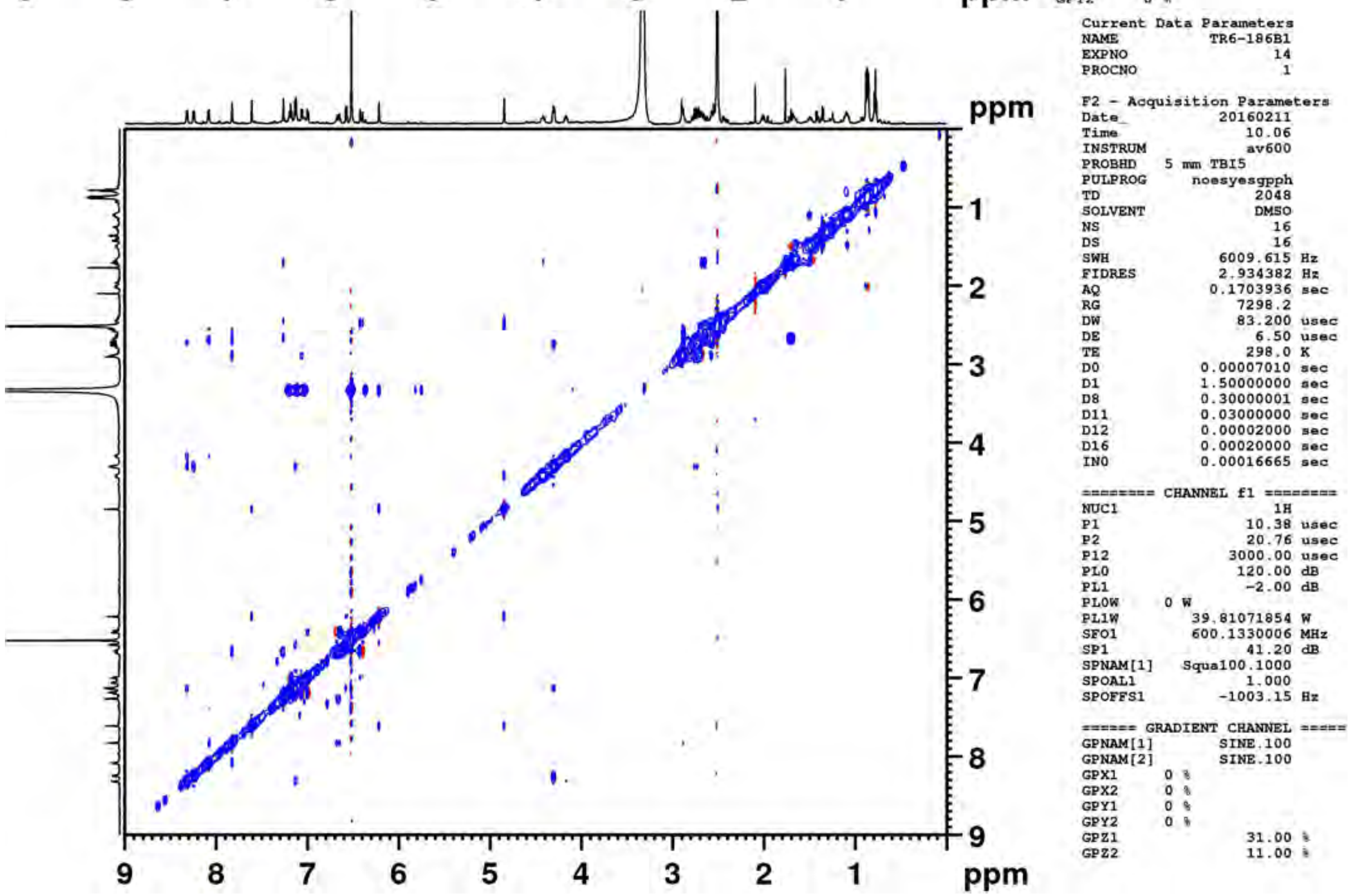
Current Data Parameters
NAME TR6-186B1
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160210
Time 17.02
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 24
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1314816 sec
RG 23170.5
DW 64.200 usec
DE 6.00 usec
TE 298.4 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS

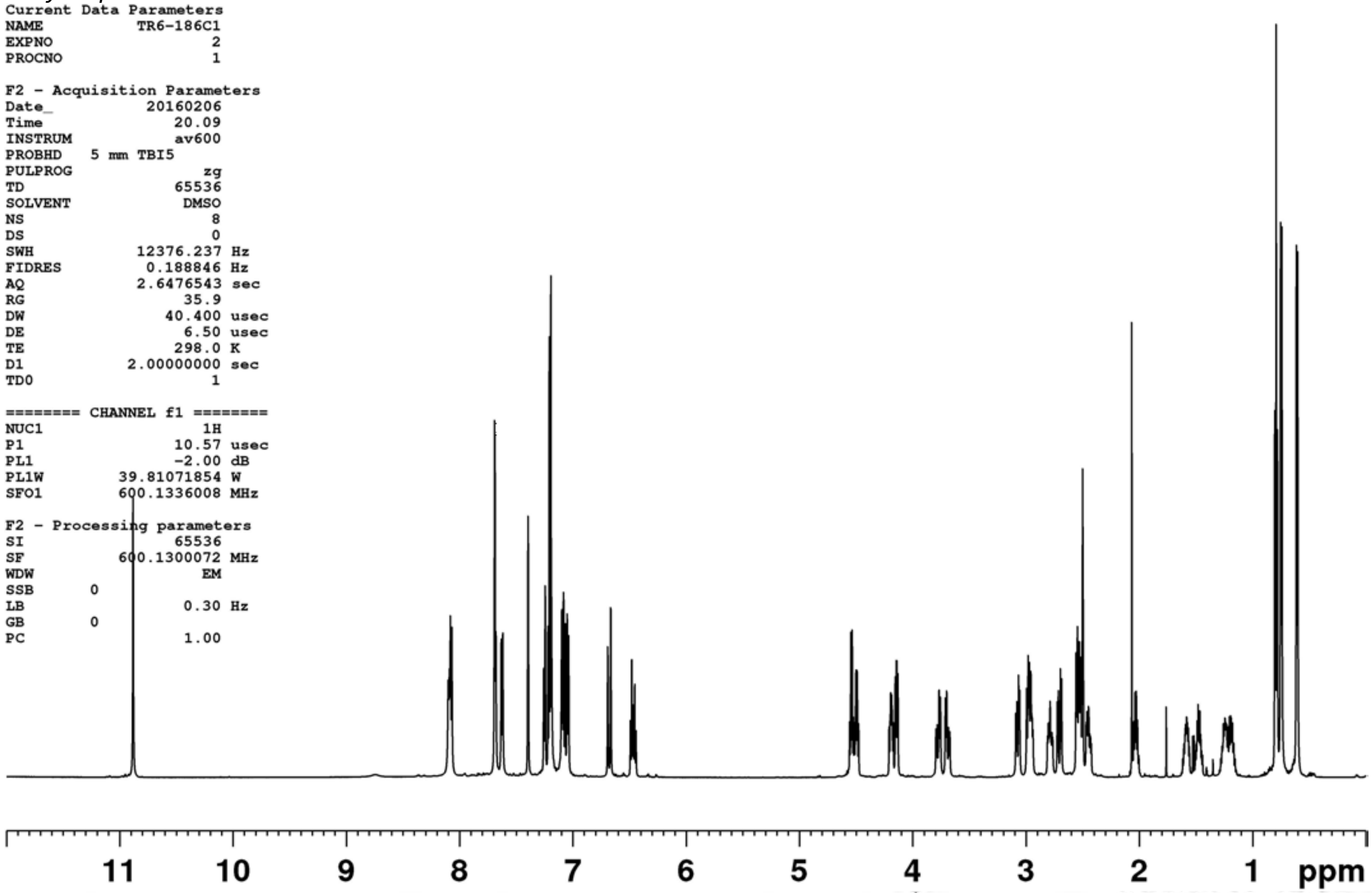
======== CHANNEL f1 ========
NUC1 1H
P1 10.32 usec
P2 20.64 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz

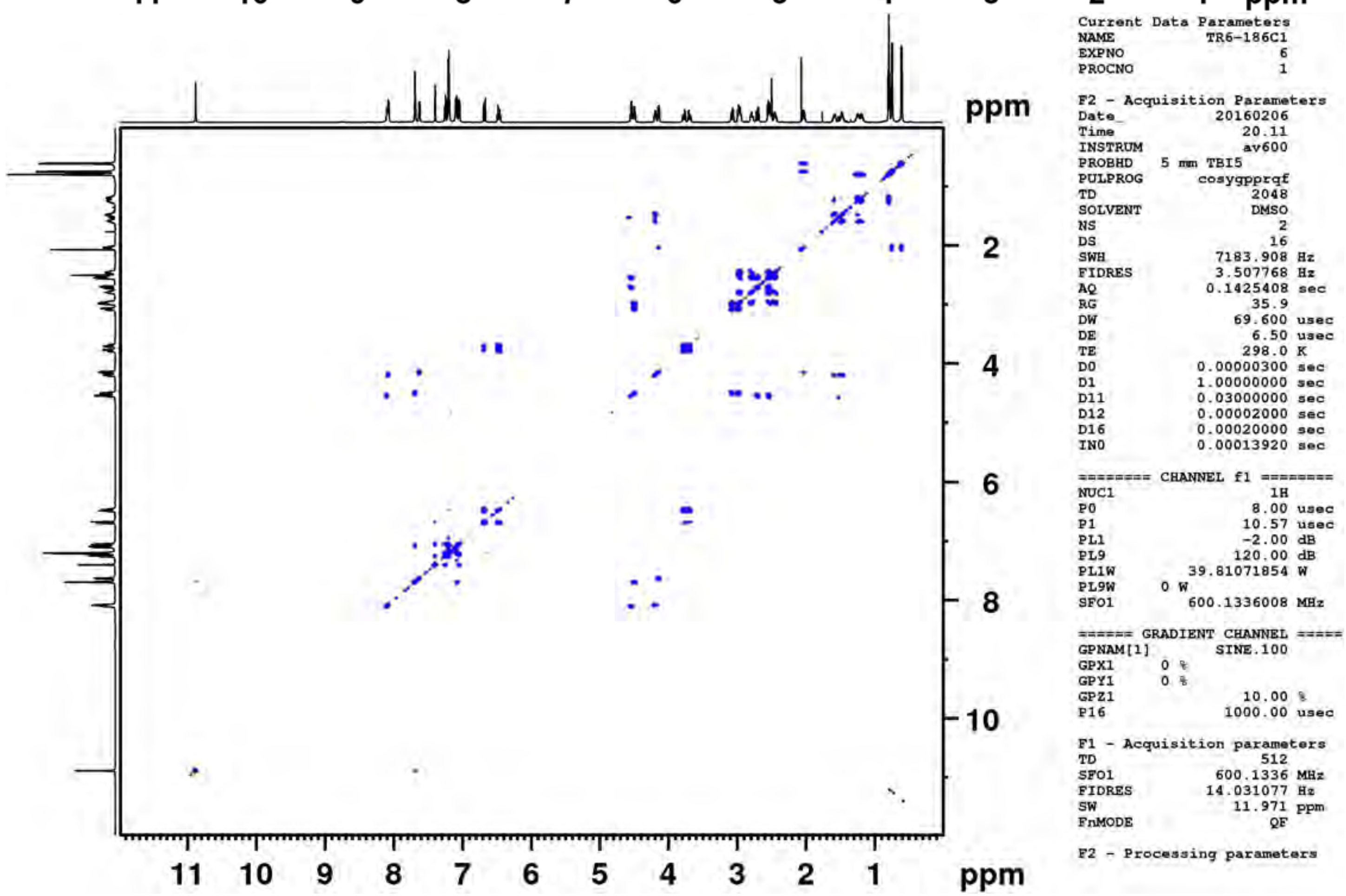
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 19.50 usec
P4 39.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.46 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 13.52450085 W
SFO2 150.9133722 MHz

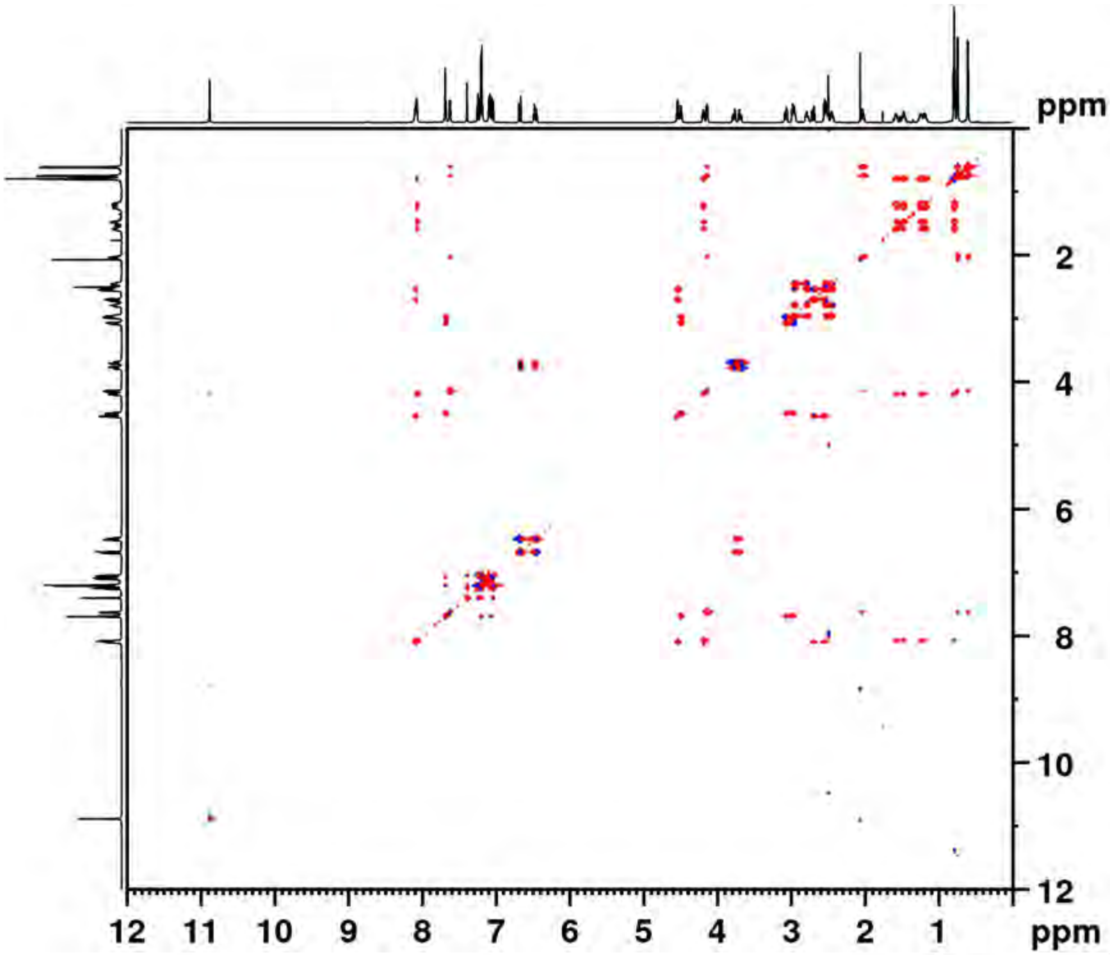

ppm
20
40
60
80
100
120
140
160
9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME TR6-186B1
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160210
Time 19.22
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 60
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1314816 sec
RG 26008
DW 64.200 usec
DE 6.00 usec
TE 297.6 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.32 usec
P2 20.64 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz
======== CHANNEL f2 ========
NUC2 13C
P3 19.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %


ppm
1
2
3
4
5
6
7
8
9
9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME TR6-186B1
EXPNO 14
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160211
Time 10.06
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG noesyesgpph
TD 2048
SOLVENT DMSO
NS 16
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 7298.2
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D0 0.00007010 sec
D1 1.50000000 sec
D8 0.30000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00016665 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.38 usec
P2 20.76 usec
P12 3000.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL0W 0 W
PL1W 39.81071854 W
SFO1 600.1330006 MHz
SP1 41.20 dB
SPNAM[1] Squa100.1000
SPOAL1 1.000
SPOFFS1 -1003.15 Hz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 31.00 %
GPZ2 11.00 %

Macrocyclic product ***S2c***





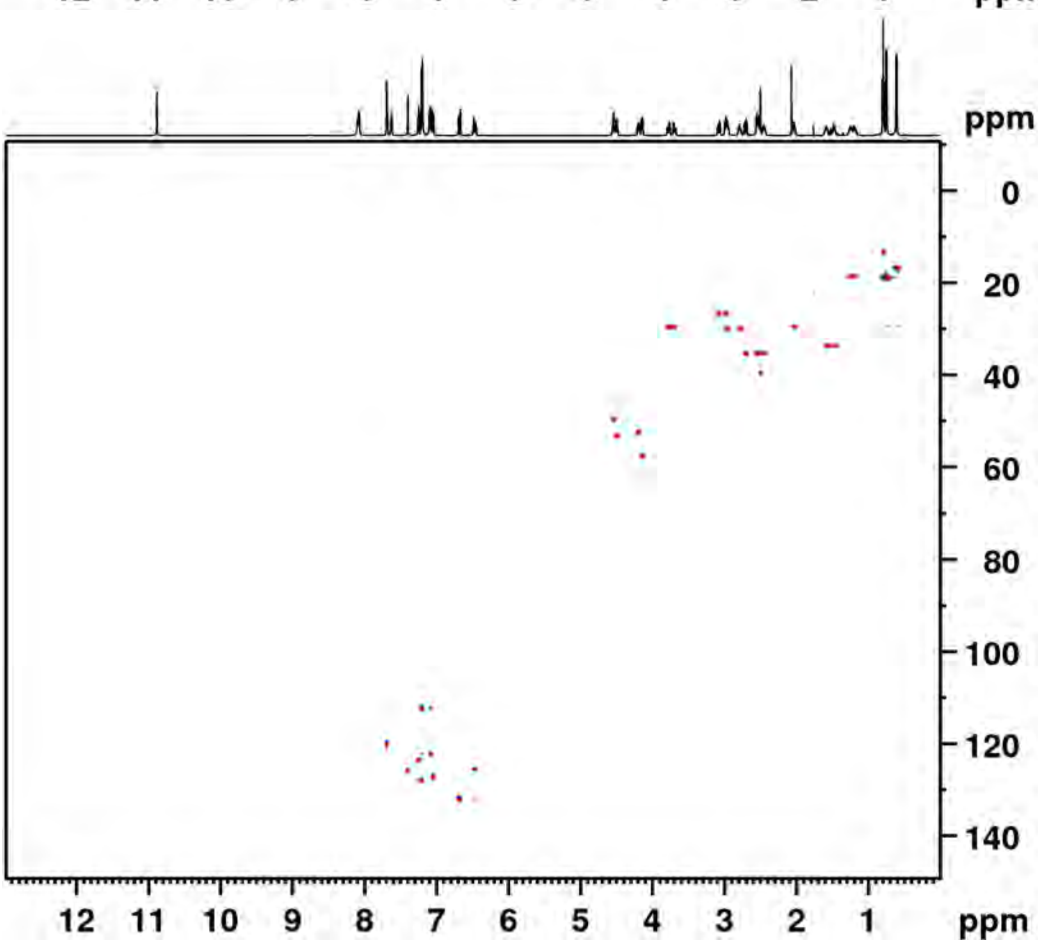


Current Data Parameters
NAME TR6-186C1
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160206
Time 20.32
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevesgpph
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1314816 sec
RG 228.1
DW 64.200 usec
DE 6.50 usec
TE 298.0 K
D0 0.00003747 sec
D1 1.00000000 sec
D9 0.06000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00012840 sec
L1 24

======== CHANNEL f1 ========
NUC1 1H
P1 10.57 usec
P2 21.14 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P12 3000.00 usec
P17 2500.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL10 9.56 dB
PL0W 0 W
PL1W 39.81071854 W
PL10W 2.77971292 W
SFO1 600.1339008 MHz
SP1 120.00 dB
SPNAM[1] Squa100.1000
SPOAL1 1.000
SPOFFS1 -1456.44 Hz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100

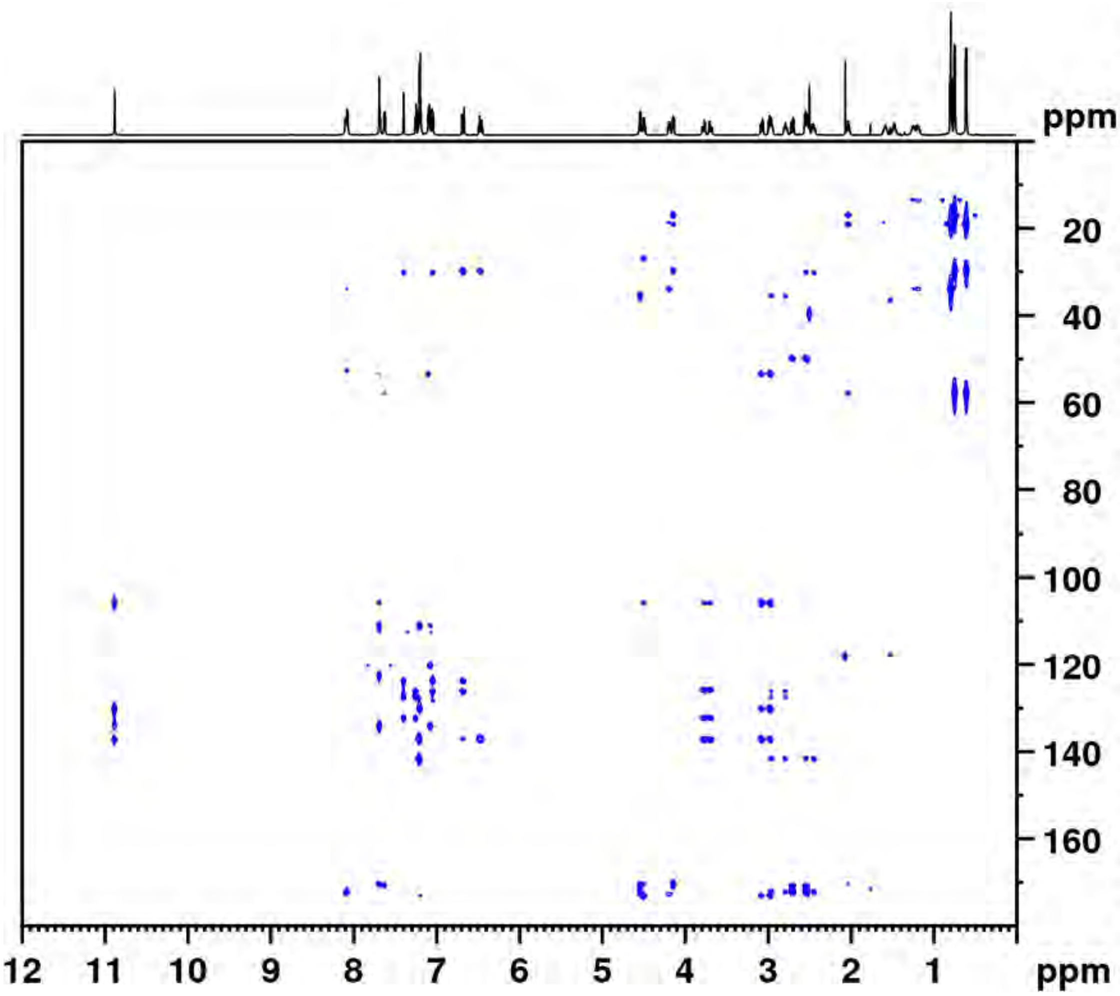


Current Data Parameters
NAME TR6-186C1
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160206
Time 20.54
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1314816 sec
RG 23170.5
DW 64.200 usec
DE 6.00 usec
TE 298.3 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1 1H
P1 10.57 usec
P2 21.14 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz

======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 19.50 usec
P4 39.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.46 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 13.52450085 W
SFO2 150.9133722 MHz



Current Data Parameters
NAME TR6-186C1
EXPNO 9
PROCNO 1

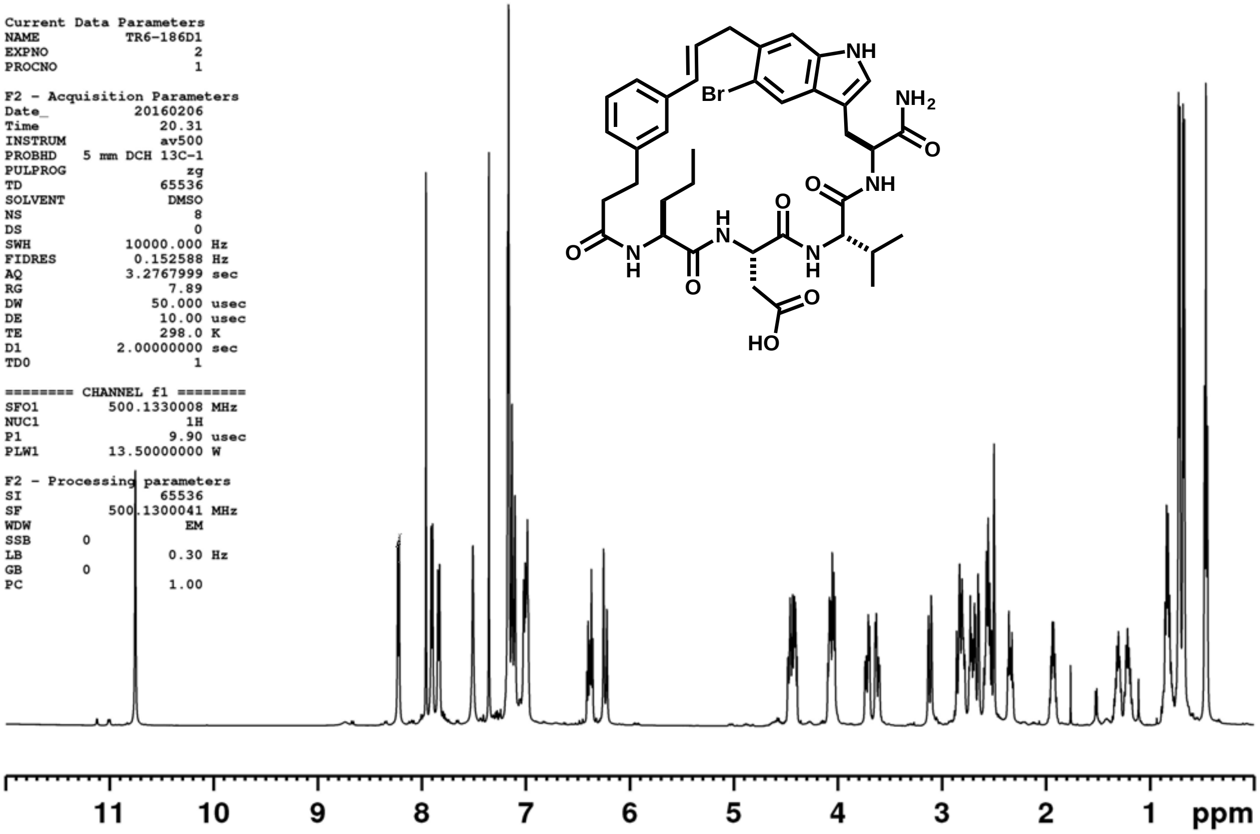
F2 - Acquisition Parameters
Date_ 20160206
Time 21.18
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 18
DS 16
SWH 7788.162 Hz
FIDRES 3.802814 Hz
AQ 0.1314816 sec
RG 26008
DW 64.200 usec
DE 6.00 usec
TE 297.5 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec

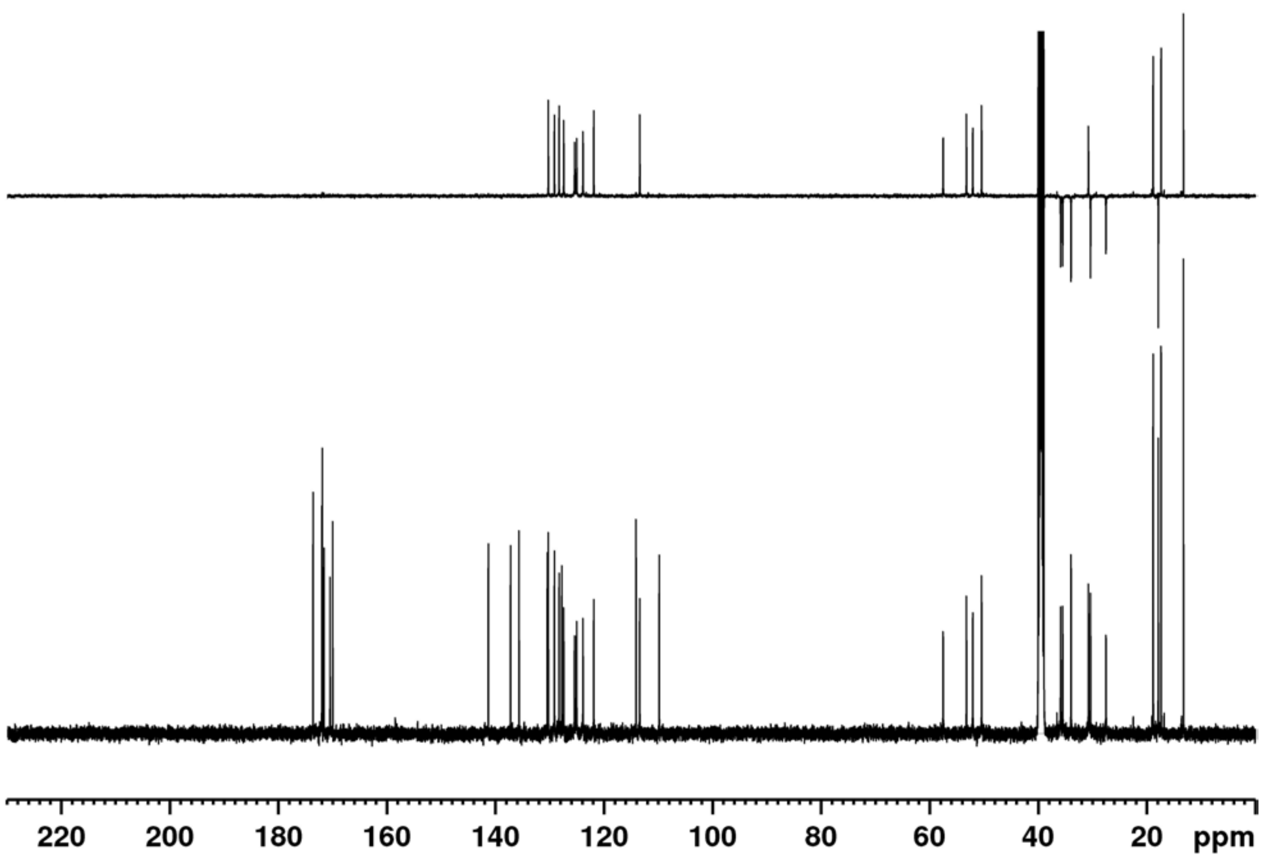
======== CHANNEL f1 ========
NUC1 1H
P1 10.57 usec
P2 21.14 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1339008 MHz

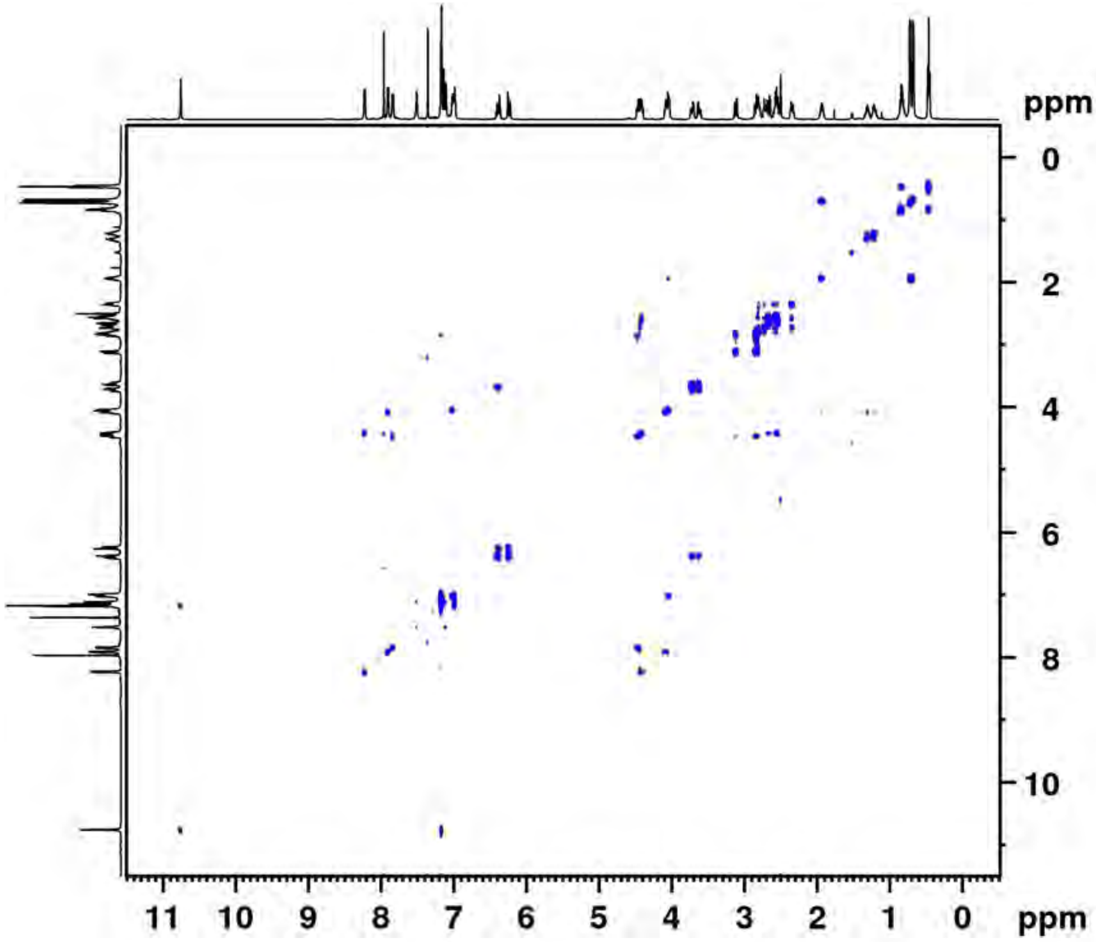
======== CHANNEL f2 ========
NUC2 13C
P3 19.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %

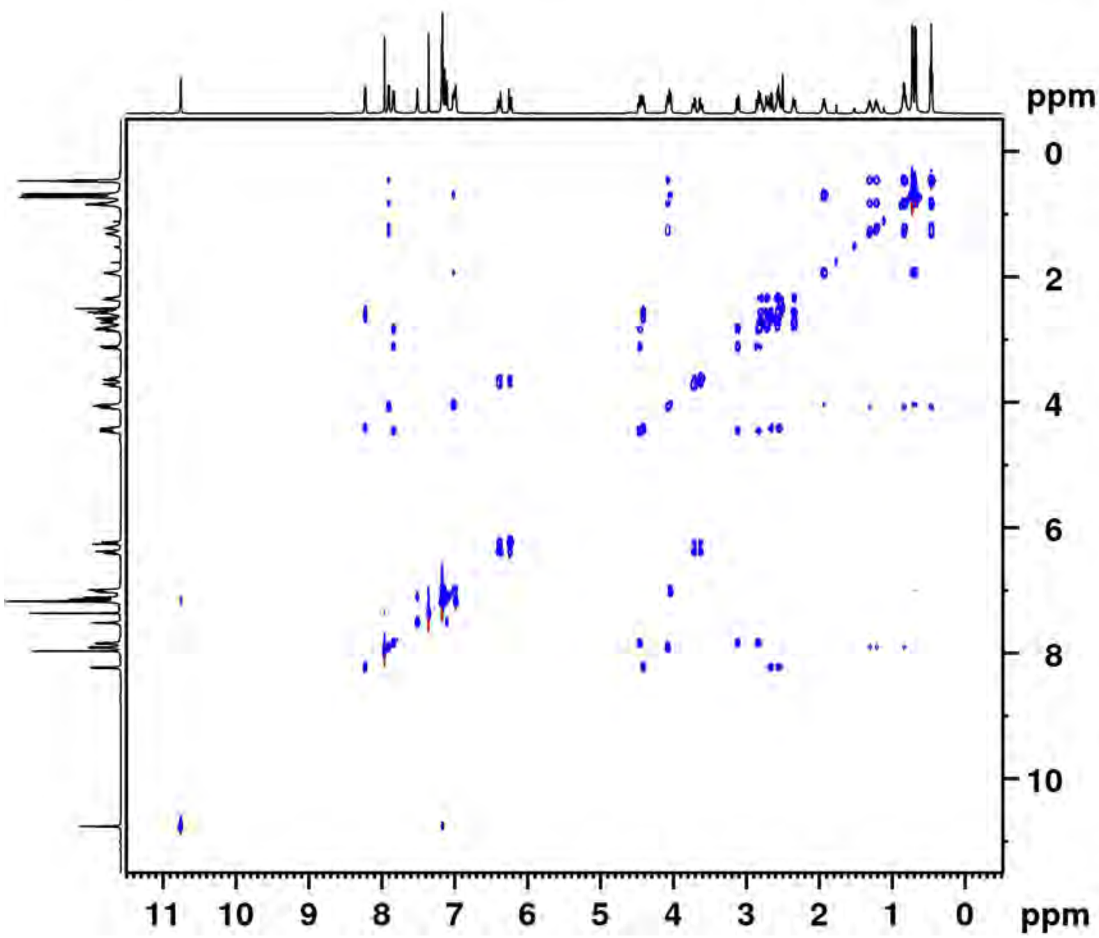
Macrocyclic Product ***S2d***



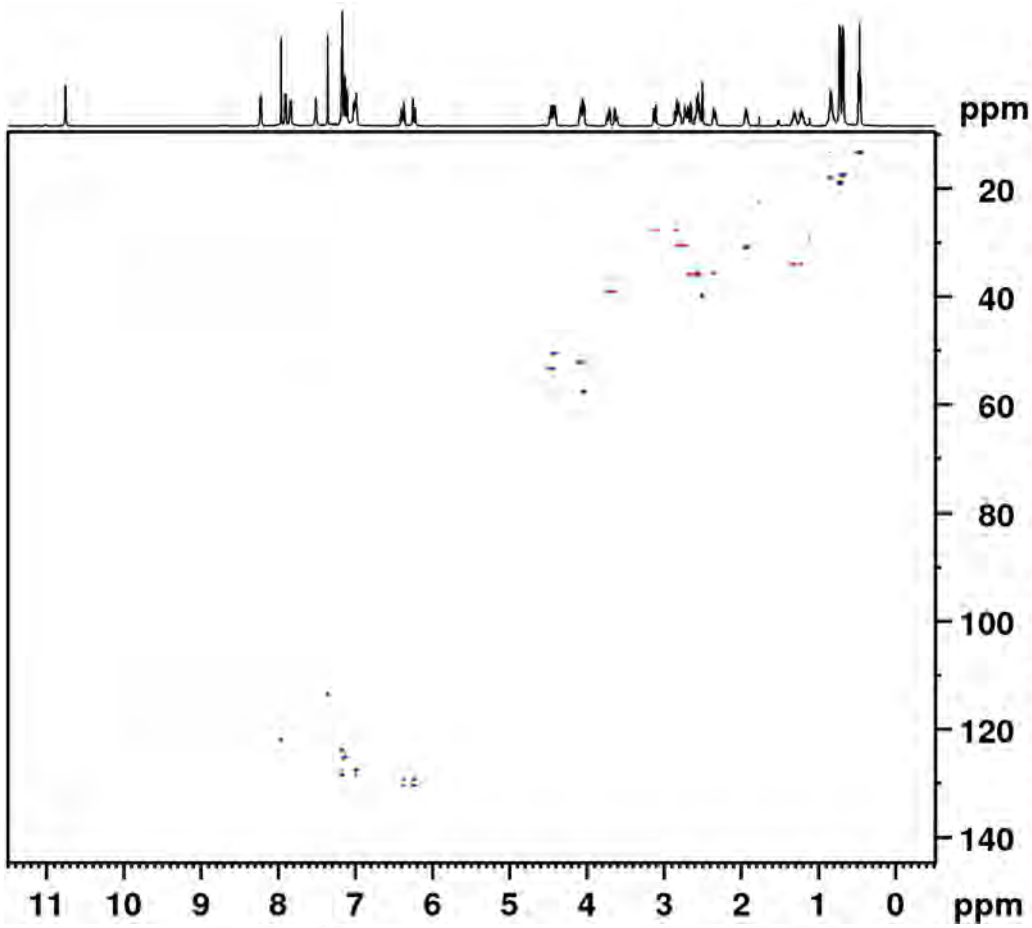





```
Current Data Parameters
NAME            TR6-186D1
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160206
Time                20.37
INSTRUM             av500
PROBHD   5 mm DCH 13C-1
PULPROG       cosygpmfph
TD                   2048
SOLVENT              DMSO
NS                      2
DS                      8
SWH              6009.615 Hz
FIDRES           2.934382 Hz
AQ              0.1703936 sec
RG                   7.89
DW                 83.200 usec
DE                  10.00 usec
TE                  298.0 K
D0             0.00007059 sec
D1             1.20000005 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00016640 sec

======== CHANNEL f1 ========
SFO1          500.1327507 MHz
NUC1                   1H
P1                   9.90 usec
P2                  19.80 usec
PLW1          13.50000000 W

====== GRADIENT CHANNEL =====
GPNAM[1]        SMSQ10.100
GPNAM[2]        SMSQ10.100
GPZ1                10.00 %
GPZ2                20.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    512
SFO1             500.1328 MHz
FIDRES          11.737530 Hz
SW                 12.016 ppm
FnMODE        States-TPPI

F2 - Processing parameters
SI                   4096
SF            500.1300028 MHz
WDW                 QSINE
SSB                     1
```




```
Current Data Parameters
NAME            TR6-186D1
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160206
Time                21.01
INSTRUM             av500
PROBHD   5 mm DCH 13C-1
PULPROG      mlevetgp.js
TD                   2048
SOLVENT              DMSO
NS                      2
DS                     16
SWH              6009.615 Hz
FIDRES           2.934382 Hz
AQ              0.1703936 sec
RG                  59.34
DW                 83.200 usec
DE                  10.00 usec
TE                  298.0 K
D0             0.00000300 sec
D1             1.50000000 sec
D9             0.06000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00016640 sec
L1                     24

======== CHANNEL f1 ========
SFO1          500.1327507 MHz
NUC1                   1H
P1                   9.90 usec
P2                  19.80 usec
P5                  26.68 usec
P6                  40.00 usec
P7                  80.00 usec
P17               2500.00 usec
PLW1          13.50000000 W
PLW10          0.92682981 W

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPZ1                30.00 %
GPZ2                30.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    256
SFO1             500.1328 MHz
FIDRES          23.475060 Hz
```

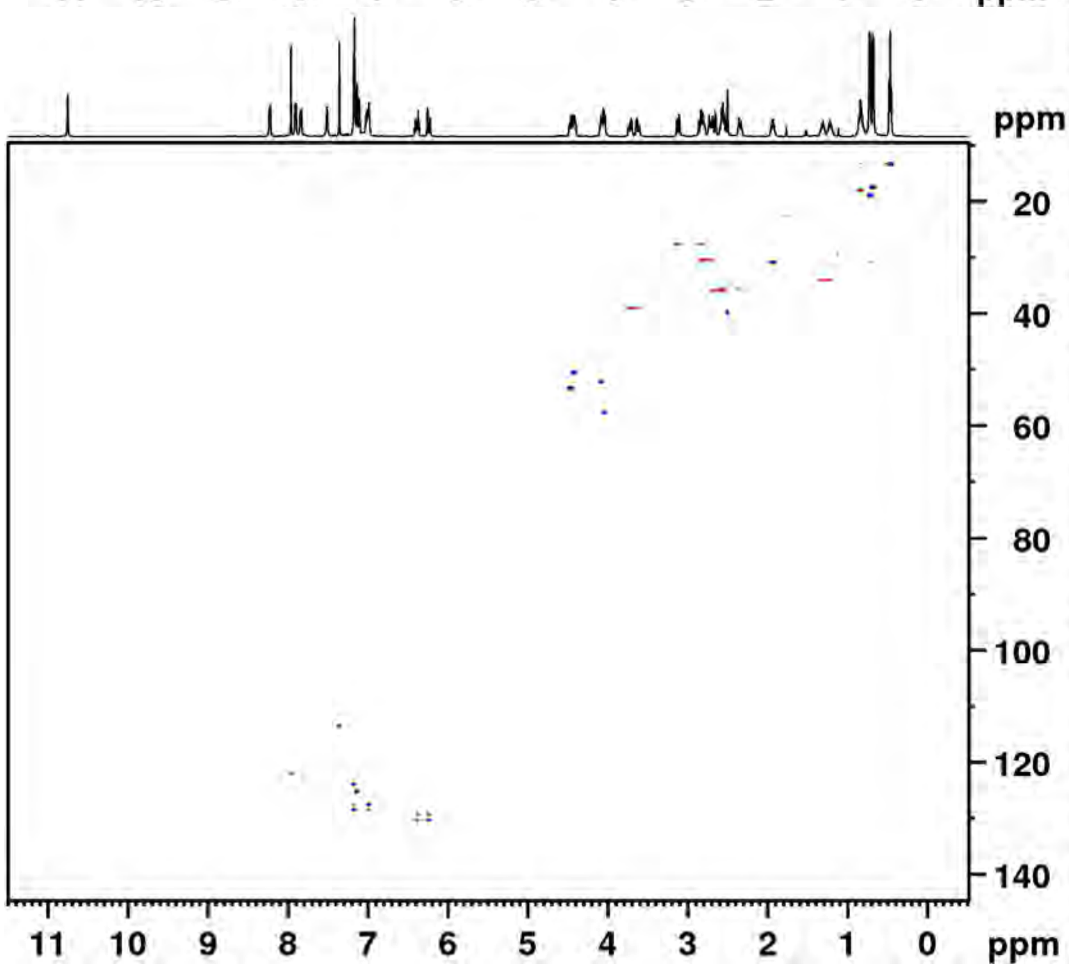


Current Data Parameters
NAME TR6-186D1
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160206
Time 21.17
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcetgpsisp.2
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 204.54
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
CNST17 -0.5000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00345000 sec
IN0 0.00002940 sec

======== CHANNEL f1 ========
SFO1 500.1327507 MHz
NUC1 1H
P1 9.90 usec
P2 19.80 usec
P28 1000.00 usec
PLW1 13.50000000 W

======== CHANNEL f2 ========
SFO2 125.7675352 MHz
NUC2 13C
CPDPRG[2 garp
P3 9.63 usec
P14 500.00 usec
P24 2000.00 usec
PCPD2 70.00 usec
PLW0 0 W
PLW2 23.01399994 W
PLW12 0.43557000 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3 0.500
SPOFFS3 0 Hz
SPW3 3.26090002 W



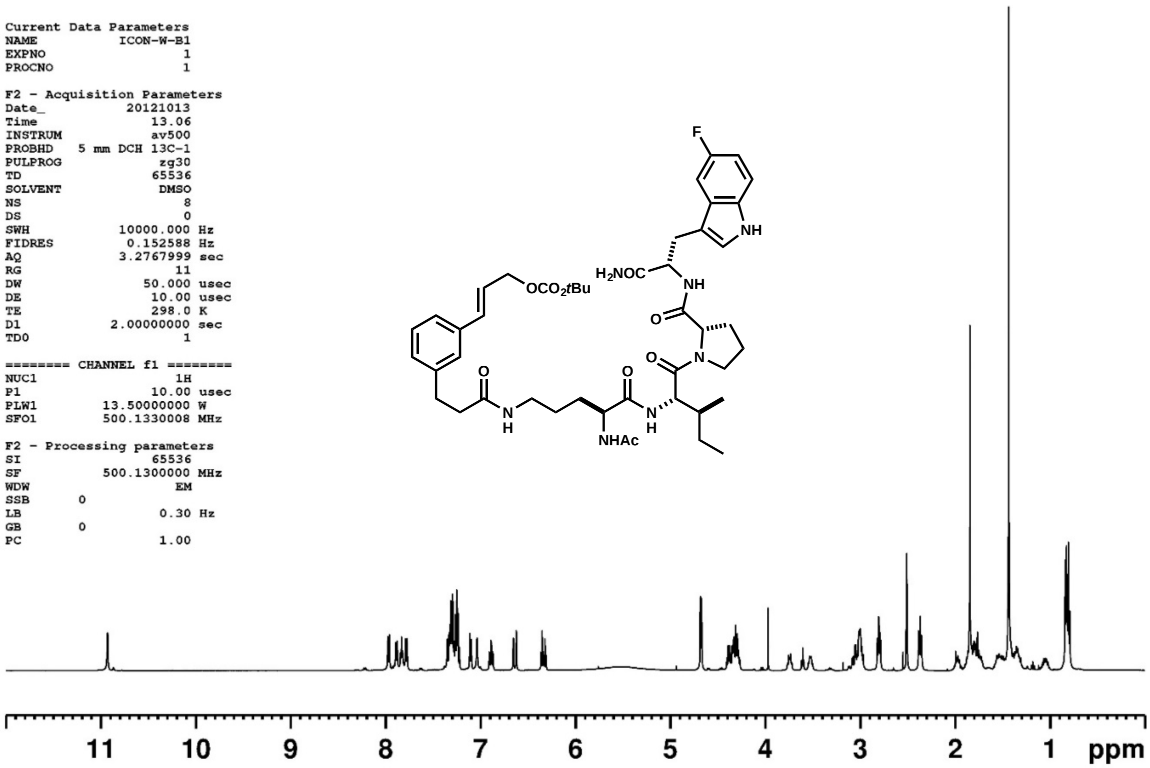
Current Data Parameters
NAME TR6-186D1
EXPNO 7
PROCNO 1

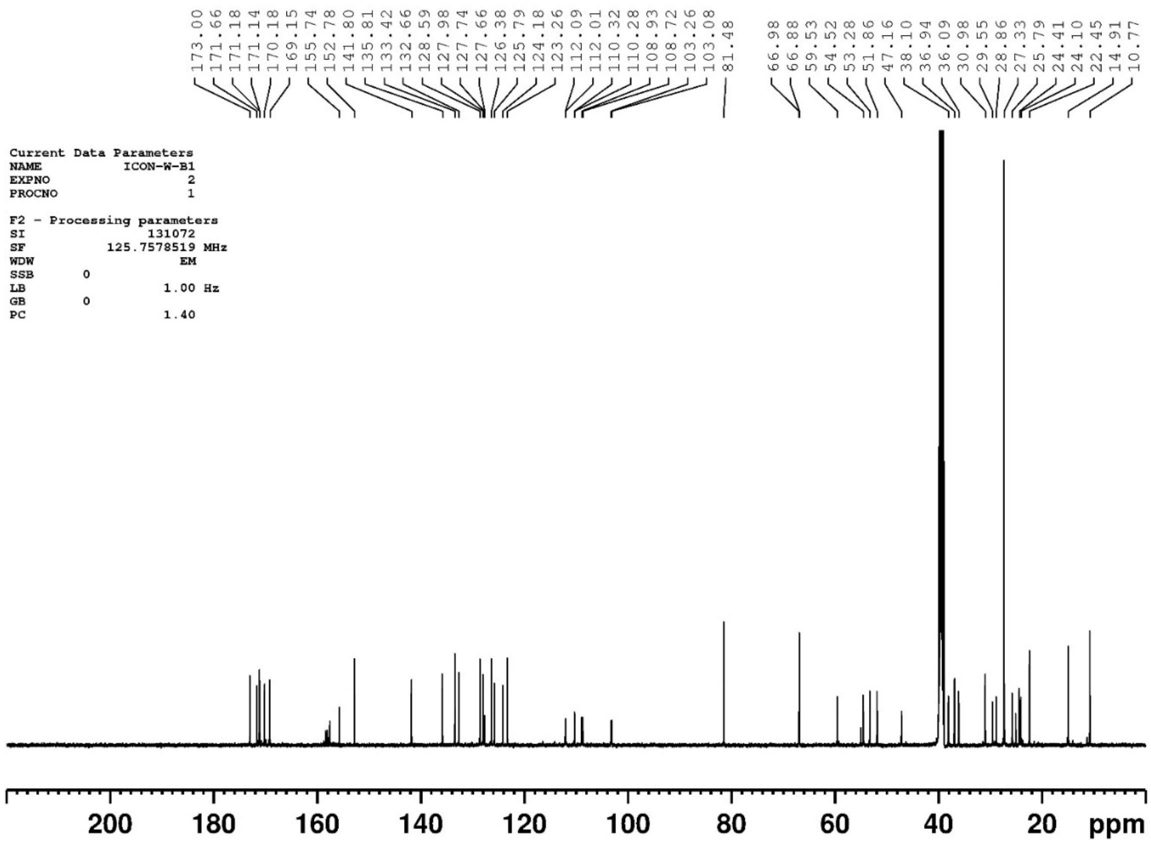
F2 - Acquisition Parameters
Date_ 20160206
Time 21.17
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcetgpsisp.2
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 204.54
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
CNST17 -0.5000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00345000 sec
IN0 0.00002940 sec

======== CHANNEL f1 ========
SFO1 500.1327507 MHz
NUC1 1H
P1 9.90 usec
P2 19.80 usec
P28 1000.00 usec
PLW1 13.50000000 W

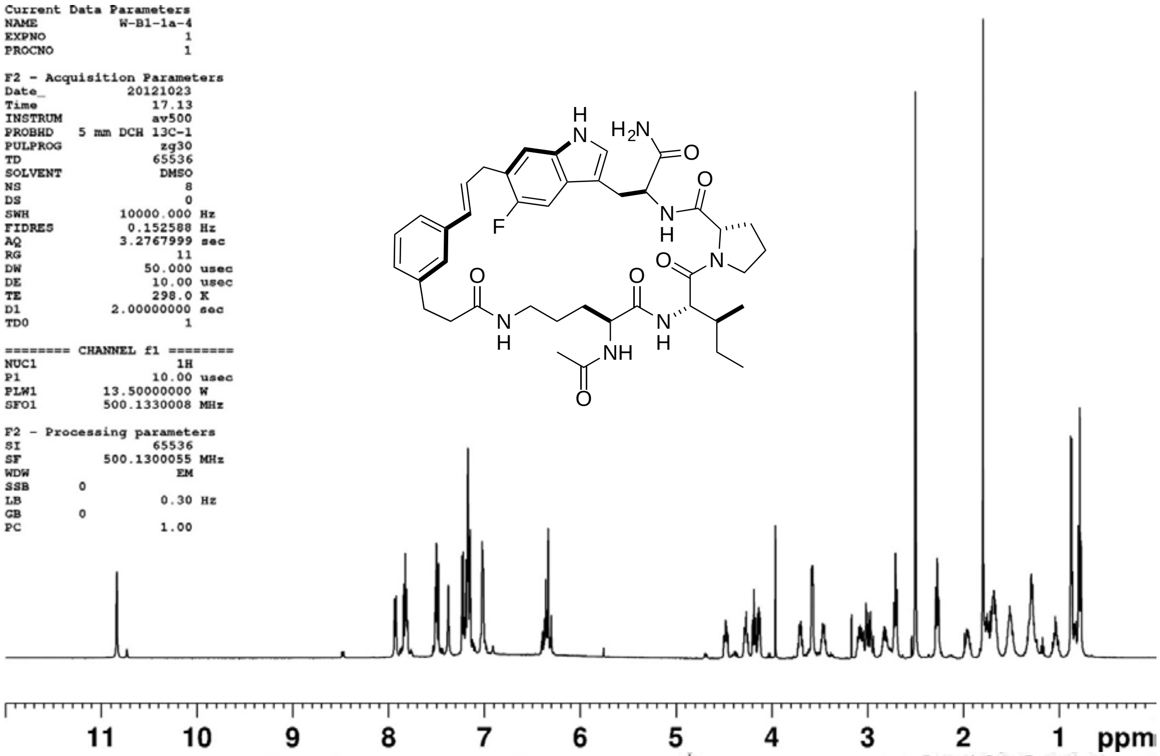
======== CHANNEL f2 ========
SFO2 125.7675352 MHz
NUC2 13C
CPDPRG[2 garp
P3 9.63 usec
P14 500.00 usec
P24 2000.00 usec
PCPD2 70.00 usec
PLW0 0 W
PLW2 23.01399994 W
PLW12 0.43557000 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3 0.500
SPOFFS3 0 Hz
SPW3 3.26090002 W

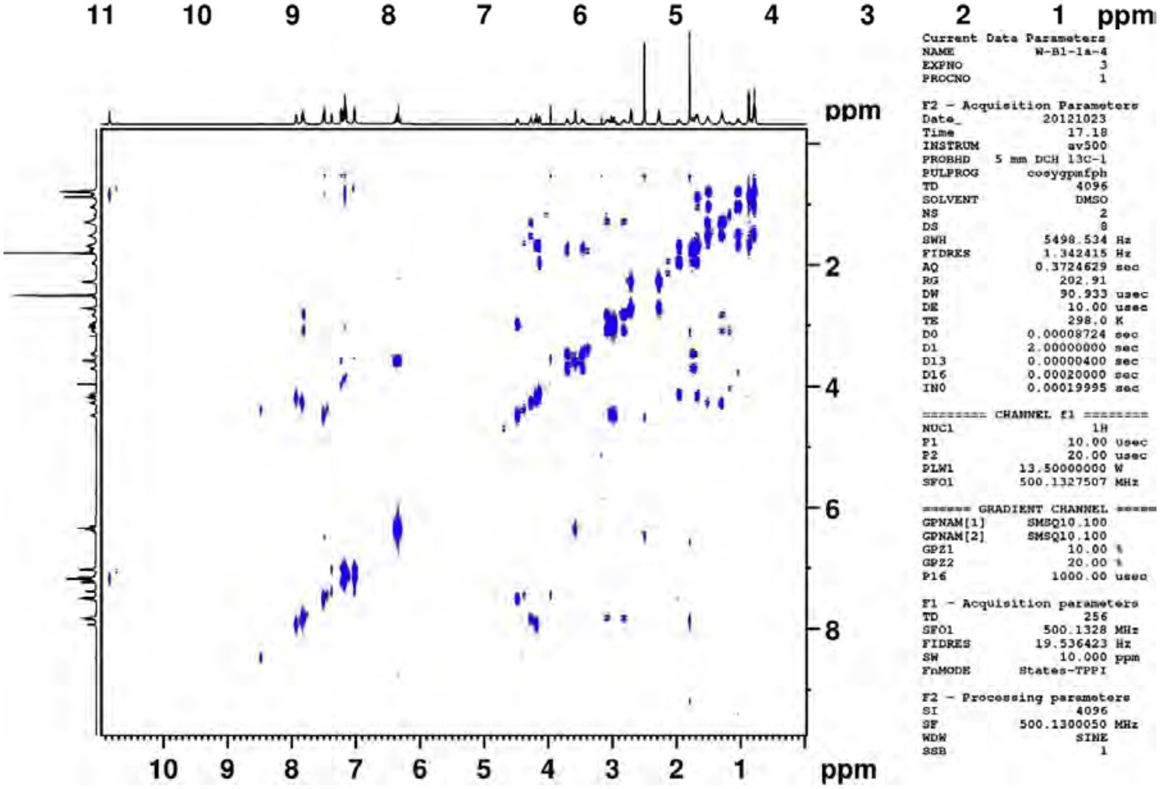
*Acyclic Precursor **S1***

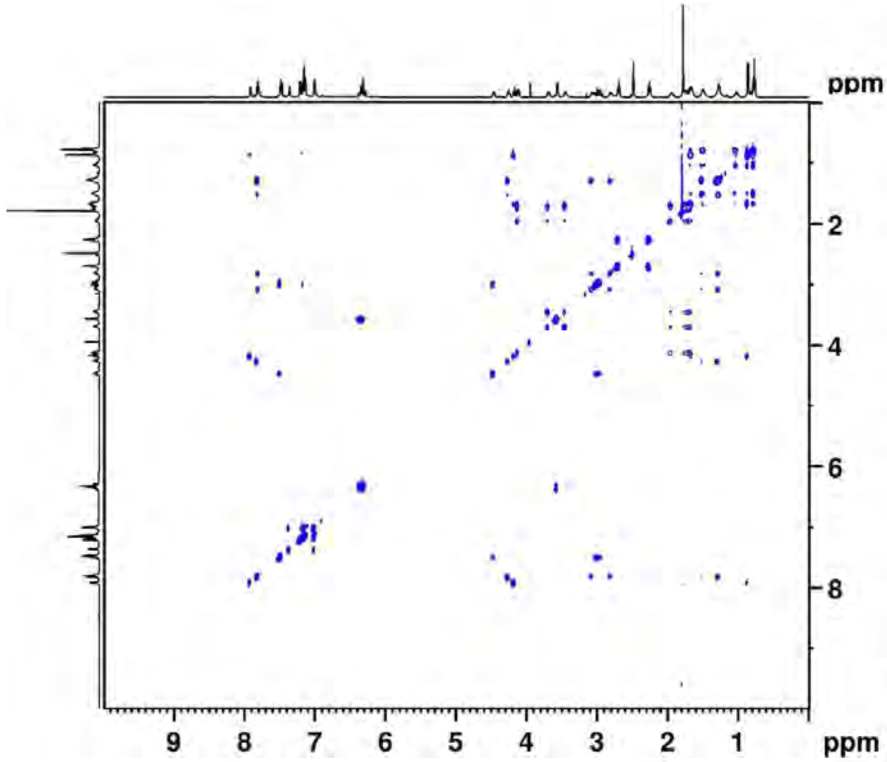




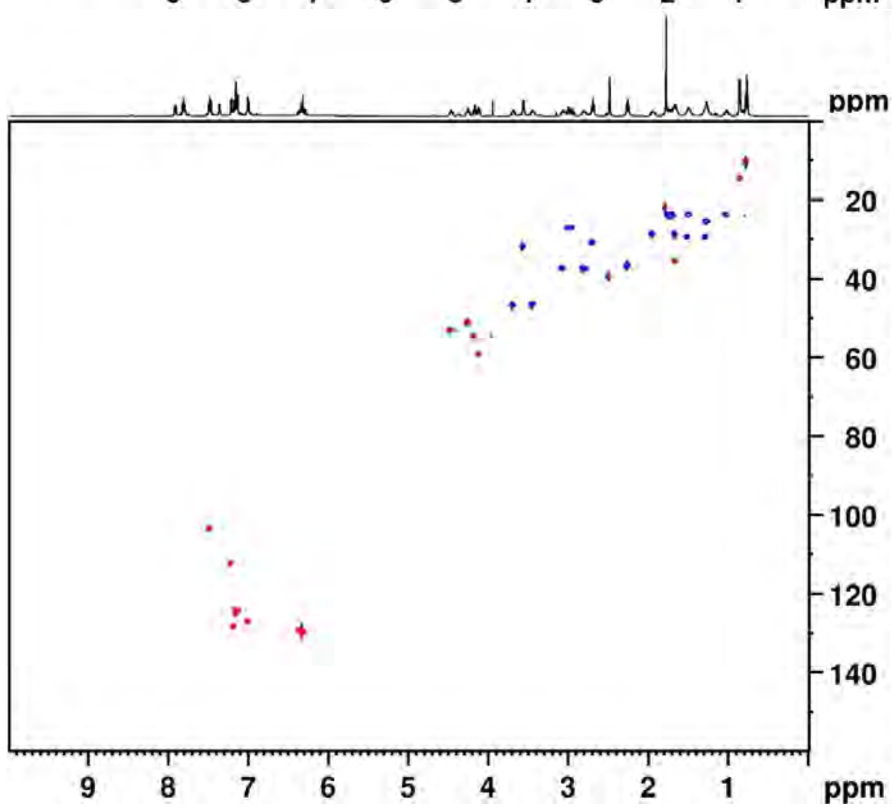
*Macrocyclic Product **S2a***



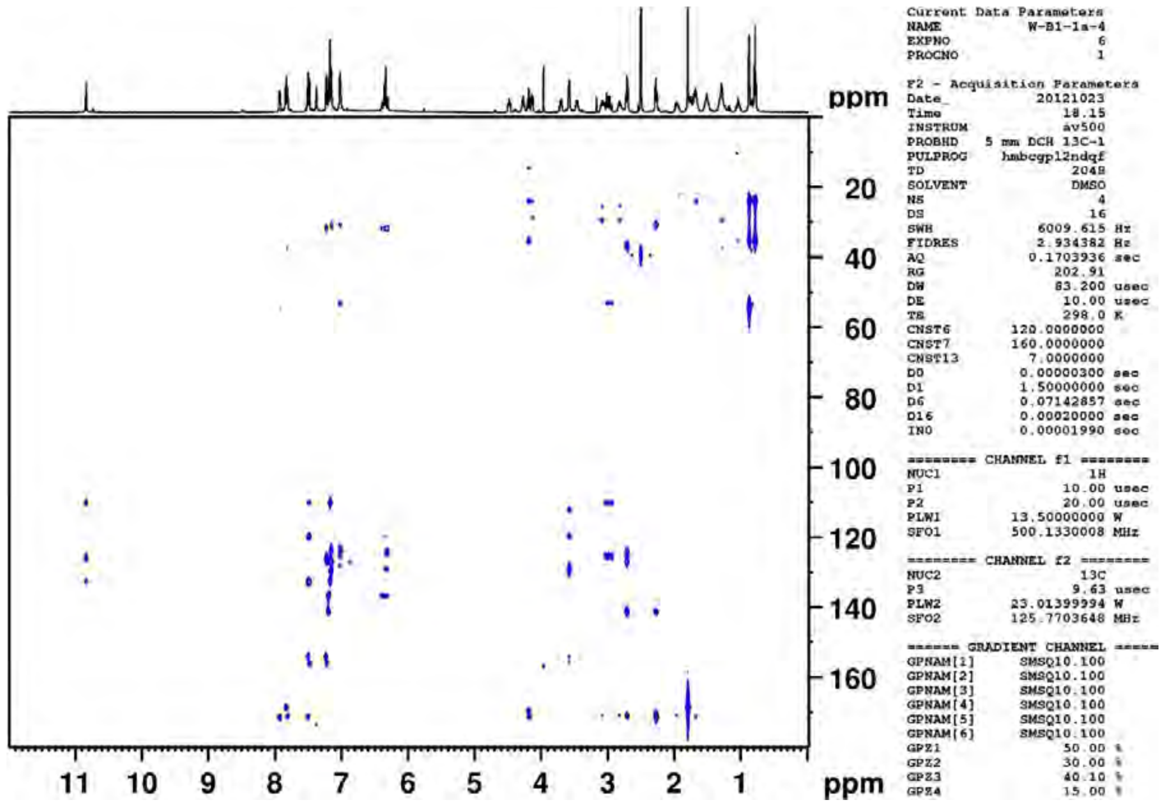




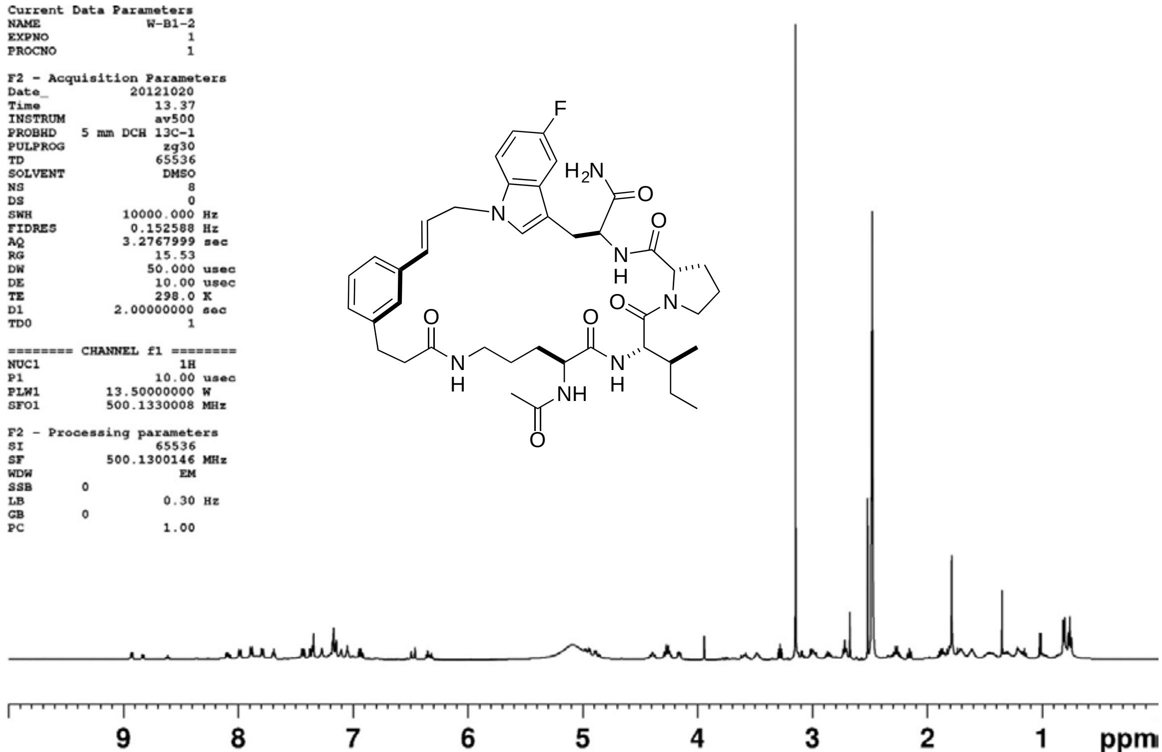

```
Current Data Parameters
NAME              W-B1-1a-4
EXPNO                     4
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121023
Time                  17.39
INSTRUM               av500
PROBHD   5 mm DCH 13C-1
PULPROG        mlevetgp.js
TD                     2048
SOLVENT                DMSO
NS                        2
DS                        8
SWH                5000.000 Hz
FIDRES             2.441406 Hz
AQ                0.2048000 sec
RG                    37.94
DW                  100.000 usec
DE                    10.00 usec
TE                    298.0 K
D0               0.00000300 sec
D1               2.00000000 sec
D9               0.06000000 sec
D11              0.03000000 sec
D12              0.00002000 sec
D16              0.00020000 sec
IN0              0.00019995 sec
L1                       24

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.00 usec
P2                    20.00 usec
P5                    26.68 usec
P6                    40.00 usec
P7                    80.00 usec
P17                 2500.00 usec
PLW1            13.50000000 W
PLW10            0.84375000 W
SFO1            500.1325007 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]           SINE.100
GPNAM[2]           SINE.100
GPZ1                  30.00 %
GPZ2                  30.00 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                      256
SFO1               500.1325 MHz
FIDRES            19.536406 Hz
```

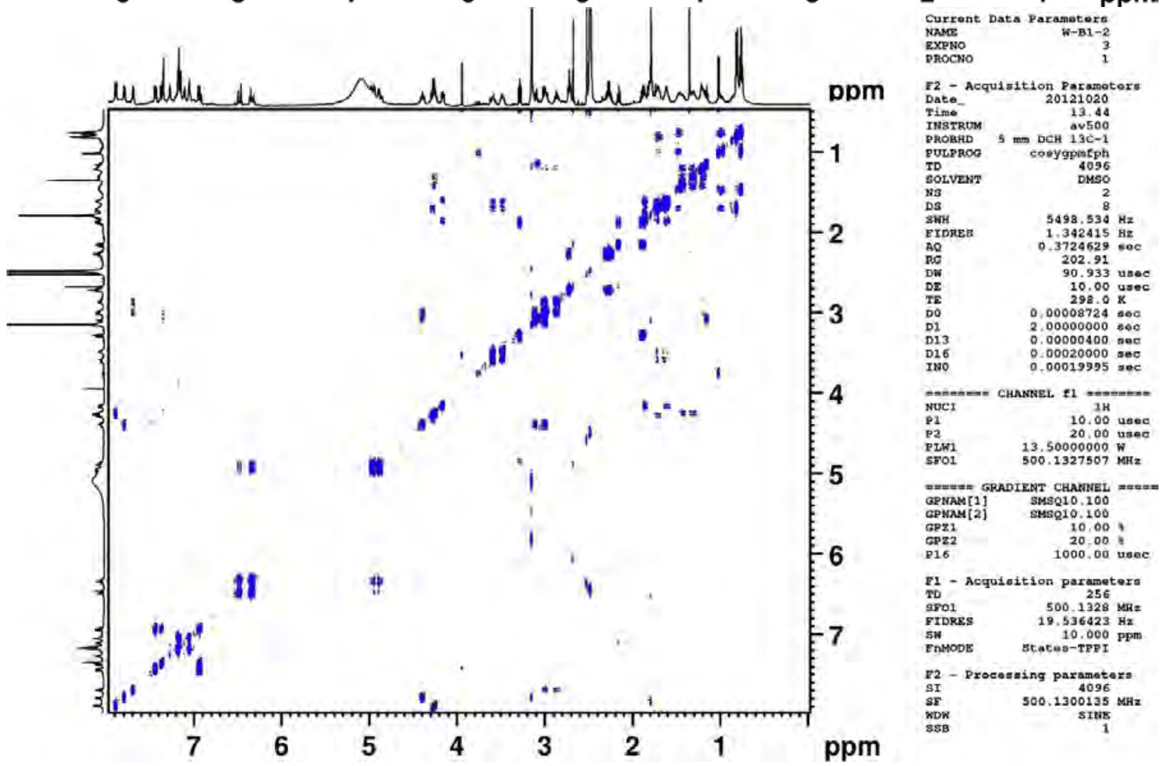


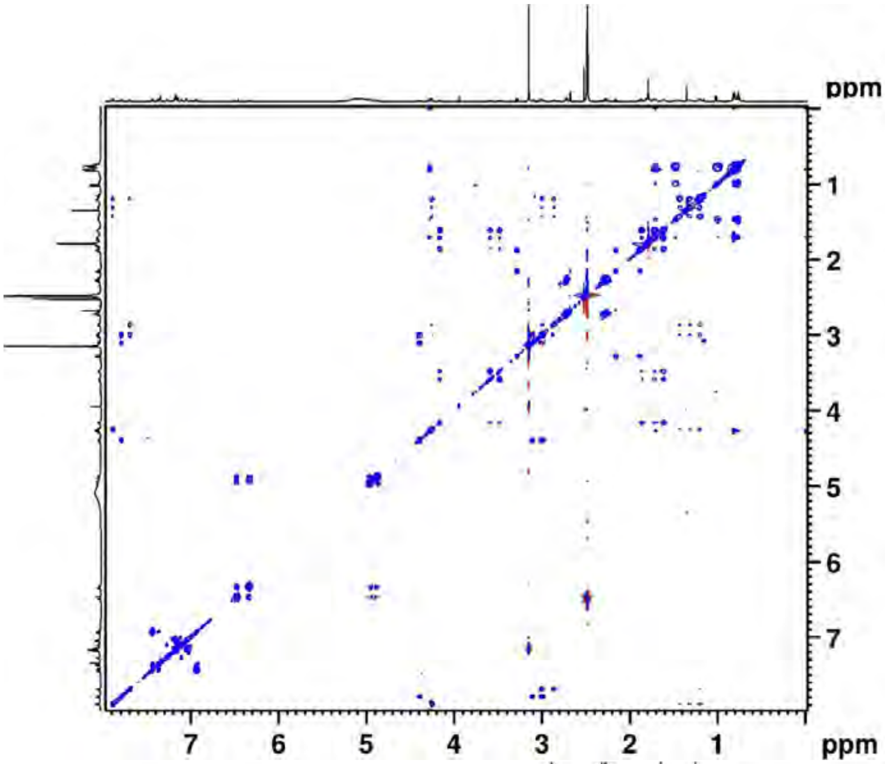

```
Current Data Parameters
NAME              W-B1-1a-4
EXPNO                     5
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121023
Time                  18.00
INSTRUM               av500
PROBHD   5 mm DCH 13C-1
PULPROG          hsqcedetgp
TD                     2048
SOLVENT                DMSO
NS                        2
DS                       16
SWH                5000.000 Hz
FIDRES             2.441406 Hz
AQ                0.2048000 sec
RG                   202.91
DW                  100.000 usec
DE                    10.00 usec
TE                    298.0 K
CNST2           145.0000000
D0               0.00000300 sec
D1               1.50000000 sec
D4               0.00172414 sec
D11              0.03000000 sec
D13              0.00000400 sec
D16              0.00020000 sec
D21              0.00345000 sec
IN0              0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.00 usec
P2                    20.00 usec
P28         0 usec
PLW1            13.50000000 W
SFO1            500.1325007 MHz

======== CHANNEL f2 ========
CPDPRG[2               garp
NUC2                    13C
P3                     9.63 usec
P4                    19.26 usec
PCPD2                 70.00 usec
PLW2            23.01399994 W
PLW12            0.43557000 W
SFO2            125.7678496 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]         SMSQ10.100
GPNAM[2]         SMSQ10.100
GPZ1                  80.00 %
```


ppm
20
40
60
80
100
120
140
160
11
10
9
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME W-B1-1a-4
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121023
Time 18.15
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 202.91
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W
SFO1 500.1330008 MHz
======== CHANNEL f2 ========
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
SFO2 125.7703648 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

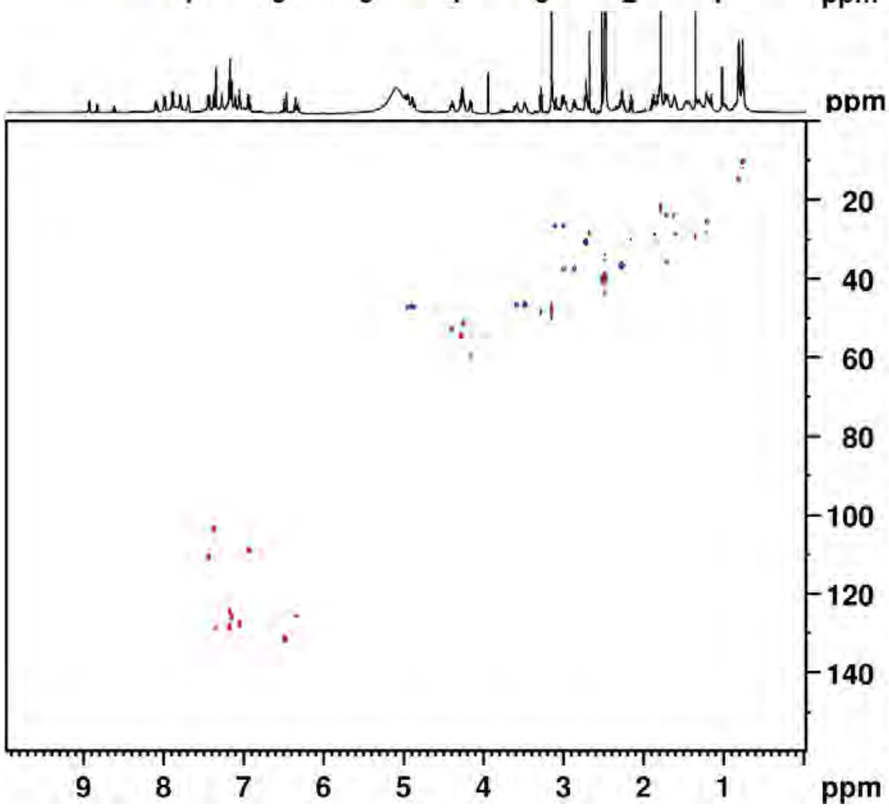
*Macrocyclic Product **S2b***



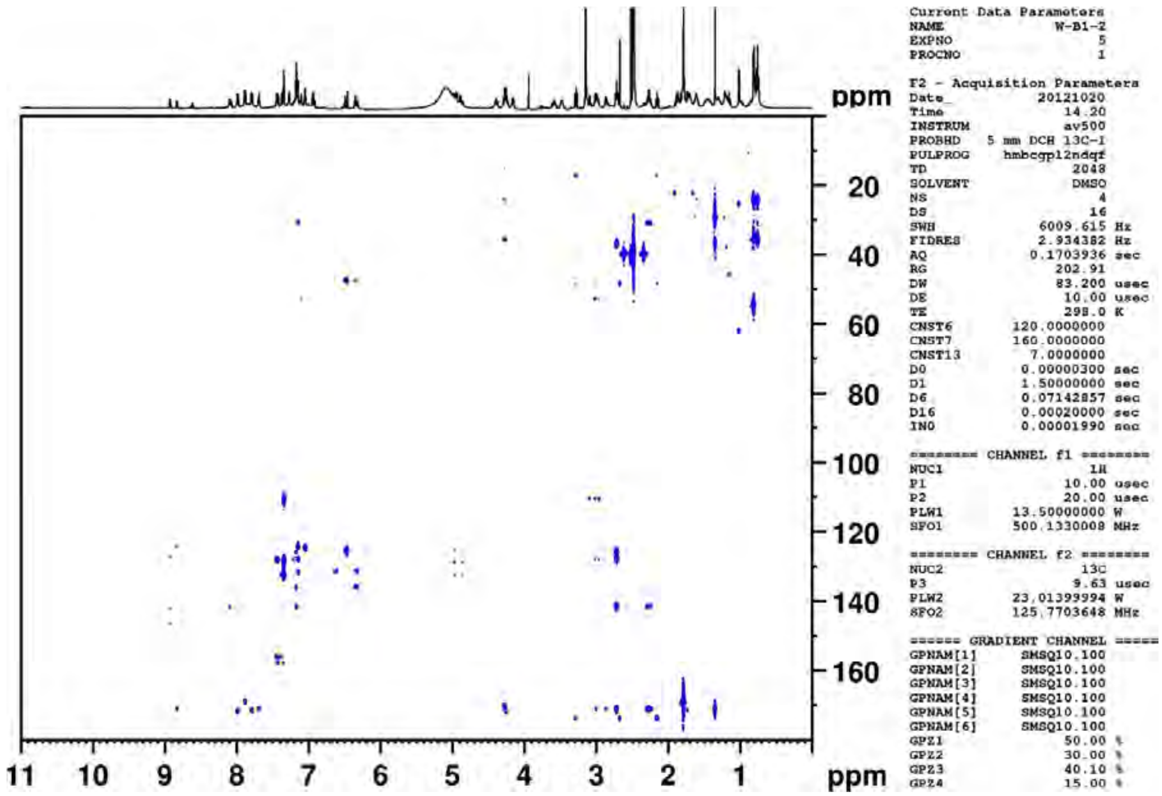




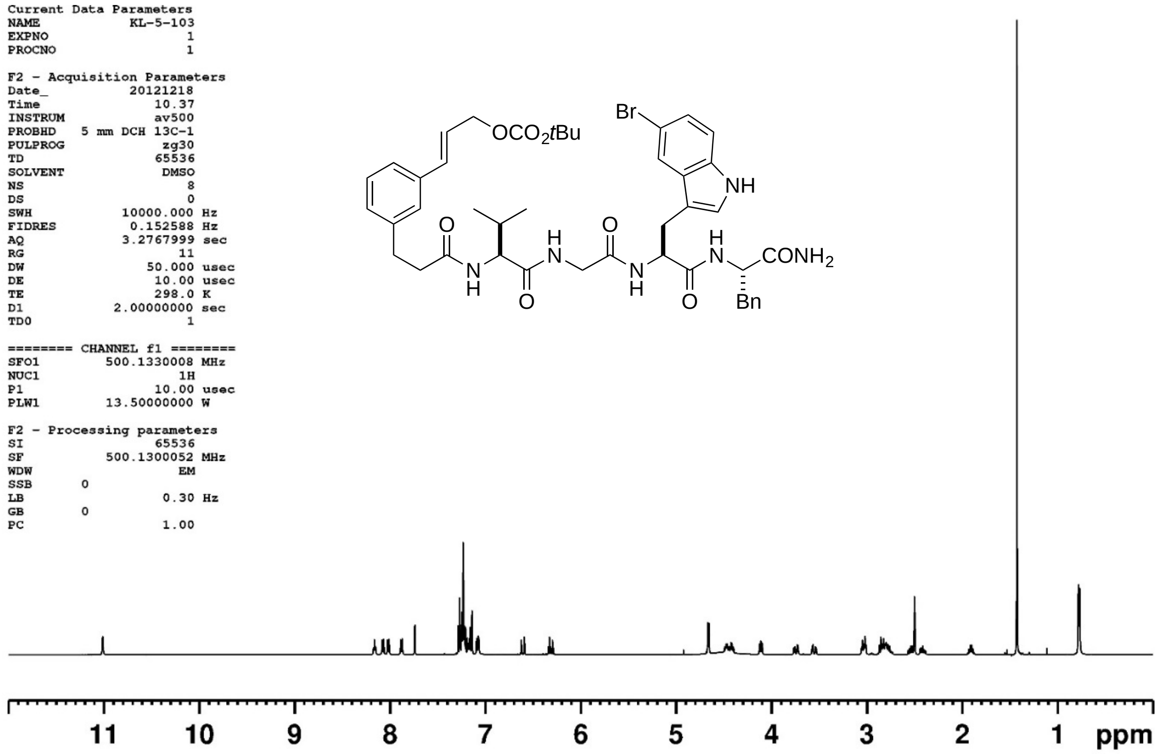
```
Current Data Parameters
NAME              W-B1-2
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121020
Time               14.51
INSTRUM            av500
PROBHD   5 mm DCH 13C-1
PULPROG      mlevetgp.js
TD                  2048
SOLVENT             DMSO
NS                     2
DS                     8
SWH          4000.000 Hz
FIDRES       1.953125 Hz
AQ         0.2560000 sec
RG                 37.94
DW           125.000 usec
DE             10.00 usec
TE                 298.0 K
D0        0.00000300 sec
D1        2.00000000 sec
D9        0.06000000 sec
D11       0.03000000 sec
D12       0.00002000 sec
D16       0.00020000 sec
IN0       0.00024995 sec
L1                    24

======== CHANNEL f1 ========
NUC1                  1H
P1             10.00 usec
P2             20.00 usec
P5             26.68 usec
P6             40.00 usec
P7             80.00 usec
P17          2500.00 usec
PLW1       13.50000000 W
PLW10       0.84375000 W
SFO1      500.1320005 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPZ1             30.00 %
GPZ2             30.00 %
P16          1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1         500.132 MHz
FIDRES      15.629138 Hz
```

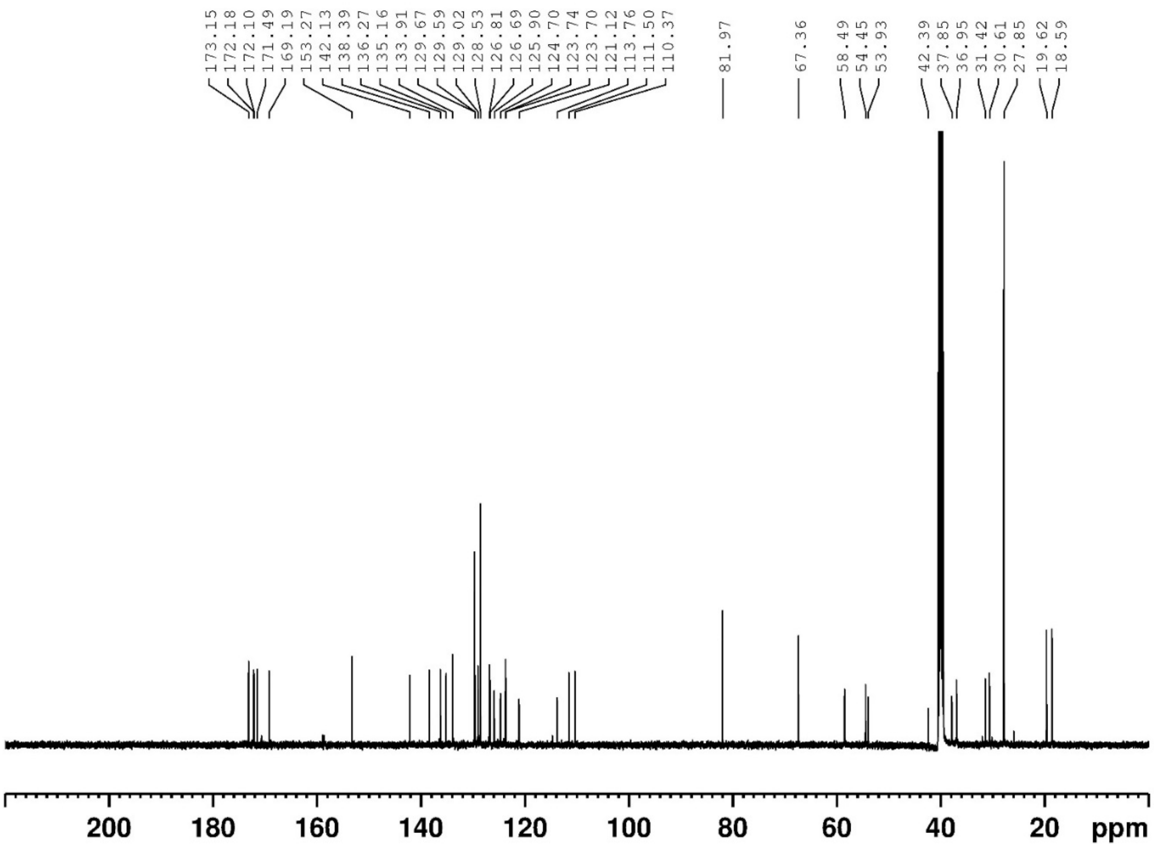


```
Current Data Parameters
NAME              W-B1-2
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121020
Time               14.05
INSTRUM            av500
PROBHD   5 mm DCH 13C-1
PULPROG      hsqcedetgp
TD                  2048
SOLVENT             DMSO
NS                     2
DS                    16
SWH          5000.000 Hz
FIDRES       2.441406 Hz
AQ         0.2048000 sec
RG                202.91
DW           100.000 usec
DE             10.00 usec
TE                 298.0 K
CNST2        145.0000000
D0        0.00000300 sec
D1        1.50000000 sec
D4        0.00172414 sec
D11       0.03000000 sec
D13       0.00000400 sec
D16       0.00020000 sec
D21       0.00345000 sec
IN0       0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                  1H
P1             10.00 usec
P2             20.00 usec
P28        0 usec
PLW1       13.50000000 W
SFO1      500.1325007 MHz

======== CHANNEL f2 ========
CPDPRG[2            garp
NUC2                 13C
P3              9.63 usec
P4             19.26 usec
PCPD2          70.00 usec
PLW2       23.01399994 W
PLW12       0.43557000 W
SFO2      125.7678496 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPNAM[2]     SMSQ10.100
GPZ1            80.00 %
```

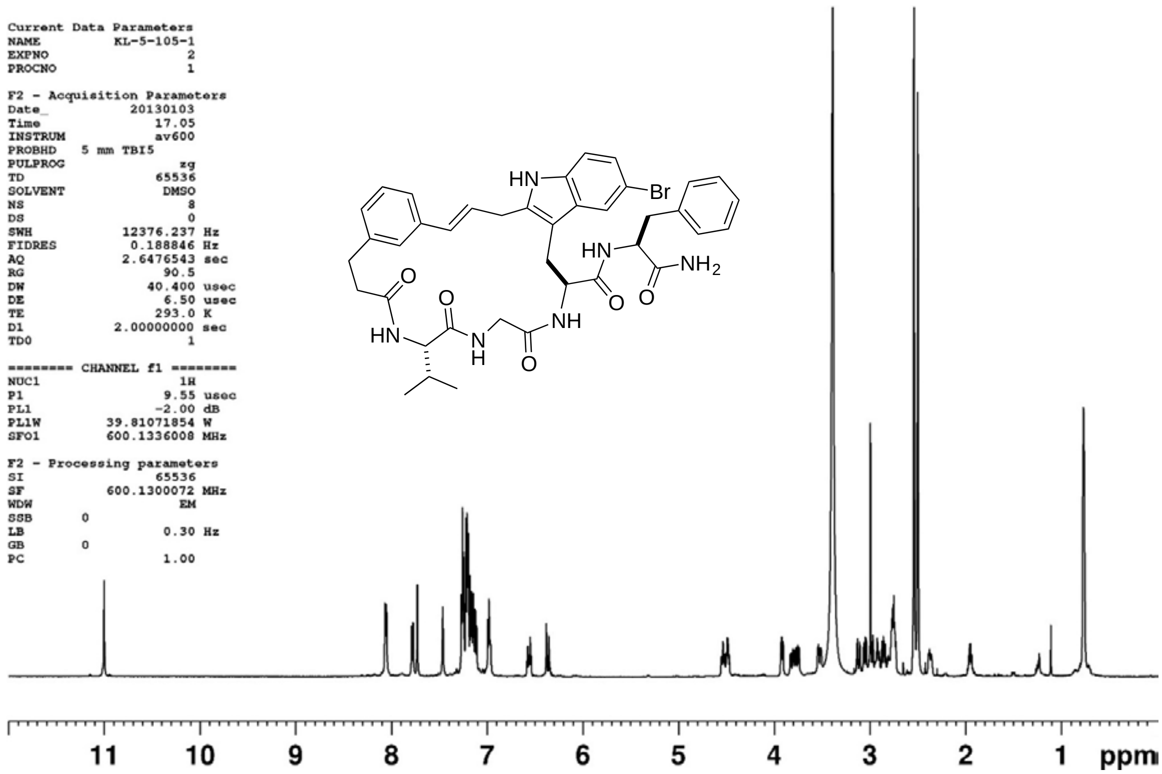

ppm
20
40
60
80
100
120
140
160
11
10
9
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME W-B1-2
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121020
Time 14.20
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 202.91
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W
SFO1 500.1330008 MHz
======== CHANNEL f2 ========
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
SFO2 125.7703648 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

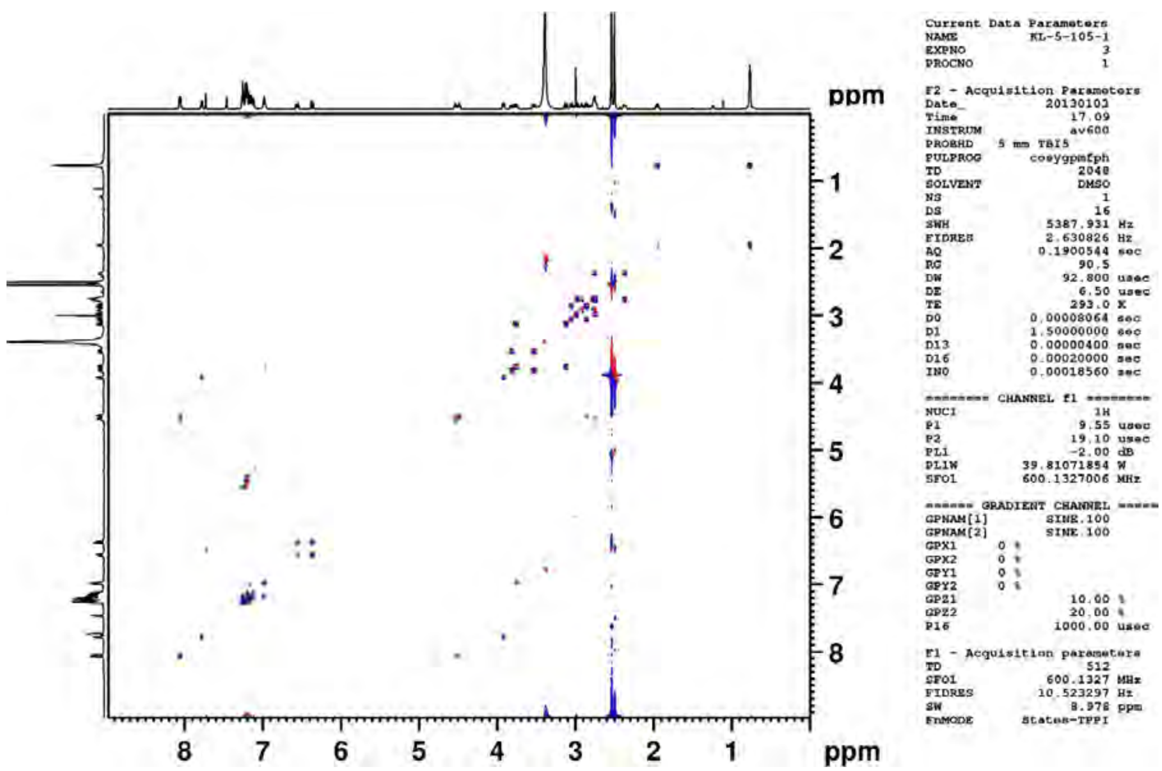
*Acyclic Precursor **S3***

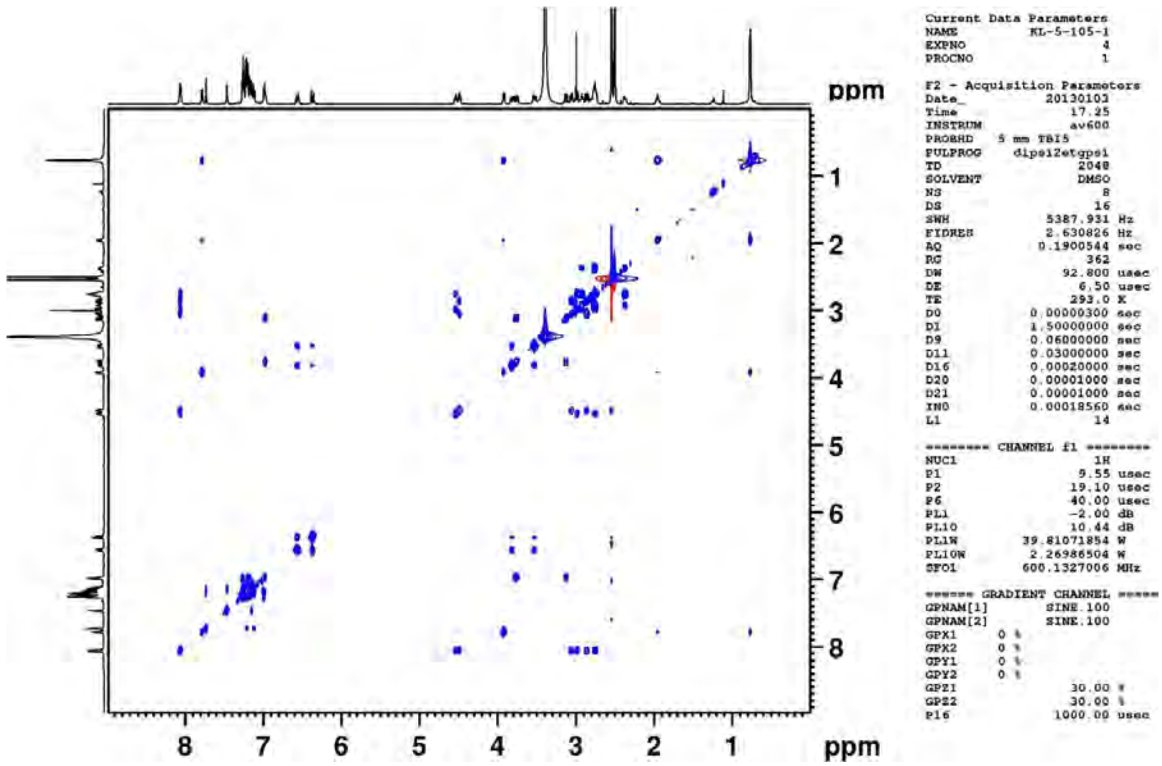


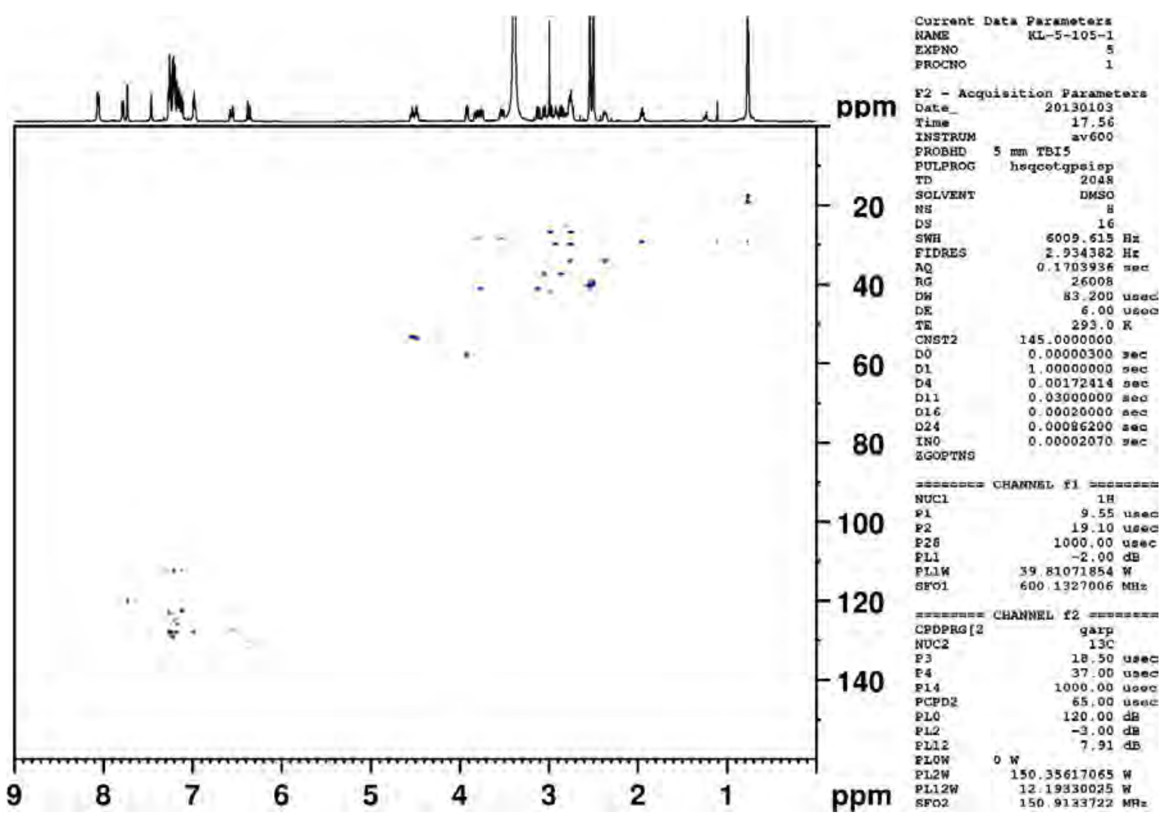


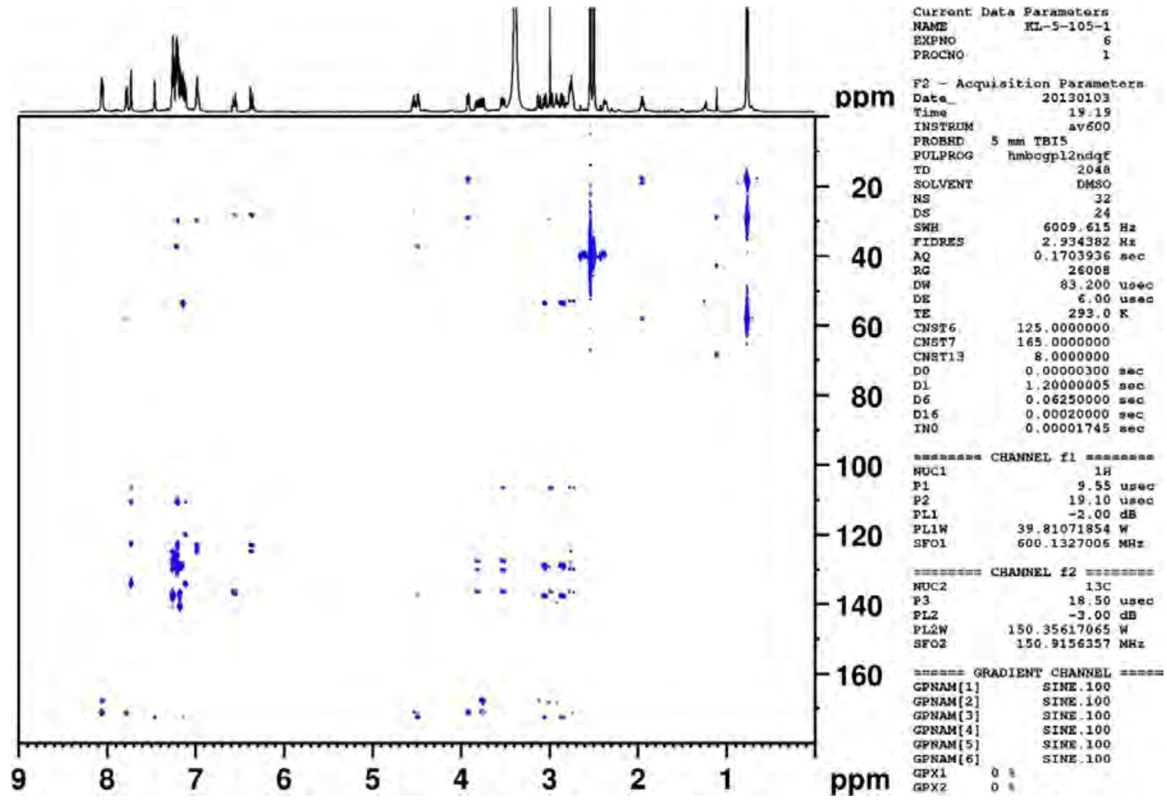
Macrocyclic Product ***S4a***



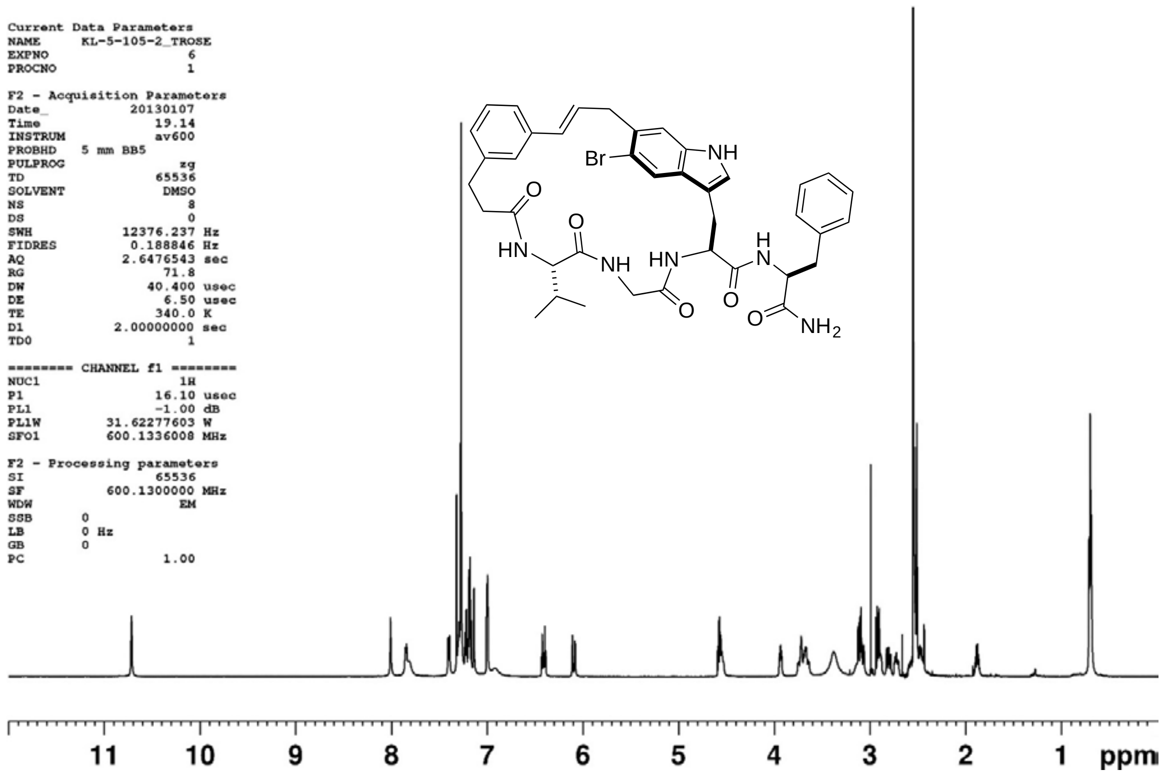


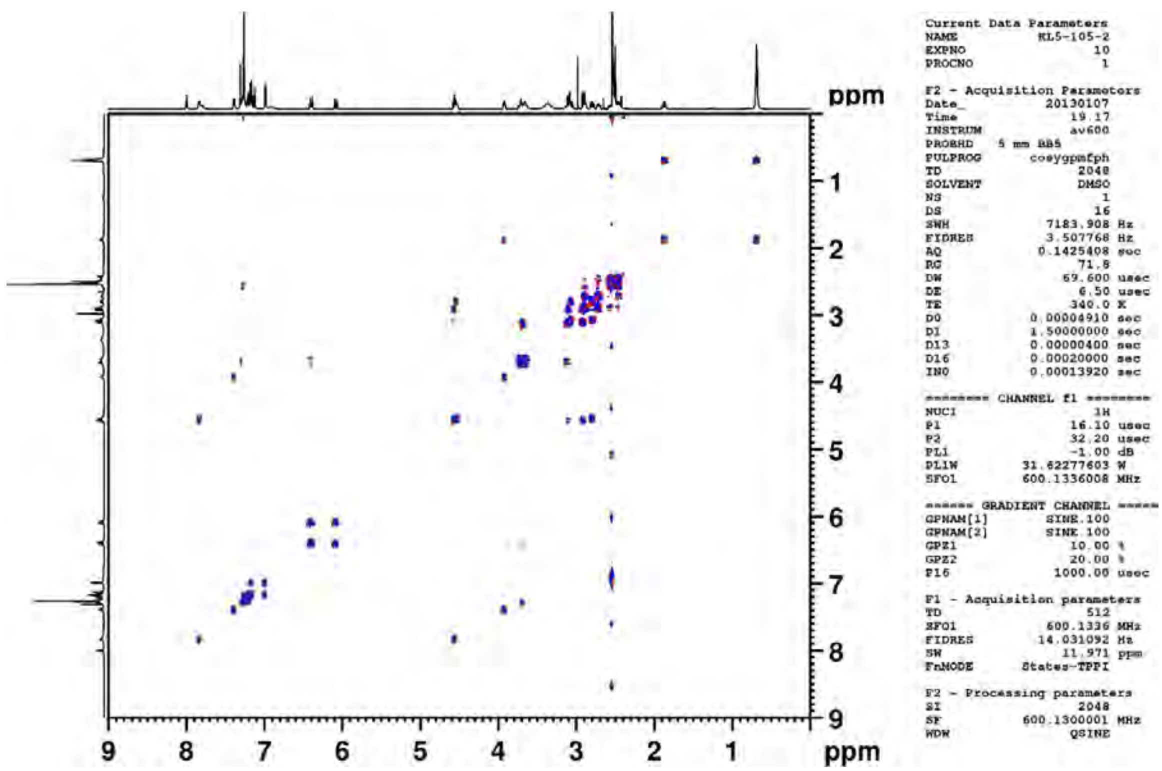

ppm
1
2
3
4
5
6
7
8
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME KL-5-105-1
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130103
Time 17.25
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG dipsi2etgpsi
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 5387.931 Hz
FIDRES 2.630826 Hz
AQ 0.1900544 sec
RG 362
DW 92.800 usec
DE 6.50 usec
TE 293.0 K
D0 0.00000300 sec
D1 1.50000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00001000 sec
D21 0.00001000 sec
IN0 0.00018560 sec
L1 14
======== CHANNEL f1 ========
NUC1 1H
P1 9.55 usec
P2 19.10 usec
P6 40.00 usec
PL1 -2.00 dB
PL10 10.44 dB
PL1W 39.81071854 W
PL10W 2.26986504 W
SFO1 600.1327006 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec

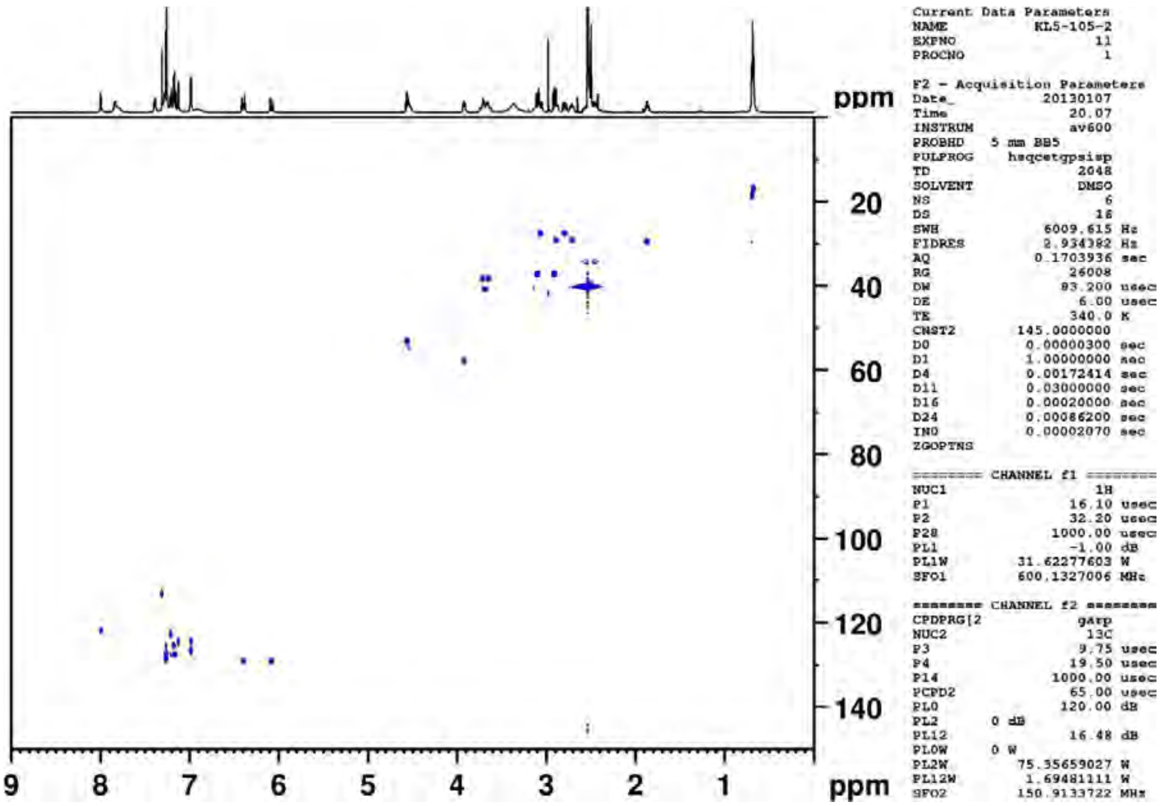

ppm
20
40
60
80
100
120
140
9
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME KL-5-105-1
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130103
Time 17.56
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 26008
DW 83.200 usec
DE 6.00 usec
TE 293.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.00000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 9.55 usec
P2 19.10 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1327006 MHz
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 18.50 usec
P4 37.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.91 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 12.19330025 W
SFO2 150.9133722 MHz

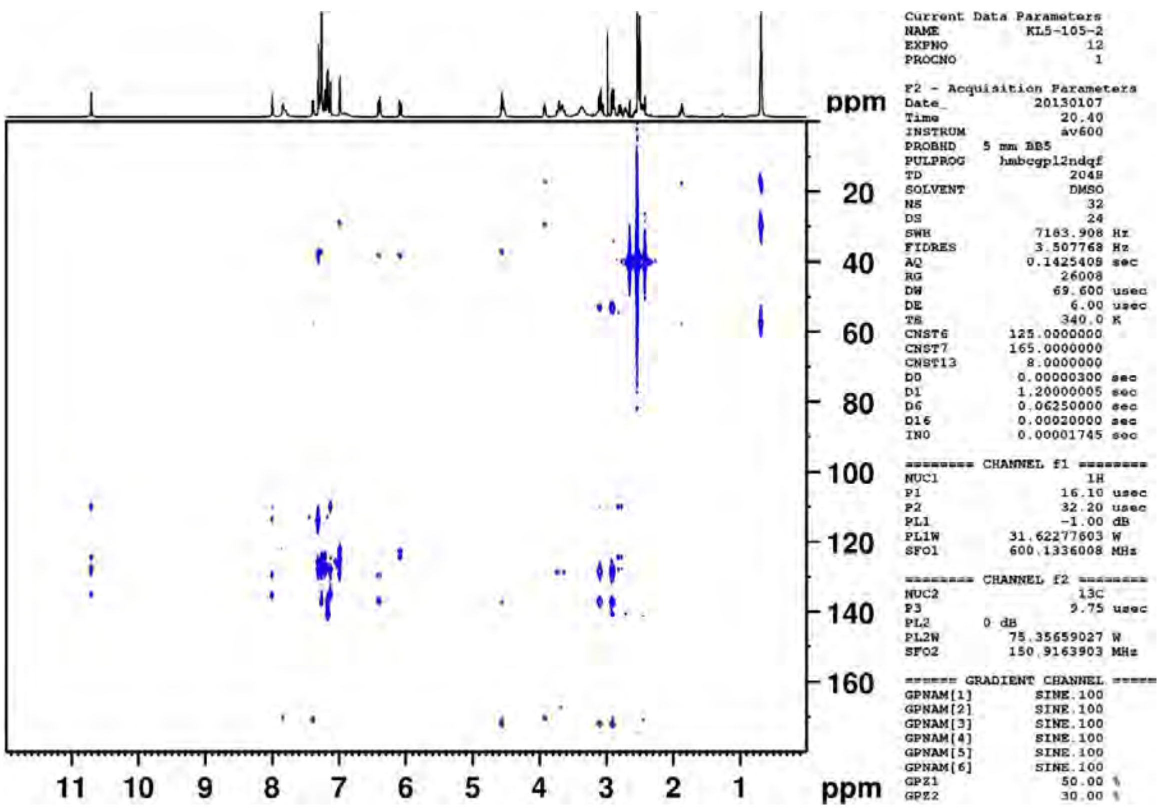

Current Data Parameters
NAME KL-5-105-1
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130103
Time 19.19
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 32
DS 24
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 26008
DW 83.200 usec
DE 6.00 usec
TE 293.0 K
CNST6 125.0000000
CNST7 165.0000000
CNST13 8.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D6 0.06250000 sec
D16 0.00020000 sec
IN0 0.00001745 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.55 usec
P2 19.10 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1327006 MHz
======== CHANNEL f2 ========
NUC2 13C
P3 18.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPNAM[4] SINE.100
GPNAM[5] SINE.100
GPNAM[6] SINE.100
GPX1 0 %
GPX2 0 %
ppm
20
40
60
80
100
120
140
160
9
8
7
6
5
4
3
2
1
ppm

*Macrocyclic Product **S4b***

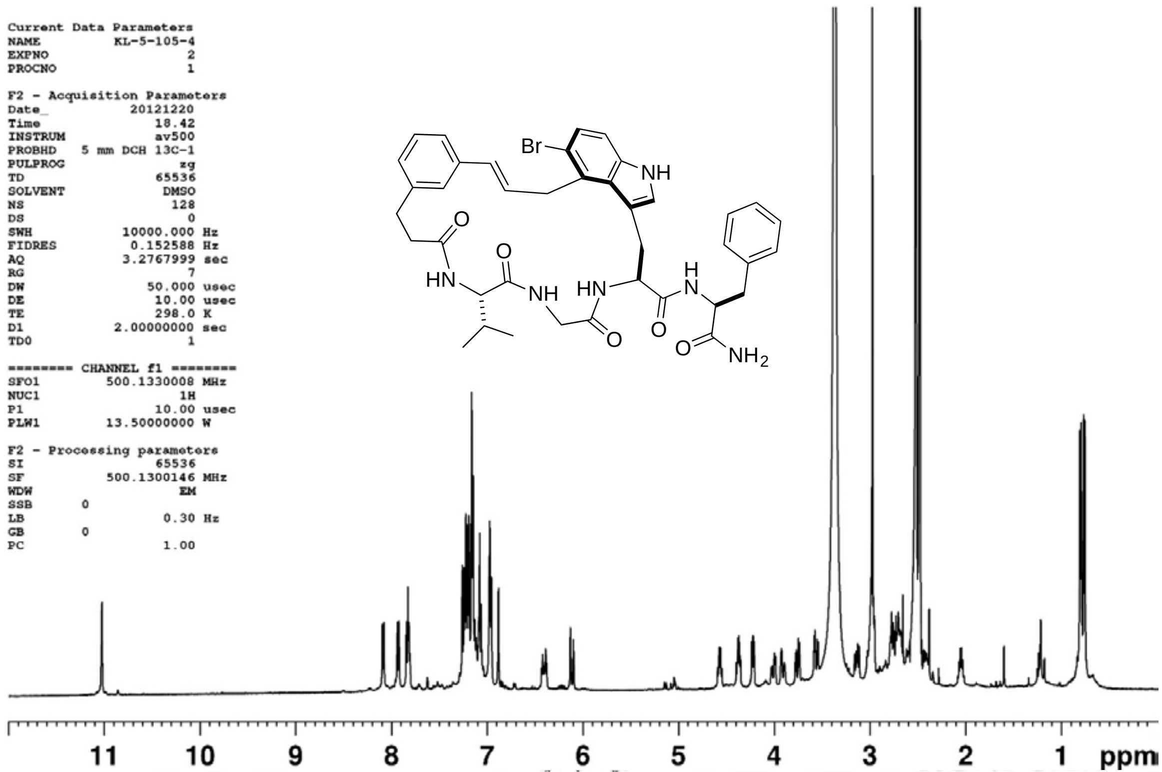


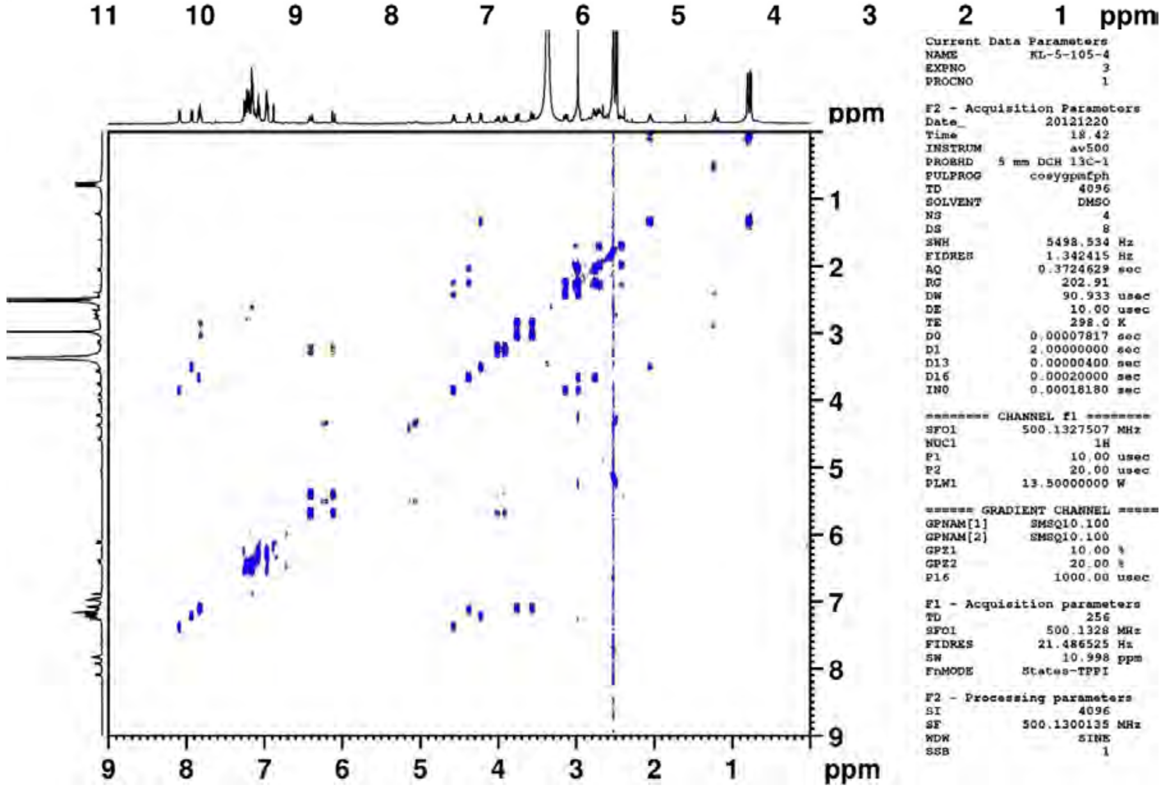


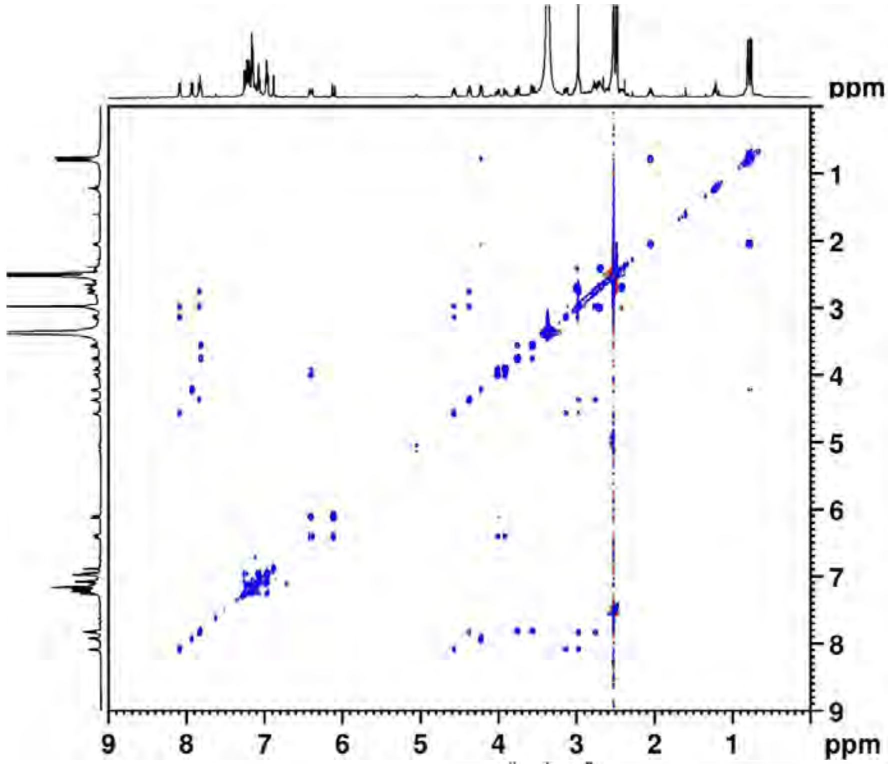

Current Data Parameters
NAME KL5-105-2
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130107
Time 20.07
INSTRUM av600
PROBHD 5 mm BB5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 6
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 26008
DW 83.200 usec
DE 6.00 usec
TE 340.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.00000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 16.10 usec
P2 32.20 usec
P28 1000.00 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1327006 MHz
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 9.75 usec
P4 19.50 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 0 dB
PL12 16.48 dB
PL0W 0 W
PL2W 75.35659027 W
PL12W 1.69481111 W
SFO2 150.9133722 MHz
ppm
20
40
60
80
100
120
140
9 8 7 6 5 4 3 2 1 ppm


Current Data Parameters
NAME KL5-105-2
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130107
Time 20.40
INSTRUM av600
PROBHD 5 mm BB5
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 32
DS 24
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425408 sec
RG 26008
DW 69.600 usec
DE 6.00 usec
TE 340.0 K
CNST6 125.0000000
CNST7 165.0000000
CNST13 8.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D6 0.06250000 sec
D16 0.00020000 sec
IN0 0.00001745 sec
======== CHANNEL f1 ========
NUC1 1H
P1 16.10 usec
P2 32.20 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1336008 MHz
======== CHANNEL f2 ========
NUC2 13C
P3 9.75 usec
PL2 0 dB
PL2W 75.35659027 W
SFO2 150.9163903 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPNAM[4] SINE.100
GPNAM[5] SINE.100
GPNAM[6] SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
ppm
20
40
60
80
100
120
140
160
11 10 9 8 7 6 5 4 3 2 1 ppm

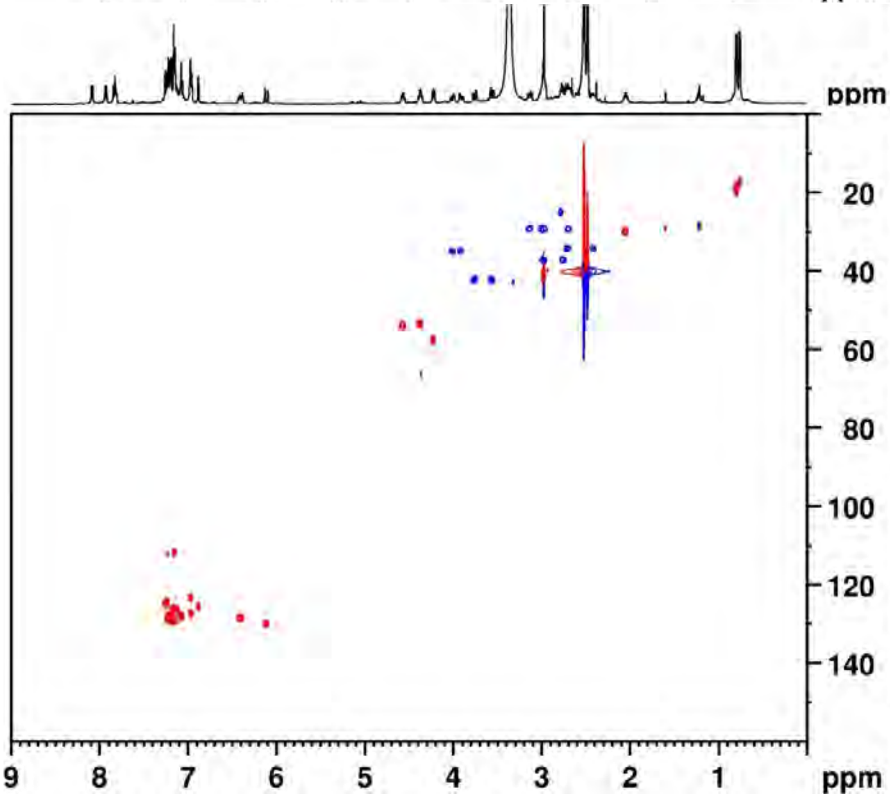
Macrocyclic Product ***S4c***



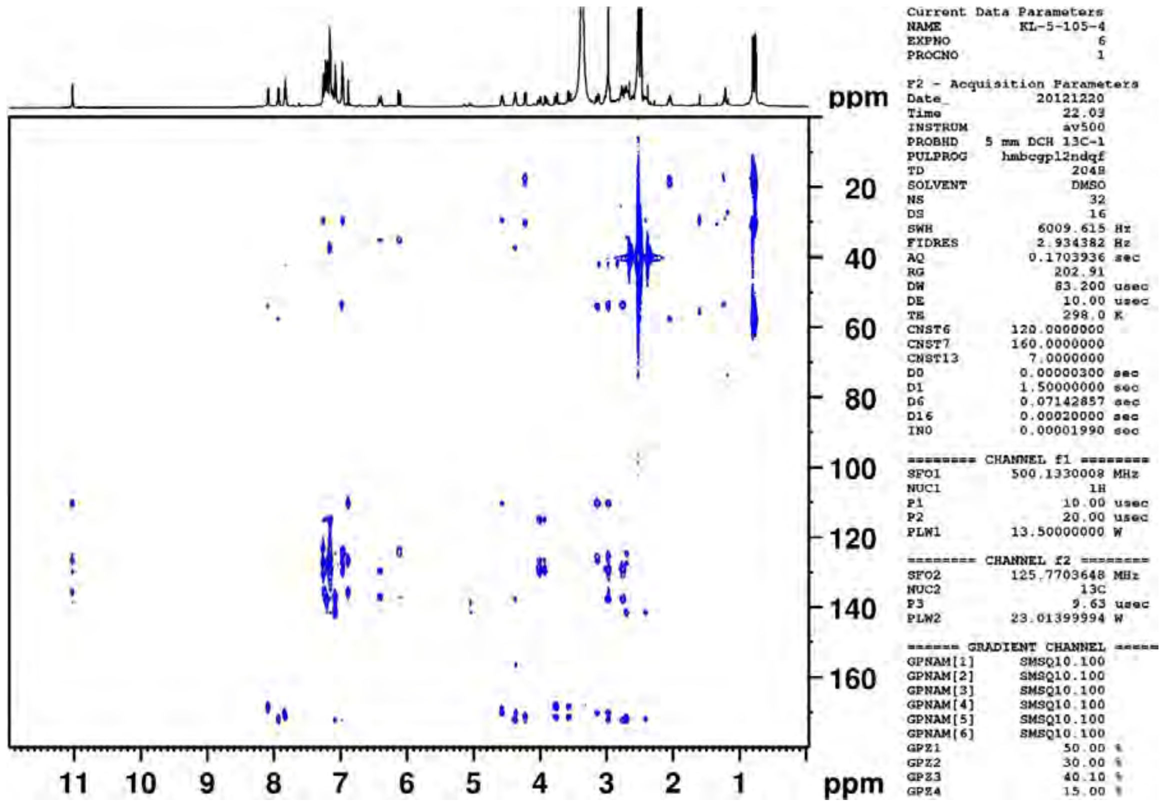





```
Current Data Parameters
NAME              KL-5-105-4
EXPNO                      4
PROCNO                     1

F2 - Acquisition Parameters
Date_               20121220
Time                   19.23
INSTRUM                av500
PROBHD   5 mm DCH 13C-1
PULPROG          mlevetgp.js
TD                      2048
SOLVENT                 DMSO
NS                         4
DS                         8
SWH                 5000.000 Hz
FIDRES              2.441406 Hz
AQ                 0.2048000 sec
RG                     37.94
DW                   100.000 usec
DE                     10.00 usec
TE                     298.0 K
D0                0.00000300 sec
D1                2.00000000 sec
D9                0.06000000 sec
D11               0.03000000 sec
D12               0.00002000 sec
D16               0.00020000 sec
IN0               0.00020000 sec
L1                        24

======== CHANNEL f1 ========
SFO1             500.1325007 MHz
NUC1                      1H
P1                     10.00 usec
P2                     20.00 usec
P5                     26.68 usec
P6                     40.00 usec
P7                     80.00 usec
P17                  2500.00 usec
PLW1             13.50000000 W
PLW10             0.84375000 W

====== GRADIENT CHANNEL =====
GPNAM[1]            SINE.100
GPNAM[2]            SINE.100
GPZ1                   30.00 %
GPZ2                   30.00 %
P16                  1000.00 usec

F1 - Acquisition parameters
TD                       256
SFO1                500.1325 MHz
FIDRES             19.531250 Hz
```

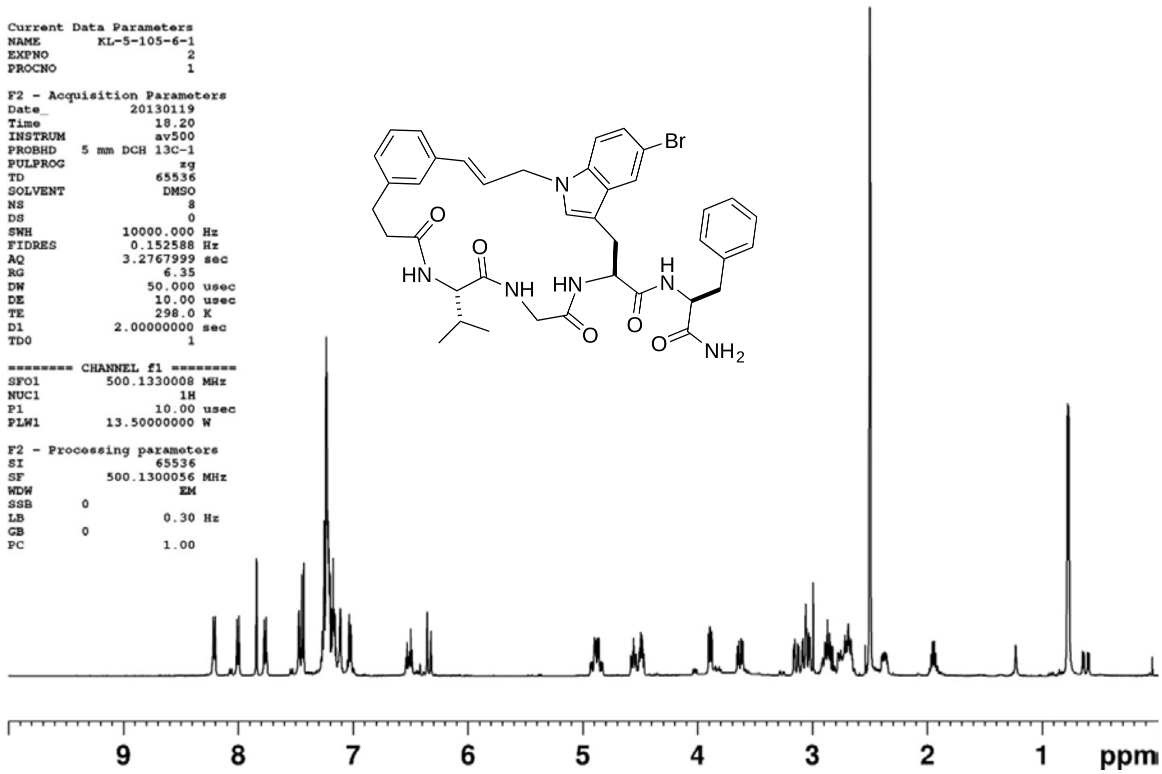


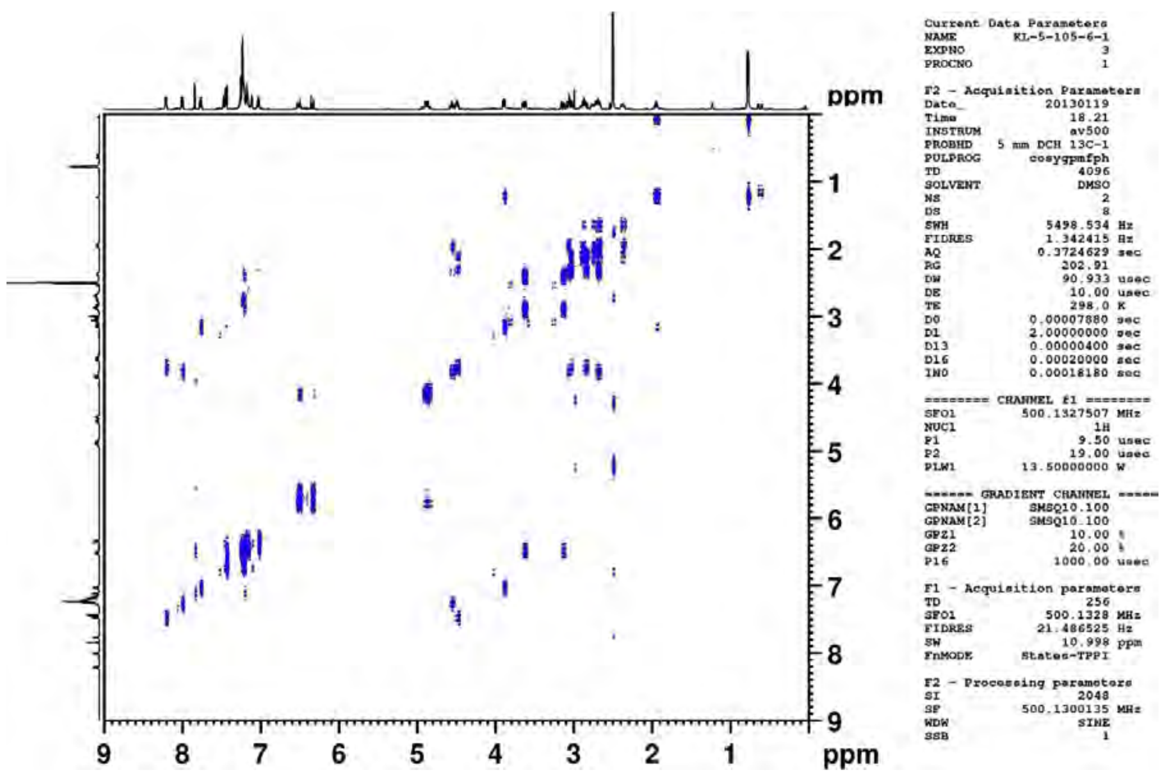

```
Current Data Parameters
NAME              KL-5-105-4
EXPNO                      5
PROCNO                     1

F2 - Acquisition Parameters
Date                20121220
Time                   20.04
INSTRUM                av500
PROBHD   5 mm DCH 13C-1
PULPROG           hsqcedetgp
TD                      2048
SOLVENT                 DMSO
NS                        16
DS                        16
SWH                 5000.000 Hz
FIDRES              2.441406 Hz
AQ                 0.2048000 sec
RG                    202.91
DW                   100.000 usec
DE                     10.00 usec
TE                     298.0 K
CNST2            145.0000000
D0                0.00000300 sec
D1                1.50000000 sec
D4                0.00172414 sec
D11               0.03000000 sec
D13               0.00000400 sec
D16               0.00020000 sec
D21               0.00345000 sec
IN0               0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
SFO1             500.1325007 MHz
NUC1                      1H
P1                     10.00 usec
P2                     20.00 usec
P28         0 usec
PLW1             13.50000000 W

======== CHANNEL f2 ========
SFO2             125.7678496 MHz
NUC2                     13C
CPDPRG[2                garp
P3                      9.63 usec
P4                     19.26 usec
PCPD2                  70.00 usec
PLW2             23.01399994 W
PLW12             0.43557000 W

====== GRADIENT CHANNEL =====
GPNAM[1]          SMSQ10.100
GPNAM[2]          SMSQ10.100
GPZ1                   80.00 %
```

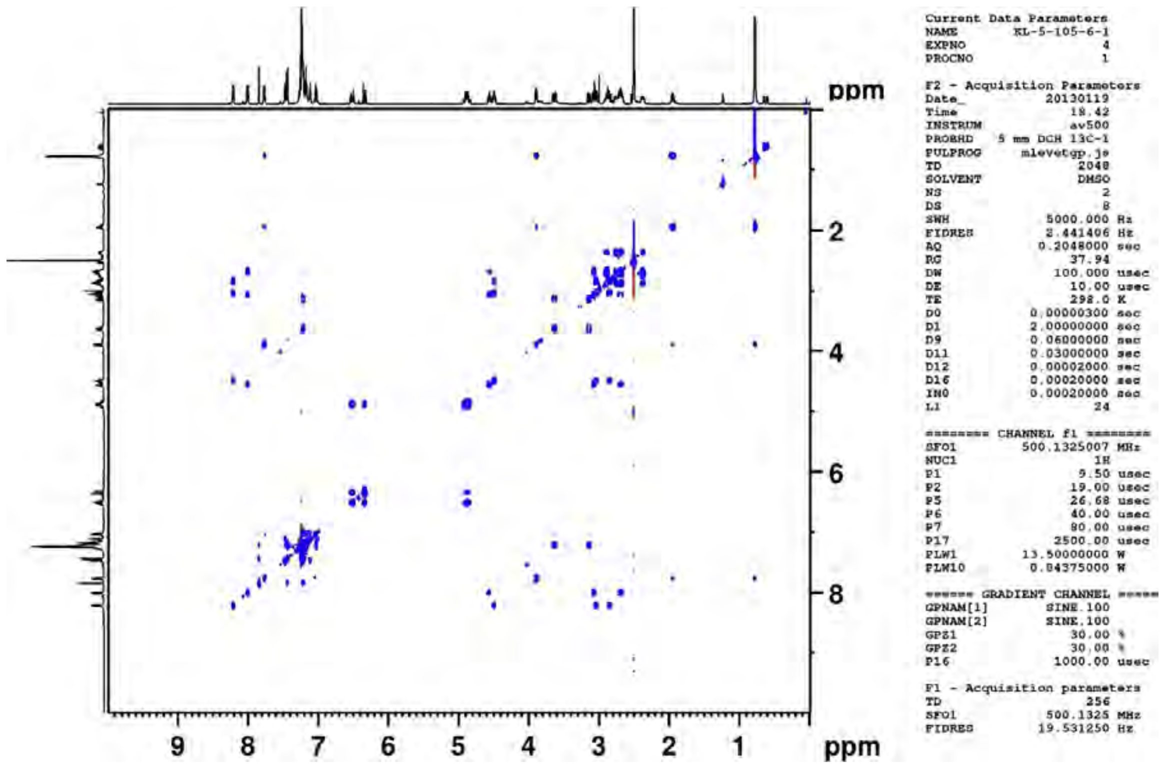


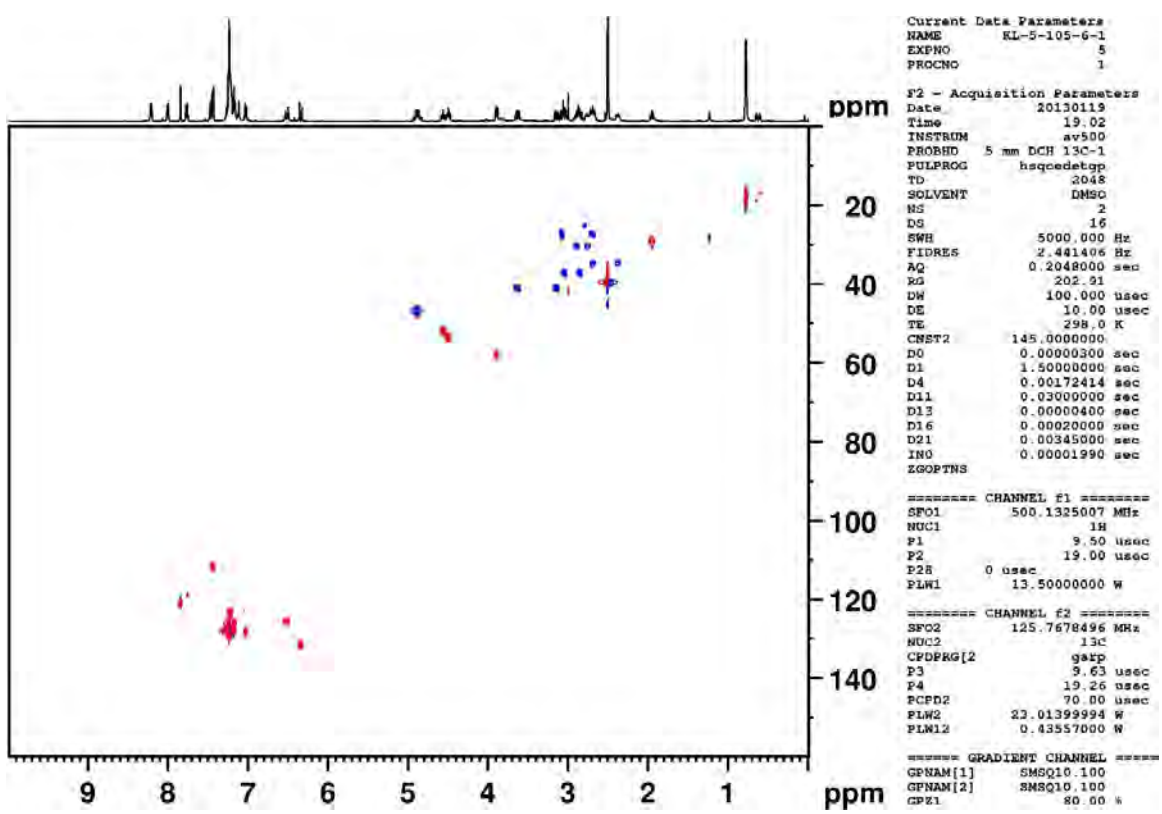
ppm
20
40
60
80
100
120
140
160
11 10 9 8 7 6 5 4 3 2 1
ppm
Current Data Parameters
NAME KL-5-105-4
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121220
Time 22.03
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 32
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 202.91
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W
======== CHANNEL f2 ========
SFO2 125.7703648 MHz
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

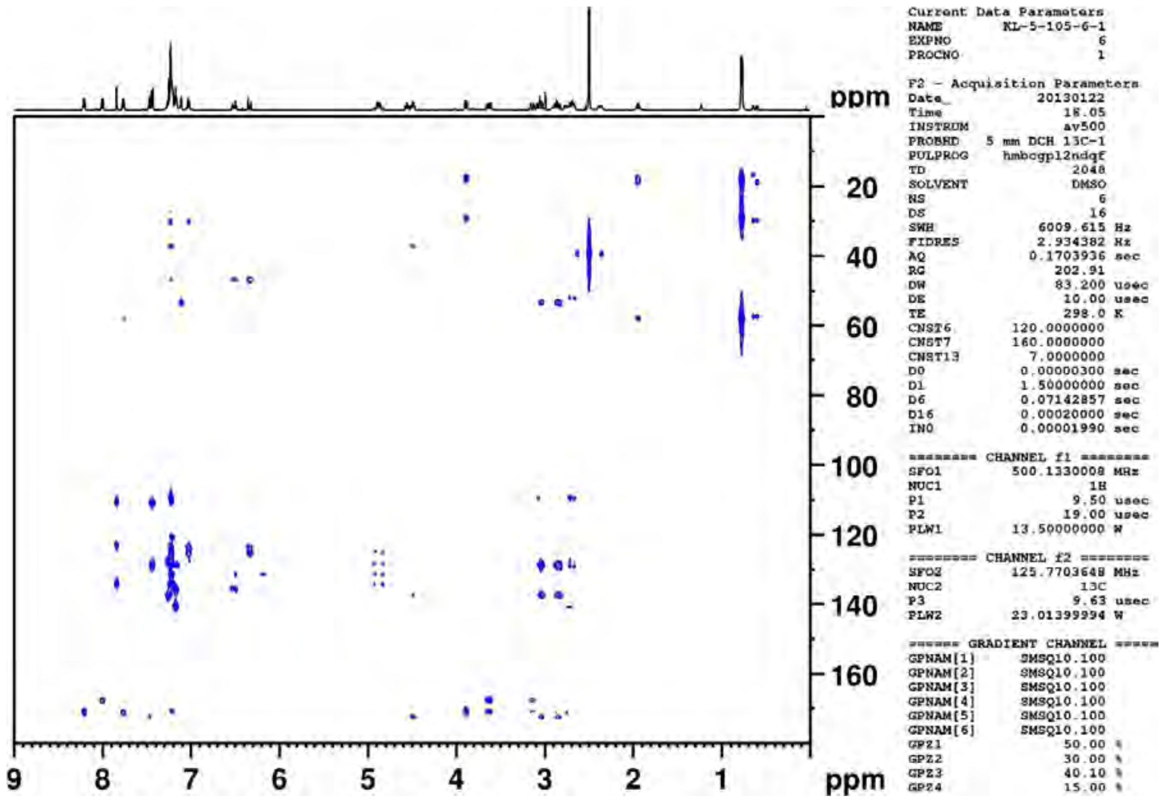
Macrocyclic Product ***S4d***



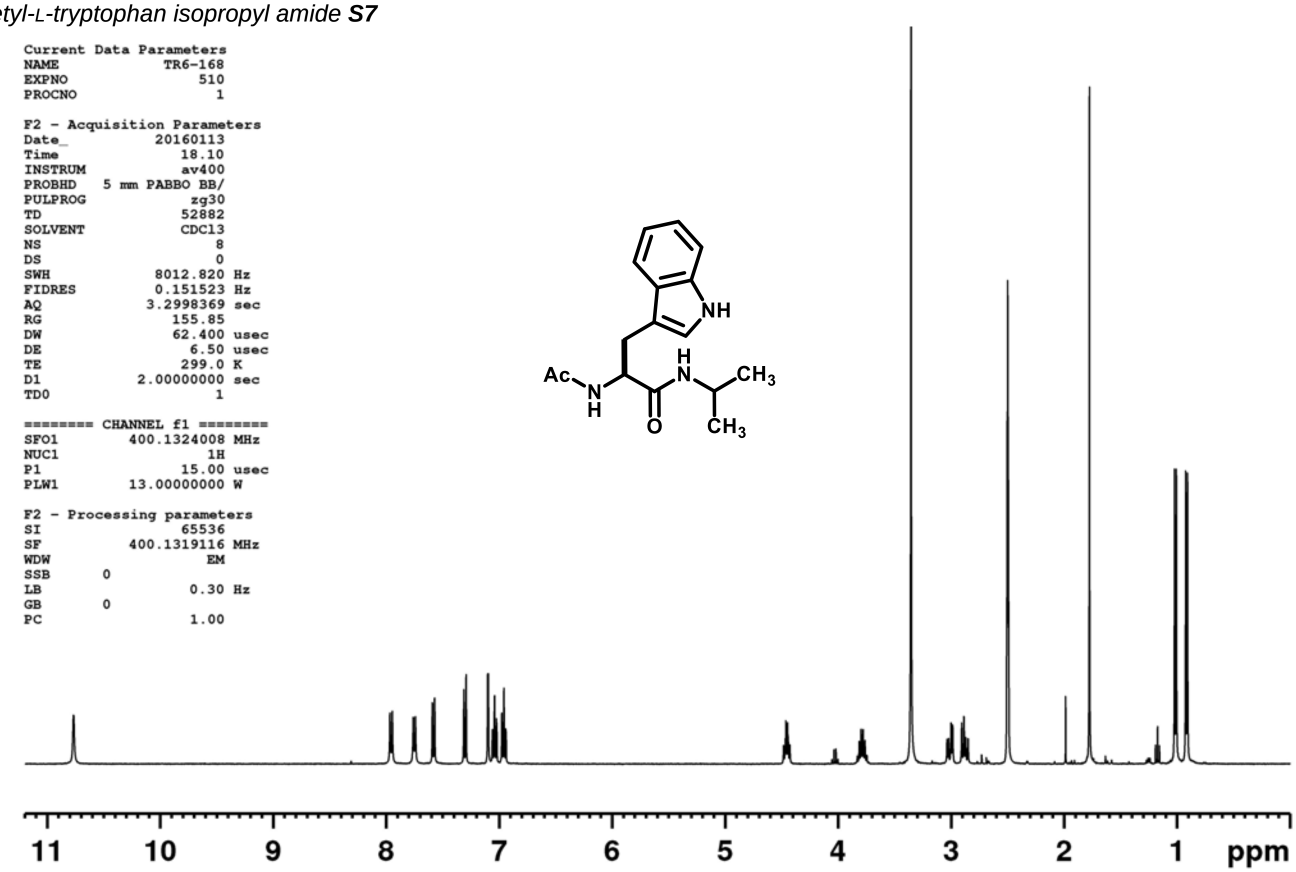


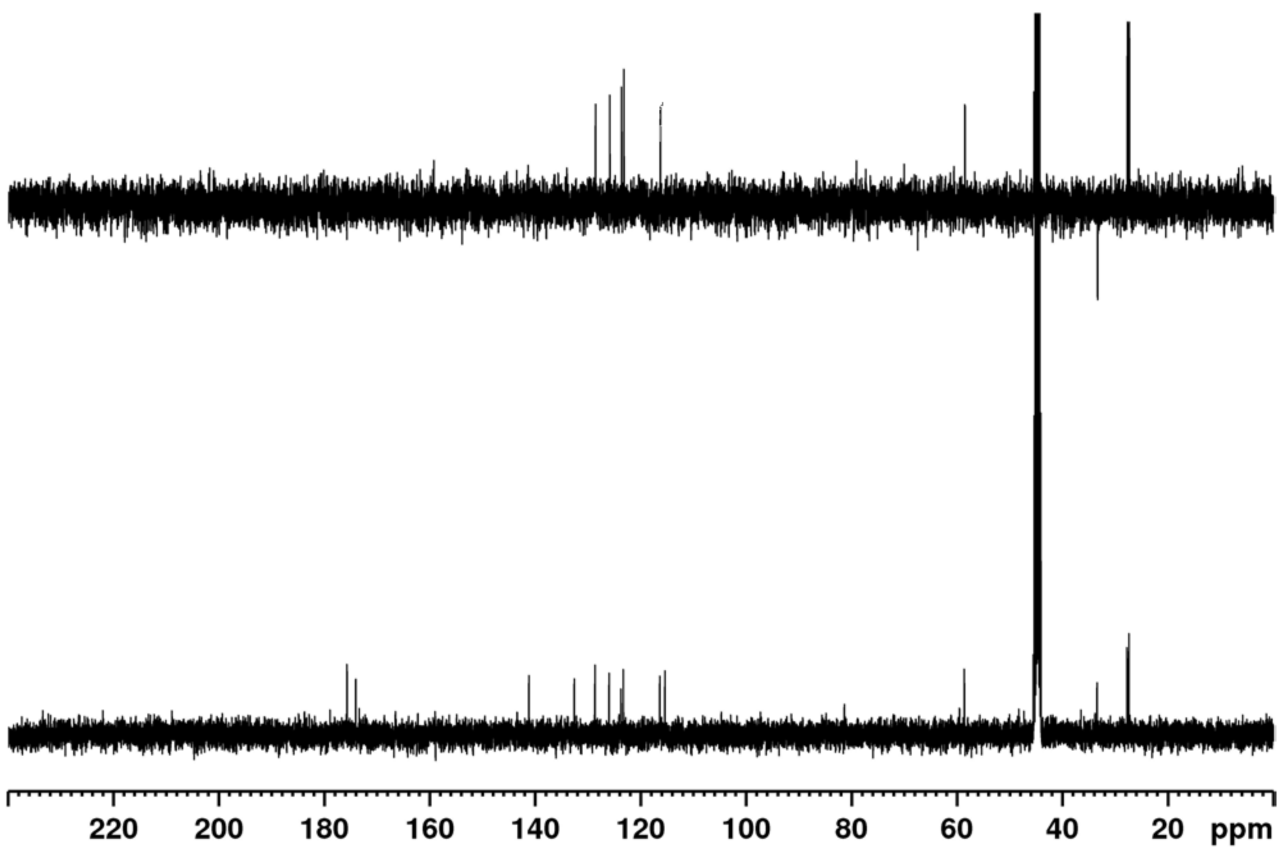

ppm
2
4
6
8
9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME KL-5-105-6-1
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130119
Time 18.42
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG mlevetgp.je
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 37.94
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00020000 sec
L1 24
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 9.50 usec
P2 19.00 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PLW1 13.50000000 W
PLW10 0.84375000 W
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.531250 Hz


ppm
20
40
60
80
100
120
140
9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME KL-5-105-6-1
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130119
Time 19.02
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcedetgp
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 202.91
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
IN0 0.00001990 sec
ZGOPTNS
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 9.50 usec
P2 19.00 usec
P28 0 usec
PLW1 13.50000000 W
======== CHANNEL f2 ========
SFO2 125.7678496 MHz
NUC2 13C
CPDPRG[2 garp
P3 9.63 usec
P4 19.26 usec
PCPD2 70.00 usec
PLW2 23.01399994 W
PLW12 0.43557000 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %


ppm
20
40
60
80
100
120
140
160
9
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME KL-5-105-6-1
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130122
Time 18.05
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 6
DS 16
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 202.91
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 9.50 usec
P2 19.00 usec
PLW1 13.50000000 W
======== CHANNEL f2 ========
SFO2 125.7703648 MHz
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %

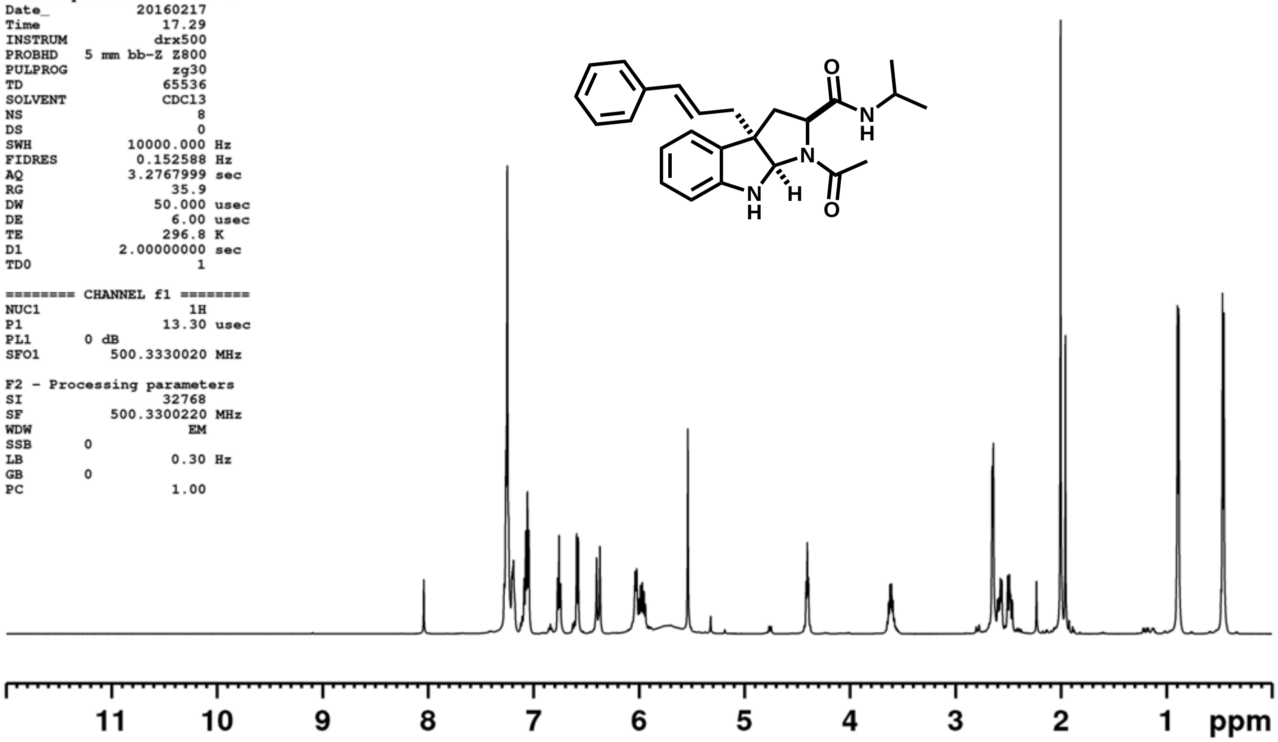
N-acetyl-L-tryptophan isopropyl amide ***S7***

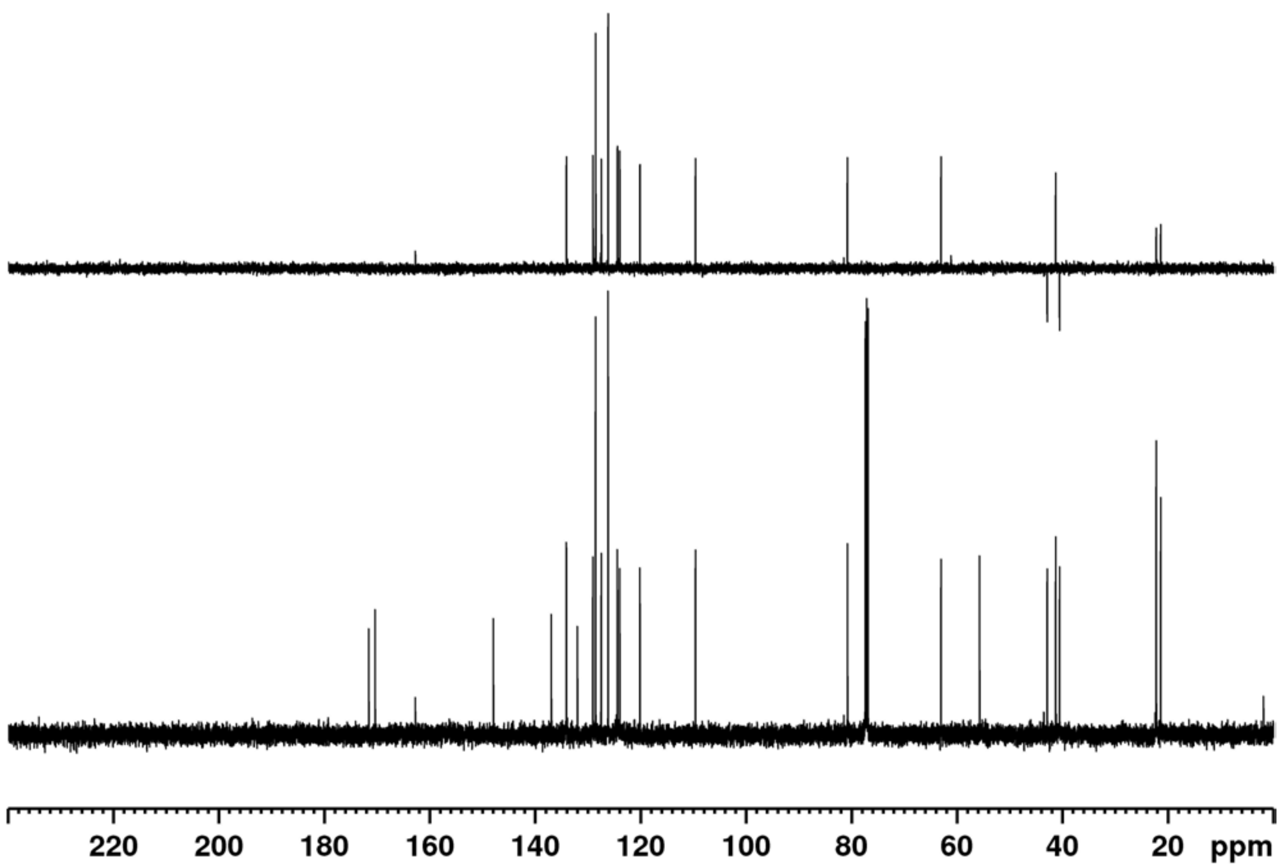


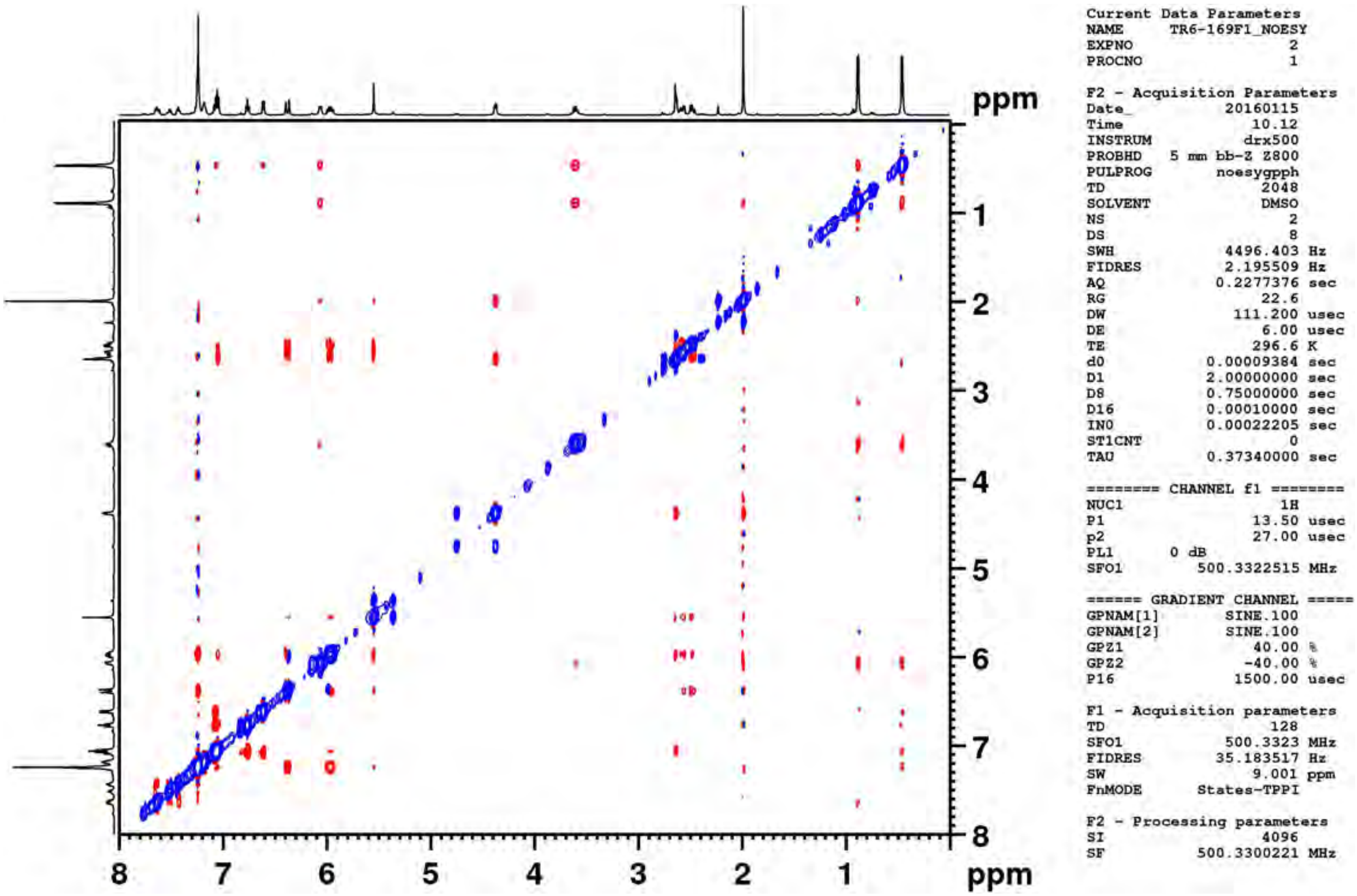


endo-pyrroloindoline ***21a***

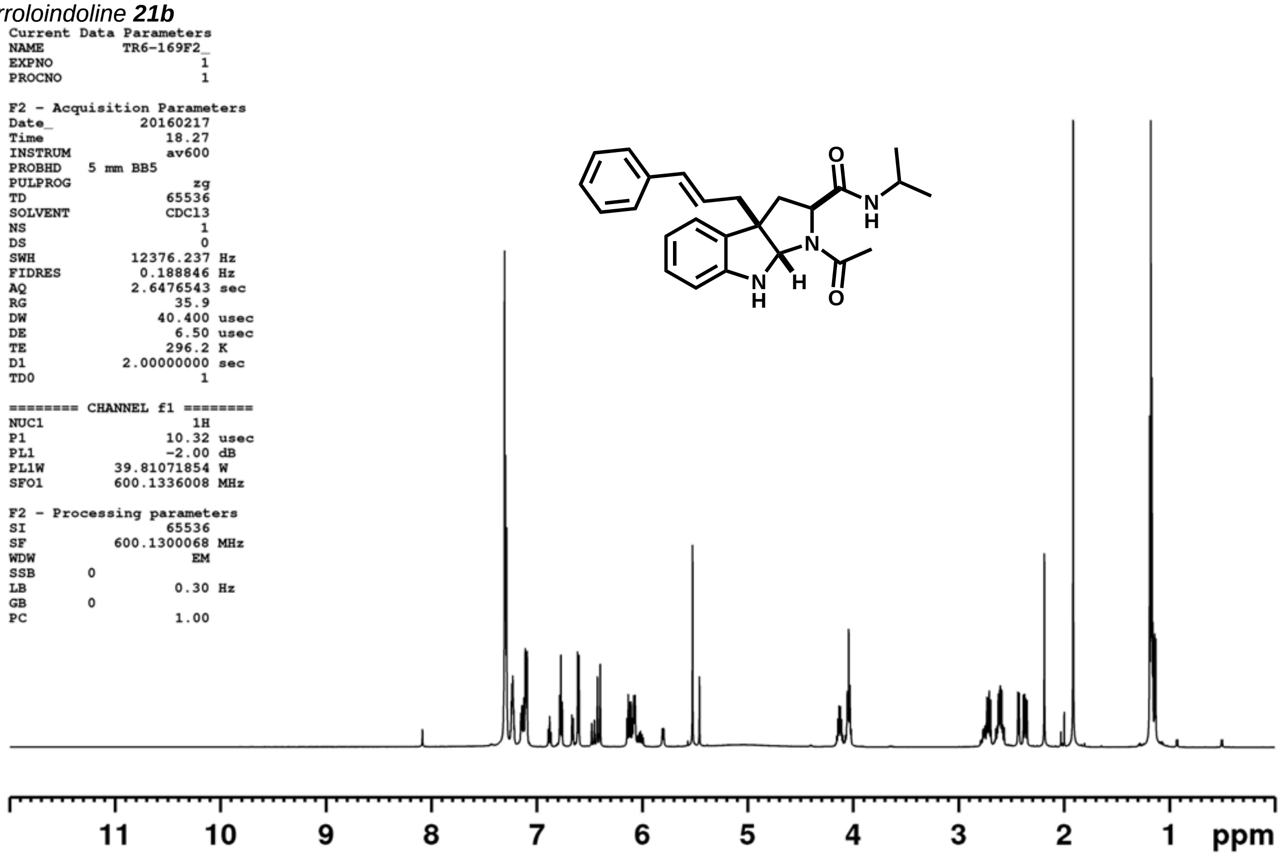
```
Current Data Parameters
NAME          TR6-169F1_
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160217
Time               17.29
INSTRUM           drx500
PROBHD   5 mm bb-Z Z800
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            10000.000 Hz
FIDRES          0.152588 Hz
AQ             3.2767999 sec
RG                  35.9
DW                50.000 usec
DE                  6.00 usec
TE                 296.8 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.30 usec
PL1         0 dB
SFO1        500.3330020 MHz

F2 - Processing parameters
SI                 32768
SF          500.3300220 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```

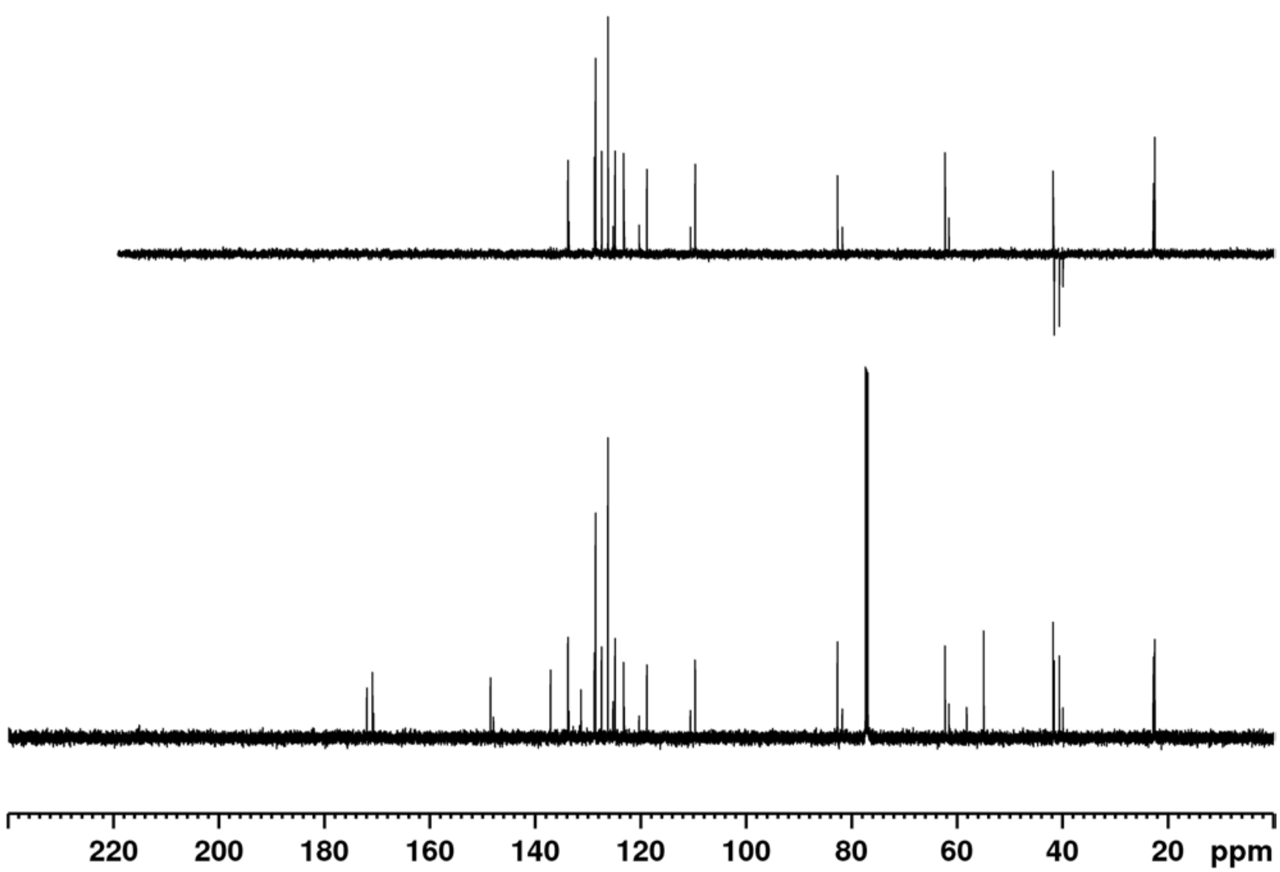


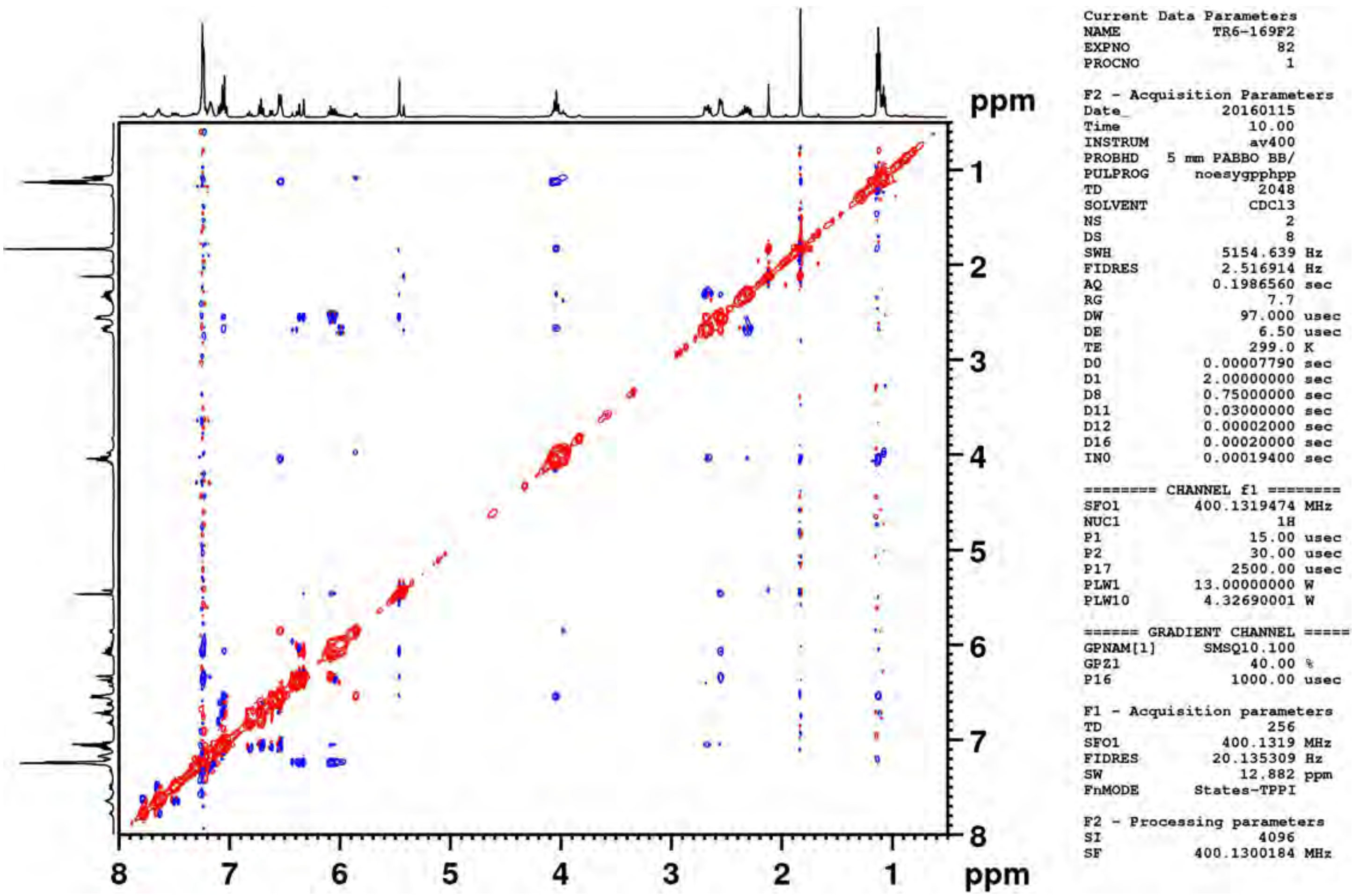



ppm
ppm
Current Data Parameters
NAME TR6-169F1_NOESY
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160115
Time 10.12
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG noesygpph
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 4496.403 Hz
FIDRES 2.195509 Hz
AQ 0.2277376 sec
RG 22.6
DW 111.200 usec
DE 6.00 usec
TE 296.6 K
d0 0.00009384 sec
D1 2.00000000 sec
D8 0.75000000 sec
D16 0.00010000 sec
IN0 0.00022205 sec
ST1CNT 0
TAU 0.37340000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
p2 27.00 usec
PL1 0 dB
SFO1 500.3322515 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 40.00 %
GPZ2 -40.00 %
P16 1500.00 usec
F1 - Acquisition parameters
TD 128
SFO1 500.3323 MHz
FIDRES 35.183517 Hz
SW 9.001 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 4096
SF 500.3300221 MHz

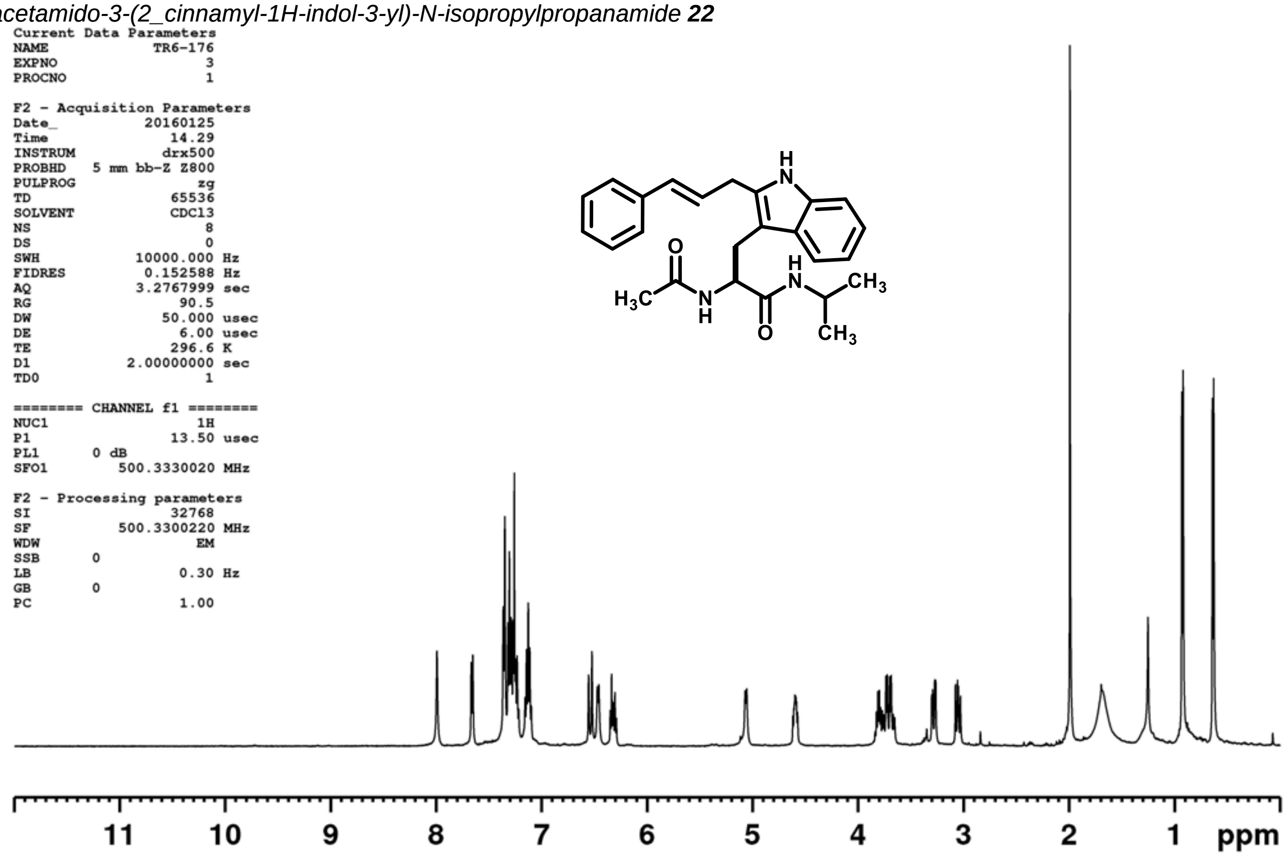
exo-pyrroloindoline ***21b***

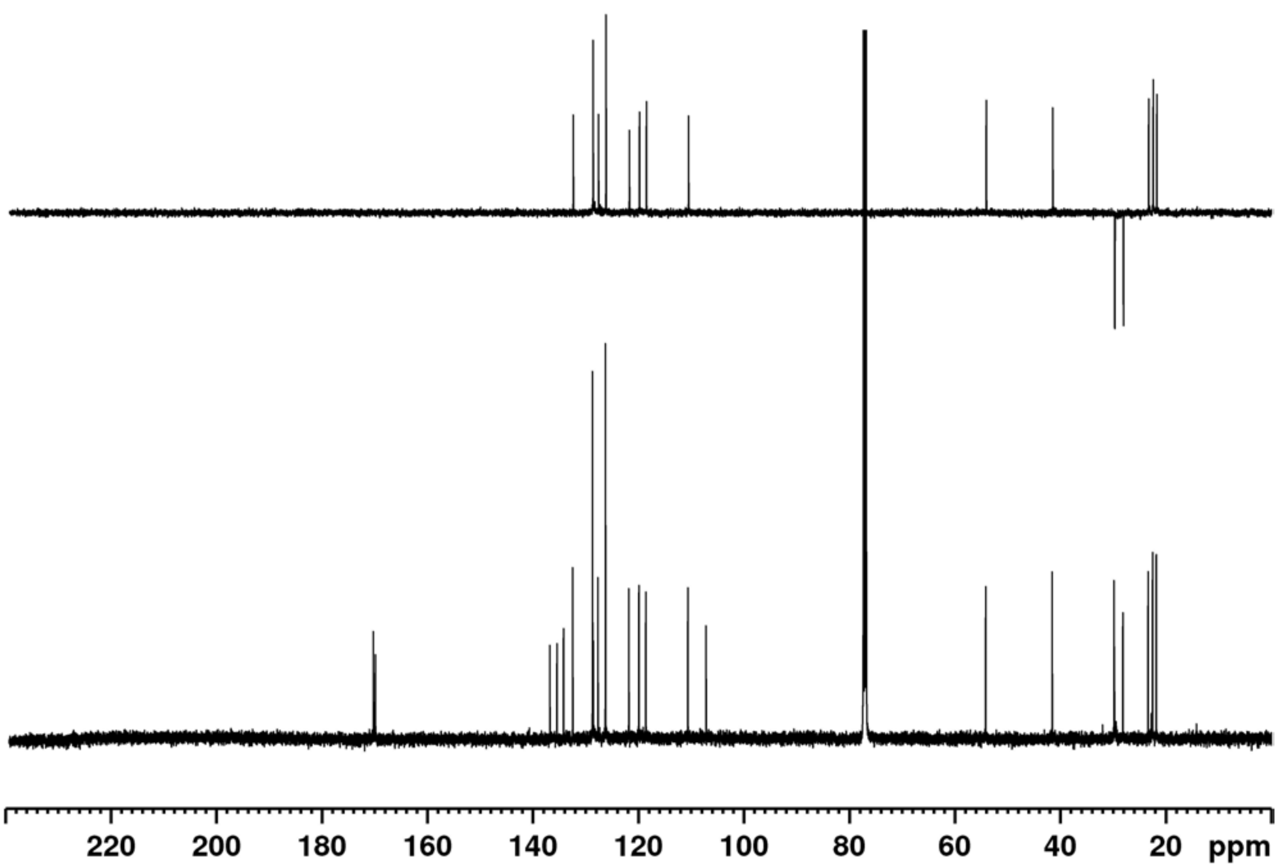




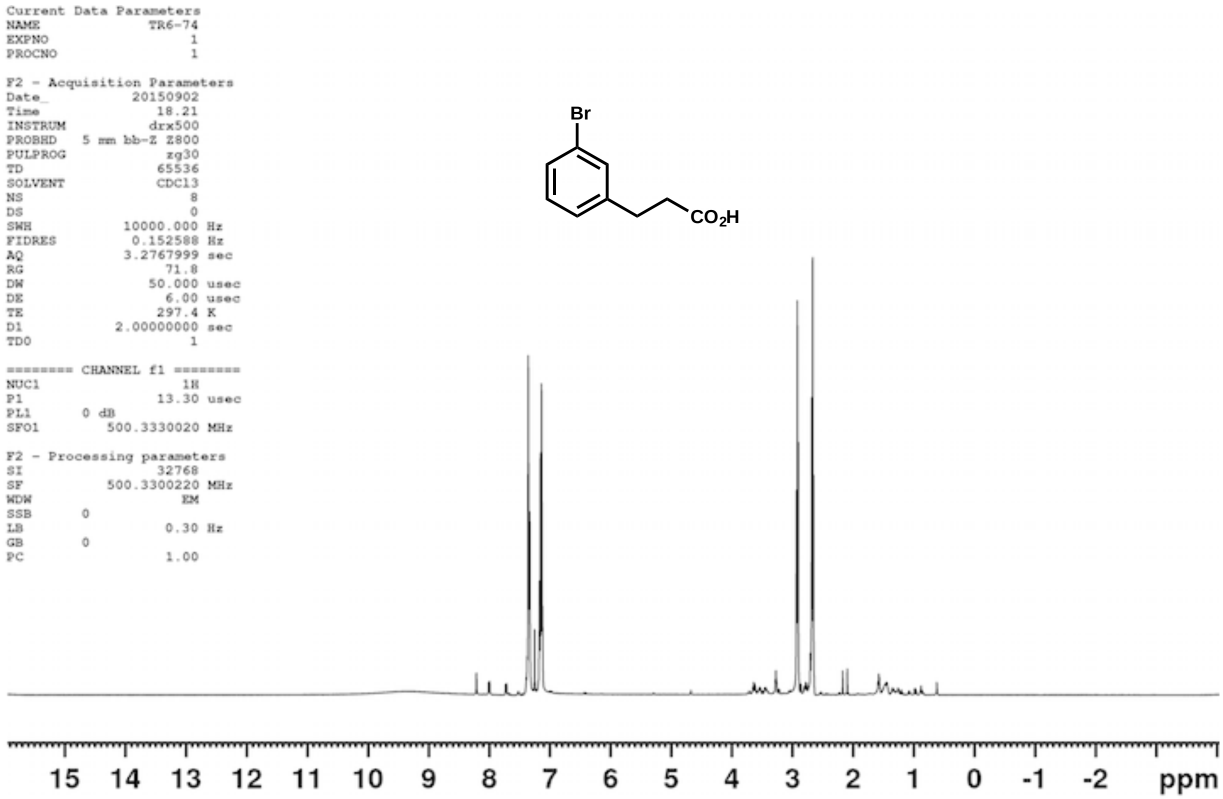

ppm
1
2
3
4
5
6
7
8
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME TR6-169F2
EXPNO 82
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160115
Time 10.00
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG noesygpphpp
TD 2048
SOLVENT CDCl3
NS 2
DS 8
SWH 5154.639 Hz
FIDRES 2.516914 Hz
AQ 0.1986560 sec
RG 7.7
DW 97.000 usec
DE 6.50 usec
TE 299.0 K
D0 0.00007790 sec
D1 2.00000000 sec
D8 0.75000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00019400 sec
======== CHANNEL f1 ========
SFO1 400.1319474 MHz
NUC1 1H
P1 15.00 usec
P2 30.00 usec
P17 2500.00 usec
PLW1 13.00000000 W
PLW10 4.32690001 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPZ1 40.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 400.1319 MHz
FIDRES 20.135309 Hz
SW 12.882 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 4096
SF 400.1300184 MHz

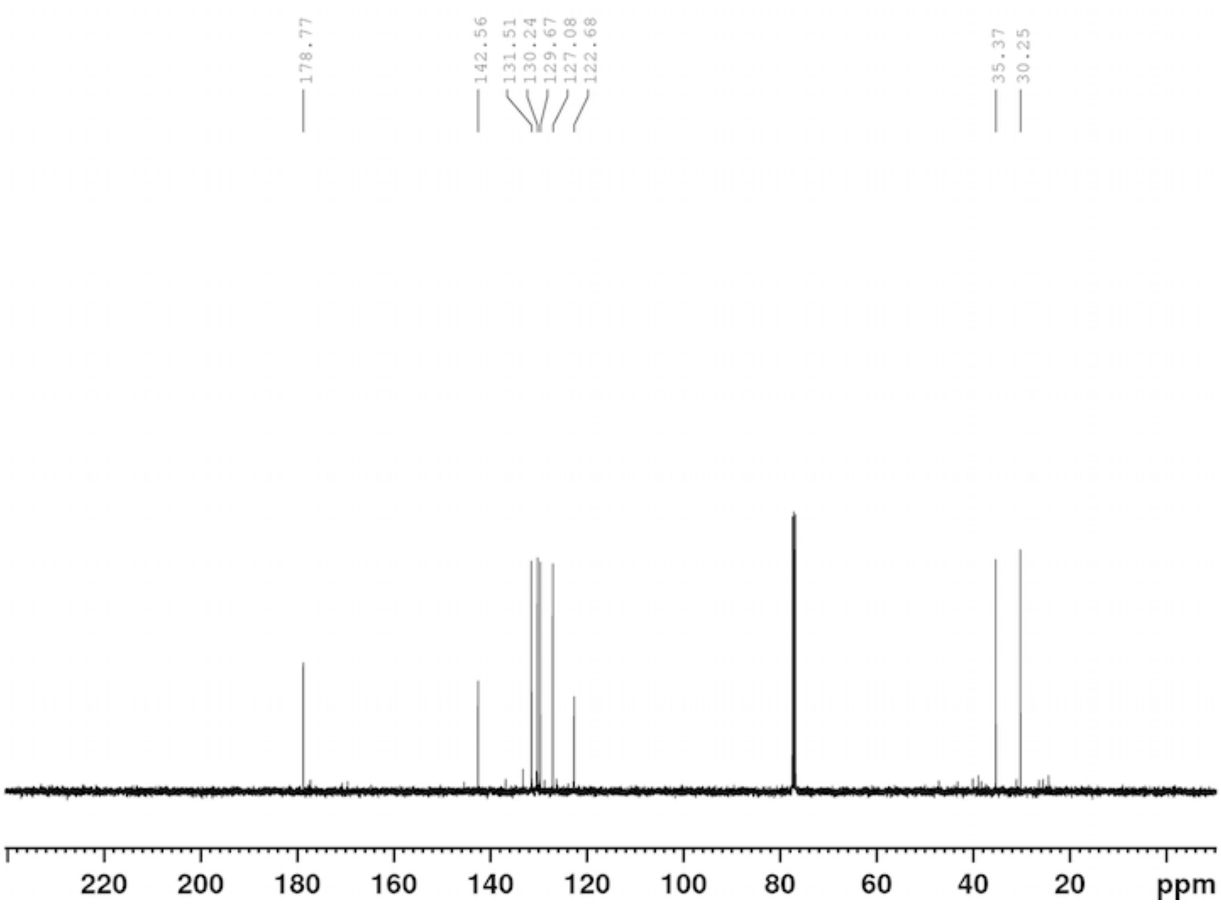
(S)-2-acetamido-3-(2_cinnamyl-1H-indol-3-yl)-N-isopropylpropanamide ***22***



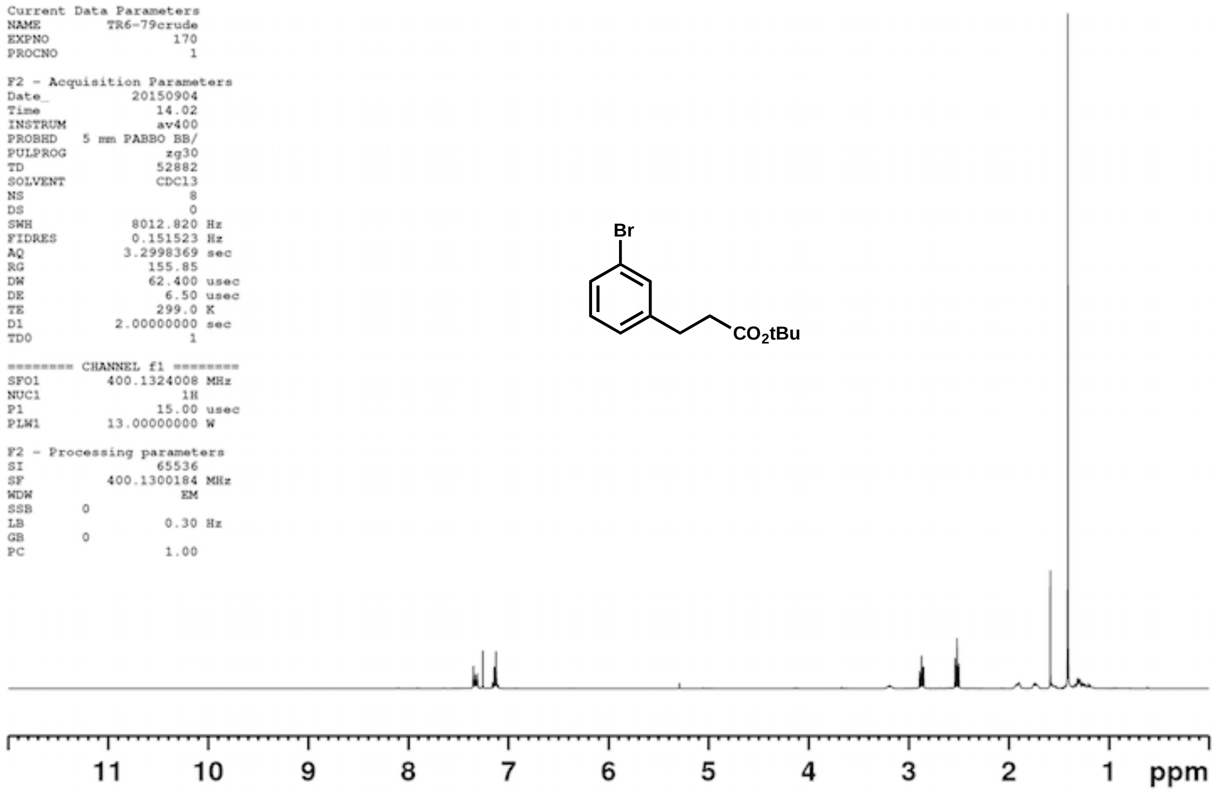


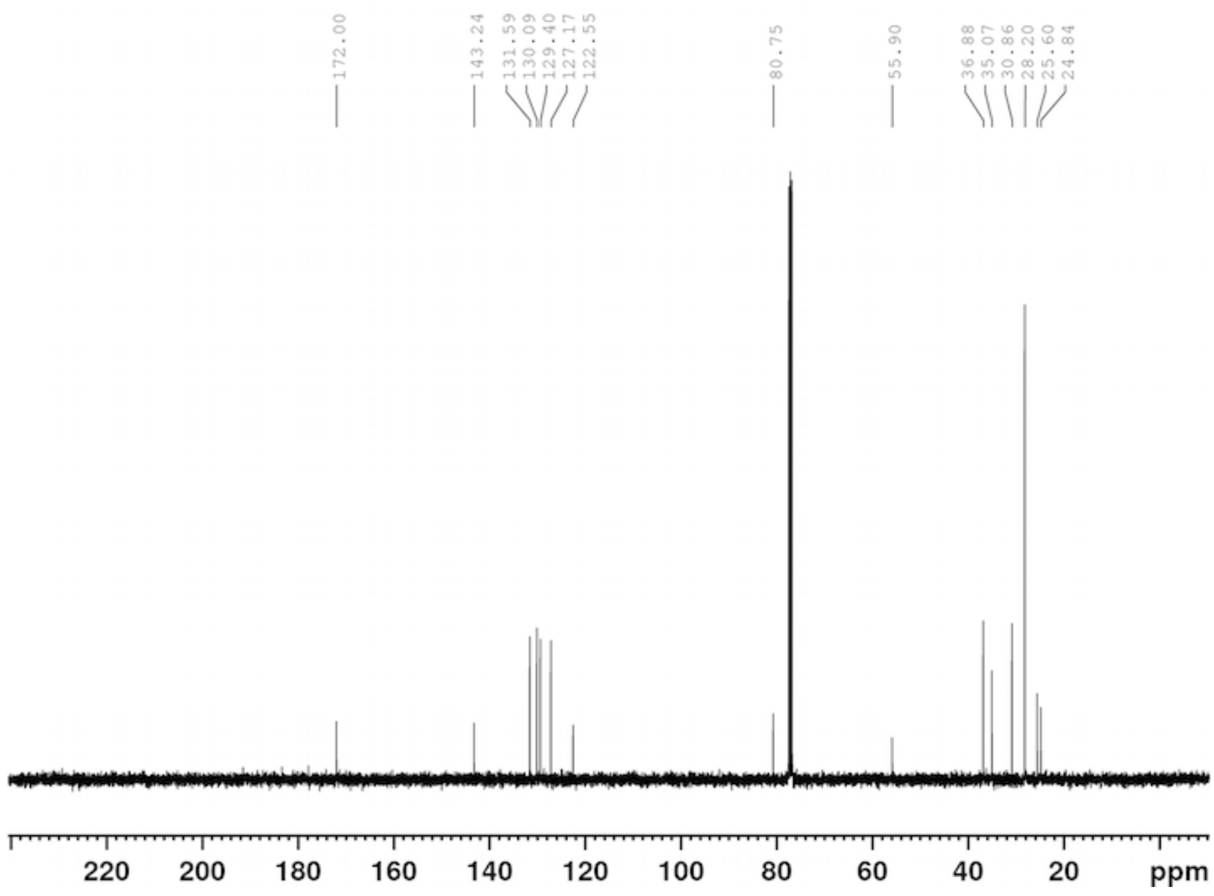
3-(3-Bromophenyl)propionic acid ***S5***



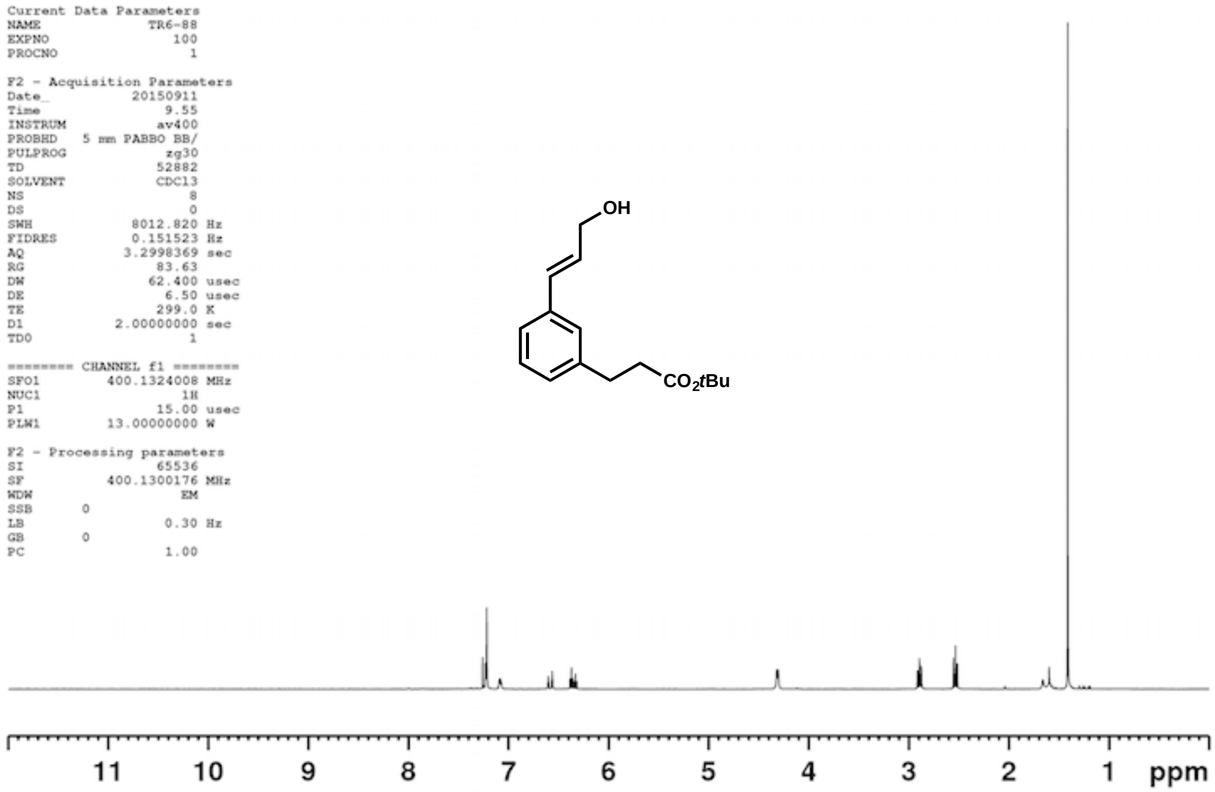


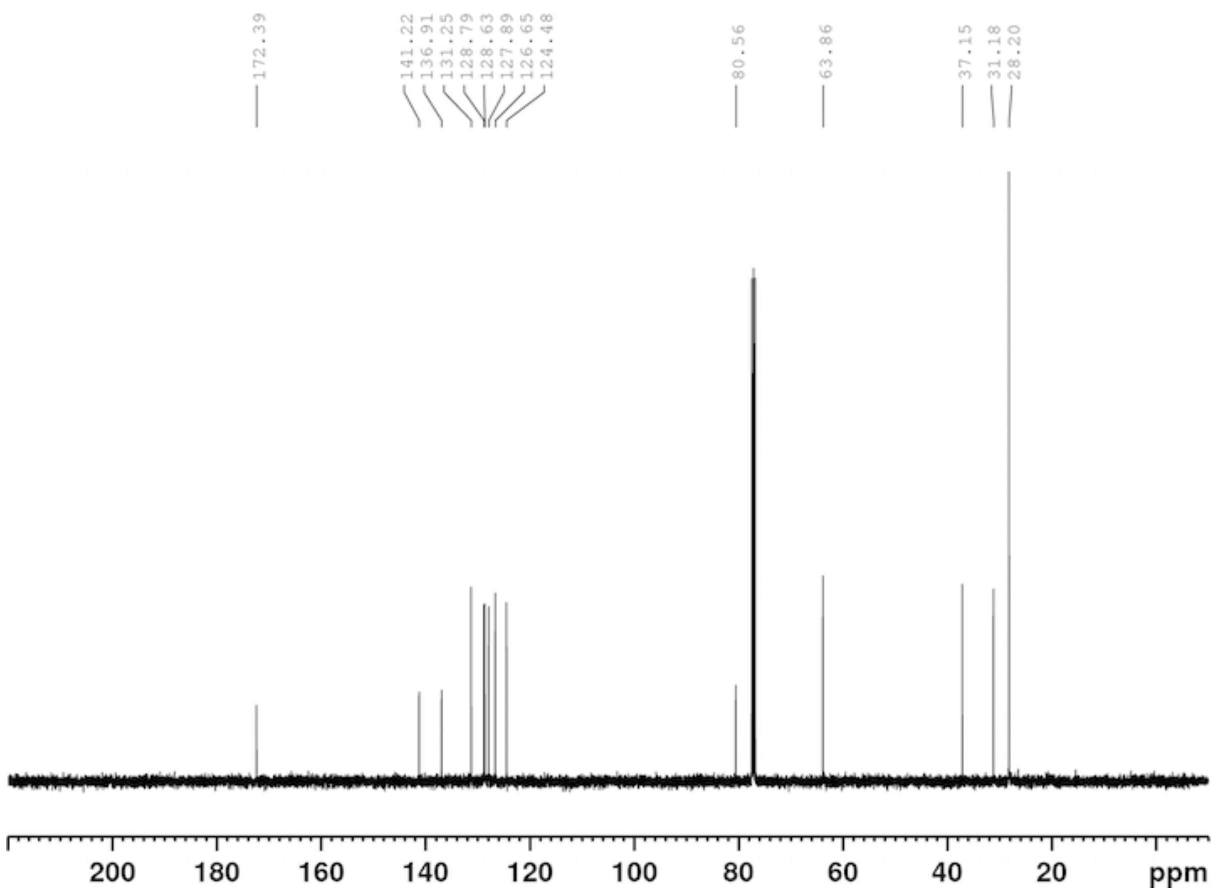
tert-Butyl 3-(3-bromophenyl)propanoate ***S6***



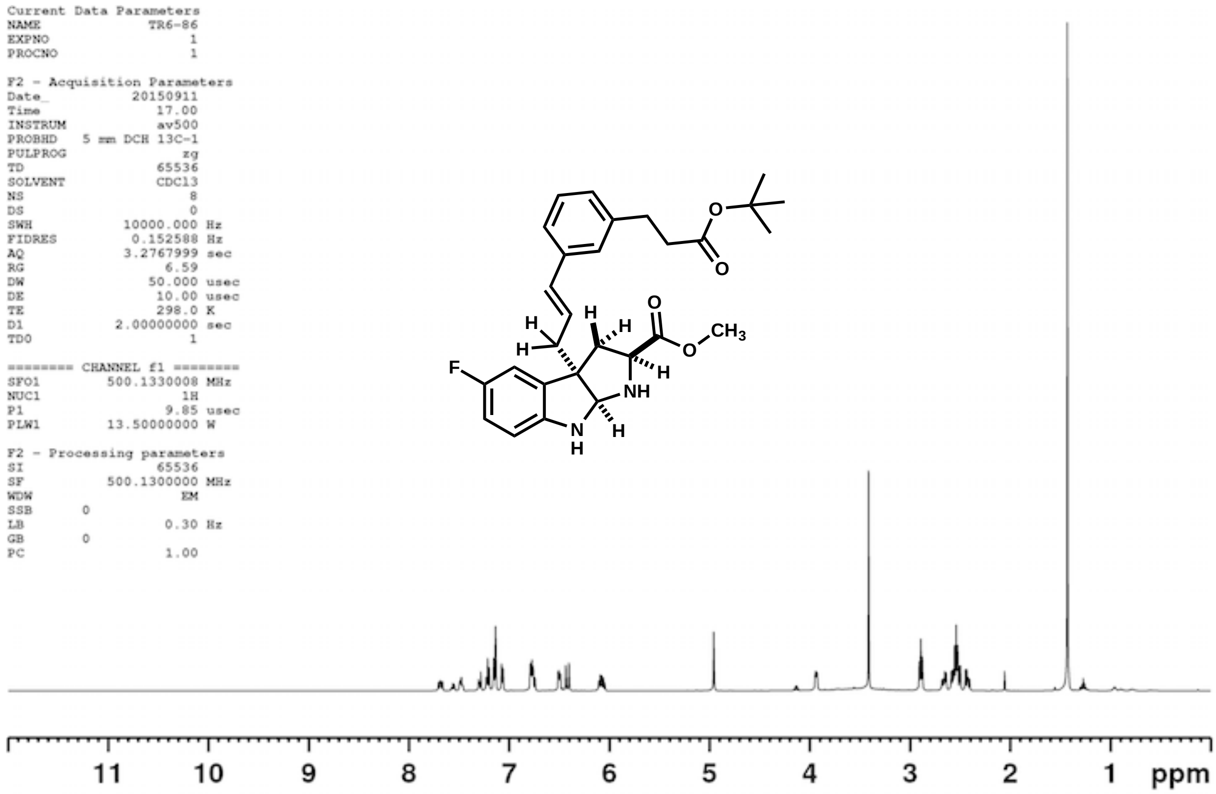


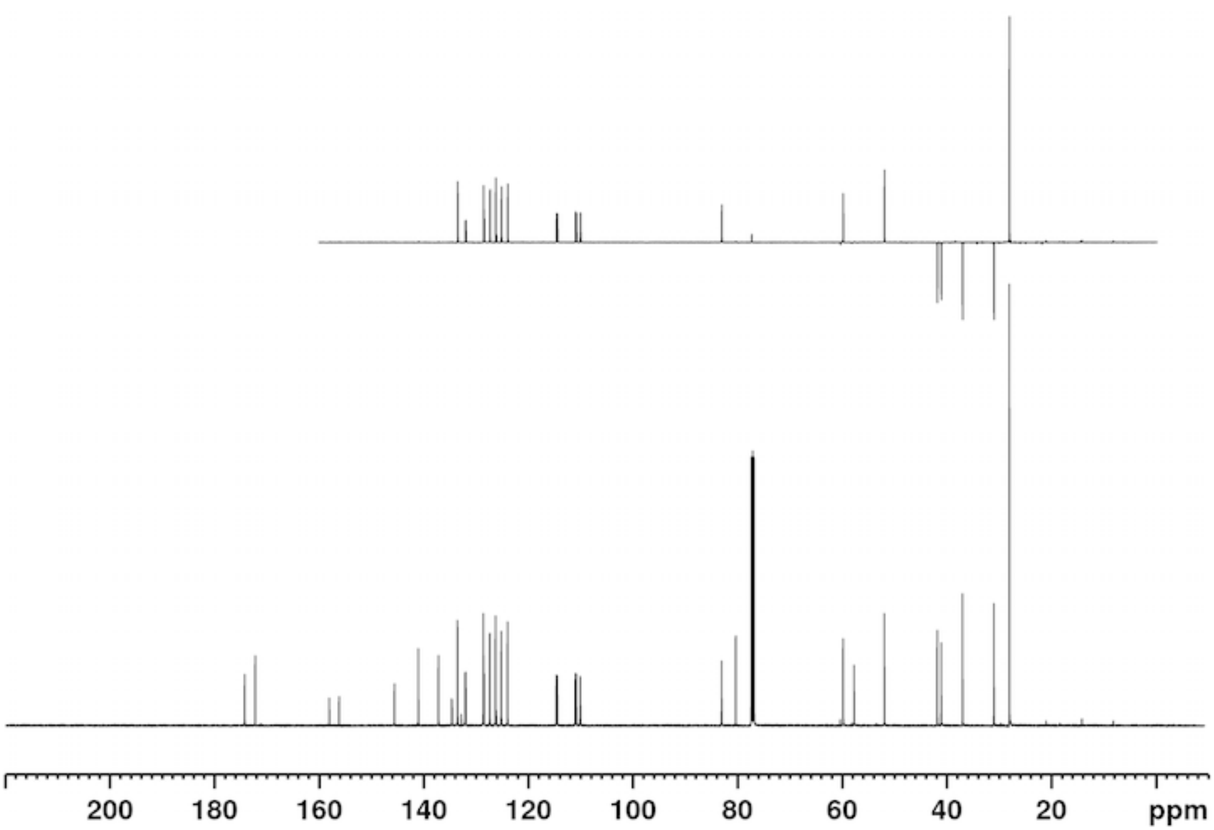
Cinnamyl Alcohol ***21***

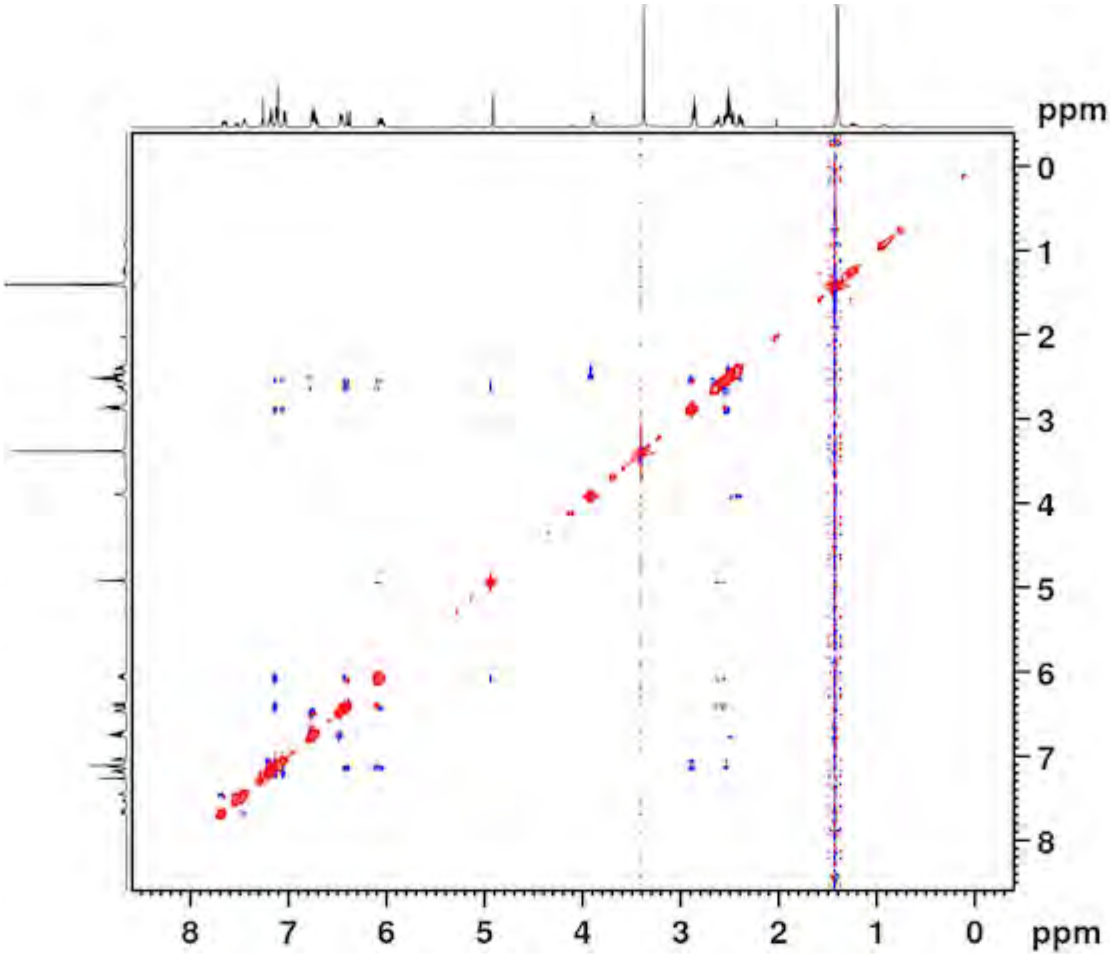




*endo-Pyrroloindoline **23***



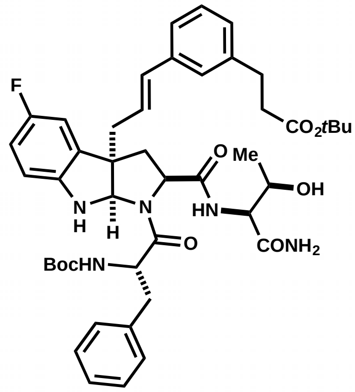


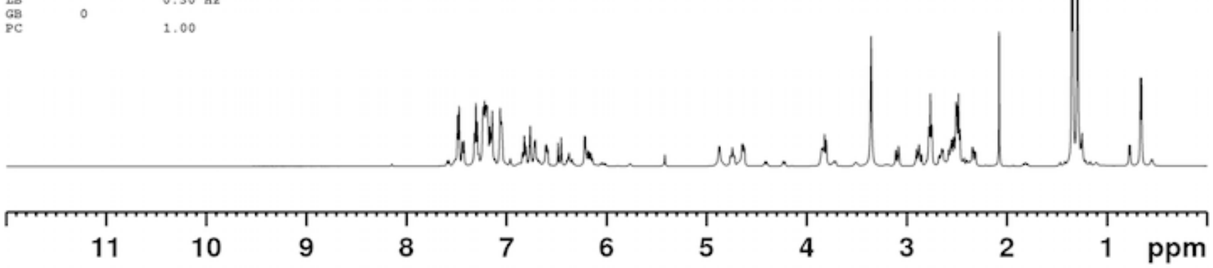


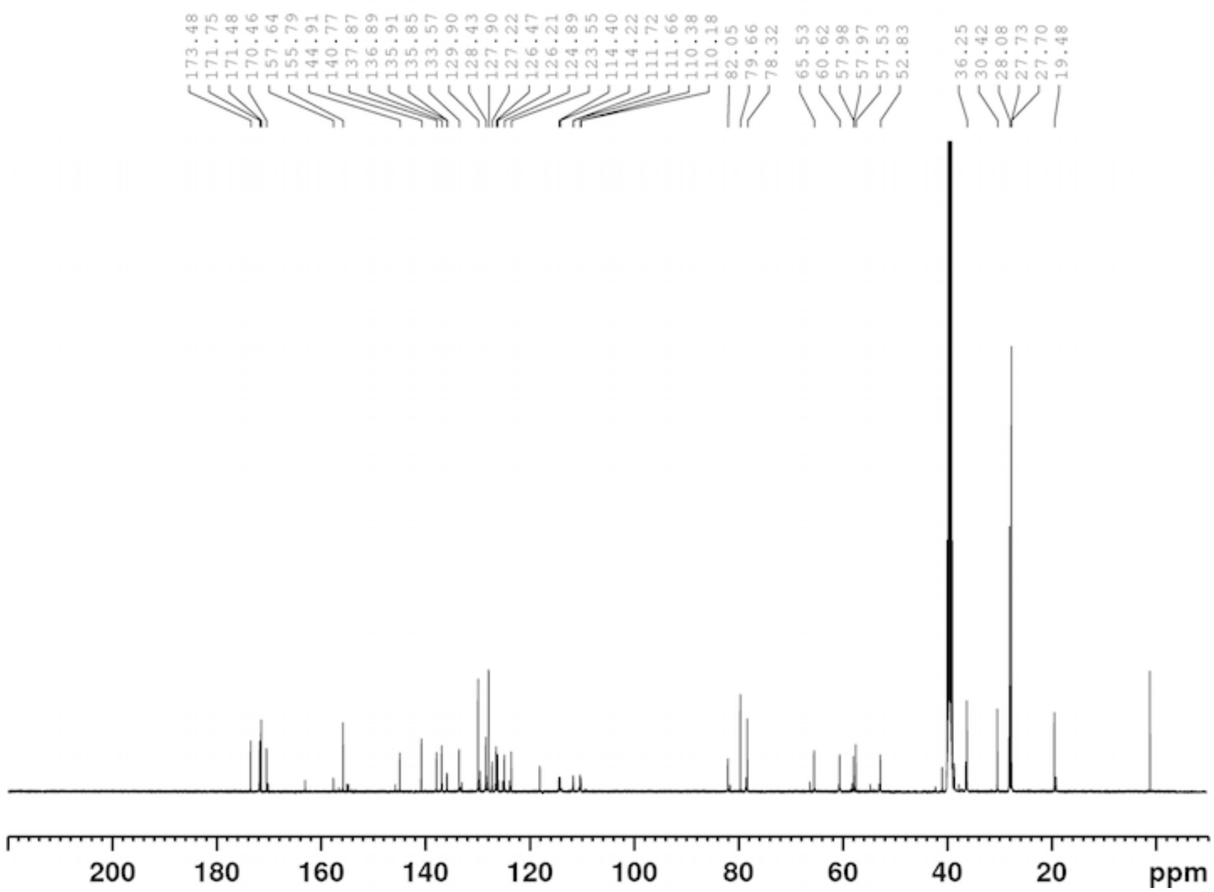
Current Data Parameters		
NAME	TR6-77B_400	
EXPNO	52	
PROCNO	1	
F2 - Acquisition Parameters		
Date_	20150904	
Time	9.27	
INSTRUM	av400	
PROBHD	5 mm PABBO BB/	
PULPROG	noesygpphpp	
TD	2048	
SOLVENT	CDCl3	
NS	2	
DS	8	
SWH	3597.122	Hz
FIDRES	1.756407	Hz
AQ	0.2846720	sec
RG	8.85	
DW	139.000	usec
DE	6.50	usec
TE	299.0	K
D0	0.00011990	sec
D1	2.00000000	sec
D8	0.75000000	sec
D11	0.03000000	sec
D12	0.00002000	sec
D16	0.00020000	sec
IN0	0.00027800	sec
======== CHANNEL f1 ========		
SFO1	400.1316472	MHz
NUC1	1H	
P1	15.00	usec
P2	30.00	usec
P17	2500.00	usec
PLW1	13.00000000	W
PLW10	4.32690001	W
====== GRADIENT CHANNEL =====		
GPNAM[1]	SMSQ10.100	
GPZ1	40.00	%
P16	1000.00	usec
F1 - Acquisition parameters		
TD	256	
SFO1	400.1316	MHz
FIDRES	14.051259	Hz
SW	8.990	ppm
FnMODE	States-TPPI	
F2 - Processing parameters		
SI	2048	
SF	400.1300062	MHz

tert-butyl ester ***24***

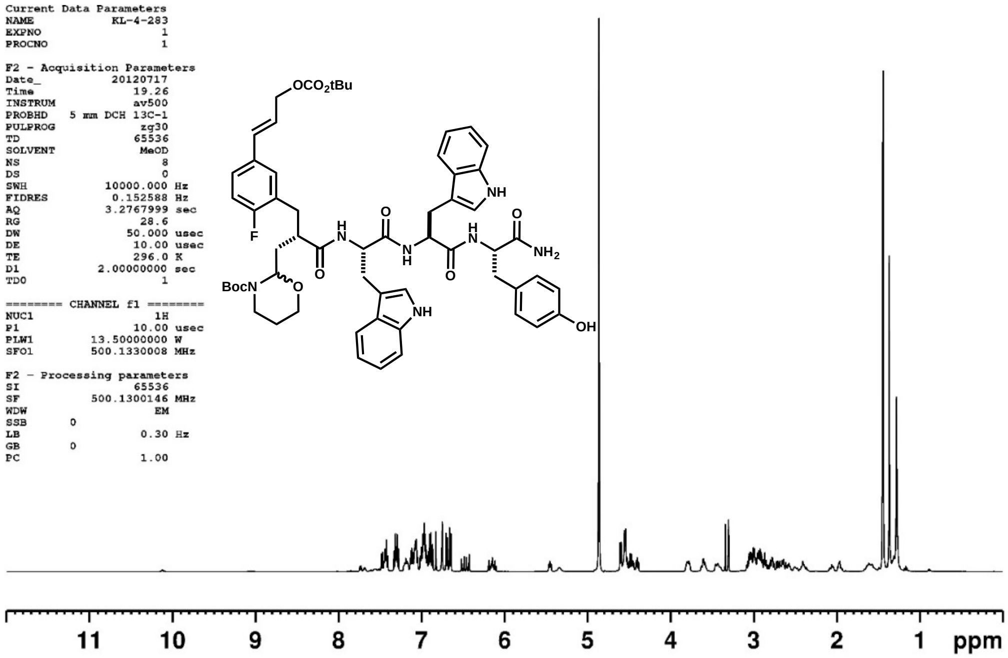
```
Current Data Parameters
NAME          TR6-98_500
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151005
Time                8.38
INSTRUM            av500
PROBHD   5 mm DCH 13C-1
PULPROG               zg
TD                 65536
SOLVENT             DMSO
NS                     8
DS                     0
SWH            10000.000 Hz
FIDRES          0.152588 Hz
AQ             3.2767999 sec
RG                  6.59
DW                50.000 usec
DE                 10.00 usec
TE                 298.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1316687 MHz
NUC1                  1H
P1                 10.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300000 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```

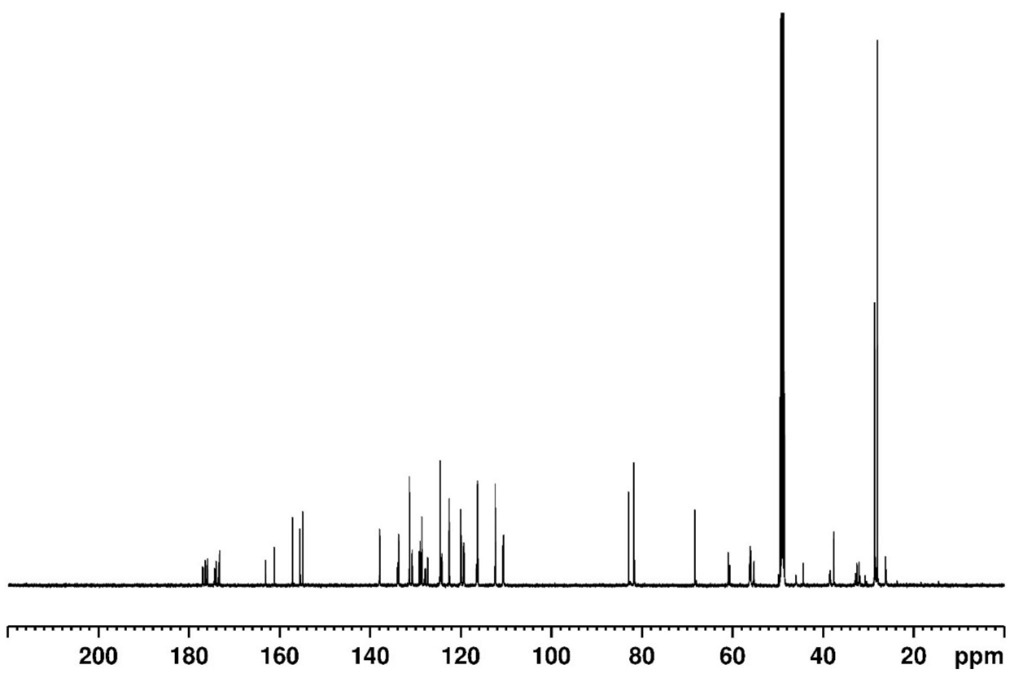




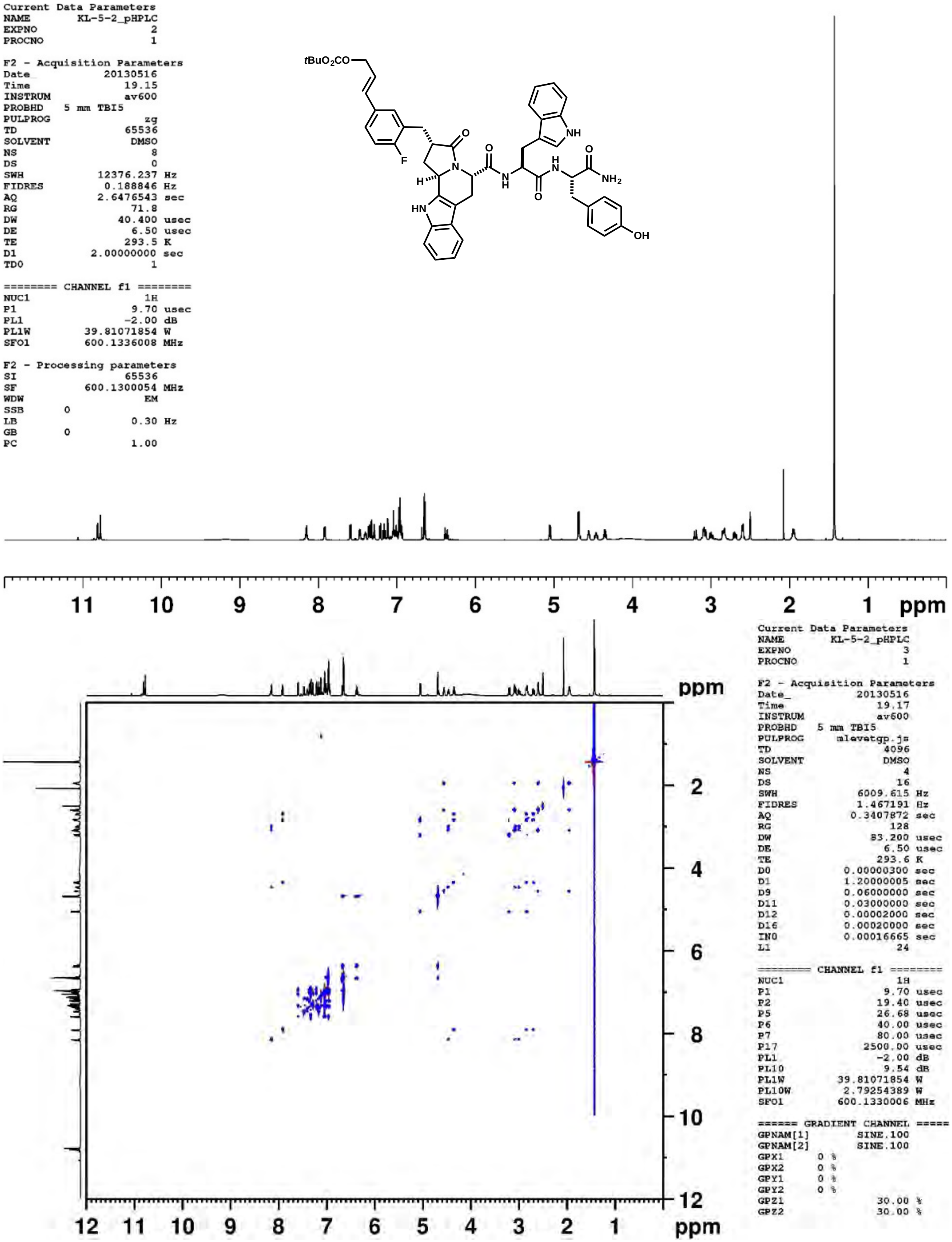


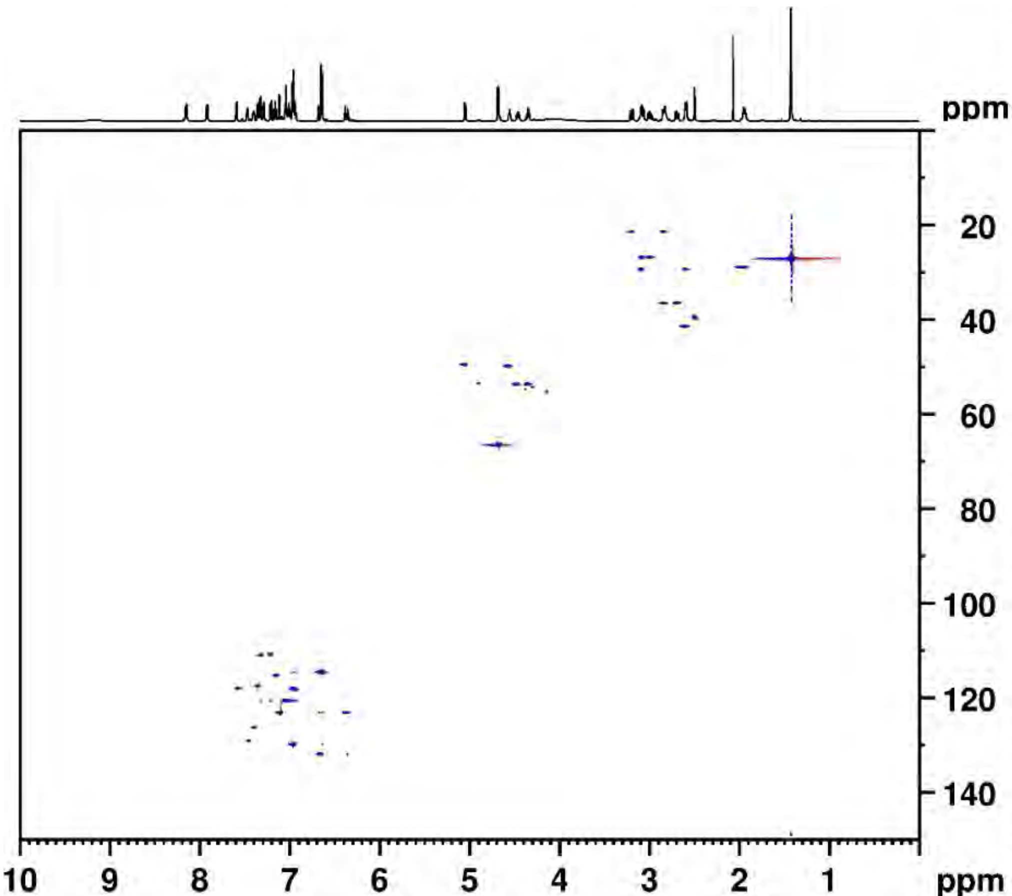
*Acyclic Precursor **S8***





*Tryptoline **27***



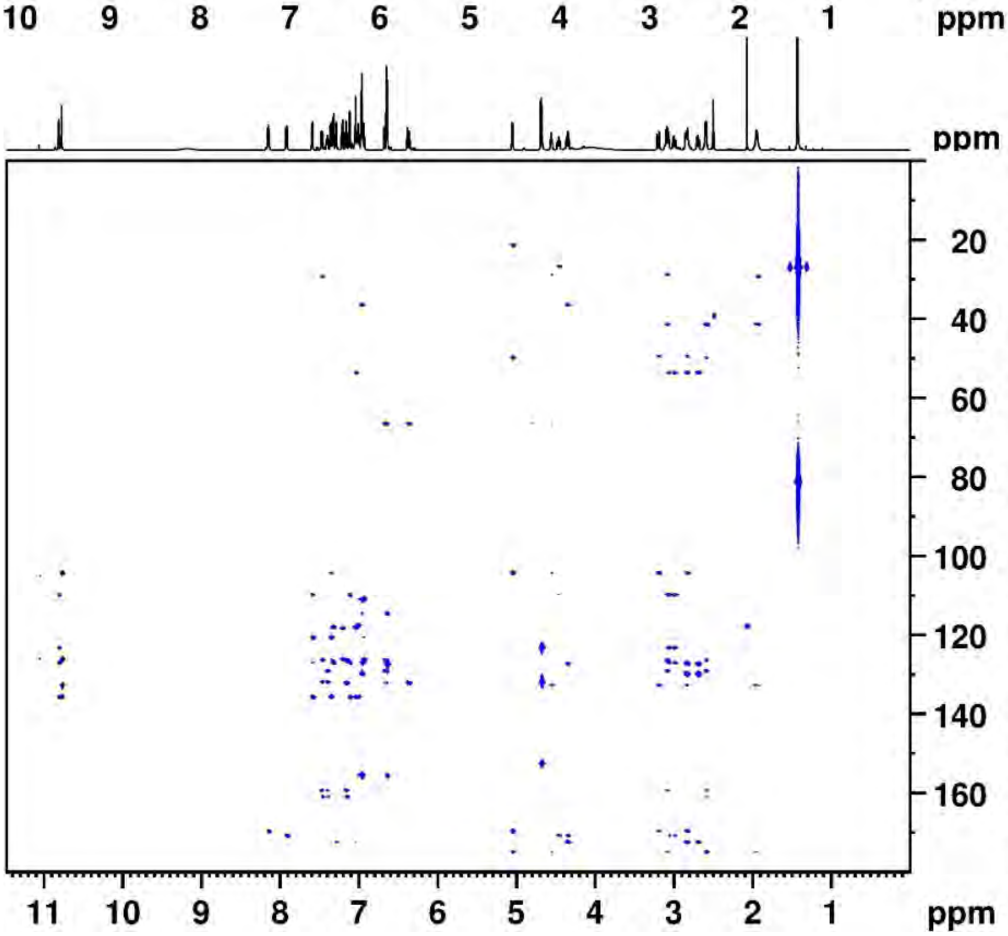


Current Data Parameters
NAME KL-5-2_pHPLC
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130516
Time 20.25
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425408 sec
RG 16384
DW 69.600 usec
DE 6.00 usec
TE 294.3 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1 1H
P1 9.70 usec
P2 19.40 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1330006 MHz

======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 18.50 usec
P4 37.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.91 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 12.19330025 W
SFO2 150.9133722 MHz



Current Data Parameters
NAME KL-5-2_pHPLC
EXPNO 5
PROCNO 1

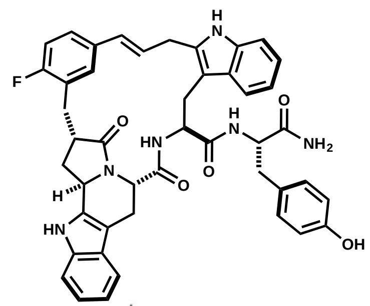
F2 - Acquisition Parameters
Date_ 20130516
Time 22.00
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 16
DS 64
SWH 6887.052 Hz
FIDRES 3.362818 Hz
AQ 0.1486848 sec
RG 26008
DW 72.600 usec
DE 6.00 usec
TE 296.0 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec

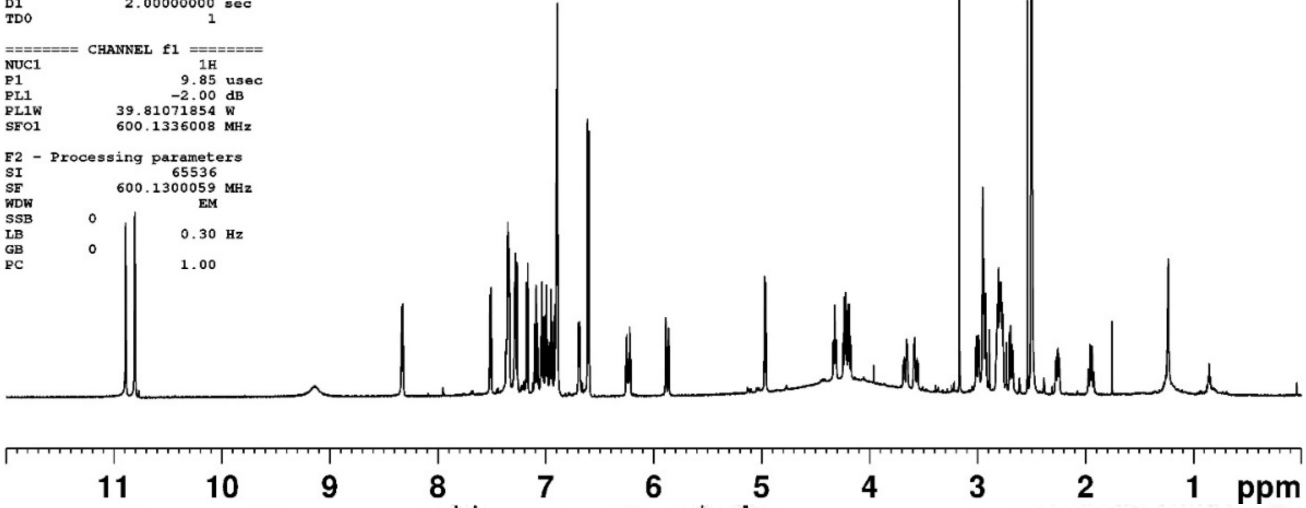
======== CHANNEL f1 ========
NUC1 1H
P1 9.70 usec
P2 19.40 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1334507 MHz

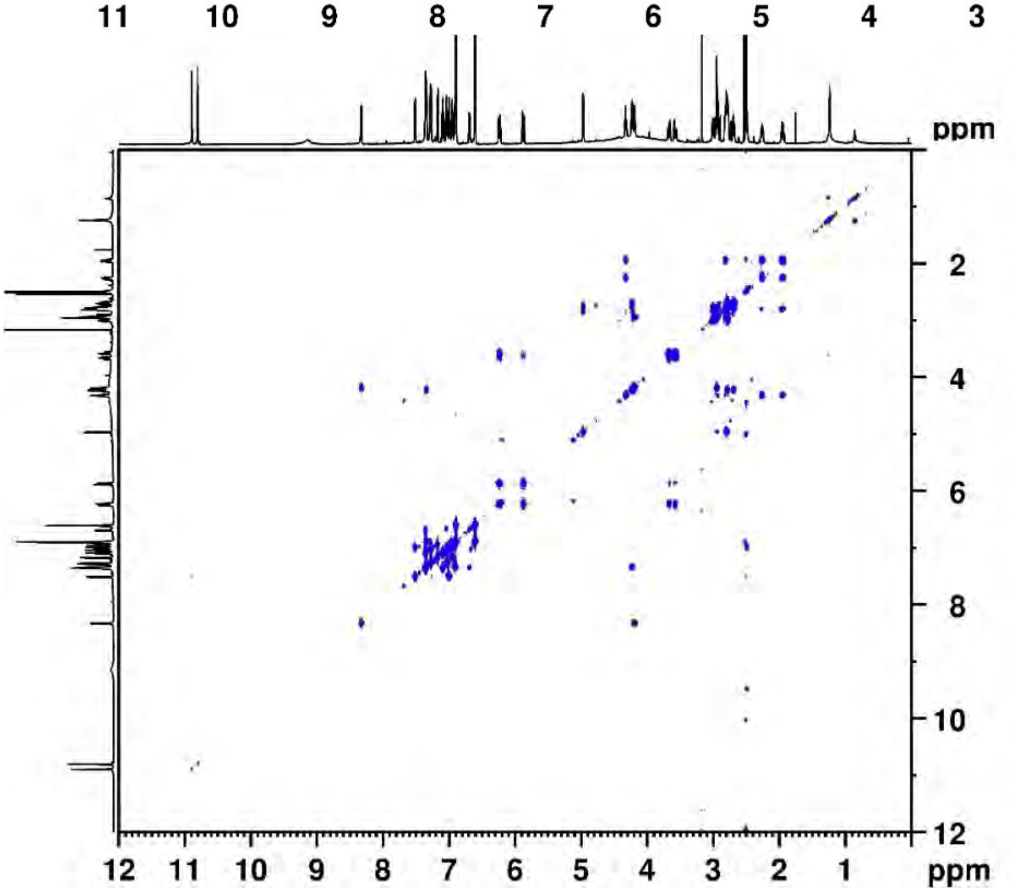
======== CHANNEL f2 ========
NUC2 13C
P3 18.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %

*Macrocyclic Product **28a***







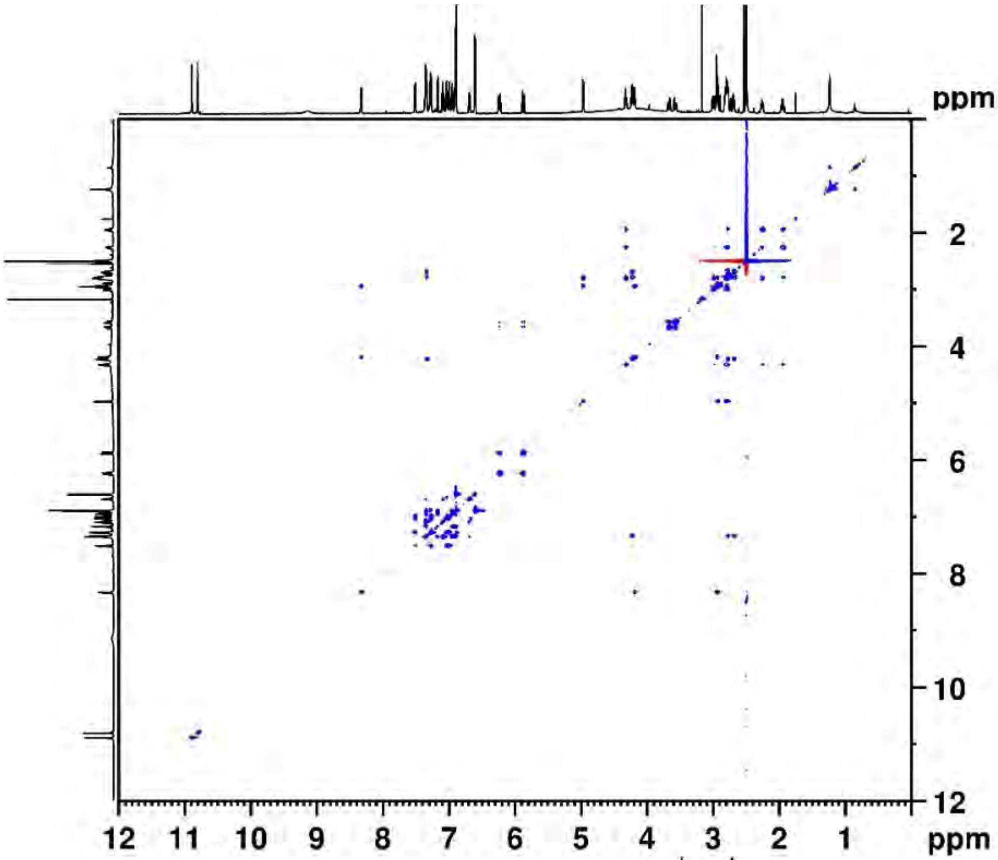
Current Data Parameters
NAME KL-5-164-1
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130522
Time 19.36
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG cosygpmfph
TD 2048
SOLVENT DMSO
NS 4
DS 16
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425408 sec
RG 90.5
DW 69.600 usec
DE 6.50 usec
TE 298.0 K
D0 0.00005689 sec
D1 2.00000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00013885 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.85 usec
P2 19.70 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1336008 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 10.00 %
GPZ2 20.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 512
SFO1 600.1336 MHz
FIDRES 14.065631 Hz
SW 12.000 ppm
FnMODE States-TPPI

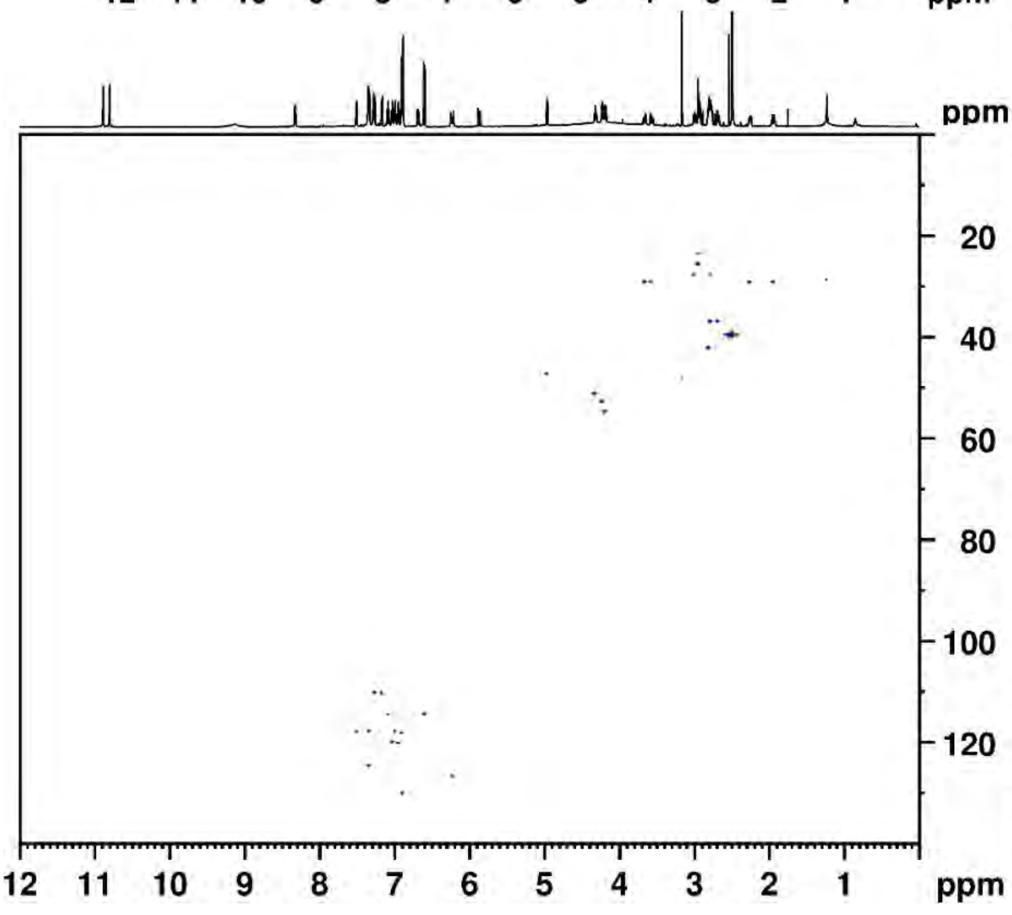


Current Data Parameters
NAME KL-5-164-1
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130522
Time 18.31
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevetgp.js
TD 4096
SOLVENT DMSO
NS 4
DS 16
SWH 7183.908 Hz
FIDRES 1.753884 Hz
AQ 0.2850816 sec
RG 90.5
DW 69.600 usec
DE 6.50 usec
TE 298.0 K
D0 0.00000300 sec
D1 1.20000005 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00013885 sec
L1 24

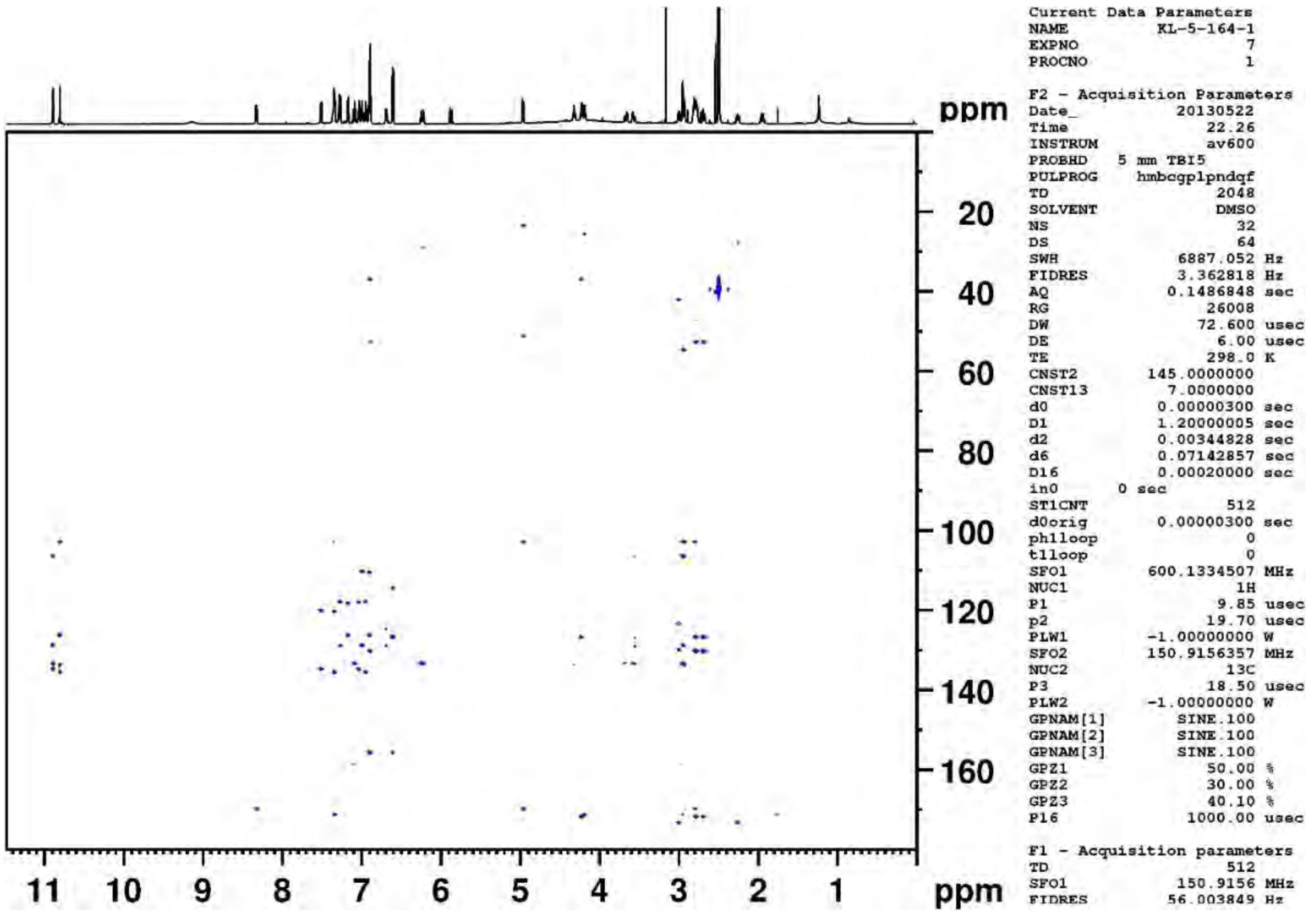
======== CHANNEL f1 ========
NUC1 1H
P1 9.85 usec
P2 19.70 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PL1 -2.00 dB
PL10 9.54 dB
PL1W 39.81071854 W
PL10W 2.79254389 W
SFO1 600.1336008 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 30.00 %
GPZ2 30.00 %



Current Data Parameters
NAME KL-5-164-1
EXPNO 6
PROCNO 1

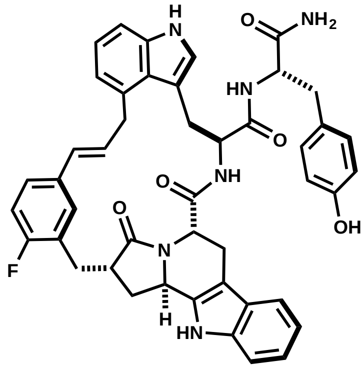
F2 - Acquisition Parameters
Date_ 20130522
Time 20.51
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425408 sec
RG 16384
DW 69.600 usec
DE 6.00 usec
TE 298.1 K
CNST2 145.0000000
d0 0.00000300 sec
D1 1.20000005 sec
d4 0.00172414 sec
d11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
DELTA 0.00127570 sec
DELTA1 0.00120632 sec
DELTA2 0.00122414 sec
in0 0 sec
ST1CNT 256
ZGOPTNS
d0orig 0.00000300 sec
ph1loop 0
t1loop 0
SFO1 600.1330006 MHz
NUC1 1H
P1 9.85 usec
p2 19.70 usec
P28 1000.00 usec
PLW1 -1.00000000 W
SFO2 150.9133722 MHz
NUC2 13C
CPDPRG[2 garp
P3 18.50 usec
p4 37.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PLW0 -1.00000000 W
PLW2 -1.00000000 W
PLW12 -1.00000000 W
SPNAM[3] Crp80.0.5.20.1

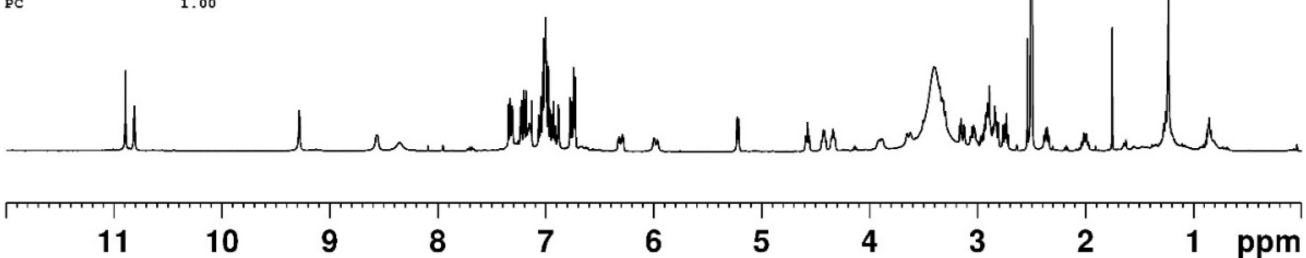


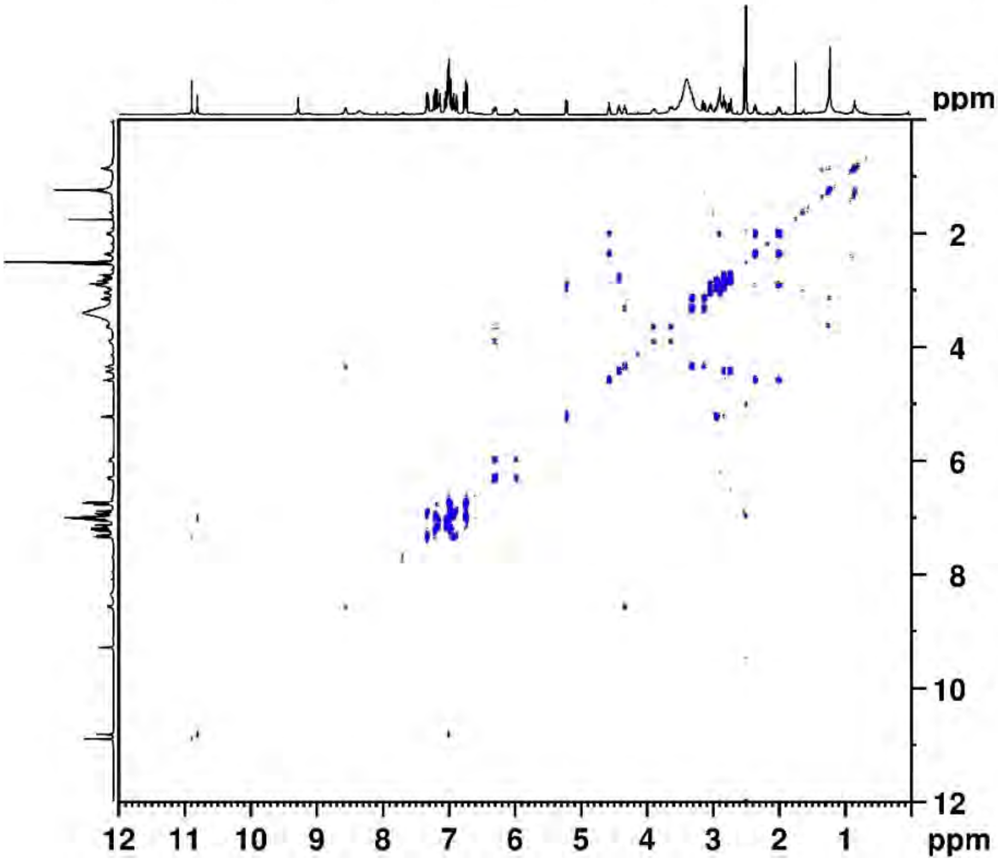
ppm
20
40
60
80
100
120
140
160
11 10 9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME KL-5-164-1
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130522
Time 22.26
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 32
DS 64
SWH 6887.052 Hz
FIDRES 3.362818 Hz
AQ 0.1486848 sec
RG 26008
DW 72.600 usec
DE 6.00 usec
TE 298.0 K
CNST2 145.0000000
CNST13 7.0000000
d0 0.00000300 sec
D1 1.20000005 sec
d2 0.00344828 sec
d6 0.07142857 sec
D16 0.00020000 sec
in0 0 sec
ST1CNT 512
d0orig 0.00000300 sec
ph1loop 0
t1loop 0
SFO1 600.1334507 MHz
NUC1 1H
P1 9.85 usec
p2 19.70 usec
PLW1 -1.00000000 W
SFO2 150.9156357 MHz
NUC2 13C
P3 18.50 usec
PLW2 -1.00000000 W
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 512
SFO1 150.9156 MHz
FIDRES 56.003849 Hz

Macrocyclic Product ***28b***

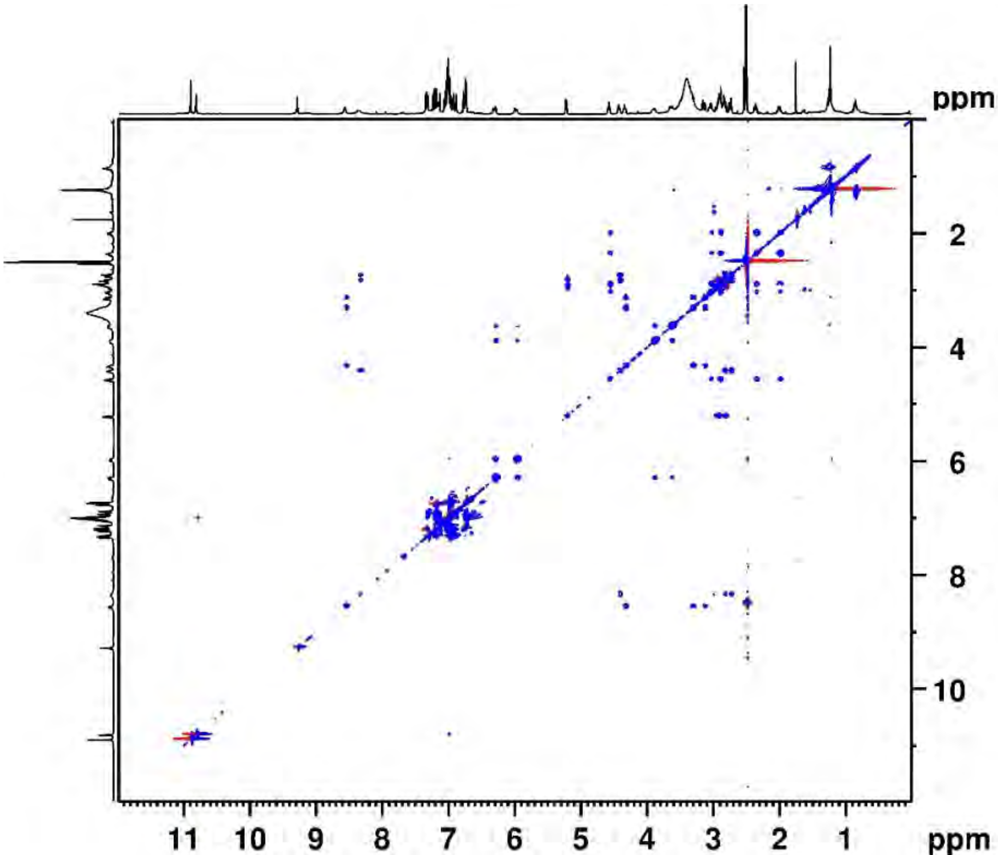
```
Current Data Parameters
NAME       KL-5-255-2 (AV500)
EXPNO                       6
PROCNO                      1

F2 - Acquisition Parameters
Date_                20140218
Time                    20.49
INSTRUM                 av500
PROBHD     5 mm DCH 13C-1
PULPROG                    zg
TD                      65536
SOLVENT                  DMSO
NS                         32
DS                          0
SWH               10000.000 Hz
FIDRES             0.152588 Hz
AQ                3.2767999 sec
RG                      19.07
DW                     50.000 usec
DE                     10.00 usec
TE                     298.0 K
D1               2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            500.1330008 MHz
NUC1                       1H
P1                       9.95 usec
PLW1             13.50000000 W

F2 - Processing parameters
SI                      65536
SF              500.1300037 MHz
WDW                        EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```








```
Current Data Parameters
NAME       KL-5-255-2 (AV500)
EXPNO                       8
PROCNO                      1

F2 - Acquisition Parameters
Date_                20140218
Time                    21.29
INSTRUM                 av500
PROBHD     5 mm DCH 13C-1
PULPROG           cosygpmfph
TD                       4096
SOLVENT                  DMSO
NS                          2
DS                          8
SWH                  6009.615 Hz
FIDRES               1.467191 Hz
AQ                  0.3407872 sec
RG                      26.58
DW                     83.200 usec
DE                      10.00 usec
TE                      298.0 K
D0                 0.00007063 sec
D1                 2.00000000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00016660 sec

======== CHANNEL f1 ========
SFO1            500.1330008 MHz
NUC1                       1H
P1                       9.95 usec
P2                      19.90 usec
PLW1             13.50000000 W

====== GRADIENT CHANNEL =====
GPNAM[1]          SMSQ10.100
GPNAM[2]          SMSQ10.100
GPZ1                    10.00 %
GPZ2                    20.00 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                        512
SFO1                  500.133 MHz
FIDRES              11.723439 Hz
SW                     12.002 ppm
FnMODE            States-TPPI

F2 - Processing parameters
SI                       2048
SF              500.1300026 MHz
WDW                      SINE
SSB                         1
```



Current Data Parameters

NAME	KL-5-255-2 (AV500)
EXPNO	7
PROCNO	1

F2 - Acquisition Parameters

Date_	20140218
Time	20.49
INSTRUM	av500
PROBHD	5 mm DCH 13C-1
PULPROG	mlevetgp.js
TD	2048
SOLVENT	DMSO
NS	2
DS	8
SWH	6009.615 Hz
FIDRES	2.934382 Hz
AQ	0.1703936 sec
RG	26.58
DW	83.200 usec
DE	10.00 usec
TE	298.0 K
D0	0.00000300 sec
D1	2.00000000 sec
D9	0.06000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
D16	0.00020000 sec
IN0	0.00016660 sec
L1	24

======== CHANNEL f1 ========

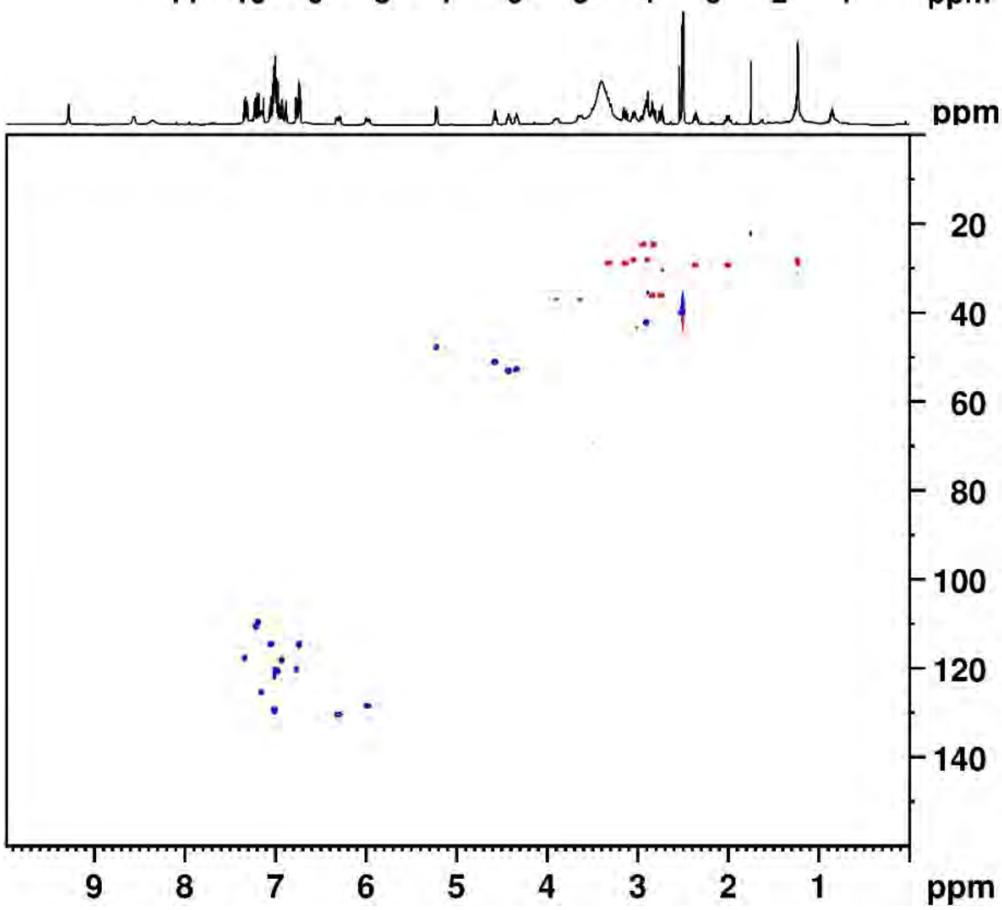
SFO1	500.1330008 MHz
NUC1	1H
P1	9.95 usec
P2	19.90 usec
P5	26.68 usec
P6	40.00 usec
P7	80.00 usec
P17	2500.00 usec
PLW1	13.50000000 W
PLW10	0.84375000 W

====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPZ1	30.00 %
GPZ2	30.00 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	512
SFO1	500.133 MHz
FIDRES	11.723439 Hz



Current Data Parameters

NAME	KL-5-255-2 (AV500)
EXPNO	9
PROCNO	1

F2 - Acquisition Parameters

Date_	20140218
Time	22.10
INSTRUM	av500
PROBHD	5 mm DCH 13C-1
PULPROG	hsqcedetgp
TD	2048
SOLVENT	DMSO
NS	16
DS	16
SWH	5000.000 Hz
FIDRES	2.441406 Hz
AQ	0.2048000 sec
RG	204.86
DW	100.000 usec
DE	10.00 usec
TE	298.0 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.50000000 sec
D4	0.00172414 sec
D11	0.03000000 sec
D13	0.00000400 sec
D16	0.00020000 sec
D21	0.00345000 sec
IN0	0.00001990 sec
ZGOPTNS	

======== CHANNEL f1 ========

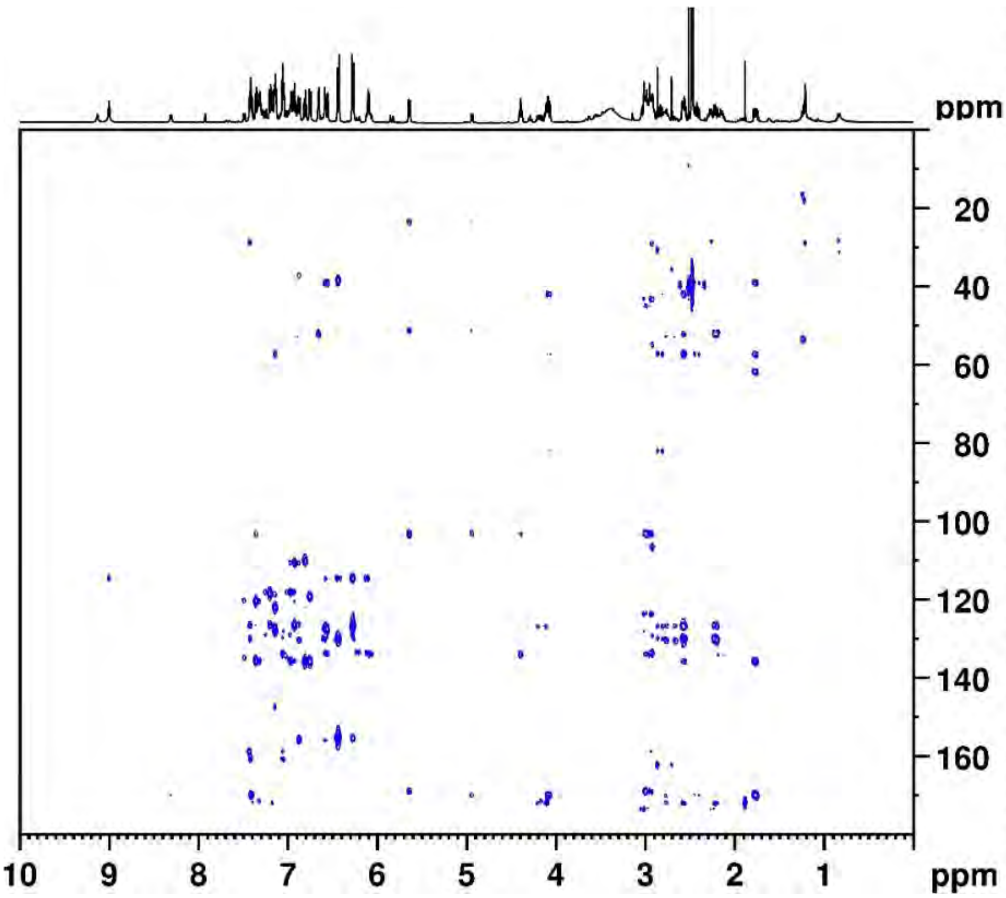
SFO1	500.1325007 MHz
NUC1	1H
P1	9.95 usec
P2	19.90 usec
P28	0 usec
PLW1	13.50000000 W

======== CHANNEL f2 ========

SFO2	125.7678496 MHz
NUC2	13C
CPDPRG[2	garp
P3	9.63 usec
P4	19.26 usec
PCPD2	70.00 usec
PLW2	23.01399994 W
PLW12	0.43557000 W

====== GRADIENT CHANNEL =====

GPNAM[1]	SMSQ10.100
GPNAM[2]	SMSQ10.100
GPZ1	80.00 %




```
Current Data Parameters
NAME            KL-5-255-5
EXPNO                    8
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140207
Time                 21.01
INSTRUM              av500
PROBHD    5 mm DCH 13C-1
PULPROG       hmbcgpl2ndqf
TD                    2048
SOLVENT               DMSO
NS                      24
DS                      16
SWH               5000.000 Hz
FIDRES            2.441406 Hz
AQ               0.2048000 sec
RG                  204.86
DW                 100.000 usec
DE                   10.00 usec
TE                   298.0 K
CNST6          120.0000000
CNST7          160.0000000
CNST13           7.0000000
D0              0.00000300 sec
D1              1.50000000 sec
D6              0.07142857 sec
D16             0.00020000 sec
IN0             0.00001990 sec

======== CHANNEL f1 ========
SFO1         500.1325007 MHz
NUC1                    1H
P1                   10.50 usec
P2                   21.00 usec
PLW1           13.50000000 W

======== CHANNEL f2 ========
SFO2         125.7703648 MHz
NUC2                   13C
P3                    9.63 usec
PLW2           23.01399994 W

====== GRADIENT CHANNEL =====
GPNAM[1]        SMSQ10.100
GPNAM[2]        SMSQ10.100
GPNAM[3]        SMSQ10.100
GPNAM[4]        SMSQ10.100
GPNAM[5]        SMSQ10.100
GPNAM[6]        SMSQ10.100
GPZ1                 50.00 %
GPZ2                 30.00 %
GPZ3                 40.10 %
GPZ4                 15.00 %
```

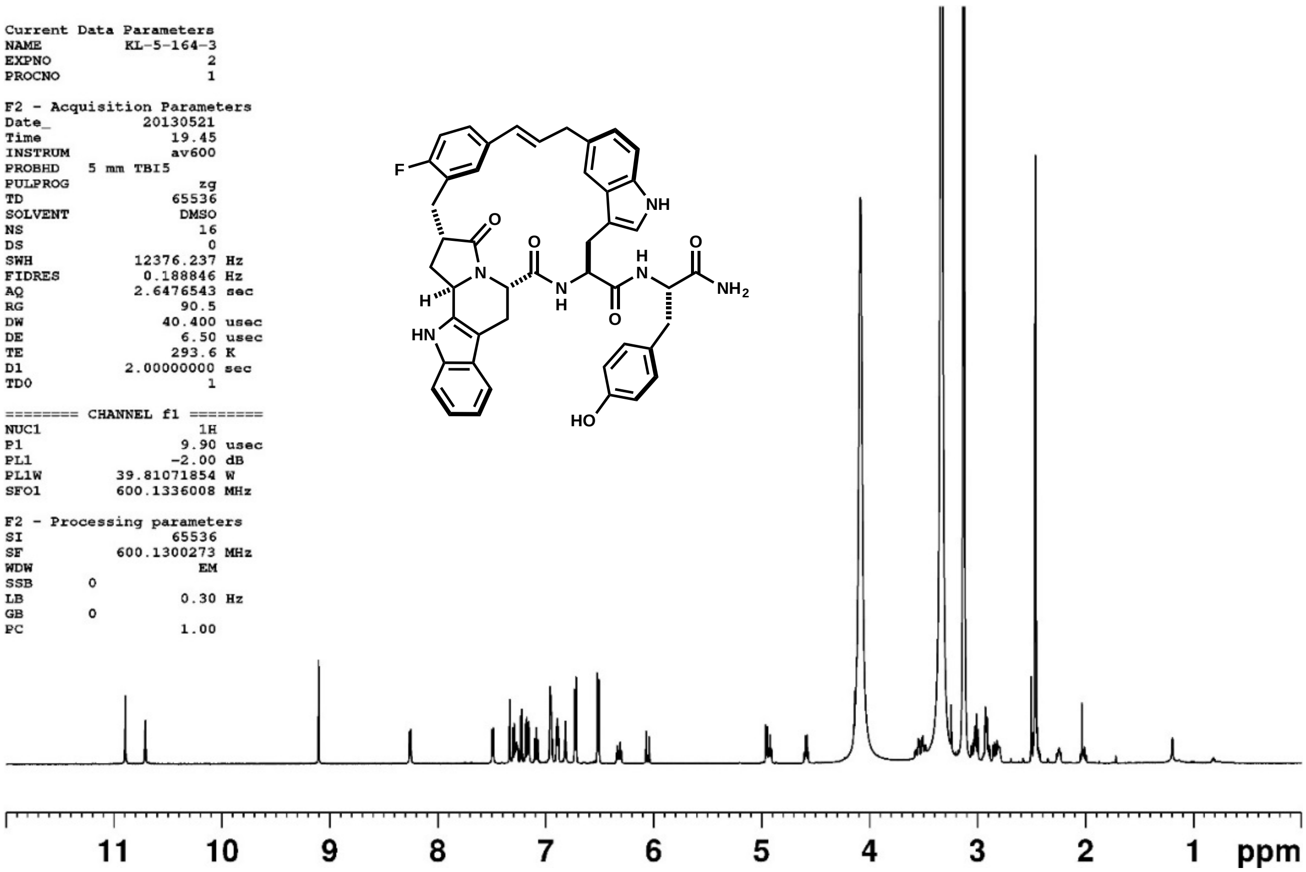
*Macrocyclic Product **28c***

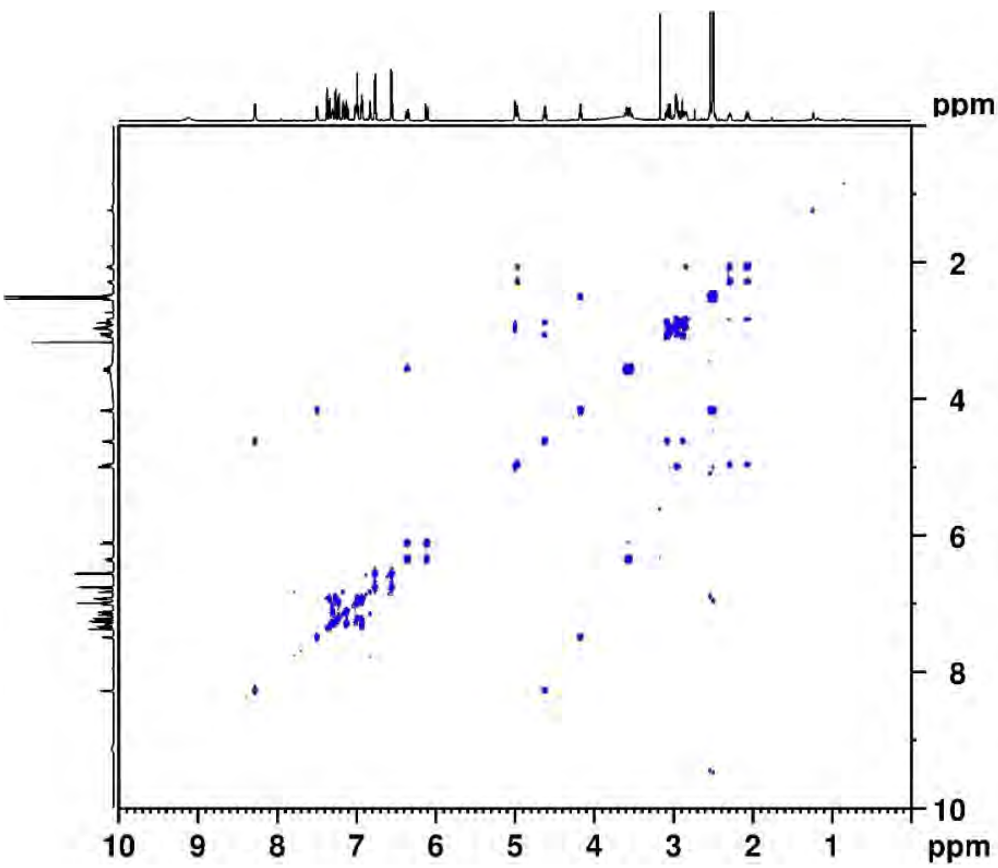
Current Data Parameters
NAME KL-5-164-3
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130521
Time 19.45
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG zg
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6476543 sec
RG 90.5
DW 40.400 usec
DE 6.50 usec
TE 293.6 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1336008 MHz

F2 - Processing parameters
SI 65536
SF 600.1300273 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





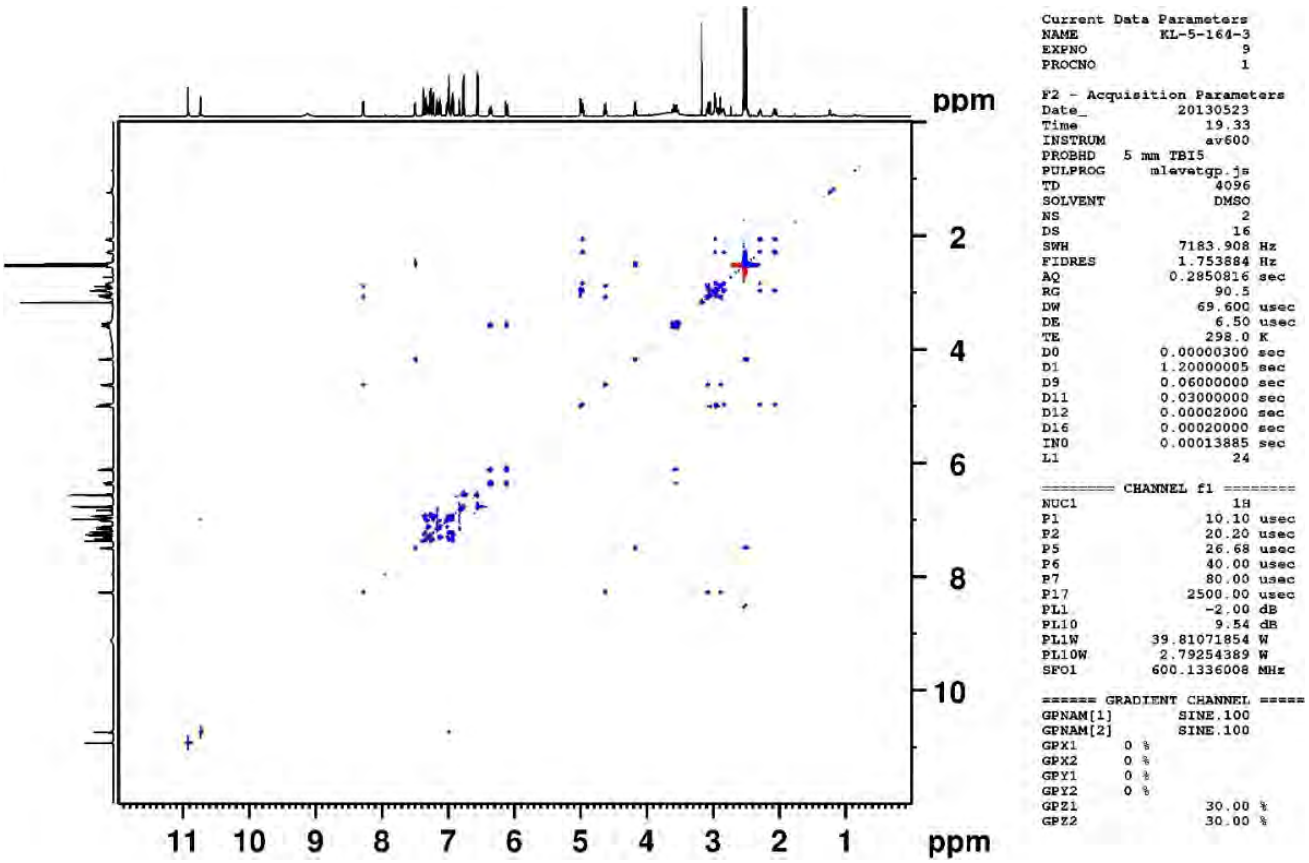
Current Data Parameters
NAME KL-5-164-3
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130523
Time 20.06
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG cosygpmfph
TD 2048
SOLVENT DMSO
NS 2
DS 16
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425408 sec
RG 90.5
DW 69.600 usec
DE 6.50 usec
TE 298.0 K
D0 0.00005657 sec
D1 2.00000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00013885 sec

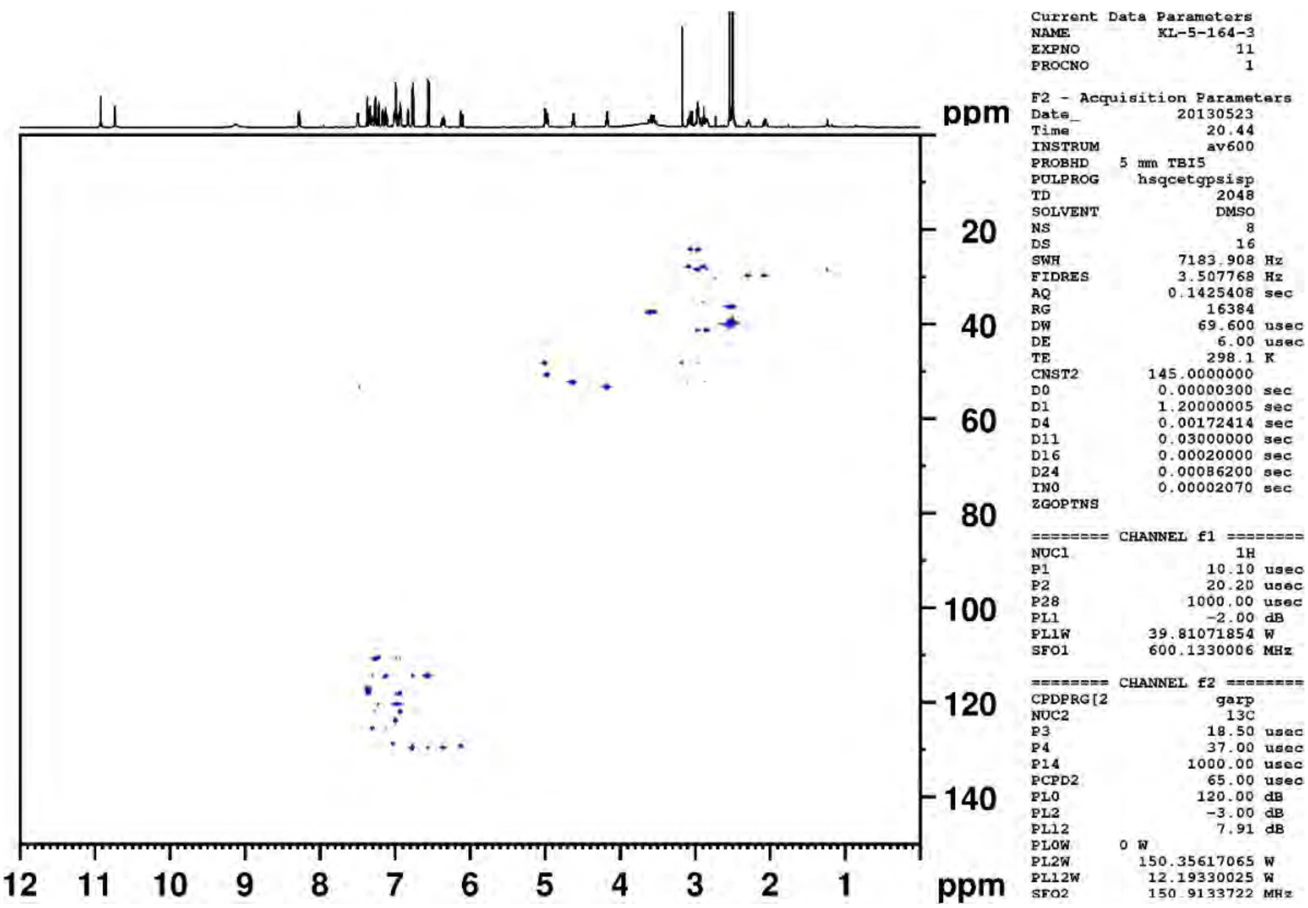
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
P2 20.20 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1336008 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 10.00 %
GPZ2 20.00 %
P16 1000.00 usec

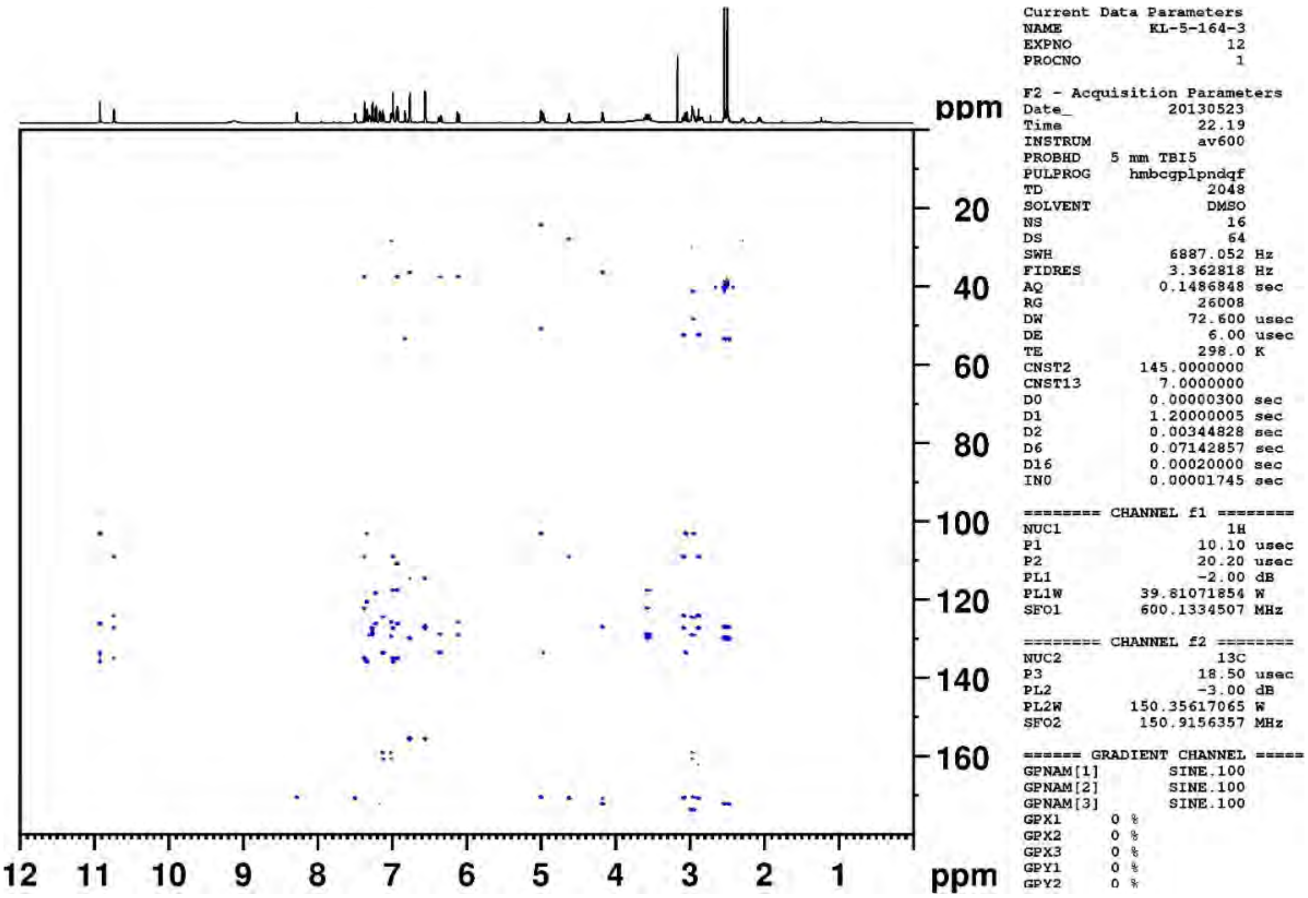
F1 - Acquisition parameters
TD 512
SFO1 600.1336 MHz
FIDRES 14.065631 Hz
SW 12.000 ppm
FnMODE States-TPPI



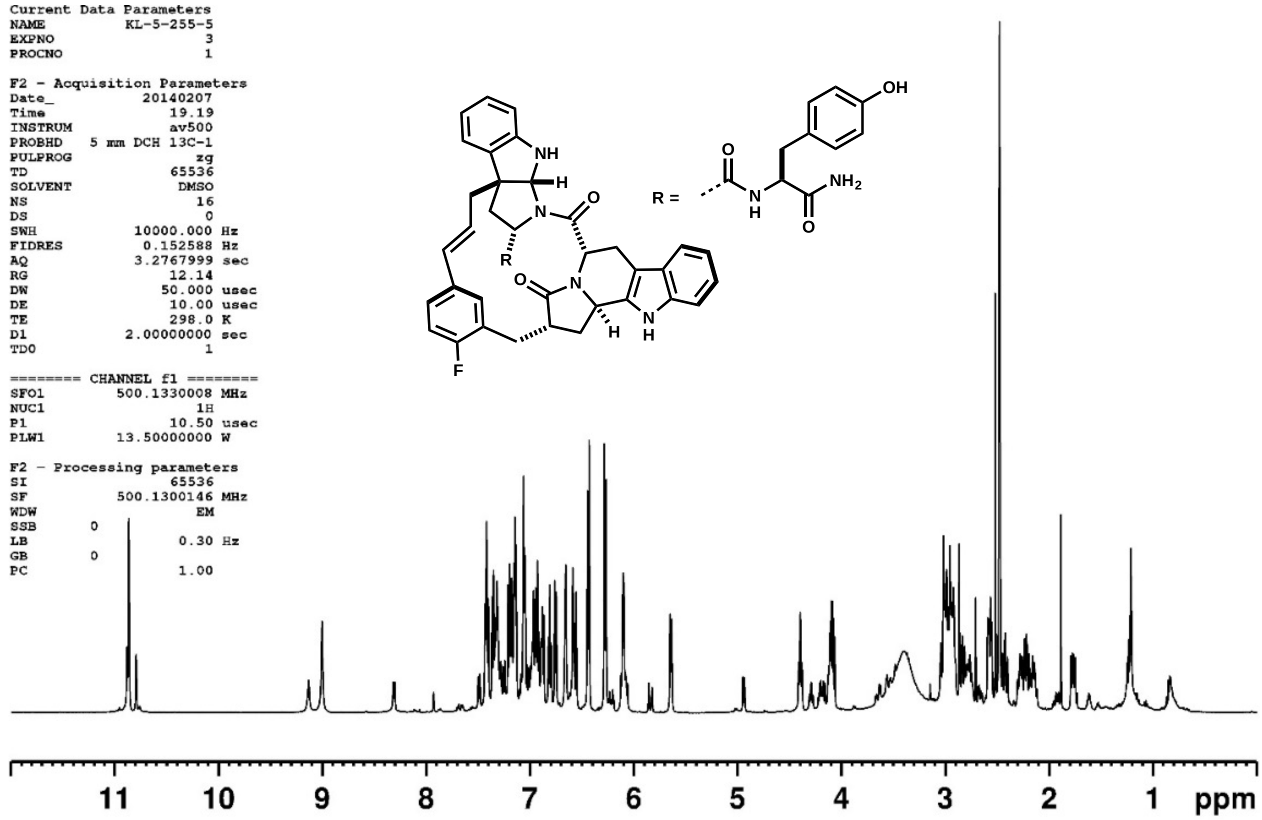
ppm
2
4
6
8
10
11 10 9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME KL-5-164-3
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130523
Time 19.33
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG mlevetgp.js
TD 4096
SOLVENT DMSO
NS 2
DS 16
SWH 7183.908 Hz
FIDRES 1.753884 Hz
AQ 0.2850816 sec
RG 90.5
DW 69.600 usec
DE 6.50 usec
TE 298.0 K
D0 0.00000300 sec
D1 1.20000005 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00013885 sec
L1 24
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
P2 20.20 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PL1 -2.00 dB
PL10 9.54 dB
PL1W 39.81071854 W
PL10W 2.79254389 W
SFO1 600.1336008 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 30.00 %
GPZ2 30.00 %

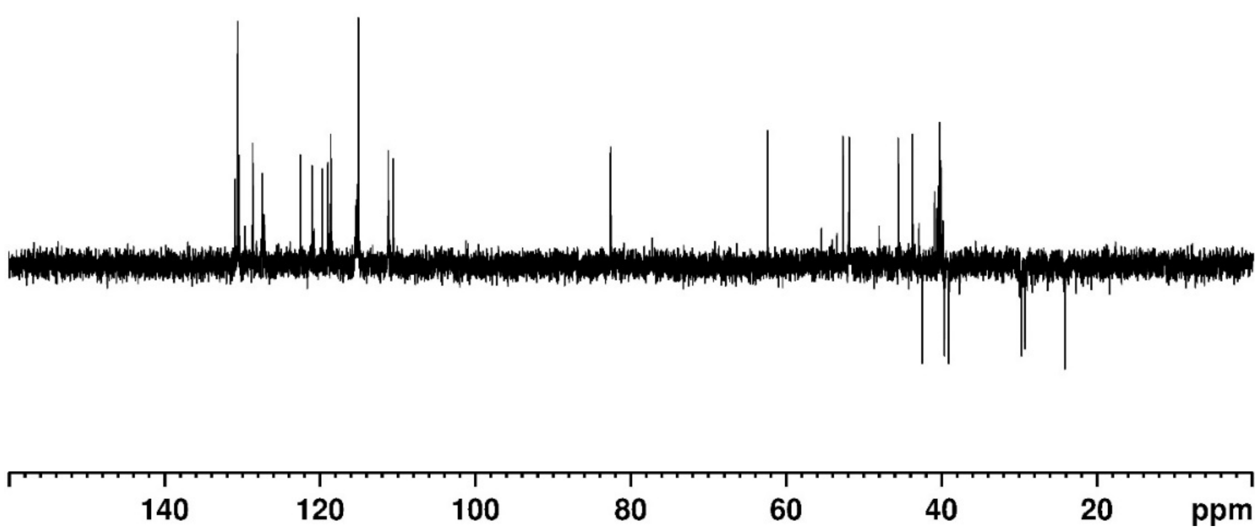


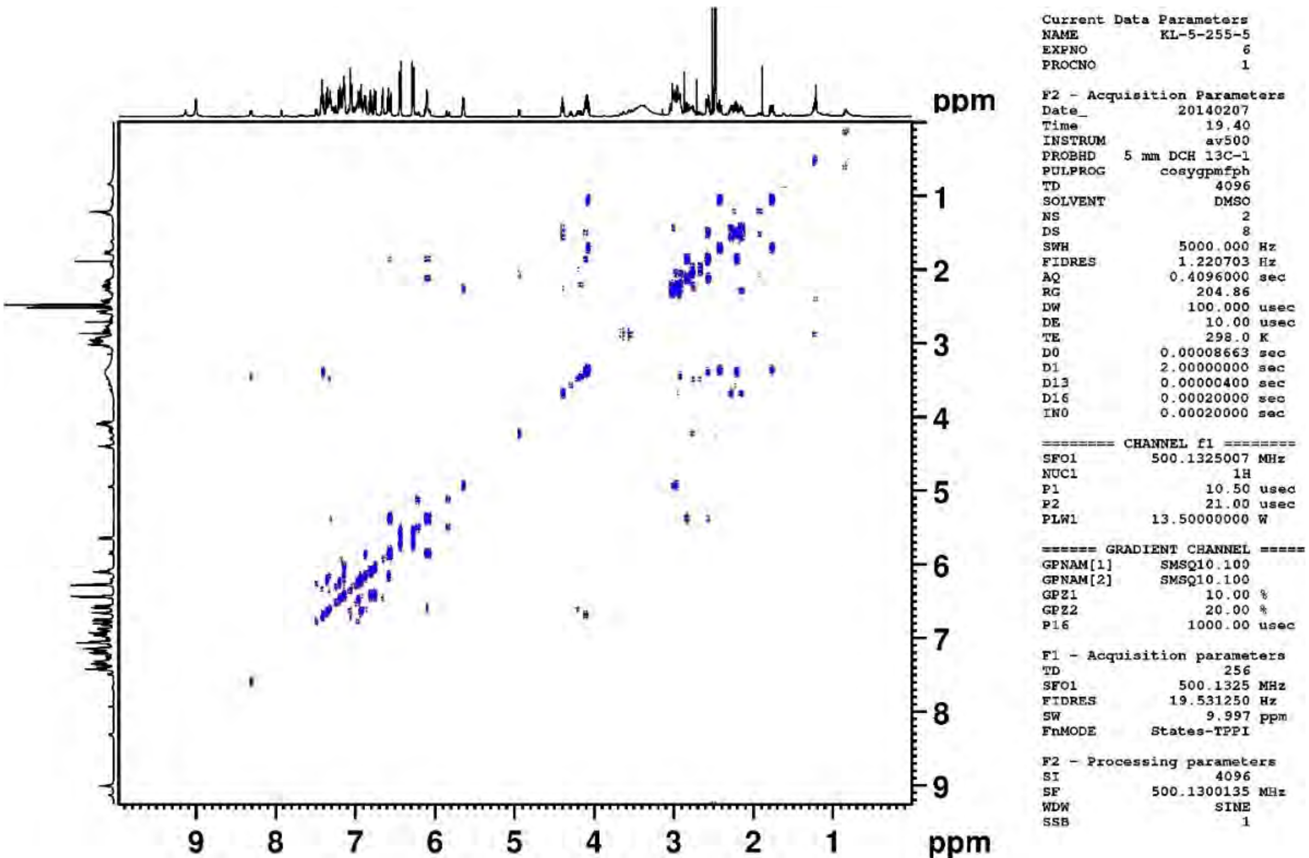
ppm
20
40
60
80
100
120
140
12 11 10 9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME KL-5-164-3
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130523
Time 20.44
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425408 sec
RG 16384
DW 69.600 usec
DE 6.00 usec
TE 298.1 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
P2 20.20 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1330006 MHz
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 18.50 usec
P4 37.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.91 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 12.19330025 W
SFO2 150.9133722 MHz

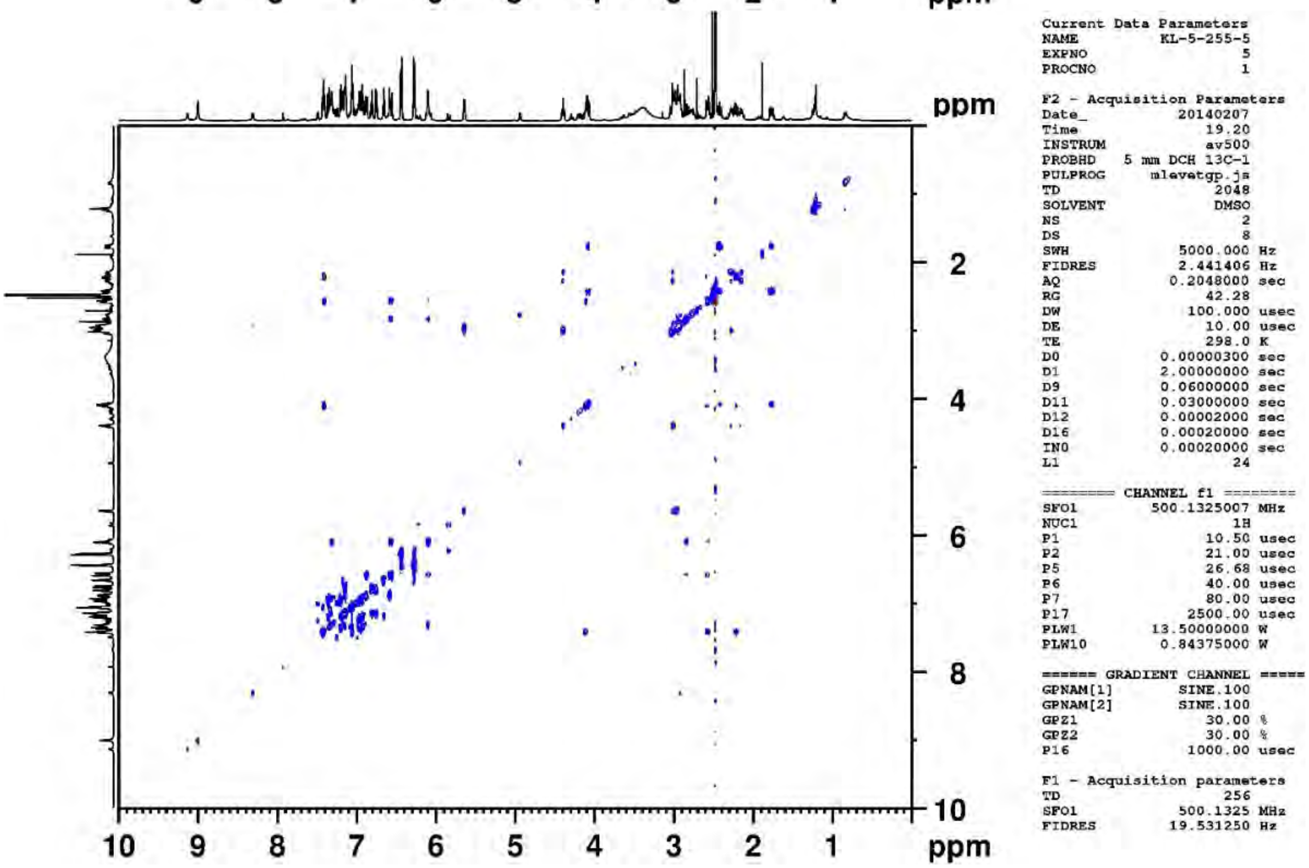

ppm
20
40
60
80
100
120
140
160
12 11 10 9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME KL-5-164-3
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130523
Time 22.19
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hmbcgplpndqf
TD 2048
SOLVENT DMSO
NS 16
DS 64
SWH 6887.052 Hz
FIDRES 3.362818 Hz
AQ 0.1486848 sec
RG 26008
DW 72.600 usec
DE 6.00 usec
TE 298.0 K
CNST2 145.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D2 0.00344828 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001745 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
P2 20.20 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1334507 MHz
======== CHANNEL f2 ========
NUC2 13C
P3 18.50 usec
PL2 -3.00 dB
PL2W 150.35617065 W
SFO2 150.9156357 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %

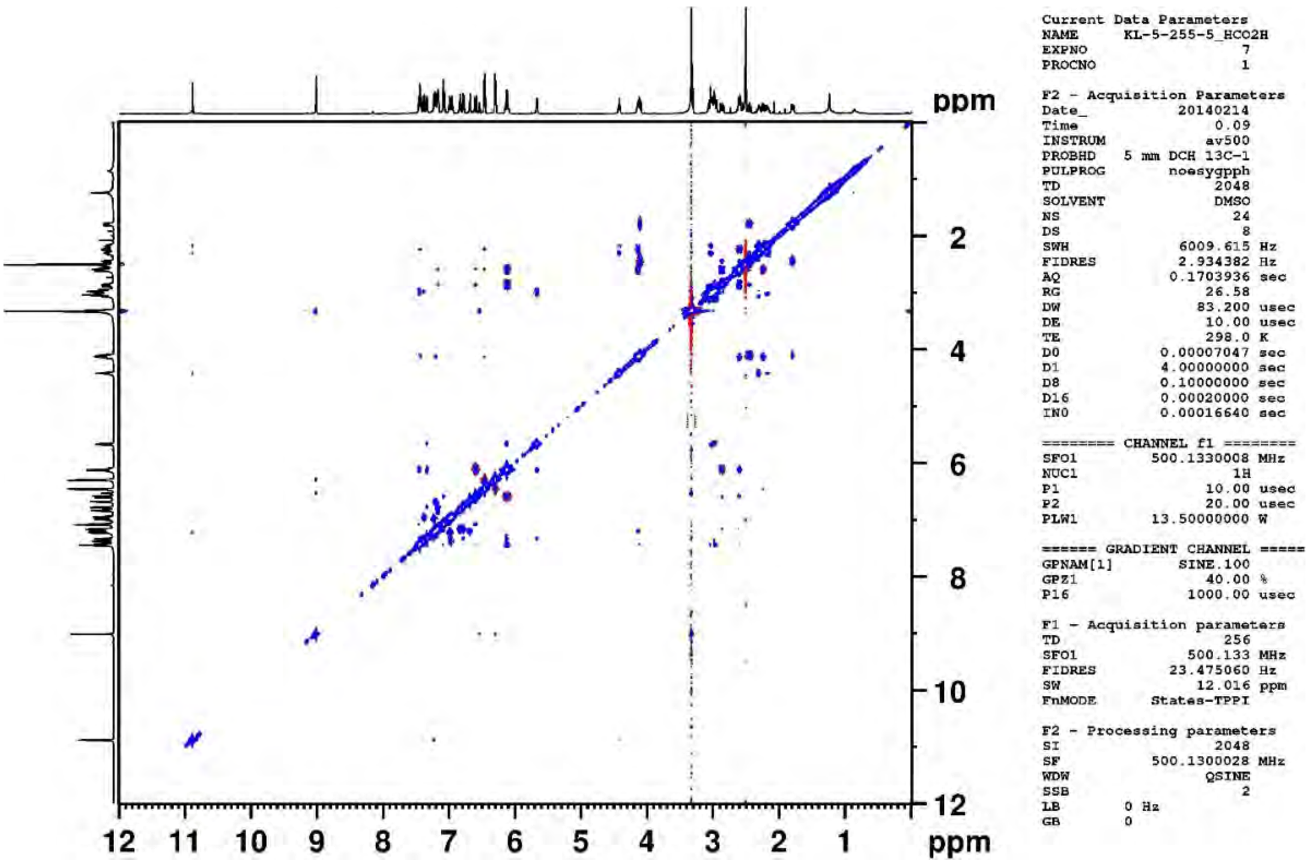
Macrocyclic Product ***28e***

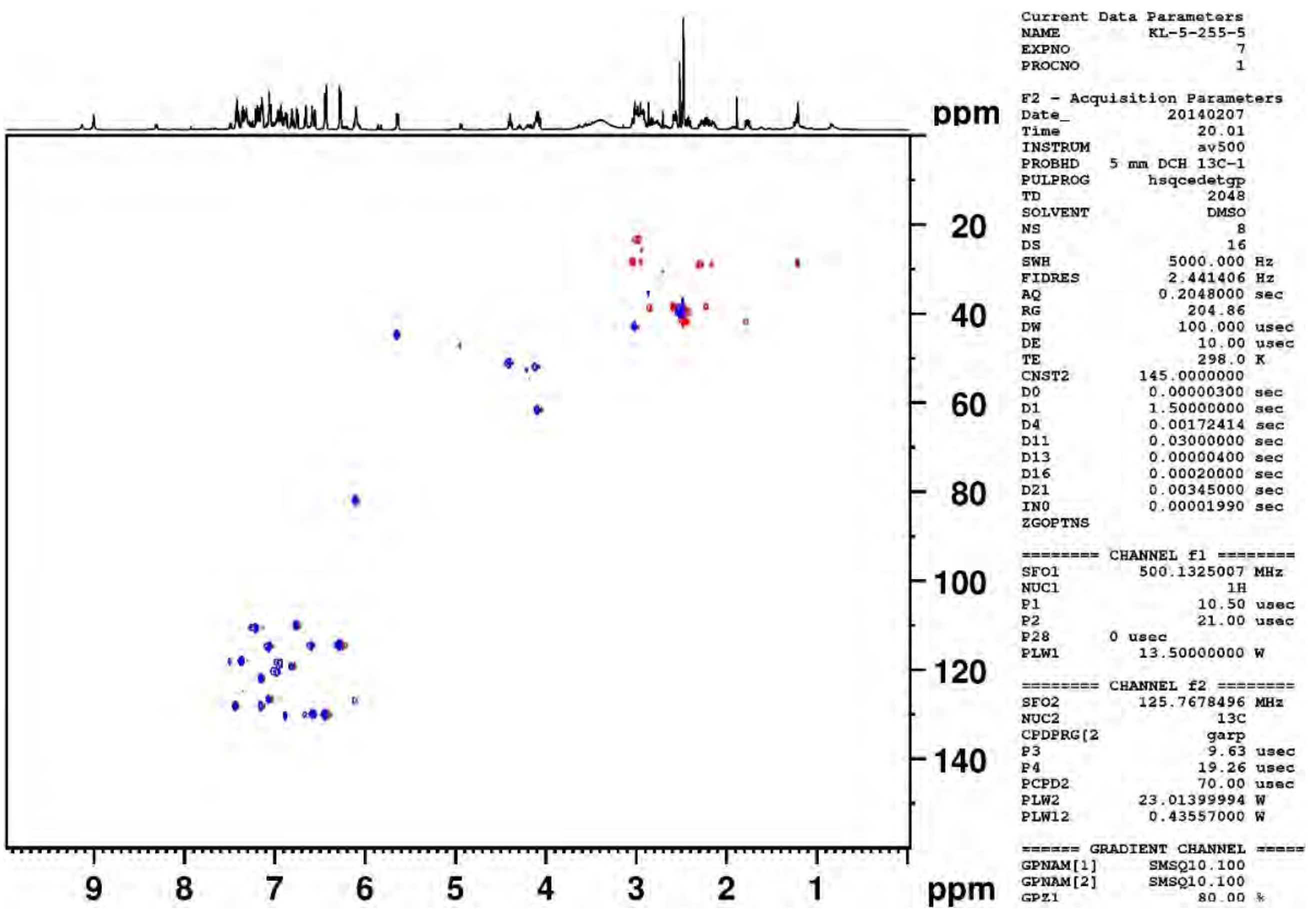


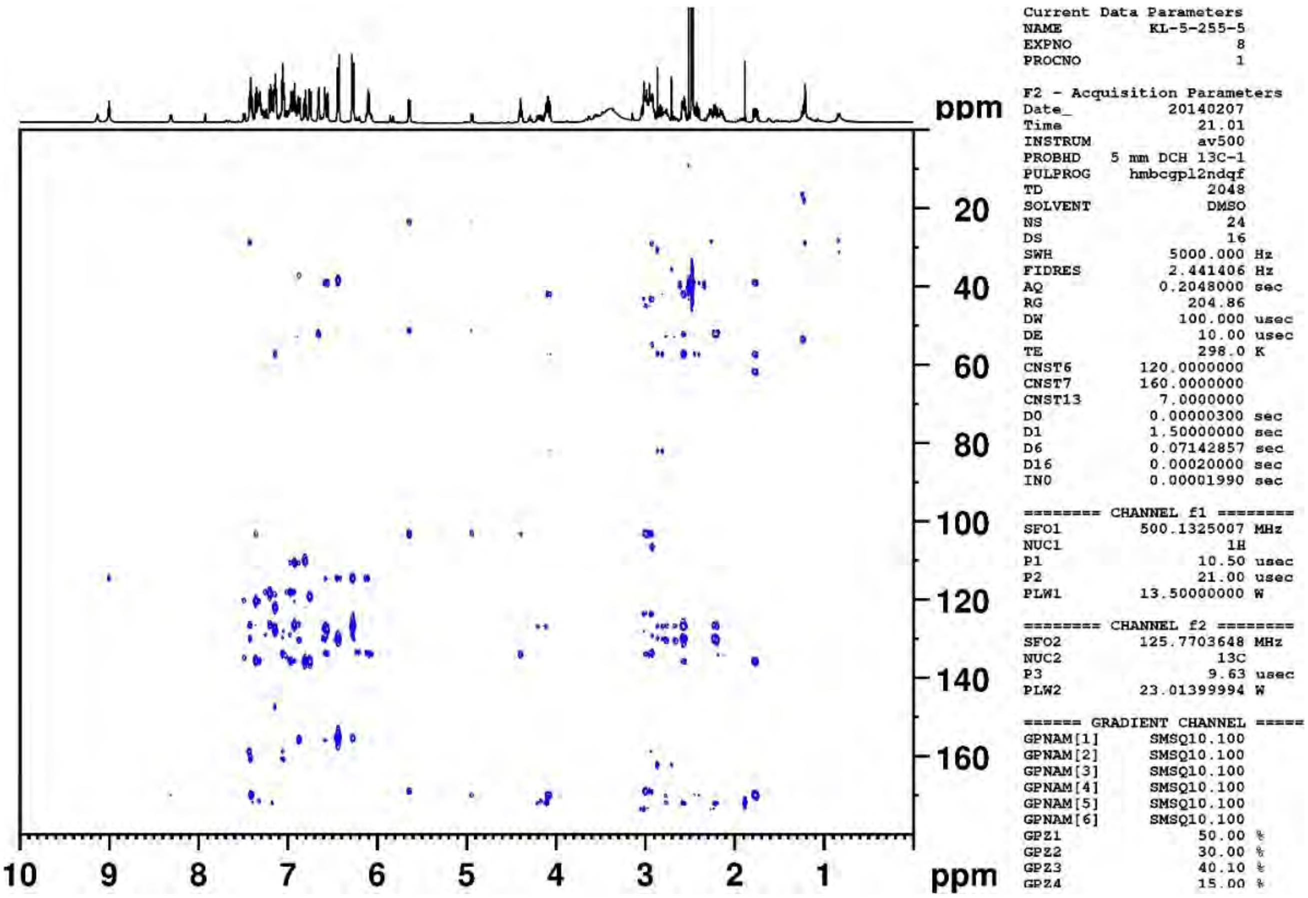



Current Data Parameters
NAME KL-5-255-5
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140207
Time 19.40
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG cosygpmfph
TD 4096
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 1.220703 Hz
AQ 0.4096000 sec
RG 204.86
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00008663 sec
D1 2.00000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00020000 sec
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.50 usec
P2 21.00 usec
PLW1 13.50000000 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 10.00 %
GPZ2 20.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.531250 Hz
SW 9.997 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 4096
SF 500.1300135 MHz
WDW SINE
SSB 1
ppm

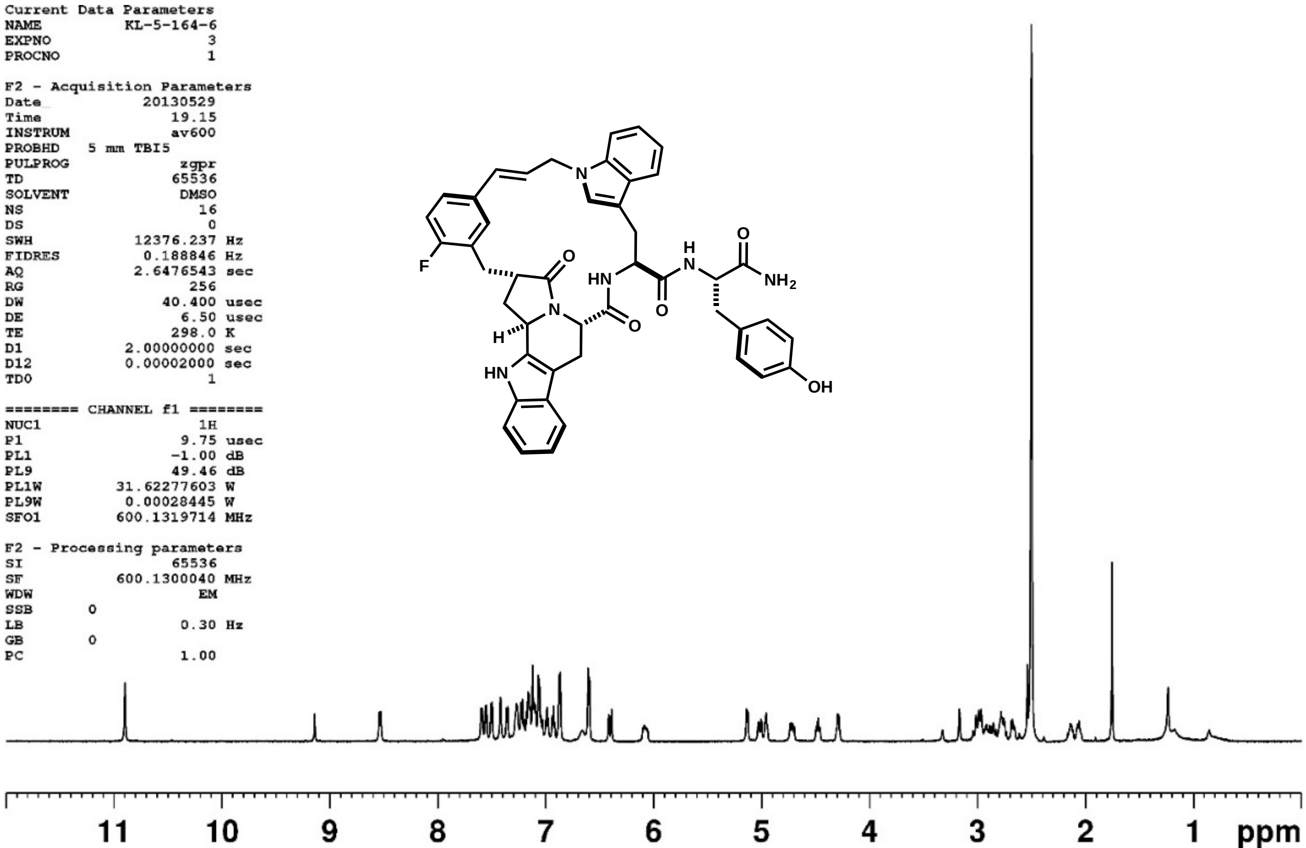

Current Data Parameters
NAME KL-5-255-5
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140207
Time 19.20
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG mlevetgp.js
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 42.28
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00020000 sec
L1 24
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.50 usec
P2 21.00 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PLW1 13.50000000 W
PLW10 0.84375000 W
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.531250 Hz
ppm

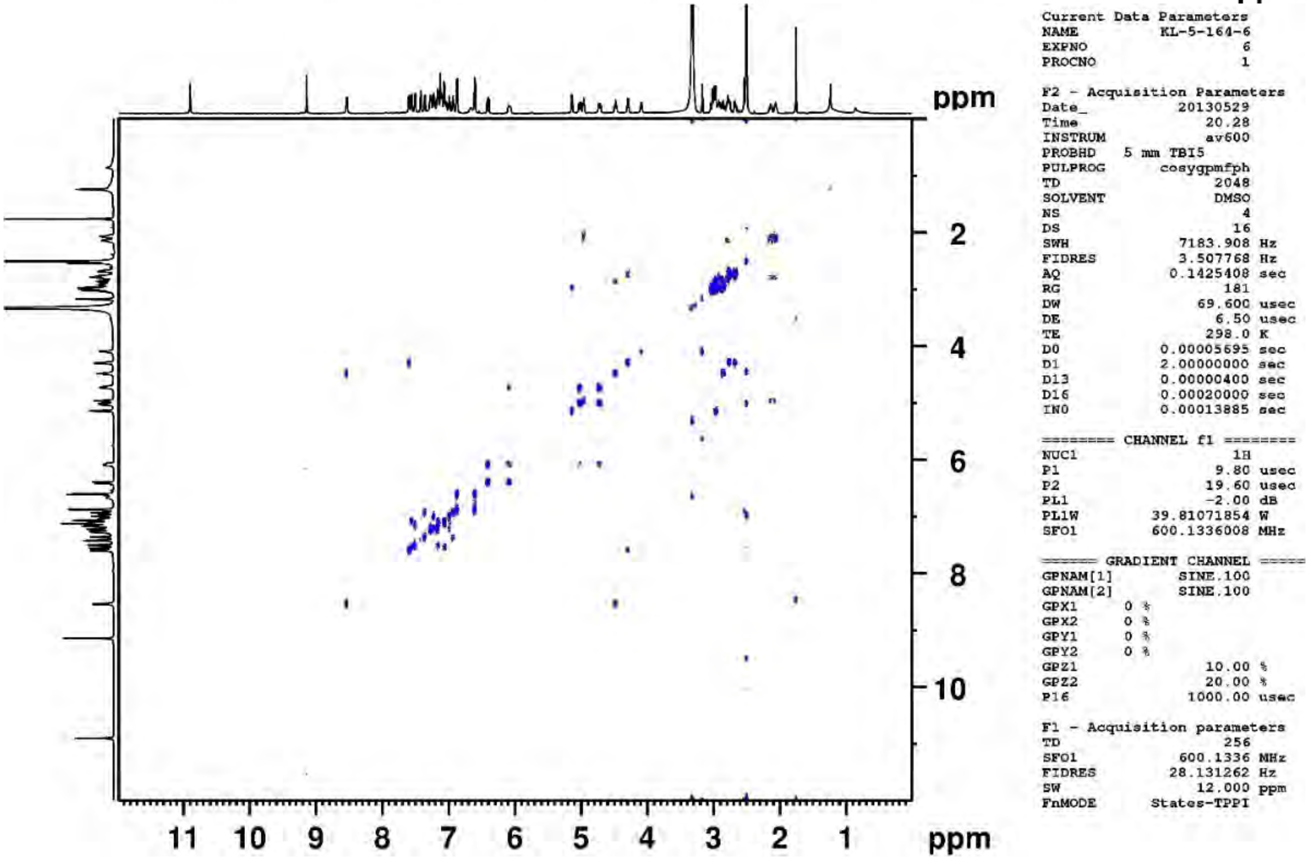

Current Data Parameters
NAME KL-5-255-5_HCO2H
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140214
Time 0.09
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG noesygpph
TD 2048
SOLVENT DMSO
NS 24
DS 8
SWH 6009.615 Hz
FIDRES 2.934382 Hz
AQ 0.1703936 sec
RG 26.58
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
D0 0.00007047 sec
D1 4.00000000 sec
D8 0.10000000 sec
D16 0.00020000 sec
IN0 0.00016640 sec
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 13.50000000 W
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPZ1 40.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.133 MHz
FIDRES 23.475060 Hz
SW 12.016 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 2048
SF 500.1300028 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
ppm
2
4
6
8
10
12
12 11 10 9 8 7 6 5 4 3 2 1
ppm

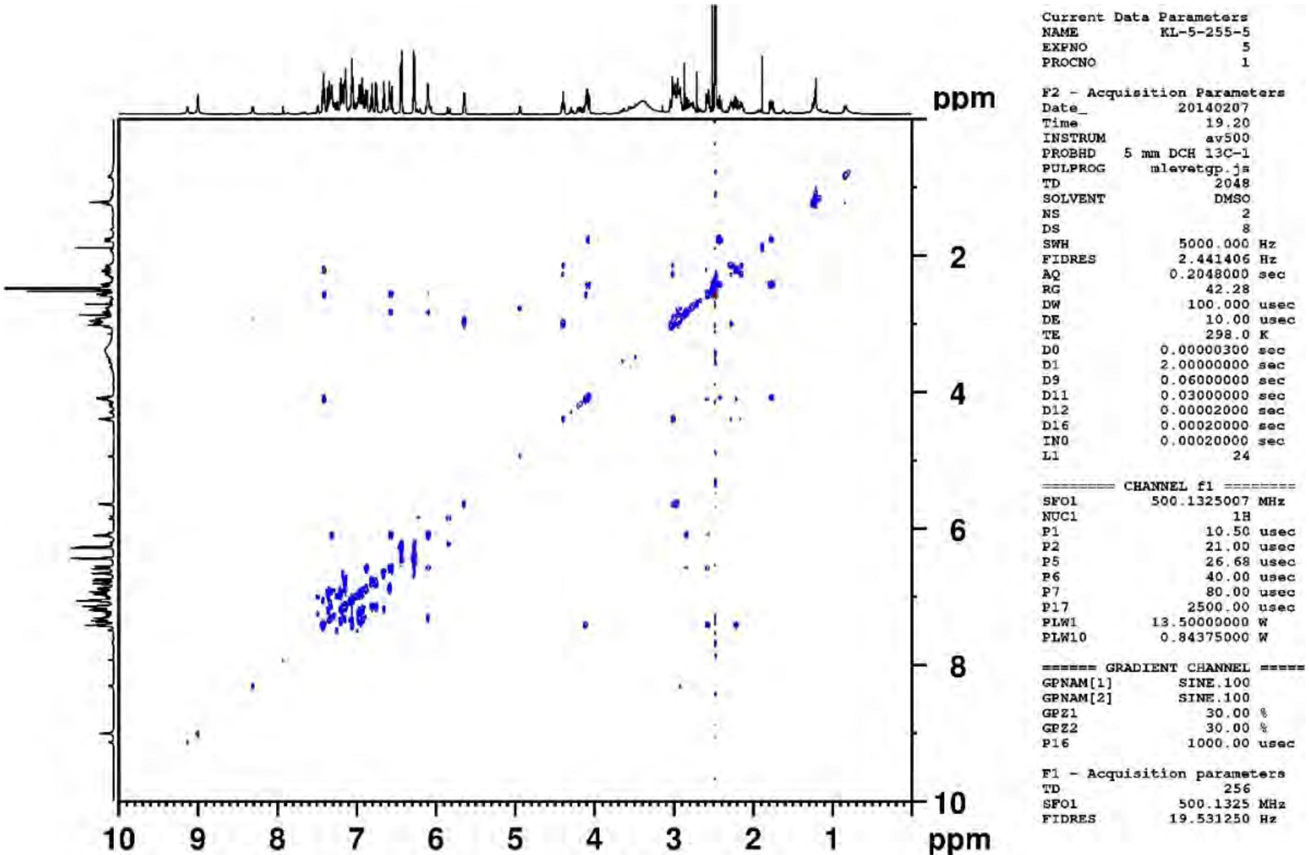

Current Data Parameters
NAME KL-5-255-5
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140207
Time 20.01
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hsqcedetgp
TD 2048
SOLVENT DMSO
NS 8
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 204.86
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
IN0 0.00001990 sec
ZGOPTNS
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.50 usec
P2 21.00 usec
P28 0 usec
PLW1 13.50000000 W
======== CHANNEL f2 ========
SFO2 125.7678496 MHz
NUC2 13C
CPDPRG[2 garp
P3 9.63 usec
P4 19.26 usec
PCPD2 70.00 usec
PLW2 23.01399994 W
PLW12 0.43557000 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %
ppm
20
40
60
80
100
120
140
9 8 7 6 5 4 3 2 1
ppm

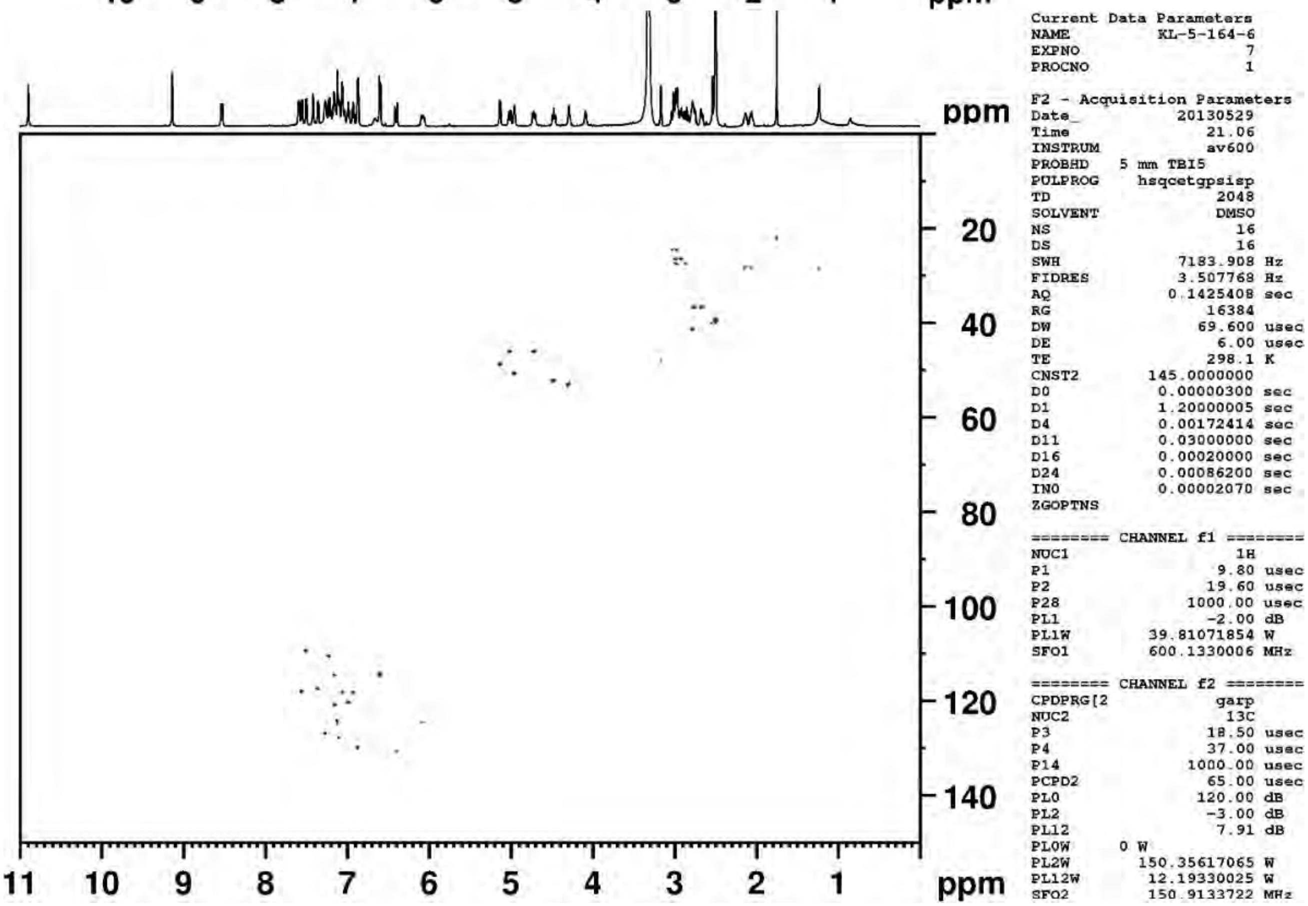

Current Data Parameters
NAME KL-5-255-5
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140207
Time 21.01
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 24
DS 16
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 204.86
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 7.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D6 0.07142857 sec
D16 0.00020000 sec
IN0 0.00001990 sec
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.50 usec
P2 21.00 usec
PLW1 13.50000000 W
======== CHANNEL f2 ========
SFO2 125.7703648 MHz
NUC2 13C
P3 9.63 usec
PLW2 23.01399994 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPNAM[4] SMSQ10.100
GPNAM[5] SMSQ10.100
GPNAM[6] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %
ppm
20
40
60
80
100
120
140
160
10 9 8 7 6 5 4 3 2 1 ppm

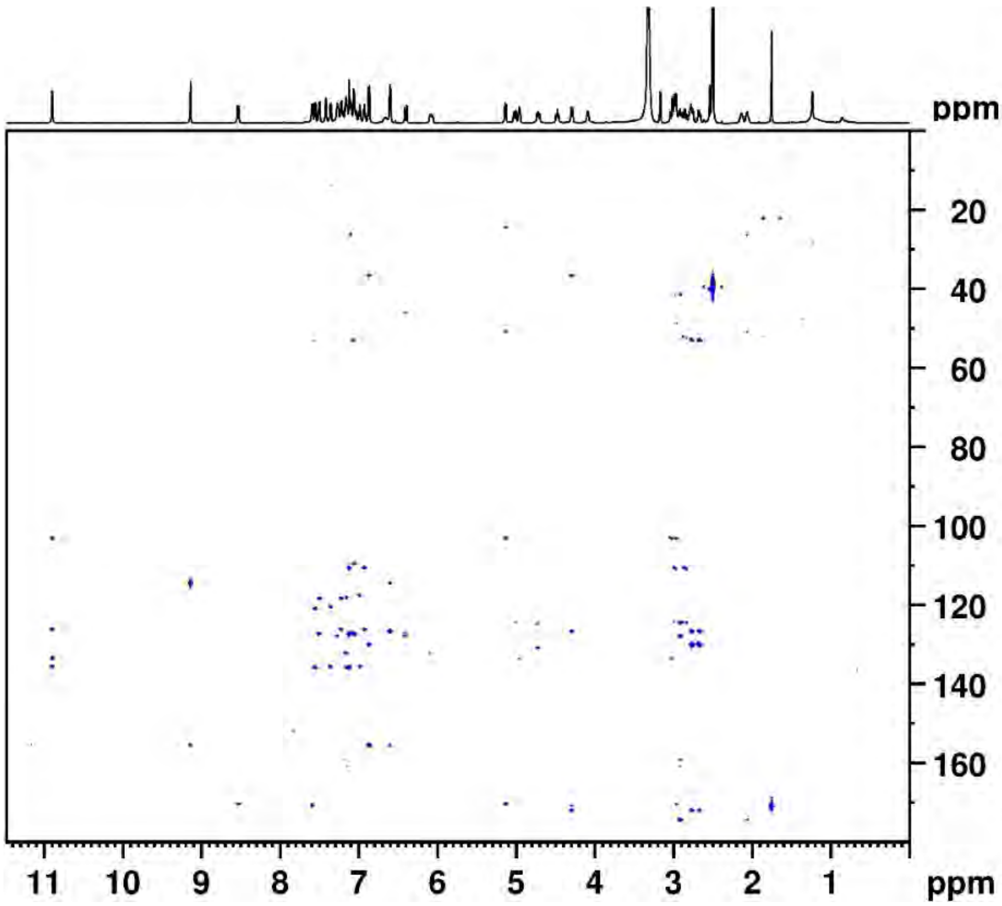
Macrocyclic Product ***28f***






Current Data Parameters
NAME KL-5-255-5
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140207
Time 19.20
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG mlevetgp.js
TD 2048
SOLVENT DMSO
NS 2
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 42.28
DW 100.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D9 0.06000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00020000 sec
L1 24
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 10.50 usec
P2 21.00 usec
P5 26.68 usec
P6 40.00 usec
P7 80.00 usec
P17 2500.00 usec
PLW1 13.50000000 W
PLW10 0.84375000 W
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.1325 MHz
FIDRES 19.531250 Hz
ppm
2
4
6
8
10
10 9 8 7 6 5 4 3 2 1 ppm


Current Data Parameters
NAME KL-5-164-6
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130529
Time 21.06
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG hsqcetgpsisp
TD 2048
SOLVENT DMSO
NS 16
DS 16
SWH 7183.908 Hz
FIDRES 3.507768 Hz
AQ 0.1425408 sec
RG 16384
DW 69.600 usec
DE 6.00 usec
TE 298.1 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.20000005 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
IN0 0.00002070 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
P2 19.60 usec
P28 1000.00 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1330006 MHz
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 18.50 usec
P4 37.00 usec
P14 1000.00 usec
PCPD2 65.00 usec
PL0 120.00 dB
PL2 -3.00 dB
PL12 7.91 dB
PL0W 0 W
PL2W 150.35617065 W
PL12W 12.19330025 W
SFO2 150.9133722 MHz
ppm
20
40
60
80
100
120
140
11 10 9 8 7 6 5 4 3 2 1 ppm




```
Current Data Parameters
NAME          KL-5-164-6
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130530
Time                0.15
INSTRUM            av600
PROBHD    5 mm TBI5
PULPROG     hmbcgplpndqf
TD                  2048
SOLVENT             DMSO
NS                    48
DS                    64
SWH             6887.052 Hz
FIDRES          3.362818 Hz
AQ             0.1486848 sec
RG                 26008
DW                72.600 usec
DE                  6.00 usec
TE                 298.0 K
CNST2        145.0000000
CNST13         7.0000000
D0            0.00000300 sec
D1            1.20000005 sec
D2            0.00344828 sec
D6            0.07142857 sec
D16           0.00020000 sec
IN0           0.00001745 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.80 usec
P2                 19.60 usec
PL1                -2.00 dB
PL1W         39.81071854 W
SFO1         600.1334507 MHz

======== CHANNEL f2 ========
NUC2                 13C
P3                 18.50 usec
PL2                -3.00 dB
PL2W        150.35617065 W
SFO2         150.9156357 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPNAM[3]        SINE.100
GPX1      0 %
GPX2      0 %
GPX3      0 %
GPY1      0 %
GPY2      0 %
```