

The Halicylindramides, Farnesoid X Receptor  
Antagonizing Depsipeptides from a *Petrosia* sp.  
Marine Sponge Collected in Korea

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**Table S1.** NMR spectroscopic data for halicyclindramide F (**1**) in DMSO-*d*<sub>6</sub> at 600 MHz (<sup>1</sup>H) and 150 MHz (<sup>13</sup>C)

position	$\delta_c$ , mult. <sup>a</sup>	$\delta_{H1}$ ( <i>J</i> in Hz)	COSY	TOCSY	NOESY	HMBC
CHO	160.5, C	7.89, s	Ala-NH	Ala-NH, Ala $\alpha$ , Ala $\beta$	Ala-NH	Ala $\alpha$
Ala $\alpha$	46.4, CH	4.33, q (7.3)	NH, $\beta$	NH, $\beta$ , CHO	Phe1-NH	$\beta$ , C=O, CHO
$\beta$	19.4, CH <sub>3</sub>	0.89, d (7.2)	$\alpha$	NH, $\alpha$		$\alpha$ , C=O
NH		8.14, brd (7.8)	$\alpha$ , CHO	CHO, $\alpha$ , $\beta$	CHO	CHO
CO	171.4, C					
Phe1 $\alpha$	51.7, CH	4.72, m	NH, $\beta$ 2	NH, $\beta$ 2		
$\beta$ 1	34.7, CH <sub>2</sub>	2.74, d (13.8)	$\beta$ 2	$\beta$ 2	Pro $\delta$	C=O
$\beta$ 2		3.01, m	$\alpha$ , $\beta$ 1	NH, $\alpha$ , $\beta$ 1		1, 2, 6, C=O
C1	137.7, C					
C2/C6	129.3, CH	7.25, m	3, 5	3, 4, 5		$\beta$ , 1, 3, 4, 5
C3/C5	128.0, CH	7.22, m	2, 4, 6	2, 4, 6		2, 4, 6
C4	126.7, CH	7.16, m	3, 5	2, 3, 5, 6		2, 3, 5, 6
NH		8.45, brd (8.4)	$\alpha$	$\alpha$ , $\beta$ 1, $\beta$ 2	Ala $\alpha$	
CO	169.4, C					
Pro $\alpha$	59.8, CH	4.46, m	$\beta$ 1, $\beta$ 2	$\beta$ 1, $\beta$ 2, $\gamma$	Val-NH	
$\beta$ 1	29.8, CH <sub>2</sub>	1.88, m	$\alpha$ , $\gamma$	$\alpha$ , $\beta$ 2, $\gamma$ , $\delta$		
$\beta$ 2		2.07 m	$\alpha$ , $\gamma$	$\alpha$ , $\beta$ 1, $\gamma$ , $\delta$		C=O
$\gamma$	24.4, CH <sub>2</sub>	1.89, m	$\beta$ 1, $\beta$ 2, $\delta$	$\alpha$ , $\beta$ 1, $\beta$ 2, $\delta$		
$\delta$	47.0, CH <sub>2</sub>	3.67, m	$\gamma$	$\beta$ , $\gamma$	Phe1 $\beta$ 1	
CO	171.1, C					
Val $\alpha$	57.2, CH	4.38, m <sup>b</sup>	NH, $\beta$	NH, $\beta$ , $\gamma$ , $\gamma'$	<i>t</i> -Leu-NH	$\beta$ , $\gamma$ , $\gamma'$ , C=O
$\beta$	31.2, CH	2.07, m	$\alpha$ , $\gamma$ , $\gamma'$	NH, $\alpha$ , $\gamma$ , $\gamma'$		$\alpha$ , $\gamma$ , $\gamma'$
$\gamma$	17.3, CH <sub>3</sub>	0.78, d (6.7)	$\beta$	$\alpha$ , $\beta$ , $\gamma'$		$\alpha$ , $\beta$ , $\gamma'$
$\gamma'$	18.7, CH <sub>3</sub>	0.83, d (7.2)	$\beta$	$\alpha$ , $\beta$ , $\gamma$		$\alpha$ , $\beta$ , $\gamma$
NH		7.81, m	$\alpha$	$\alpha$ , $\beta$	Pro $\alpha$	Pro C=O
CO	171.0, C					
<i>t</i> -Leu $\alpha$	60.4, CH	4.18, d (8.7)	NH	NH	Trp-NH	$\beta$ , $\gamma$ , C=O
$\beta$	33.7, C					
$\gamma$	26.5, CH <sub>3</sub>	0.70, s				$\alpha$ , $\beta$
NH		7.76, m	$\alpha$	$\alpha$	Val $\alpha$	
CO	169.9, C					
Trp $\alpha$	54.0, CH	4.57, m	NH, $\beta$ 1, $\beta$ 2	$\beta$ 1, $\beta$ 2, NH	Arg-NH	$\beta$ , 3, C=O
$\beta$ 1	26.8, CH <sub>2</sub>	3.16, m	$\alpha$ , $\beta$ 2	$\alpha$ , $\beta$ 2, NH		2, 3
$\beta$ 2		2.96, m	$\alpha$ , $\beta$ 1	$\alpha$ , $\beta$ 1, NH		2, 3
C2	124.1, CH	7.15, s	1-NH	1-NH		3, 3a, 7a
C3	109.8, C					
C3a	136.6, C					
C7a	119.5, C					
C4	118.1, CH	7.60, d (7.9)	5	5, 6, 7		2, 3, 3a, 6
C5	120.8, CH	6.96, dd (7.5, 7.1)	4, 6	4, 6, 7		3a, 6, 7
C6	111.2, CH	7.03, dd (7.4, 7.6)	5, 7	4, 5, 7		5, 7a
C7	127.2, CH	7.31, d (8.0)	6	4, 5, 6		5, 7a
1-NH		10.6, brs	2	2		
NH		8.29, brd (6.5)	$\alpha$	$\alpha$	<i>t</i> -Leu $\alpha$	<i>t</i> -Leu C=O
CO	170.0, C					
Arg $\alpha$	51.5, CH	4.39, m	$\beta$ , NH	$\beta$ , $\gamma$ , NH	Cys(SO <sub>3</sub> H)-NH	$\beta$ , $\gamma$ , C=O
$\beta$	29.5, CH <sub>2</sub>	1.61, m	$\alpha$ , $\gamma$	$\alpha$ , $\gamma$ , $\delta$		
$\gamma$	23.6, CH <sub>2</sub>	1.28, m	$\beta$ , $\delta$	$\alpha$ , $\beta$ , $\delta$		
$\delta$	39.7, CH <sub>2</sub>	2.94, m	$\gamma$	$\beta$ , $\gamma$		
guanidine	156.7, C					
NH		7.99, brs	$\alpha$	$\alpha$	Trp $\alpha$	Trp C=O
CO	171.4, C					
Cys(SO <sub>3</sub> H) $\alpha$	50.9, CH	4.60, m	$\beta$ , NH	$\beta$ , NH	Thr1-NH	$\beta$ , C=O
$\beta$	52.4, CH <sub>2</sub>	2.93, m	$\alpha$	$\alpha$ , NH		
NH		8.35, brd (6.5)	$\alpha$	$\alpha$ , $\beta$	Arg $\alpha$	Arg C=O
CO	170.8, C					
Thr1 $\alpha$	51.8, CH	4.83, d (8.5)	NH	$\beta$ , NH	NMeGln-NMe	C=O
$\beta$	69.5, CH	5.14, d (6.7)	$\gamma$	$\alpha$ , $\gamma$		C=O, Sar C=O
$\gamma$	17.3, CH <sub>3</sub>	1.16, d (6.5)	$\beta$	$\alpha$		$\alpha$ , $\beta$
NH		7.98, m	$\alpha$	$\alpha$ , $\beta$	Cys(SO <sub>3</sub> H) $\alpha$	Cys(SO <sub>3</sub> H) C=O
CO	167.9, C					
NMeGln $\alpha$	54.9, CH	4.97, m	$\beta$ 1	$\beta$ , $\gamma$	Phe-NH	
$\beta$ 1	24.7, CH <sub>2</sub>	1.70, m	$\alpha$ , $\gamma$	$\alpha$ , $\gamma$		CONH <sub>2</sub>
$\beta$ 2		1.89, m <sup>b</sup>		$\alpha$ , $\beta$ 1, $\gamma$		
$\gamma$	31.6, CH <sub>2</sub>	1.87, m <sup>b</sup>	$\beta$ 1	$\alpha$ , $\beta$ 1, $\beta$ 2		$\alpha$ , $\beta$ , CONH <sub>2</sub>
NMe	30.6, CH <sub>3</sub>	2.93, s			Thr1 $\alpha$	$\alpha$ , Thr1 C=O
CONH <sub>2</sub>	173.6, C	6.68, brs				$\gamma$ , CONH <sub>2</sub>
		7.17, brs				$\gamma$ , CONH <sub>2</sub>
CO	168.8, C					

Phe2 $\alpha$	54.2, CH	4.67, d (7.2)	NH	NH, $\beta$	Thr2-NH	$\beta$ , 1, C=O
$\beta$	38.0, CH <sub>2</sub>	3.00, m	$\alpha$	NH, $\alpha$		$\alpha$ , 1, 2, 6, C=O
C1	137.3, C					
C2/C6	129.4, CH	7.22, m	3, 5	3, 4, 5		$\beta$ , 1, 3, 4, 5
C3/C5	128.0, CH	7.14, m	2, 4, 6	2, 4, 6		1, 2, 4, 6
C4	126.3, CH	7.12, m	3, 5	2, 3, 5, 6		2, 3, 5, 6
NH		7.55, brd (6.6)	$\alpha$	$\alpha$ , $\beta$	NMeGln $\alpha$	NMeGln C=O
CO	171.1, C					
Thr2 $\alpha$	60.9, CH	3.85, m	NH, $\beta$	NH, $\beta$ , $\gamma$ , OH	Asn-NH	C=O
$\beta$	65.6, CH	3.96, m	$\alpha$ , $\gamma$ , OH	$\gamma$ , NH, OH		$\alpha$
$\gamma$	20.2, CH <sub>3</sub>	0.89, d (4.8)	$\beta$	$\alpha$ , $\beta$ , OH		$\alpha$ , $\beta$
OH		4.87, d (5.1)	$\beta$	$\alpha$ , $\beta$ , $\gamma$		
NH		7.92, brd (7.9)	$\alpha$	$\alpha$ , $\beta$	Phe $\alpha$	
CO	169.8, C					
Asn $\alpha$	45.9, CH	4.98, m	NH, $\beta$ 1, $\beta$ 2	NH, $\alpha$ , $\beta$ 1, $\beta$ 2	Sar-NMe	$\beta$ , CONH <sub>2</sub>
$\beta$ 1	36.9, CH <sub>2</sub>	2.08, m	$\alpha$	NH, $\alpha$ , $\beta$ 1, $\beta$ 2		$\alpha$ , C=O, CONH <sub>2</sub>
$\beta$ 2		2.64, m	$\alpha$	NH, $\alpha$ , $\beta$ 1, $\beta$ 2		
CONH <sub>2</sub>	171.2, C	6.78, brs	CONH <sub>2</sub>	CONH <sub>2</sub>		C=O, Thr2 C=O
		7.32, brs	CONH <sub>2</sub>	CONH <sub>2</sub>		$\beta$ , CONH <sub>2</sub>
NH		7.50, brd (9.6)	$\alpha$	NH, $\alpha$ , $\beta$ 1, $\beta$ 2	Thr2 $\alpha$	$\beta$ , CONH <sub>2</sub>
CO	172.1, C					
Sar $\alpha$	48.6, CH <sub>2</sub>	3.46, d (15.7)	$\alpha$ 2	$\alpha$ 2		C=O
		4.40, m	$\alpha$ 1	$\alpha$ 1		C=O
NMe	35.6, CH <sub>3</sub>	2.73, s			Asn $\alpha$	$\alpha$ , Asn C=O
CO	167.6, C					

<sup>a</sup>Derived from the combination of <sup>13</sup>C NMR, HSQC and HMBC data. <sup>b</sup>Singals overlapped.

**Table S2.** Absolute configuration of halicyclindramide F (**1**) by advanced Marfey's method and Edman degradation

Parent peptide	Constitutive amino acids	<i>m/z</i>	Retention time		Absolute configuration of amino acids
			AA-L-DLA	AA-D-DLA	
<b>1</b>	Ala <sup>a</sup>	495-496	39.08	33.99	D
	Pro <sup>a</sup>	521.5-522.5	33.89	37.28	L
	Val <sup>a</sup>	523-524	46.90	38.97	D
	<i>t</i> -Leu <sup>a</sup>	537-538	41.33	50.39	L
	Trp <sup>a</sup>	610.5-611.5	46.25	41.42	D
	Arg <sup>b,c</sup>	694-695	26.28	21.32	L
	Cys(SO <sub>3</sub> H) <sup>a,c</sup>	575-576	11.30	12.74	D
	Thr1, Thr2 <sup>a</sup>	525-526	27.73	33.90	L
	NMeGln <sup>a</sup>	567-568	28.84	29.69	L
	Phe1, Phe2 <sup>d</sup>	457.5-458.8	49.32/43.57	49.32/43.50	D & L
	Asn <sup>d</sup>	524.5-525.0	36.99	36.99	
Standard amino acids	L- <i>allo</i> -Thr	525-526	28.82	31.27	
	L-Asn <sup>e</sup>	384.5-385.5	47.80	56.20	
<b>1</b>	Asn <sup>e</sup>	384.5-385.5	L-AA-L-DAA 47.76	D-AA-L-DAA 56.20	L
Dodecapeptide of <b>1</b> <sup>f</sup>	Phe2 <sup>d</sup>	457.5-458.8	48.78	42.99	D

<sup>a</sup> The ions of advanced Marfey's reaction products were detected as form of [M+CF<sub>3</sub>COO]<sup>-</sup> under negative ion detection mode.

<sup>b</sup> The ions of advanced Marfey's reaction products were detected as form of [M+2CF<sub>3</sub>COOH-H]<sup>-</sup> under negative ion detection mode.

<sup>c</sup> The L-AA-D-DLA or D-AA-L-DLA products derived from the amino acids with highly polar residues such as Arg and Cys(SO<sub>3</sub>H) elute faster than L-AA-L-DLA or D-AA-D-DLA products (Fuji et al., *Anal. Chem.* **1997**, *69*, 5146-5151).

<sup>d</sup> The ions of advanced Marfey's reaction products were detected as form of [M-H]<sup>-</sup> under negative ion detection mode.

<sup>e</sup> The absolute configuration of Asn was determined by Marfey's method (with based on the comparison of retention time of natural Asn-L-DAA to those of two authentic standards (L-Asn-L-DAA 47.80/ D-Asn-L-DAA 56.20 min).

<sup>f</sup> Dodecapeptide of **1** was prepared by deformylation followed by two cycles of Edman degradation.

**Table S3.** NMR spectroscopic data for halicylindramide G (**2**) in DMSO-*d*<sub>6</sub> at 600 MHz (<sup>1</sup>H) and 150 MHz (<sup>13</sup>C)

position	$\delta_c$ , mult. <sup>a</sup>	$\delta_{H_1}$ , ( <i>J</i> in Hz)	COSY	TOCSY	NOESY	HMBC (H to C)
NCHO	160.5, C	7.88, s	Ala-NH	Ala-NH, Ala $\alpha$ , Ala $\beta$	Ala-NH	
Ala $\alpha$	46.4, CH	4.31, q (7.3)	NH, $\beta$	NH, $\beta$ , formyl	$\beta$ , formyl, Br-Phe-NH	$\beta$ , formyl C=O
$\beta$	18.6, CH <sub>3</sub>	0.93, d (7.0)	$\alpha$	NH, $\alpha$	$\alpha$	$\alpha$ , C=O
NH		8.18, brs	$\alpha$ , formyl	formyl, $\alpha$ , $\beta$	formyl	
CO	171.4, C					
BrPhe $\alpha$	51.9, CH	4.71, m <sup>b</sup>	NH, $\beta$ 1, $\beta$ 2	NH, $\beta$ 1, $\beta$ 2	Pro $\delta$	
$\beta$ 1	36.2, CH <sub>2</sub>	2.69-2.72 m	$\alpha$ , $\beta$ 2	NH, $\alpha$ , $\beta$ 2	$\alpha$	1, 2, 6
$\beta$ 2		3.02, m <sup>b</sup>	$\alpha$ , $\beta$ 1	NH, $\alpha$ , $\beta$ 1	$\alpha$	1, 2, 6
C1	137.1, C					
C2/C6	131.8, CH	7.26, m <sup>b</sup>	3, 5	3, 5	3, 5	$\beta$ , 1, 3, 4, 6
C3/C5	130.8, CH	7.42, m	2, 6	2, 6	2, 6	1, 4
C4	119.5, C					
NH		8.44, brd (8.3)	$\alpha$	$\alpha$ , $\beta$	Ala $\alpha$	C=O
CO	171.0, C					
Pro $\alpha$	59.5, CH	4.46, m	$\beta$ 1, $\beta$ 2	$\beta$ 1, $\beta$ 2, $\gamma$ , $\delta$	Val-NH	
$\beta$ 1	29.5, CH <sub>2</sub>	1.89, m	$\alpha$ , $\gamma$	$\alpha$ , $\beta$ 2, $\gamma$ , $\delta$		
$\beta$ 2		2.10, m	$\alpha$ , $\gamma$	$\alpha$ , $\beta$ 1, $\gamma$ , $\delta$		C=O
$\gamma$	24.6, CH <sub>2</sub>	1.90, m	$\beta$ 1, $\beta$ 2, $\delta$	$\alpha$ , $\beta$ 1, $\beta$ 2, $\delta$		
$\delta$	46.9, CH <sub>2</sub>	3.70-3.62 m	$\gamma$	$\alpha$ , $\beta$ , $\gamma$	BrPhe $\alpha$	
CO	169.8, C					
Val $\alpha$	57.2, CH	4.38, m <sup>b</sup>	NH, $\beta$	NH	t-Leu-NH	C=O
$\beta$	30.7, CH	2.08, m	$\alpha$ , $\gamma$ , $\gamma'$	$\alpha$ , $\gamma$ , $\gamma'$		C=O
$\gamma$	17.4, CH <sub>3</sub>	0.76, d (6.7)	$\beta$	$\beta$ , $\gamma'$		$\alpha$ , $\beta$ , $\gamma'$
$\gamma'$	19.4, CH <sub>3</sub>	0.81, d (6.7)	$\beta$	$\beta$		$\alpha$ , $\beta$ , $\gamma$
NH		7.80, m <sup>b</sup>	$\alpha$	$\alpha$ , $\beta$	Pro $\alpha$	
CO	171.9, C					
t-Leu $\alpha$	60.2, CH	4.13, m <sup>b</sup>	NH	NH	Dioia-NH	$\beta$ , $\gamma$ , C=O
$\beta$	34.1, C					
$\gamma$	26.6, CH <sub>3</sub>	0.86, s				$\alpha$ , $\beta$
NH		7.82, m <sup>b</sup>	$\alpha$	$\alpha$	Val $\alpha$	
CO	170.0, C					
Dioia $\alpha$	48.5, CH	4.46, m <sup>b</sup>	NH, $\beta$ 1, $\beta$ 2	NH, $\beta$ 1, $\beta$ 2	Arg-NH	C=O
$\beta$ 1	39.0, CH <sub>2</sub>	2.30, m	$\alpha$ , $\beta$ 2	NH, $\alpha$		2, 3
$\beta$ 2		1.89, m	$\alpha$ , $\beta$ 1	NH, $\alpha$		2, 3
C2	178.2, C					
C3	74.0, C					
C3a	131.7, C					
C7a	141.7, C					
C4	124.0, CH	7.35, d (7.6)	5	5, 6, 7		3, 7a
C5	122.0, CH	6.90, dd (7.6, 7.3)	4, 6	4, 6, 7		3a, 7
C6	128.0, CH	7.18, m <sup>b</sup>	5, 7	4, 5, 7		7a
C7	109.8, CH	6.78, d (8.0)	6	4, 5, 6		3a, 5
1-NH		10.24, brs				3
3-OH		6.16, brs				
NH		8.21, brd (7.9)	$\alpha$		t-Leu $\alpha$	
CO	169.8, C					
Arg $\alpha$	52.0, CH	4.22, m <sup>b</sup>	NH, $\beta$	NH, $\beta$	Cys(SO <sub>3</sub> H)-NH	
$\beta$	29.1, CH <sub>2</sub>	1.65, m	$\alpha$ , $\gamma$	$\alpha$ , $\gamma$ , $\delta$		C=O
$\gamma$	23.2, CH <sub>2</sub>	1.40, m	$\beta$ , $\delta$	$\alpha$ , $\beta$ , $\delta$		
$\delta$	40.2, CH <sub>2</sub>	3.01, m <sup>b</sup>	$\gamma$	$\beta$ , $\gamma$		
guanidine	156.8, C					
NH		7.81, brs <sup>b</sup>	$\alpha$	$\alpha$	Dioia $\alpha$	Dioia C=O
CO	171.0, C					
Cys(SO <sub>3</sub> H) $\alpha$	50.9, CH	4.59, m	NH, $\beta$	NH, $\beta$	Thr1-NH	C=O
$\beta$	52.5, CH <sub>2</sub>	2.90, m	$\alpha$	$\alpha$		
NH		8.30, brd (8.3)	$\alpha$	$\alpha$ , $\beta$	Arg $\alpha$	Arg C=O
CO	169.4, C					
Thr1 $\alpha$	51.9, CH	4.84, d (8.5)	NH	NH	NMeGln-NMe	$\gamma$ , C=O, Cys(SO <sub>3</sub> H) C=O
$\beta$	69.2, CH	5.14, d (6.7)	$\gamma$	$\gamma$		$\alpha$ , $\gamma$ , Sar C=O
$\gamma$	17.4, CH <sub>3</sub>	1.17, d (6.5)	$\beta$	$\beta$		$\alpha$
NH		7.96, m	$\alpha$	$\alpha$	Cys(SO <sub>3</sub> H) $\alpha$	
CO	169.2, C					
NMeGln $\alpha$	54.5, CH	4.98, m <sup>b</sup>	$\beta$	$\beta$ , $\gamma$	$\beta$ , $\gamma$ , Phe-NH	C=O, NMe
$\beta$	24.7, CH <sub>2</sub>	1.70, m	$\alpha$ , $\gamma$	$\alpha$ , $\gamma$	$\alpha$ , $\gamma$	$\alpha$
$\gamma$	31.5, CH <sub>2</sub>	1.89, m	$\beta$	$\alpha$ , $\beta$		CONH <sub>2</sub>
NMe	30.4, CH <sub>3</sub>	2.94, s			$\alpha$ , Thr1 $\alpha$ , Thr1 $\beta$	$\alpha$ , Thr1 C=O
CONH <sub>2</sub>	173.5, C	6.64, brs 7.17, brs <sup>b</sup>				$\gamma$ $\gamma$
CO	167.5, C					
Phe $\alpha$	54.0, CH	4.69, d (7.2)	NH, $\beta$	NH, $\beta$	Thr2-NH	$\beta$ , C=O

$\beta$	37.9, CH <sub>2</sub>	2.98, m	$\alpha$	NH, $\alpha$		$\alpha$
C1	137.1, C					
C2/C6	129.3, CH	7.18, m <sup>b</sup>	3, 5	3, 4, 5		$\beta$ , 4
C3/C5	129.0, CH	7.23, m <sup>b</sup>	2, 4, 6	2, 4, 6		1, 2, 6
C4	126.2, CH	7.15, m <sup>b</sup>	3, 5	2, 3, 5, 6		2, 3, 5, 6
NH		7.56, brd (6.6)	$\alpha$	$\alpha$ , $\beta$	NMeGln $\alpha$	
CO	170.7, C					
Thr2 $\alpha$	60.7, CH	3.85, m	NH, $\beta$	NH, $\beta$ , $\gamma$ , OH	Asn-NH	$\beta$ , C=O
$\beta$	65.5, CH	3.96, m	$\alpha$ , $\gamma$ , OH	$\gamma$ , NH, OH	Asn-NH	
$\gamma$	20.1, CH <sub>3</sub>	0.89, d (6.3)	$\beta$	$\alpha$ , $\beta$ , OH		$\alpha$ , $\beta$
OH		4.88, d (4.9)				
NH		7.95, brs	$\alpha$	$\alpha$ , $\beta$	Phe $\alpha$	
CO	169.6, C					
Asn $\alpha$	46.0, CH	4.98, m <sup>b</sup>	NH, $\beta$ 1, $\beta$ 2	NH, $\alpha$ , $\beta$ 1, $\beta$ 2		CONH <sub>2</sub>
$\beta$ 1	36.9, CH <sub>2</sub>	2.09, m	$\alpha$	NH, $\alpha$ , $\beta$ 1, $\beta$ 2		C=O, CONH <sub>2</sub>
$\beta$ 2		2.30, m	$\alpha$	NH, $\alpha$ , $\beta$ 1, $\beta$ 2		$\alpha$ , CONH <sub>2</sub>
CONH <sub>2</sub>	172.1, C	6.72, brs				CONH <sub>2</sub>
		7.33, brs				
NH		7.51, brd (7.6)	$\alpha$	NH, $\alpha$ , $\beta$ 1, $\beta$ 2	Thr2 $\alpha$ , Thr2 $\beta$	C=O
CO	169.4, C					
Sar $\alpha$ 1	49.5, CH <sub>2</sub>	3.49, d (17.3)	$\alpha$ 2	$\alpha$ 2		C=O, Asn C=O
$\alpha$ 2		4.42, m <sup>b</sup>	$\alpha$ 1	$\alpha$ 1		C=O
NMe	35.4, CH <sub>3</sub>	2.74, s				$\alpha$
CO	167.5, C					

<sup>a</sup>Derived from the combination of <sup>13</sup>C NMR, HSQC and HMBC data. <sup>b</sup>Singals overlapped.

**Table S4.** NMR spectroscopic data for halicylindramide H (**3**) in DMSO-*d*<sub>6</sub> at 600 MHz (<sup>1</sup>H) and 150 MHz (<sup>13</sup>C)

position	$\delta_c$ , mult. <sup>a</sup>	$\delta_{H_i}$ ( <i>J</i> in Hz)	COSY	TOCSY	NOESY	HMBC (H to C)
NCHO	161.4, C	7.90, s	Ala-NH	Ala-NH, Ala $\alpha$ , Ala $\beta$	Ala-NH	
Ala $\alpha$	47.6, CH	4.31, m <sup>b</sup>	NH, $\beta$	NH, $\beta$	$\beta$ , Br-Phe-NH	$\beta$
$\beta$	19.5, CH <sub>3</sub>	0.92, d (7.0)	$\alpha$	NH, $\alpha$		$\alpha$ , C=O
NH		8.18, brs	formyl, $\alpha$	formyl, $\alpha$ , $\beta$	formyl	
CO	172.3, C					
BrPhe $\alpha$	52.6, CH	4.72, m <sup>b</sup>	NH, $\beta$ 1, $\beta$ 2	NH, $\beta$ 1, $\beta$ 2	Pro $\delta$	$\beta$ , 1
$\beta$ 1	37.1, CH <sub>2</sub>	2.71, m	$\beta$ 2	NH, $\alpha$ , $\beta$ 2	$\alpha$	$\alpha$ , C=O, 1, 2, 6
$\beta$ 2		3.01, m <sup>b</sup>	$\alpha$ , $\beta$ 1	NH, $\alpha$ , $\beta$ 1	$\alpha$ , Pro $\delta$	$\alpha$ , C=O, 1, 2, 6
C1	138.2, C					
C2/C6	132.6, CH	7.26, m <sup>b</sup>	3, 5	3, 5	$\beta$ 1, $\beta$ 2, Ala $\beta$ 1, Ala $\beta$ 2	$\beta$ , 1, 3, 4, 6
C3/C5	131.7, CH	7.43, d (8.1)	2, 6	2, 6	$\beta$ 1, $\beta$ 2, Ala $\beta$ 1, Ala $\beta$ 2,	1, 2, 4, 5
C4	120.3, C					
NH		8.42, m <sup>b</sup>	$\alpha$	$\alpha$ , $\beta$ 1, $\beta$ 2	Ala $\alpha$	C=O
CO	171.4, C					
Pro $\alpha$	59.8, C	4.40, m <sup>b</sup>	$\beta$ 1, $\beta$ 2	$\beta$ 1, $\beta$ 2, $\gamma$	Val-NH	$\beta$ , $\gamma$
$\beta$ 1	30.5, CH <sub>2</sub>	1.80, m <sup>b</sup>	$\alpha$ , $\gamma$	$\alpha$ , $\beta$ 2, $\gamma$ , $\delta$		$\alpha$ , $\gamma$ , $\delta$ , C=O
$\beta$ 2		2.02, m <sup>b</sup>	$\alpha$ , $\gamma$	$\alpha$ , $\beta$ 1, $\gamma$ , $\delta$		C=O
$\gamma$	25.2, CH <sub>2</sub>	1.83, m <sup>b</sup>	$\beta$ 1, $\beta$ 2, $\delta$ 1, $\delta$ 2	$\alpha$ , $\beta$ 1, $\beta$ 2, $\delta$ 1, $\delta$ 2	$\alpha$ , $\delta$	$\alpha$ , $\beta$ , $\delta$
$\delta$ 1	47.8, CH <sub>2</sub>	3.60, m	$\gamma$	$\beta$ , $\gamma$	BrPhe $\alpha$ , BrPhe $\beta$ 2	$\beta$ , $\gamma$
$\delta$ 2		3.71, m	$\gamma$	$\beta$ , $\gamma$		$\beta$ , $\gamma$
CO	168.7, C					
Val $\alpha$	58.1, CH	4.37, m <sup>b</sup>	NH, $\beta$	NH, $\beta$ , $\gamma$ , $\gamma'$	<i>t</i> -Leu-NH	$\beta$ , $\gamma$ , $\gamma'$ , C=O
$\beta$	31.8, CH	2.09, m <sup>b</sup>	$\alpha$ , $\gamma$ , $\gamma'$	NH, $\alpha$ , $\gamma$ , $\gamma'$		$\alpha$ , $\gamma$ , $\gamma'$ , C=O
$\gamma$	18.2, CH <sub>3</sub>	0.77, d (6.7)	$\beta$	$\alpha$ , $\beta$ , $\gamma'$		$\alpha$ , $\beta$ , $\gamma'$
$\gamma'$	20.3, CH <sub>3</sub>	0.82, d (6.5)	$\beta$	$\alpha$ , $\beta$ , $\gamma$		$\alpha$ , $\beta$ , $\gamma$
NH		7.78, m <sup>b</sup>	$\alpha$	$\alpha$ , $\beta$	Pro $\alpha$	
CO	171.4, C					
<i>t</i> -Leu $\alpha$	61.3, CH	4.13, m <sup>b</sup>	NH	NH	Dioia-NH	$\gamma$ , C=O
$\beta$	34.9, C					
$\gamma$	27.6, CH <sub>3</sub>	0.89, s			Dioia-NH	$\alpha$ , $\beta$
NH		7.91, m <sup>b</sup>	$\alpha$	$\alpha$	Val $\alpha$	
CO	170.9, C					
Dioia $\alpha$	50.8, CH	4.42, m <sup>b</sup>	NH	NH, $\beta$ 1, $\beta$ 2	Arg-NH	$\beta$ , 3, C=O
$\beta$ 1	39.9, CH <sub>2</sub>	2.18, m	$\alpha$	NH, $\alpha$		$\alpha$ , 3
$\beta$ 2		1.96, m	$\alpha$	NH, $\alpha$		$\alpha$ , 2, 3
C2	179.8, C					
C3	75.2, C					
C3a	132.0, C					
C7a	142.1, C					
C4	125.0, CH	7.23, m <sup>b</sup>	5	5, 6, 7	5	3a, 6, 7a
C5	122.6, CH	6.94, brt (7.3)	4, 6	4, 6, 7	4, 6	3a, 7
C6	129.3, CH	7.19, m <sup>b</sup>	5, 7	4, 5, 7	5, 7	4, 7a
C7	110.8, CH	6.80, brd (7.5)	6	4, 5, 6	6	3a, 5
1-NH		10.32, brs				3
3-OH		6.18, brs			$\alpha$ , $\beta$ 1, $\beta$ 2	
NH		8.33, brs	$\alpha$	$\alpha$ , $\beta$ 1, $\beta$ 2	$\beta$ , <i>t</i> -Leu $\alpha$ , <i>t</i> -Leu $\gamma$	
CO	169.1, C					
Arg $\alpha$	53.0, CH	4.27, m <sup>b</sup>	NH, $\beta$	NH, $\beta$	Cys(SO <sub>3</sub> H)-NH	$\beta$
$\beta$	29.5, CH <sub>2</sub>	1.68, m <sup>b</sup>	$\alpha$ , $\gamma$	$\alpha$ , $\gamma$		$\alpha$ , C=O
$\gamma$	24.8, CH <sub>2</sub>	1.45, m	$\beta$ , $\delta$	$\beta$ , $\delta$		$\beta$ , $\delta$
$\delta$	41.2, CH <sub>2</sub>	3.01, m <sup>b</sup>	$\gamma$	$\beta$ , $\gamma$		$\beta$ , $\gamma$
guanidine	157.7, C					
NH		7.82, brs <sup>b</sup>	$\alpha$	$\alpha$		Dioia C=O
CO	172.3, C					
Cys(SO <sub>3</sub> H) $\alpha$	51.8, CH	4.59, m	NH, $\beta$	NH, $\beta$	Thr1-NH, Thr1- $\gamma$	$\beta$ , C=O
$\beta$	53.4, CH <sub>2</sub>	2.96, m <sup>b</sup>	$\alpha$	NH, $\alpha$	NH	$\alpha$
NH		8.30, brd (7.2)	$\alpha$	$\alpha$ , $\beta$	$\beta$ , Arg $\alpha$	Arg C=O
CO	169.0, C					
Thr1 $\alpha$	52.8, CH	4.83, d (8.6)	NH	NH	NMeGln-NMe, NMeGln $\gamma$	$\gamma$ , C=O, Cys(SO <sub>3</sub> H) C=O
$\beta$	69.8, CH	5.13, brq (6.6)	$\alpha$ , $\gamma$	$\gamma$	NMeGln-NMe	$\gamma$ , Sar C=O
$\gamma$	18.2, CH <sub>3</sub>	1.16, d (6.5)	$\beta$	$\beta$	NH, $\alpha$ , $\beta$ , Cys(SO <sub>3</sub> H) $\alpha$	$\alpha$ , $\beta$
NH		8.01, brs	$\alpha$	$\alpha$	$\gamma$ , Cys(SO <sub>3</sub> H) $\alpha$ , Cys(SO <sub>3</sub> H) $\beta$	
CO	170.5, C					
NMeGln $\alpha$	55.2, CH	4.96, m <sup>b</sup>	$\beta$ 1, $\beta$ 2	$\beta$ 1, $\beta$ 2, $\gamma$ 1, $\gamma$ 2	$\gamma$ , Thr1 $\gamma$ , Phe-NH	C=O, NMe
$\beta$ 1	26.7, CH <sub>2</sub>	1.70, m <sup>b</sup>	$\alpha$ , $\gamma$ 2	$\alpha$ , $\beta$ 2, $\gamma$ 1, $\gamma$ 2		$\gamma$
$\beta$ 2		1.89, m <sup>b</sup>	$\alpha$ , $\gamma$ 2	$\alpha$ , $\beta$ 1, $\gamma$ 1, $\gamma$ 2		
$\gamma$ 1	32.4, CH <sub>2</sub>	1.87, m <sup>b</sup>	$\beta$ 1, $\beta$ 2, $\gamma$ 2	$\alpha$ , $\beta$ 1, $\beta$ 2, $\gamma$ 2	Thr1 $\alpha$	CONH <sub>2</sub>

$\gamma$ 2		1.92, m <sup>b</sup>	$\beta$ 1, $\beta$ 2, $\gamma$ 1	$\alpha$ , $\beta$ 1, $\beta$ 2, $\gamma$ 1		
NMe	31.3, CH <sub>3</sub>	2.94, s			Thr1 $\alpha$ , Thr1 $\beta$	$\alpha$ , Thr1 C=O
CONH <sub>2</sub>	174.4, C	6.67, brs 7.16, brs <sup>b</sup>				$\gamma$ $\gamma$
CO	168.6, C					
Phe $\alpha$	55.0, CH	4.69, m <sup>b</sup>	NH, $\beta$ 1, $\beta$ 2	NH, $\beta$ 1, $\beta$ 2	Phe-NH, Thr2-NH	$\beta$
$\beta$ 1	39.2, CH <sub>2</sub>	2.96, m <sup>b</sup>	$\alpha$	NH, $\alpha$	Thr2-NH	C=O, 1, 2, 6
$\beta$ 2		3.02, m <sup>b</sup>	$\alpha$	NH, $\alpha$		
C1	138.0, C					
C2/C6	130.2, CH	7.18, m <sup>b</sup>	3, 5	3, 4, 5	$\beta$ 1, $\beta$ 2, NMeGln $\gamma$ , Thr2 $\gamma$	$\beta$ , 4
C3/C5	128.9, CH	7.23, m <sup>b</sup>	2, 4, 6	2, 4, 6	$\beta$ 1, $\beta$ 2	1
C4	127.2, CH	7.17, m <sup>b</sup>	3, 5	2, 3, 5, 6		2, 6
NH		7.56, brs			NMeGln $\alpha$	
CO	171.9, C					
Thr2 $\alpha$	61.9, CH	3.86, m	NH, $\beta$	NH, $\beta$ , $\gamma$ , OH	Asn-NH	Phe C=O
$\beta$	66.3, CH	3.96, brs	$\alpha$ , $\gamma$	$\alpha$ , $\gamma$ , OH		
$\gamma$	21.0, CH <sub>3</sub>	0.85, m <sup>b</sup>	$\beta$	$\alpha$ , $\beta$ , OH	Asn-NH	$\alpha$ , $\beta$
OH		4.90, brs				
NH		7.96, brd (6.8)	$\alpha$	$\alpha$ , $\beta$	$\gamma$ , Phe $\alpha$ , Phe $\beta$	
CO	170.1, C					
Asn $\alpha$	46.8, CH	4.99, m <sup>b</sup>	NH, $\beta$ 1, $\beta$ 2	NH, $\alpha$ , $\beta$ 1, $\beta$ 2	Sar-NMe	CONH <sub>2</sub>
$\beta$ 1	38.1, CH <sub>2</sub>	2.08, m <sup>b</sup>	$\alpha$	NH, $\alpha$ , $\beta$ 1, $\beta$ 2		C=O, CONH <sub>2</sub>
$\beta$ 2		2.66, m <sup>b</sup>	$\alpha$	NH, $\alpha$ , $\beta$ 1, $\beta$ 2		$\alpha$ , CONH <sub>2</sub>
NH		7.50, brd (9.1)	$\alpha$	NH, $\alpha$ , $\beta$ 1, $\beta$ 2	$\beta$ 1, Thr2 $\alpha$ , Thr2 $\gamma$ , Sar-NMe,	C=O
CONH <sub>2</sub>	173.0, C	6.77, brs 7.32, brs			$\beta$ 1, $\beta$ 2 $\beta$ 1, $\beta$ 2	$\beta$ , CONH <sub>2</sub> $\beta$
CO	170.2, C					
Sar $\alpha$ 1	50.6, CH <sub>2</sub>	3.48, m <sup>b</sup>	$\alpha$ 2	$\alpha$ 2		C=O, Asn C=O
$\alpha$ 2		4.42, m <sup>b</sup>	$\alpha$ 1	$\alpha$ 1		C=O
NMe	36.4, CH <sub>3</sub>	2.73, s				$\alpha$ , Asn C=O
CO	168.5, C					

<sup>a</sup>Derived from the combination of <sup>13</sup>C NMR, HSQC and HMBC data. <sup>b</sup>Singals overlapped.

**Table S5.** Absolute configuration of halicyclindramides G (**2**) and H (**3**) by advanced Marfey's method and Edman degradation

Parent peptide	Constitutive amino acids	<i>m/z</i>	Retention time		Absolute configuration of amino acids
			AA-L-DLA	AA-D-DLA	
<b>2</b>	Ala <sup>a</sup>	495-496	38.40	34.49	D
	BrPhe <sup>a</sup>	648.5-649.5	48.32	55.27	L
	Pro <sup>a</sup>	521.5-522.5	33.71	37.28	L
	Val <sup>a</sup>	523-524	46.90	38.97	D
	<i>t</i> -Leu <sup>a</sup>	537-538	41.42	50.39	L
	Arg <sup>b,c</sup>	694-695	25.98	21.32	L
	Cys(SO <sub>3</sub> H) <sup>a,c</sup>	575-576	11.66	12.74	D
	Thr1, Thr2 <sup>a</sup>	525-526	27.73	33.90	L
	NMeGln <sup>a</sup>	567-568	28.75	29.69	L
	Phe <sup>d</sup>	457.5-458.8	43.50	49.38	D
Asn <sup>d</sup>	524.5-525.0	36.31	36.31		
<b>3</b>	Ala <sup>a</sup>	495-496	38.27	34.37	D
	BrPhe <sup>a</sup>	648.5-649.5	48.17	55.11	L
	Pro <sup>a</sup>	521.5-522.5	33.59	37.16	L
	Val <sup>a</sup>	523-524	46.76	38.84	D
	<i>t</i> -Leu <sup>a</sup>	537-538	41.29	50.24	L
	Arg <sup>b,c</sup>	694-695	25.88	21.23	L
	Cys(SO <sub>3</sub> H) <sup>a,c</sup>	575-576	11.59	12.66	D
	Thr1, Thr2 <sup>a</sup>	525-526	27.62	33.78	L
	NMeGln <sup>a</sup>	567-568	28.64	29.58	L
	Phe <sup>d</sup>	457.5-458.8	43.36	49.24	D
Asn <sup>d</sup>	524.5-525.0	36.85	36.85		
Standard amino acids	L- <i>allo</i> -Thr	525-526	28.82	31.27	
	L-Asn <sup>e</sup>	384.5-385.5	47.80 L-AA-L-DAA	56.20 D-AA-L-DAA	
<b>2</b>	Asn <sup>e</sup>	384.5-385.5	47.73		L
<b>3</b>	Asn <sup>e</sup>	384.5-385.5	47.70		L

<sup>a</sup> The ions of advanced Marfey's reaction products were detected as form of [M+CF<sub>3</sub>COO]<sup>-</sup> under negative ion detection mode.

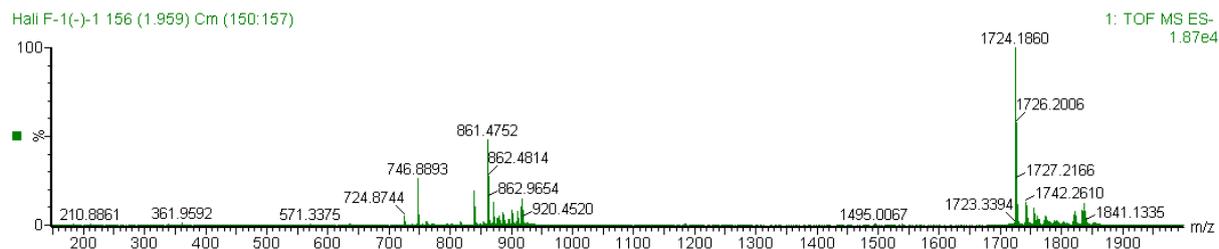
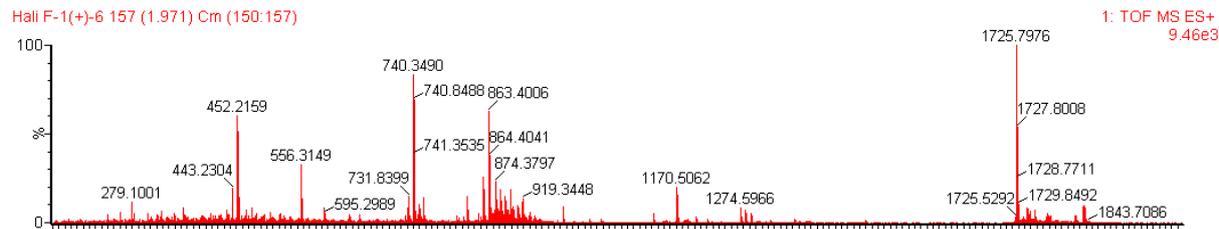
<sup>b</sup> The ions of advanced Marfey's reaction products were detected as form of [M+2CF<sub>3</sub>COOH-H]<sup>-</sup> under negative ion detection mode.

<sup>c</sup> The L-AA-D-DLA or D-AA-L-DLA products derived from the amino acids with highly polar residues such as Arg and Cys(SO<sub>3</sub>H) elute faster than L-AA-L-DLA or D-AA-D-DLA products (Fuji et al., *Anal. Chem.* **1997**, *69*, 5146-5151).

<sup>d</sup> The ions of advanced Marfey's reaction products were detected as form of [M-H]<sup>-</sup> under negative ion detection mode.

<sup>e</sup> The absolute configuration of Asn was determined by Marfey's method based on the comparison of retention time of natural Asn-L-DAA to those of two authentic standards (L-Asn-L-DAA 47.80/ D-Asn-L-DAA 56.20 min on positive ion detection [M+H]<sup>+</sup>).

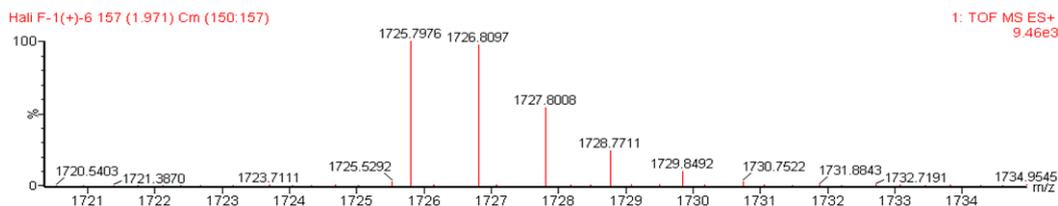
# S6. HR-ESI-ToF-MS spectrum of halicyclindramide F (1)



Calculated  
Isotope  
Model



Analysis  
Result



**Single Mass Analysis**  
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0  
Selected filters: None  
Monoisotopic Mass, Even Electron ions  
12 formula[e] evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

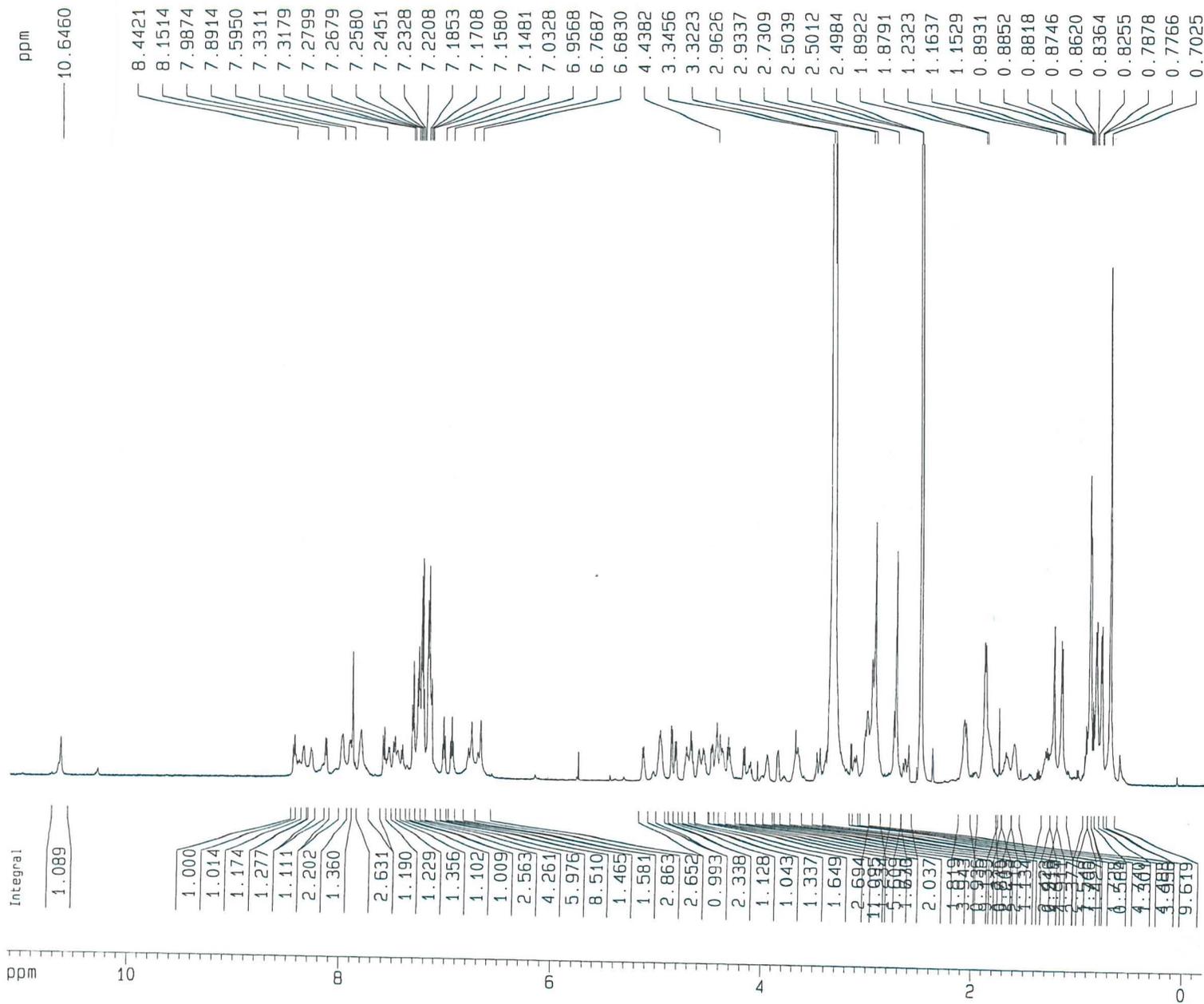
Mass	Calc. Mass	mDa	PPM	DBE	Formula	IFIT	C	H	N	O	S
1725.7976	1725.7947	2.9	1.7	33.5	C80 H113 N18 O23 S	5550753.0	80	113	18	23	1
	1725.8059	-8.3	-4.8	33.5	C79 H113 N20 O22 S	5550753.5	79	113	20	22	1

MCP 2300, CP 2200  
Hall F-1(+)-6 157 (1.971) Cm (150:157) 1: TOF MS ES+ 9.46e3

For Help, press F1

S7. <sup>1</sup>H NMR (600 MHz) spectrum of halicylindramide F (1) in DMSO-d<sub>6</sub>

6



Current Data Parameters  
 NAME jul07-snu-khj  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20040707  
 Time 19.29  
 INSTRUM spect  
 PROBHD 5 mm CPTXI 1H/  
 PULPROG zg30  
 TD 65536  
 SOLVENT DMSO  
 NS 8  
 DS 0  
 SWH 8741.259 Hz  
 FIDRES 0.133381 Hz  
 AQ 3.7487664 sec  
 RG 32  
 DW 57.200 usec  
 DE 6.00 usec  
 TE 297.9 K  
 D1 1.0000000 sec  
 MCREST 0.0000000 sec  
 MCWRK 0.0150000 sec

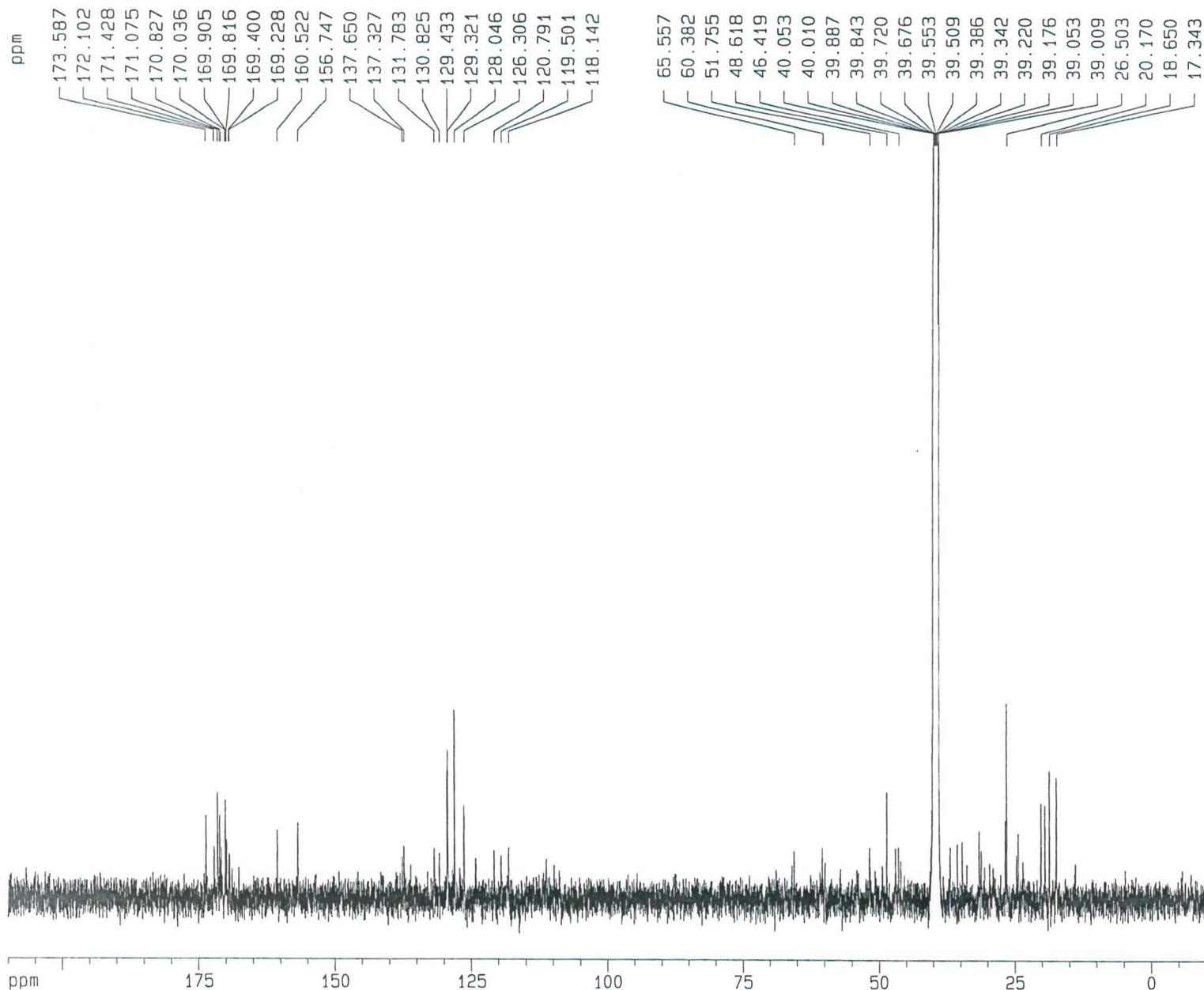
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 PL1 -5.00 dB  
 SFO1 600.1336696 MHz

F2 - Processing parameters  
 SI 32768  
 SF 600.1300063 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 1000.00 cm  
 F1P 11.126 ppm  
 F1 6677.05 Hz  
 F2P -0.198 ppm  
 F2 -119.04 Hz  
 PPMCM 0.56622 ppm/cm  
 HZCM 339.80441 Hz/cm

# S8. <sup>13</sup>C NMR (150 MHz) spectrum of halicylindramide F (1) in DMSO-d6

/C13



Current Data Parameters  
 NAME aug13-ee-chj  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20040816  
 Time 8.56  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H/2H  
 PULPROG zgdc  
 TD 65536  
 SOLVENT DMSO  
 NS 64657  
 DS 0  
 SWH 30303.031 Hz  
 FIDRES 0.462388 Hz  
 AQ 1.0814105 sec  
 RG 32768  
 DW 16.500 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec

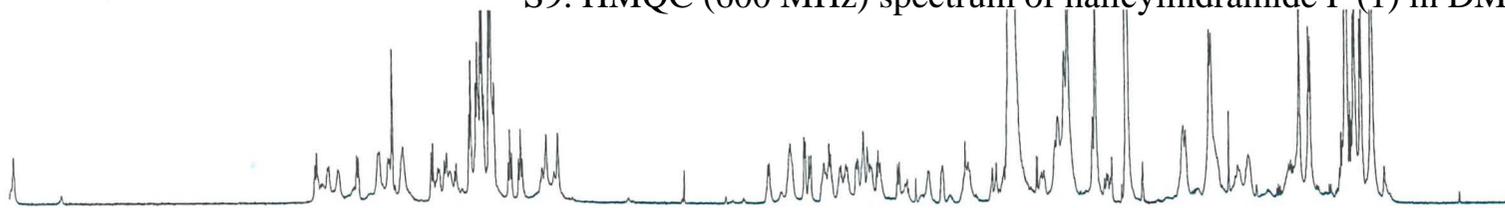
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 P1 18.00 usec  
 PL1 0.00 dB  
 SFO1 125.7709436 MHz

==== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 100.00 usec  
 PL2 0.00 dB  
 PL12 23.50 dB  
 SFO2 500.1320005 MHz

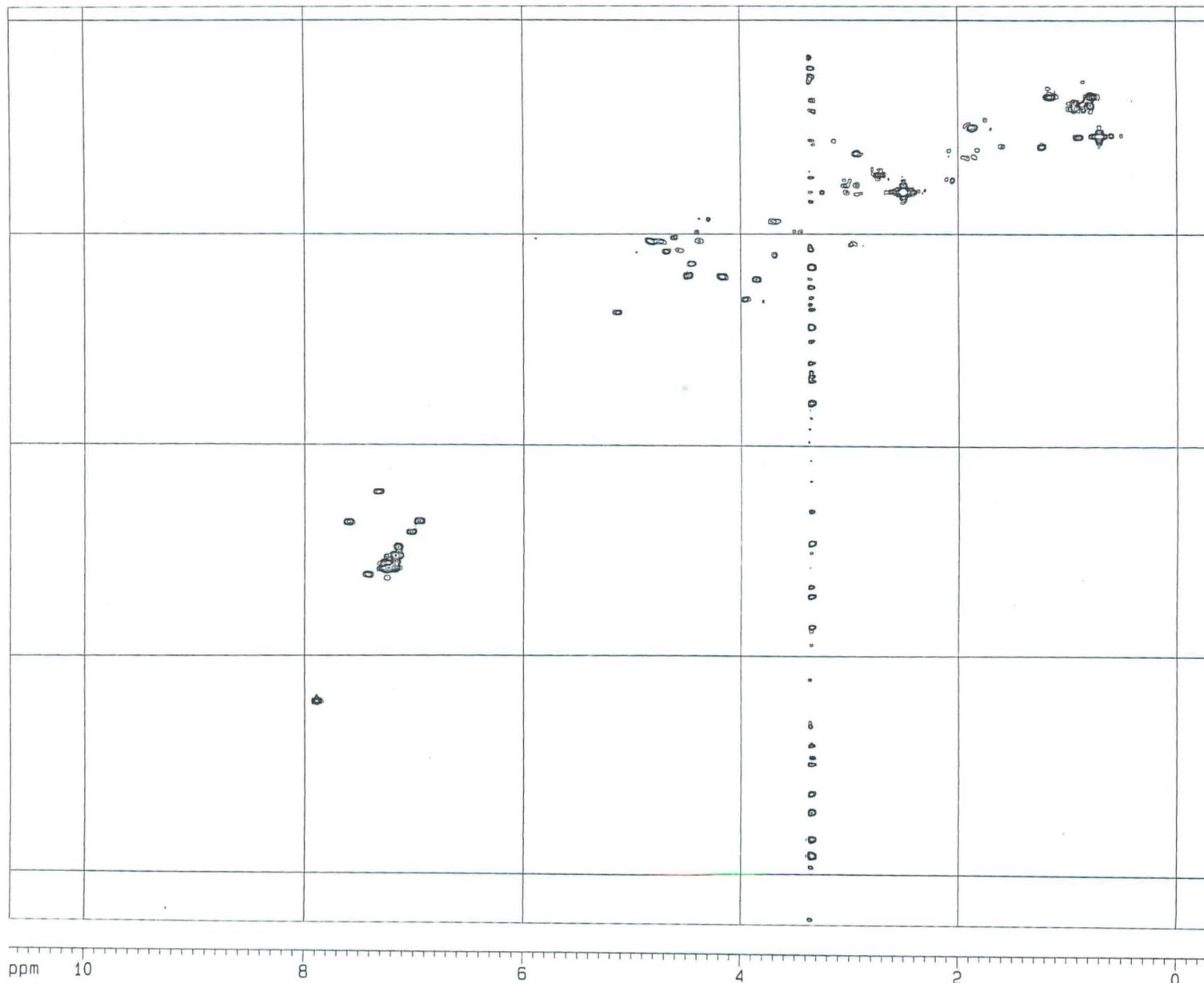
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 SF 125.7578452 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 CY 200.00 cm  
 F1P 210.000 ppm  
 F1 26409.15 Hz  
 F2P -10.000 ppm  
 F2 -1257.58 Hz  
 PPMCM 11.00000 ppm/cm  
 HZCM 1383.33630 Hz/cm

S9. HMQC (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/HMQC



```

Current Data Parameters
NAME      ju107-snu-hh)
EXPNO     5
PROCNO    1

F2 - Acquisition Parameters
Date_     20040707
Time      23.35
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   magpprof
TD         1024
SOLVENT   DMSO
NS         16
DS         4
SFO1      7786.162 Hz
FIDRES    7.60527 Hz
AQ         0.0658950 sec
RG         20642.5
DM         64.200 usec
DE         8.00 usec
TE         298.0 K
CNST2     145.0000000
d0         0.0000300 sec
d1         1.5000000 sec
d2         0.00344608 sec
d12        0.0002000 sec
d13        0.0000400 sec
d16        0.0002000 sec
DELTA1    0.0022408 sec
IN0        0.00001440 sec
MCREST    0.0000000 sec
MCKRR     1.5000000 sec

***** CHANNEL f1 *****
NUC1       1H
P1         10.00 usec
P2         21.20 usec
PL1        -5.00 dB
PL2        -5.00 dB
SF01       600.1336008 MHz

***** CHANNEL f2 *****
CPDPRG2   gpg
NUC2       13C
P3         15.00 usec
P4         70.00 usec
PL3        -6.00 dB
PL4        7.40 dB
SF02       150.9194083 MHz

***** GRADIENT CHANNEL *****
GPNAM1    SINE.100
GPNAM2    SINE.100
GPNAM3    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPX3      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPY3      0.00 %
GPZ1      50.00 %
GPZ2      30.00 %
GPZ3      40.10 %
P16       1000.00 usec

F1 - Acquisition parameters
ND0        2
TD         256
SF01       150.9194 MHz
FIDRES    135.633602 Hz
SN         230.071 cpm
FMODE     OF

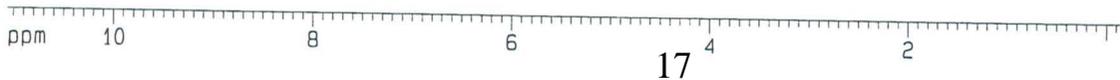
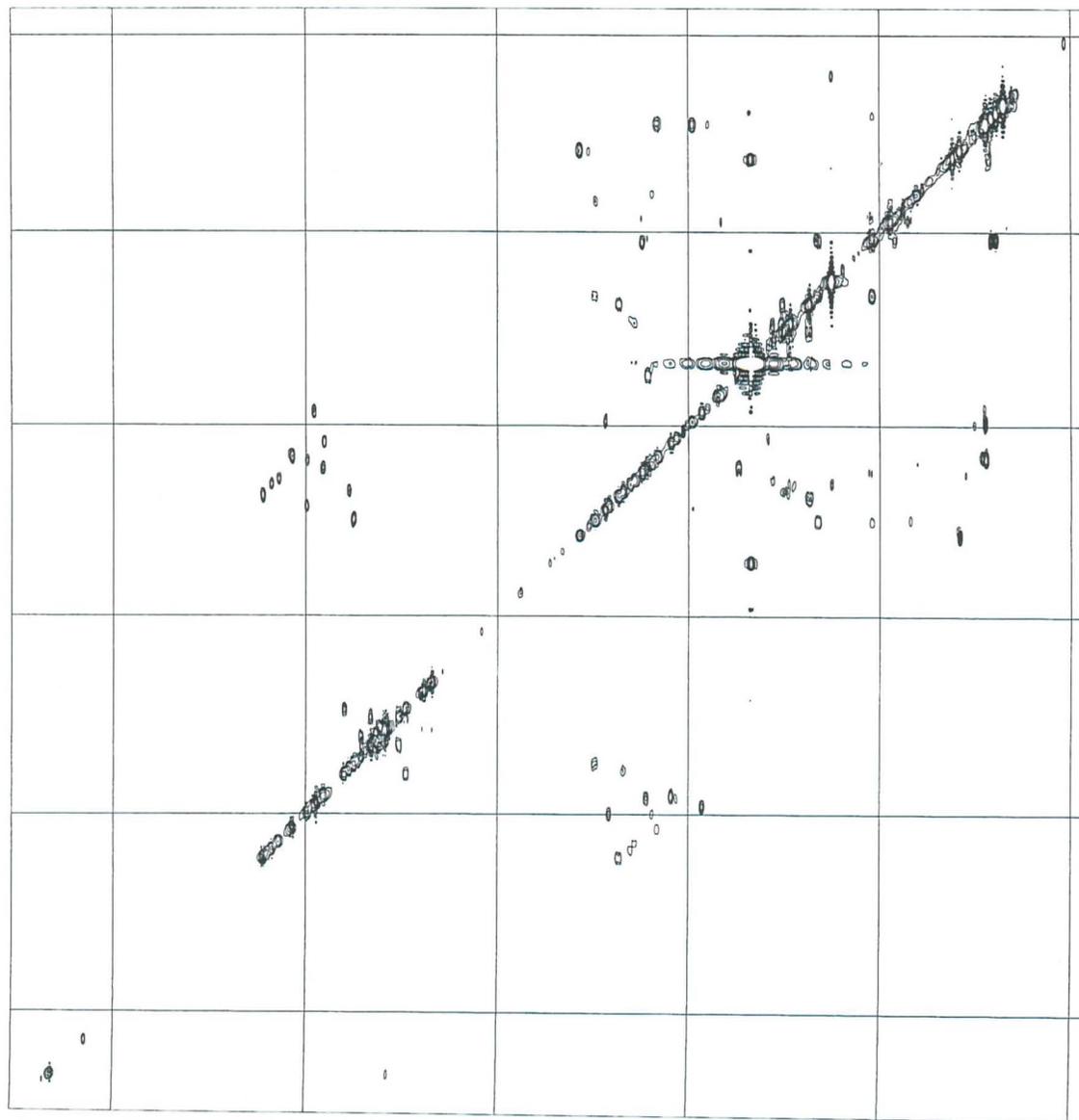
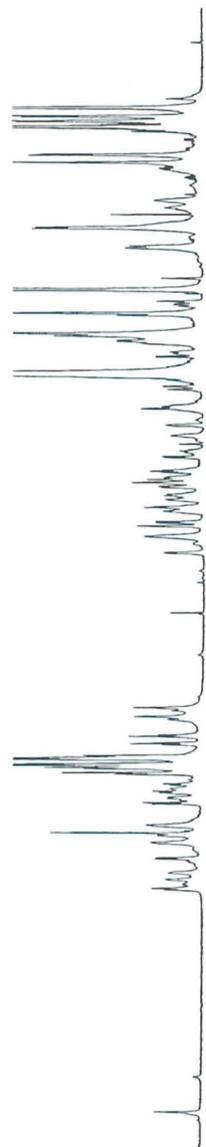
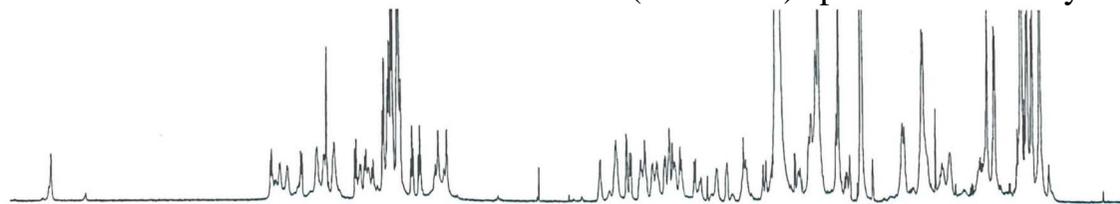
F2 - Processing parameters
SI         1024
SF         600.130063 MHz
WDW        SINE
SSB        1
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MC2        OF
SF         150.9028748 MHz
WDW        SINE
SSB        1
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        20.00 cm
CX1         15.00 cm
F2P1LO     10.679 ppm
F2LO       6408.53 Hz
F2P1HI     -0.284 ppm
F2HI       -170.34 Hz
F1P1LO     211.806 ppm
F1LO       31962.09 Hz
F1P1HI     -3.236 ppm
F1HI       -480.27 Hz
F2PPMCH    0.54612 ppm/cm
F2HZCM     328.94336 Hz/cm
F1PPMCH    14.33609 ppm/cm
F1HZCM     2163.25718 Hz/cm
    
```

# S10. COSY (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6

/COSY



```

Current Data Parameters
NAME      jul07-snu-kh
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20040707
Time      19.30
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   cosygpdf
TD        2048
SOLVENT   DMSO
NS        15
DS        8
SWH       7788.162 Hz
FIDRES    3.802814 Hz
AQ        0.1315958 sec
RG        64
DM        64.200 usec
DE        6.00 usec
TE        298.0 K
d0        0.0000000 sec
d1        1.48689198 sec
d13       0.0000400 sec
d16       0.0002000 sec
INDO     0.00012818 sec
MCREST    0.0000000 sec
MCWRK     1.48689198 sec

----- CHANNEL f1 -----
NUC1      1H
PQ        10.60 usec
P1        10.60 usec
PL1       -5.00 dB
SFO1      600.1336008 MHz

----- GRADIENT CHANNEL -----
GPNAM1    SINE.100
GPNAM2    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPZ1      10.00 %
GPZ2      10.00 %
P15       1000.00 usec

F1 - Acquisition parameters
NDO       1
TD        128
SFO1      600.1336 MHz
FIDRES    60.951824 Hz
SW        13.000 ppm
FHM00E    GF

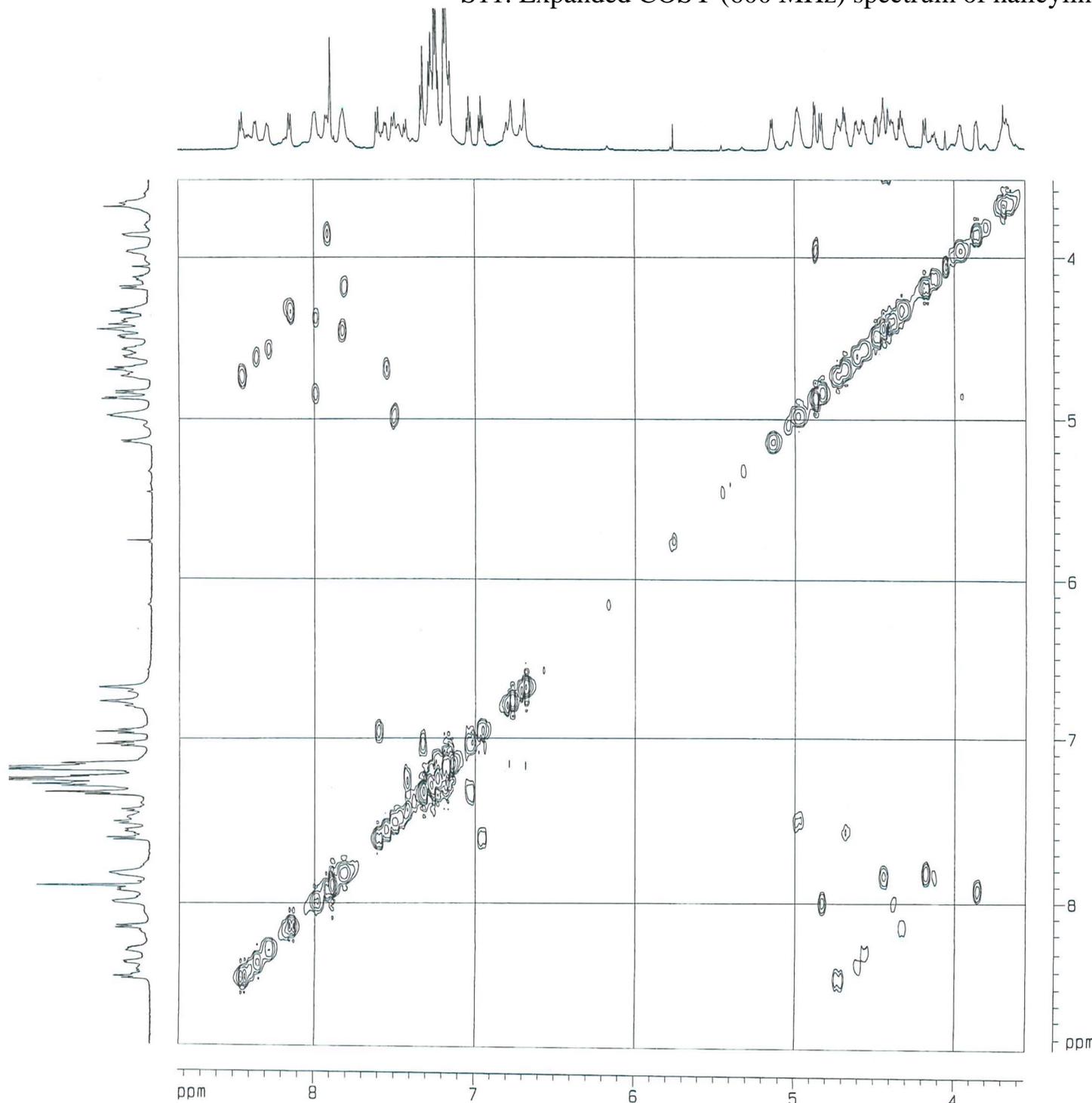
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SI        1024
SF        600.1300063 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
MC2       GF
SF        600.1300063 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     11.059 ppm
F2L0      6636.70 Hz
F2PHI     -0.233 ppm
F2HI      -139.91 Hz
F1PLO     11.042 ppm
F1L0      6626.80 Hz
F1PHI     -0.269 ppm
F1HI      -161.71 Hz
F2PPHCM   0.75279 ppm/cm
F2HZCM    451.77423 Hz/cm
F1PPHCM   0.75412 ppm/cm
F1HZCM    452.56729 Hz/cm
    
```

17

# S11. Expanded COSY (600 MHz) spectrum of halicyclindramide F (1) in DMSO-d6



/COSY

Current Data Parameters  
 NAME jul07-snu-kh  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20040707  
 Time 19.30  
 INSTRUM spect  
 PROBD 5 mm CPTXI 1H/  
 PULPROG cosyppaf  
 TO 2048  
 SOLVENT DMSO  
 NS 16  
 DS 8  
 SWH 7788.162 Hz  
 FIDRES 3.602914 Hz  
 AQ 0.1315959 sec  
 RG 64  
 DW 64.200 usec  
 DE 6.00 usec  
 TE 298.0 K  
 d0 0.0000300 sec  
 D1 1.46689198 sec  
 d13 0.0000400 sec  
 D16 0.0002000 sec  
 INO 0.00012818 sec  
 MCREST 0.0000000 sec  
 MCWPK 1.46689198 sec

----- CHANNEL f1 -----  
 NUC1 1H  
 P0 10.60 usec  
 P1 10.60 usec  
 PL1 -5.00 dB  
 SFO1 600.1336008 MHz

===== GRADIENT CHANNEL =====  
 GPNAM1 SINE.100  
 GPNAM2 SINE.100  
 GPX1 0.00 %  
 GPX2 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 GPZ1 10.00 %  
 GPZ2 10.00 %  
 P16 1000.00 usec

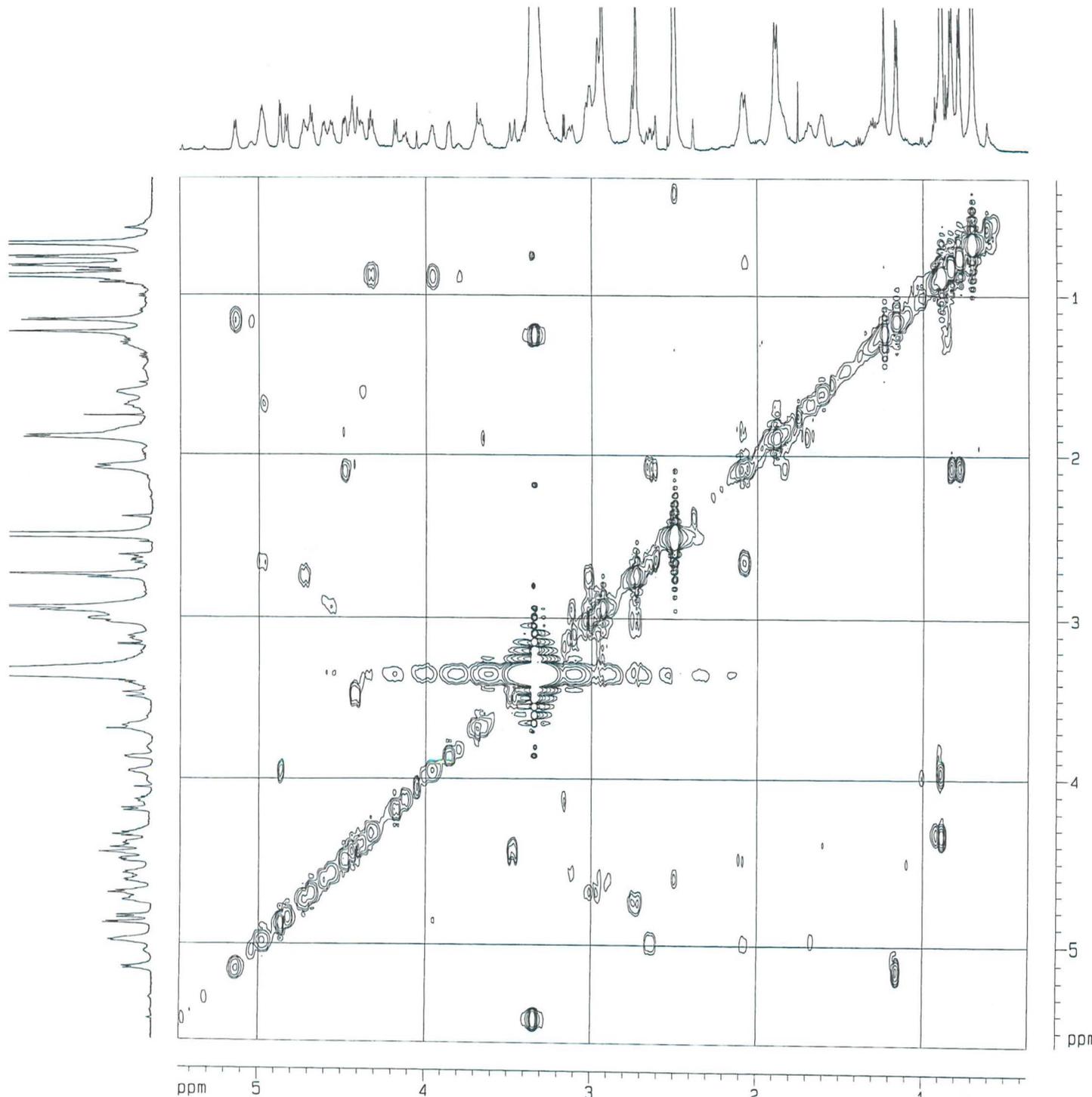
F1 - Acquisition parameters  
 NDD 1  
 TO 128  
 SFO1 600.1336 MHz  
 FIDRES 60.951824 Hz  
 SH 13.000 ppm  
 FmMODE DF

F2 - Processing parameters  
 SI 1024  
 SF 600.1300053 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 MC2 DF  
 SF 600.1300053 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 8.841 ppm  
 F2L0 5305.72 Hz  
 F2PHI 3.556 ppm  
 F2HI 2134.17 Hz  
 F1PLO 8.871 ppm  
 F1L0 5323.96 Hz  
 F1PHI 3.527 ppm  
 F1HI 2116.37 Hz  
 F2PDMC 0.35232 ppm/cm  
 F2HZCM 211.43643 Hz/cm  
 F1PDMC 0.35532 ppm/cm  
 F1HZCM 213.83931 Hz/cm

S12. Expanded COSY (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



/COSY

```

Current Data Parameters
NAME      ju107-snu-kh)
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20040707
Time      19.30
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPROG   cosygpgf
TD         2048
SOLVENT   DMSO
NS         16
DS         8
SWH        7788.162 Hz
FIDRES     3.802814 Hz
AQ         0.1315968 sec
RG         64
DW         64.200 usec
DE         6.00 usec
TE         298.0 K
d0         0.00000300 sec
d1         1.48689198 sec
d13        0.00000400 sec
d16        0.00020000 sec
IND        0.00012818 sec
MCREST     0.00000000 sec
MCKRST     1.48689198 sec

----- CHANNEL f1 -----
NUC1       1H
P0         10.60 usec
P1         10.60 usec
PL1        -5.00 dB
SF01       600.1336008 MHz

----- GRADIENT CHANNEL -----
GPNAM1     SINE.100
GPNAM2     SINE.100
GPX1       0.00 %
GPX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       10.00 %
GPZ2       10.00 %
P16        1000.00 usec

F1 - Acquisition parameters
ND0        1
TD         128
SF01       600.1336 MHz
FIDRES     60.951824 Hz
SW         13.000 ppm
FMODE      GF

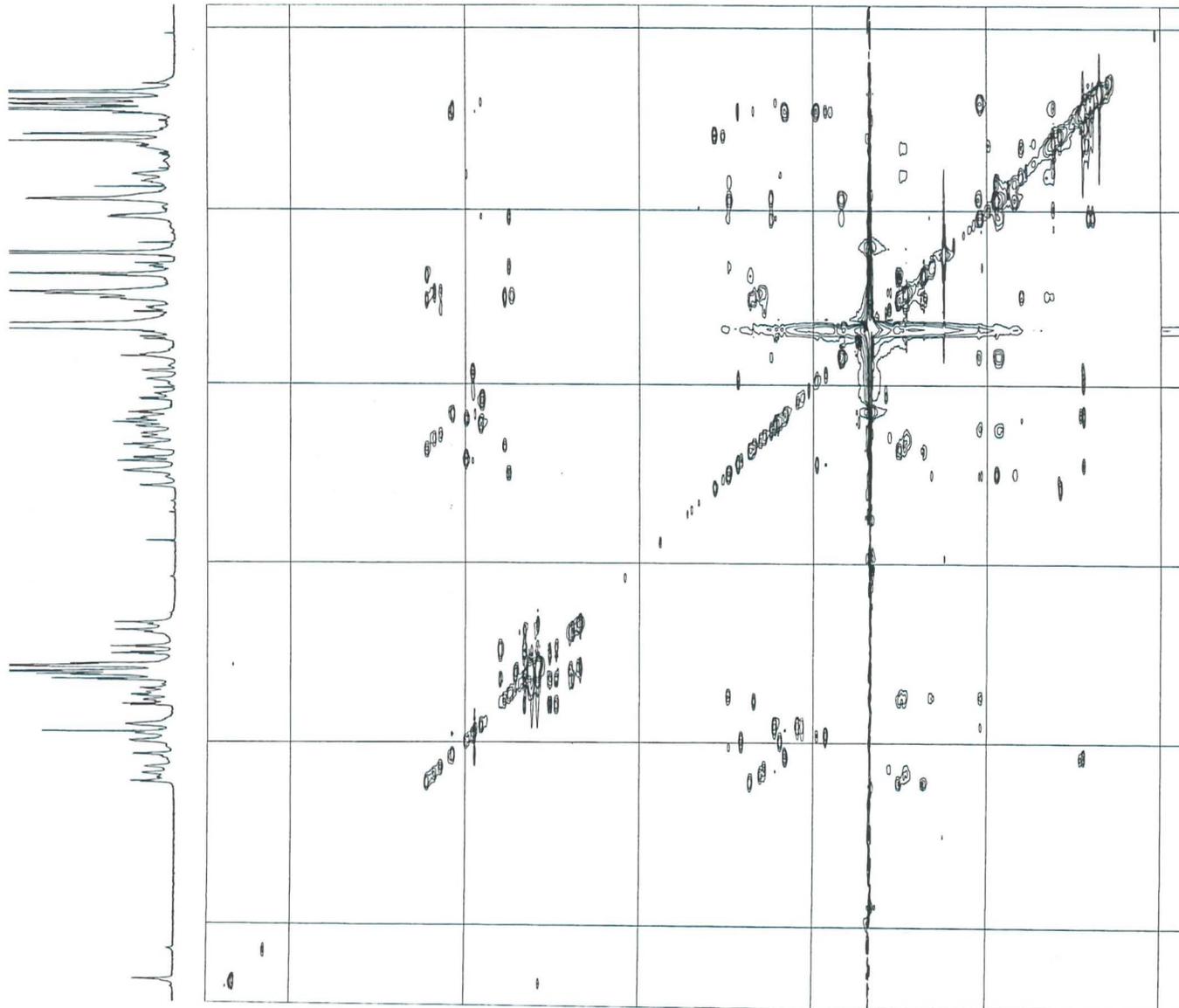
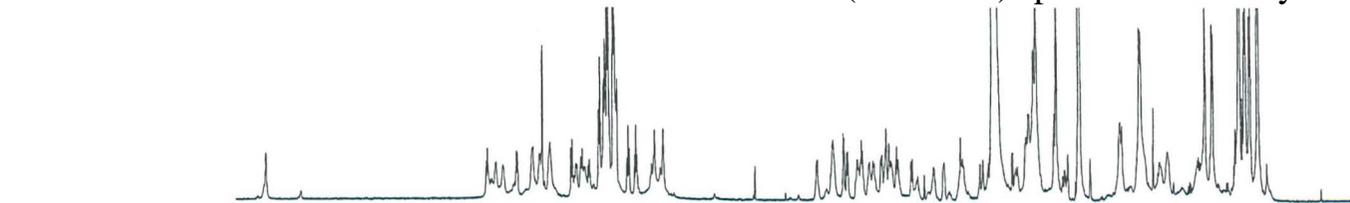
F2 - Processing parameters
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SF         600.1300063 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MC2        GF
SF         600.1300063 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      5.470 ppm
F2LO       3282.62 Hz
F2PHI      0.363 ppm
F2HI       217.55 Hz
F2PLD      5.583 ppm
F1LO       3350.64 Hz
F1PHI      0.315 ppm
F1HI       188.77 Hz
F2PPMCM    0.34049 ppm/cm
F2HZCM     204.33784 Hz/cm
F1PPMCM    0.35124 ppm/cm
F1HZCM     210.79172 Hz/cm
    
```

S13. TOCSY (600 MHz) spectrum of halicyclindramide F (1) in DMSO-d6

6/TOCSY



0  
2  
4  
6  
8  
10  
ppm

```

Current Data Parameters
NAME      10107-gnu-4h
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_    20040701
Time     22.52
INSTRUM  spect
PROBHD   5 mm CPXI 1H/
PULPROG  mlevupg
TD        2048
SOLVENT  DMSO
NS        16
DS        16
SWH       7788.162 Hz
FIDRES   3.802814 Hz
AQ        0.1315958 sec
RG        203.2
Dw        64.200 usec
DE        6.00 usec
TE        298.0 K
d0        0.0000000 sec
D1        1.0000000 sec
D2        0.0800000 sec
d11       0.0300000 sec
D12       0.0000000 sec
D16       0.0000000 sec
DELTA     0.00118625 sec
DELTA1    0.00120900 sec
FACTOR1   6
IN0       0.00012818 sec
I1        36
MCREST    0.0000000 sec
MCWRR     0.00750000 sec
SCALEF    6
STICNT    64

***** CHANNEL f1 *****
NUC1      1H
P1        10.60 usec
P2        21.20 usec
P3        16.67 usec
P6        25.00 usec
P7        50.00 usec
P17       1500.00 usec
PL1       -5.00 dB
PL10      2.70 dB
SFO1      600.1336000 MHz

***** GRADIENT CHANNEL *****
GPNAM1    SINE 100
GPNAM2    SINE 100
GPK1      0.00 %
GPK2      0.00 %
GPK3      0.00 %
GPK4      0.00 %
GPK5      0.00 %
GPK6      30.00 %
GPK7      30.00 %
GPK8      1000.00 usec

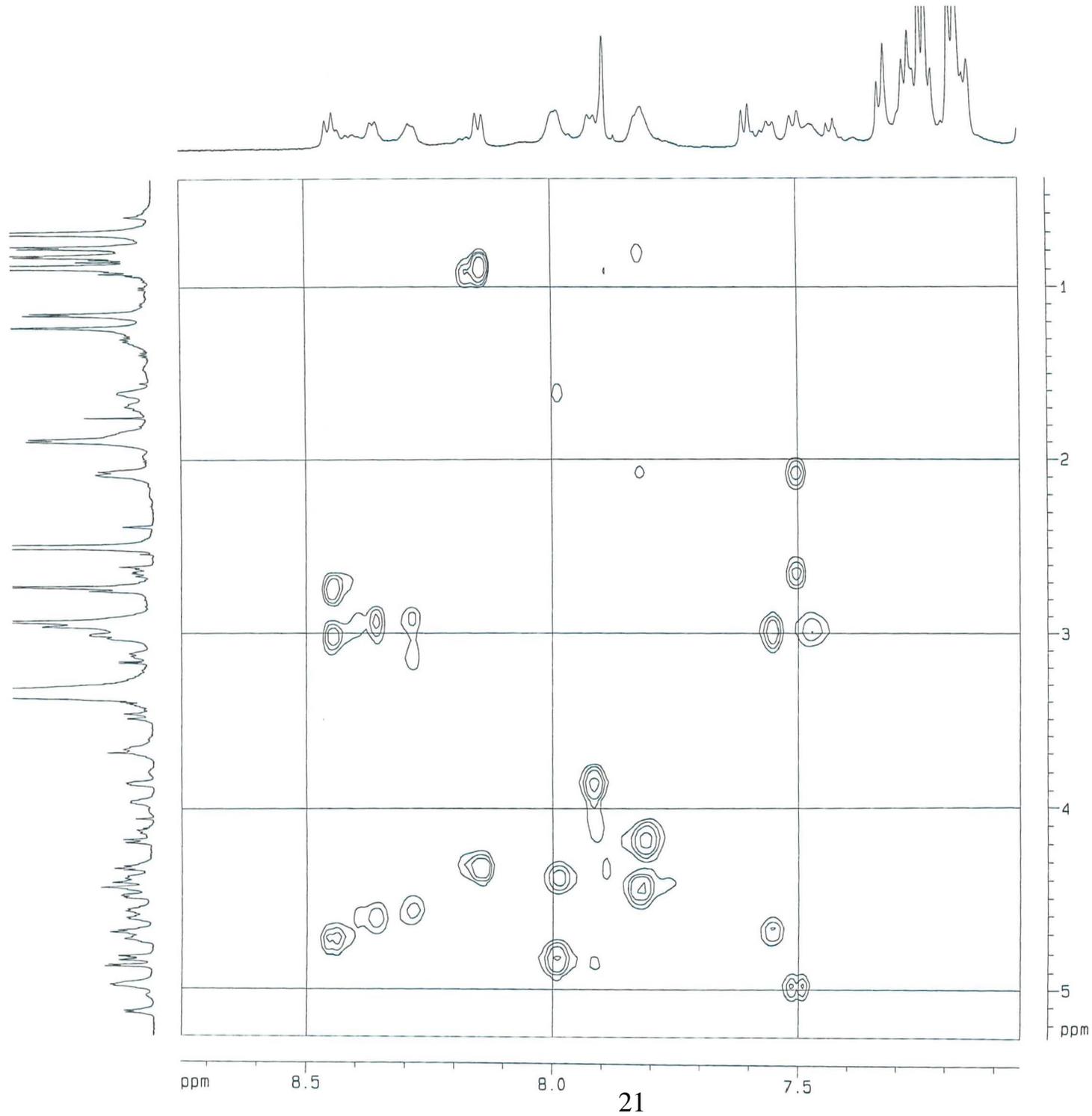
F1 - Acquisition parameters
ND0       1
TD         128
SFO1      600.1336 MHz
FIDRES    60.351824 Hz
SW         13.000 ppm
FMODE     Echo-Antiecho

F2 - Processing parameters
SI         2048
SF         600.1300063 MHz
WDW        0SINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         256
MC2        echo-antjecho
SF         600.1300063 MHz
WDW        0SINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1         15.00 cm
F2PL0      10.951 ppm
F2L0       6572.05 Hz
F2PH1      -0.309 ppm
F2H1       -195.55 Hz
F1PL0      10.915 ppm
F1L0       6550.61 Hz
F1PH1      -0.257 ppm
F1H1       -154.09 Hz
F2PPMCM    0.75068 ppm/cm
F2HZCM     450.50665 Hz/cm
F1PPMCM    0.74481 ppm/cm
F1HZCM     446.98004 Hz/cm
    
```

S14. Expanded TOCSY (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/TOCSY

```

Current Data Parameters
NAME          ju107-snu-kh)
EXPNO         4
PROCNO        1

F2 - Acquisition Parameters
Date_         20040707
Time          22.52
INSTRUM       spect
PROBHD        5 mm CPTX1 1H/
PULPROG       mlevetgp
TO            2048
SOLVENT       DMSO
NS            16
DS            16
SMH           7788.162 Hz
FIDRES        3.802814 Hz
AQ            0.1315958 sec
RG            203.2
DW            64.200 usec
DE            6.00 usec
TE            299.0 K
d0            0.0000000 sec
d1            1.0000000 sec
d9            0.0500000 sec
d11           0.0300000 sec
d12           0.0000200 sec
d16           0.0000000 sec
DELTA         0.0015625 sec
DELTA1        0.0012000 sec
FACTOR1       6
IN0           0.00012818 sec
I1            36
MCREST        0.0000000 sec
MCMRK         0.00750000 sec
SCALEF        6
STICNT        64

***** CHANNEL f1 *****
NUC1          1H
P1            10.60 usec
p2            21.20 usec
p5            16.67 usec
p6            25.00 usec
p7            50.00 usec
P17           1500.00 usec
PL1           -5.00 dB
PL10          2.70 dB
SFO1          600.1336000 MHz

***** GRADIENT CHANNEL *****
GPNAM1        SINE.100
GPNAM2        SINE.100
GPK1          0.00 X
GPK2          0.00 X
GPY1          0.00 X
GPY2          0.00 X
GPT1          30.00 X
GPT2          30.00 X
P16           1000.00 usec

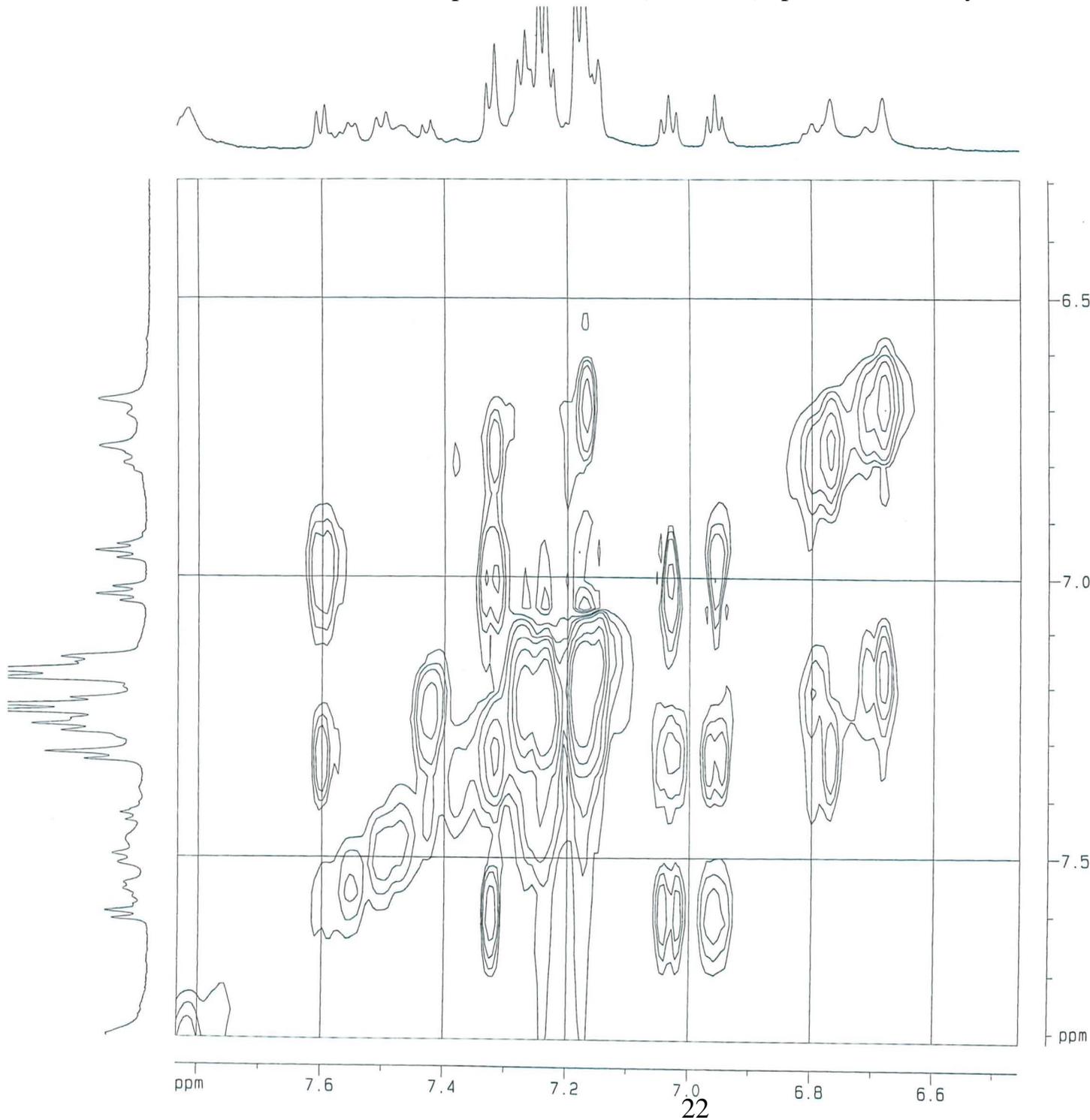
F1 - Acquisition parameters
NOF           1
TO            120
SFO1          600.1336 MHz
FIDRES        60.951824 Hz
SW            13.000 ppm
FMODE         Echo-Antiecho

F2 - Processing parameters
SI            2048
SF            600.130063 MHz
WDW           GSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00

F1 - Processing parameters
SI            256
MC2           echo-antiecho
SF            600.130063 MHz
WDW           GSINE
SSB           2
LB            0.00 Hz
GB            0

20 NMR plot parameters
CX2           15.00 cm
CX1           15.00 cm
FPLD          8.752 ppm
FZLO          5252.48 Hz
F2PH1         7.046 ppm
F2PH2         4829.52 Hz
F1PLD         5.278 ppm
F1LO          3167.79 Hz
F1PH1         0.403 ppm
F1H1          242.10 Hz
F2PPMCH       0.11364 ppm/cm
F2HZCM        68.19713 Hz/cm
F1PPMCH       0.32501 ppm/cm
F1HZCM        195.04584 Hz/cm
    
```

S15. Expanded TOCSY (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/TOCSY

```

Current Data Parameters
NAME      jul107-sru-4h1
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20040707
Time      22:52
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPROG   m1evetgp
TD         2048
SOLVENT   DMSO
NS         16
DS         16
SWH        7788.162 Hz
FIDRES     3.802814 Hz
AQ         0.1315958 sec
RG         203.2
DM         64.200 usec
DE         8.00 usec
TE         298.0 K
D0         0.0000300 sec
D1         1.0000000 sec
D9         0.0600000 sec
d11        0.0300000 sec
D12        0.0002000 sec
D18        0.0002000 sec
DELTA     0.00119625 sec
DELTA1    0.00128800 sec
FACTOR1   6
INO        0.00012818 sec
J1         30
MCREST    0.0000000 sec
MCHWR     0.00750000 sec
SCALEF    6
STICENT   64

***** CHANNEL f1 *****
NUC1      1H
P1         10.60 usec
P2         21.20 usec
P3         16.67 usec
P6         25.00 usec
P7         50.00 usec
P17        1500.00 usec
PL1        -5.00 dB
PL10       2.70 dB
SFO1       600.1336008 MHz

***** GRADIENT CHANNEL *****
GPNAM1    SINE.100
GPNAM2    SINE.100
GPR1      0.00 %
GPR2      0.00 %
GPR3      0.00 %
GPR4      0.00 %
GPR5      30.00 %
GPR6      30.00 %
GPR7      30.00 %
GPR8      1000.00 usec

F1 - Acquisition parameters
TD         1
FIDRES     60.951824 Hz
SW         13.000 ppm
FNMDOE     Echo-Antiecho

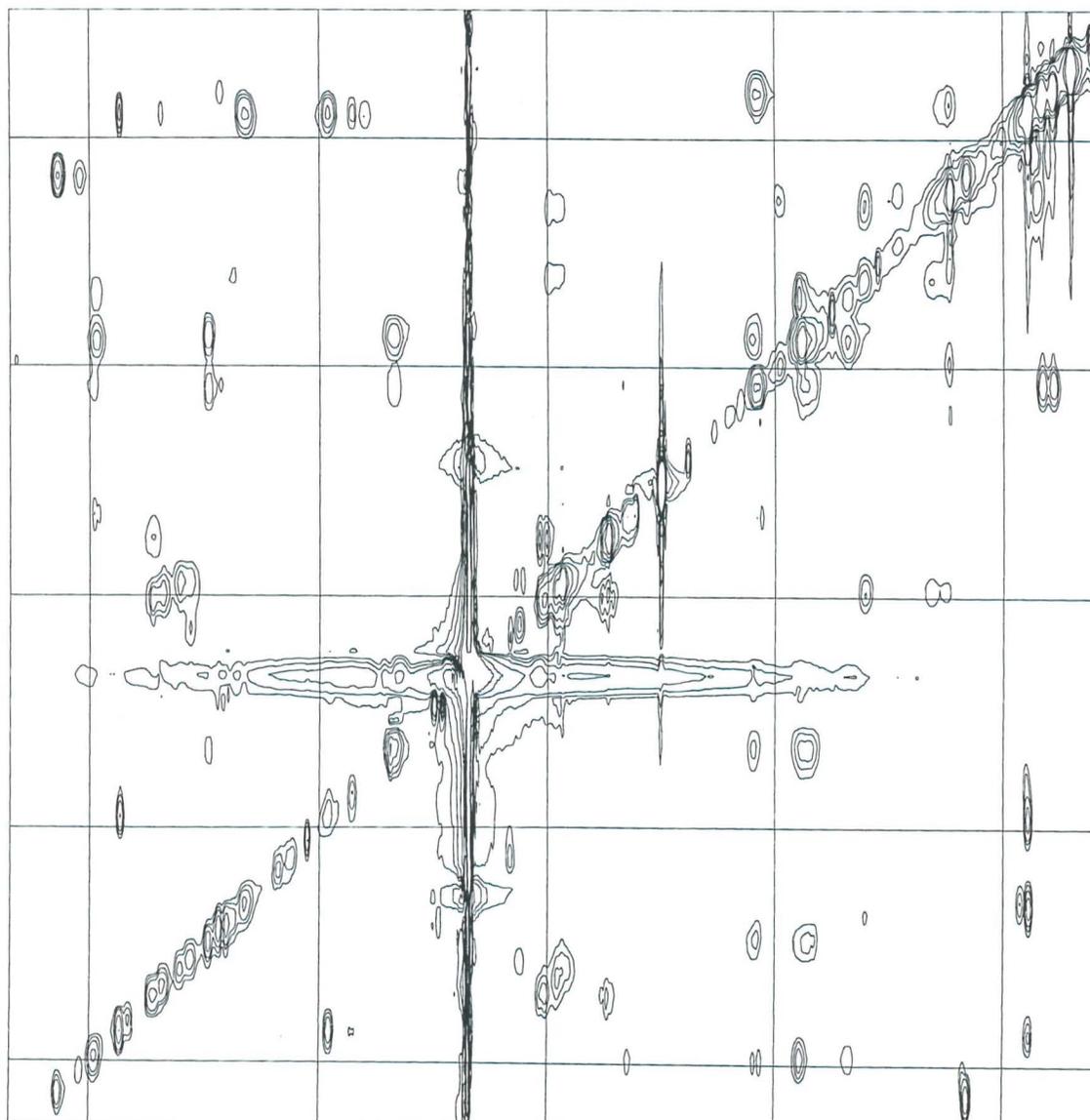
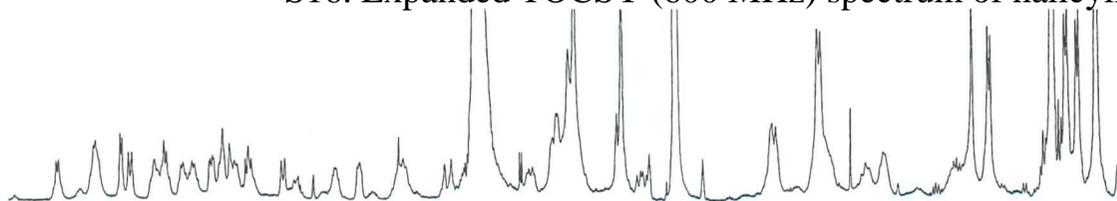
F2 - Processing parameters
SI         2048
SF         600.1300063 MHz
WDW        0SINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         256
MC2        echo-antiecho
SF         600.1300063 MHz
WDW        0SINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
FAPLO      7.833 ppm
F2L0       4701.07 Hz
F2PH1      6.458 ppm
F2HI       3875.86 Hz
F1PLO      7.818 ppm
F1L0       4691.58 Hz
F1PH1      3777.30 Hz
F1HI       3777.30 Hz
F2PPMCM    0.09167 ppm/cm
F2HZCM     55.01403 Hz/cm
F1PPMCM    0.10156 ppm/cm
F1HZCM     60.95183 Hz/cm
    
```

S16. Expanded TOCSY (600 MHz) spectrum of halicyclindramide F (1) in DMSO-d6

6/TOCSY



ppm 5 4 3 2 1

1  
2  
3  
4  
5  
ppm

```

Current Data Parameters
NAME      ju107-sru-kh
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20040707
Time      22.52
INSTRUM   spect
PROBHD    5 mm CP1X1 1H/
PULPROG   mjevetop
TD         2048
SOLVENT   DMSO
NS         16
DS         16
SWH        7786.162 Hz
FIDRES     3.802814 Hz
AQ         0.1315596 sec
RG         203.2
DW         64.200 usec
DE         6.00 usec
TE         298.0 K
d0         0.0000000 sec
d1         1.0000000 sec
d2         0.0600000 sec
d11        0.0300000 sec
d12        0.0002000 sec
d15        0.0002000 sec
DELTA     0.0019625 sec
DELTA1    0.0012080 sec
FACTOR1    6
IN0        0.0012819 sec
I1         36
MCREST    0.0000000 sec
MCWRK     0.0075000 sec
SCALEF    6
ST1CNT    64

***** CHANNEL f1 *****
NUC1       1H
P1         10.60 usec
p2         21.20 usec
p5         16.67 usec
p6         25.00 usec
p7         50.00 usec
P17        1500.00 usec
PL1        -5.00 dB
PL10       2.70 dB
SFO1       600.1336008 MHz

***** GRADIENT CHANNEL *****
GPRAM1     SINE.100
GPRAM2     SINE.100
GPX1       0.00 %
GPX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPI1       30.00 %
GPI2       30.00 %
GPI3       30.00 %
P16        1000.00 usec

F1 - Acquisition parameters
NOF        1
TD         128
SFO1       600.1336 MHz
FIDRES     60.321624 Hz
SK         13.000 dBm
FMODE      Echo-Antiecho

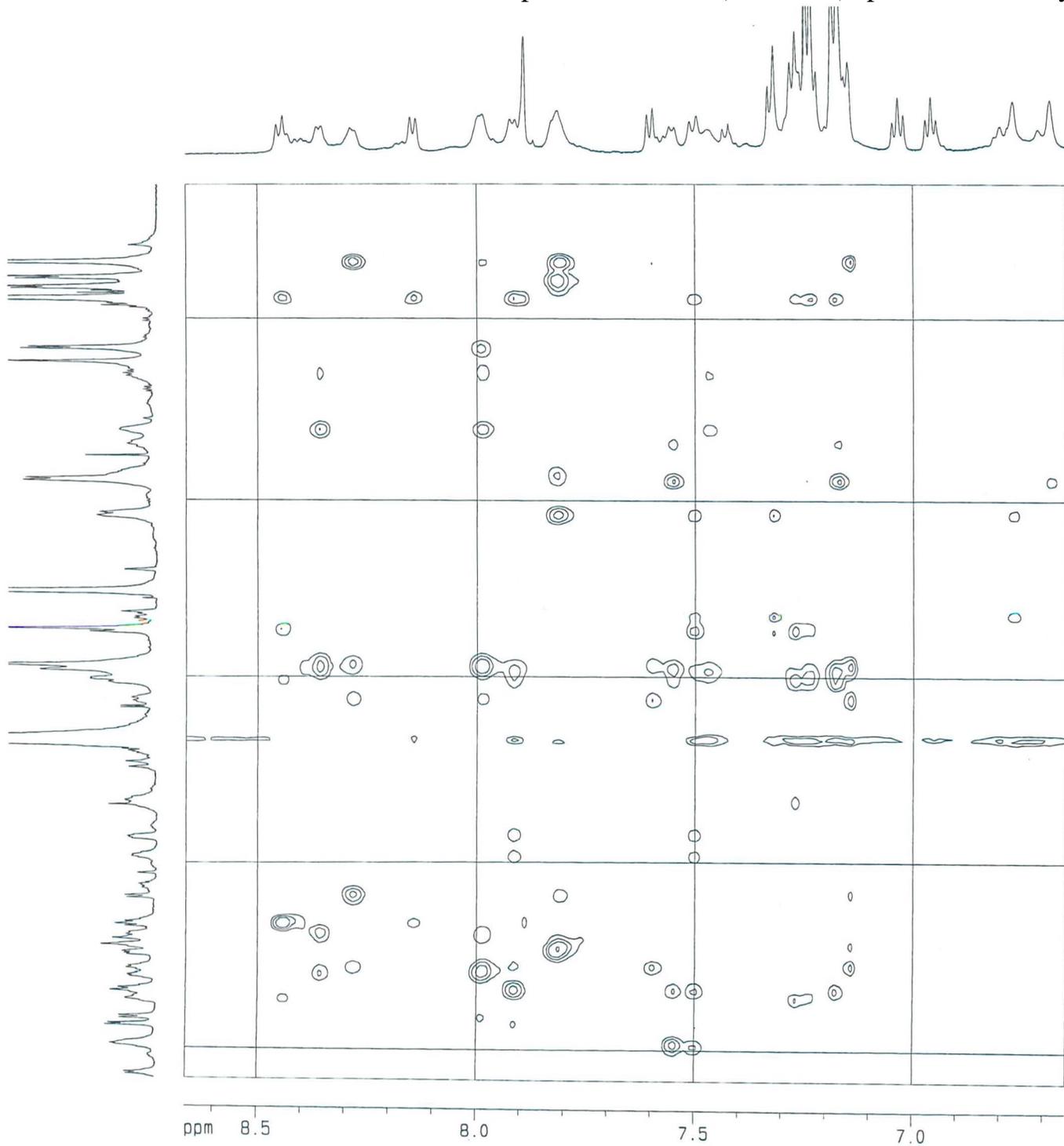
F2 - Processing parameters
SI         2048
SF         600.1300063 MHz
WDW        GSTINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         256
MC2        echo-antiecho
SF         600.1300063 MHz
WDW        GSTINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2P0       5.349 dBm
F2L0       3210.36 Hz
F2PHI      0.559 dBm
F2HT       335.44 Hz
F2PL0     5.278 dBm
F2L0       3167.79 Hz
F2PHI      0.454 dBm
F2HT       272.57 Hz
F2PPMCM    0.31937 ppm/cm
F2HZCM     193.66179 Hz/cm
F2PPMCM    0.32162 dBm/cm
F2HZCM     193.01411 Hz/cm
    
```



S18. Expanded NOESY (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/NOESY

```

Current Data Parameters
NAME      ju107-snu-knj
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20040707
Time      20.26
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   noesyph
TD         2048
SOLVENT   DMSO
NS         16
DS         4
SWH        7788.162 Hz
FIDRES     3.802814 Hz
AQ         0.1315958 sec
RG         25.4
DW         64.200 usec
DE         6.00 usec
TE         298.0 K
d0         0.00005059 sec
O1         1.00000000 sec
O2         1.00000000 sec
INO        0.00012818 sec
MCREST     0.00000000 sec
MCMARK     0.50000000 sec
ST1CNT     128

***** CHANNEL f1 *****
NUC1       1H
P1         10.60 usec
PL1        -5.00 dB
SF01       600.1336008 MHz

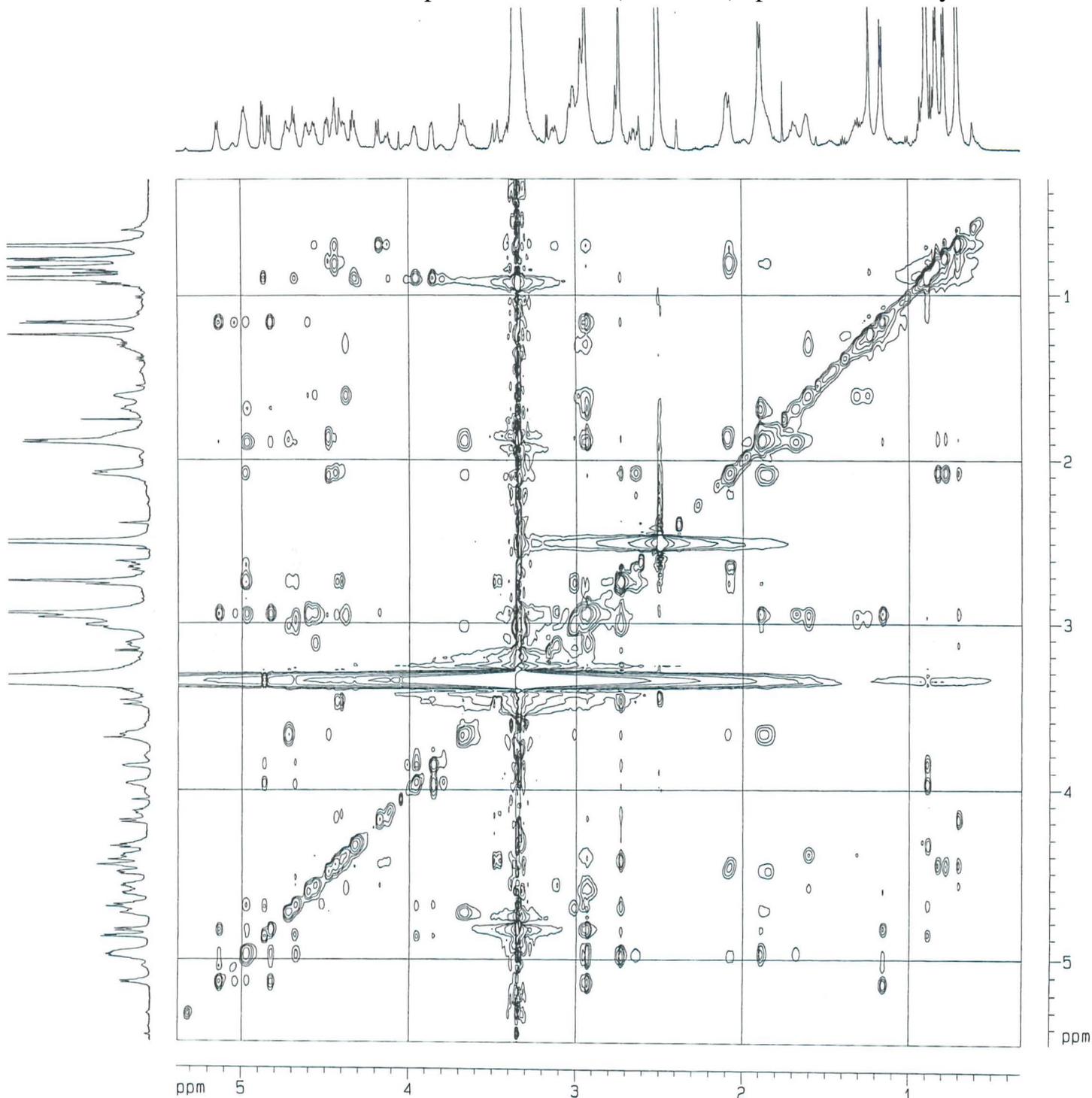
F1 - Acquisition parameters
ND0        1
TD         256
SF01       600.1336 MHz
FIDRES     30.475912 Hz
SW         13.000 ppm
F1MODE     States-TPPI

F2 - Processing parameters
SI         1024
SF         600.1300063 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300063 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      8.664 ppm
F2LO       5199.24 Hz
F2PHI      6.648 ppm
F2HI       3989.94 Hz
F1PLO      5.164 ppm
F1LO       3099.22 Hz
F1PHI      0.289 ppm
F1HI       173.53 Hz
F2PPMCM    0.13434 ppm/cm
F2HZCM     80.61965 Hz/cm
F1PPMCM    0.32501 ppm/cm
F1HZCM     195.04584 Hz/cm
    
```

S19. Expanded NOESY (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/NOESY

```

Current Data Parameters
NAME      ju107-snu-khj
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20040707
Time      20.26
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   noesyph
TD         2048
SOLVENT   DMSO
NS         16
DS         4
SWH        7788.162 Hz
FIDRES     3.802814 Hz
AQ         0.1315958 sec
RG         25.4
DW         64.200 usec
DE         6.00 usec
TE         298.0 K
d0         0.00005059 sec
d1         1.00000000 sec
d2         1.00000000 sec
DB         0.0012818 sec
INO        0.00000000 sec
MCREST    0.00000000 sec
MCWARR    0.50000000 sec
ST1CNT    128

----- CHANNEL f1 -----
NUC1      1H
P1        10.60 usec
PL1       -5.00 dB
SFO1      600.1336008 MHz

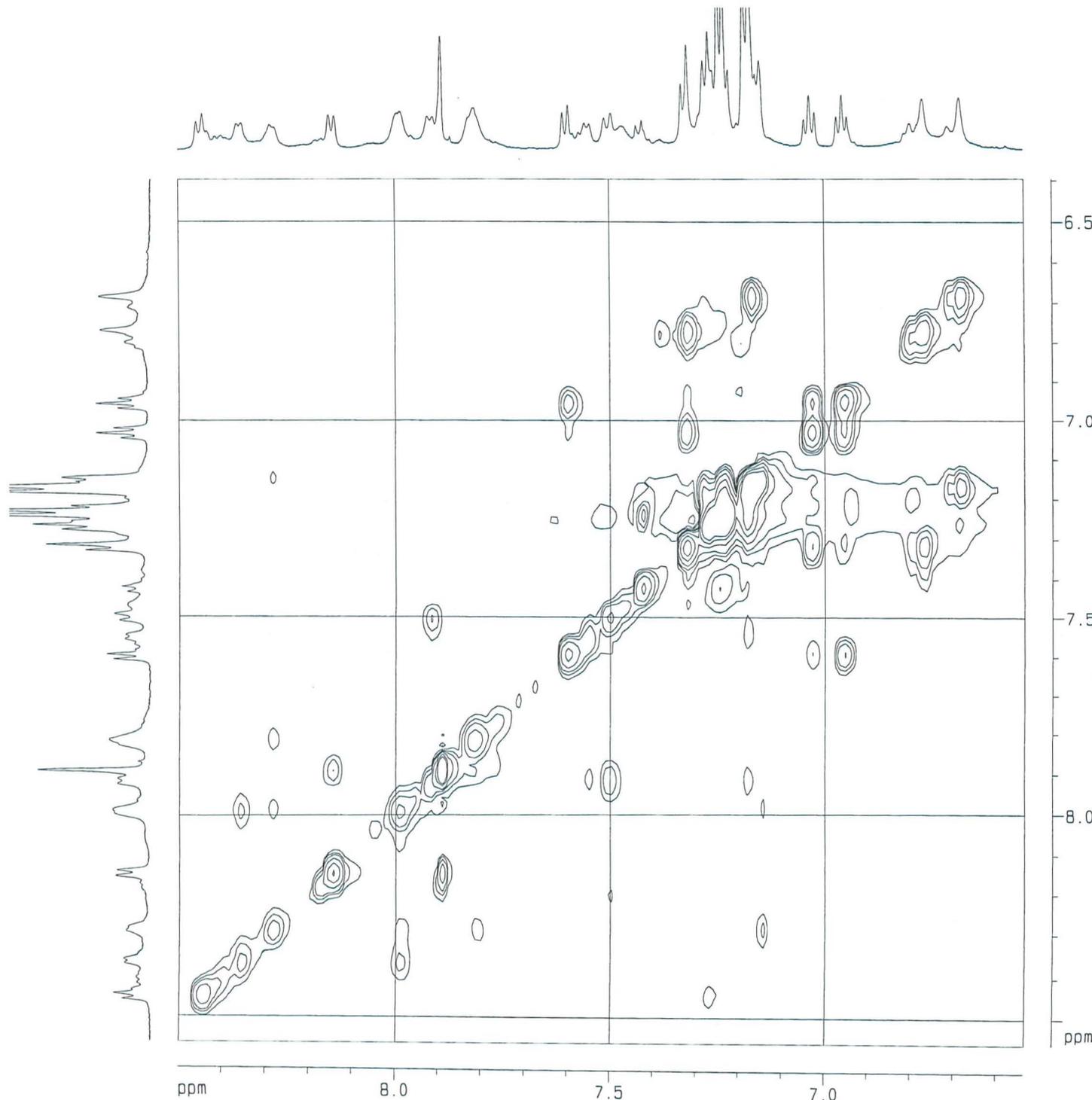
F1 - Acquisition parameters
ND0       1
TD        256
SFO1      600.1336 MHz
FIDRES     30.475912 Hz
SW         13.000 ppm
FnMODE    States-TPPI

F2 - Processing parameters
SI        1024
SF        600.1300063 MHz
WDW       QSINE
SSB       2
LB        0.00 Hz
GB        0
PC        1.00

F1 - Processing parameters
SI        1024
MC2       States-TPPI
SF        600.1300063 MHz
WDW       QSINE
SSB       2
LB        0.00 Hz
GB        0

20 NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     5.381 ppm
F2LO      3229.38 Hz
F2PHI     0.324 ppm
F2HI      194.73 Hz
F1PLO     5.494 ppm
F1LO      3297.31 Hz
F1PHI     0.315 ppm
F1HI      188.77 Hz
F2PPMCM   0.33711 ppm/cm
F2HZCM    202.30966 Hz/cm
F1PPMCM   0.34532 ppm/cm
F1HZCM    207.23619 Hz/cm
    
```

S20. Expanded NOESY (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/NOESY

```

Current Data Parameters
NAME      jul07-snu-khj
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20040707
Time      20.26
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   noesyph
TD         2048
SOLVENT   DMSO
NS         16
DS         4
SMH        7788.162 Hz
FIDRES     3.802814 Hz
AQ         0.1315958 sec
RG         25.4
DW         64.200 usec
DE         6.00 usec
TE         298.0 K
d0         0.00005059 sec
d1         1.00000000 sec
d8         1.00000000 sec
IN0        0.00012818 sec
MCREST     0.00000000 sec
MCWRK     0.50000000 sec
ST1CNT     128

===== CHANNEL f1 =====
NUC1       1H
P1         10.60 usec
PL1        -5.00 dB
SFO1       600.1336008 MHz

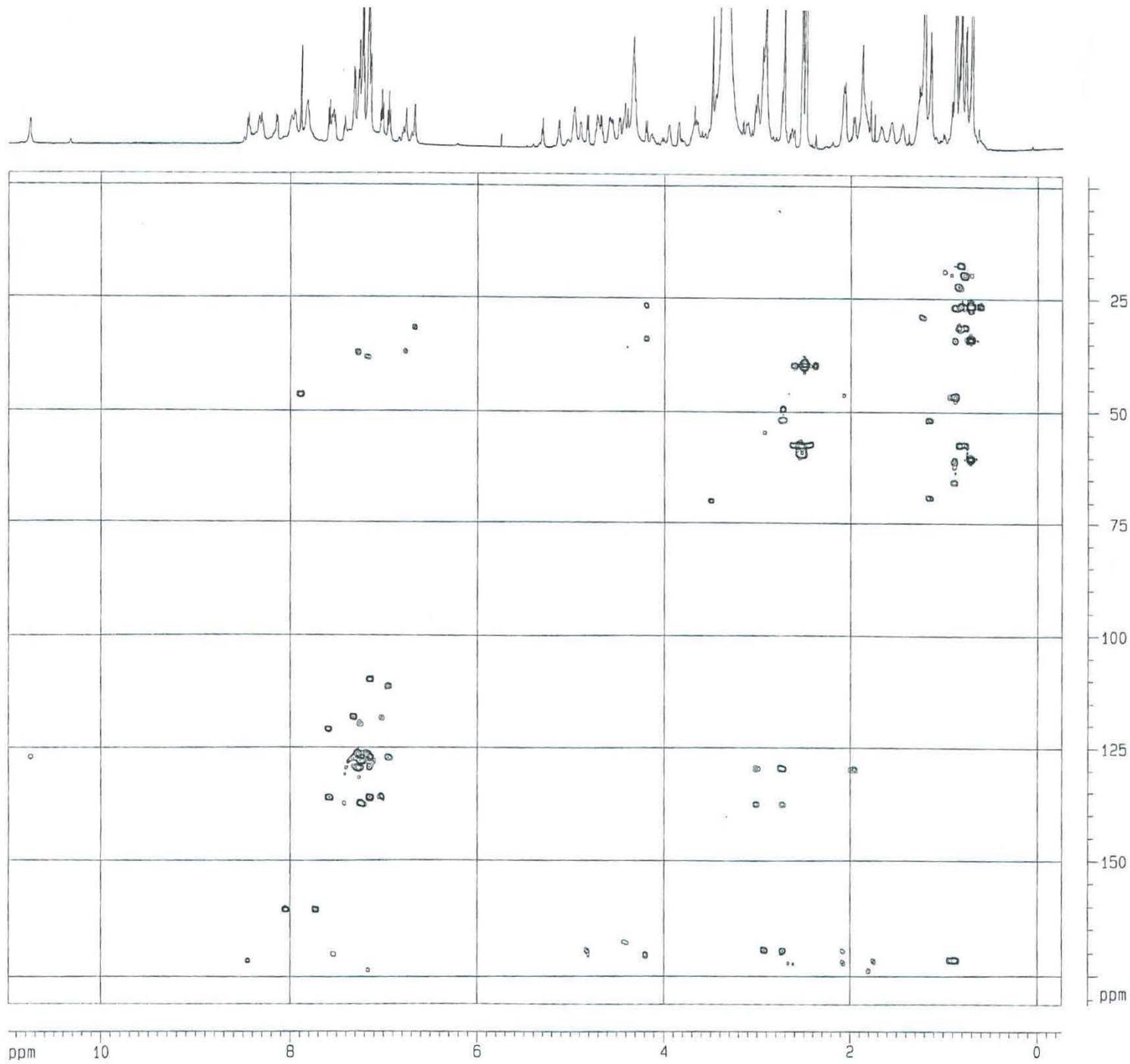
F1 - Acquisition parameters
ND0        1
TD         256
SFO1       600.1336 MHz
FIDRES     30.475912 Hz
SW         13.000 ppm
FnMODE     States-TPPI

F2 - Processing parameters
SI         1024
SF         600.1300063 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300063 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      8.499 ppm
F2LO       5100.36 Hz
F2PHI      6.534 ppm
F2HI       3921.49 Hz
F1PLO      8.567 ppm
F1LO       5141.10 Hz
F1PHI      6.396 ppm
F1HI       3838.26 Hz
F2PPMCM    0.13096 ppm/cm
F2HZCM     78.59148 Hz/cm
F1PPMCM    0.14473 ppm/cm
F1HZCM     86.85635 Hz/cm
    
```

S21. HMBC (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/HMBC

Current Data Parameters  
 NAME jul19-nu-41  
 EXPNO 6  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080720  
 Time 1:30  
 INSTRUM spect  
 PULPROG zgpg30  
 POLARIZ none  
 TD 1224  
 SOLVENT DMSO  
 NS 32  
 DS 4  
 SWH 7780.162 Hz  
 FIDRES 7.505527 Hz  
 AQ 0.0208550 sec  
 RG 20482.5  
 DW 84.250 usec  
 DE 0.00 usec  
 TE 298.0 K  
 CH212 140.500000  
 CH213 16.000000  
 D0 0.0000000 sec  
 D1 1.5000000 sec  
 D2 0.00344928 sec  
 D3 0.0000000 sec  
 C16 0.0000000 sec  
 INO 0.00001440 sec  
 HRCALC 0.0000000 sec  
 HMRK 1.5000000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 1H  
 P1 10.70 usec  
 PL1 21.40 dB  
 PL2 -5.00 dB  
 SF01 600.130608 MHz

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 NUC2 13C  
 P3 15.00 usec  
 PL3 -8.00 dB  
 SF02 100.6254983 MHz

\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*  
 GPNAM1 SINE 100  
 GPNAM2 SINE 100  
 GPNAM3 SINE 100  
 GPR1 0.00 %  
 GPR2 0.00 %  
 GPR3 0.00 %  
 GPR4 0.00 %  
 GPR5 0.00 %  
 GPR6 50.00 %  
 GPR7 30.00 %  
 GPR8 40.00 %  
 P16 1000.00 usec

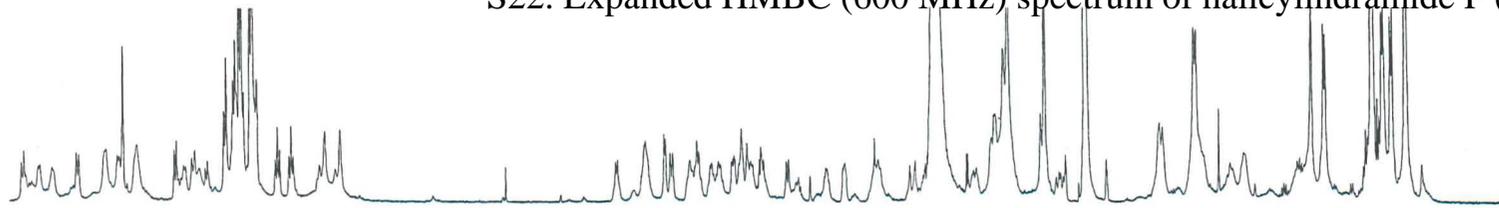
F1 - Acquisition parameters  
 NS 2  
 TD 256  
 SF01 150.9194 MHz  
 FIDRES 120.633682 Hz  
 SW 230.571 ppm  
 FWHM 67

F2 - Processing parameters  
 SI 1024  
 SF 600.1306081 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

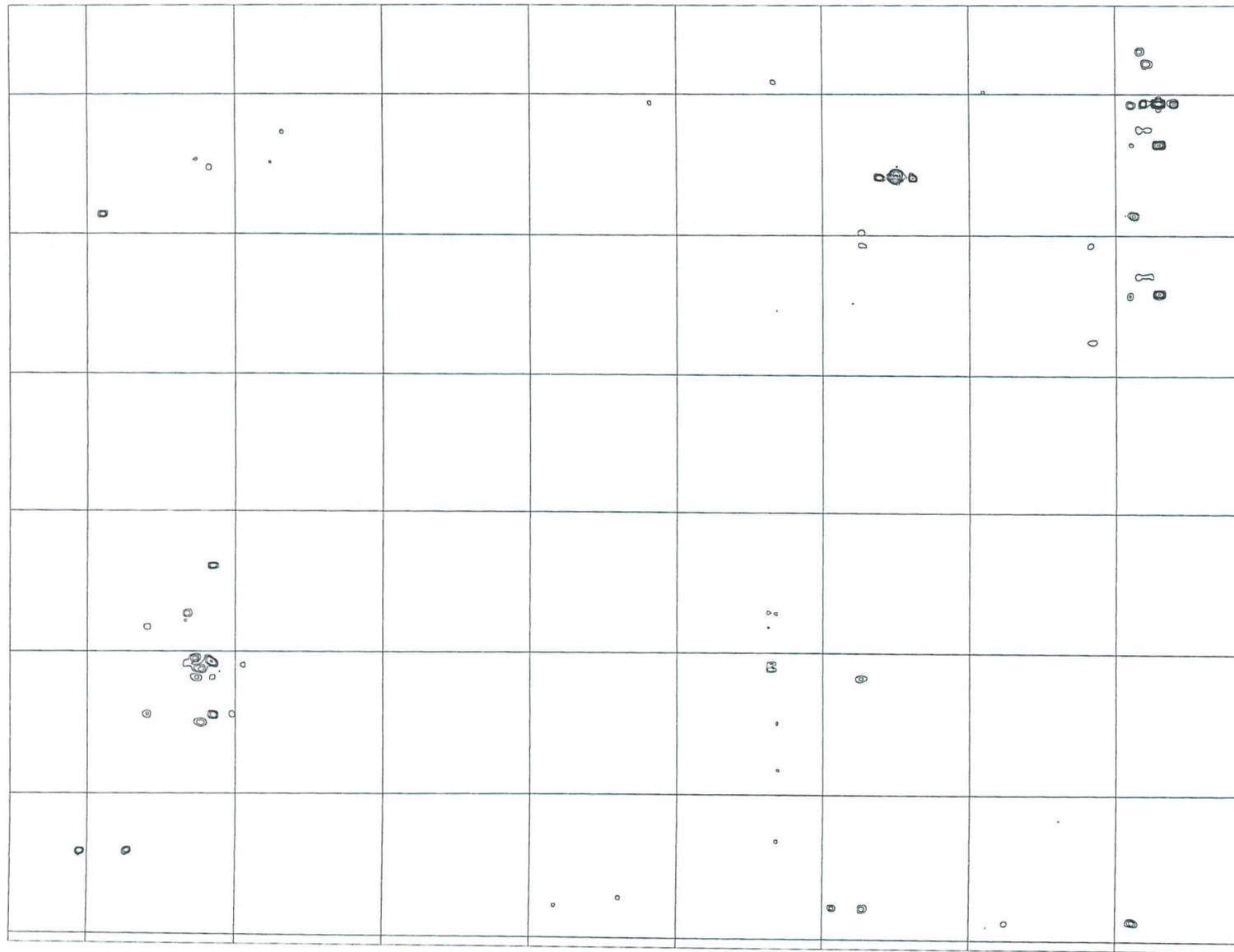
F1 - Processing parameters  
 SI 1024  
 NC2 67  
 SF 150.9028550 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 20.00 cm  
 CX1 10.00 cm  
 FZPLD 10.981 ppm  
 FZLD 8590.27 Hz  
 FZPH 0.260 ppm  
 FZM -155.92 Hz  
 F1PLD 181.148 ppm  
 F1LD 27335.72 Hz  
 F1PH1 -2.435 ppm  
 F1M -367.46 Hz  
 F2PHMCH 0.50206 ppm/cm  
 F2MCH 337.30954 Hz/cm  
 F1PHMCH 12.33866 ppm/cm  
 F2ZM 1046.67866 Hz/cm

S22. Expanded HMBC (600 MHz) spectrum of halicyclindramide F (1) in DMSO-d6 .....



6/HMBC



```

Current Data Parameters
NAME      jul107-snu-khj
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20040708
Time     1.23
INSTRUM spect
PROBHD   5 mm CPXI 1H/
PULPROG  mbcpp1dntof
TD       1024
SOLVENT  DMSO
NS       16
DS       4
SWH      7388.165 Hz
FIDRES   7.805627 Hz
AQ       0.0658550 sec
RG       20642 5
DM       64.200 usec
DE       6.00 usec
TE       298.0 K
CNS12    145.0000000
CNS13    10.0000000
DO       0.00000300 sec
D1       1.5000000 sec
d2       0.00344028 sec
d5       0.05000000 sec
D16      0.00200000 sec
IND      0.00001446 sec
MCREST   0.0000000 sec
MCHRR    1.50000000 sec

***** CHANNEL f1 *****
NUC1     1H
P1       10.60 usec
p2       21.20 usec
PL1      -5.00 dB
SFO1     600.1336008 MHz

***** CHANNEL f2 *****
NUC2     13C
P3       15.00 usec
PL2      -6.00 dB
SFO2     150.9194083 MHz

***** GRADIENT CHANNEL *****
GPNAM1   SINE 100
GPNAM2   SINE 100
GPNAM3   SINE 100
GPX1     0.00 %
GPX2     0.00 %
GPX3     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPY3     0.00 %
GPR1     50.00 %
GPR2     30.00 %
GPR3     40.10 %
P16      1000.00 usec

F1 - Acquisition parameters
ND0      2
TD       256
SFO1     150.9194 MHz
FIDRES   135.633682 Hz
SW       230.071 ppm
FhMODE   GF

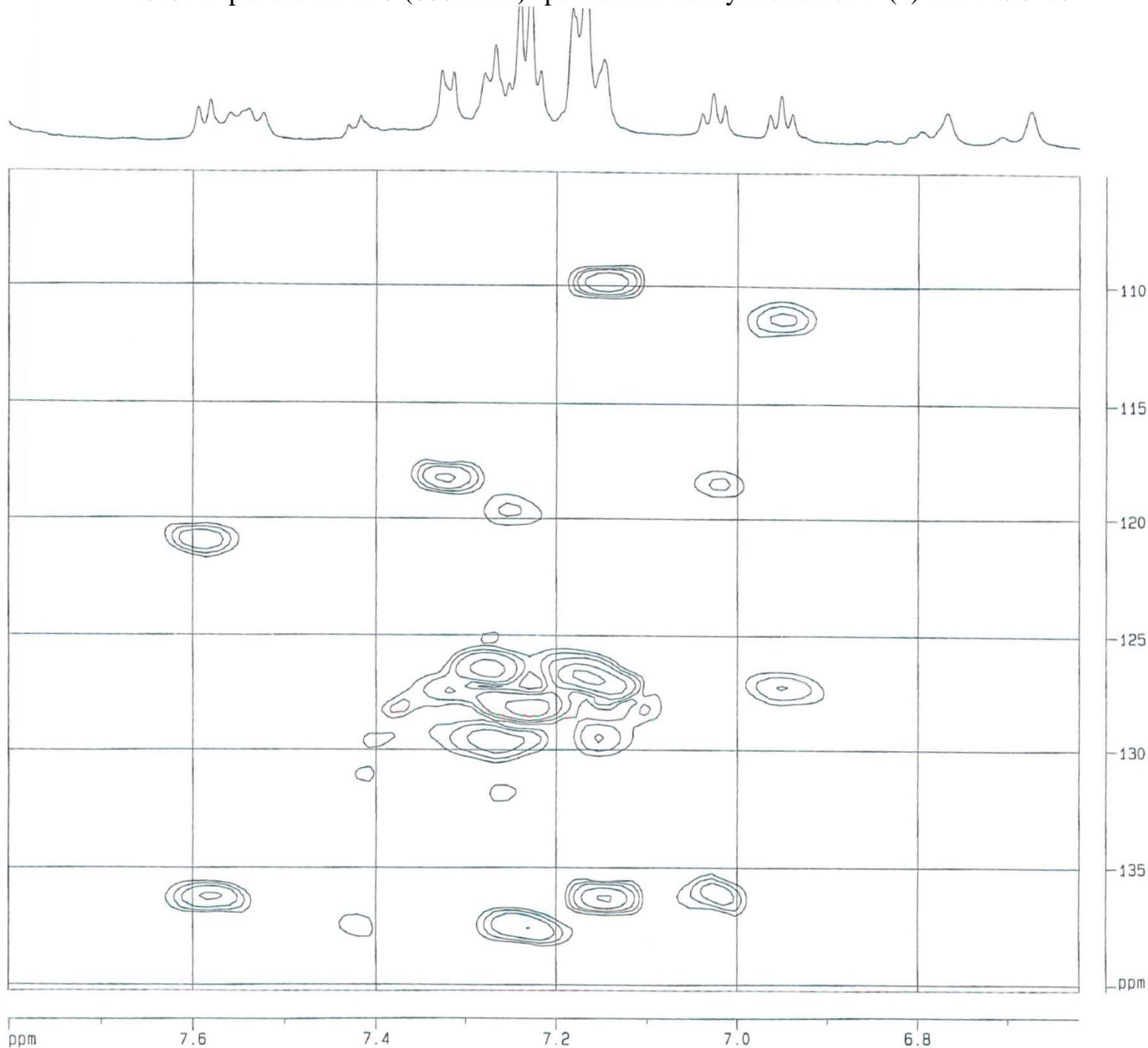
F2 - Processing parameters
SI       1024
SF       600.1300063 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
MC2      GF
SF       150.9028746 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      20.00 cm
CX1      15.00 cm
FPPLO    9.524 ppm
F2LO     5115.57 Hz
F2PHI    0.134 ppm
F2HI     80.65 Hz
F1PLO    176.752 ppm
F1LO     26672.38 Hz
F1PHI    9.123 ppm
F1HI     1376.69 Hz
F2PPMCM 0.41949 ppm/cm
F2HZCM   251.74625 Hz/cm
F1PPMCM 11.17526 ppm/cm
F1HZCM   1686.37878 Hz/cm
    
```



S23. Expanded HMBC (600 MHz) spectrum of halicyclindramide F (1) in DMSO-d6



6/HMBC

```

Current Data Parameters
NAME      ju119-amu-htj
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20040720
Time     1:30
INSTRUM  spect
PROBHD   5 mm CPTXI 1H/
PULPROG  hmcgpg1ndat
TD       1024
SOLVENT  DMSO
NS       32
DS       4
SWH      7780.160 Hz
FIDRES   7.625627 Hz
AQ       0.5658550 sec
RG       20642.5
DW       84.200 usec
DE       6.00 usec
TE       298.0 K
CNS12    145.0000000
CNS13    10.0000000
d0       0.00000300 sec
d1       1.500000000 sec
d2       0.00344828 sec
d6       0.050000000 sec
d16      0.000200000 sec
lrd      0.000144000 sec
MCREST1 0.000000000 sec
MCMR1    1.500000000 sec

***** CHANNEL f1 *****
NUC1     1H
P1       19.70 usec
p2       21.40 usec
PL1      -5.00 dB
SFO1     600.133608 MHz

***** CHANNEL f2 *****
NUC2     13C
P2       15.00 usec
PL2      -0.00 dB
SFO2     150.9154083 MHz

***** GRADIENT CHANNEL *****
GRNAM1   SINE.100
GRNAM2   SINE.100
GRNAM3   SINE.100
GRF1     0.00 %
GRF2     0.00 %
GRF3     0.00 %
GRF4     0.00 %
GRF5     0.00 %
GRF6     50.00 %
GRF7     30.00 %
GRF8     40.10 %
P16      1000.00 usec

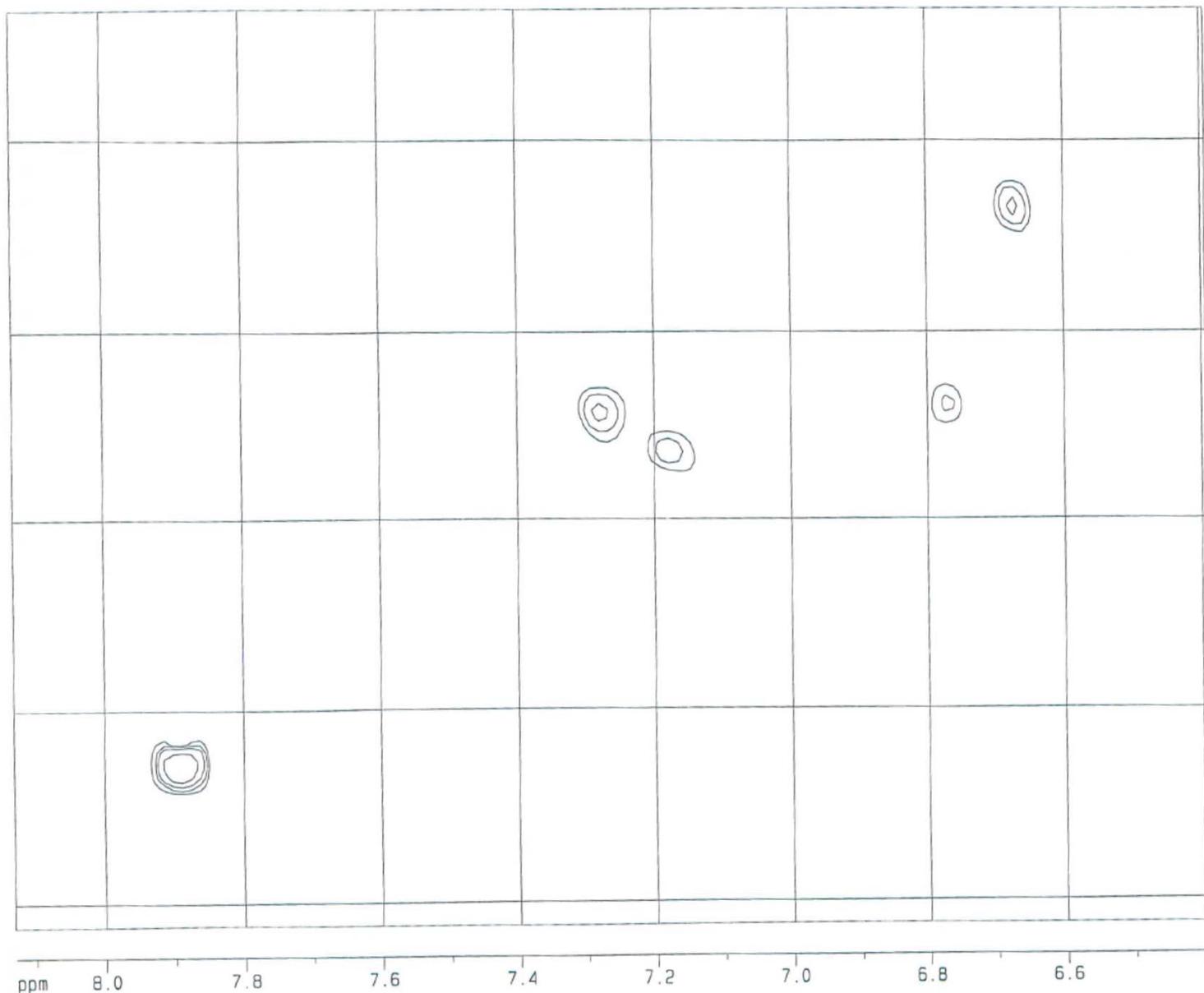
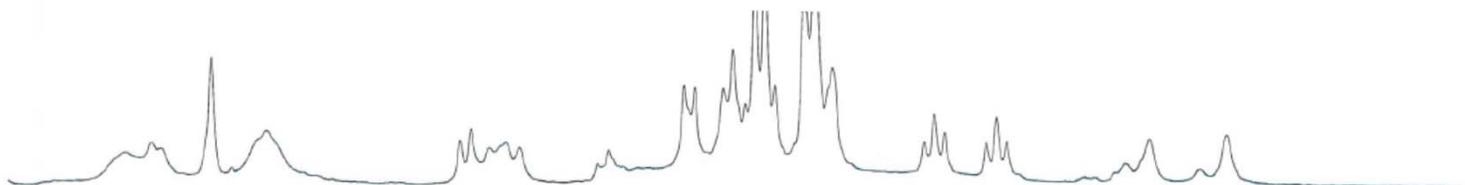
F1 - Acquisition parameters
NUC      13C
TD       256
SFO1     150.9194 MHz
FIDRES   120.833602 Hz
SW       230.8710000
FWDODE   0F

F2 - Processing parameters
SI       1024
SF       600.1300731 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
HC2      0F
SF       150.9028250 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      20.00 cm
CX1      15.00 cm
F2PL0    7.800 ppm
F2RL0    4881.25 Hz
F2PH1    6.622 ppm
F2H1     3973.93 Hz
F1PL0    140.252 ppm
F1RL0    21104.20 Hz
F1PH1    105.196 ppm
F1H1     15874.67 Hz
F2DWDCH  0.05893 ppm/cm
F2H2CH   35.36616 Hz/cm
F1DWDCH  2.33632 ppm/cm
    
```

S24. Expanded HMBC (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/HMBC

```

Current Data Parameters
NAME          jul19-nmr-hy
EXPNO         6
PROCNO        1

F2 - Acquisition Parameters
Date_         20040720
Time          1.30
INSTRUM       spect
PROBHD        5 mm CP1X1 1H/
PULPROG       hmcpgp[psdpt]
TD            1024
SOLVENT       DMSO
NS            32
DS            4
SWH           7799.162 Hz
FIDRES        7.62527 Hz
AQ            0.0058550 sec
RG            20640.5
OW            64.200 usec
DE            0.90 usec
TE            298.0 K
CNSF2         145.000000
CNS113        10.000000
GB            0.0000000 sec
G1            1.5000000 sec
G2            0.0034000 sec
G3            0.0500000 sec
G16           0.0002000 sec
MG            0.0001440 sec
MCHRES        0.0000000 sec
MCHW          1.0000000 sec

***** CHANNEL f1 *****
NUC1           1H
P1            10.70 usec
S2            21.40 usec
PL1           -5.00 dB
SFO1          600.1336008 MHz

***** CHANNEL f2 *****
NUC2           13C
P2            15.00 usec
PL2           -6.00 dB
SFO2          150.9134083 MHz

***** GRADIENT CHANNEL *****
GPRAMS        SINE 100
GPRAME        SINE 100
GPRAMS3       SINE 100
GPK1           0.00 %
GPK2           3.00 %
GPK3           3.00 %
GPK4           0.00 %
GPK5           0.00 %
GPK6           0.00 %
GPK7           0.00 %
GPK8           0.00 %
GPK9           0.00 %
GPK10          0.00 %
GPK11          0.00 %
GPK12          0.00 %
GPK13          0.00 %
GPK14          0.00 %
GPK15          0.00 %
GPK16          0.00 %
GPK17          0.00 %
GPK18          0.00 %
GPK19          0.00 %
GPK20          0.00 %
GPK21          0.00 %
GPK22          0.00 %
GPK23          0.00 %
GPK24          0.00 %
GPK25          0.00 %
GPK26          0.00 %
GPK27          0.00 %
GPK28          0.00 %
GPK29          0.00 %
GPK30          0.00 %
GPK31          0.00 %
GPK32          0.00 %
GPK33          0.00 %
GPK34          0.00 %
GPK35          0.00 %
GPK36          0.00 %
GPK37          0.00 %
GPK38          0.00 %
GPK39          0.00 %
GPK40          0.00 %
GPK41          0.00 %
GPK42          0.00 %
GPK43          0.00 %
GPK44          0.00 %
GPK45          0.00 %
GPK46          0.00 %
GPK47          0.00 %
GPK48          0.00 %
GPK49          0.00 %
GPK50          0.00 %
GPK51          0.00 %
GPK52          0.00 %
GPK53          0.00 %
GPK54          0.00 %
GPK55          0.00 %
GPK56          0.00 %
GPK57          0.00 %
GPK58          0.00 %
GPK59          0.00 %
GPK60          0.00 %
GPK61          0.00 %
GPK62          0.00 %
GPK63          0.00 %
GPK64          0.00 %
GPK65          0.00 %
GPK66          0.00 %
GPK67          0.00 %
GPK68          0.00 %
GPK69          0.00 %
GPK70          0.00 %
GPK71          0.00 %
GPK72          0.00 %
GPK73          0.00 %
GPK74          0.00 %
GPK75          0.00 %
GPK76          0.00 %
GPK77          0.00 %
GPK78          0.00 %
GPK79          0.00 %
GPK80          0.00 %
GPK81          0.00 %
GPK82          0.00 %
GPK83          0.00 %
GPK84          0.00 %
GPK85          0.00 %
GPK86          0.00 %
GPK87          0.00 %
GPK88          0.00 %
GPK89          0.00 %
GPK90          0.00 %
GPK91          0.00 %
GPK92          0.00 %
GPK93          0.00 %
GPK94          0.00 %
GPK95          0.00 %
GPK96          0.00 %
GPK97          0.00 %
GPK98          0.00 %
GPK99          0.00 %
GPK100         0.00 %

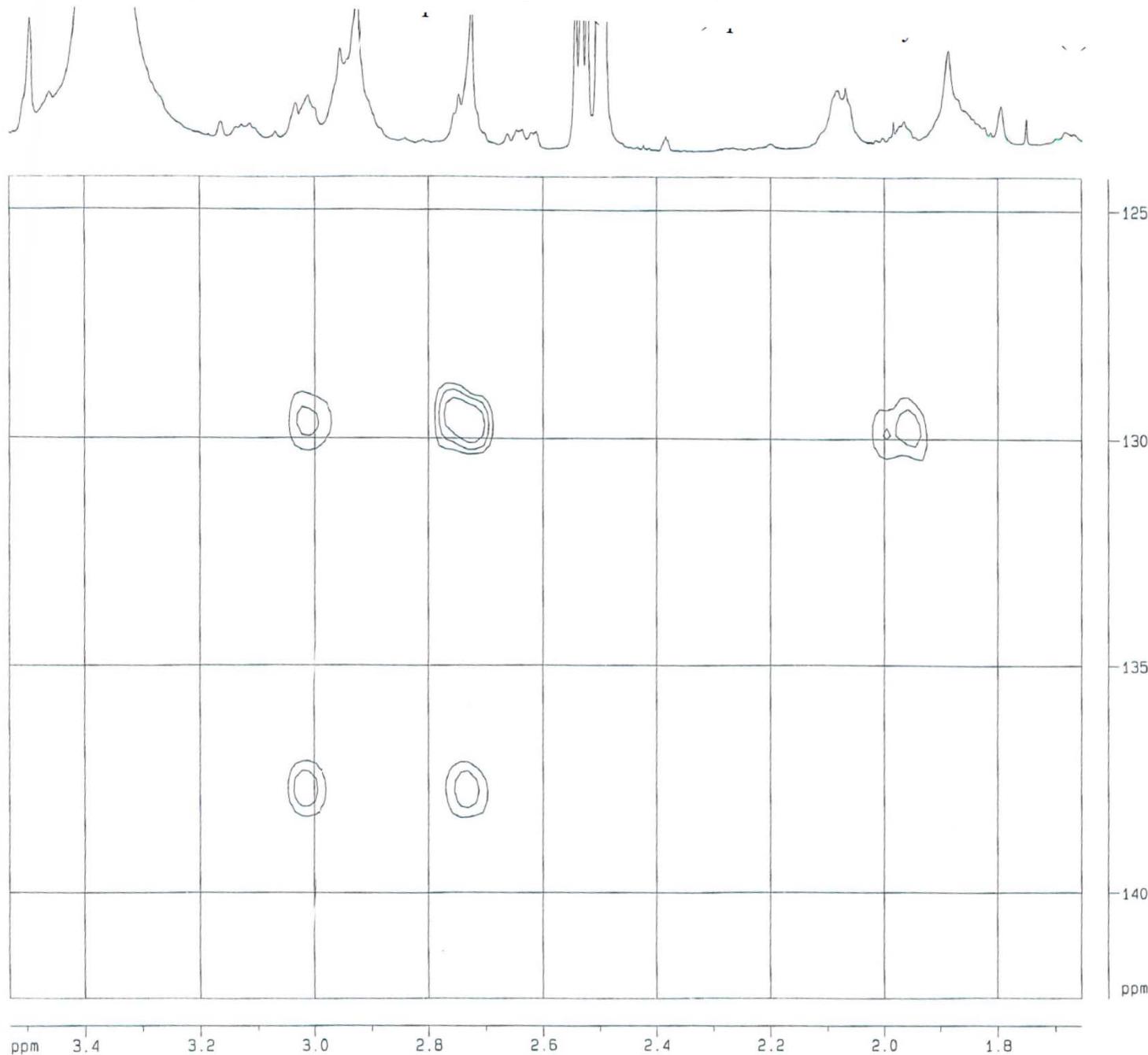
F1 - Acquisition parameters
ND0            2
TD             256
SFO1          150.9134 MHz
FIDRES        135.623680 Hz
AQ            0.003971 sec
F0MODE         DF

F2 - Processing parameters
SI             1024
SF            600.130071 MHz
WDW            SINE
SSB            0
LB             0 Hz
GB             0
PC             1.40

F1 - Processing parameters
SI             1024
SF            150.9029555 MHz
WDW            SINE
SSB            0
LB             0 Hz
GB             0

TD NMR 0101 parameters
C4Z            20.26 cm
CX1            15.00 cm
F2PLD          8.130 ppm
F2LO          4979.00 Hz
F2PHI         5.794 ppm
F2PLH        3637.93 Hz
F2PLB        50.595 ppm
F2LO         7634.93 Hz
F2PHI         26.552 ppm
F2PLH        4006.73 Hz
F2PHCH        0.28865 ppm/cm
F2CHCM        52.09464 Hz/cm
F1PHMCH       1.60299 ppm/cm
F1HZCM        241.88009 Hz/cm
    
```

S25. Expanded HMBC (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/HMBC

```

Current Data Parameters
NAME      jmlb-hm-171
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_    20040720
Time     1.30
INSTRUM spect
PROBHD   5 mm CP131 300
PULPROG  mzgpg1endzf
TD        1024
SOLVENT  DMSO
NS        32
DS         4
SWH       7388.162 Hz
FIDRES    7.402827 Hz
AQ         0.0594950 sec
RG         29542.5
DM         94.200 usec
DE         6.00 usec
TE         298.0 K
CNS12     145.000000 sec
CNS13     10.000000 sec
AQ         0.0000000 sec
D1         1.5000000 sec
D2         0.00344828 sec
DB         0.0500000 sec
D18        0.0002000 sec
IND        0.00001440 sec
MCREST    0.0000000 sec
MCHWR     1.5000000 sec

***** CHANNEL f1 *****
NUC1      1H
P1         10.70 usec
P2         21.40 usec
PL1        -5.00 dB
SFO1      600.1336008 MHz

***** CHANNEL f2 *****
NUC2      13C
P3         15.00 usec
P4         -6.00 dB
SFO2      150.9194003 MHz

***** GRADIENT CHANNEL *****
GPM1      SINE 100
GPM2      SINE 100
GPM3      SINE 100
GP11      0.00 %
GP12      0.00 %
GP13      0.00 %
GP21      0.00 %
GP22      36.00 %
GP23      45.10 %
P16       1000.00 usec

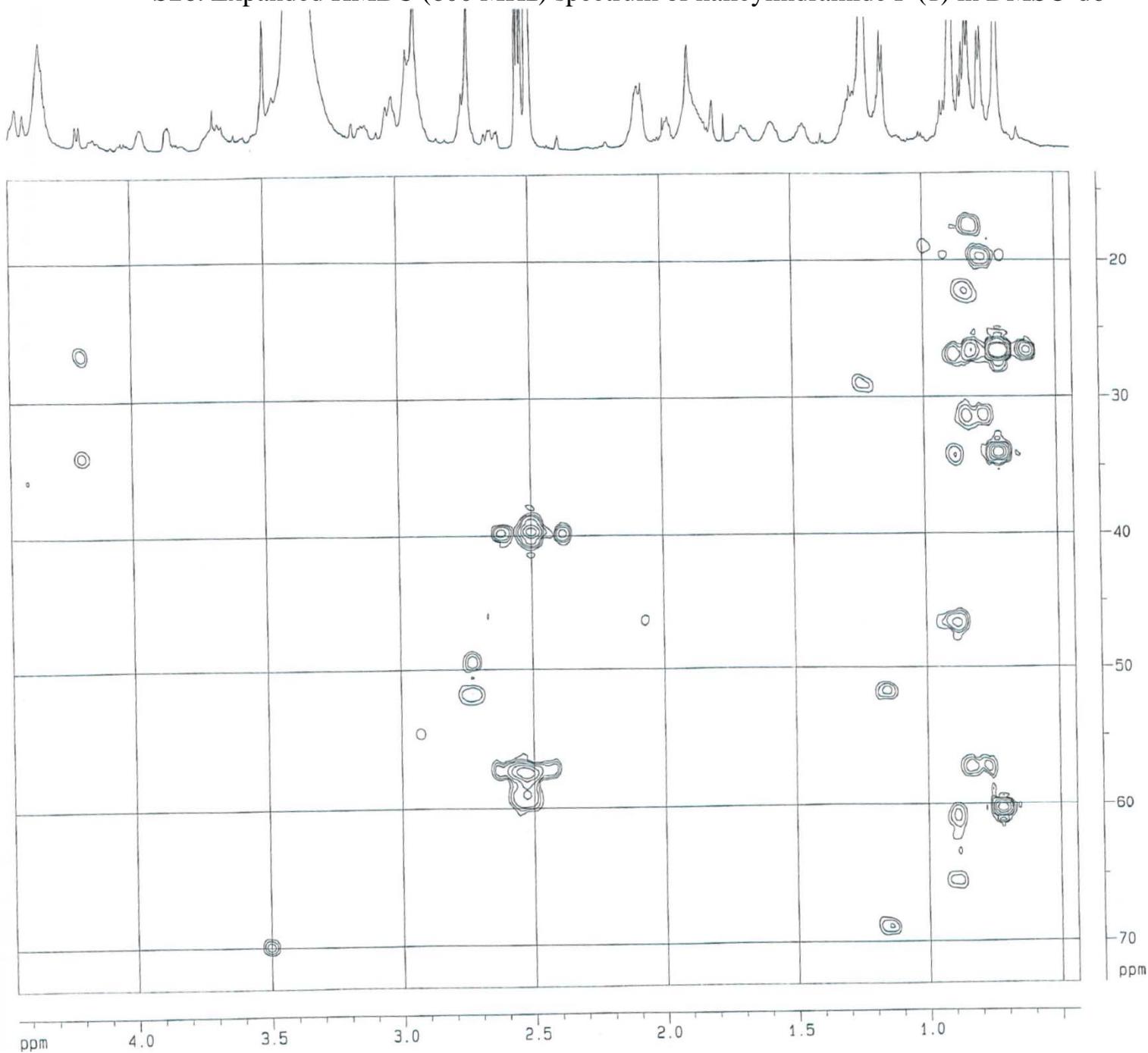
F1 - Acquisition Parameters
NUC1      1H
TD         2
SF         600.1336008 MHz
FIDRES     125.033800 Hz
SW         230.671 ppm
FMODE      DF

F2 - Processing Parameters
SI         1024
SF         800.1300071 MHz
WDW        SINE
SSB         0
LB          0.00 Hz
GB          0
PC          1.40

F1 - Processing Parameters
SI         1024
SF         150.9028555 MHz
WDW        SINE
SSB         0
LB          0.00 Hz
GB          0

2D NMR plot parameters
CAZ        29.00 cm
CX1        15.00 cm
FPLD       3.530 ppm
FPLD0      2118.16 Hz
FSDHI      1.654 ppm
FZHI       932.53 Hz
FPLD1      142.274 ppm
FPLD01     21469.56 Hz
F1PH1      124.296 ppm
F1H1       16756.89 Hz
F1PHMCH    0.09376 ppm/cm
F1HMCH     56.28164 Hz/cm
F1PHMCH    1.19842 ppm/cm
F1HMCH     180.84480 Hz/cm
    
```

S26. Expanded HMBC (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/HMBC

```

Current Data Parameters
NAME      jul19-snu-4h
EXPNO    6
PROCNO    1

#2 - Acquisition Parameters
Date_     20040720
Time      1.30
INSTRUM   spect
PROBHD    5 mm CPTAI 1H/
PULPROG   hmcpgp1m001
TD         1024
SOLVENT   DMSO
NS         32
DS         4
SWH        7700.162 Hz
FIDRES     7.60567 Hz
AQ         0.052050 sec
RG         256/2
DM         94.200 usec
DE         6.00 usec
TE         298.0 K
ENST2     145.000000
ENST13    10.000000
SFO        600.000000 sec
D1         1.5000000 sec
d2         0.0094085 sec
d8         0.0000000 sec
D15        0.0002000 sec
IND        0.0001149 sec
HCHWST     0.0000000 sec
HCHWTR     1.5000000 sec

***** CHANNEL f1 *****
NUC1       1H
P1         19.70 usec
P2         21.40 usec
PL1        -5.00 dB
SFO1       600.1336009 MHz

***** CHANNEL f2 *****
NUC2       13C
P3         15.00 usec
PL2        -5.00 dB
SFO2       150.9194083 MHz

***** GRADIENT CHANNEL *****
GPMAG1     SINE 100
GPMAG2     SINE 100
GPMAG3     SINE 100
GPA1       0.00 %
GPA2       0.00 %
GPA3       0.00 %
GPA4       0.00 %
GPA5       0.00 %
GPA6       0.00 %
GPA7       0.00 %
GPA8       0.00 %
GPA9       50.00 %
GPB1       30.00 %
GPB2       40.10 %
GPB3       1000.00 usec

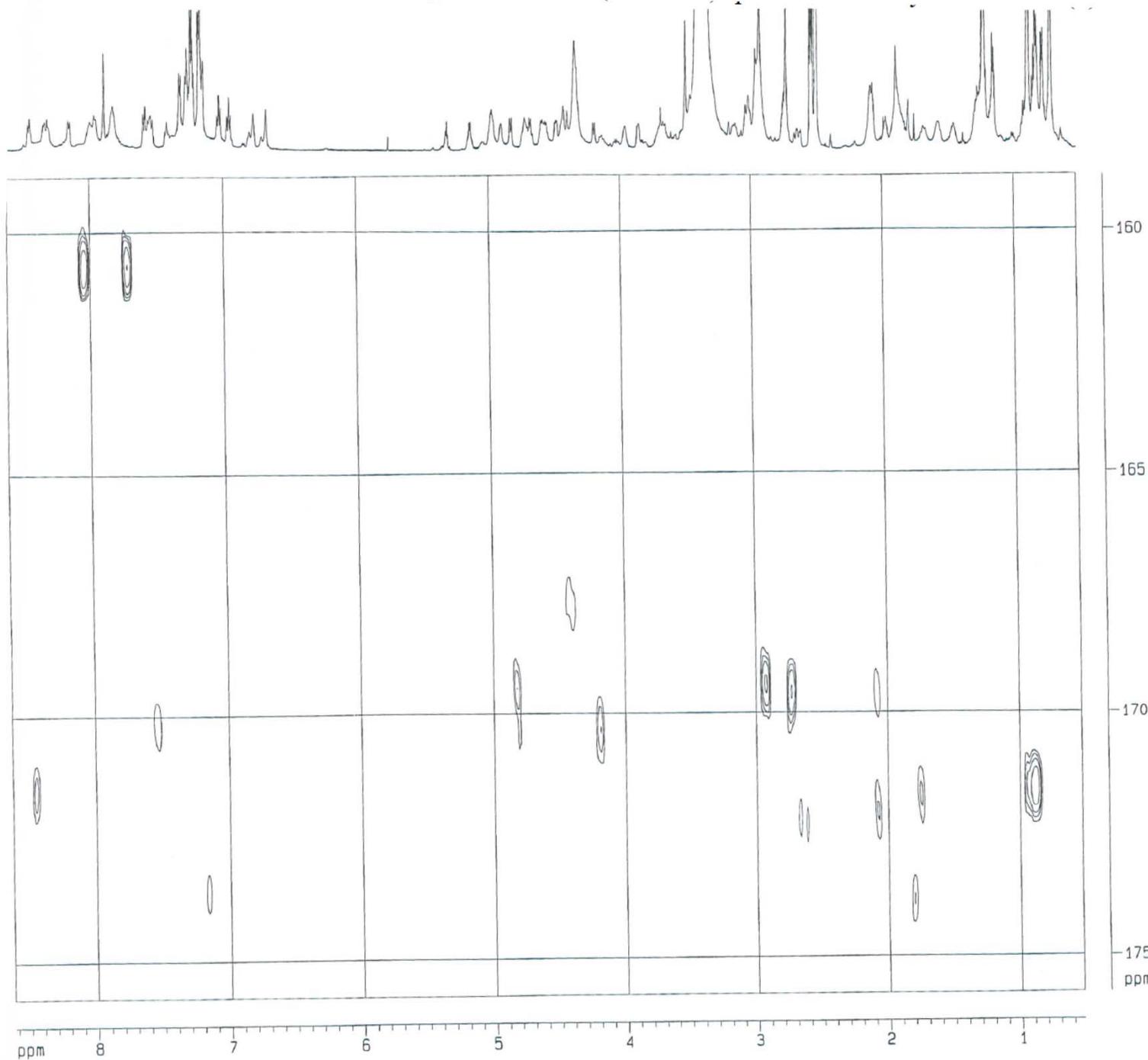
f1 - Acquisition parameters
NUC1       1
TD         256
SFO1       150.9194 MHz
FIDRES     135.639602 Hz
SF         252.071 ppm
FAMODE     DF

f2 - Processing parameters
SI         1024
SF         600.1305071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

f1 - Processing parameters
SI         1024
SF         150.9020555 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        20.00 cm
CX1        15.00 cm
F2PULPROG  4.455 ppm
F2FLD      2673.32 Hz
F2PH1      0.437 ppm
F2PH2      296.39 Hz
F2PULPROG  73.905 ppm
F2FLD      11025.77 Hz
F2PH3      13.744 ppm
F2PH4      2073.95 Hz
F2PH5CHM   0.00001 ppm/cm
F2PH5CH    120.54918 Hz/cm
F2PH5CHM   1.35478 ppm/cm
F2H2CHM    596.78821 Hz/cm
    
```

S27. Expanded HMBC (600 MHz) spectrum of halicylindramide F (1) in DMSO-d6



6/HMBC

```

Current Data Parameters
NAME      ju119-en-uhj
EXPNO    5
PROCNO   1

F2 - Acquisition Parameters
Date_    20040720
Time     11.30
INSTRUM  spect
PROBHD   5 mm CPX13 1H/
PULPROG  hmcgcp13001
TD        1024
SOLVENT  DMSO
NS        32
DS         4
SWH       7708.182 Hz
FIDRES    7.665827 Hz
AQ         0.0658550 sec
RG         20648.5
CW         84.200 usec
DE         0.00 usec
TE         308.0 K
CNS12     145.0000000
CNS13     10.0000000
DD         0.00002000 sec
D1         1.50000000 sec
d2         0.00344820 sec
d3         0.00000000 sec
D18        0.00000000 sec
INQ        0.00001440 sec
MCREST     0.00000000 sec
MCMK       1.68000000 sec

***** CHANNEL f1 *****
NUC1       1H
P1         10.70 usec
PL1        21.40 dB
PL2        -5.00 dB
SFO1       600.1330000 MHz

***** CHANNEL f2 *****
NUC2       13C
P2         15.00 usec
PL2        -8.00 dB
SFO2       150.9184083 MHz

***** GRADIENT CHANNEL *****
GRNAM1     SINE 100
GRNAM2     SINE 100
GRNAM3     SINE 100
GPX1       0.00 %
GPX2       0.00 %
GPX3       0.00 %
GPI1       0.00 %
GPY2       0.00 %
GPY3       0.00 %
GPI2       50.00 %
GPZ2       30.00 %
GPZ3       48.10 %
F1G        1000.00 usec

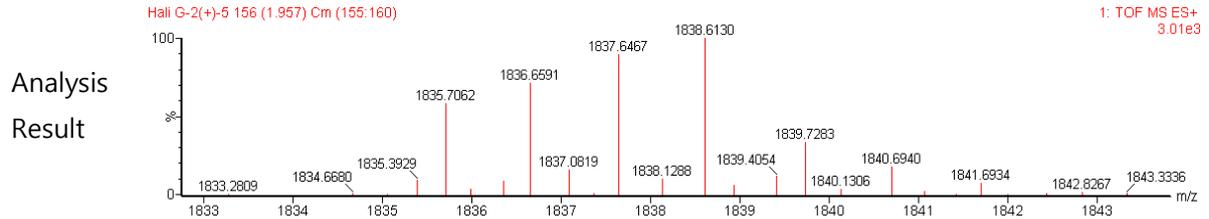
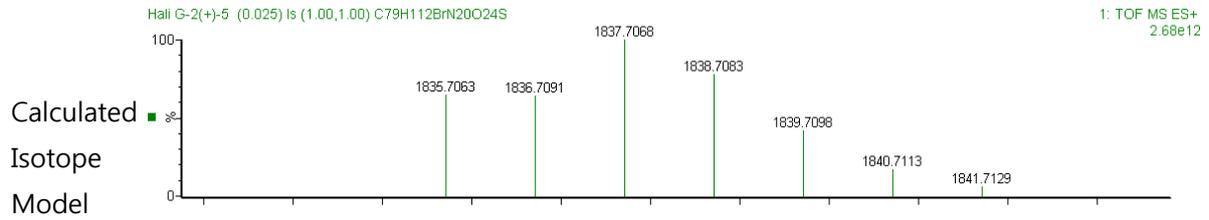
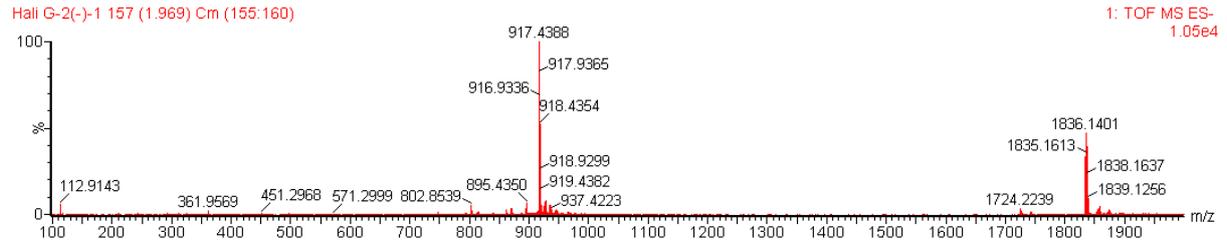
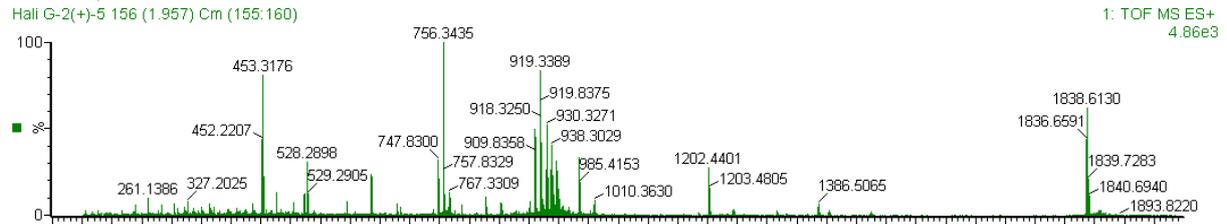
F1 - Acquisition Parameters
ND0        2
TD         256
SFO1       150.9184 MHz
FIDRES     136.433680 Hz
SW         210.071 ppm
FHMDC      0

F2 - Processing parameters
SI         1024
SF         600.1300073 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
WDW        SF
SF         150.9026955 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR Plot Parameters
CH0        20.00 cm
CX1        15.00 cm
F2P1O      8.624 ppm
F2L0       5175.82 Hz
F2P1H      0.526 ppm
F2H1       319.83 Hz
F1P1O      175.755 ppm
F1L0       26521.92 Hz
F1P1H      158.902 ppm
F1H1       23378.79 Hz
F2P1MCM    0.40493 ppm/cm
F2H1ZDM    242.59379 Hz/cm
F1P1MCM    1.12352 ppm/cm
F1H1ZDM    109.54307 Hz/cm
    
```

## S28. HR-ESI-ToF-MS spectrum of halicyclindramide G (2)



**Single Mass Analysis**  
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0  
Selected filters: None  
Monoisotopic Mass, Even Electron Ions  
19 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

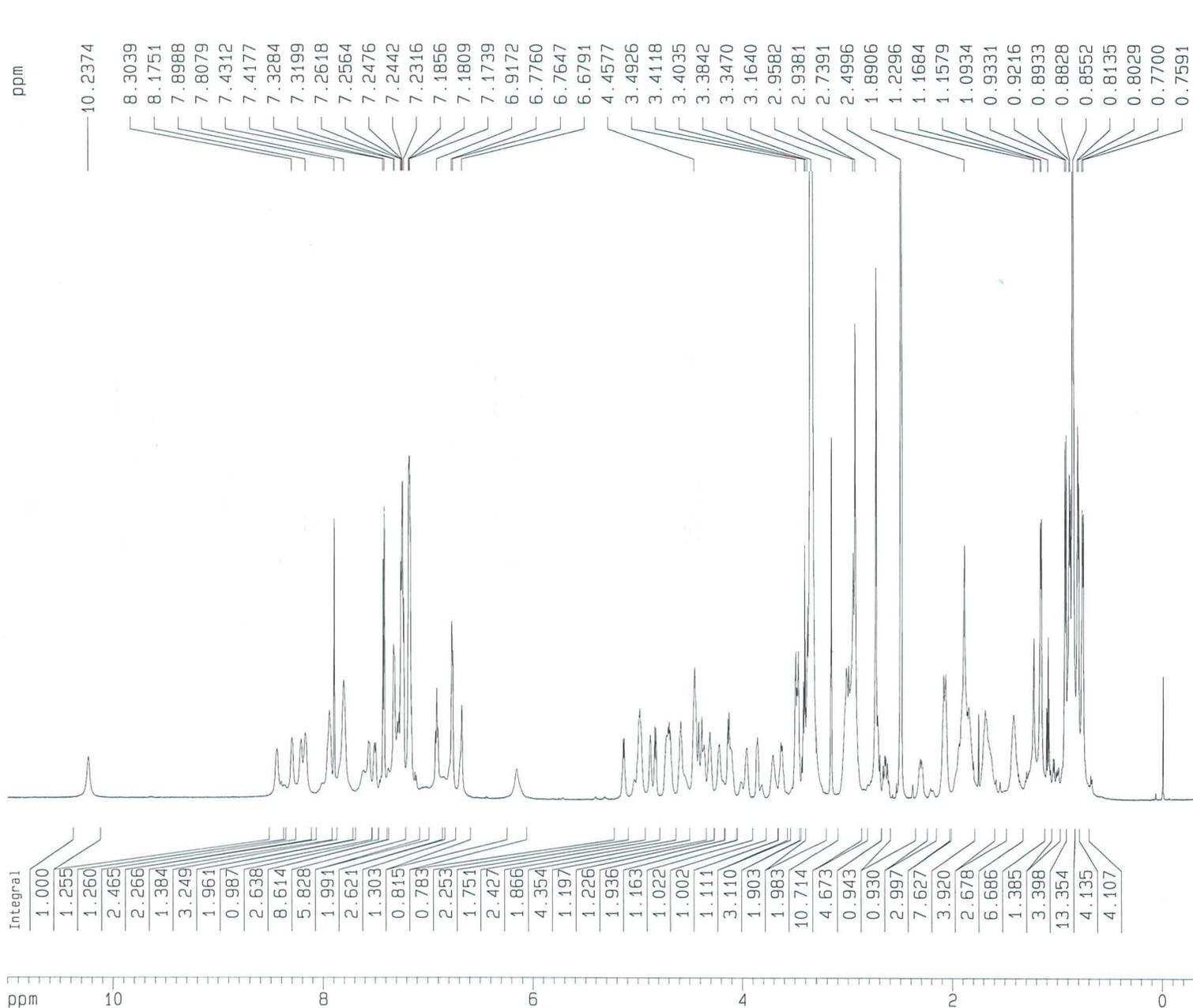
Mass	Calc. Mass	mDa	PPM	DBE	Formula	±FIT	C	H	N	O	S	Br
1835.7062	1835.7062	0.0	0.0	33.5	C79 H112 N20 O24 S Br	554690.0	79	112	20	24	1	1
1835.6997	1835.6997	6.5	-3.5	33.5	C78 H112 N22 O21 S2 Br	554690.0	78	112	22	21	2	1

MCP 2300, CP 2200  
Hali G-2(+)-5 156 (1.957) Cm (155:160) 1: TOF MS ES+  
1.75e3

For Help, press F1

S29. <sup>1</sup>H NMR (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6

5/1H



Current Data Parameters

NAME ju104-snu-chj  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20050704  
Time 16.06  
INSTRUM spect  
PROBHD 5 mm CPTXI 1H/  
PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 8  
DS 0  
SWH 8741.259 Hz  
FIDRES 0.133381 Hz  
AQ 3.7487664 sec  
RG 20.2  
DW 57.200 usec  
DE 6.00 usec  
TE 298.0 K  
D1 1.00000000 sec  
MCREST 0.00000000 sec  
MCWRK 0.01500000 sec

==== CHANNEL f1 =====

NUC1 1H  
P1 10.30 usec  
PL1 -5.00 dB  
SFO1 600.1336696 MHz

F2 - Processing parameters

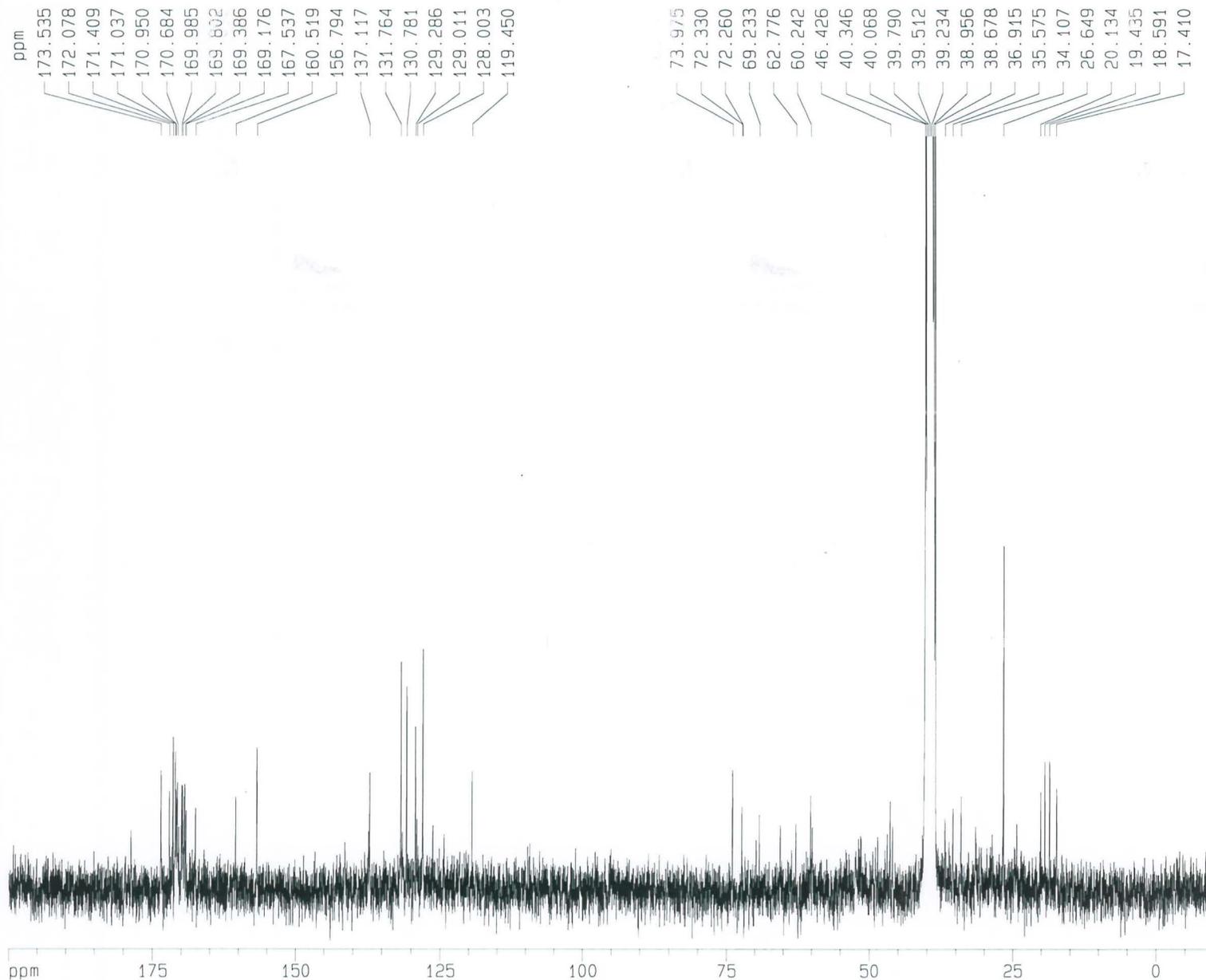
SI 32768  
SF 600.1300074 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters

CX 20.00 cm  
CY 160.00 cm  
F1P 11.000 ppm  
F1 6601.43 Hz  
F2P -0.300 ppm  
F2 -180.04 Hz  
PPMCM 0.56500 ppm/cm  
HZCM 339.07346 Hz/cm

# S30. <sup>13</sup>C NMR (150 MHz) spectrum of halicylindramide G (2) in DMSO-d6

5/C13



## Current Data Parameters

NAME jul105-snu-chj  
 EXPNO 1  
 PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20050706  
 Time 5.29  
 INSTRUM spect  
 PROBHD 5 mm Dual 13  
 PULPROG zgdc  
 TD 65536  
 SOLVENT DMSO  
 NS 15360  
 DS 0  
 SWH 21097.047 Hz  
 FIDRES 0.321915 Hz  
 AQ 1.5532532 sec  
 RG 8192  
 DW 23.700 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
 P1 10.60 usec  
 PL1 0.00 dB  
 SFO1 75.4760204 MHz

## ===== CHANNEL f2 =====

CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 100.00 usec  
 PL2 -5.00 dB  
 PL12 15.00 dB  
 SFO2 300.1312005 MHz

## F2 - Processing parameters

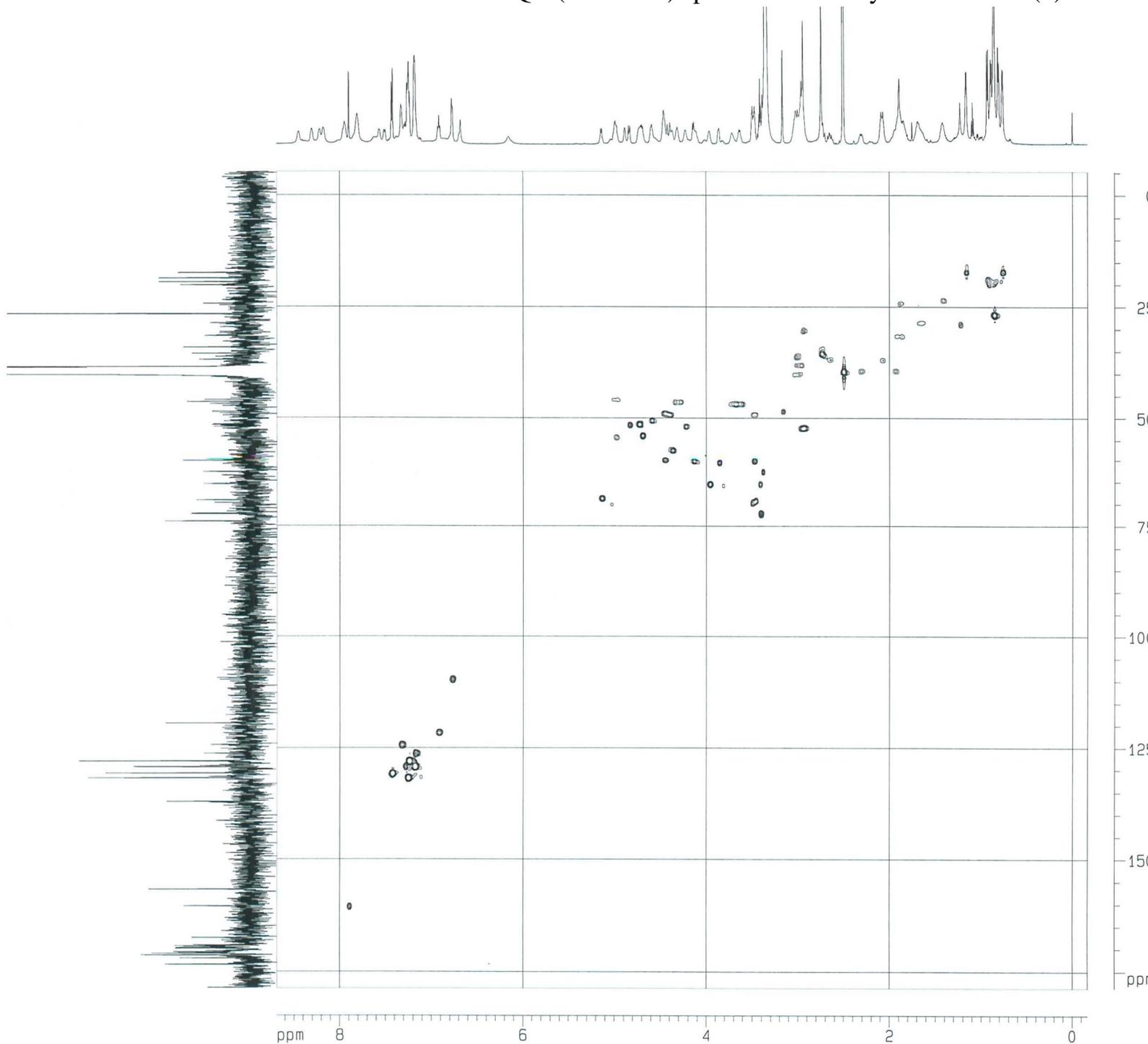
SI 32768  
 SF 75.4677839 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
 F1P 200.000 ppm  
 F1 15093.56 Hz  
 F2P -10.000 ppm  
 F2 -754.68 Hz  
 PPMCM 10.50000 ppm/cm  
 HZCM 792.41168 Hz/cm

S31. HSQC (600 MHz) spectrum of halicyclindramide G (2) in DMSO-d6

5/hsqctgpp



```

Current Data Parameters
NAME          ju104-snu-ch)
EXPNO         7
PROCNO        1

F2 - Acquisition Parameters
Date_         20050705
Time          16:44
INSTRUM       spect
PROBHD        5 mm CPTXI 1H/
PULPROG       hsqctgpp
TD            1024
SOLVENT       DMSO
NS            8
DS            32
SWH           6613.757 Hz
FIDRES        6.458747 Hz
AQ            0.0775400 sec
RG            26008
DK            75.600 usec
DE            6.6 usec
TE            299.0 K
CNST2         145.000000
d0            0.00000300 sec
d1            1.5000000 sec
d4            0.00172414 sec
d11           0.03000000 sec
d13           0.00000400 sec
d16           0.00000000 sec
DELTA         0.0182680 sec
DELTA1        0.00071614 sec
IN0           0.00001380 sec
MCOREST       0.00000000 sec
MCMRR         0.30000001 sec
STICNT        128

***** CHANNEL f1 *****
NUC1          1H
P1            10.40 usec
P2            20.80 usec
P2B           0.10 usec
PL1           -5.00 dB
PL2           -5.00 dB
SFO1          600.133006 MHz

***** CHANNEL f2 *****
CPDPRG2       gprg
NUC2          13C
P3            15.00 usec
P4            30.00 usec
PCPD2         70.00 usec
PL2           -6.00 dB
PL12          7.40 dB
SFO2          150.9194083 MHz

***** GRADIENT CHANNEL *****
GPNAM1        SINE 100
GPNAM2        SINE 100
GPX1          0.00 X
GPX2          0.00 X
GPY1          0.00 X
GPY2          0.00 X
GPI1          80.00 X
GPI2          20.10 X
P16           1000.00 usec

F1 - Acquisition parameters
NDO           2
TD            256
SFO1          150.9194 MHz
FIDRES        141.920792 Hz
SH            240.074 cm
FmMCOE        Echo-Antiecho

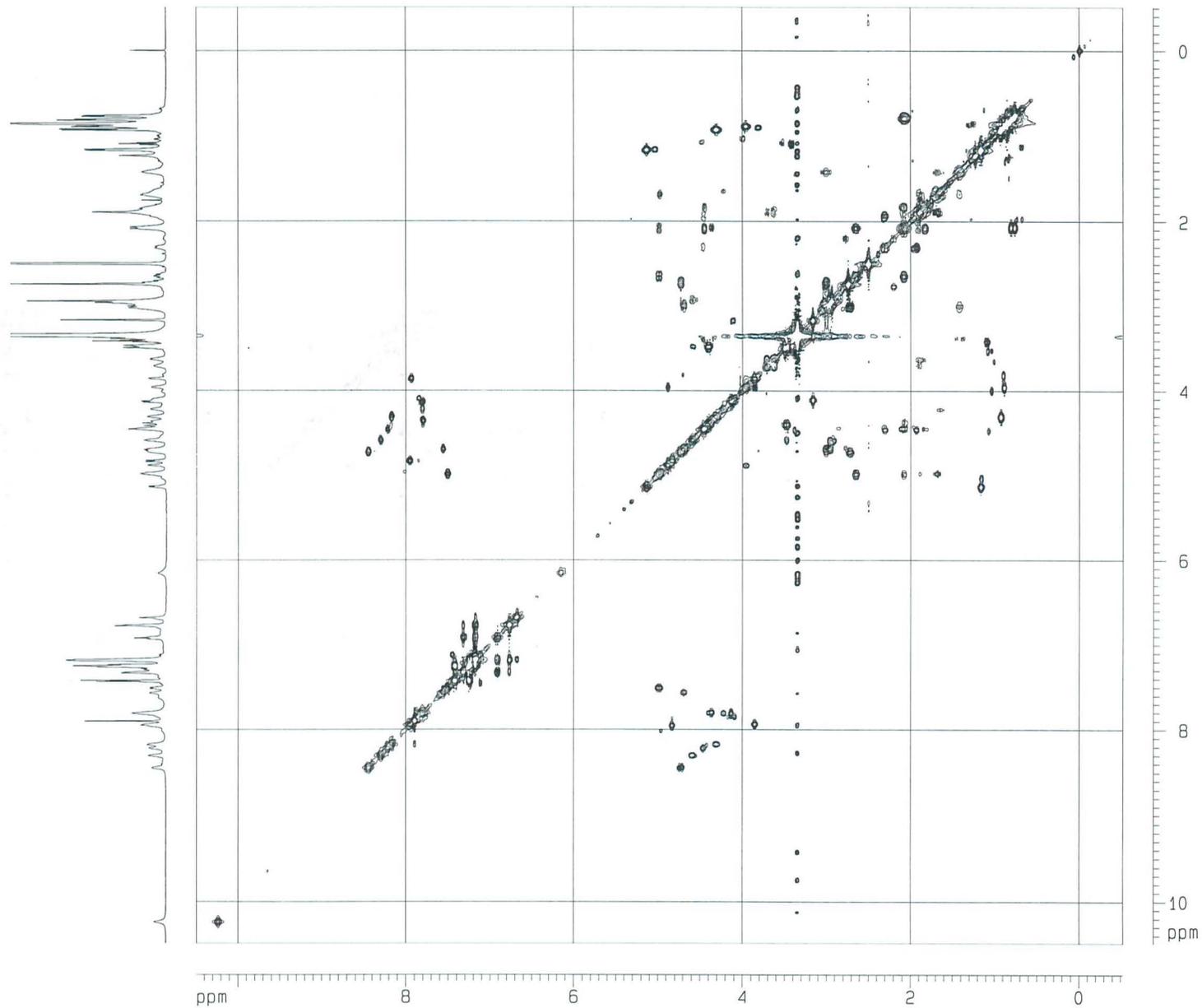
F2 - Processing parameters
SI            1024
SF            600.1300074 MHz
WDW           G5SINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.40

F1 - Processing parameters
SI            1024
MC2           echo-antiecho
SF            150.9028739 MHz
WDW           G5SINE
SSB           2
LB            0.00 Hz
GB            0

3D NMR plot parameters
CX2           15.00 cm
CX1           15.00 cm
F2PULO        0.690 ppm
F2PL0         9215.06 Hz
F2PH1         -0.167 ppm
F2H1          -100.49 Hz
F1PULO        178.739 ppm
F1PL0         26972.29 Hz
F1PH1         -5.322 ppm
F1H1          -803.13 Hz
F2PPMCM       0.59049 ppm/cm
F2HZCM        354.36990 Hz/cm
F1PPMCM       12.27077 ppm/cm
F1HZCM        1851.69470 Hz/cm
    
```

S32. COSY (600 MHz) spectrum of halicyclindramide G (2) in DMSO-d6

5/COSY



```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO     5
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      12.57
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   cosyprogf
TD         2048
SOLVENT   DMSO
NS         8
DS         8
SWH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         35.9
DW         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00000300 sec
d1         1.48689198 sec
d13        0.00000400 sec
d16        0.00020000 sec
JNO        0.00015149 sec
MCREST     0.00000000 sec
MCWRK     1.48689198 sec

===== CHANNEL f1 =====
NUC1       1H
P0         10.40 usec
P1         10.40 usec
PL1        -5.00 dB
SF01       600.133006 MHz

===== GRADIENT CHANNEL =====
GPNAM1     SINE 100
GPNAM2     SINE 100
GPX1       0.00 %
GPX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       10.00 %
GPZ2       10.00 %
P16        1000.00 usec

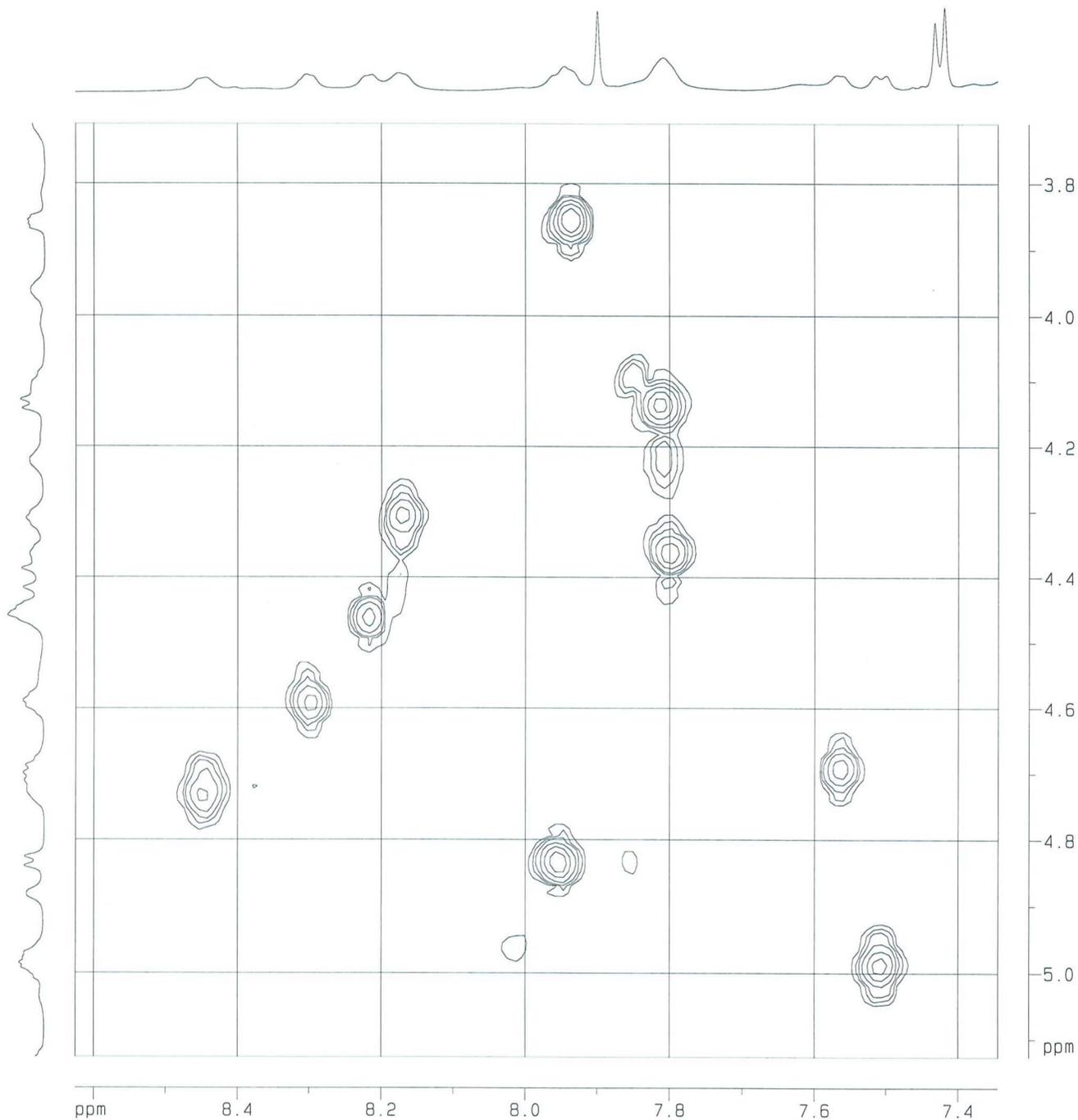
F1 - Acquisition parameters
ND0        1
TD         256
SF01       600.133 MHz
FIDRES     25.785955 Hz
SW         11.000 ppm
FMODE      GF

F2 - Processing parameters
SI         1024
SF         600.1300074 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        GF
SF         600.1300074 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      10.498 ppm
F2LO       6300.13 Hz
F2PHI      -0.523 ppm
F2HI       -313.63 Hz
F1PLO      10.487 ppm
F1LO       6293.85 Hz
F1PHI      -0.512 ppm
F1HI       -307.35 Hz
F2PPMCM    0.73470 ppm/cm
F2HZCM     440.93711 Hz/cm
F1PPMCM    0.73331 ppm/cm
F1HZCM     440.08035 Hz/cm
    
```

S33. Expanded COSY (600 MHz) spectrum of halicyclindramide G (2) in DMSO-d6



5/COSY

```

Current Data Parameters
NAME      jul04-snu-ch)
EXPNO    5
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      12.57
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPRG    cosygpqf
TD        2048
SOLVENT   DMSO
NS        16
DS        8
SWH       6613.757 Hz
FIDRES    3.229373 Hz
AQ        0.1549544 sec
RG        35.9
DM        75.600 usec
DE        6.00 usec
TE        298.0 K
d0        0.0000300 sec
d1        1.48669199 sec
d13       0.0000400 sec
d16       0.0020000 sec
IND       0.0015149 sec
MCREST    0.0000000 sec
MCWRK     1.48669199 sec

***** CHANNEL f1 *****
NUC1      1H
PC        10.40 usec
P1        10.40 usec
PL1       -5.00 dB
SFO1      600.1330006 MHz

***** GRADIENT CHANNEL *****
GPNAM1    SINE.100
GPNAM2    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPZ1      10.00 %
GPZ2      10.00 %
P16       1000.00 usec

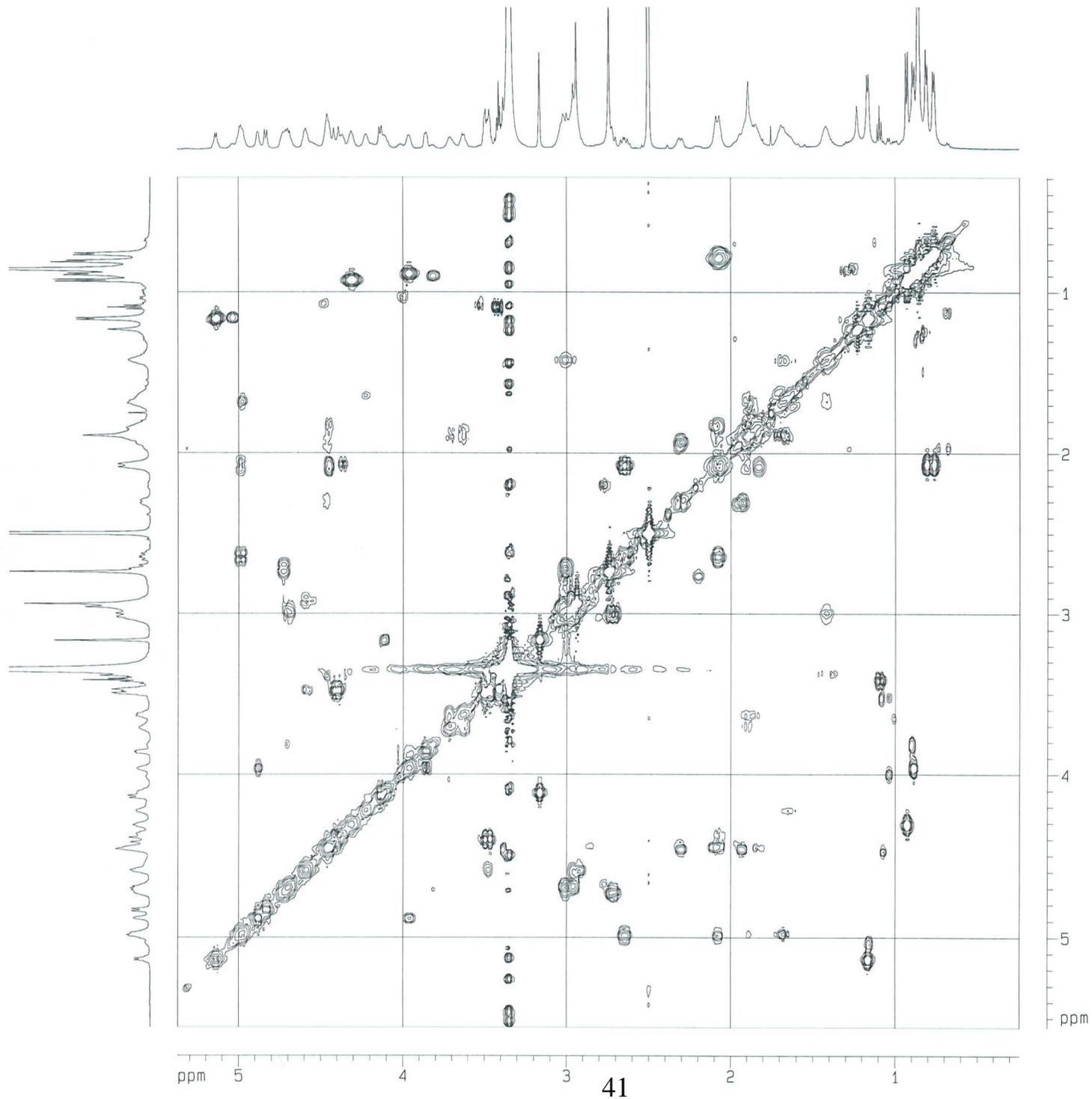
F1 - Acquisition parameters
ND0       1
TD        256
SFO1      600.133 MHz
FIDRES    25.785955 Hz
SFO2      11.000 ppm
FRMODE    GF

F2 - Processing parameters
SI        1024
SF        600.1300074 MHz
KDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.00

F1 - Processing parameters
SI        1024
MC2       GF
SF        600.1300074 MHz
KDW       SINE
SSB       0
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     8.625 ppm
F2LO      5176.31 Hz
F2PHI     7.345 ppm
F2HI      4407.72 Hz
F1PLO     5.127 ppm
F1LO      3077.05 Hz
F1PHI     3.709 ppm
F1HI      2226.12 Hz
F2PPMCM   0.08538 ppm/cm
F2HZCM    51.23940 Hz/cm
F1PPMCM   0.09453 ppm/cm
F1HZCM    56.72910 Hz/cm
    
```

S34. Expanded COSY (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/COSY

```

Current Data Parameters
NAME      ju104-snu-chj
EXPNO     5
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      12:57
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPROG   cosygpdf
TD         2048
SOLVENT   DMSO
NS         16
DS         8
SWH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         35.9
RW         75.600 usec
DE         5.00 usec
TE         298.0 K
d0         0.0000300 sec
d1         1.48689198 sec
d13        0.0000400 sec
d16        0.0002000 sec
INO        0.00015149 sec
MCREST     0.0000000 sec
MCWRK     1.48689198 sec

----- CHANNEL f1 -----
NUC1       1H
P0         10.40 usec
P1         10.40 usec
PL1        -5.00 dB
SF01       600.133006 MHz

===== GRADIENT CHANNEL =====
GRNAM1     SINE 100
GRNAM2     SINE 100
GPX1       0.00 %
GPX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       10.00 %
GPZ2       10.00 %
P16        1000.00 usec

F1 - Acquisition parameters
ND0        1
TD         256
SF01       600.133 MHz
FIDRES     25.785985 Hz
SW         11.000 ppm
FMODE      DF

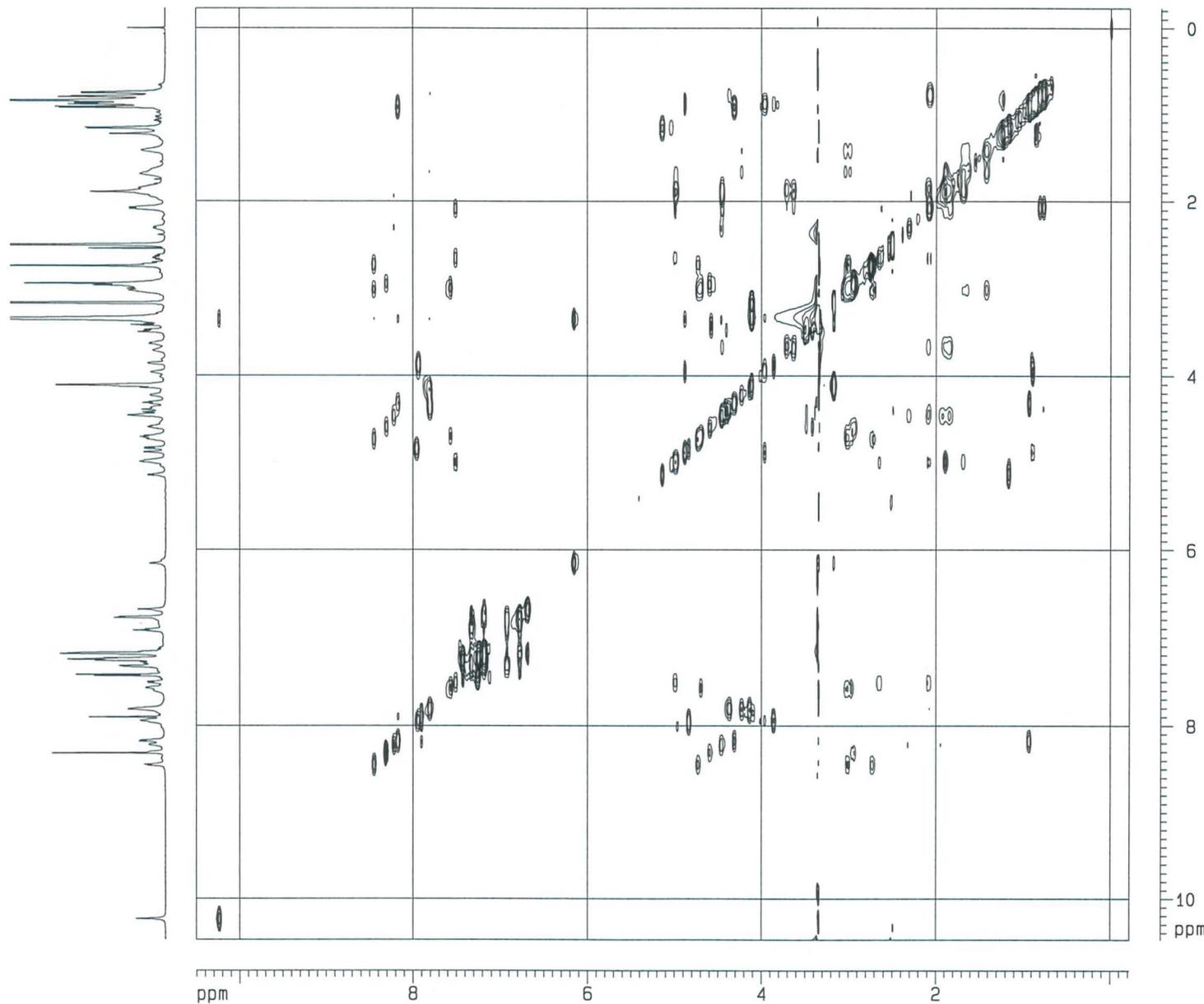
F2 - Processing parameters
SI         1024
SF         600.1300074 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        DF
SF         600.1300074 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1         15.00 cm
F2PL0      5.375 ppm
F2RL0      3225.77 Hz
F2PH1      0.242 ppm
F2H1       144.84 Hz
F1PL0      5.557 ppm
F1L0       3334.91 Hz
F1PH1      0.293 ppm
F1H1       176.13 Hz
F2PPMCM    0.34224 ppm/cm
F2HZCM     205.38814 Hz/cm
F1PPMCM    0.35090 ppm/cm
F1HZCM     210.58830 Hz/cm
    
```

S35. TOCSY (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6

5/TOCSY



```

Current Data Parameters
NAME      oct25-snu-chj
EXPNO     23
PROCNO    1

F2 - Acquisition Parameters
Date_     20051028
Time      13.26
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   mlevgh
TD         2048
SOLVENT   DMSO
NS         16
DS         16
SWH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         90.5
DW         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00006506 sec
D1         2.00000000 sec
D9         0.06000000 sec
d12        0.00002000 sec
FACTOR1    6
fNO        0.00015149 sec
f1         35
MCREST     0.00000000 sec
MCWRK     1.00000000 sec
SCALEF     6
STICNT     64

***** CHANNEL f1 *****
NUC1       1H
P1         10.50 usec
p5         16.67 usec
P6         25.00 usec
p7         50.00 usec
P17        1500.00 usec
PL1        -5.00 dB
PL10       2.54 dB
SFO1       600.1330006 MHz

F1 - Acquisition parameters
ND0        1
TD         128
SFO1       600.133 MHz
FIDRES     51.571911 Hz
SW         11.000 ppm
FnMODE     States-TPPI

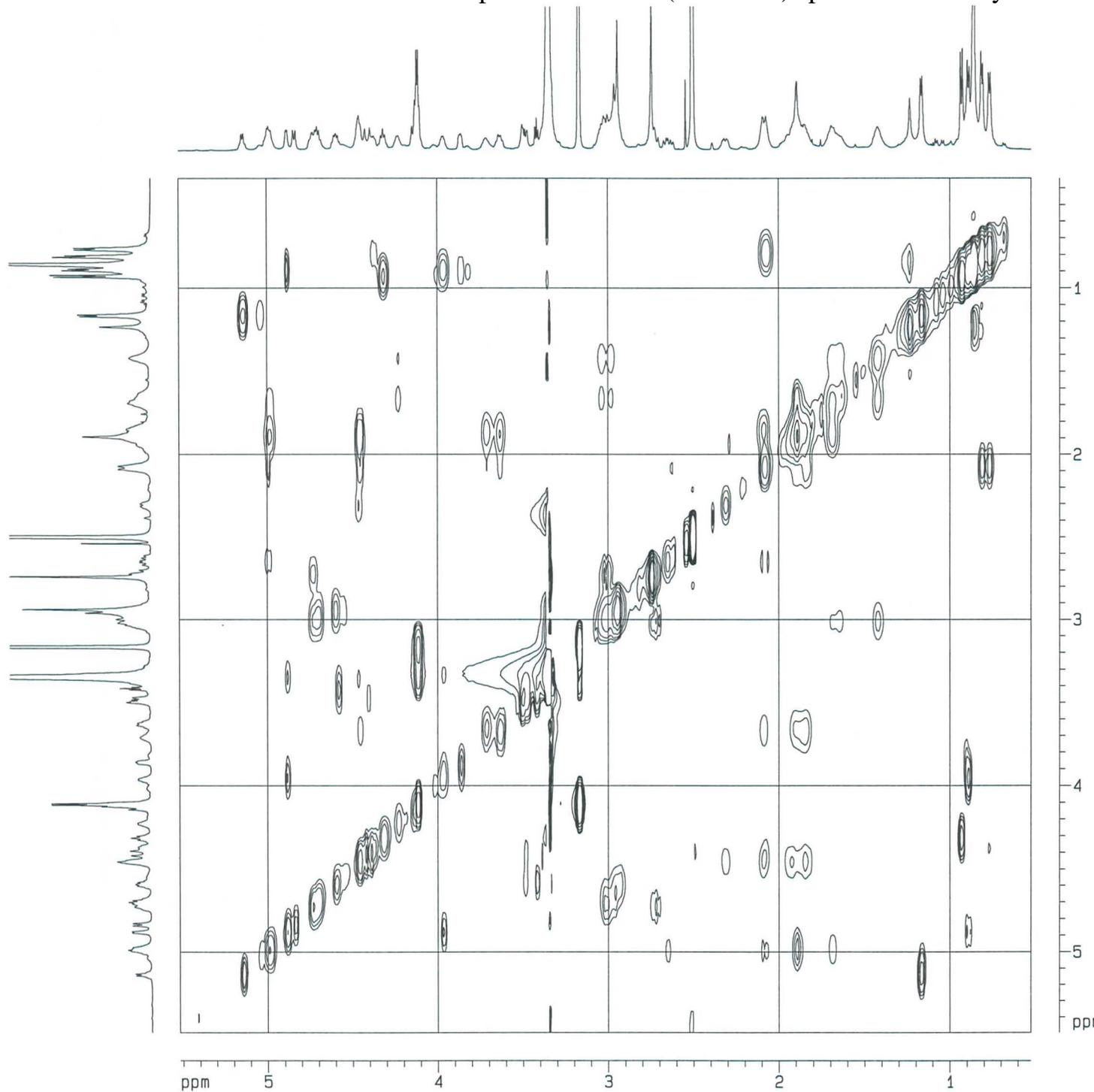
F2 - Processing parameters
SI         1024
SF         600.1300052 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300052 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      10.502 ppm
F2LO       6302.35 Hz
F2PH1      -0.218 ppm
F2HI       -130.55 Hz
F1PLO      10.480 ppm
F1LO       6289.63 Hz
F1PH1      -0.229 ppm
F1HI       -137.52 Hz
F2PPMCM    0.71461 ppm/cm
F2HZCM     428.86081 Hz/cm
F1PPMCM    0.71397 ppm/cm
F1HZCM     428.47662 Hz/cm
    
```

S36. Expanded TOCSY (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6

5/TOCSY



```

Current Data Parameters
NAME      oct25-snu-chj
EXPNO    23
PROCNO    1

F2 - Acquisition Parameters
Date_    20051028
Time     13.26
INSTRUM  spect
PROBHD   5 mm CPTXI 1H/
PULPROG  mlevph
TD        2048
SOLVENT  DMSO
NS        16
DS        16
SWH       6613.757 Hz
FIDRES    3.229373 Hz
AQ        0.1549544 sec
RG        90.5
DM        75.600 usec
DE        6.00 usec
TE        298.0 K
d0        0.0000506 sec
d1        2.0000000 sec
d9        0.0600000 sec
d12       0.0000200 sec
FACTOR1   6
IN0       0.00015149 sec
I1        36
MCREST    0.0000000 sec
MCWRK     1.0000000 sec
SCALEF    6
ST1CNT    64

----- CHANNEL f1 -----
NUC1      1H
P1        10.50 usec
p5        16.67 usec
P6        25.00 usec
p7        50.00 usec
P17       1500.00 usec
PL1       -5.00 dB
PL10      2.54 dB
SFO1      600.1330006 MHz

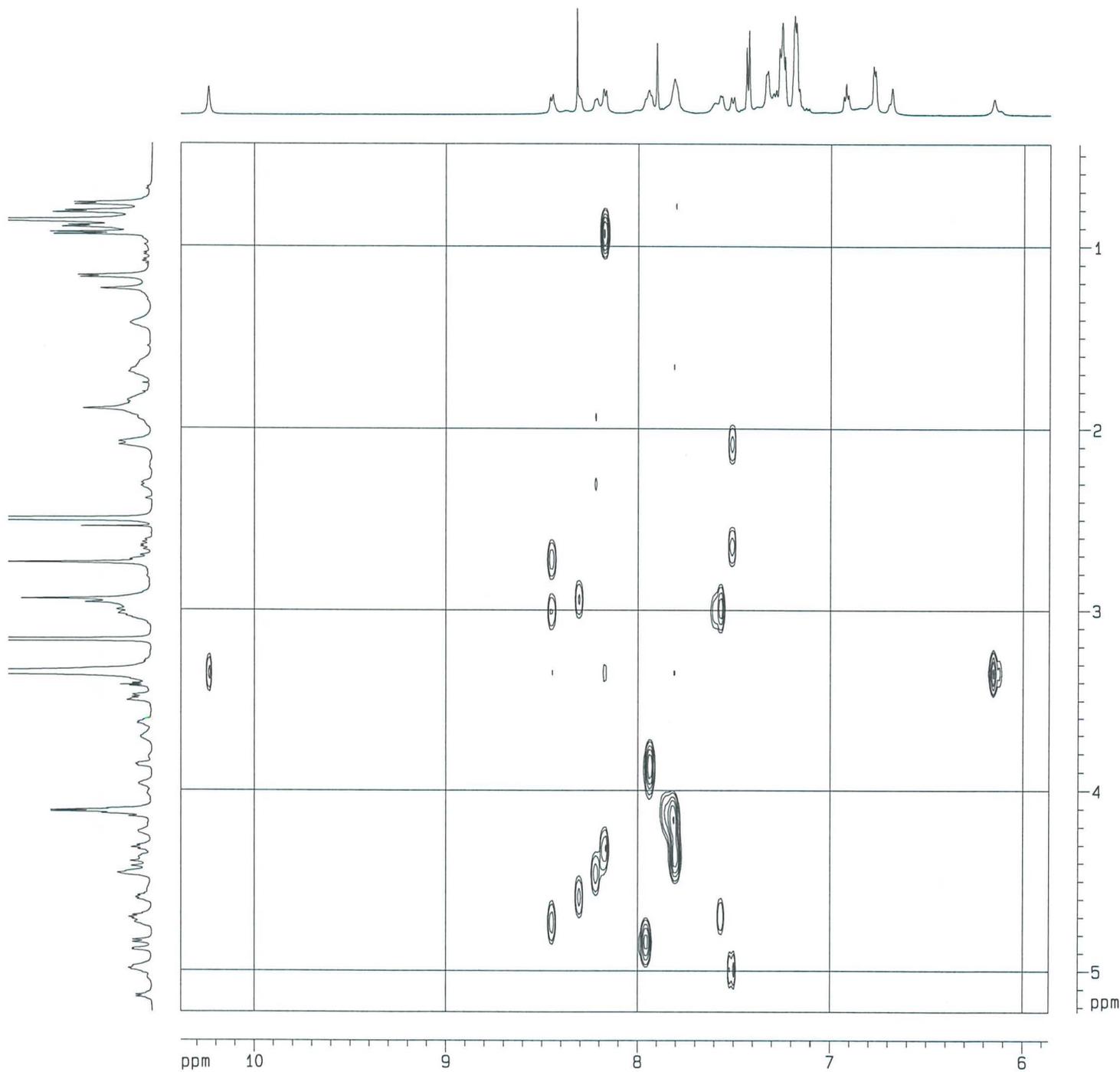
F1 - Acquisition parameters
ND0       1
TD        128
SFO1      600.133 MHz
FIDRES    51.571911 Hz
SW        11.000 ppm
F1MODE    States-TPPI

F2 - Processing parameters
SI        1024
SF        600.1300052 MHz
WDW       OSINE
SSB       2
LB        0.00 Hz
GB        0
PC        1.00

F1 - Processing parameters
SI        1024
MC2       States-TPPI
SF        600.1300052 MHz
WDW       OSINE
SSB       2
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO    5.519 ppm
F2LO     3311.95 Hz
F2PHI    0.525 ppm
F2HI     315.09 Hz
F1PLO    5.496 ppm
F1LO     3298.45 Hz
F1PHI    0.340 ppm
F1HI     204.14 Hz
F2PPMCM  0.33291 ppm/cm
F2HZCM   199.79056 Hz/cm
F1PPMCM  0.34374 ppm/cm
F1HZCM   206.28764 Hz/cm
    
```

S37. Expanded TOCSY (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/TOCSY

```

Current Data Parameters
NAME      oct25-snu-chj
EXPNO     23
PROCNO    1

F2 - Acquisition Parameters
Date_     20051028
Time      13.26
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPROG   mlevph
TD         2048
SOLVENT   DMSO
NS         16
DS         16
SMH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         90.5
DW         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00006505 sec
D1         2.00000000 sec
D9         0.06000000 sec
d12        0.00002000 sec
FACTOR1    6
IN0        0.00015149 sec
I1         36
MCREST     0.00000000 sec
MCWRK     1.00000000 sec
SCALEF     6
STICNT     64

***** CHANNEL f1 *****
NUC1       1H
P1         10.50 usec
p5         16.67 usec
P6         25.00 usec
p7         50.00 usec
P17        1500.00 usec
PL1        -5.00 dB
PL10       2.54 dB
SFO1       600.1330005 MHz

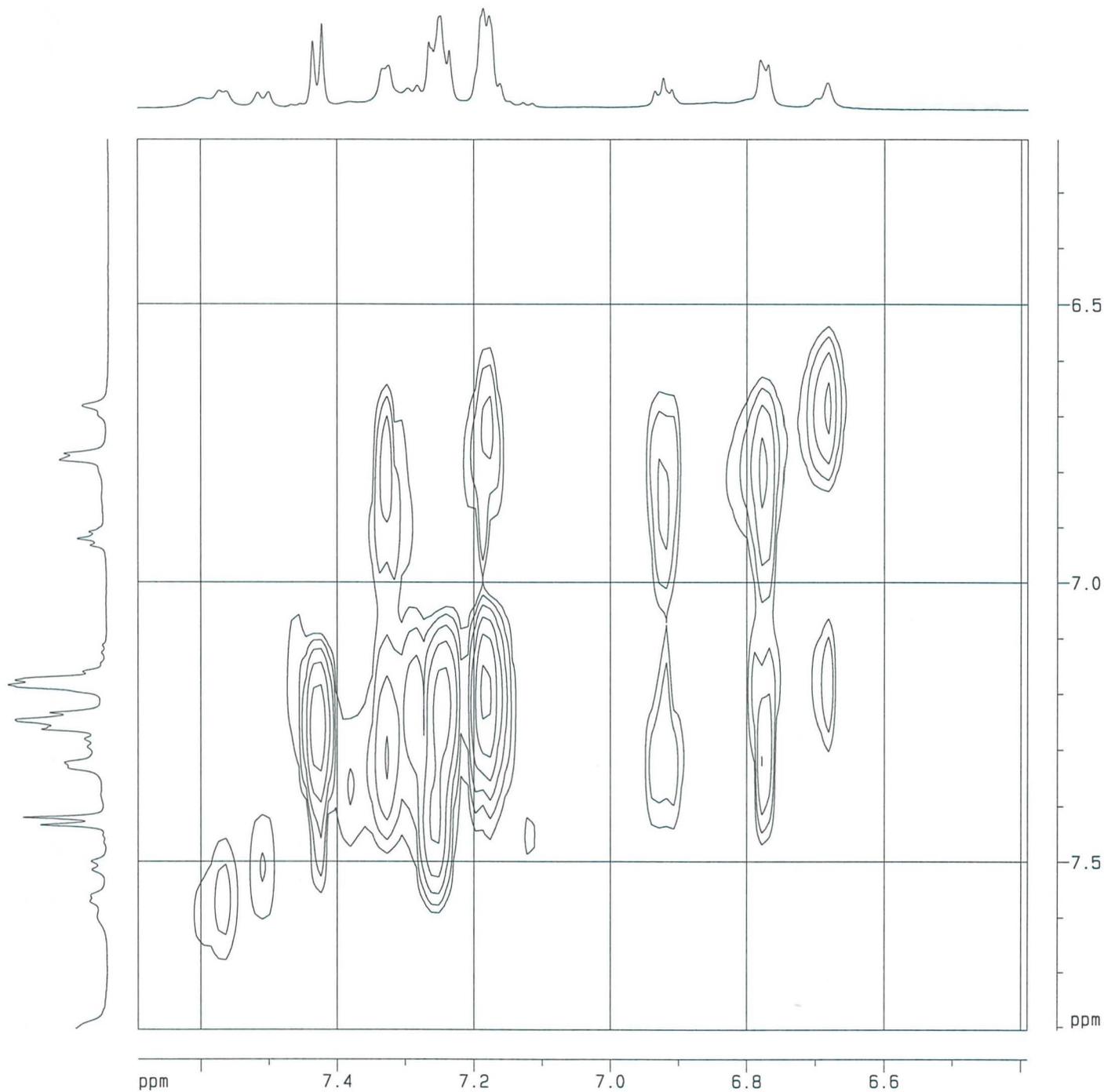
F1 - Acquisition parameters
ND0        1
TD         128
SFO1       600.133 MHz
FIDRES     51.571911 Hz
SW         11.000 ppm
FnMODE     States-TPPI

F2 - Processing parameters
SI         1024
SF         600.1300052 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300052 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      10.383 ppm
F2L0       6231.30 Hz
F2PHI      5.863 ppm
F2H1       3518.63 Hz
F1PLO      5.228 ppm
F1L0       3137.29 Hz
F1PHI      0.437 ppm
F1H1       262.16 Hz
F2PPMCM    0.30134 ppm/cm
F2HZCM     180.84491 Hz/cm
F1PPMCM    0.31939 ppm/cm
F1HZCM     191.67560 Hz/cm
    
```

S38. Expanded TOCSY (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/TOCSY

```

Current Data Parameters
NAME      oct25-anu-chj
EXPNO     23
PROCNO    1

F2 - Acquisition Parameters
Date_     20051028
Time      13.26
INSTRUM   spect
PROBHD    5 mm CPTXI 4H/
PULPROG   mjvph
TD         2048
SOLVENT   DMSO
NS         16
DS         16
SMH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         90.5
DM         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00006506 sec
D1         2.00000000 sec
D9         0.06000000 sec
d12       0.00002000 sec
FACTOR1    5
IN0        0.00015149 sec
I1         36
MCREST    0.00000000 sec
MCMRK     1.00000000 sec
SCALEF    6
ST1CNT     64

***** CHANNEL f1 *****
NUC1       1H
P1         10.50 usec
p5         16.67 usec
P6         25.00 usec
p7         50.00 usec
P17        1500.00 usec
PL1        -5.00 dB
PL10       2.54 dB
SFO1       600.1330005 MHz

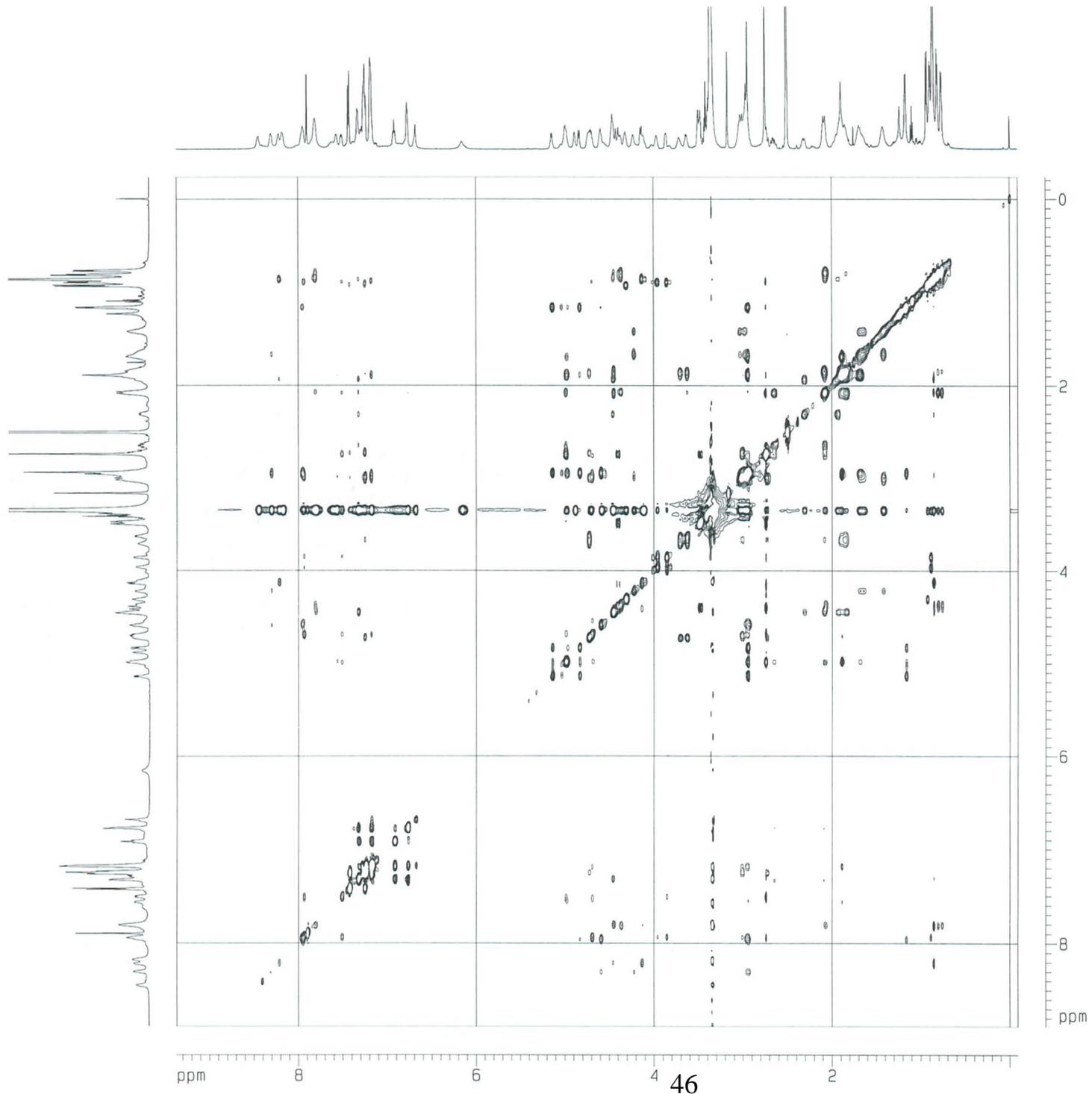
F1 - Acquisition parameters
ND0        1
TD         128
SFO1       600.133 MHz
FIDRES     51.571911 Hz
SM         11.000 ppm
FnMODE     States-TPPI

F2 - Processing parameters
SI         1024
SF         600.1300052 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300052 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      7.693 ppm
F2LO       4616.62 Hz
F2PHI      6.390 ppm
F2H1       3835.11 Hz
F1PLO      7.806 ppm
F1LO       4684.45 Hz
F1PHI      6.205 ppm
F1H1       3723.92 Hz
F2PPMCM    0.08682 ppm/cm
F2HZCM     52.10056 Hz/cm
F1PPMCM    0.10670 ppm/cm
F1HZCM     64.03512 Hz/cm
    
```

S39. NOESY (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/NOESY

Current Data Parameters  
 NAME ju104-snu-chj  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20050705  
 Time 12.04  
 INSTRUM spect  
 PROBHD 5 mm CPTXI 1H/  
 PULPROG noesyph  
 TD 4096  
 SOLVENT DMSO  
 NS 8  
 DS 8  
 SWH 6613.757 Hz  
 FIDRES 1.614687 Hz  
 AQ 0.3097832 sec  
 RG 28.5  
 OW 75.600 usec  
 DE 6.00 usec  
 TE 298.0 K  
 d0 0.0006250 sec  
 D1 1.0000000 sec  
 DB 0.8999998 sec  
 INO 0.00015149 sec  
 MCREST 0.0000000 sec  
 MCWRK 0.5000000 sec  
 ST1CNT 128

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 1H  
 P1 10.40 usec  
 PL1 -5.00 dB  
 SFO1 600.1330006 MHz

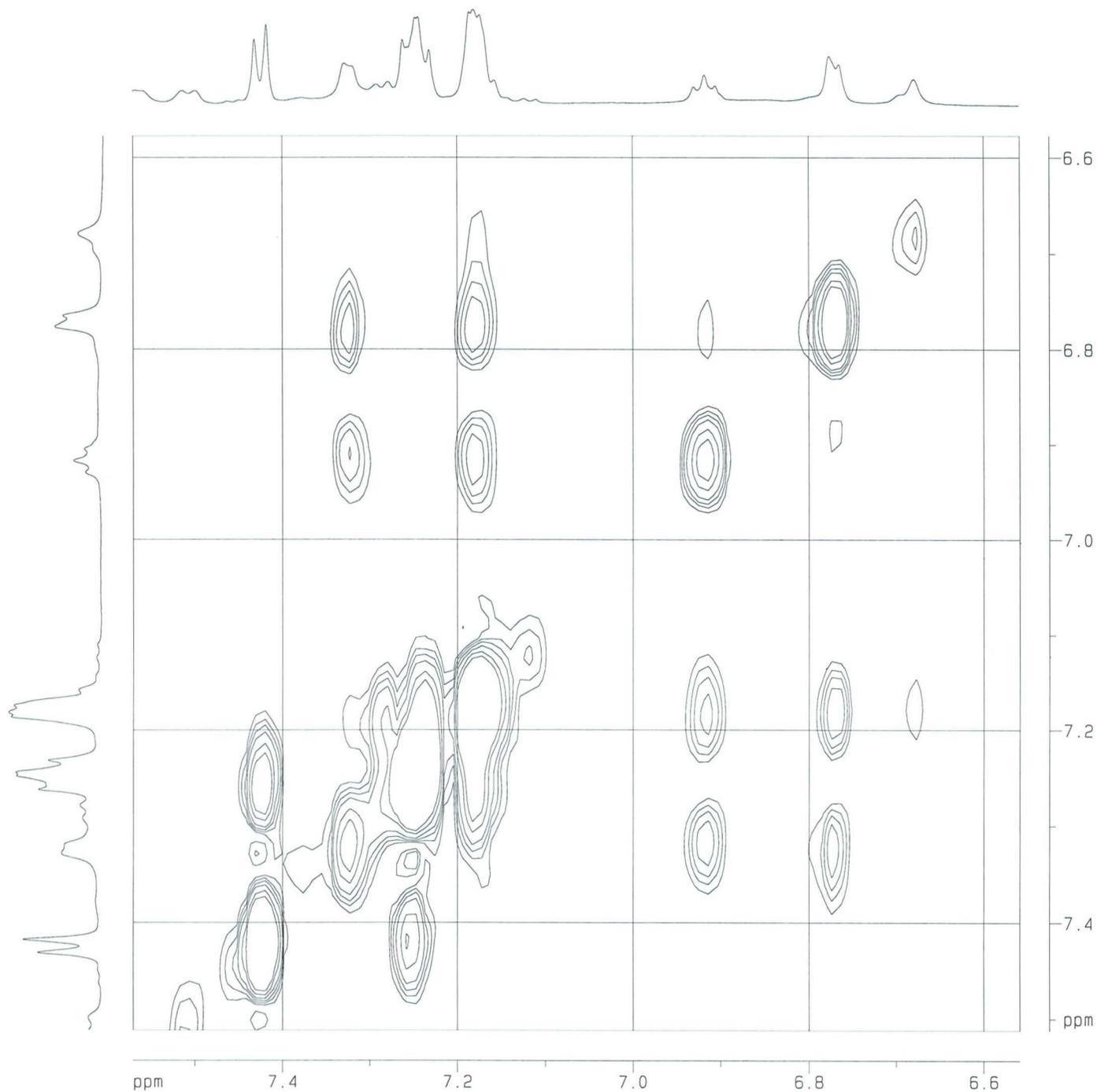
F1 - Acquisition parameters  
 NDO 1  
 TD 177  
 SFO1 600.133 MHz  
 FIDRES 37.294941 Hz  
 SW 11.000 ppm  
 FhMODE States-TPPI

F2 - Processing parameters  
 SI 1024  
 SF 600.1300074 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 MC2 States-TPPI  
 SF 600.1300074 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 9.368 ppm  
 F2LO 5621.96 Hz  
 F2PHI -0.092 ppm  
 F2HI -55.28 Hz  
 F1PLO 8.887 ppm  
 F1LO 5333.33 Hz  
 F1PHI -0.233 ppm  
 F1HI -139.74 Hz  
 F2PPMCM 0.63067 ppm/cm  
 F2HZCM 378.48251 Hz/cm  
 F1PPMCM 0.60799 ppm/cm  
 F1HZCM 364.87131 Hz/cm

S40. Expanded NOESY (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/NOESY

```

Current Data Parameters
NAME      ju104-snu-cnj
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_    20050705
Time     12.04
INSTRUM  spect
PROBHD   5 mm CPTXI 1H/
PULPROG  noesyph
TD       4096
SOLVENT  DMSO
NS       8
DS       8
SWH      6613.757 Hz
FIDRES   1.614687 Hz
AQ       0.3097832 sec
RG       28.5
DM       75.600 usec
DE       6.00 usec
TE       298.0 K
d0       0.00006250 sec
D1       1.00000000 sec
D8       0.89999998 sec
IN0      0.00015149 sec
MCREST   0.00000000 sec
MCWRK    0.50000000 sec
ST1CNT   128

***** CHANNEL f1 *****
NUC1     1H
P1       10.40 usec
PL1      -5.00 dB
SFO1     600.1330006 MHz

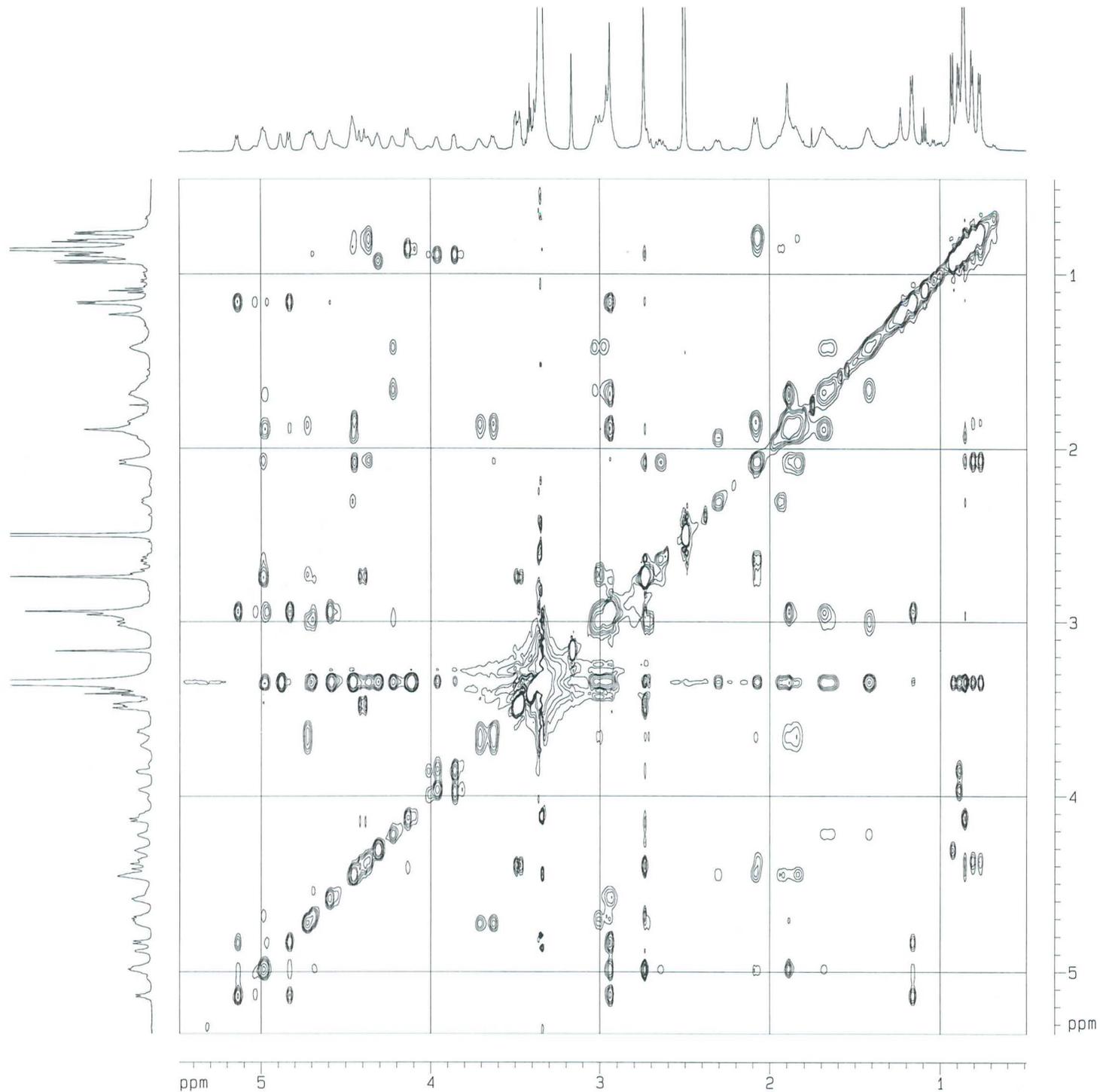
F1 - Acquisition parameters
ND0      1
TD       177
SFO1     600.133 MHz
FIDRES   37.294941 Hz
SK       11.000 ppm
FnMODE   States-TPPI

F2 - Processing parameters
SI       1024
SF       600.1300074 MHz
WDW      QSINE
SSB      2
LB       0.00 Hz
GB       0
PC       1.00

F1 - Processing parameters
SI       1024
MC2      States-TPPI
SF       600.1300074 MHz
WDW      QSINE
SSB      2
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2PLO    7.571 ppm
F2LO     4543.35 Hz
F2PHI    6.559 ppm
F2HI     3936.23 Hz
F1PLO    7.512 ppm
F1LO     4508.17 Hz
F1PHI    6.577 ppm
F1HI     3947.33 Hz
F2PPMCM  0.06744 ppm/cm
F2HZCM   40.47481 Hz/cm
F1PPMCM  0.06230 ppm/cm
F1HZCM   37.38963 Hz/cm
    
```

S41. Expanded NOESY (600 MHz) spectrum of halicyclindramide G (2) in DMSO-d6



5/NOESY

```

Current Data Parameters
NAME      ju104-snu-ch]
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      12.04
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   noesyph
TD         4096
SOLVENT   DMSO
NS         8
DS         8
SWH        6613.757 Hz
FIDRES     1.614687 Hz
AQ         0.3097832 sec
RG         28.5
DW         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00006250 sec
D1         1.00000000 sec
D8         0.89999998 sec
INO        0.00015149 sec
MCREST     0.00000000 sec
MCHWK      0.50000000 sec
ST1CNT     128

===== CHANNEL f1 =====
NUC1       1H
P1         10.40 usec
PL1        -5.00 dB
SFO1       600.1330006 MHz

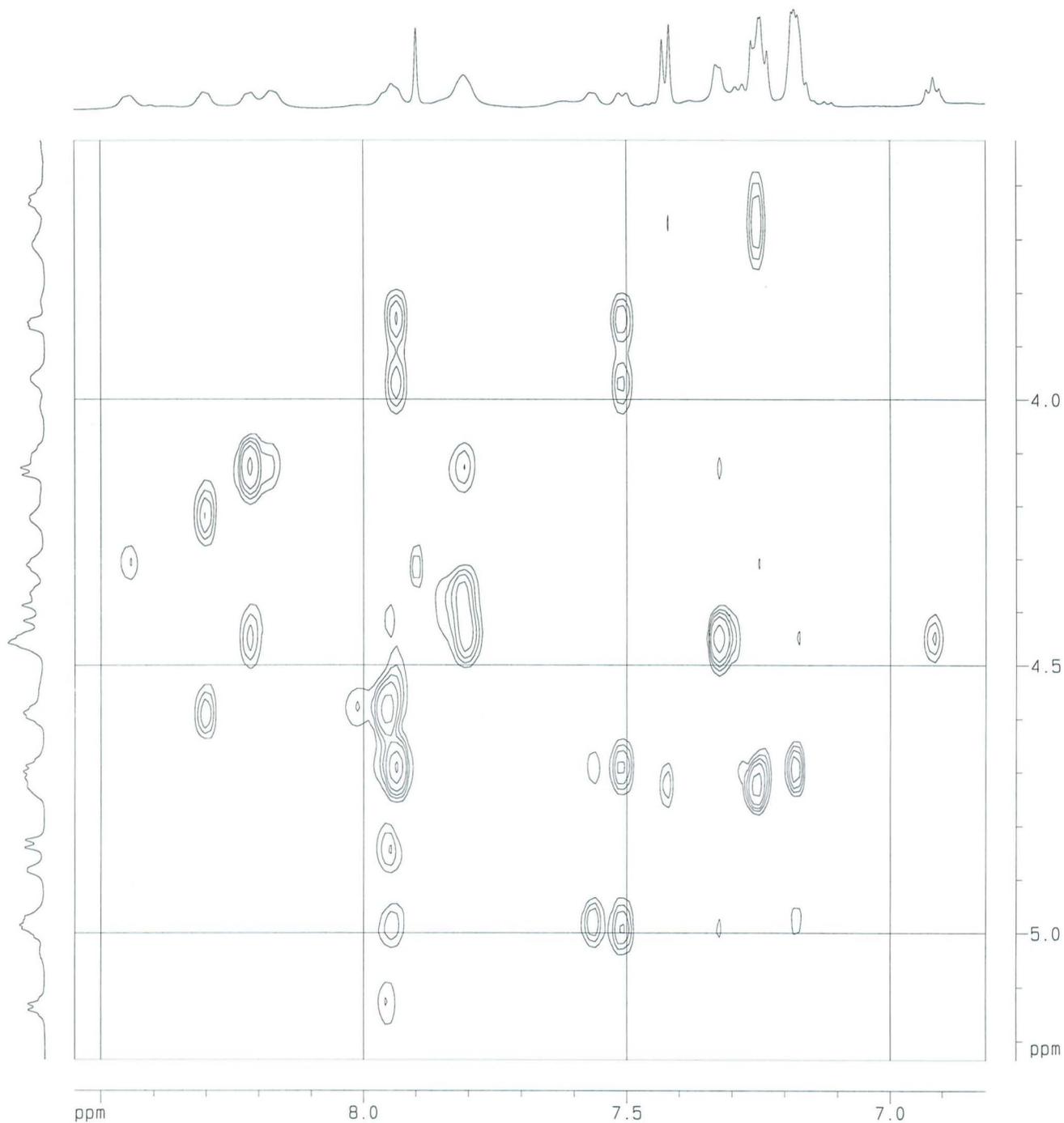
F1 - Acquisition parameters
ND0         1
TD          177
SFO1        600.133 MHz
FIDRES      37.294941 Hz
SW          11.000 ppm
FnMODE      States-TPPI

F2 - Processing parameters
SI          1024
SF          600.1300074 MHz
WDW         QSINE
SSB         2
LB          0.00 Hz
GB          0
PC          1.00

F1 - Processing parameters
SI          1024
MC2         States-TPPI
SF          600.1300074 MHz
WDW         QSINE
SSB         2
LB          0.00 Hz
GB          0

2D NMR plot parameters
CX2         15.00 cm
CX1         15.00 cm
F2PLO       5.483 ppm
F2LO        3290.35 Hz
F2PHI       0.489 ppm
F2HI        293.49 Hz
F1PLO       5.353 ppm
F1LO        3212.43 Hz
F1PHI       0.444 ppm
F1HI        266.38 Hz
F2PPMCM     0.33291 ppm/cm
F2HZCM      199.79056 Hz/cm
F1PPMCM     0.32727 ppm/cm
F1HZCM      196.40302 Hz/cm
    
```

S42. Expanded NOESY (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/NOESY

```

Current Data Parameters
NAME      ju104-snu-ch
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      12.04
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   noesyph
TD         4096
SOLVENT   DMSO
NS         8
DS         8
SWH        6613.757 Hz
FIDRES     1.614687 Hz
AQ         0.3097832 sec
RG         28.5
OW         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00006250 sec
D1         1.00000000 sec
DB         0.89999998 sec
IN0        0.00015149 sec
MCREST    0.00000000 sec
MCWRK     0.50000000 sec
ST1CNT    128

***** CHANNEL f1 *****
NUC1      1H
P1        10.40 usec
PL1       -5.00 dB
SFO1      600.1330006 MHz

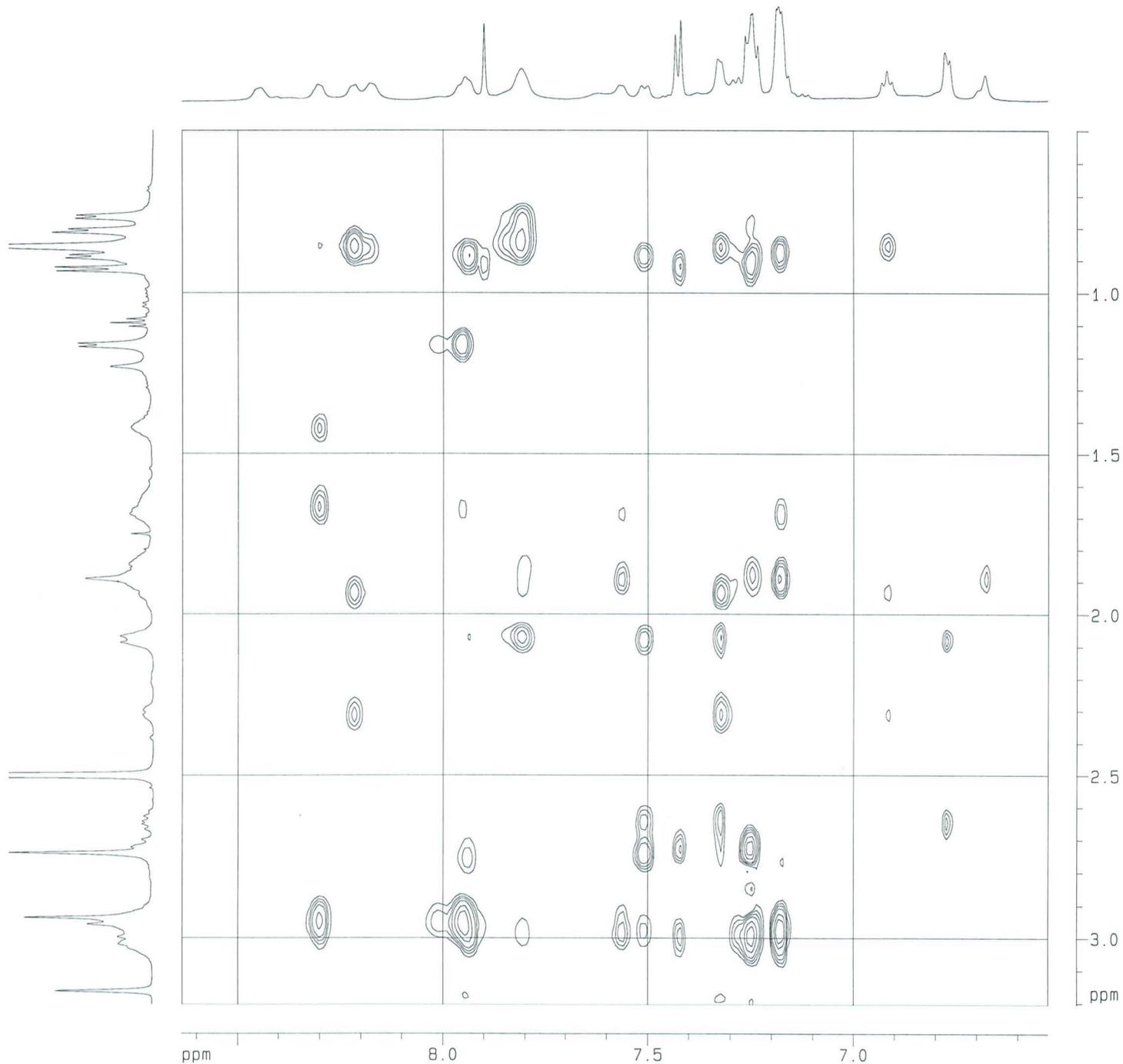
F1 - Acquisition parameters
ND0       1
TD        177
SFO1      600.133 MHz
FIDRES    37.294941 Hz
SW        11.000 ppm
FnmODE    States-TPPI

F2 - Processing parameters
SI        1024
SF        600.1300074 MHz
WDW       QSINE
SSB       2
LB        0.00 Hz
GB        0
PC        1.00

F1 - Processing parameters
SI        1024
MC2       States-TPPI
SF        600.1300074 MHz
WDW       QSINE
SSB       2
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     8.550 ppm
F2LO      5131.10 Hz
F2PHI     6.817 ppm
F2HI      4091.24 Hz
F1PLO     5.235 ppm
F1LO      3141.52 Hz
F1PHI     3.516 ppm
F1HI      2110.08 Hz
F2PPMCM   0.11551 ppm/cm
F2HZCM    69.32387 Hz/cm
F1PPMCM   0.11458 ppm/cm
F1HZCM    68.76255 Hz/cm
    
```

S43. Expanded NOESY (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/NOESY

```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      12.04
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   noesyph
TD         4096
SOLVENT   DMSO
NS         8
DS         8
SWH        6613.757 Hz
FIDRES     1.614687 Hz
AQ         0.3097832 sec
RG         28.5
DM         75.800 usec
DE         6.00 usec
TE         298.0 K
d0         0.00006250 sec
D1         1.00000000 sec
D8         0.89999998 sec
IN0        0.00015149 sec
MCREST    0.00000000 sec
MCWRK     0.50000000 sec
ST1CNT    128

===== CHANNEL f1 =====
NUC1      1H
P1        10.40 usec
PL1       -5.00 dB
SFO1      600.1330006 MHz

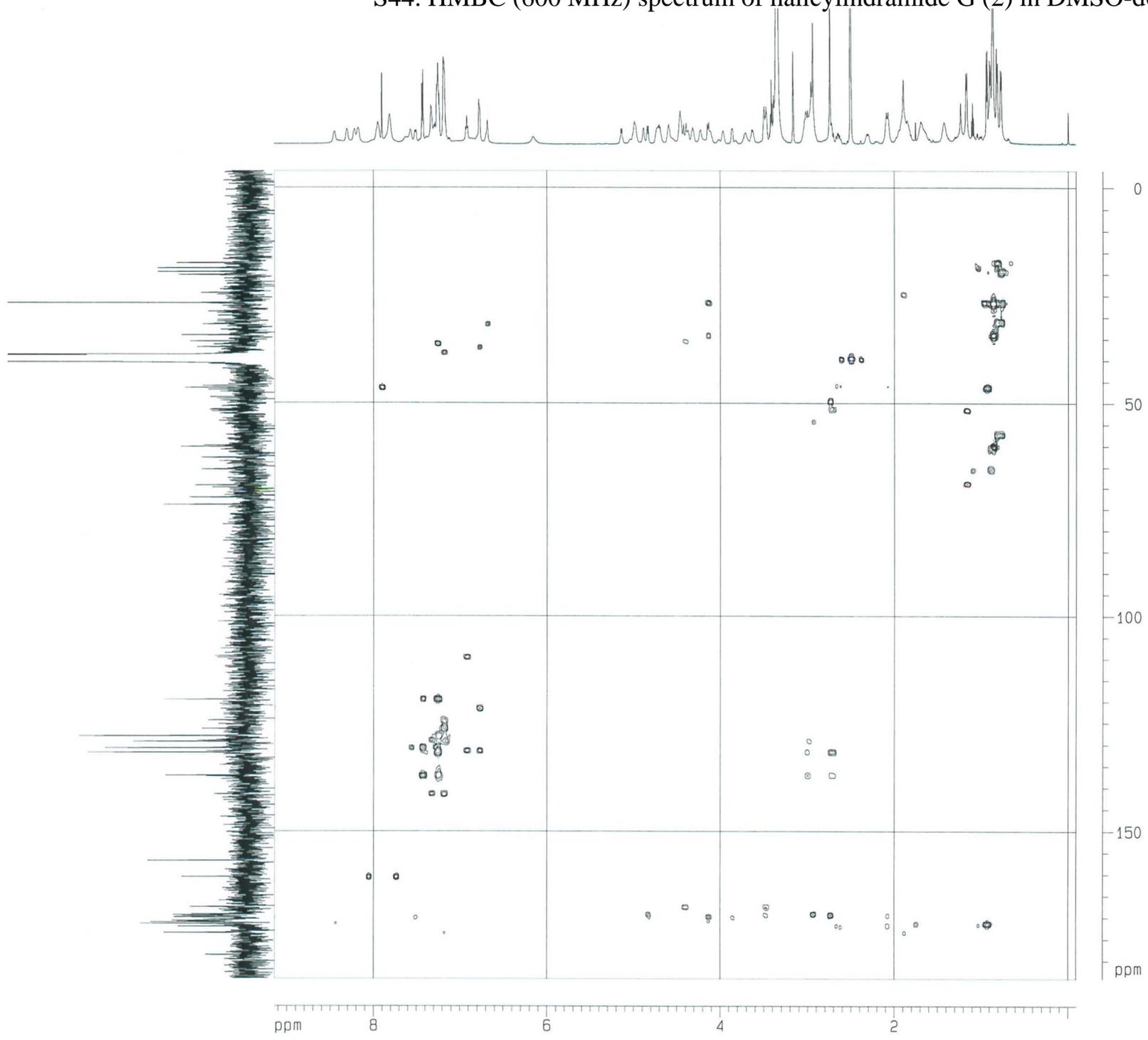
F1 - Acquisition parameters
ND0       1
TD        177
SFO1      600.133 MHz
FIDRES    37.294941 Hz
SK        11.000 ppm
FnMODE    States-TPPI

F2 - Processing parameters
SI        1024
SF        600.1300074 MHz
WDW       QSINE
SSB       2
LB        0.00 Hz
GB        0
PC        1.00

F1 - Processing parameters
SI        1024
MC2       States-TPPI
SF        600.1300074 MHz
WDW       QSINE
SSB       2
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     6.636 ppm
F2LO      5182.77 Hz
F2PHI     6.527 ppm
F2HI      3916.85 Hz
F1PLO     3.205 ppm
F1LO      1923.13 Hz
F1PHI     0.498 ppm
F1HI      298.62 Hz
F2PPMCM   0.14063 ppm/cm
F2HZCM    84.39429 Hz/cm
F1PPMCM   0.18046 ppm/cm
F1HZCM    108.30102 Hz/cm
    
```

S44. HMBC (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/HMBC

```

Current Data Parameters
NAME      jul04-snu-ch)
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20050705
Time     14.51
INSTRUM  spect
PROBHD   5 mm CPTX1 1H/
PULPROG  hmcgpcplndaf
TD       1024
SOLVENT  DMSO
NS       16
DS       4
SWH      6613.757 Hz
FIDRES   6.458747 Hz
AQ       0.0775400 sec
RG       26008
DK       75.600 usec
DE       6.00 usec
TE       298.0 K
CHST2    145.000000
CHST13   10.000000
d0       0.00000300 sec
D1       1.50000000 sec
d2       0.00344828 sec
d6       0.05000000 sec
D16      0.00050000 sec
IN0      0.0001380 sec
MCREST   0.0000000 sec
MCWFK    1.5000000 sec

***** CHANNEL f1 *****
NUC1     1H
P1       10.40 usec
p2       20.80 usec
PL1      -5.00 dB
SFO1     600.1330006 MHz

***** CHANNEL f2 *****
NUC2     13C
P3       15.00 usec
PL2      -6.00 dB
SFO2     150.9194083 MHz

***** GRADIENT CHANNEL *****
GRNAM1   SINE 100
GRNAM2   SINE 100
GRNAM3   SINE 100
GPX1     0.00 %
GPX2     0.00 %
GPX3     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPY3     0.00 %
GPZ1     50.00 %
GPZ2     30.00 %
GPZ3     40.10 %
P16      1000.00 usec

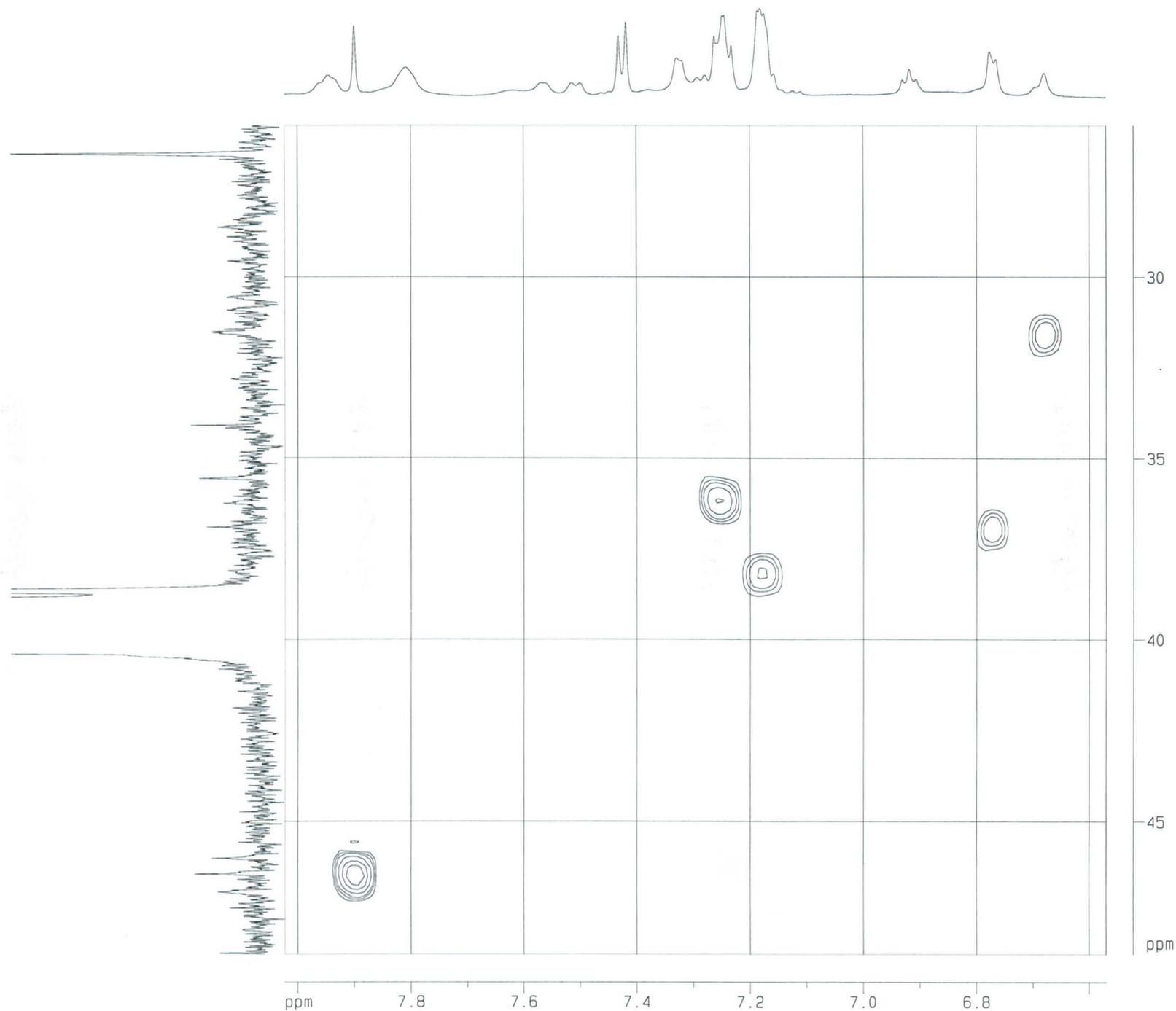
F1 - Acquisition parameters
RG      2
TD      256
SFO1    150.9194 MHz
FIDRES   141.530792 Hz
SW       240.074 ppm
FHMDCOE  GF

F2 - Processing parameters
SI      1024
SF      600.1300074 MHz
WDW     SINE
SSB     0
LB      0.00 Hz
GB      0
PC      1.40

F1 - Processing parameters
SI      1024
MC2     GF
SF      150.9028739 MHz
WDW     SINE
SSB     0
LB      0.00 Hz
GB      0

2D NMR plot parameters
CX2     15.00 cm
CX1     15.00 cm
F2PLG   9.142 ppm
FZLO    5486.33 Hz
F2PHI   -0.092 ppm
FPHI    -55.38 Hz
F1PLG   184.132 ppm
F1LO    27786.09 Hz
F1PHI   -3.915 ppm
F1HI    -590.83 Hz
F2FPMCM 0.61560 ppm/cm
F2FZCM  365.44031 Hz/cm
F1FPMCM 12.53651 ppm/cm
F1FZCM  1891.79504 Hz/cm
    
```

S45. Expanded HMBC (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/HMBC

```

Current Data Parameters
NAME      ju104-sru-ch]
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20050705
Time     14.51
INSTRUM  spect
PROBHD   5 mm CPTX1 1H/
PULPROG  hmbcge1pndqf
TO       1024
SOLVENT  DMSO
NS       16
DS       4
SWH      6613.757 Hz
FIDRES   6.458747 Hz
AQ       0.0775400 sec
RG       28000
DW       75.600 usec
DE       6.00 usec
TE       298.0 K
CHST12   145.0000000
CHST13   10.0000000
d0       0.00000300 sec
D1       1.50000000 sec
d2       0.00344828 sec
d8       0.05000000 sec
O16      0.00000000 sec
IN0      0.00001380 sec
MCREST   0.00000000 sec
MCWRR    1.50000000 sec

***** CHANNEL f1 *****
NUC1     1H
P1       10.40 usec
p2       20.80 usec
PL1      -5.00 dB
SFO1     600.1330074 MHz

***** CHANNEL f2 *****
NUC2     13C
P2       15.00 usec
PL2      -8.00 dB
SFO2     150.9194083 MHz

***** GRADIENT CHANNEL *****
GPNAM1   SINE 100
GPNAM2   SINE 100
GPNAM3   SINE 100
GPX1     0.00 %
GPX2     0.00 %
GPX3     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPY3     0.00 %
GPZ1     50.00 %
GPZ2     30.00 %
GPZ3     40.10 %
P16      1000.00 usec

F1 - Acquisition parameters
NUC      13C
TD       256
SFO1     150.9194 MHz
FIDRES   141.530792 Hz
SW       240.074 ppm
FHMDC    GF

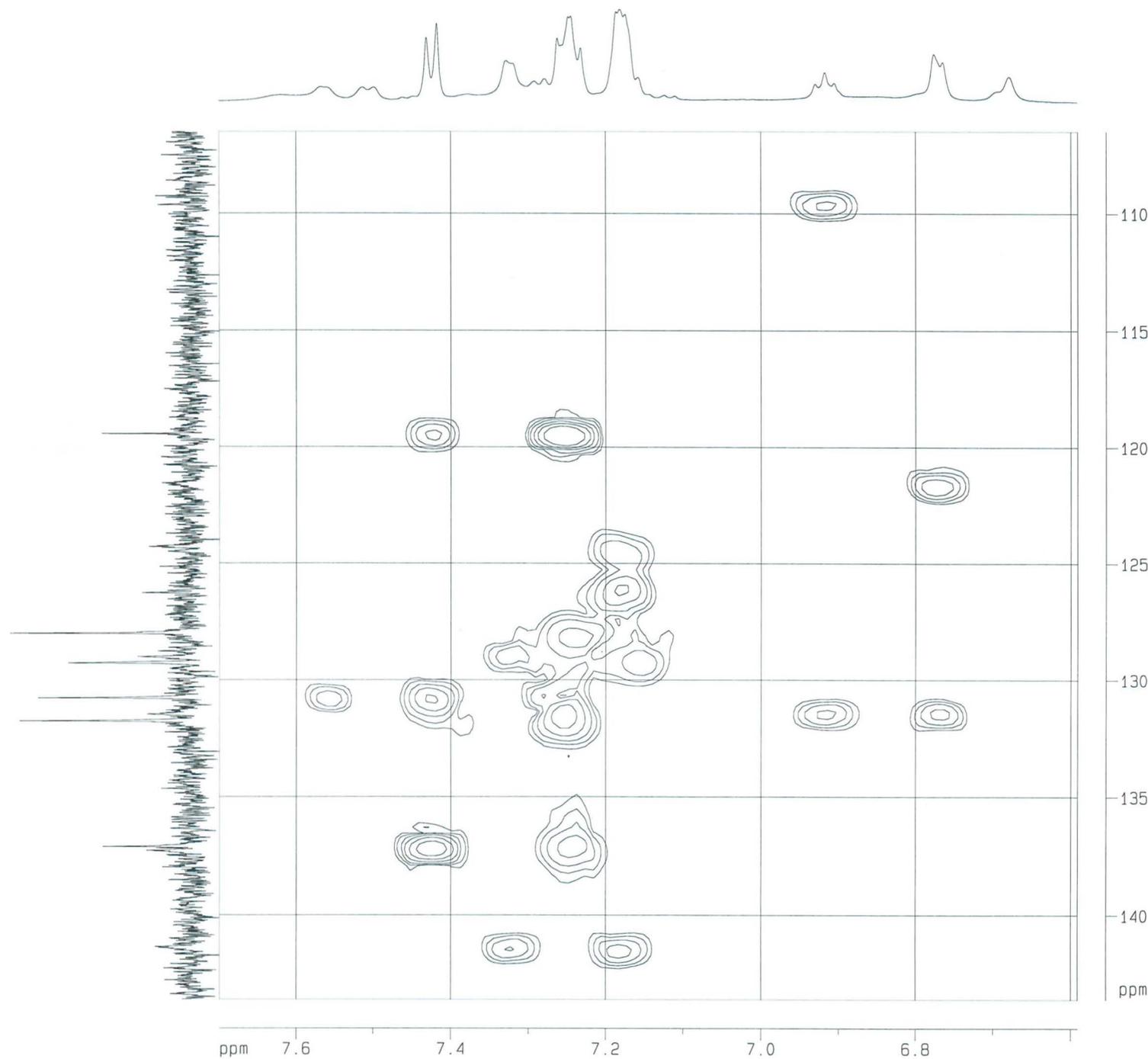
F2 - Processing parameters
SI       1024
SF       600.1330074 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
MC2      GF
SF       150.9028739 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2PL0    8.023 ppm
F2L0     4814.62 Hz
F2PHI    6.570 ppm
F2HI     3942.60 Hz
F2PL0    48.607 ppm
F2L0     7334.89 Hz
F2PHI    25.863 ppm
F2HI     3902.77 Hz
F2PPMCM 0.09696 ppm/cm
F2HZCM   58.12872 Hz/cm
F1PPMCM  1.51626 ppm/cm
F1HZCM   228.80814 Hz/cm
    
```

S46. Expanded HMBC (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6

5/HMBC



```

Current Data Parameters
NAME          j1104-gnu-cm1
EXPNO         6
PROCNO       1

F2 - Acquisition Parameters
Date_         20050705
Time          14.51
INSTRUM       spect
PROBHD        5 mm CPTX1 1H/
PULPROG       hmcgplndqf
TD            1024
SOLVENT       DMSO
NS            16
DS            4
SWH           6613.757 Hz
FIDRES        6.458747 Hz
AQ            0.0775400 sec
RG            26008
DM            75.600 usec
DE            6.00 usec
TE            298.0 K
CNS12         145.000000
CNS13         10.000000
d0            0.00000300 sec
D1            1.50000000 sec
d2            0.00344828 sec
d3            0.00000000 sec
D16           0.00000000 sec
IND           0.0001360 sec
MCREST        0.00000000 sec
MCHWK         1.50000000 sec

***** CHANNEL f1 *****
NUC1           1H
P1             10.40 usec
p2             20.80 usec
PL1            -6.00 dB
SFO1           600.1330006 MHz

***** CHANNEL f2 *****
NUC2           13C
P3             15.00 usec
PL2            -6.00 dB
SFO2           150.9194083 MHz

***** GRADIENT CHANNEL *****
GPNAM1        SINE 100
GPNAM2        SINE 100
GPNAM3        SINE 100
GPX1           0.00 %
GPX2           0.00 %
GPX3           0.00 %
GPY1           0.00 %
GPY2           0.00 %
GPY3           0.00 %
GPZ1           50.00 %
GPZ2           30.00 %
GPZ3           40.10 %
P16           1000.00 usec

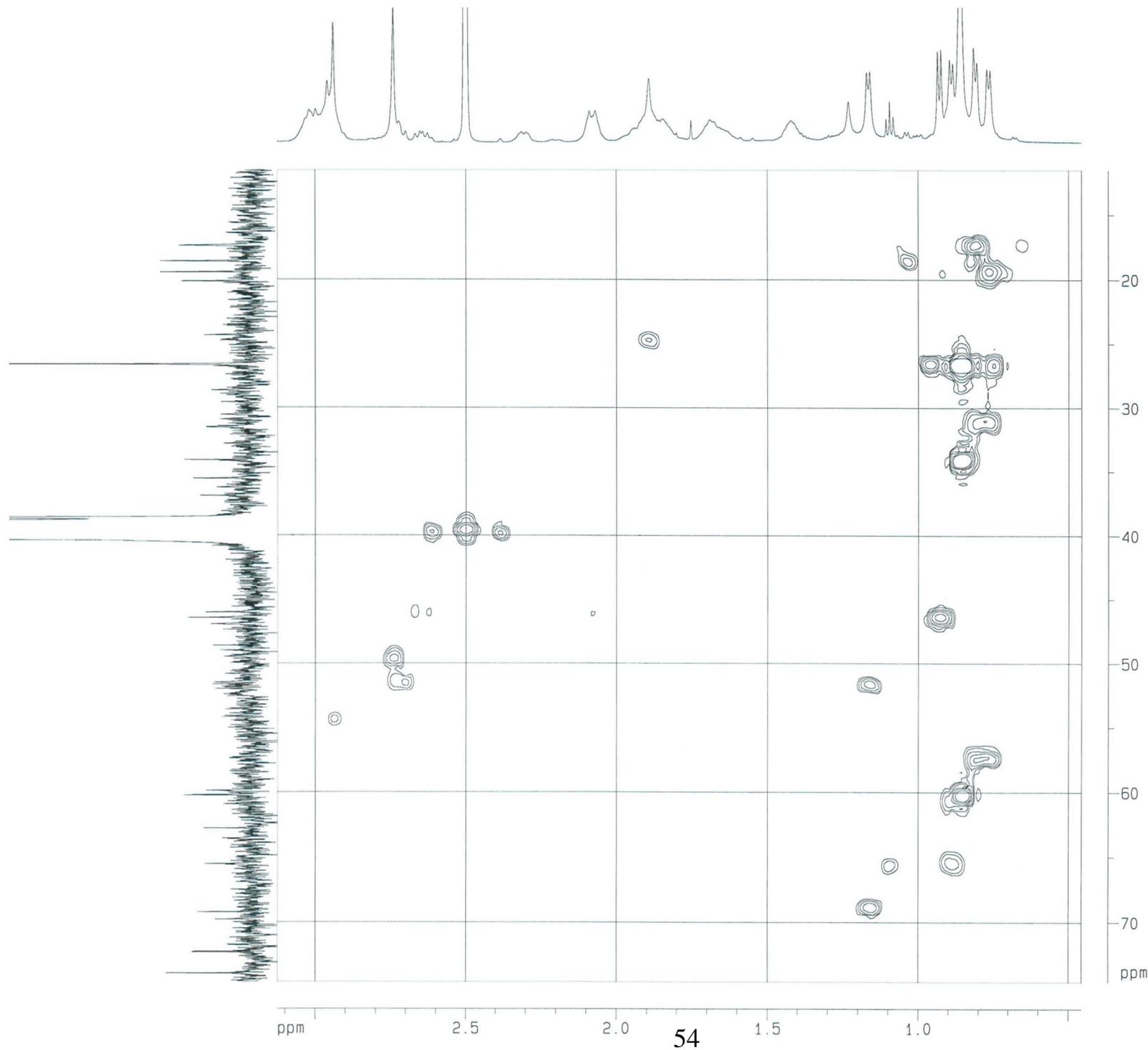
F1 - Acquisition parameters
ND0            2
TD             256
SFO1           150.9194 MHz
FIDRES         141.550792 Hz
SW             240.074 ppm
FHMDCOE        GF

F2 - Processing parameters
SI             1024
SF             600.1300074 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0
PC             1.40

F1 - Processing parameters
SI             1024
MC2            GF
SF             150.9028739 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0

2D NMR plot parameters
CX2            15.00 cm
CX1            15.00 cm
F2PLD          7.700 ppm
F2LD           4620.85 Hz
F2PHI          6.591 ppm
F2H1           3955.60 Hz
F1PLD          143.568 ppm
F1LO           21664.88 Hz
F1PHI         106.528 ppm
F1H1           16074.42 Hz
F2FPMCM        0.07398 ppm/cm
F2HZCM         44.35007 Hz/cm
F1FPMCM        2.46979 ppm/cm
F1HZCM         372.69775 Hz/cm
    
```

S47. Expanded HMBC (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/HMBC

```

Current Data Parameters
NAME      jul04-sru-01
EXPNO    5
PROCNO   1

F2 - Acquisition Parameters
Date_    20050705
Time     14.51
INSTRUM  spect
PROBHD   5 mm CPTXI 1H/
PULPROG  hmcgpgndgf
TD       1024
SOLVENT  DMSO
NS       16
DS       4
SWH      6613.757 Hz
FIDRES   6.458747 Hz
AQ       0.0775408 sec
RG       26008
Dw       75.600 usec
DE       6.00 usec
TE       298.0 K
CST2     145.000000
CNST13   10.000000
d0       0.00000300 sec
D1       1.50000000 sec
D2       0.00344828 sec
d5       0.00000000 sec
D16      0.00020000 sec
IN0      0.0001380 sec
MCREST   0.00000000 sec
MCHRR    1.50000000 sec

***** CHANNEL f1 *****
NUC1     1H
P1       10.40 usec
P2       20.80 usec
FL1      -5.00 dB
SFO1     600.1330006 MHz

***** CHANNEL f2 *****
NUC2     13C
P3       15.00 usec
P4       -6.00 dB
SFO2     150.9194083 MHz

***** GRADIENT CHANNEL *****
GPNAM1   SINE 100
GPNAM2   SINE 100
GPNAM3   SINE 100
GPX1     0.00 %
GPX2     0.00 %
GPX3     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPY3     0.00 %
GR1      50.00 %
GR2      30.00 %
GR3      40.10 %
P16      1000.00 usec

F1 - Acquisition parameters
ND0      2
TD       256
SFO1     150.9194 MHz
FIDRES   141.530792 Hz
SW       240.074 ppm
FRMDCOE  GF

F2 - Processing parameters
SI       1024
SF       600.1300074 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
MC2      GF
SF       150.9028739 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2PLO    3.126 ppm
F2LO     1875.89 Hz
F2PHI    0.457 ppm
F2HI     274.12 Hz
F1PLO    74.633 ppm
F1LO     11262.37 Hz
F1PHI    11.560 ppm
F1HI     1744.42 Hz
F2PRMCM 0.17784 ppm/cm
F2HZCM   106.78461 Hz/cm
F1PRMCM  4.20489 ppm/cm
F1HZCM   634.52879 Hz/cm
    
```

S48. Expanded HMBC (600 MHz) spectrum of halicyclindramide G (2) in DMSO-d6

5/HMBC

```

Current Data Parameters
NAME          jul04-snu-chn
EXPNO         5
PROCNO       1

F2 - Acquisition Parameters
Date_         20050705
Time          14.51
INSTRUM       spect
PROBHD        5 mm CPTX1 1H/
PULPROG       hmbcgp1rndqf
TD            1024
SOLVENT       DMSO
NS            16
DS            4
SWH           6613.757 Hz
FIDRES        6.458747 Hz
AQ            0.0775400 sec
RG            26300
DM            75.600 usec
DE            6.00 usec
TE            298.0 K
CNST2         145.000000
CNST13        10.000000
d0            0.0000000 sec
d1            1.5000000 sec
d2            0.0034828 sec
d6            0.0500000 sec
D16           0.0000000 sec
IN0           0.0001980 sec
MCREST        0.0000000 sec
MCNPRK        1.5000000 sec

***** CHANNEL f1 *****
NUC1          1H
P1            10.40 usec
p2            20.80 usec
PL1           -5.00 dB
SFO1          600.1330006 MHz

***** CHANNEL f2 *****
NUC2          13C
P3            15.00 usec
PL2           -5.00 dB
SFO2          150.9194083 MHz

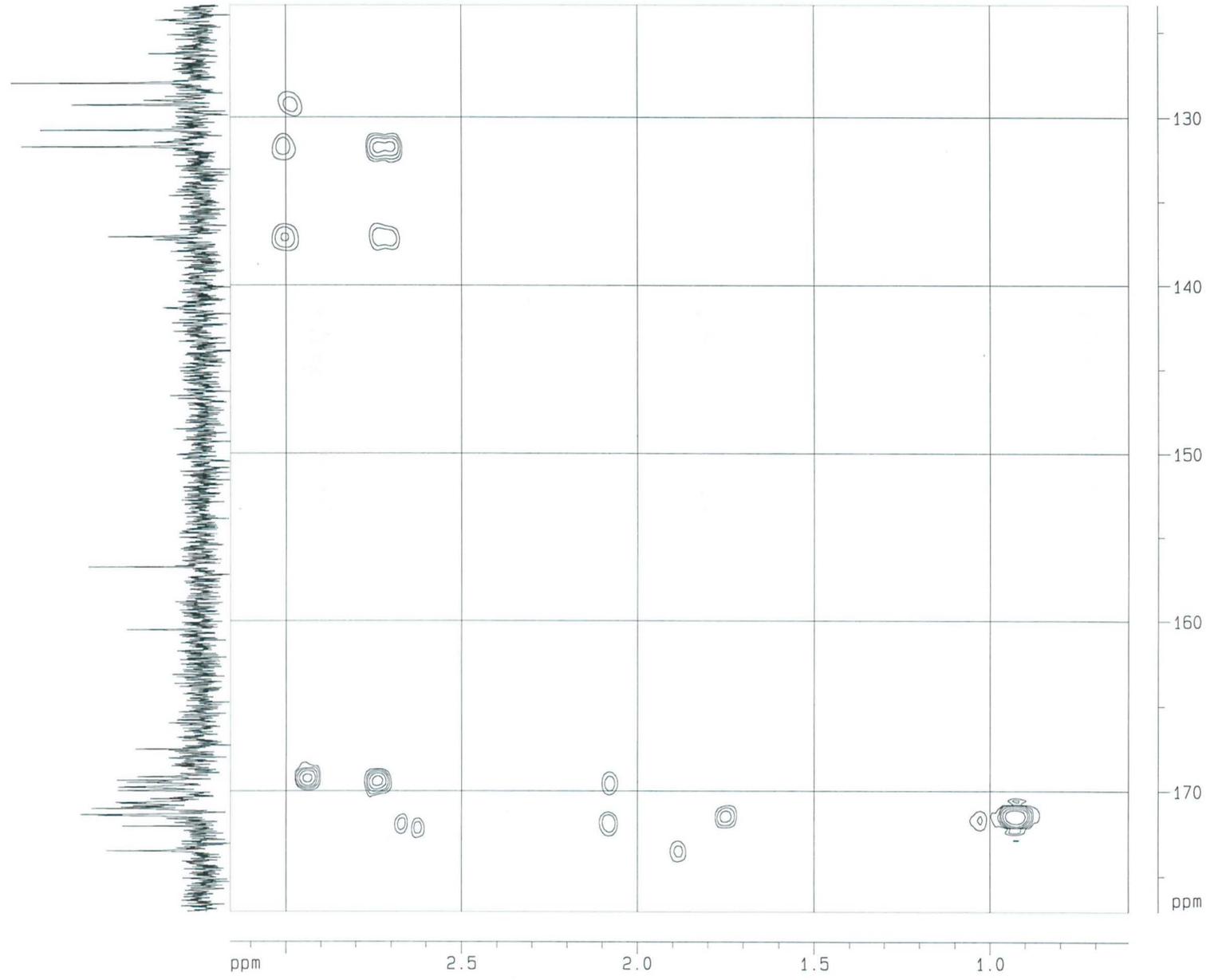
***** GRADIENT CHANNEL *****
GPNAM1        SINE 100
GPNAM2        SINE 100
GPNAM3        SINE 100
GPX1           0.00 %
GPX2           0.00 %
GPX3           0.00 %
GPY1           0.00 %
GPY2           0.00 %
GPY3           0.00 %
GRZ1           50.00 %
GRZ2           30.00 %
GRZ3           40.10 %
P16           1000.00 usec

F1 - Acquisition parameters
NU0            0
TD             256
SFO1           150.9194 MHz
FIDRES         141.530792 Hz
SW             240.074 ppm
FHM0DE         GF

F2 - Processing parameters
SI             1024
SF             600.1300074 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0
PC             1.40

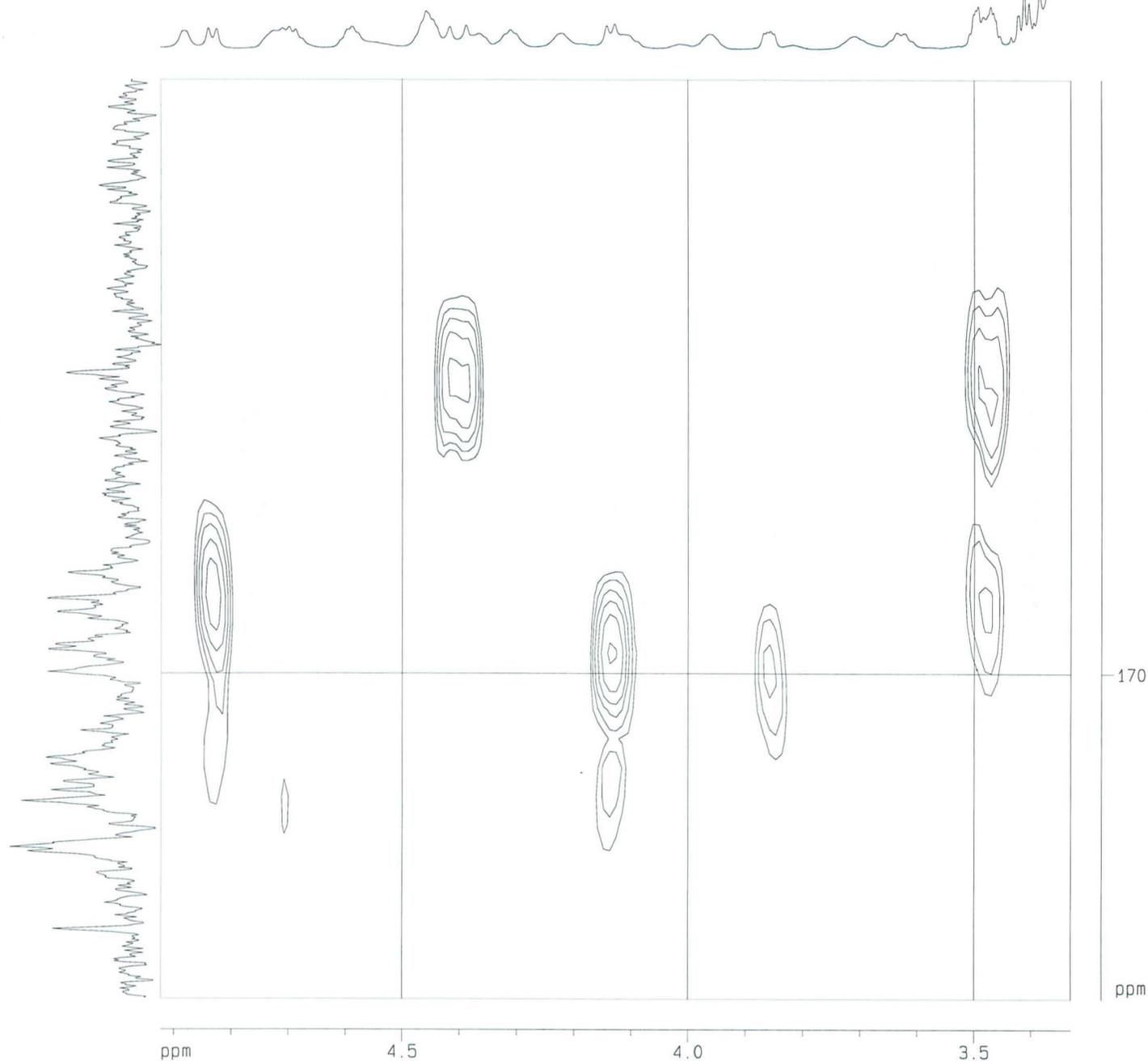
F1 - Processing parameters
SI             1024
MC2            GF
SF             150.9028739 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0

2D NMR plot parameters
CX2            15.00 cm
CX1            15.00 cm
F2PL0          3.158 ppm
F2L0           1895.26 Hz
F2PHI          0.607 ppm
F2FH1          364.54 Hz
F1PL0          177.098 ppm
F1L0           26724.61 Hz
F1PH1          123.404 ppm
F1HI           18821.97 Hz
F2PPMCH        0.17004 ppm/cm
F2HZCM         102.04819 Hz/cm
F1PPMCM        3.57953 ppm/cm
F1HZCM         540.17956 Hz/cm
    
```



S49. Expanded HMBC (600 MHz) spectrum of halicyclindramide G (2) in DMSO-d6

5/HMBC



Current Data Parameters  
 NAME ju104-sru-chj  
 EXPNO 6  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20050705  
 Time 14.51  
 INSTRUM spect  
 PROBHD 5 mm CPTX1 1H/  
 PULPROG hmcgpp1ndqf  
 TO 1024  
 SOLVENT DMSO  
 NS 16  
 DS 4  
 SWH 6613.757 Hz  
 FIDRES 6.458747 Hz  
 AQ 0.0775400 sec  
 RG 26008  
 DW 75.600 usec  
 DE 6.00 usec  
 TE 298.0 K  
 CNST2 145.000000  
 CNST13 10.000000  
 d0 0.0000300 sec  
 D1 1.5000000 sec  
 d2 0.00344829 sec  
 d6 0.0500000 sec  
 D16 0.0000000 sec  
 IN0 0.0001300 sec  
 MCREST 0.0000000 sec  
 MCWFK 1.5000000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NU1 1H  
 P1 10.40 usec  
 p2 20.80 usec  
 PL1 -5.00 dB  
 SFO1 600.133006 MHz

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 NU2 13C  
 P3 15.00 usec  
 PL2 -6.00 dB  
 SFO2 150.9194083 MHz

\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*  
 GPRAM1 SINE 100  
 GPRAM2 SINE 100  
 GPRAM3 SINE 100  
 GPX1 0.00 X  
 GPX2 0.00 X  
 GPX3 0.00 X  
 GPY1 0.00 X  
 GPY2 0.00 X  
 GPY3 0.00 X  
 GPZ1 50.00 X  
 GPZ2 30.00 X  
 GPZ3 40.10 X  
 P16 1000.00 usec

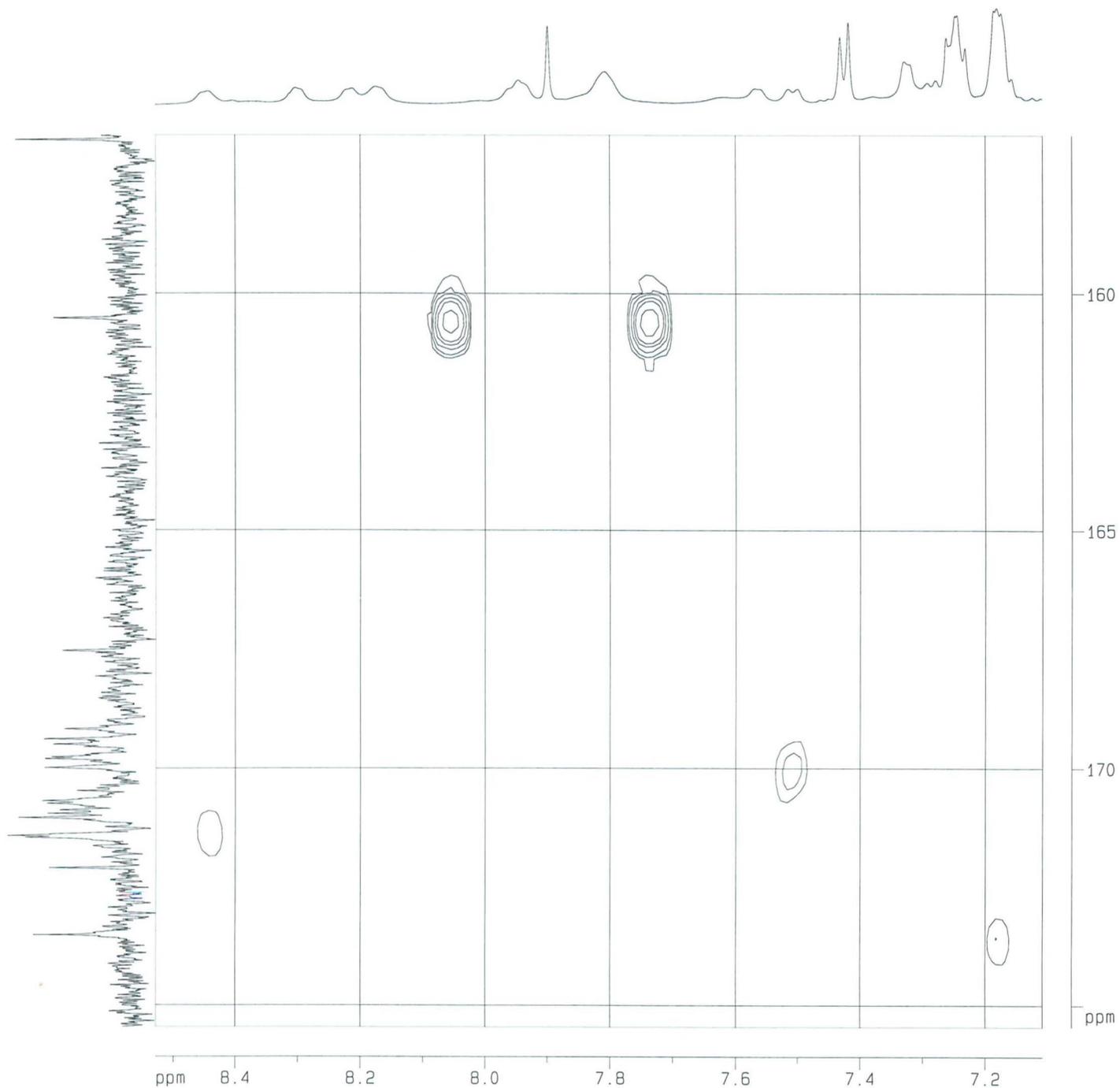
F1 - Acquisition parameters  
 ND0 2  
 TD 256  
 SFO1 150.9194 MHz  
 FIDRES 141.530792 Hz  
 SW 240.074 ppm  
 FMODE QF

F2 - Processing parameters  
 SI 1024  
 SF 600.1300074 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 MC2 QF  
 SF 150.9028739 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 4.923 ppm  
 F2LO 2954.50 Hz  
 F2PHI 3.330 ppm  
 F2H 1998.60 Hz  
 F2PLO 172.543 ppm  
 F1LO 26052.34 Hz  
 F1PHI 165.140 ppm  
 F1H 24920.09 Hz  
 F2PPMCM 0.10619 ppm/cm  
 F2HZCM 63.72631 Hz/cm  
 F1PPMCM 0.50021 ppm/cm  
 F1HZCM 75.48306 Hz/cm

S50. Expanded HMBC (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/HMBC

```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20050705
Time     14.51
INSTRUM  spect
PROBHD   5 mm CPTX1 3H/
PULPROG  hmbcgalpndaf
TD       1024
SOLVENT  DMSO
NS       16
DS       4
SWH      6613.757 Hz
FIDRES   6.458747 Hz
AQ       0.0775400 sec
RG       26008
DM       75.600 usec
DE       6.00 usec
TE       298.0 K
CNST2    145.0000000
CNST13   10.0000000
d0       0.00000000 sec
d1       1.50000000 sec
d2       0.00344828 sec
d6       0.05000000 sec
d16      0.00000000 sec
IN0      0.0001380 sec
MCREST   0.00000000 sec
MCWRK    1.50000000 sec

***** CHANNEL f1 *****
NUC1     1H
P1       10.40 usec
p2       20.80 usec
PL1      -5.00 dB
SFO1     600.1330006 MHz

***** CHANNEL f2 *****
NUC2     13C
P3       15.00 usec
PL2      -8.00 dB
SFO2     150.9194083 MHz

***** GRADIENT CHANNEL *****
GPRAM1   SINE 100
GPRAM2   SINE 100
GPRAM3   SINE 100
GPX1     0.00 %
GPX2     0.00 %
GPX3     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPY3     0.00 %
GPZ1     50.00 %
GPZ2     30.00 %
GPZ3     40.10 %
P15      1000.00 usec

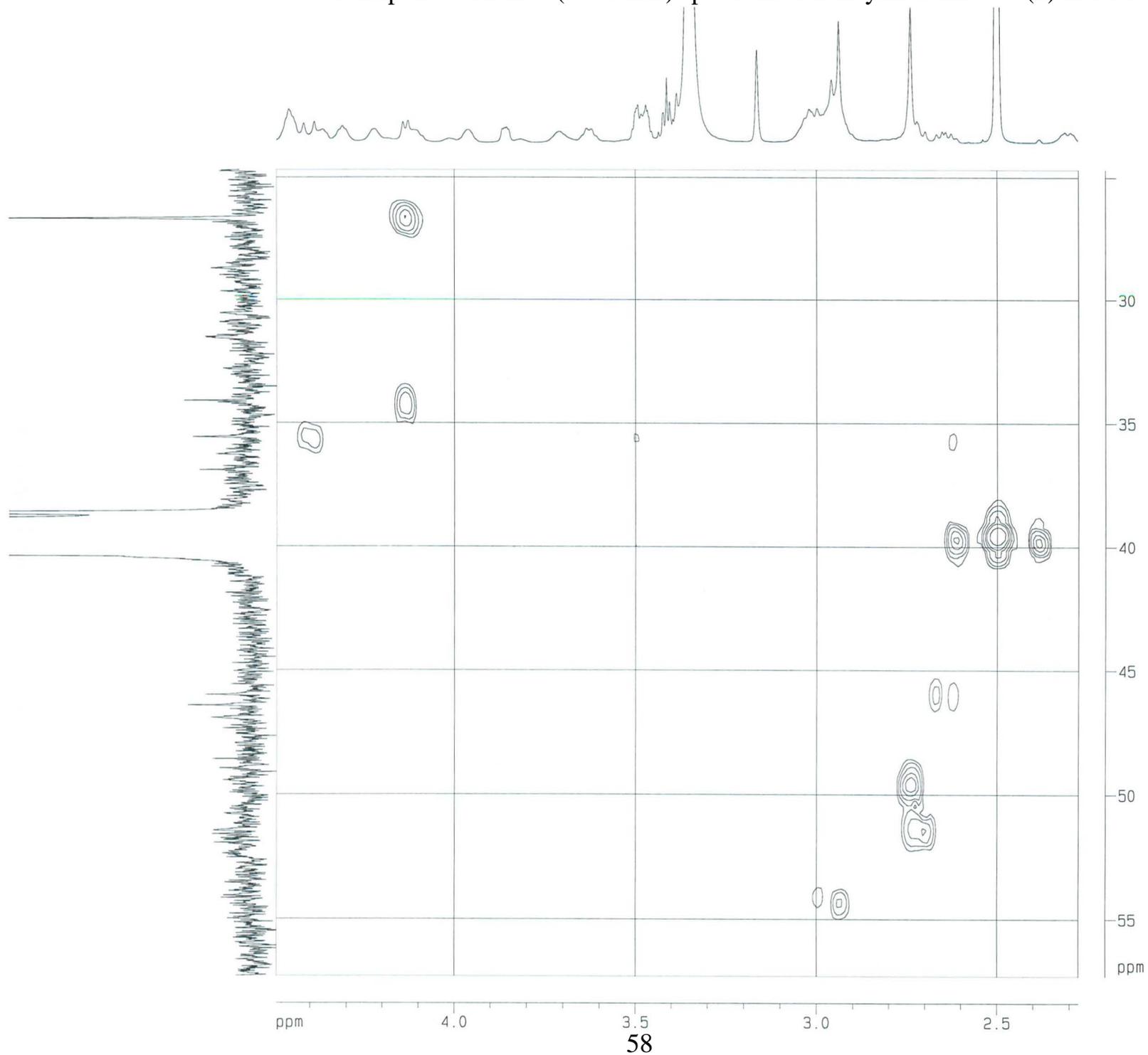
F1 - Acquisition parameters
ND0      2
TD       256
SFO1     150.9194 MHz
FIDRES   141.530792 Hz
SW       240.074 ppm
FHMDOE   GF

F2 - Processing parameters
SI       1024
SF       600.1300074 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
MC2      GF
SF       150.9028739 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2PLO    8.528 ppm
F2LO     5118.18 Hz
F2PHI    7.108 ppm
F2HZ     4285.62 Hz
F1PLO    175.457 ppm
F1LO     26476.93 Hz
F1PHI    156.699 ppm
F1HZ     23646.30 Hz
F2P2MCM  0.09471 ppm/cm
F2HZCM   56.83696 Hz/cm
F1P2MCM  1.25052 ppm/cm
F1HZCM   188.70766 Hz/cm
    
```

S51. Expanded HMBC (600 MHz) spectrum of halicylindramide G (2) in DMSO-d6



5/HMBC

```

Current Data Parameters
NAME      ju[04-snu-ch]
EXPNO     6
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      14.51
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPROG   hmcgplndqf
TD         1024
SOLVENT   DMSO
NS         16
DS         4
SWH        6613.757 Hz
FIDRES     6.458747 Hz
AQ         0.0775400 sec
RG         38608
DW         75.600 usec
DE         6.00 usec
TE         298.0 K
CNS12     145.0000000
CNS13     10.0000000
d0         0.0000000 sec
D1         1.5000000 sec
d2         0.00344828 sec
d5         0.0500000 sec
D16        0.0002000 sec
IN0        0.00001380 sec
MCREST     0.0000000 sec
MCWRK     1.5000000 sec

***** CHANNEL f1 *****
NUC1       1H
P1         10.40 usec
p2         20.80 usec
FL1        -5.00 dB
SFO1       600.1330006 MHz

***** CHANNEL f2 *****
NUC2       13C
P3         15.00 usec
FL2        -6.00 dB
SFO2       150.9194083 MHz

***** GRADIENT CHANNEL *****
GPNAM1     SINE 100
GPNAM2     SINE 100
GPNAM3     SINE 100
GPX1       0.00 %
GPX2       0.00 %
GPX3       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPY3       0.00 %
GPZ1       50.00 %
GPZ2       30.00 %
GPZ3       40.10 %
P16        1000.00 usec

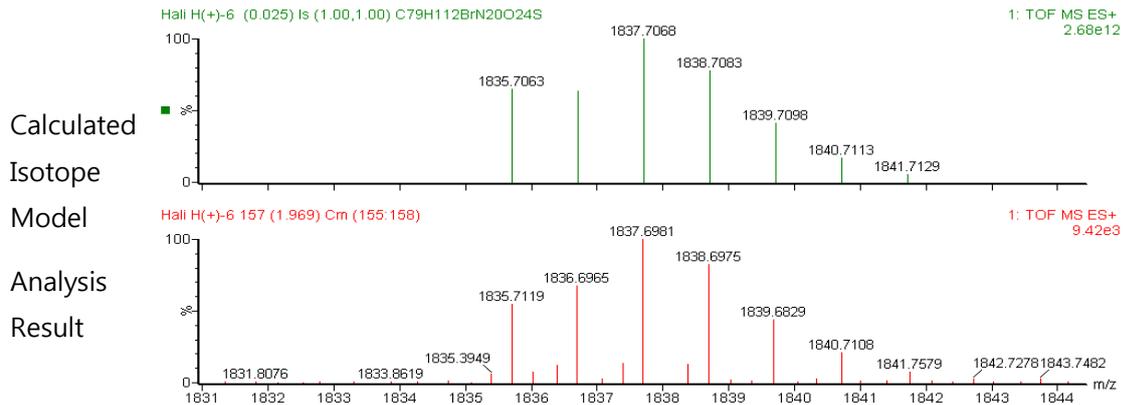
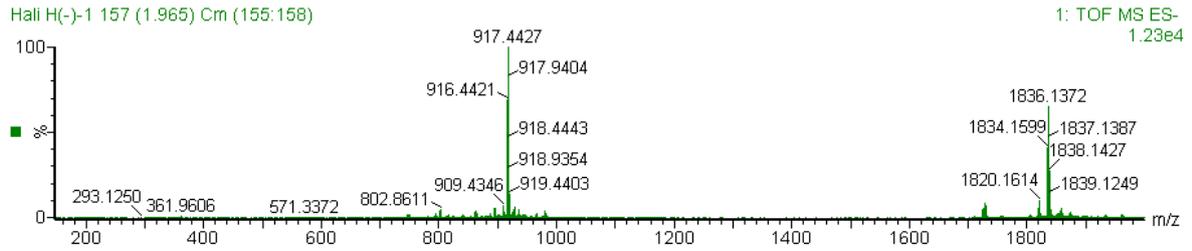
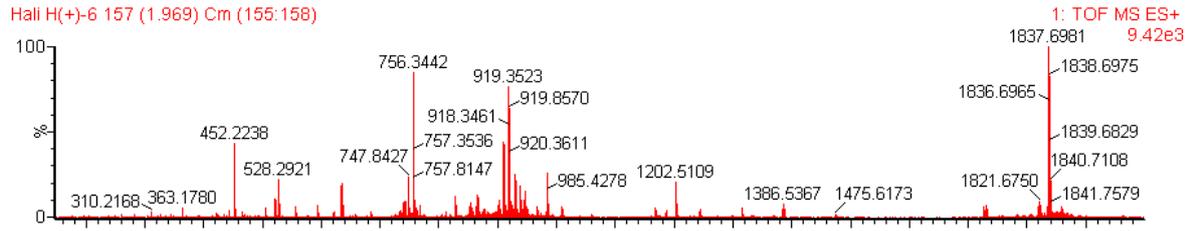
F1 - Acquisition parameters
NUC        2
TD         256
SFO1       150.9194 MHz
FIDRES     141.530792 Hz
SW         240.074 ppm
FHM000     GF

F2 - Processing parameters
SI         1024
SF         600.1300074 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
NUC2       GF
SF         150.9028739 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      4.493 ppm
F2LO       2696.15 Hz
F2PHI      2.276 ppm
F2HI       1365.65 Hz
F1PLO      57.282 ppm
F1LO       8644.05 Hz
F1PHI      24.690 ppm
F1HI       3725.95 Hz
F1PPMCM    0.14780 ppm/cm
F2HZCM     88.70012 Hz/cm
F1PPMCM    2.17279 ppm/cm
F2HZCM     327.87970 Hz/cm
    
```

## S52. HR-ESI-ToF-MS spectrum of halicyclindramide H (3)



**Single Mass Analysis**  
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0  
Selected filters: None  
Monoisotopic Mass, Even Electron Ions  
19 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

Mass	Calc. Mass	ΔDa	PPM	DBE	Formula	LEW	C	H	N	O	S	Br
1835.7119	1835.7062	5.7	-3.1	33.5	C79 H112 N22 O24 S Br	5549614.0	79	112	22	24	1	1
1835.7176	1835.7176	-5.6	-3.1	33.5	C78 H112 N22 O23 S Br	5549614.0	78	112	22	23	1	1

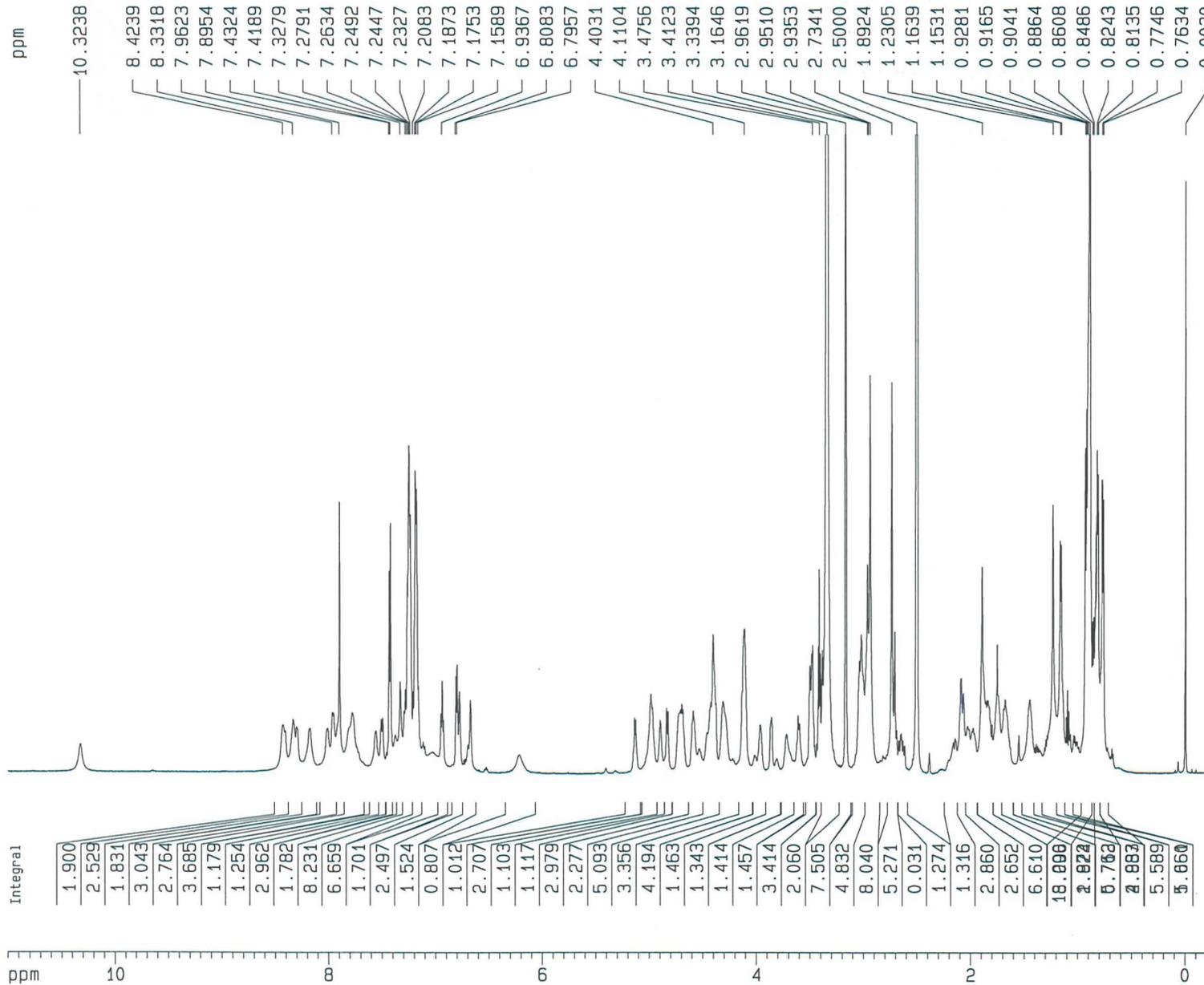
MCP 2300, CP 2200  
Hali H(+)-6 157 (1.969) Cm (155:158)

1: TOF MS ES+ 5.18e3

For Help, press F1

# S53. 1H NMR (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6

5-1/1H



## Current Data Parameters

NAME jul04-snu-chj  
 EXPNO 11  
 PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20050704  
 Time 16.09  
 INSTRUM spect  
 PROBHD 5 mm CPTXI 1H/  
 PULPROG zg30  
 TD 65536  
 SOLVENT DMSO  
 NS 8  
 DS 0  
 SWH 8741.259 Hz  
 FIDRES 0.133381 Hz  
 AQ 3.7487664 sec  
 RG 28.5  
 DW 57.200 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 1.00000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

## ==== CHANNEL f1 =====

NUC1 1H  
 P1 10.30 usec  
 PL1 -5.00 dB  
 SF01 600.1336696 MHz

## F2 - Processing parameters

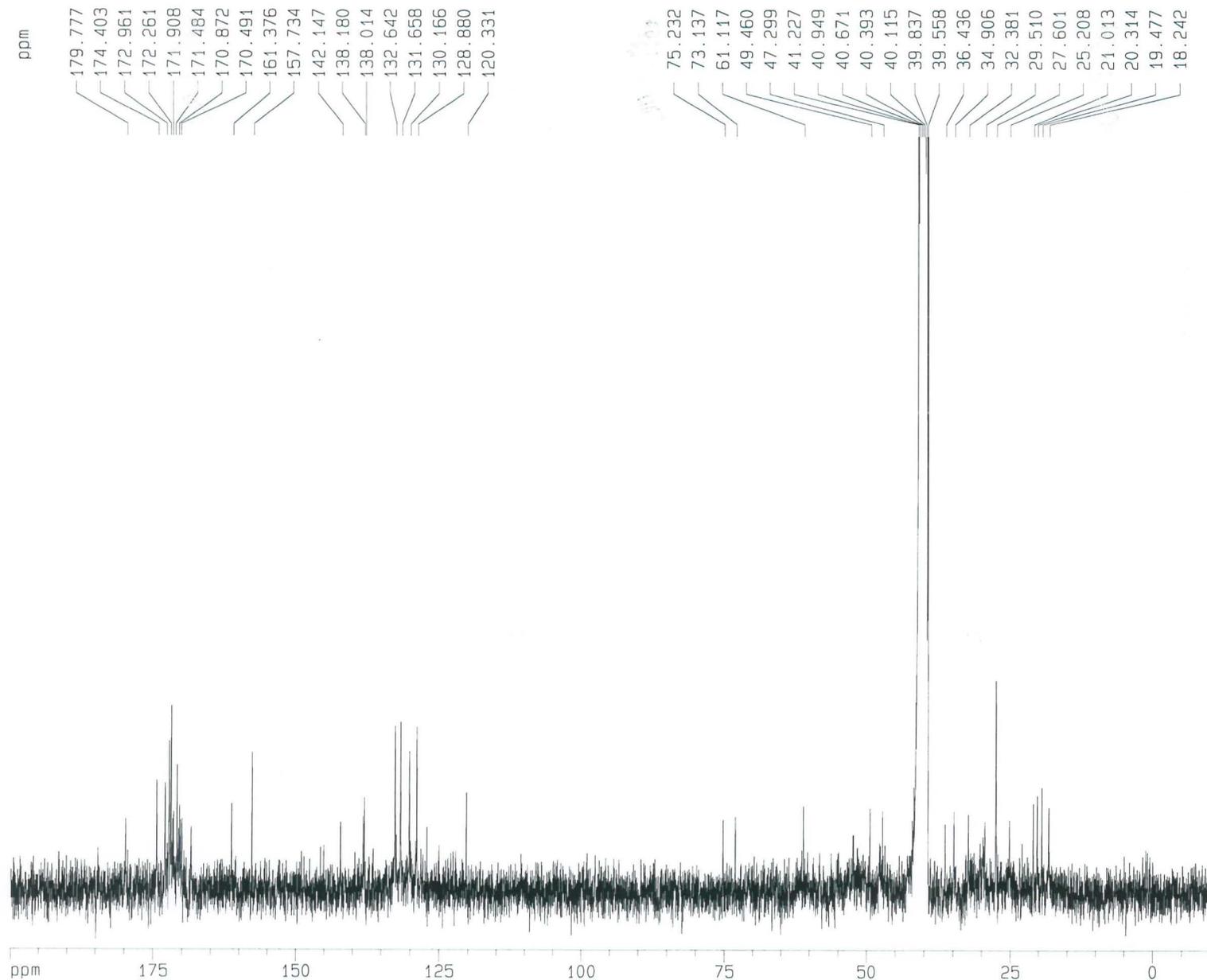
SI 32768  
 SF 600.1300071 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
 CY 200.00 cm  
 F1P 11.000 ppm  
 F1 6601.43 Hz  
 F2P -0.300 ppm  
 F2 -180.04 Hz  
 PPMCM 0.56500 ppm/cm  
 HZCM 339.07346 Hz/cm

# S54. <sup>13</sup>C NMR (150 MHz) spectrum of halicyclindramide H (3) in DMSO-d<sub>6</sub>

5-1/C13



## Current Data Parameters

NAME ju108-snu-chj  
 EXPNO 5  
 PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20050717  
 Time 11.27  
 INSTRUM spect  
 PROBHD 5 mm Dual 13  
 PULPROG zgdc  
 TD 65536  
 SOLVENT DMSO  
 NS 46080  
 DS 0  
 SWH 21097.047 Hz  
 FIDRES 0.321915 Hz  
 AQ 1.5532532 sec  
 RG 8192  
 DW 23.700 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
 P1 10.60 usec  
 PL1 0.00 dB  
 SF01 75.4760204 MHz

## ===== CHANNEL f2 =====

CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 100.00 usec  
 PL2 -5.00 dB  
 PL12 15.00 dB  
 SF02 300.1312005 MHz

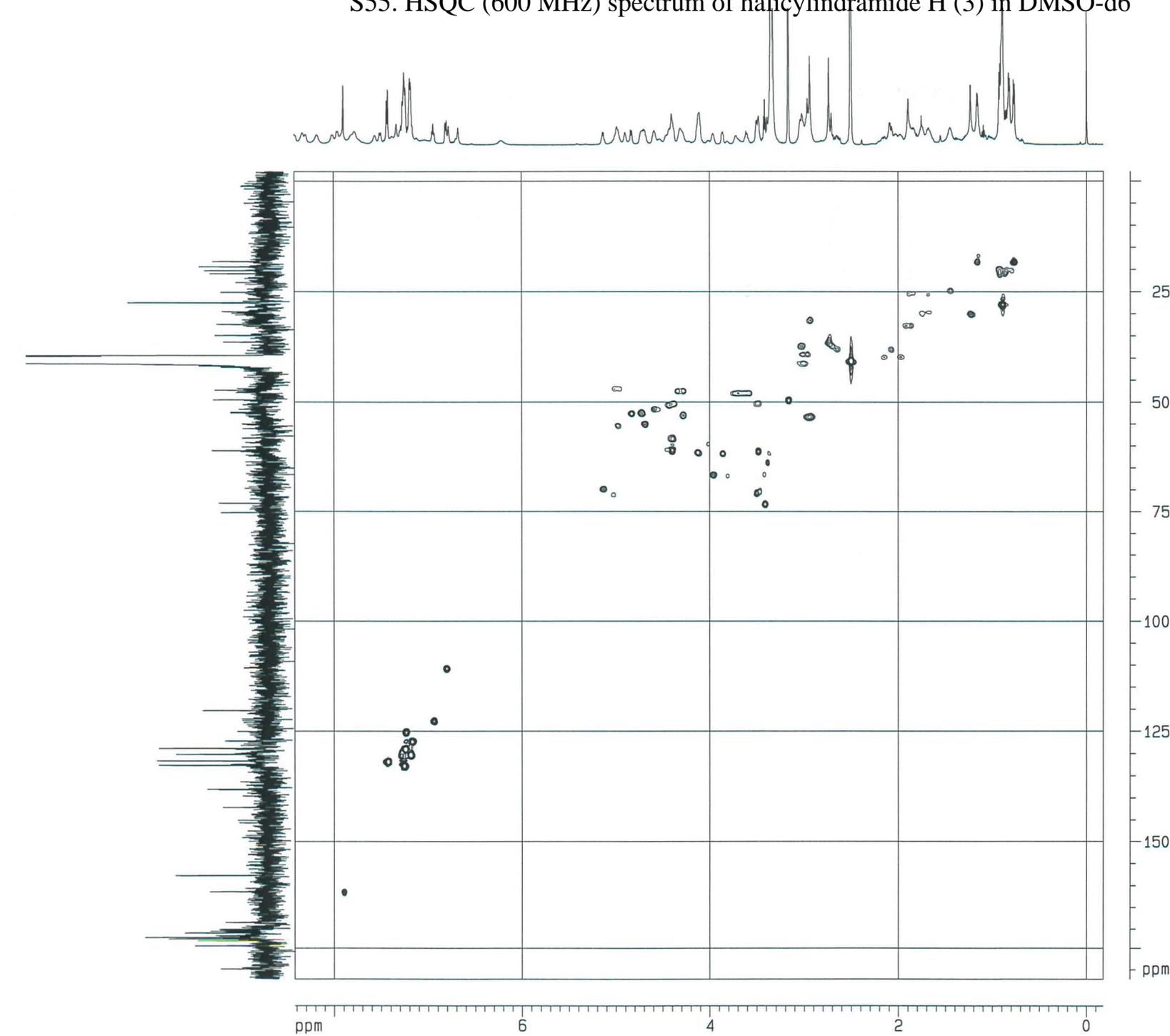
## F2 - Processing parameters

SI 32768  
 SF 75.4677190 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
 F1P 200.000 ppm  
 F1 15093.54 Hz  
 F2P -10.000 ppm  
 F2 -754.68 Hz  
 PPMCM 10.50000 ppm/cm  
 HZCM 792.41107 Hz/cm

S55. HSQC (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6



5-1/hsqcetgp

```

Current Data Parameters
NAME      jul04-snu-chn
EXPNO    17
PROCNO   1

F2 - Acquisition Parameters
Date_    20050708
Time     0.14
INSTRUM  spect
PROBHD   5 mm CPTXI 1H/
PULPROG  hsqcetgp
TD       1024
SOLVENT  DMSO
NS       16
DS       32
SWH      6513.737 Hz
FIDRES   6.458747 Hz
AQ       0.0775400 sec
RG       20642.5
DE       75.600 usec
TE       298.0 K
CNS12    145.000000
GB       0.0000300 sec
D1       1.50000000 sec
d4       0.00172414 sec
d11      0.03000000 sec
g13      0.00000400 sec
D16      0.00020000 sec
DELTA    0.00122700 sec
DELTA1   0.00071614 sec
IN0      0.00001360 sec
MCRETST  0.00000000 sec
MCRWK    0.30000001 sec
STICNT   128

----- CHANNEL f1 -----
NUC1     1H
P1       10.50 usec
p2       21.00 usec
P2B      0.10 usec
PL1      -5.00 dB
SFO1     600.1330006 MHz

----- CHANNEL f2 -----
CPDPRG2  gbrs
NUC2     13C
P3       15.00 usec
p4       30.00 usec
PCPD2    70.00 usec
PL2      -5.00 dB
PL12     7.40 dB
SFO2     150.9194083 MHz

----- GRADIENT CHANNEL -----
GPMAM1   SINE.100
GPMAM2   SINE.100
GPX1     0.00 %
GPX2     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPZ1     80.00 %
GPZ2     20.10 %
P16      1000.00 usec

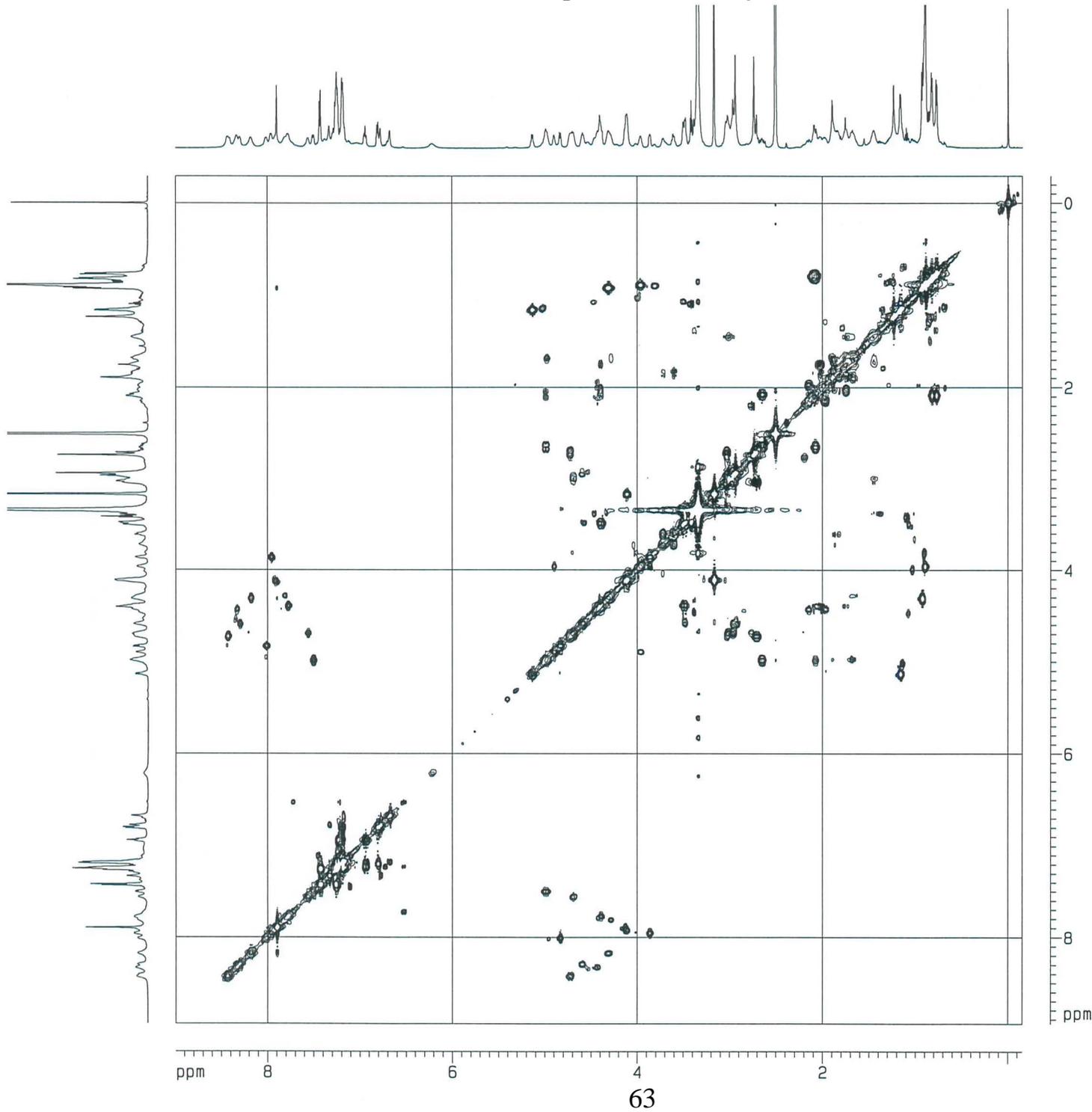
F1 - Acquisition parameters
ND0      2
TD       256
SFO1     150.9194 MHz
FIDRES   141.530792 Hz
SW       240.074 ppm
FMODE    Echo-Antiecho

F2 - Processing parameters
SI       1024
SF       600.1300071 MHz
WDW      GSINE
SSB      2
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
MC2      echo-antiecho
SF       150.9027205 MHz
WDW      GSINE
SSB      2
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      16.00 cm
F2PLD0   8.421 ppm
F2L0     5053.66 Hz
F2PHI    -0.178 ppm
F2HI     -106.68 Hz
F2PLD    182.101 ppm
F2L0     27479.50 Hz
F2PHI    -2.195 ppm
F2HI     -331.28 Hz
F2PPMCM  0.57327 ppm/cm
F2HZCM   344.02689 Hz/cm
F1PPMCM  12.28641 ppm/cm
F1HZCM   1854.05237 Hz/cm
    
```

S56. COSY (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6



5-1/COSY

```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO     15
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      20.27
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   cosyppaf
TD         2048
SOLVENT   DMSO
NS         15
DS         8
SWH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         57
DW         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.0000300 sec
d1         1.48689198 sec
d13        0.0000400 sec
d16        0.00020000 sec
INO        0.00015149 sec
MCREST    0.0000000 sec
MCKWK     1.48689198 sec

***** CHANNEL f1 *****
NUC1      1H
P0        10.50 usec
P1        10.50 usec
PL1       -5.00 dB
SFO1      600.133006 MHz

***** GRADIENT CHANNEL *****
GPNAM1    SINE.100
GPNAM2    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPZ1      10.00 %
GPZ2      10.00 %
P15       1000.00 usec

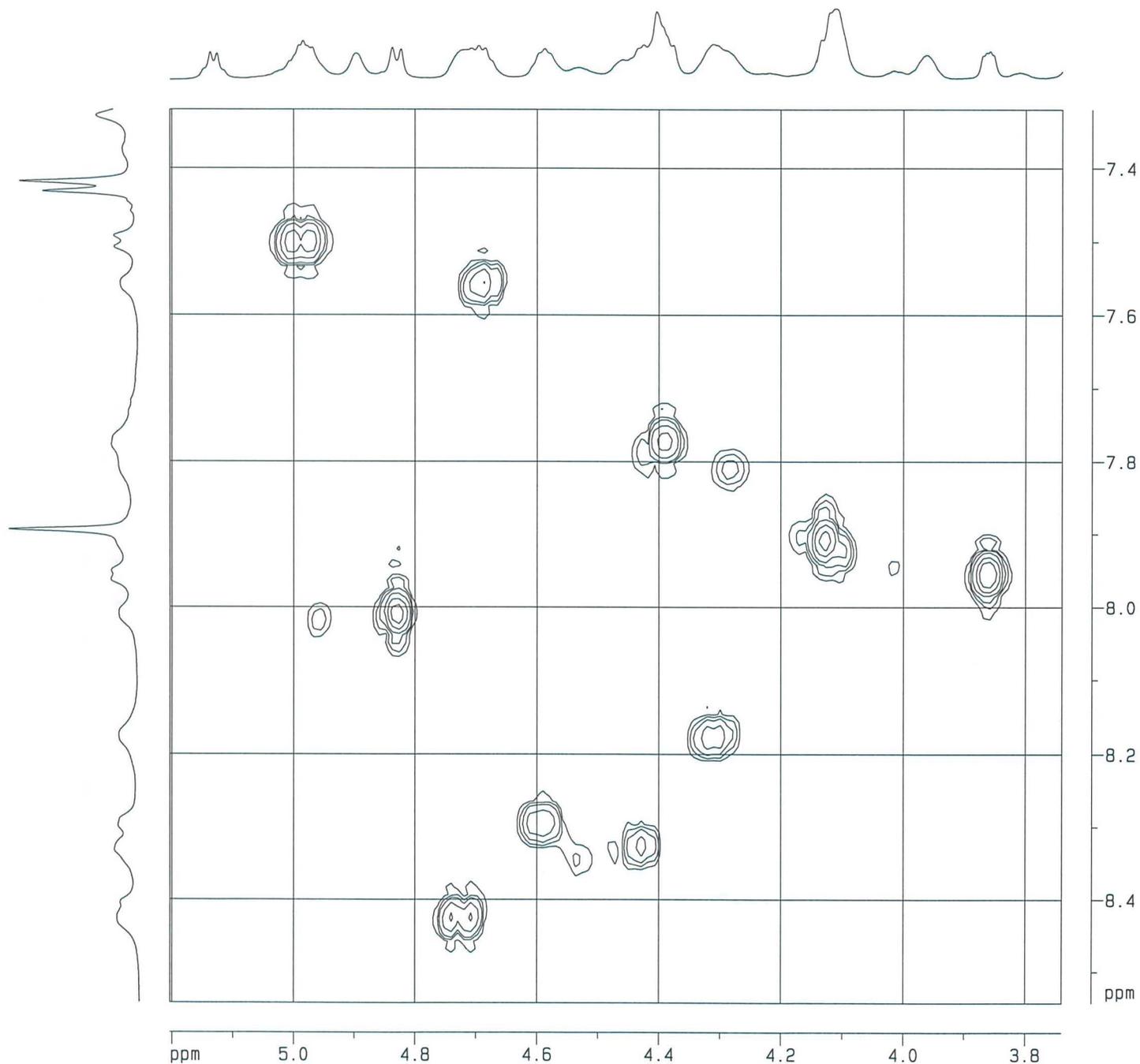
F1 - Acquisition parameters
NO0       1
TD         256
SFO1      600.133 MHz
FIDRES     25.785955 Hz
SW         11.000 ppm
FnMODE    QF

F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        QF
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      8.992 ppm
F2LO       5396.17 Hz
F2PHI      -0.156 ppm
F2HI       -93.75 Hz
F1PLO      8.992 ppm
F1LO       5372.27 Hz
F1PHI      -0.297 ppm
F1HI       -178.15 Hz
F2PPMCM    0.60986 ppm/cm
F2HZCM     365.99567 Hz/cm
F1PPMCM    0.61658 ppm/cm
F1HZCM     370.02847 Hz/cm
    
```

S57. Expanded COSY (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6



5-1/COSY

```

Current Data Parameters
NAME      ju104-gnu-ch)
EXPNO     15
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      20.27
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   cosygpaf
TD         2048
SOLVENT   DMSO
NS         16
DS         8
SWH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         57
DW         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00000300 sec
D1         1.46689198 sec
d13        0.00000400 sec
d16        0.00020000 sec
IN0        0.00015149 sec
MCREST     0.00000000 sec
MCWRR      1.46689198 sec

***** CHANNEL f1 *****
NUC1       1H
P0         10.50 usec
P1         10.50 usec
PL1        -5.00 dB
SFO1       600.1330006 MHz

***** GRADIENT CHANNEL *****
GPNAM1     SINE:100
GPNAM2     SINE:100
GPX2       0.00 %
GPY2       0.00 %
GPZ2       0.00 %
GPR1       10.00 %
GPR2       10.00 %
GPR3       10.00 %
P16        1000.00 usec

F1 - Acquisition parameters
ND0        1
TD         256
SFO1       600.133 MHz
FIDRES     25.785955 Hz
SW         11.000 ppm
FnMODE     GF

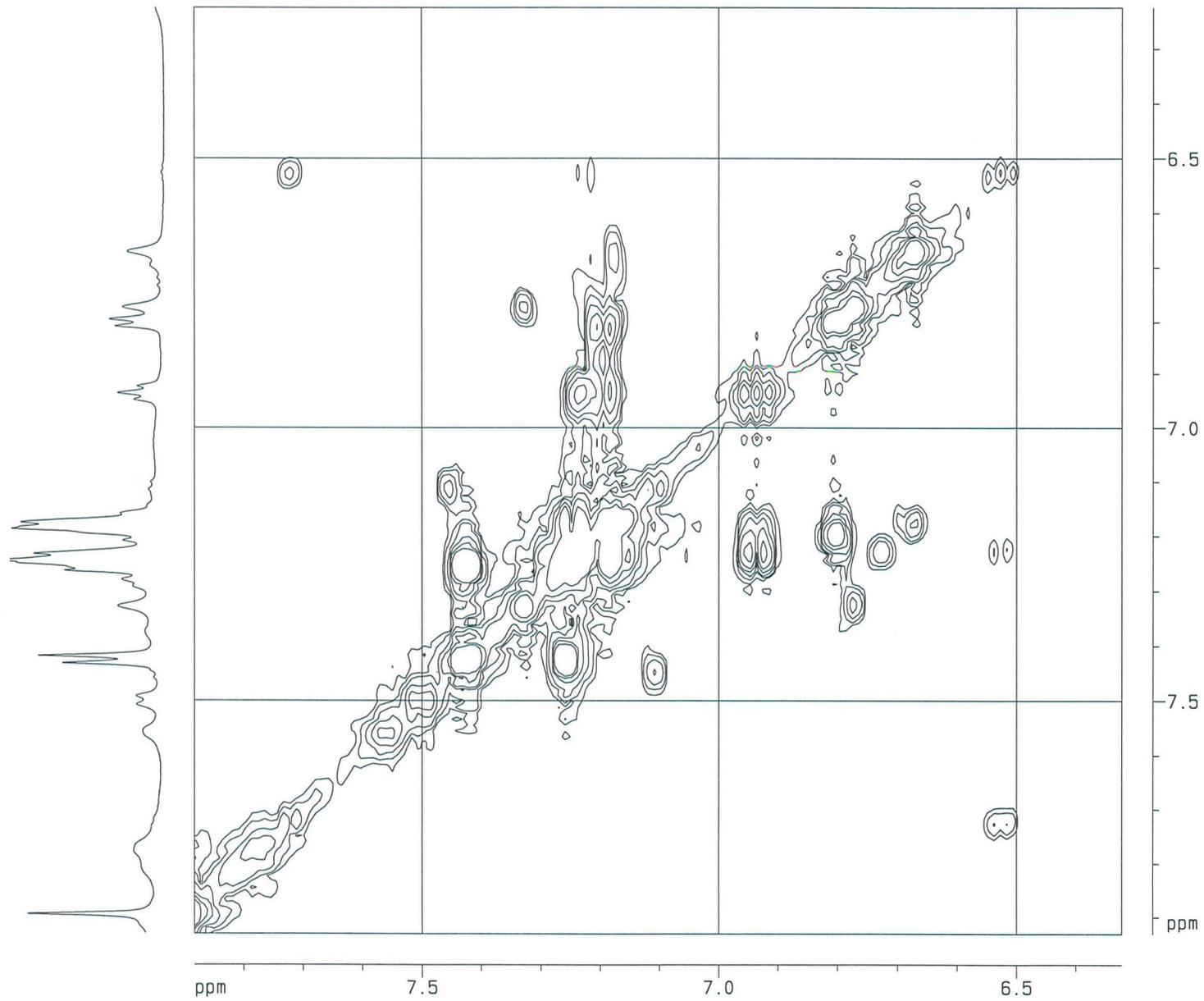
F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        GF
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      5.203 ppm
F2LO       3122.70 Hz
F2PHI      3.740 ppm
F2HI       2244.31 Hz
F1PLO      8.544 ppm
F1LO       5127.31 Hz
F1PHI      7.319 ppm
F1HI       4392.41 Hz
F2PPMCM    0.09758 ppm/cm
F2HZCM     58.55930 Hz/cm
F1PPMCM    0.08164 ppm/cm
F1HZCM     48.99331 Hz/cm
    
```

S58. Expanded COSY (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6

5-1/COSY



```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO     15
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      20.27
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   cosygpgf
TD         2048
SOLVENT   DMSO
NS         16
DS         8
SWH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         57
DM         75.500 usec
DE         5.00 usec
TE         298.0 K
d0         0.0000300 sec
d1         1.48689198 sec
d13        0.0000400 sec
d16        0.0002000 sec
INO        0.00015149 sec
MCREST     0.0000000 sec
MCWRRK     1.48689198 sec

===== CHANNEL f1 =====
NUC1       1H
P0         10.50 usec
P1         10.50 usec
PL1        -5.00 dB
SFO1       600.1330006 MHz

===== GRADIENT CHANNEL =====
GPNAM1     SINE.100
GPNAM2     SINE.100
GPX1       0.00 %
GPX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       10.00 %
GPZ2       10.00 %
P16        1000.00 usec

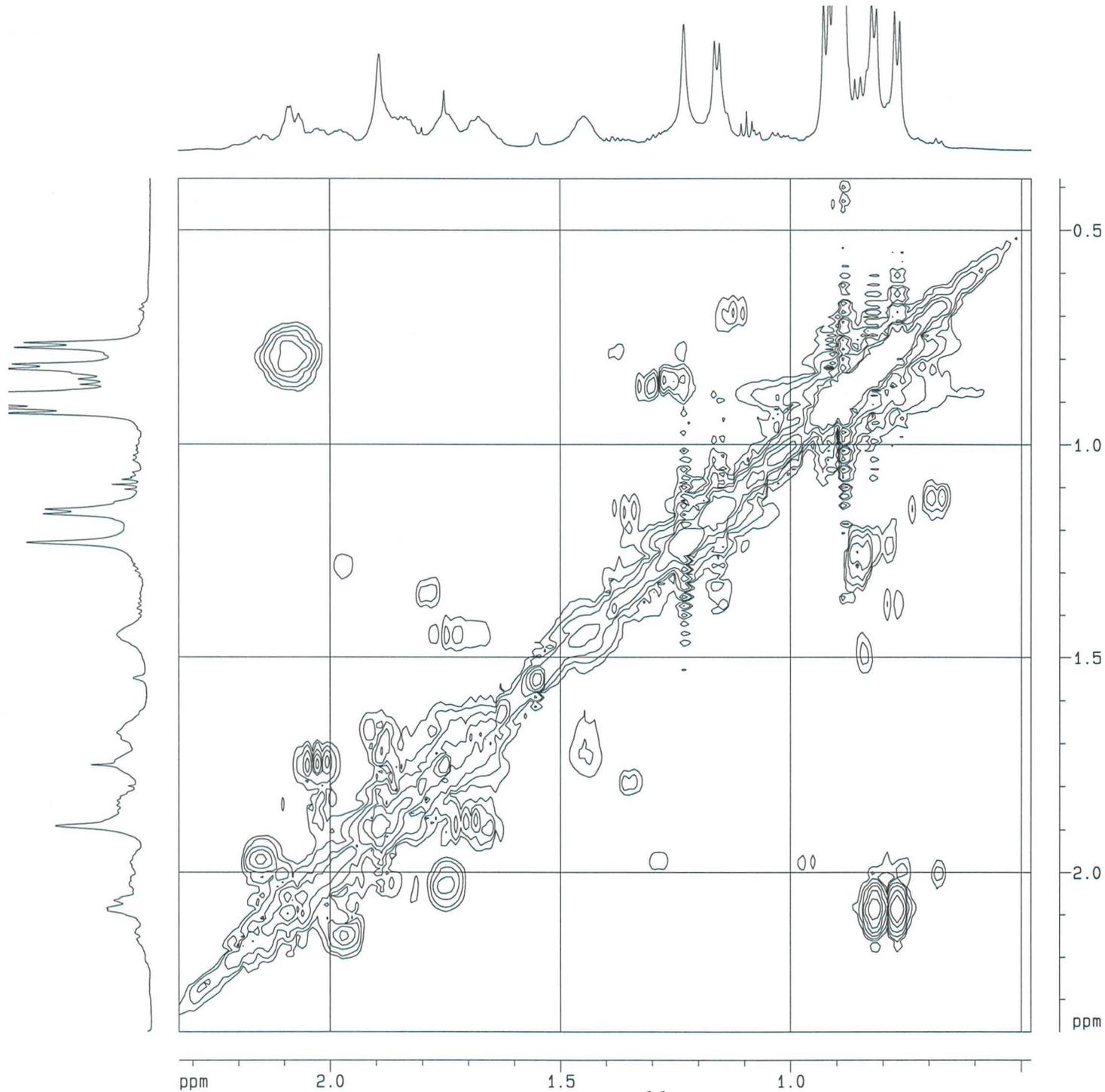
F1 - Acquisition parameters
ND0        1
TD         266
SFO1       600.133 MHz
FIDRES     25.785955 Hz
SW         11.000 ppm
FhMODE     GF

F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        GF
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      7.883 ppm
F2LO       4730.92 Hz
F2PHI      6.323 ppm
F2HI       3794.41 Hz
F1PLO      7.931 ppm
F1LO       4759.86 Hz
F1PHI      6.223 ppm
F1HI       3734.87 Hz
F2PPMCM    0.10404 ppm/cm
F2HZCM     62.43456 Hz/cm
F1PPMCM    0.11386 ppm/cm
F1HZCM     68.33279 Hz/cm
    
```

S59. Expanded COSY (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/COSY

```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO     15
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      20.27
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   cosygpgf
TD         2048
SOLVENT   DMSO
NS         16
DS         8
SWH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         67
DM         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00000300 sec
d1         1.48689198 sec
d13        0.00000400 sec
d16        0.0020000 sec
INO        0.00015149 sec
MCREST     0.00000000 sec
MCWPRK     1.48689198 sec

***** CHANNEL f1 *****
NUC1       1H
P0         10.50 usec
P1         10.50 usec
PL1        -5.00 dB
SF01       600.1330005 MHz

***** GRADIENT CHANNEL *****
GPNAM1     SINE.100
GPNAM2     SINE.100
GPX1       0.00 %
GPX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       10.00 %
GPZ2       10.00 %
P16        1000.00 usec

F1 - Acquisition parameters
ND0        1
TD         256
SF01       600.133 MHz
FIDRES     25.785955 Hz
SW         11.000 ppm
FhMODE     QF

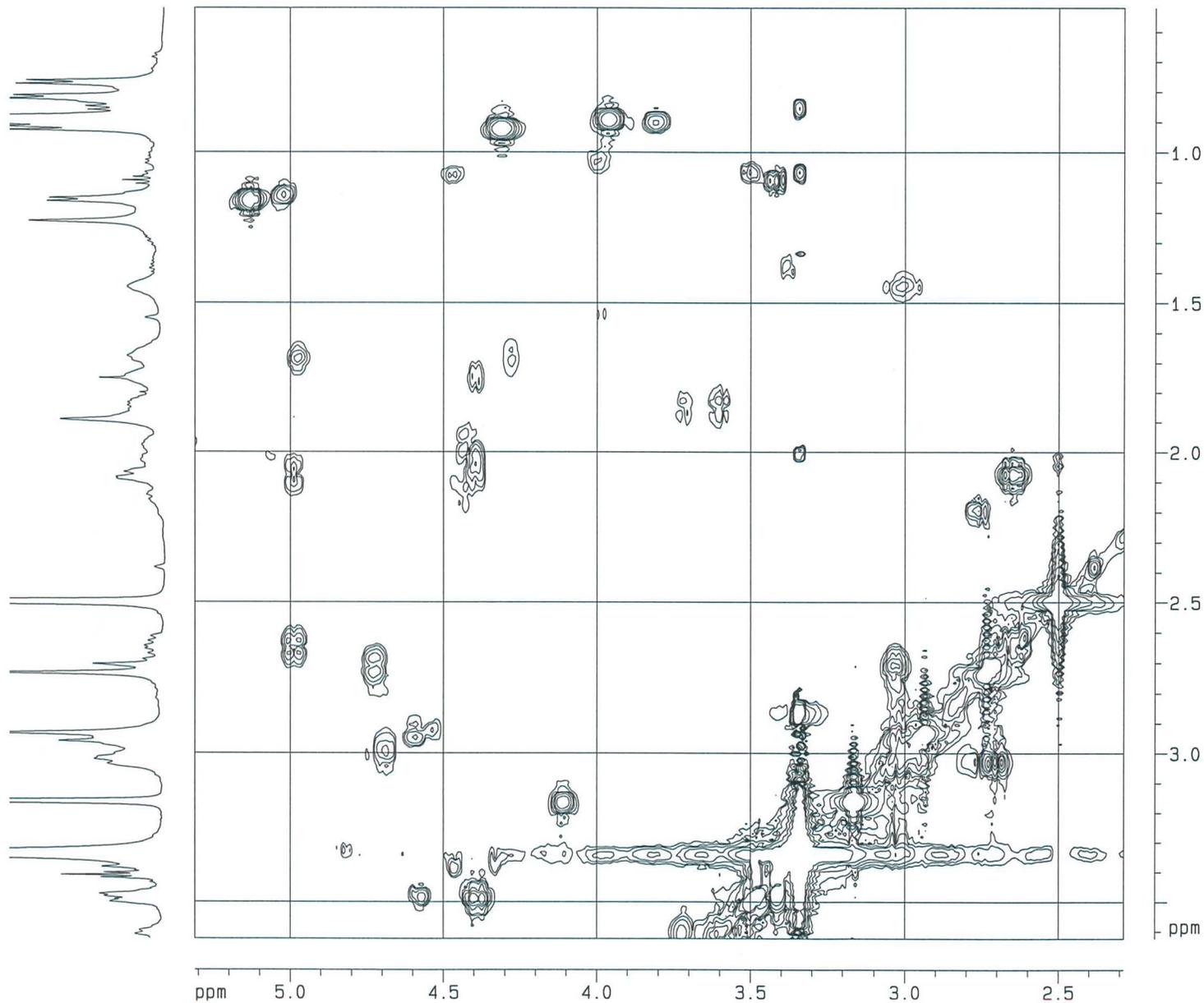
F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        QF
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1         15.00 cm
F2PL0      2.330 ppm
F2L0       1398.21 Hz
F2PHI      0.479 ppm
F2HI       287.31 Hz
F1PL0      2.378 ppm
F1L0       1427.02 Hz
F1PHI      0.380 ppm
F1HI       227.98 Hz
F2PPMCM    0.12341 ppm/cm
F2HZCM     74.06030 Hz/cm
F1PPMCM    0.13320 ppm/cm
F1HZCM     79.93646 Hz/cm
    
```

S60. Expanded COSY (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6

5-1/COSY



```

Current Data Parameters
NAME      ju104-snu-chj
EXPNO     15
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      20:27
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   cosygpqf
TD        2048
SOLVENT   DMSO
NS        16
DS        8
SWH       6613.757 Hz
FIDRES    3.229373 Hz
AQ        0.1549544 sec
RG        57
DW        75.600 usec
DE        6.00 usec
TE        298.0 K
d0        0.0000300 sec
D1        1.48689198 sec
d13       0.0000400 sec
D16       0.0002000 sec
IN0       0.00015149 sec
MCREST    0.0000000 sec
MCWRK     1.48689198 sec

***** CHANNEL f1 *****
NUC1      1H
P0        10.50 usec
P1        10.50 usec
PL1       -5.00 dB
SF01      600.1330005 MHz

***** GRADIENT CHANNEL *****
GPNAM1    SINE.100
GPNAM2    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPZ1      10.00 %
GPZ2      10.00 %
P16       1000.00 usec

F1 - Acquisition parameters
NDO       1
TD        256
SF01      600.133 MHz
FIDRES    25.785955 Hz
SW        11.000 ppm
FRMODE    GF

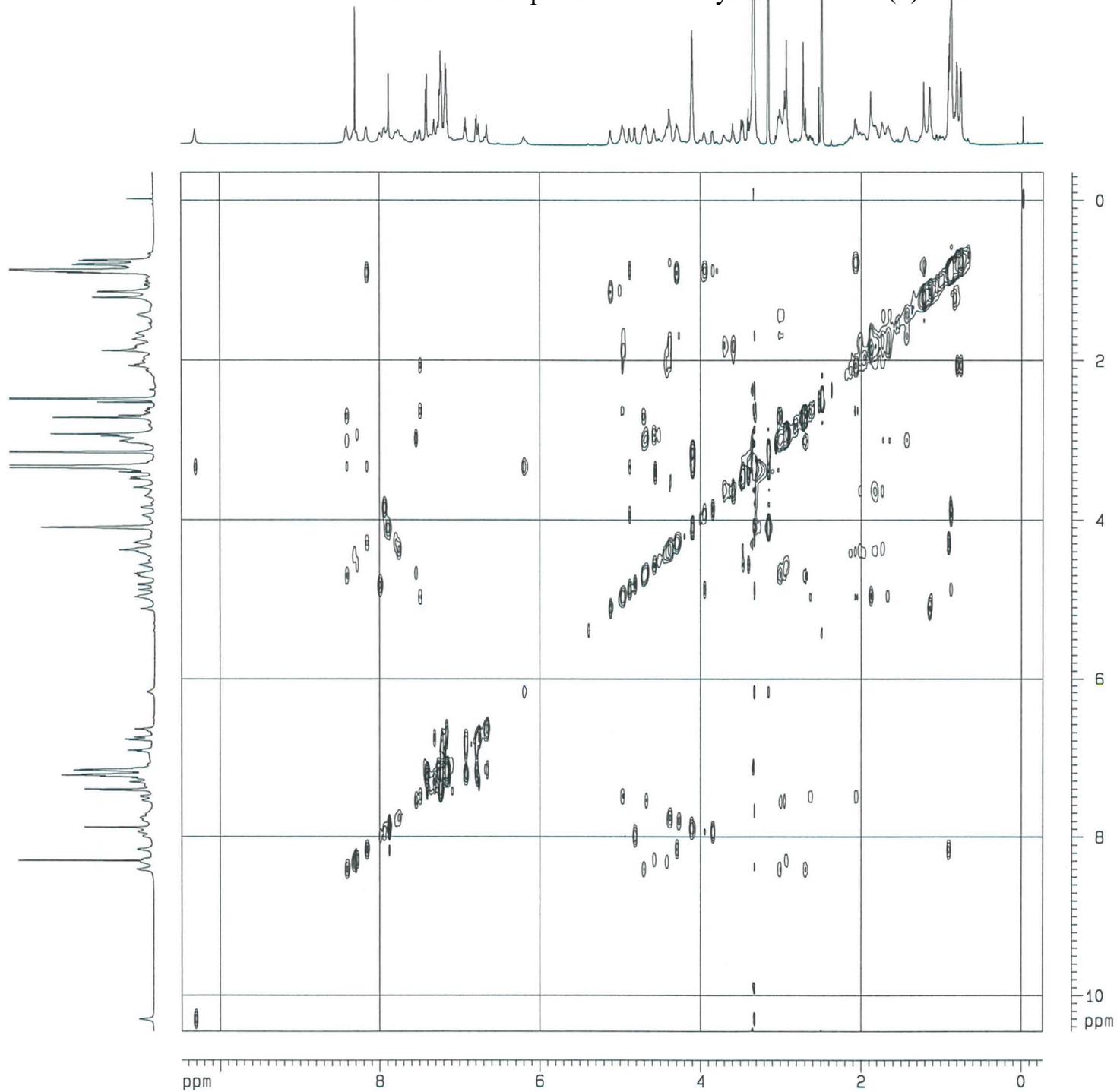
F2 - Processing parameters
SI        1024
SF        600.1300071 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.00

F1 - Processing parameters
SI        1024
MC2       GF
SF        600.1300071 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     5.311 ppm
F2LO      3167.28 Hz
F2PHI     2.287 ppm
F2HI      1372.38 Hz
F1PLO     3.624 ppm
F1LO      2174.82 Hz
F1PHI     0.520 ppm
F1HI      311.78 Hz
F2PPMCM   0.20161 ppm/cm
F2HZCM    120.99286 Hz/cm
F1PPMCM   0.30086 ppm/cm
F1HZCM    124.20236 Hz/cm
    
```

# S61. TOCSY spectrum of halicylindramide H (3) in DMSO-d6

5-1/TOCSY



Current Data Parameters  
 NAME oct25-snu-chj  
 EXPNO 13  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20051028  
 Time 11.51  
 INSTRUM spect  
 PROBHD 5 mm CPTX1 1H/  
 PULPROG m1evprb  
 TD 2048  
 SOLVENT DMSO  
 NS 16  
 DS 16  
 SWH 6613.757 Hz  
 FIDRES 3.229373 Hz  
 AQ 0.1549544 sec  
 RG 80.6  
 DW 75.600 usec  
 DE 6.00 usec  
 TE 298.0 K  
 d0 0.00006506 sec  
 D1 2.00000000 sec  
 D9 0.06000000 sec  
 d12 0.00002000 sec  
 FACTOR1 6  
 INO 0.00015149 sec  
 J1 36  
 MCREST 0.00000000 sec  
 MCWPK 1.00000000 sec  
 SCALEF 6  
 ST1CNT 64

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 1H  
 P1 10.50 usec  
 p5 16.67 usec  
 P6 25.00 usec  
 p7 50.00 usec  
 P17 1500.00 usec  
 PL1 -5.00 dB  
 PL10 2.54 dB  
 SFO1 600.1330006 MHz

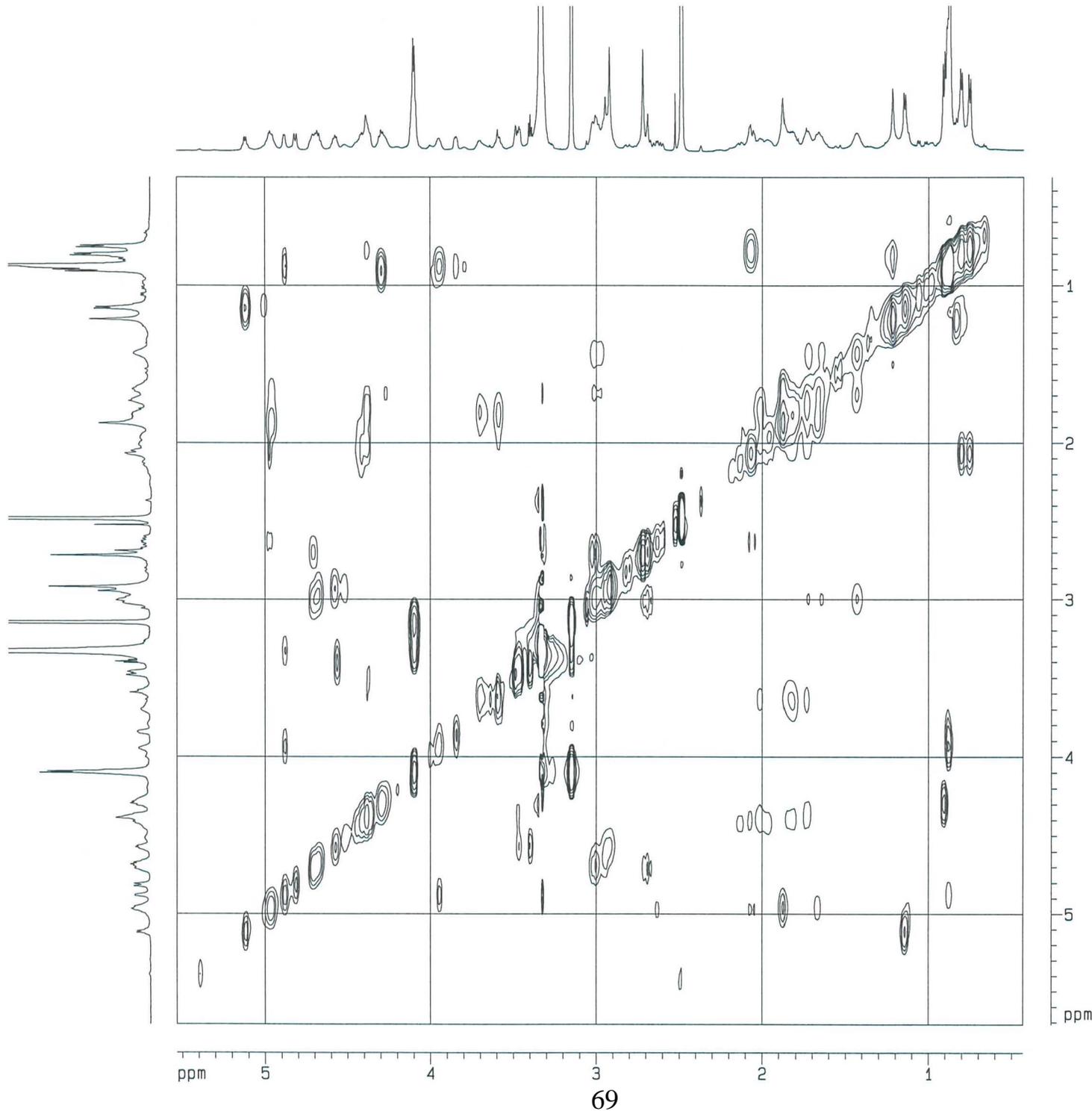
F1 - Acquisition parameters  
 MD 1  
 TD 128  
 SFO1 600.133 MHz  
 FIDRES 31.571911 Hz  
 SW 11.000 ppm  
 FhMODE States-TPPI

F2 - Processing parameters  
 SI 1024  
 SF 600.1300162 MHz  
 MDW OSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 MC2 States-TPPI  
 SF 600.1300162 MHz  
 MDW OSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 10.483 ppm  
 F2LO 6291.33 Hz  
 F2PHI -0.268 ppm  
 F2HI -160.96 Hz  
 F1PLO 10.462 ppm  
 F1LO 6278.61 Hz  
 F1PHI -0.355 ppm  
 F1HI -213.01 Hz  
 F2PPMCM 0.71677 ppm/cm  
 F2HZCM 430.15253 Hz/cm  
 F1PPMCM 0.72113 ppm/cm  
 F1HZCM 432.77426 Hz/cm

S62. Expanded TOCSY (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/TOCSY

Current Data Parameters  
 NAME oct25-snu-chj  
 EXPNO 13  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20051028  
 Time 11.51  
 INSTRUM spect  
 PROBHD 5 mm CPTXI 1H/  
 PULPROG mlevgh  
 TD 2048  
 SOLVENT DMSO  
 NS 16  
 DS 16  
 SWH 6613.757 Hz  
 FIDRES 3.229373 Hz  
 AQ 0.1549544 sec  
 RG 80.6  
 DW 75.600 usec  
 DE 6.00 usec  
 TE 298.0 K  
 d0 0.0006506 sec  
 D1 2.0000000 sec  
 D9 0.06000000 sec  
 d12 0.00002000 sec  
 FACTOR1 6  
 INO 0.00015148 sec  
 J1 35  
 MCREST 0.00000000 sec  
 MCNRK 1.00000000 sec  
 SCALEF 6  
 STICNT 64

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 1H  
 P1 10.50 usec  
 p5 16.67 usec  
 P6 25.00 usec  
 p7 50.00 usec  
 P17 1500.00 usec  
 PL1 -5.00 dB  
 PL10 2.54 dB  
 SF01 600.1330006 MHz

F1 - Acquisition parameters  
 NDO 1  
 TD 128  
 SF01 600.133 MHz  
 FIDRES 51.571911 Hz  
 SW 11.000 ppm  
 FnMODE States-TPPI

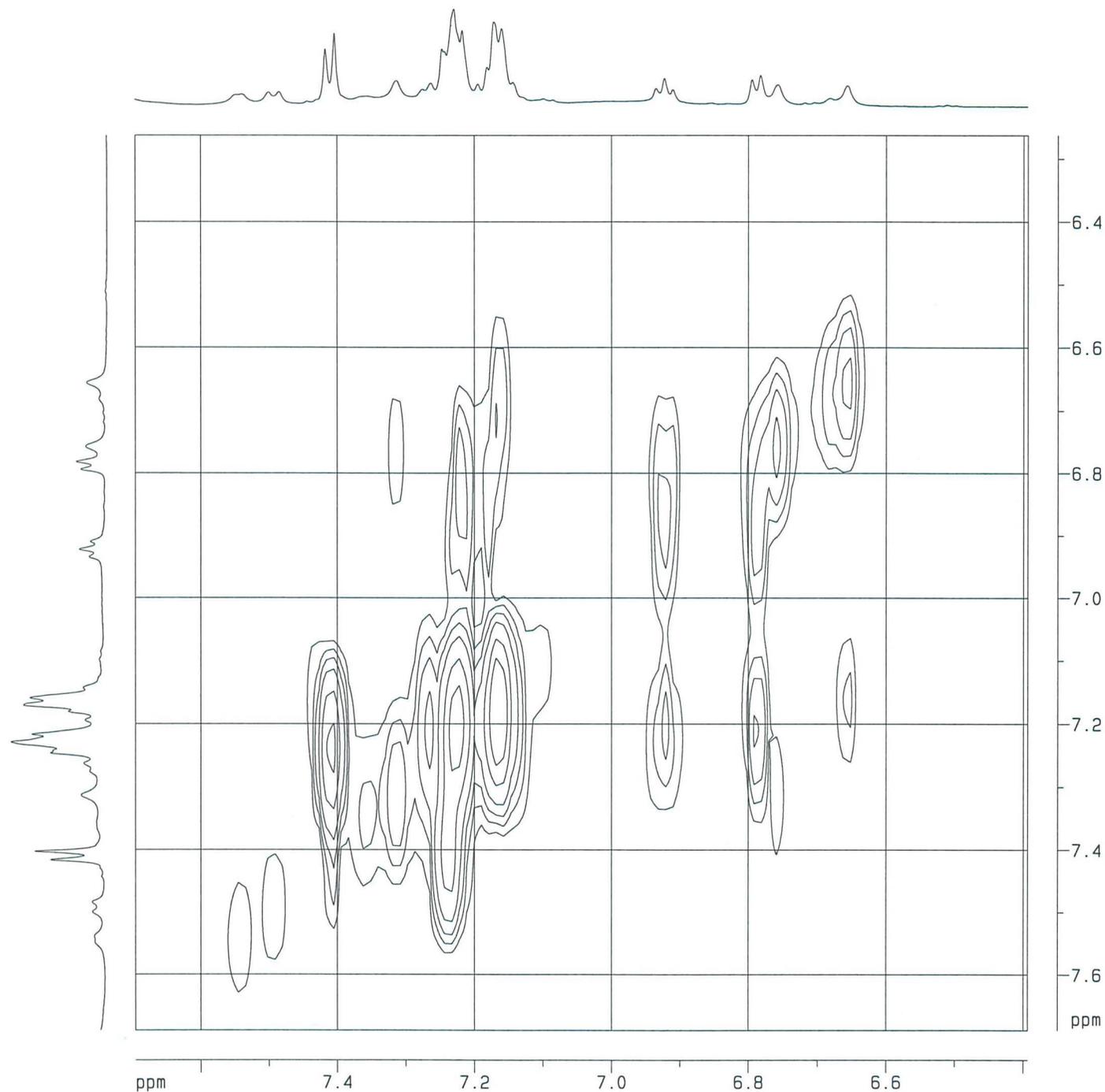
F2 - Processing parameters  
 SI 1024  
 SF 600.1300162 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 MC2 States-TPPI  
 SF 600.1300162 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 5.533 ppm  
 F2PHI 3320.31 Hz  
 F2PHI 0.431 ppm  
 F2HI 258.88 Hz  
 F2HI 5.714 ppm  
 F1PLO 3429.26 Hz  
 F1PHI 0.311 ppm  
 F1HI 186.68 Hz  
 F2PPMCM 0.34009 ppm/cm  
 F2HZCM 204.09639 Hz/cm  
 F1PPMCM 0.36021 ppm/cm  
 F1HZCM 216.17226 Hz/cm

S63. Expanded TOCSY (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6

5-1/TOCSY



```

Current Data Parameters
NAME      oct25-snu-chj
EXPNO     13
PROCNO    1

F2 - Acquisition Parameters
Date_     20051028
Time      11:51
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   mlevph
TD         2048
SOLVENT   DMSO
NS         16
DS         16
SWH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         80.6
DM         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00006506 sec
D1         2.00000000 sec
D9         0.06000000 sec
d12        0.00002000 sec
FACTOR1    6
INO        0.00015149 sec
I1         36
MCREST     0.00000000 sec
MCMARK     1.00000000 sec
SCALEF     6
ST1CNT     64

***** CHANNEL f1 *****
NUC1       1H
P1         10.50 usec
p5         16.67 usec
P6         25.00 usec
p7         50.00 usec
P17        1500.00 usec
PL1        -5.00 dB
PL10       2.54 dB
SFO1       600.1330006 MHz

F1 - Acquisition parameters
ND0        1
TD         128
SFO1       600.133 MHz
FIDRES     51.571911 Hz
SW         11.000 ppm
FnMODE     States-TPPI

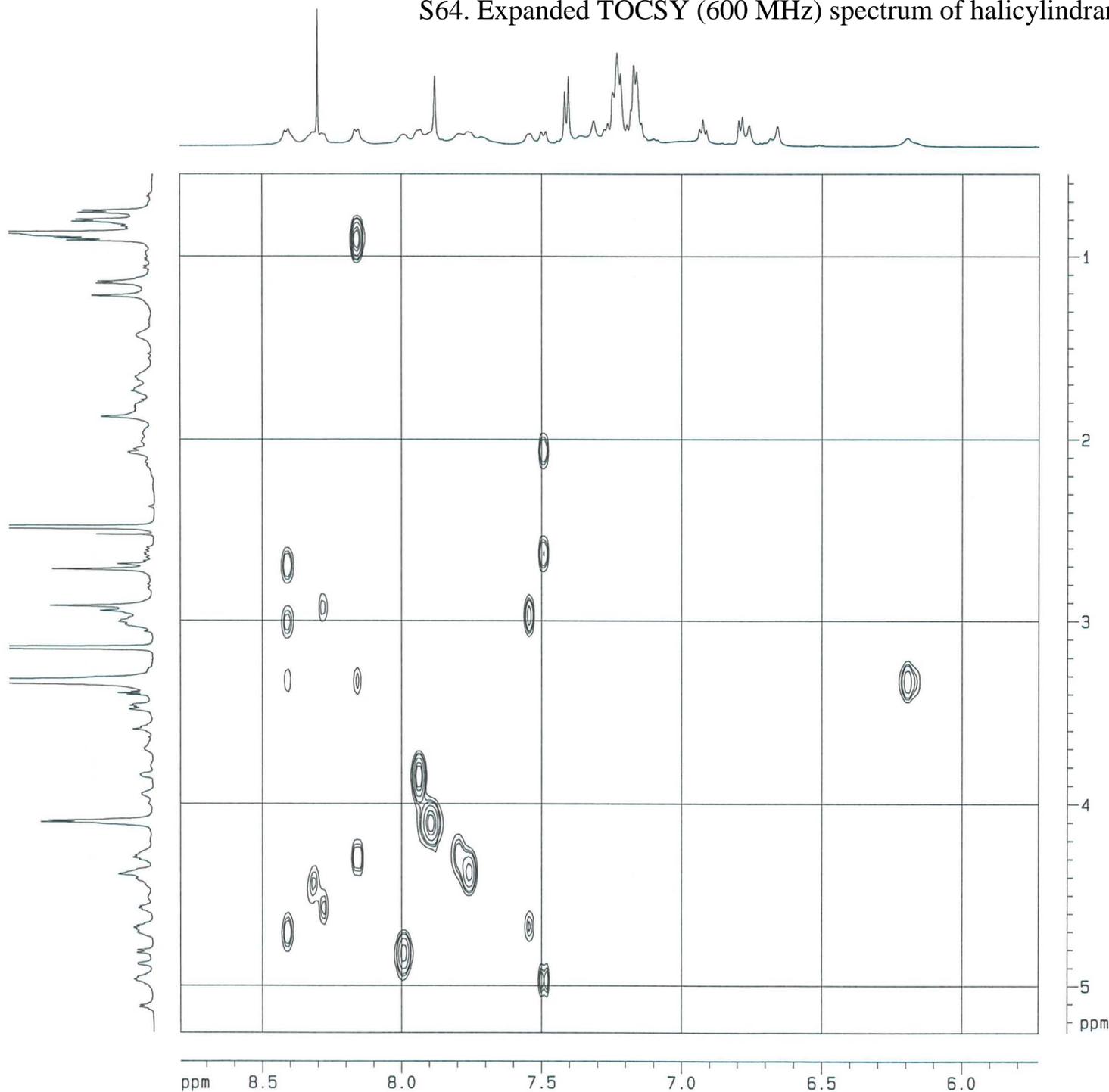
F2 - Processing parameters
SI         1024
SF         600.1300162 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300162 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      7.696 ppm
F2LO       4618.52 Hz
F2PHI      6.394 ppm
F2HI       3837.01 Hz
F1PLO      7.691 ppm
F1LO       4615.41 Hz
F1PHI      6.262 ppm
F1HI       3758.03 Hz
F2PPMCM    0.08682 ppm/cm
F2HZCM     52.10056 Hz/cm
F1PPMCM    0.09524 ppm/cm
F1HZCM     57.15887 Hz/cm
    
```

S64. Expanded TOCSY (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6

5-1/TOCSY



```

Current Data Parameters
NAME      oct25-snu-chj
EXPNO    13
PROCNO    1

F2 - Acquisition Parameters
Date_     20051028
Time      11.51
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   mlevgh
TD         2048
SOLVENT   DMSO
NS         16
DS         16
SWH        6613.757 Hz
FIDRES     3.229373 Hz
AQ         0.1549544 sec
RG         80.6
DW         75.600 usec
DE         6.00 usec
TE         298.0 K
d0         0.00006506 sec
D1         2.00000000 sec
D9         0.06000000 sec
d12        0.00002000 sec
FACTOR1    5
IND        0.00015148 sec
I1         35
MCREST     0.00000000 sec
MCNRK      1.00000000 sec
SCALEF     6
STICNT     64

***** CHANNEL f1 *****
NUC1       1H
P1         10.50 usec
p5         16.67 usec
P6         25.00 usec
p7         50.00 usec
P17        1500.00 usec
PL1        -5.00 dB
PL10       2.54 dB
SF01       600.1330006 MHz

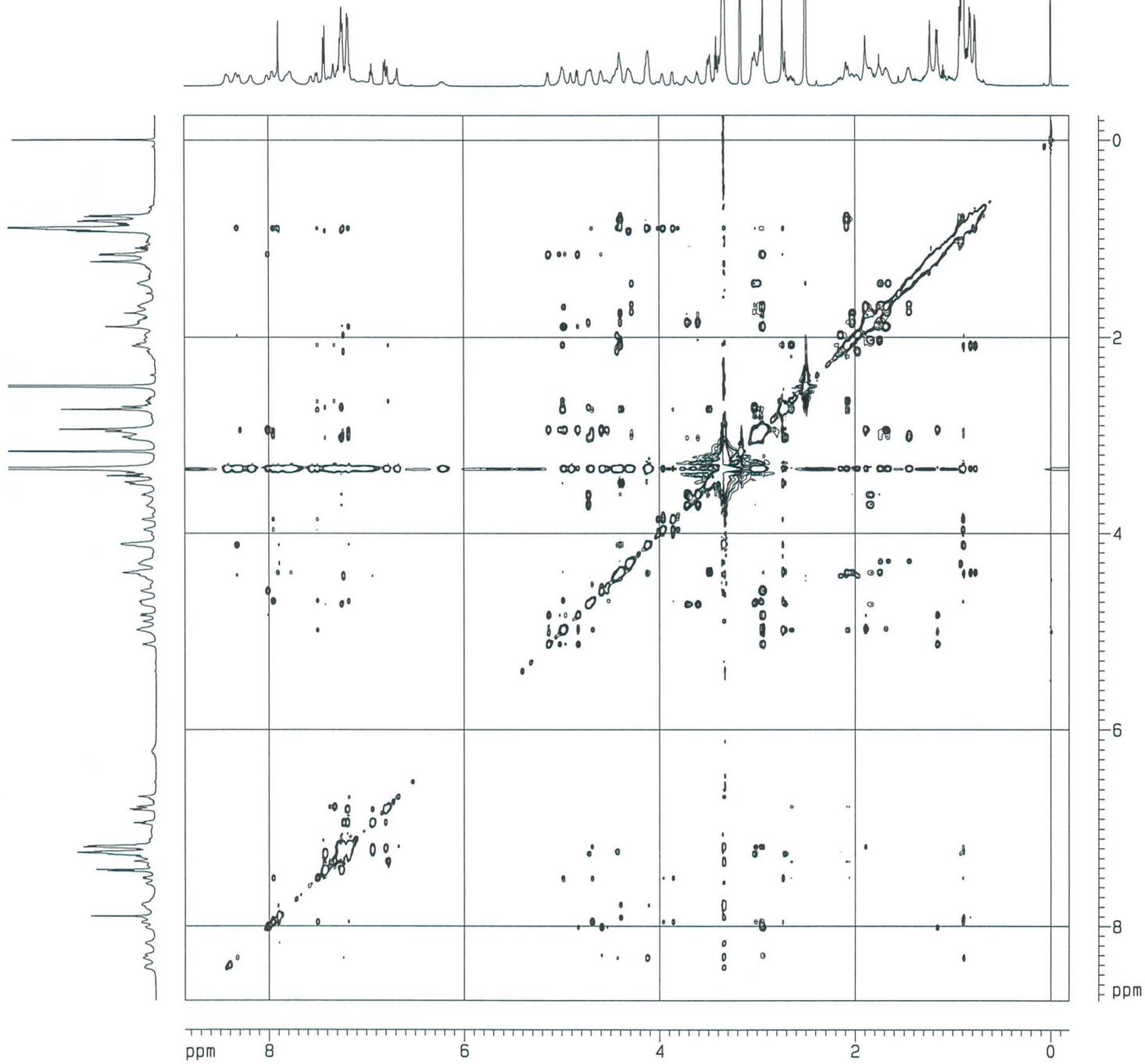
F1 - Acquisition parameters
ND0        1
TD         128
SF01       600.133 MHz
FIDRES     51.571911 Hz
SW         11.000 ppm
FnMODE     States-TPPI

F2 - Processing parameters
SI         1024
SF         600.1300162 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300162 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      8.794 ppm
F2L0       5277.31 Hz
F2PH1      5.726 ppm
F2H1       3436.57 Hz
F1PL0      5.263 ppm
F1L0       3158.57 Hz
F1PH1      0.547 ppm
F1H1       328.50 Hz
F2PPMCM    0.20448 ppm/cm
F2HZCM     122.71617 Hz/cm
F1PPMCM    0.31438 ppm/cm
F1HZCM     188.66724 Hz/cm
    
```

S65. NOESY (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/NOESY

Current Data Parameters  
 NAME ju104-snu-chj  
 EXPNO 14  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20050705  
 Time 17.55  
 INSTRUM spect  
 PROBHD 5 mm CPTXI 1H/  
 PULPROG noesyph  
 TD 4096  
 SOLVENT DMSO  
 NS 16  
 DS 8  
 SWH 6613.757 Hz  
 FIDRES 1.614687 Hz  
 AQ 0.3097832 sec  
 RG 28.5  
 DW 75.600 usec  
 DE 6.00 usec  
 TE 298.1 K  
 d0 0.0006237 sec  
 D1 1.0000000 sec  
 D8 0.89999998 sec  
 INO 0.00015149 sec  
 MCREST 0.0000000 sec  
 MCMK 0.5000000 sec  
 ST1CNT 128

----- CHANNEL f1 -----  
 NUC1 1H  
 P1 10.50 usec  
 PL1 -5.00 dB  
 SF01 600.1330006 MHz

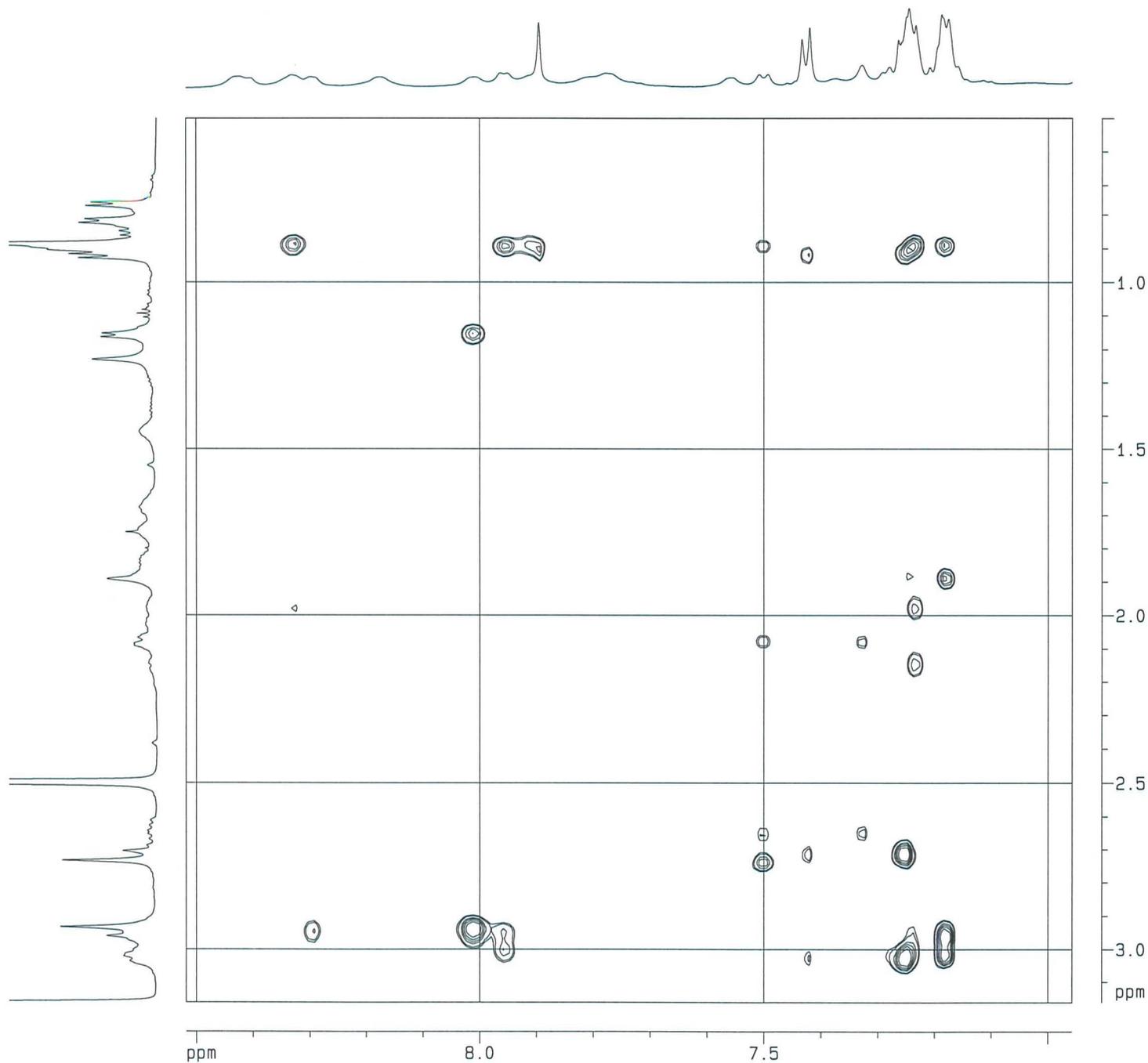
F1 - Acquisition parameters  
 ND0 1  
 TD 256  
 SF01 600.133 MHz  
 FIDRES 25.785955 Hz  
 SW 11.000 ppm  
 FnmODE States-TPPI

F2 - Processing parameters  
 SI 1024  
 SF 600.1300071 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 MC2 States-TPPI  
 SF 600.1300071 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 8.863 ppm  
 F2LO 5318.67 Hz  
 F2PHI -0.189 ppm  
 F2HI -113.14 Hz  
 F1PLO 8.769 ppm  
 F1LO 5262.68 Hz  
 F1PHI -0.254 ppm  
 F1HI -152.37 Hz  
 F2PPMCM 0.60340 ppm/cm  
 F2HZCM 362.12042 Hz/cm  
 F1PPMCM 0.60154 ppm/cm  
 F1HZCM 361.00339 Hz/cm

S66. Expanded NOESY (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6



5-1/NOESY

```

Current Data Parameters
NAME      ju104-snu-chj
EXPNO     14
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      17.55
INSTRUM   spect
PROBHD    5 mm CPX 1 4H/
PULPROG   noesyph
TD         4096
SOLVENT   DMSO
NS         16
DS         8
SWH        6613.757 Hz
FIDRES     1.614687 Hz
AQ         0.3097832 sec
RG         28.5
DW         75.600 usec
DE         6.00 usec
TE         298.1 K
d0         0.0006237 sec
D1         1.0000000 sec
DB         0.89999998 sec
INO        0.0015149 sec
MCREST     0.0000000 sec
MCWRK     0.5000000 sec
ST1CNT    128

===== CHANNEL f1 =====
NUC1      4H
P1        10.50 usec
PL1       -5.00 dB
SF01      600.1330006 MHz

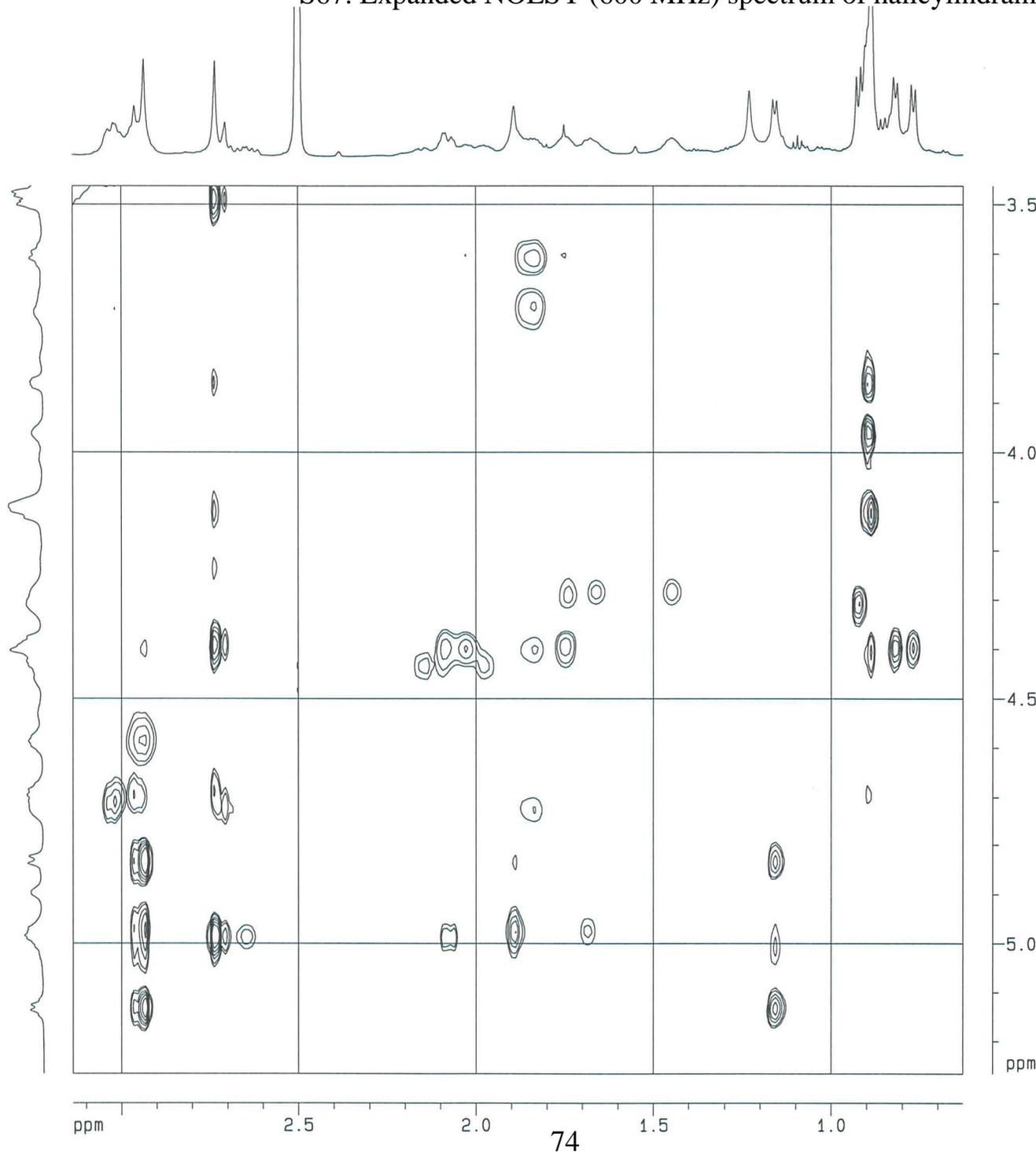
F1 - Acquisition parameters
ND0       1
TD        256
SF01      600.133 MHz
FIDRES    25.785955 Hz
SW        11.000 ppm
FnMODE    States-TPPI

F2 - Processing parameters
SI        1024
SF        600.1300071 MHz
WDW       QSINE
SSB       2
LB        0.00 Hz
GB        0
PC        1.00

F1 - Processing parameters
SI        1024
MC2       States-TPPI
SF        600.1300071 MHz
WDW       QSINE
SSB       2
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     8.518 ppm
F2LO      5111.99 Hz
F2PHI     6.958 ppm
F2HI      4175.47 Hz
F1PLO     3.162 ppm
F1LO      1897.62 Hz
F1PHI     0.498 ppm
F1HI      298.89 Hz
F2PPMCM   0.10404 ppm/cm
F2HZCM    62.43456 Hz/cm
F1PPMCM   0.17760 ppm/cm
F1HZCM    106.58195 Hz/cm
    
```

S67. Expanded NOESY (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6



5-1/NOESY

```

Current Data Parameters
NAME      jul04-snu-chj
EXPNO     14
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      17.55
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   noesyph
TD         4096
SOLVENT   DMSO
NS         16
DS         8
SWH        6613.757 Hz
FIDRES     1.614687 Hz
AQ         0.3097832 sec
RG         28.5
DW         75.600 usec
DE         6.00 usec
TE         298.1 K
d0         0.0006237 sec
D1         1.0000000 sec
DB         0.89999998 sec
INO        0.00015149 sec
MCREST     0.0000000 sec
MCWRK     0.5000000 sec
ST1CNT     128

===== CHANNEL f1 =====
NUC1      1H
P1         10.50 usec
PL1        -5.00 dB
SF01      600.1330005 MHz

F1 - Acquisition parameters
ND0        1
TD         256
SF01       600.133 MHz
FIDRES     25.785955 Hz
SW         11.000 ppm
FnMODE     States-TPPI

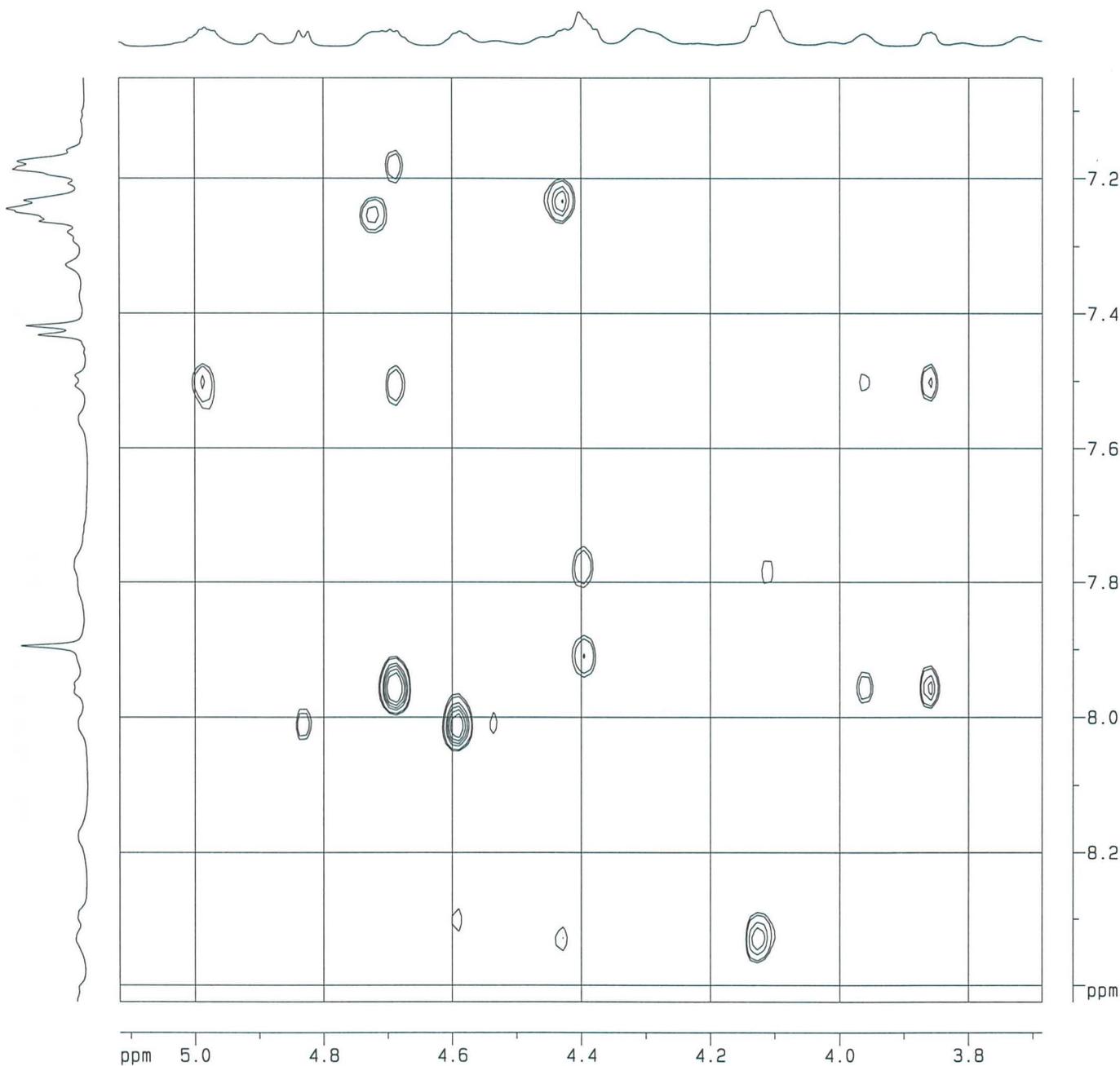
F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300071 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      3.137 ppm
F2LO       1882.62 Hz
F2PHI      0.629 ppm
F2HI       377.73 Hz
F1PLO      5.267 ppm
F1LO       3161.13 Hz
F1PHI      3.463 ppm
F1HI       2078.12 Hz
F2PPMCM    0.16717 ppm/cm
F2HZCM     100.32587 Hz/cm
F1PPMCM    0.12031 ppm/cm
F1HZCM     72.20068 Hz/cm
    
```

S68. Expanded NOESY (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6

5-1/NOESY



```

Current Data Parameters
NAME      ju104-snu-chj
EXPNO     14
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      17.55
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   noesyph
TD         4096
SOLVENT   DMSO
NS         16
DS         8
SWH        6613.757 Hz
FIDRES     1.614687 Hz
AQ         0.3097832 sec
RG         28.5
DW         75.600 usec
DE         6.00 usec
TE         298.1 K
d0         0.00006237 sec
D1         1.0000000 sec
DB         0.89999998 sec
INO        0.00015149 sec
MCREST     0.0000000 sec
MCWRK     0.5000000 sec
ST1CNT     128

===== CHANNEL f1 =====
NUC1       1H
P1         10.50 usec
PL1        -5.00 dB
SF01       600.1330006 MHz

F1 - Acquisition parameters
ND0        1
TD         256
SF01       600.133 MHz
FIDRES     25.785955 Hz
SW         11.000 ppm
FnMODE     States-TPPI

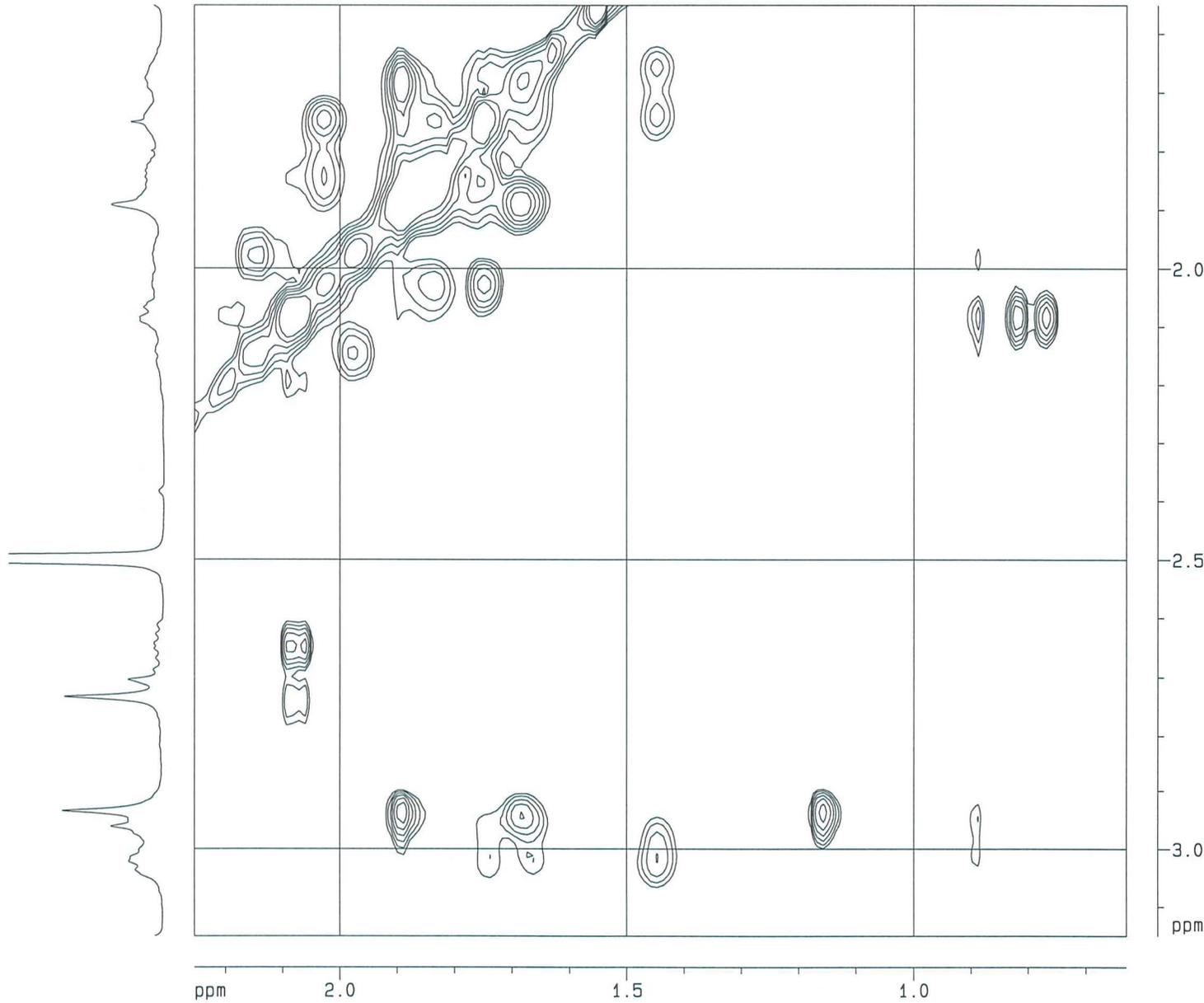
F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300071 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      5.117 ppm
F2LO       3071.03 Hz
F2PHI      3.686 ppm
F2HI       2212.01 Hz
F1PLO      8.426 ppm
F1LO       5056.40 Hz
F1PHI      7.051 ppm
F1HI       4231.25 Hz
F2PPMCM    0.09543 ppm/cm
F2HZCM     57.26755 Hz/cm
F1PPMCM    0.09166 ppm/cm
F1HZCM     55.01005 Hz/cm
    
```

S69. Expanded NOESY (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6

5-1/NOESY



```

Current Data Parameters
NAME      ju104-snu-chj
EXPNO    14
PROCNO   1

F2 - Acquisition Parameters
Date_    20050705
Time     17.55
INSTRUM  spect
PROBHD   5 mm CPTXI 1H/
PULPROG  noesyph
TD       4096
SOLVENT  DMSO
NS       16
DS       8
SWH      6613.757 Hz
FIDRES   1.614687 Hz
AQ       0.3097832 sec
RG       28.5
DW       75.600 usec
DE       6.00 usec
TE       298.1 K
d0       0.00005237 sec
D1       1.00000000 sec
D8       0.89999998 sec
INO      0.00015149 sec
MCREST   0.00000000 sec
MCWRK    0.50000000 sec
ST1CNT   128

===== CHANNEL f1 =====
NUC1     1H
P1       10.50 usec
PL1      -5.00 dB
SFO1     600.1330006 MHz

F1 - Acquisition parameters
ND0      1
TD       256
SFO1     600.133 MHz
FIDRES   25.785955 Hz
SW       11.000 ppm
FnMODE   States-TPPI

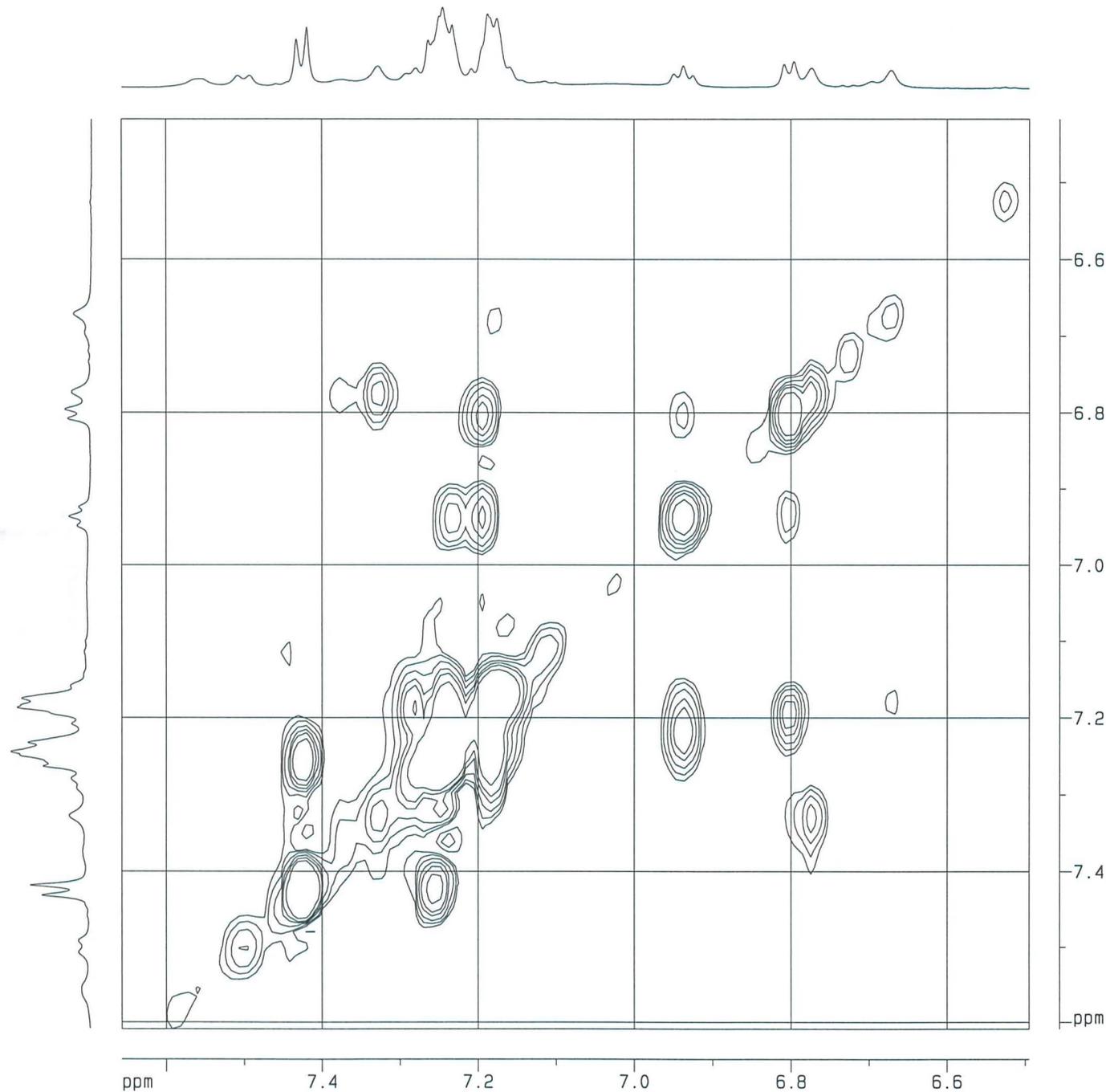
F2 - Processing parameters
SI       1024
SF       600.1300071 MHz
WDW      QSINE
SSB      2
LB       0.00 Hz
GB       0
PC       1.00

F1 - Processing parameters
SI       1024
MC2     States-TPPI
SF       600.1300071 MHz
WDW      QSINE
SSB      2
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2PLO    2.255 ppm
F2L0     1353.00 Hz
F2PHI    0.629 ppm
F2HI     377.73 Hz
F1PLO    3.151 ppm
F1L0     1891.17 Hz
F1PHI    1.551 ppm
F1HI     930.64 Hz
F2PPMCM  0.10834 ppm/cm
F2HZCM   65.01804 Hz/cm
F1PPMCM  0.10670 ppm/cm
F1HZCM   64.03512 Hz/cm
    
```

S70. Expanded NOESY (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6

5-1/NOESY



```

Current Data Parameters
NAME      ju104-snu-chj
EXPNO     14
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      17.55
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   noesyph
TD         4096
SOLVENT   DMSO
NS         16
DS         8
SWH        6613.757 Hz
FIDRES     1.614687 Hz
AQ         0.3097832 sec
RG         28.5
DW         75.600 usec
DE         6.00 usec
TE         298.1 K
d0         0.0006237 sec
D1         1.0000000 sec
D8         0.89999998 sec
INO        0.00015149 sec
MCREST    0.0000000 sec
MCWRK     0.5000000 sec
ST1CNT    128

===== CHANNEL f1 =====
NUC1      1H
P1         10.50 usec
PL1        -5.00 dB
SF01      600.1330006 MHz

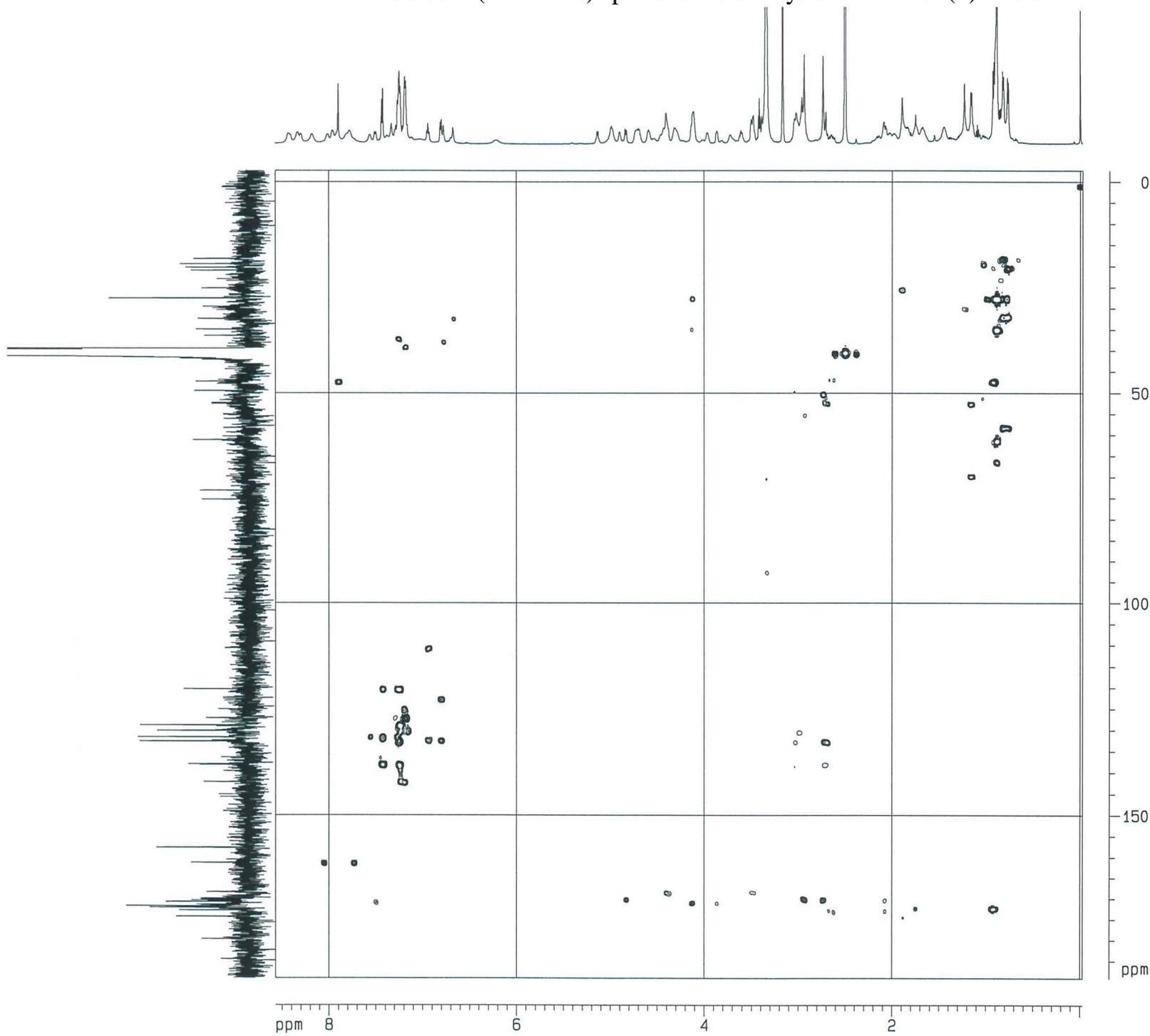
F1 - Acquisition parameters
ND0        1
TD         256
SF01       600.133 MHz
FIDRES     25.785955 Hz
SW         11.000 ppm
FnMODE     States-TPPI

F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         600.1300071 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      7.657 ppm
F2LO       4595.29 Hz
F2PHI      6.495 ppm
F2HI       3897.75 Hz
F1PLO      7.609 ppm
F1LO       4566.46 Hz
F1PHI      6.417 ppm
F1HI       3850.90 Hz
F2PPMCM    0.07749 ppm/cm
F2HZCM     46.50298 Hz/cm
F1PPMCM    0.07949 ppm/cm
F1HZCM     47.70401 Hz/cm
    
```

S71. HMBC (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO    16
PROCNO    1

F2 - Acquisition Parameters
Date_    20050705
Time     22.21
INSTRUM  spect
PROBHD   5 mm CPTX1 1H/
PULPROG  hmcgpp100af
TD       1024
SOLVENT  DMSO
NS       16
DS       4
SWH      6513.757 Hz
FIDRES   6.458747 Hz
AQ       0.0775400 sec
RG       18390.4
DM       75.500 usec
DE       5.00 usec
TE       298.0 K
CHST2    145.0000000
CHST13   10.0000000
d0       0.00000300 sec
d1       1.50000000 sec
d2       0.00344828 sec
d6       0.05000000 sec
d16      0.00020000 sec
IN0      0.00001380 sec
MCREST   0.00000000 sec
MCWRK    1.50000000 sec

----- CHANNEL f1 -----
NUC1     1H
P1       10.50 usec
p2       21.00 usec
PL1      -5.00 dB
SFO1     600.1330006 MHz

----- CHANNEL f2 -----
NUC2     13C
P3       15.00 usec
PL2      -5.00 dB
SFO2     150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1   SINE.100
GPNAM2   SINE.100
GPNAM3   SINE.100
GPX1     0.00 %
GPX2     0.00 %
GPX3     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPY3     0.00 %
GPZ1     50.00 %
GPZ2     30.00 %
GPZ3     40.10 %
P16      1000.00 usec

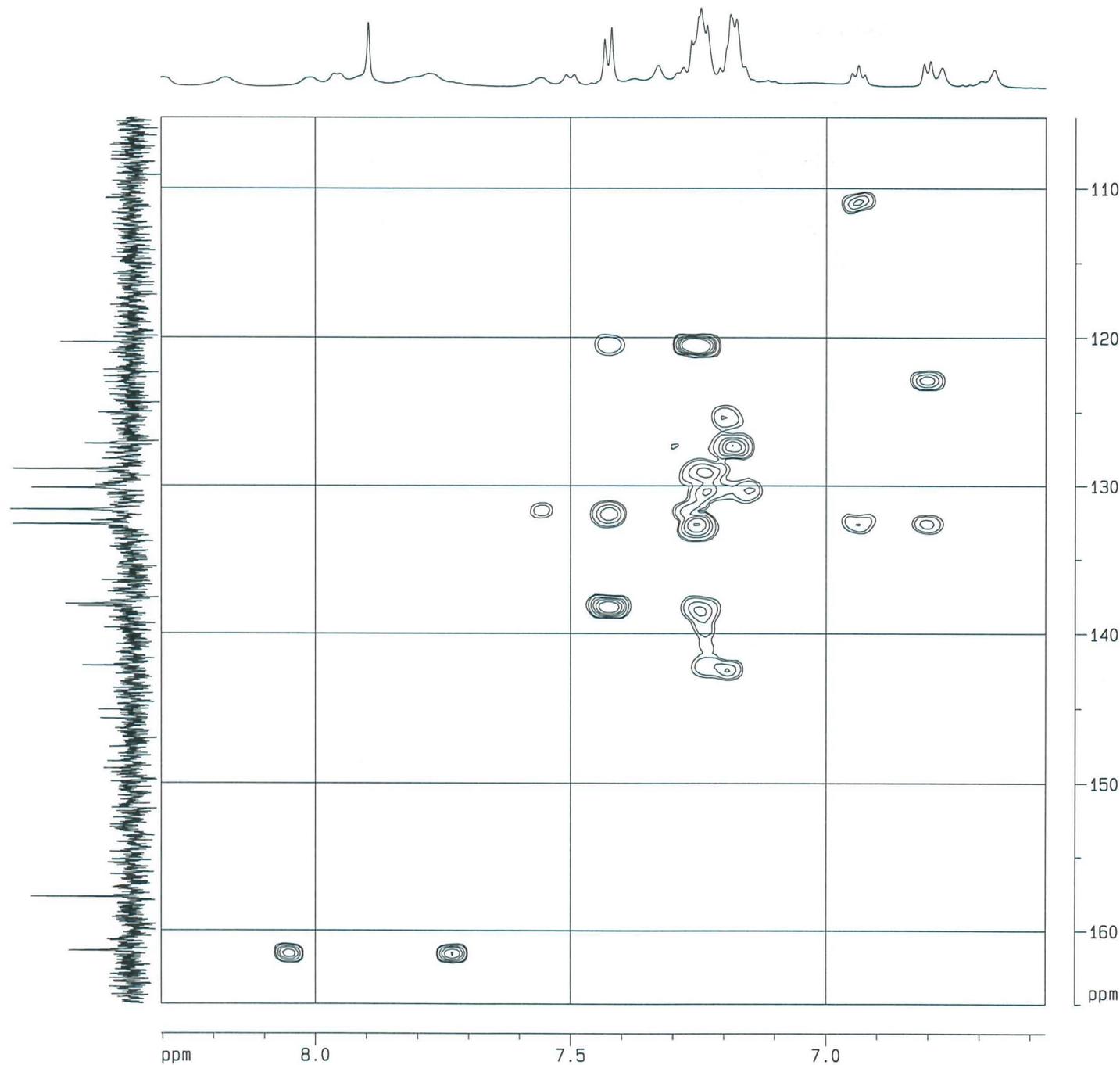
F1 - Acquisition parameters
ND0      2
TD       256
SFO1     150.9194 MHz
FIDRES   141.530792 Hz
SW       240.074 ppm
FMODE    GF

F2 - Processing parameters
SI       1024
SF       600.1300071 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
MC       GF
SF       150.9027181 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2P0LO   0.5772 ppm
F2L0     5144.28 Hz
F2PHI    -0.027 ppm
F2HI     -16.26 Hz
F1P0LO   189.151 ppm
F1L0     28543.43 Hz
F1PHI    -2.648 ppm
F1HI     -399.62 Hz
F2PPMCM  0.57327 ppm/cm
F2HZCM   344.03589 Hz/cm
F1PPMCM  12.78662 ppm/cm
F1HZCM   1929.53662 Hz/cm
    
```

S72. Expanded HMBC (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO     16
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      22.21
INSTRUM   spect
PROBHD    5 mm CPTXI 1H/
PULPROG   hmcgcp1pndrf
TD         1024
SOLVENT   DMSO
NS         16
DS         4
SWH        6613.757 Hz
FIDRES     6.458747 Hz
AQ         0.0775400 sec
RG         18390.4
DM         75.600 usec
DE         6.00 usec
TE         298.0 K
CNST2     145.0000000
CNST13    10.0000000
d0         0.00000300 sec
D1         1.50000000 sec
d2         0.00344628 sec
d5         0.05000000 sec
D16        0.00020000 sec
IN0        0.0001380 sec
MCREST    0.00000000 sec
MCHWK     1.50000000 sec

----- CHANNEL f1 -----
NUC1       1H
P1         10.50 usec
P2         21.00 usec
PL1        -5.00 dB
SFO1       600.1330006 MHz

----- CHANNEL f2 -----
NUC2       13C
P3         15.00 usec
PL2        -6.00 dB
SFO2       150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1    SINE.100
GPNAM2    SINE.100
GPNAM3    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPX3      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPY3      0.00 %
GPZ1      50.00 %
GPZ2      30.00 %
GPZ3      40.10 %
P16       1000.00 usec

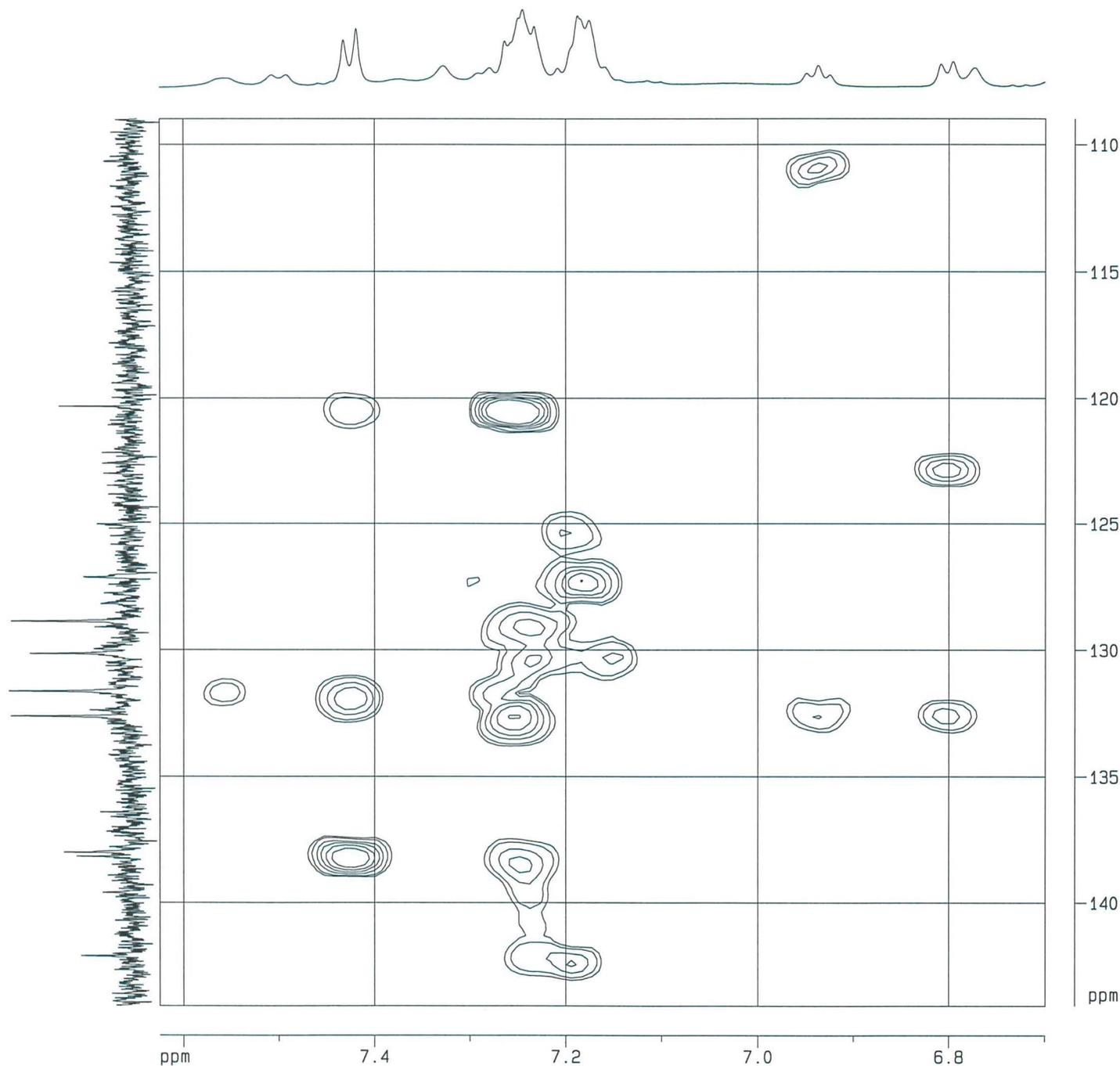
F1 - Acquisition parameters
ND0        2
TD          256
SFO1       150.9194 MHz
FIDRES     141.530792 Hz
SW         240.074 ppm
FNM0DE     OF

F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MC2        OF
SF         150.9027181 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      8.303 ppm
F2L0       4982.81 Hz
F2PH1      6.570 ppm
F2H1       3942.96 Hz
F1PLO      165.000 ppm
F1L0       24899.02 Hz
F1PH2      105.210 ppm
F1H1       15876.44 Hz
F2PPMCM    0.11551 ppm/cm
F2HZCM     69.32387 Hz/cm
F1PPMCM    3.98605 ppm/cm
F1HZCM     601.50555 Hz/cm
    
```

S73. Expanded HMBC (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      ju104-mu-chn
EXPNO     16
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      22.21
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPROG   hmbcpgpindrf
TD         1024
SOLVENT   DMSO
NS         16
DS         4
SWH        6613.757 Hz
FIDRES     6.458747 Hz
AQ         0.0775400 sec
RG         18390.4
DM         75.600 usec
DE         6.00 usec
TE         298.0 K
CNST2     145.0000000
CNST13    10.0000000
d0         0.0000300 sec
D1         1.5000000 sec
d2         0.00346028 sec
d5         0.0500000 sec
D16        0.0002000 sec
IN0        0.0001380 sec
MCREST     0.0000000 sec
MCRWK      1.5000000 sec

----- CHANNEL f1 -----
NUC1       1H
P1         10.50 usec
PL1        -5.00 dB
PL12       -5.00 dB
SFO1       600.1330006 MHz

----- CHANNEL f2 -----
NUC2       13C
P2         15.00 usec
PL2        -6.00 dB
SFO2       150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1     SINE.100
GPNAM2     SINE.100
GPNAM3     SINE.100
GPX1       0.00 %
GPX2       0.00 %
GPX3       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPY3       0.00 %
GPZ1       50.00 %
GPZ2       30.00 %
GPZ3       40.10 %
P16        1000.00 usec

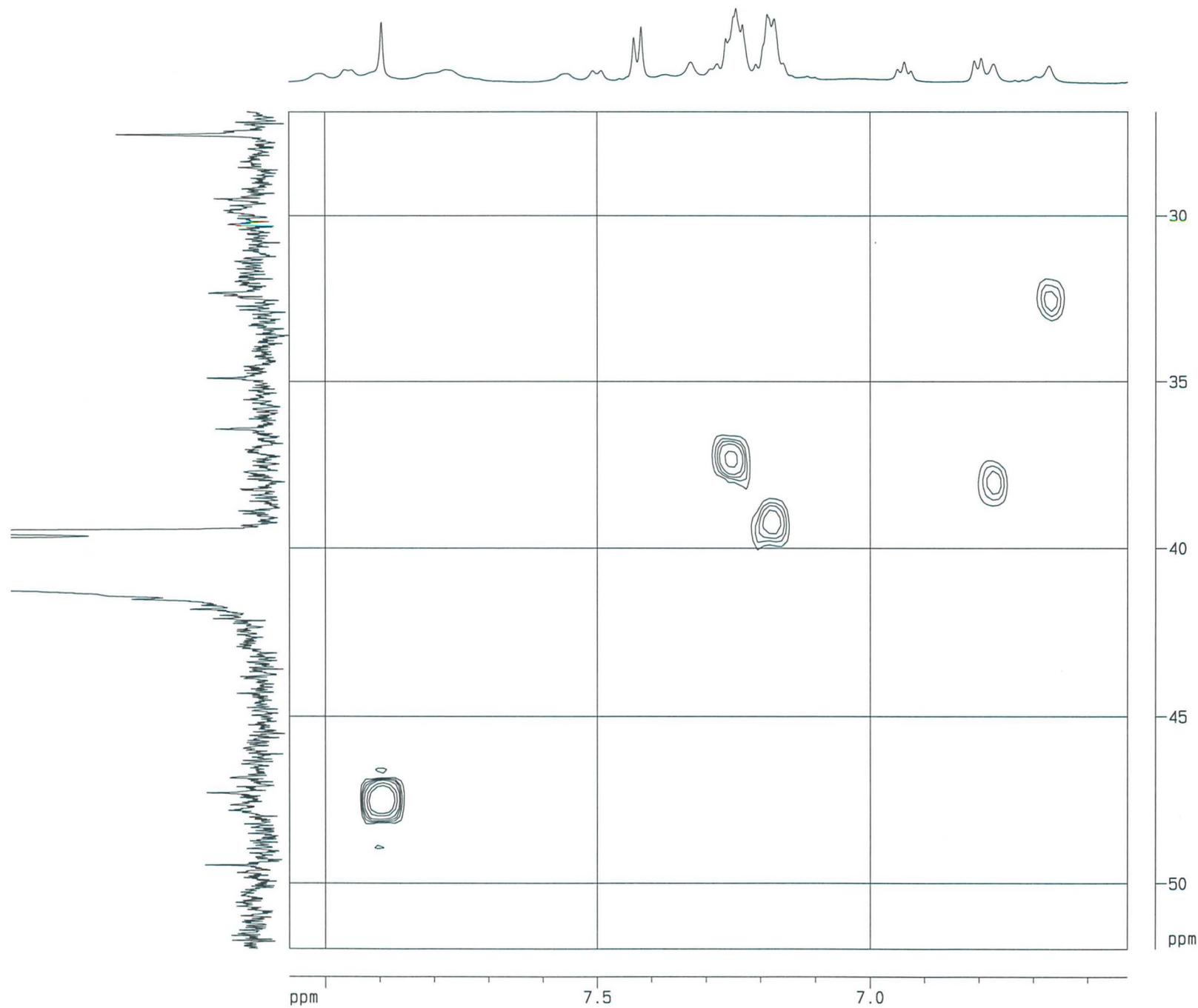
F1 - Acquisition parameters
ND0        2
TD         256
SFO1       150.9194 MHz
FIDRES     141.530792 Hz
SW         240.074 ppm
FNM0DE     gF

F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MC2        gF
SF         150.9027181 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1         5.00 cm
F2P1LO     7.625 ppm
F2L0       4575.31 Hz
F2PHI      6.899 ppm
F2HI       4020.46 Hz
F1P1LO     144.132 ppm
F1L0       21749.96 Hz
F1PHI     108.961 ppm
F1HI       16442.56 Hz
F2PPMCM    0.06170 ppm/cm
F2HZCM     37.03016 Hz/cm
F1PPMCM    2.34473 ppm/cm
F1HZCM     353.82678 Hz/cm
    
```

S74. Expanded HMBC (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      jul04-gnu-cn1
EXPNO    16
PROCNO   1

F2 - Acquisition Parameters
Date_    20050705
Time     22.21
INSTRUM  spect
PROBHD   5 mm CPTXI 1H/
PULPROG  hbcbcp1prndf
TD       1024
SOLVENT  DMSO
NS       16
DS       4
SMH      6613.757 Hz
FIDRES   0.408747 Hz
AQ       0.0775400 sec
RG       18390.4
DM       75.600 usec
DE       6.00 usec
TE       298.0 K
CNST2    145.0000000
CNST13   10.0000000
d0       0.00000300 sec
D1       1.50000000 sec
d2       0.00344028 sec
d5       0.05000000 sec
D16      0.00020000 sec
IN0      0.00001380 sec
MCREST   0.00000000 sec
MCMRK    1.50000000 sec

----- CHANNEL f1 -----
NUC1     1H
P1       10.50 usec
P2       21.00 usec
PL1      -5.00 dB
SFO1     600.1330006 MHz

----- CHANNEL f2 -----
NUC2     13C
P3       15.00 usec
PL2      -6.00 dB
SFO2     150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1   SINE.100
GPNAM2   SINE.100
GPNAM3   SINE.100
GPX1     0.00 %
GPX2     0.00 %
GPX3     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPY3     0.00 %
GPZ1     50.00 %
GPZ2     30.00 %
GPZ3     40.10 %
P16      1000.00 usec

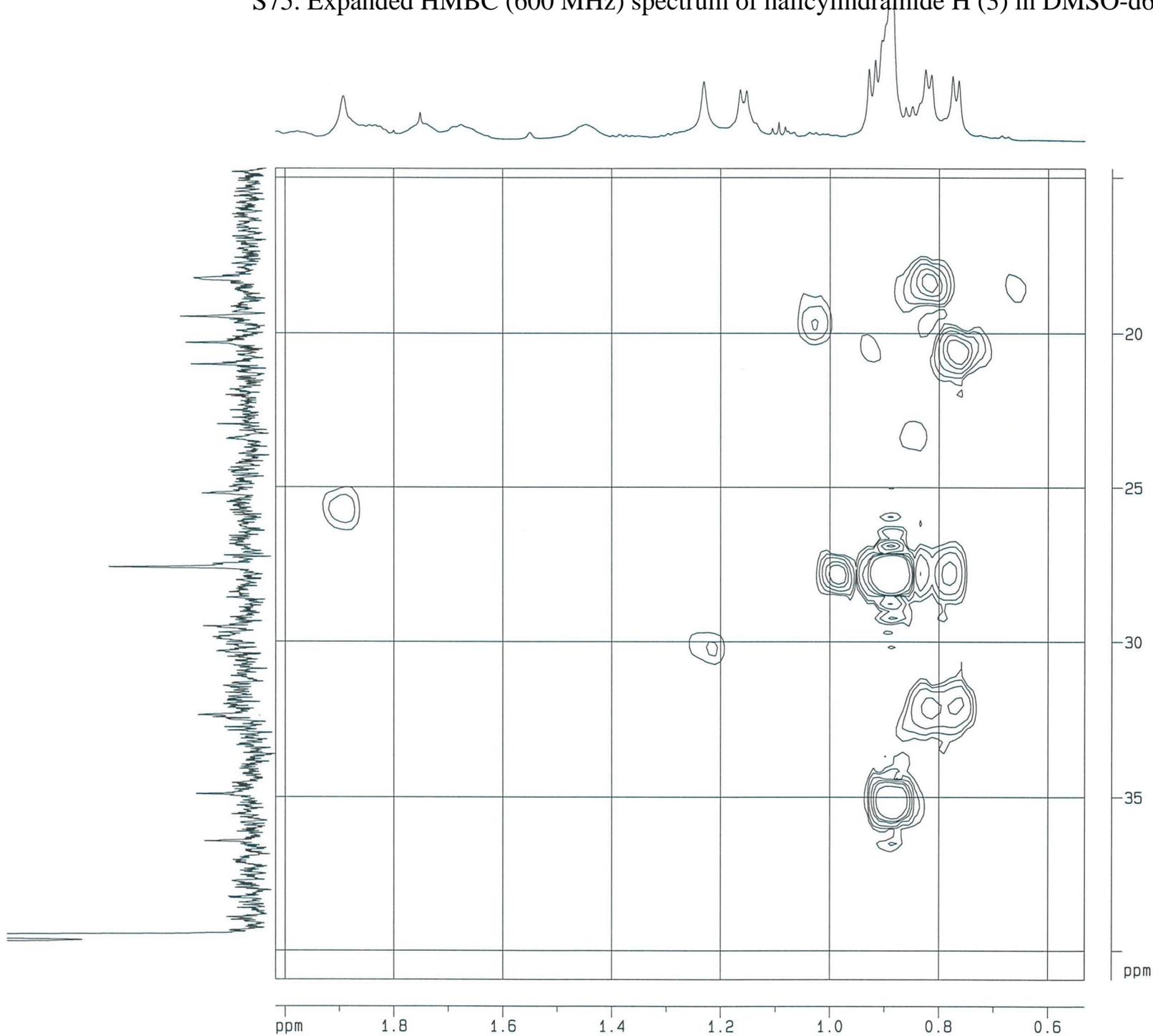
F1 - Acquisition parameters
ND0      2
TD       256
SFO1     150.9194 MHz
FIDRES   141.530792 Hz
SW       240.074 ppm
FNM0DE   gF

F2 - Processing parameters
SI       1024
SF       600.1300071 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
MC2      gF
SF       150.9027181 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2PLO    8.066 ppm
F2L0     4840.72 Hz
F2PHI    6.527 ppm
F2HI     3917.12 Hz
F1PLO    51.984 ppm
F1L0     7844.57 Hz
F1PHI    26.896 ppm
F1HI     4058.62 Hz
F2PPMCM  0.10260 ppm/cm
F2HZCM   61.57340 Hz/cm
F1PPMCM  1.67258 ppm/cm
F1HZCM   252.39644 Hz/cm
    
```

S75. Expanded HMBC (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      jul04-shu-chn
EXPNO    16
PROCNO    1

F2 - Acquisition Parameters
Date_    20050705
Time     22.21
INSTRUM  spect
PROBHD   5 mm CPTXI 1H/
PULPROG  hbcbgplndrf
TD        1024
SOLVENT  DMSO
NS        16
DS        4
SWH       6613.757 Hz
FIDRES    6.458747 Hz
AQ        0.0775400 sec
RG        18390.4
DM        75.600 usec
DE        6.00 usec
TE        298.0 K
CNST2    145.0000000
CNST13   10.0000000
d0        0.00000300 sec
D1        1.50000000 sec
D2        0.00344828 sec
d5        0.00000000 sec
D16       0.00020000 sec
IN0       0.0001380 sec
MCREST   0.0000000 sec
MCWRK    1.50000000 sec

----- CHANNEL f1 -----
NUC1      1H
P1        10.50 usec
P2        21.00 usec
PL1       -5.00 dB
PL2       -5.00 dB
SFO1      600.1330006 MHz

----- CHANNEL f2 -----
NUC2      13C
P3        15.00 usec
P4        15.00 usec
PL2       -6.00 dB
PL4       -6.00 dB
SFO2      150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1    SINE.100
GPNAM2    SINE.100
GPNAM3    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPX3      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPY3      0.00 %
GPZ1      50.00 %
GPZ2      30.00 %
GPZ3      40.10 %
P16       1000.00 usec

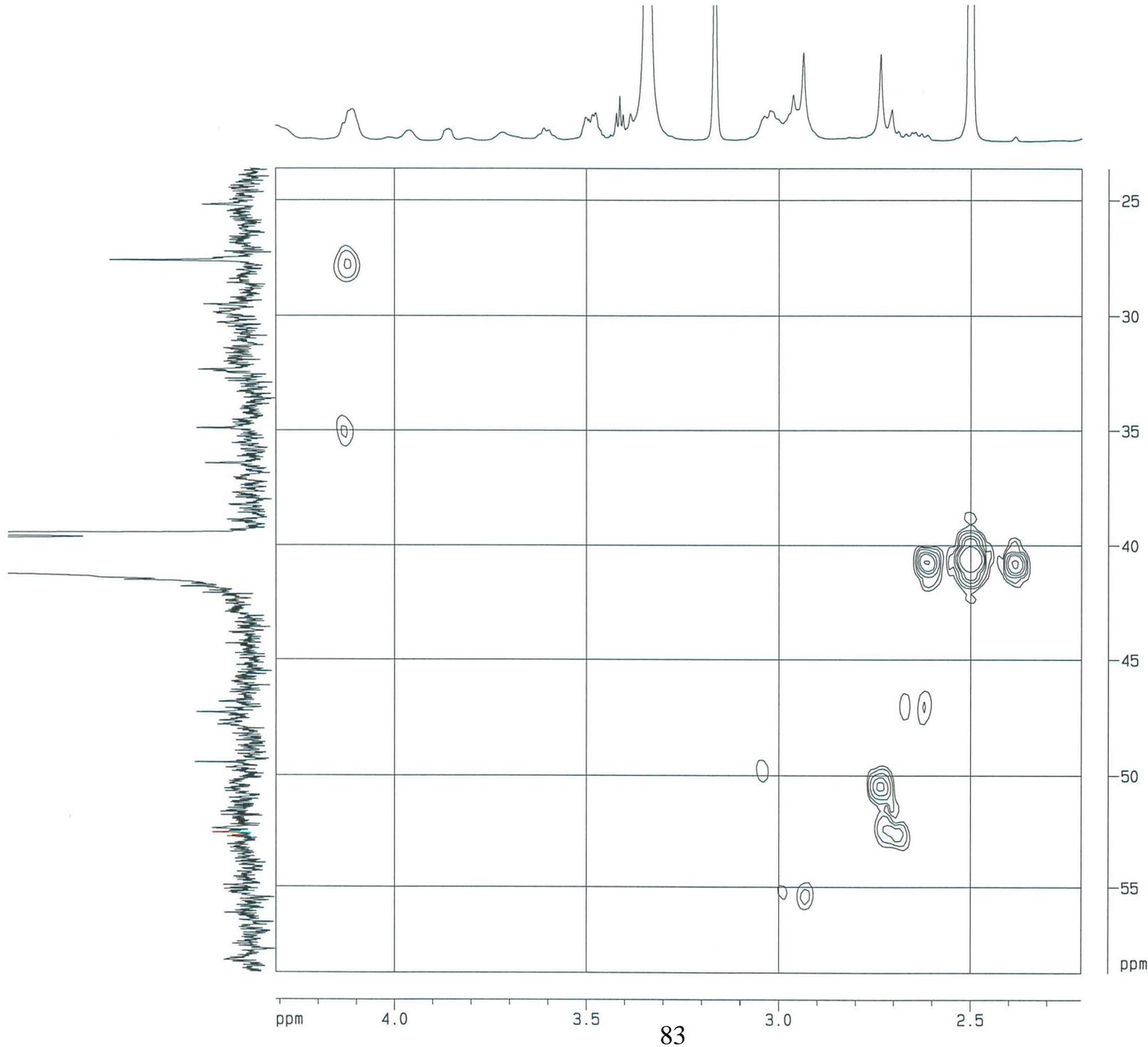
F1 - Acquisition parameters
ND0       2
TD        256
SFO1      150.9194 MHz
FIDRES    141.530792 Hz
SW        240.074 ppm
FHM0DE    GF

F2 - Processing parameters
SI        1024
SF        600.1300071 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
MC2       GF
SF        150.9027181 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     2.018 ppm
F2LO      1210.91 Hz
F2PH1     0.533 ppm
F2H1      319.60 Hz
F1PLO     40.964 ppm
F1LO      6181.58 Hz
F1PH1     14.703 ppm
F1H1      2218.72 Hz
F2PPMCM   0.09901 ppm/cm
F2HZCM    59.42047 Hz/cm
F1PPMCM   1.75073 ppm/cm
F1HZCM    264.19067 Hz/cm
    
```

S76. Expanded HMBC (600 MHz) spectrum of halicyclindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO     16
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      22.21
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPROG   hmcgcp1pndsf
TD        1024
SOLVENT   DMSO
NS        16
DS        4
SWH       6613.757 Hz
FIDRES    6.458747 Hz
AQ        0.0775400 sec
RG        18390.4
DW        75.600 usec
DE        6.00 usec
TE        298.0 K
CNST2     145.0000000
CNST13    10.0000000
d0        0.00000300 sec
d1        1.50000000 sec
d2        0.00346028 sec
d6        0.05000000 sec
d16       0.00020000 sec
IN0       0.00001380 sec
MCREST    0.00000000 sec
MCMWK     1.50000000 sec

----- CHANNEL f1 -----
NUC1      1H
P1        10.50 usec
p2        21.00 usec
PL1       -5.00 dB
SFO1      600.1330006 MHz

----- CHANNEL f2 -----
NUC2      13C
P3        15.00 usec
PL2       -6.00 dB
SFO2      150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1    SINE.100
GPNAM2    SINE.100
GPNAM3    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPX3      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPY3      0.00 %
GPZ1      50.00 %
GPZ2      30.00 %
GPZ3      40.10 %
P16       1000.00 usec

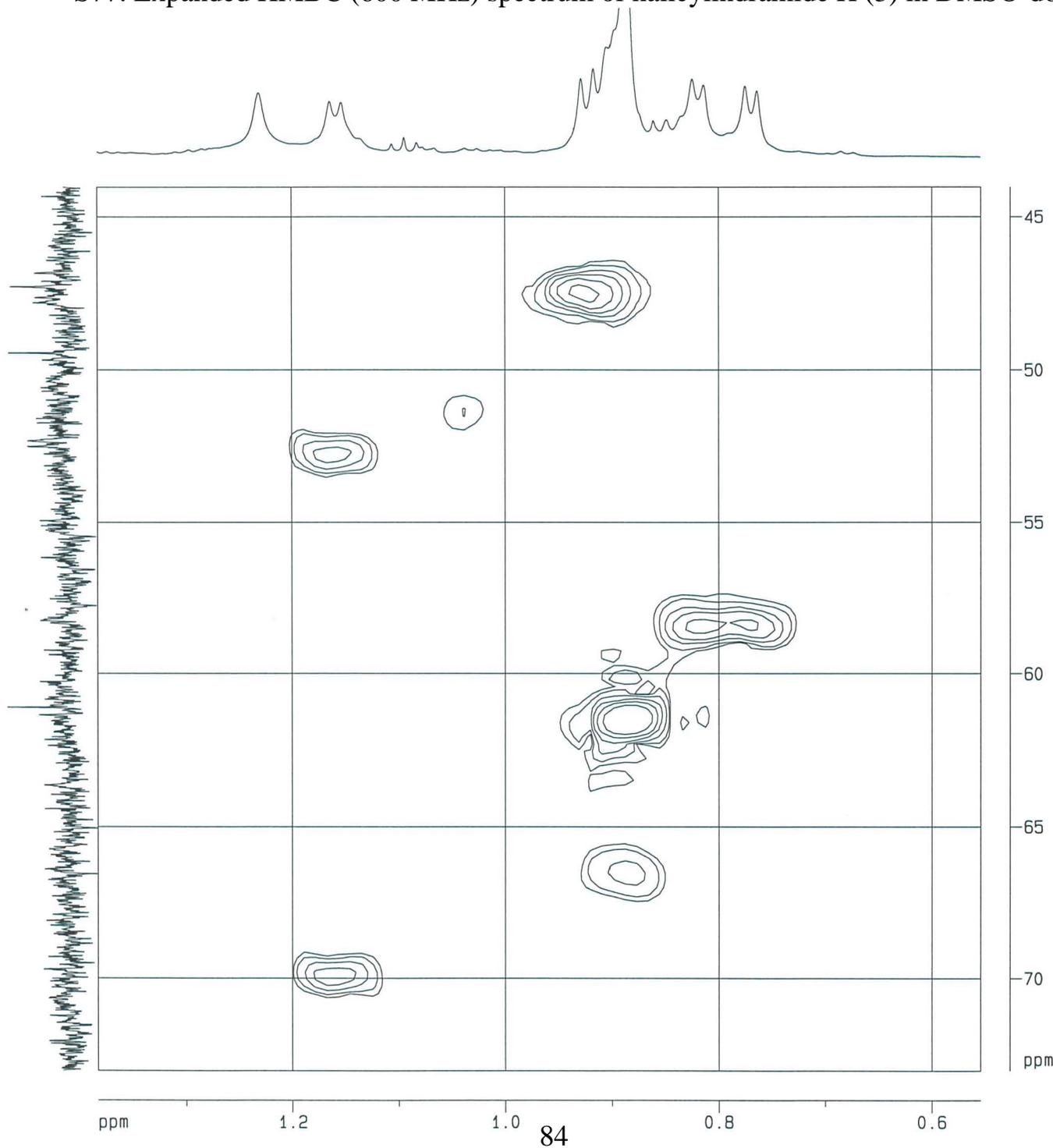
F1 - Acquisition parameters
ND0       2
TD        256
SFO1      150.9194 MHz
FIDRES    141.530792 Hz
SW        240.074 ppm
FNUCDE    gf

F2 - Processing parameters
SI        1024
SF        600.1300071 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
MC2       gf
SF        150.9027181 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     4.310 ppm
F2LO      2586.62 Hz
F2PHI     2.211 ppm
F1PHI     1367.16 Hz
F1PLO     58.784 ppm
F1LO      8870.67 Hz
F1PHI     23.613 ppm
F1HI      3563.27 Hz
F2PPMCH   0.13991 ppm/cm
F2HZCM    83.96371 Hz/cm
F1PPMCH   2.34473 ppm/cm
F1HZCM    353.82678 Hz/cm
    
```

S77. Expanded HMBC (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      jul04-stu-eh
EXPNO     15
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      22.21
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPROG   hmbcpg1pndrf
TD         1024
SOLVENT   DMSO
NS         16
DS         4
SMH        6613.757 Hz
FIDRES     6.458747 Hz
AQ         0.0775400 sec
RG         18380.4
DW         75.600 usec
DE         6.00 usec
TE         298.0 K
CNS12     145.0000000
CNS13     10.0000000
d0         0.00000300 sec
D1         1.50000000 sec
d2         0.00344828 sec
d5         0.00000000 sec
D16        0.00020000 sec
IN0        0.00001380 sec
MCREST    0.00000000 sec
MCWRK     1.50000000 sec

----- CHANNEL f1 -----
NUC1       1H
P1         10.50 usec
p2         21.00 usec
PL1        -6.00 dB
SFO1       600.1330006 MHz

----- CHANNEL f2 -----
NUC2       13C
P3         15.00 usec
PL2        -6.00 dB
SFO2       150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1    SINE.100
GPNAM2    SINE.100
GPNAM3    SINE.100
GPX1       0.00 %
GPX2       0.00 %
GPX3       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPY3       0.00 %
GPT1       50.00 %
GPT2       30.00 %
GPT3       40.10 %
P16        1000.00 usec

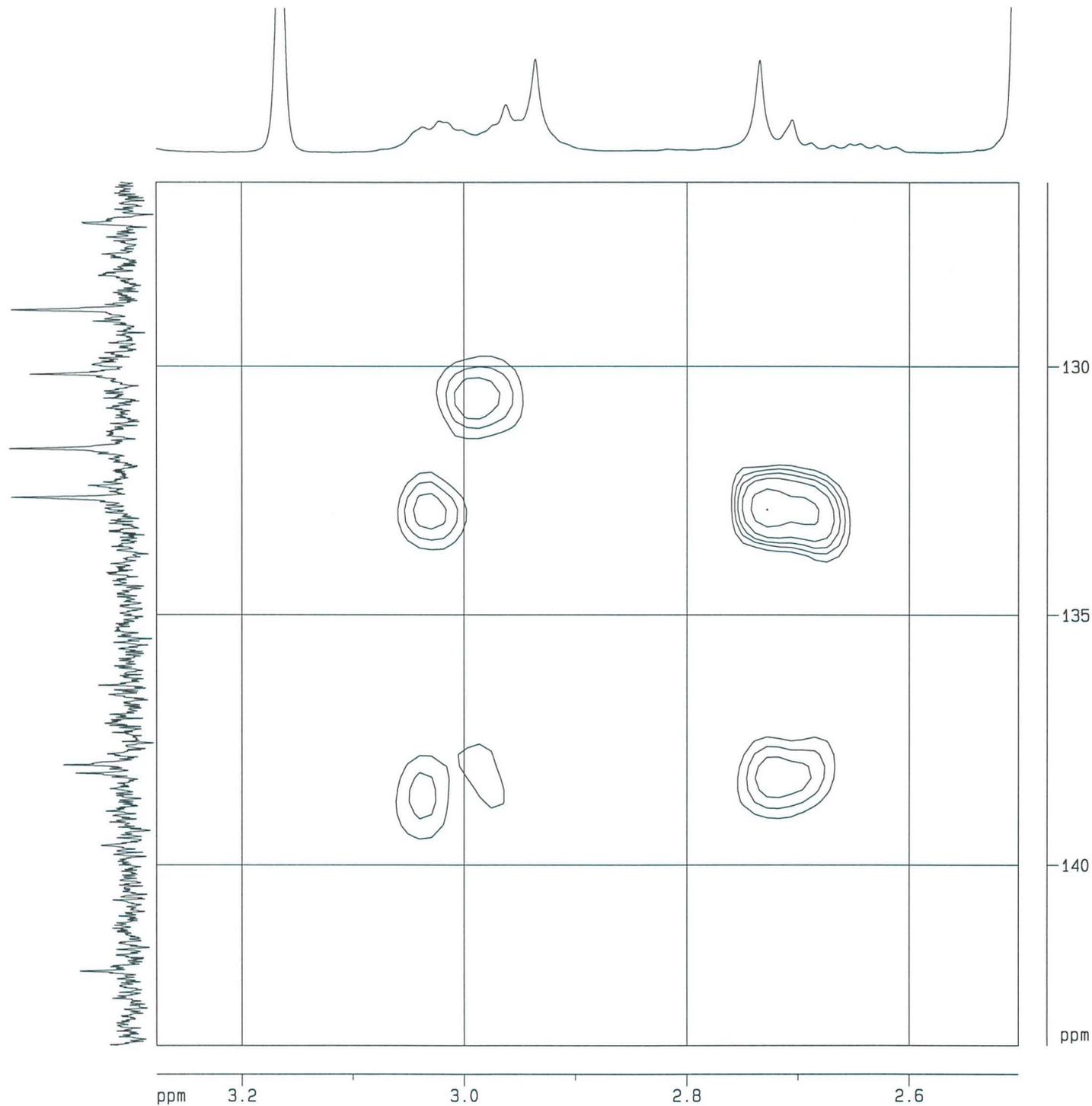
F1 - Acquisition parameters
ND0        2
T0         256
SFO1       150.9194 MHz
FIDRES     141.530792 Hz
SW         240.074 ppm
FHM0DE     GF

F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        SINE
SSB         0
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MC2        GF
SF         150.9027181 MHz
WDW        SINE
SSB         0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1         15.00 cm
F2PLO      1.383 ppm
F2PLO      809.84 Hz
F2PHI      0.554 ppm
F2HI       332.52 Hz
F1PLO      73.087 ppm
F1LO       11029.01 Hz
F1PHI      44.012 ppm
F1HI       6641.55 Hz
F2PPMCM    0.05525 ppm/cm
F2HZCM     33.15490 Hz/cm
F1PPMCM    1.93831 ppm/cm
F1HZCM     292.49683 Hz/cm
    
```

S78. Expanded HMBC (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME          ju104-snu-ch
EXPNO         16
PROCNO        1

F2 - Acquisition Parameters
Date_         20050705
Time          22.21
INSTRUM       spect
PROBHD        5 mm CPTXI 1H/
PULPROG       hmbcgp1dqdf
TD            1024
SOLVENT       DMSO
NS            16
DS            4
SWH           6613.757 Hz
FIDRES        6.458747 Hz
AQ            0.0775400 sec
RG            18390.4
DM            75.600 usec
DE            6.00 usec
TE            298.0 K
CNST2         145.0000000
CNST13        10.0000000
d0            0.00000300 sec
d1            1.50000000 sec
d2            0.00344828 sec
d5            0.05000000 sec
d16           0.00020000 sec
INO           0.00001380 sec
MCREST        0.00000000 sec
MCWRK         1.50000000 sec

----- CHANNEL f1 -----
NUC1           1H
P1             10.50 usec
P2             21.00 usec
PL1            -5.00 dB
SFO1           600.1330006 MHz

----- CHANNEL f2 -----
NUC2           13C
P3             15.00 usec
PL2            -6.00 dB
SFO2           150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1        SINE.100
GPNAM2        SINE.100
GPNAM3        SINE.100
GPX1           0.00 %
GPX2           0.00 %
GPX3           0.00 %
GPY1           0.00 %
GPY2           0.00 %
GPY3           0.00 %
GPZ1           50.00 %
GPZ2           30.00 %
GPZ3           40.10 %
P1B            1000.00 usec

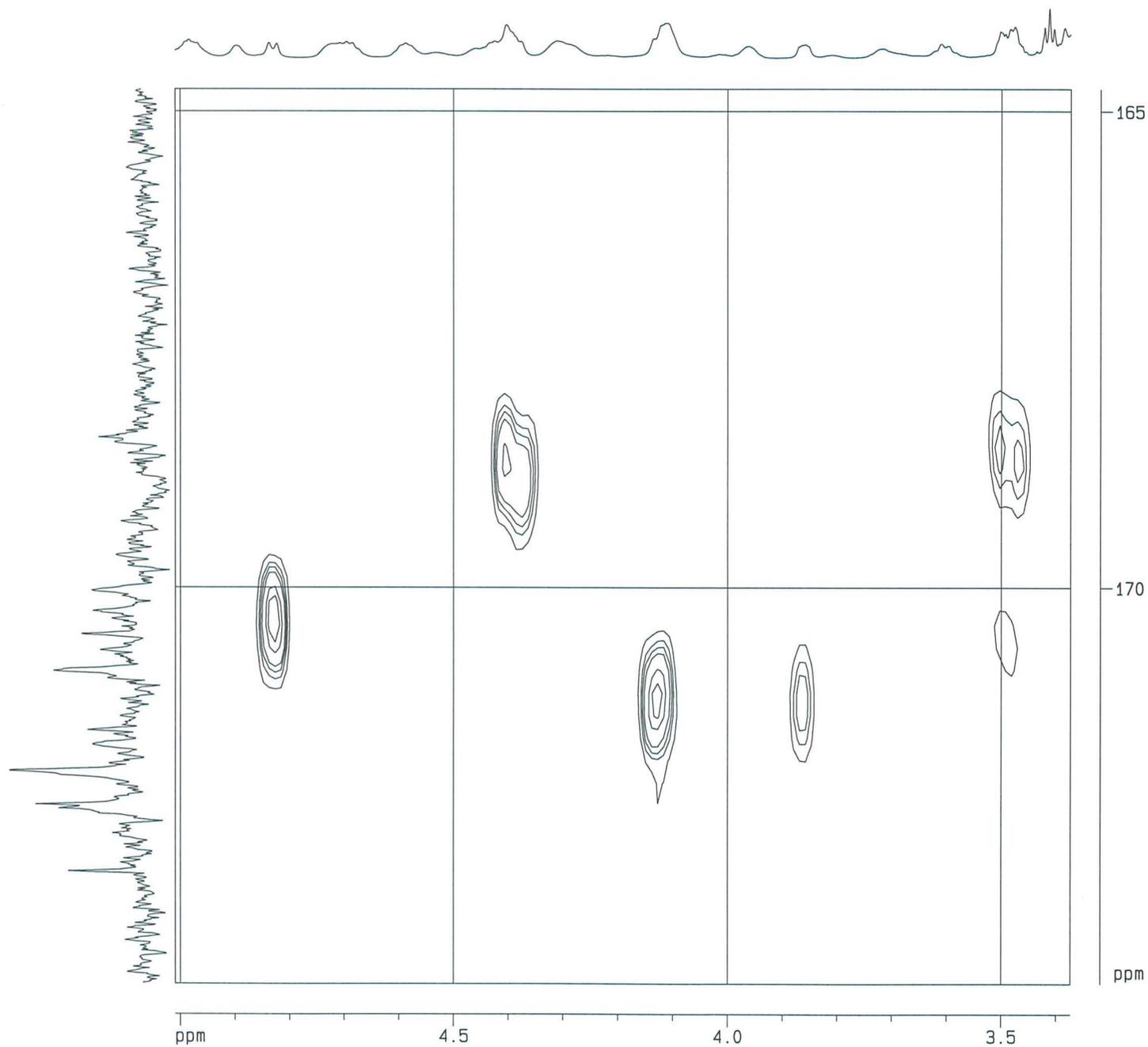
F1 - Acquisition parameters
MDO            2
TD             256
SFO1           150.9194 MHz
FIDRES         141.530792 Hz
SW             240.074 ppm
FHM0DE         GF

F2 - Processing parameters
SI             1024
SF             600.1300071 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0
PC             1.40

F1 - Processing parameters
SI             1024
MC2            GF
SF             150.9027161 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0

2D NMR plot parameters
CX2            15.00 cm
CX1            15.00 cm
F2PLO          3.277 ppm
F2L0           1965.58 Hz
F2PHI          2.502 ppm
F2H1           1501.55 Hz
F1PLO          143.663 ppm
F1L0           21679.20 Hz
F1PHI          125.312 ppm
F1H1           19060.88 Hz
F2PPMCM        0.05166 ppm/cm
F2HZCM         31.00198 Hz/cm
F1PPMCM        1.15674 ppm/cm
F1HZCM         174.95458 Hz/cm
    
```

S79. Expanded HMBC (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      ju104-snu-chn
EXPNO    16
PROCNO   1

F2 - Acquisition Parameters
Date_    20090705
Time     22.21
INSTRUM  spect
PROBHD   5 mm CPXI 1H/
PULPROG  hmbcgp1pndqf
TD       1024
SOLVENT  DMSO
NS       16
DS       4
SMH      6613.757 Hz
FIDRES   6.458747 Hz
AQ       0.0775400 sec
RG       18390.4
DM       75.600 usec
DE       6.00 usec
TE       298.0 K
CNST2    145.0000000
CNST13   10.0000000
d0       0.00000300 sec
D1       1.50000000 sec
D2       0.00344628 sec
D5       0.00000000 sec
D16      0.00020000 sec
IN0      0.00001380 sec
MCREST   0.00000000 sec
MCWRK    1.50000000 sec

----- CHANNEL f1 -----
NUC1      1H
P1        10.50 usec
P2        21.00 usec
PL1       -5.00 dB
SFO1      600.1330006 MHz

----- CHANNEL f2 -----
NUC2      13C
P3        15.00 usec
P4        -6.00 dB
SFO2      150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1    SINE.100
GPNAM2    SINE.100
GPNAM3    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPX3      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPY3      0.00 %
GPT1      50.00 %
GPT2      30.00 %
GPT3      40.10 %
P16       1000.00 usec

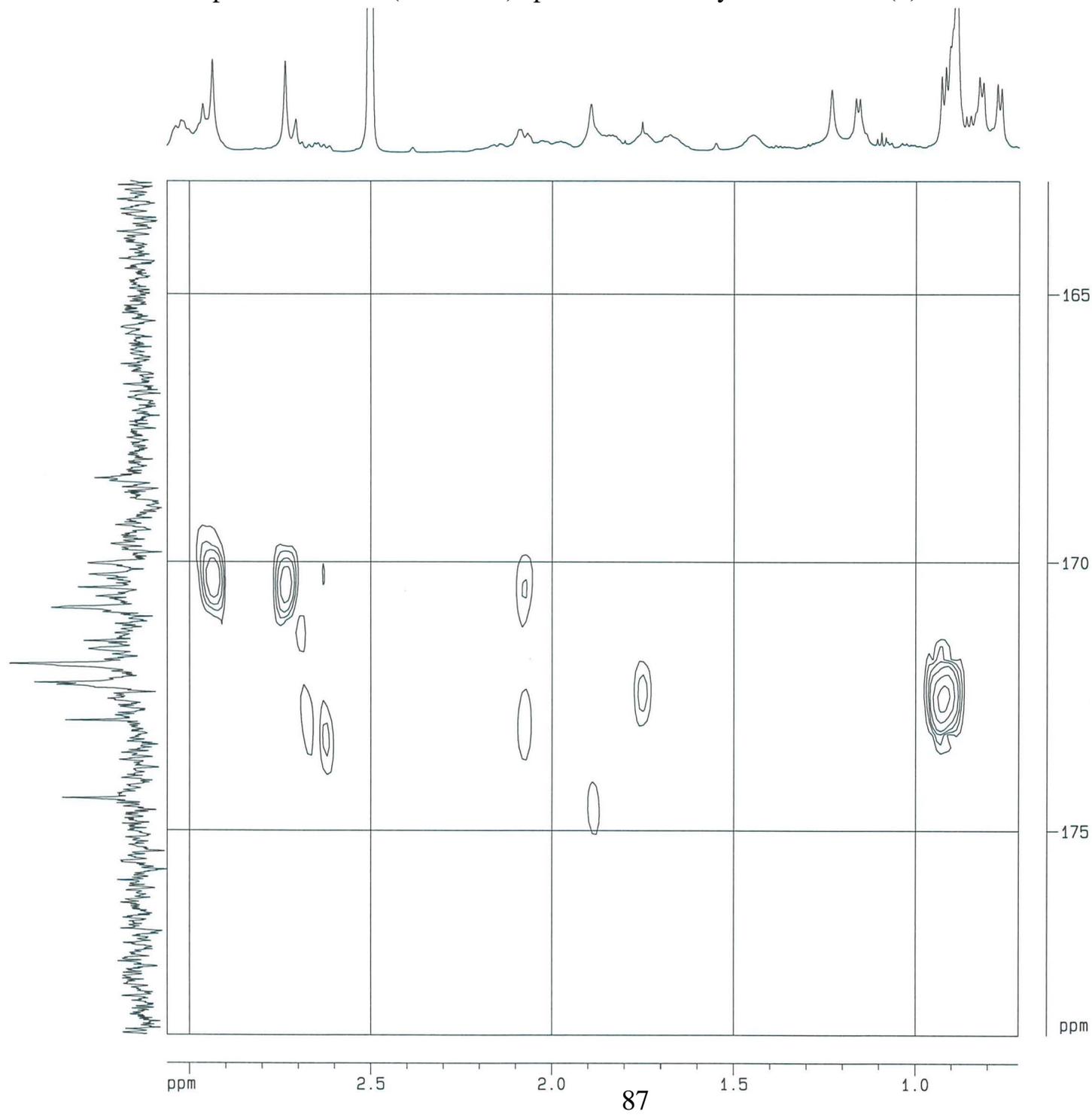
F1 - Acquisition parameters
ND0       2
TD        256
SFO1      150.9194 MHz
FIDRES    141.530792 Hz
SH        240.074 ppm
FNMDC2    GF

F2 - Processing parameters
SI        1024
SF        600.1300071 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
MC2       GF
SF        150.9027181 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     5.010 ppm
F2LO      3006.44 Hz
F2PHI     3.374 ppm
F2HI      2024.71 Hz
F2PLO     174.145 ppm
F2LO      26278.95 Hz
F2PHI     184.786 ppm
F2HI      24863.64 Hz
F2PPMCM   0.10806 ppm/cm
F2HZCM    65.44863 Hz/cm
F1PPMCM   0.62526 ppm/cm
F1HZCM    94.35381 Hz/cm
    
```

S80. Expanded HMBC (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      ju104-snu-ch
EXPNO     16
PROCNO    1

F2 - Acquisition Parameters
Date_     20050705
Time      22.21
INSTRUM   spect
PROBHD    5 mm CPTX1 1H/
PULPROG   hmcgpp1pndqf
TD         1024
SOLVENT   DMSO
NS         16
DS         4
SMH        6613.757 Hz
FIDRES     6.458747 Hz
AQ         0.0775400 sec
RG         16390.4
DM         75.600 usec
DE         6.00 usec
TE         298.0 K
CNST2     145.0000000
CNST13    10.0000000
d0         0.0000000 sec
D1         1.5000000 sec
d2         0.00344828 sec
d5         0.05000000 sec
D15        0.00020000 sec
lAQ        0.00001360 sec
MCREST     0.00000000 sec
MWRK      1.50000000 sec

----- CHANNEL f1 -----
NUC1       1H
P1         10.50 usec
p2         21.00 usec
PL1        -5.00 dB
SF01       600.1330006 MHz

----- CHANNEL f2 -----
NUC2       13C
P3         15.00 usec
PL2        -6.00 dB
SF02       150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1     SINE:100
GPNAM2     SINE:100
GPNAM3     SINE:100
GPX1       0.00 %
GPX2       0.00 %
GPX3       0.00 %
GPH1       0.00 %
GPH2       0.00 %
GPH3       0.00 %
GPZ1       50.00 %
GPZ2       30.00 %
GPZ3       40.10 %
P15        1000.00 usec

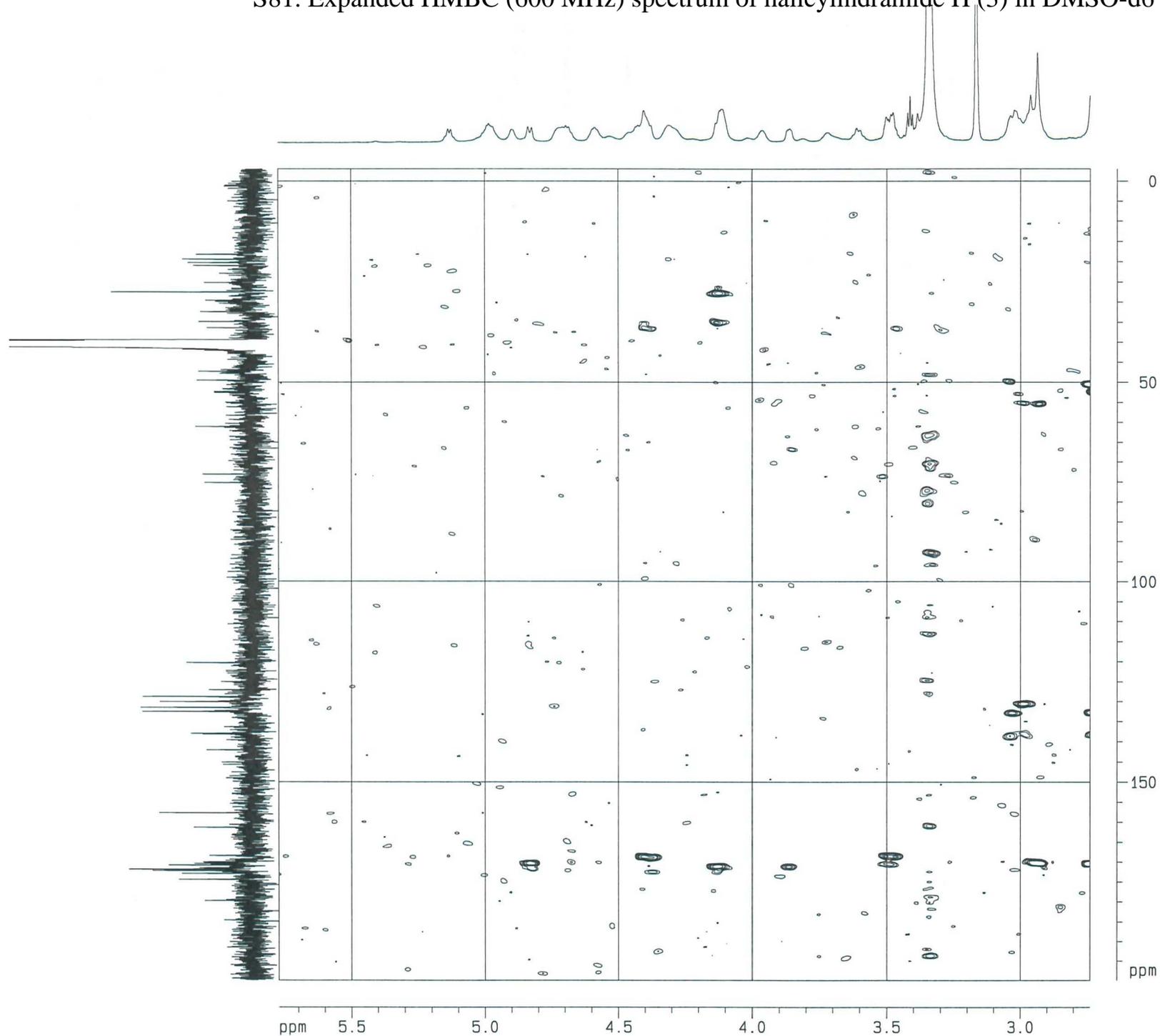
F1 - Acquisition parameters
ND0        2
TD         256
SF01       150.9194 MHz
FIDRES     141.530792 Hz
SM         240.074 ppm
FnMODE     QF

F2 - Processing parameters
SI         1024
SF         600.1300071 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MC2        QF
SF         150.9027181 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1         15.00 cm
F2PLO      3.062 ppm
F2L0       1837.40 Hz
F2PHI      0.716 ppm
F2HL       429.40 Hz
F1PLO      178.634 ppm
F1L0       26986.6 Hz
F1PHI      162.890 ppm
F1HI       24580.58 Hz
F2PVMCH    0.15541 ppm/cm
F2HZCM     93.85712 Hz/cm
F1PVMCH    1.05295 ppm/cm
F1HZCM     160.40154 Hz/cm
    
```

S81. Expanded HMBC (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      ju104-smu-chj
EXPNO    16
PROCNO   1

F2 - Acquisition Parameters
Date_    20050705
Time     22.21
INSTRUM  spect
PROBHD   5 mm CPTX1 1H/
PULPROG  hmcpcp1ndqf
TD       1024
SOLVENT  DMSO
NS       15
DS       4
SMH      6613.757 Hz
FIDRES   6.458747 Hz
AQ       0.0775400 sec
RG       18390.4
DW       75.600 usec
DE       6.00 usec
TE       298.0 K
CNST2    145.000000
CNST13   10.000000
d0       0.00000300 sec
d1       1.50000000 sec
d2       0.00344828 sec
d6       0.05000000 sec
d16      0.00020000 sec
IND      0.0001360 sec
MCREST   0.0000000 sec
MCMRK    1.5000000 sec

----- CHANNEL f1 -----
NUC1     1H
P1       10.50 usec
p2       21.00 usec
PL1      -5.00 dB
SFO1     600.1330006 MHz

----- CHANNEL f2 -----
NUC2     13C
P3       15.00 usec
PL2      -5.00 dB
SFO2     150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1   SINE:100
GPNAM2   SINE:100
GPNAM3   SINE:100
GPX1     0.00 %
GPX2     0.00 %
GPX3     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPY3     0.00 %
GPZ1     50.00 %
GPZ2     30.00 %
GPZ3     40.10 %
P16      1000.00 usec

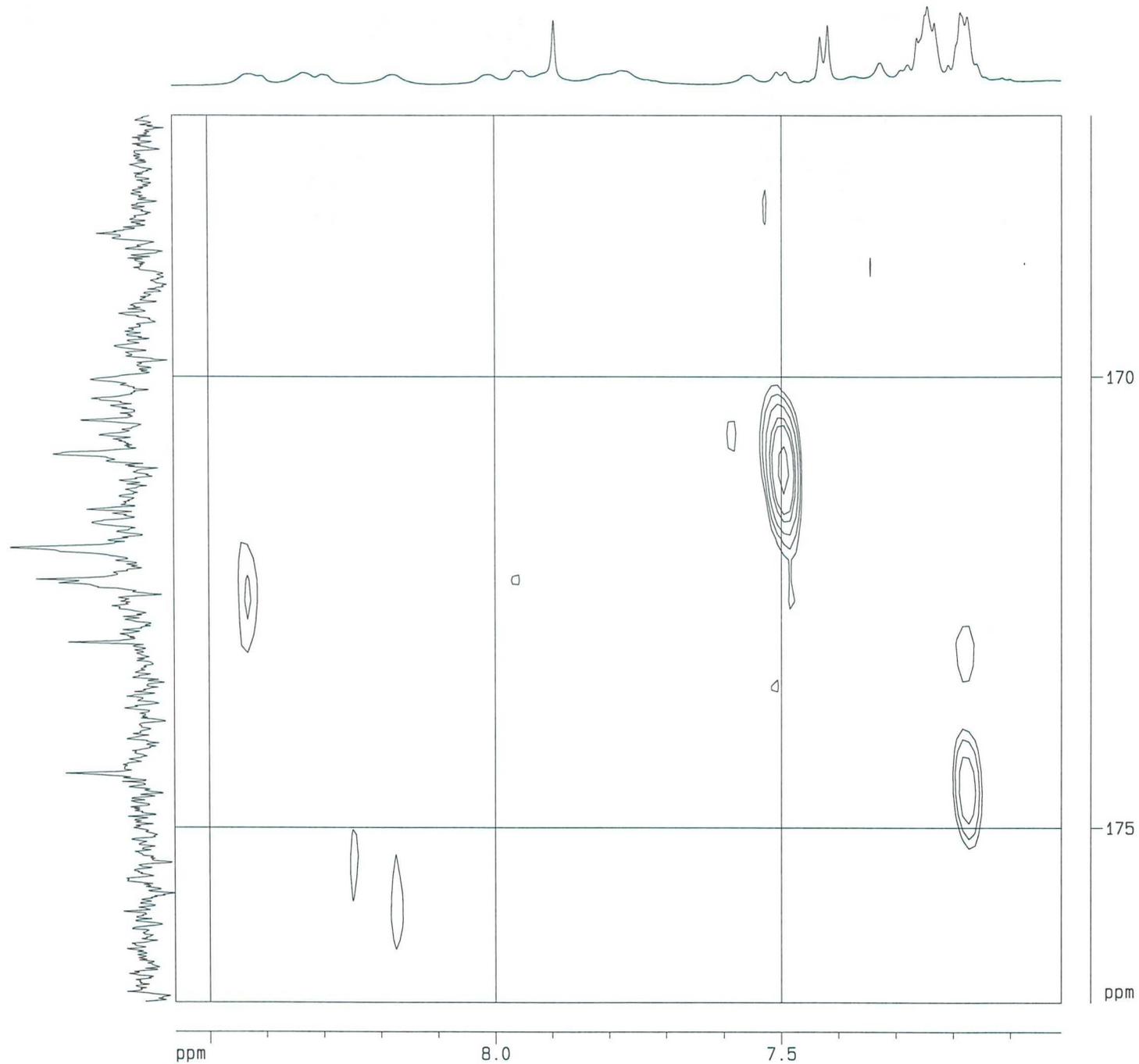
F1 - Acquisition parameters
ND0      2
TD       256
SFO1     150.9194 MHz
FIDRES   141.530792 Hz
SW       240.074 ppm
FMODE    QF

F2 - Processing parameters
SI       1024
SF       600.1300071 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
MC2      QF
SF       150.9027181 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2PLO    5.774 ppm
F2LO     3485.01 Hz
F2PHI    2.739 ppm
F2HI     1643.54 Hz
F1PLO    193.837 ppm
F1LO     30171.03 Hz
F1PHI    -3.117 ppm
F1HI     -470.39 Hz
F2P2MCM 0.20233 ppm/cm
F2HZCM   121.42444 Hz/cm
F1P2MCM  13.53694 ppm/cm
F1HZCM   2042.76135 Hz/cm
    
```

S82. Expanded HMBC (600 MHz) spectrum of halicylindramide H (3) in DMSO-d6



5-1/HMBC

```

Current Data Parameters
NAME      ju104-snu-ch)
EXPNO    16
PROCNO    1

F2 - Acquisition Parameters
Date_    20050705
Time     22.21
INSTRUM  spect
PROBHD   5 mm CPTX1 1H/
PULPROG  hmcgplndqf
TD       1024
SOLVENT  DMSO
NS       16
DS       4
SWH      6613.757 Hz
FIDRES   6.458747 Hz
AQ       0.0775400 sec
RG       18390.4
DM       75.600 usec
DE       6.00 usec
TE       298.0 K
CNS12    145.0000000
CNS113   10.0000000
d0       0.00000300 sec
D1       1.50000000 sec
d2       0.00344628 sec
d5       0.05000000 sec
D16      0.0002000 sec
IN0      0.00001380 sec
MCREST   0.0000000 sec
MCMRK    1.5000000 sec

----- CHANNEL f1 -----
NUC1     1H
P1       10.50 usec
p2       21.00 usec
PL1      -5.00 dB
SFO1     600.1330005 MHz

----- CHANNEL f2 -----
NUC2     13C
P3       15.00 usec
PL2      -6.00 dB
SFO2     150.9194083 MHz

----- GRADIENT CHANNEL -----
GPNAM1   SINE.100
GPNAM2   SINE.100
GPNAM3   SINE.100
GPX1     0.00 %
GPX2     0.00 %
GPX3     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPY3     0.00 %
GPZ1     50.00 %
GPZ2     30.00 %
GPZ3     40.10 %
P16      1000.00 usec

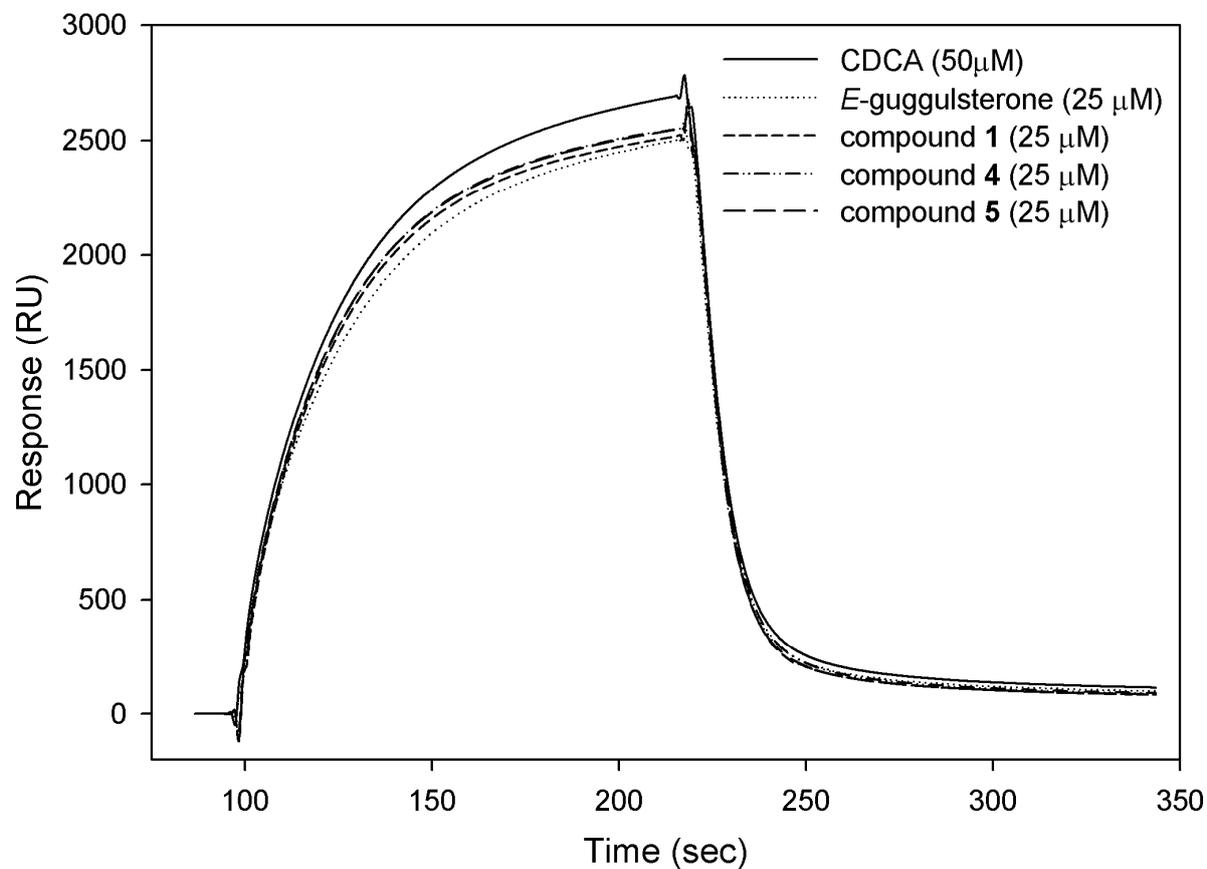
F1 - Acquisition parameters
NDO      2
TD       256
SFO1     150.9194 MHz
FIDRES   141.530792 Hz
SW       240.074 ppm
FhMODE   GF

F2 - Processing parameters
SI       1024
SF       600.1300071 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
MC2      GF
SF       150.9027181 MHz
WDW      SINE
SSB      0
LB       0.00 Hz
GB       0

2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2PLO    8.561 ppm
F2LD     5137.82 Hz
F2PHI    7.011 ppm
F2HI     4207.76 Hz
F1PLO    176.959 ppm
F1LO     26703.53 Hz
F1PHI    167.111 ppm
F1HI     25217.46 Hz
F2PPMCM  0.10332 ppm/cm
F2HZCM   62.00398 Hz/cm
F1PPMCM  0.65653 ppm/cm
F1HZCM   99.07151 Hz/cm
    
```

**S83.** Indirect binding of **1**, **4** and **5** to the LBD of hFXR on BIAcore experiments



**S84.** Photograph of the animal specimen and taxonomic description



Porifera Demospongiae Haplosclerida Petrosina Petrosiidae

ID: Petrosia (Petrosia) 4921

Growth Form: Encrusting or lobate plate 2-5 mm thick

Colour: underwater pinky/brown grey with white rings around the oscules. Maroon on deck. Tints ethanol green

Oscules: on top of short conical fistules, 2 mm diameter.

Texture: smooth, compressible and brittle

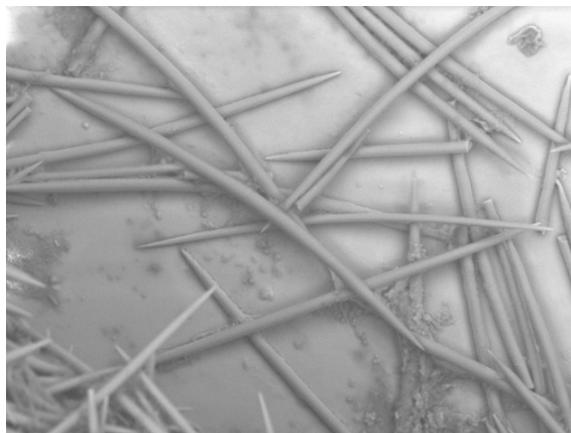
Surface Features Fine microscopic honeycomb pattern of ostia

Ectosome: Tangential reticulation of oxeas forming the surface mesh, with rarely protruding oxeas

Choanosome: unispicular Petrosid isotropic reticulation forming variable circular meshes, smaller at the surface and increasing in size deeper within the choanosome. No fibres. Abundant collagen pigmented red. Occasional paucispicular tracts leading to the ectosome.

Megascleres: One dominant size oxea, slightly curved with sharp tips. (216-254 x 4.8-7.5  $\mu\text{m}$ ), plus rare thin oxeas 196 x 1.63  $\mu\text{m}$ .

Microscleres; Nil



TM-1000\_2821 2015/12/14 12:17 L D5.8 x800 100  $\mu\text{m}$

**S85.** Specific rotation values,  $^1\text{H}$  NMR (600 MHz) data and LR-ESI-MS data of four synthetic Dioias

**D-Dioia-1:**  $[\alpha]_{\text{D}}^{25} + 20$  (*c* 0.1,  $\text{H}_2\text{O}$ ); ECD (0.5 mg/mL,  $\text{CH}_3\text{CN}$ ),  $\lambda_{\text{max}}$  ( $\Delta\epsilon$ ) 318 (-0.24), 265 (21.49), and 239 (-50.77) nm;  $^1\text{H}$  NMR  $\delta_{\text{H}}$  ( $\text{DMSO}-d_6$ ): 10.31(1H, brs), 7.75 (2H, brs), 7.24 (1H, d,  $J = 7.6$  Hz), 7.21 (1H, t,  $J = 7.7$  Hz), 6.97 (1H, t,  $J = 7.3$  Hz), 6.82 (1H, d,  $J = 7.6$  Hz), 4.26 (1H, dd,  $J = 9.5$  Hz, 2.1Hz), 3.33 (1H, brs) 2.31 (1H, dd,  $J = 14.5$  Hz, 9.7 Hz), 1.67 (1H, dd,  $J = 14.5$  Hz, 2.4 Hz), LR-ESI-MS  $m/z$   $[\text{M}+\text{H}]^+$  237.

**D-Dioia-2:**  $[\alpha]_{\text{D}}^{25} - 39$  (*c* 0.1,  $\text{H}_2\text{O}$ ); ECD (0.5 mg/mL,  $\text{CH}_3\text{CN}$ ),  $\lambda_{\text{max}}$  ( $\Delta\epsilon$ ) 315 (-0.42), 263 (20.07), and 240 (52.64) nm;  $^1\text{H}$  NMR  $\delta_{\text{H}}$  ( $\text{DMSO}-d_6$ ): 10.31(1H, brs), 7.91 (2H, brs), 7.37 (1H, d,  $J = 7.1$  Hz), 7.22 (1H, t,  $J = 7.7$  Hz), 6.99 (1H, t,  $J = 7.4$  Hz), 6.84 (1H, d,  $J = 7.6$  Hz), 3.81 (1H, dd,  $J = 9.4$  Hz, 3.9 Hz), 2.23 (1H, dd,  $J = 14.3$  Hz, 9.5 Hz), 1.76 (1H, dd,  $J = 14.3$  Hz, 3.8 Hz), LR-ESI-MS  $m/z$   $[\text{M}+\text{H}]^+$  237.

**L-Dioia-1:**  $[\alpha]_{\text{D}}^{25} - 20$  (*c* 0.1,  $\text{H}_2\text{O}$ ); ECD (0.5 mg/mL,  $\text{CH}_3\text{CN}$ ),  $\lambda_{\text{max}}$  ( $\Delta\epsilon$ ) 318 (0.57), 266 (-20.53), and 238 (51.54) nm;  $^1\text{H}$  NMR  $\delta_{\text{H}}$  ( $\text{DMSO}-d_6$ ): 10.31(1H, brs), 7.75 (2H, brs), 7.24 (1H, d,  $J = 7.6$  Hz), 7.21 (1H, t,  $J = 7.7$  Hz), 6.97 (1H, t,  $J = 7.3$  Hz), 6.82 (1H, d,  $J = 7.6$  Hz), 4.26 (1H, dd,  $J = 9.5$  Hz, 2.1Hz), 3.33 (1H, brs) 2.31 (1H, dd,  $J = 14.5$  Hz, 9.7 Hz), 1.67 (1H, dd,  $J = 14.5$  Hz, 2.4 Hz), LR-ESI-MS  $m/z$   $[\text{M}+\text{H}]^+$  237.

**L-Dioia-2:**  $[\alpha]_{\text{D}}^{25} + 39$  (*c* 0.1,  $\text{H}_2\text{O}$ ); ECD (0.5 mg/mL,  $\text{CH}_3\text{CN}$ ),  $\lambda_{\text{max}}$  ( $\Delta\epsilon$ ) 315 (-0.42), 263 (20.07), and 240 (52.64) nm;  $^1\text{H}$  NMR  $\delta_{\text{H}}$  ( $\text{DMSO}-d_6$ ): 10.31(1H, brs), 7.91 (2H, brs), 7.37 (1H, d,  $J = 7.1$  Hz), 7.22 (1H, t,  $J = 7.7$  Hz), 6.99 (1H, t,  $J = 7.4$  Hz), 6.84 (1H, d,  $J = 7.6$  Hz), 3.81 (1H, dd,  $J = 9.4$  Hz, 3.9 Hz), 2.23 (1H, dd,  $J = 14.3$  Hz, 9.5 Hz), 1.76 (1H, dd,  $J = 14.3$  Hz, 3.8 Hz), LR-ESI-MS  $m/z$   $[\text{M}+\text{H}]^+$  237.