SUPPORTING INFORMATION FOR

The Basis of a Humeomics Science: Chemical Fractionation and Molecular Characterization of Humic Biosuprastructures

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Table S-1. Amount ($\mu g.g^{-1}$ of original HA weight) and standard deviation (in parentheses) of compounds detected in RES0 by HPSEC-ESI-MS. Peak numbers (PNo) refer to labelling in Figure 2.

Even-numbered alkanoic acids	PNo		Unsaturated Hydroxyacids	PNo	
Decanoic acid ($C_{10}H_{20}O_2$)	C6	109 (±29.3)	Hydroxyhexenoic acid ($C_6H_{10}O_3$)	C1	98.8 (±30.1)
Dodecanoic acid ($C_{12}H_{24}O_2$)	C7	128 (±34.5)	Hydroxyeptenoic acid ($C_7H_{12}O_3$)	C2	39.7 (±8.58)
Tetradecanoic acid ($C_{14}H_{28}O_2$)	C9	491 (±23.1)	Hydroxytetradecenoic acid ($C_{14}H_{26}O_3$)	C11	40.3 (±21.5)
Hexadecanoic acid ($C_{16}H_{32}O_2$)	C14	2045 (±74.9)	Hydroxypentadecenoic acid ($C_{15}H_{28}O_3$)	C15	10.0 (±6.01)
Octadecanoic acid ($C_{18}H_{36}O_2$)	C22	281 (±20.6)	Hydroxyhexadecenoic acid ($C_{16}H_{30}O_3$)	C19	207 (±122)
Eicosanoic acid ($C_{20}H_{40}O_2$)	C30	50.6 (±6.64)	Hydroxyoctadecenoic acid ($C_{18}H_{34}O_3$)	C25	425 (±285)
Docosanoic acid ($C_{22}H_{44}O_2$)	C40	126 (±5.69)	Hydroxynonadecenoic acid ($C_{19}H_{36}O_3$)	C31	21.5 (±3.11)
Tetracosanoic acid ($C_{24}H_{48}O_2$)	C43	84.9 (±10.1)	Hydroxyeicosenoic acid ($C_{19}H_{38}O_3$)	C35	5.71 (±4.64)
Triacontanoic acid ($C_{30}H_{60}O_2$)	C45	75.6 (±16.2)	Total		848 (±313)
Total		3391 (±83.6)	Hydroxydioic acids		
Odd-numbered alkanoic acids			Hydroxyhexadecanedioic acid ($C_{16}H_{30}O_5$)	B7	71.2 (±9.77)
Pentadecanoic acid ($C_{15}H_{30}O_2$)	C10	784 (±26.4)	Hydroxyoctadecanedioic acid ($C_{18}H_{34}O_5$)	B9	85.4 (±30.3)
Heptadecanoic acid ($C_{17}H_{34}O_2$)	C18	480 (±10.5)	Hydroxyeicosanedioic acid ($C_{20}H_{38}O_5$)	B11	53.8 (±6.57)
Tricosanoic acid ($C_{23}H_{46}O_2$)	C41	28.8 (±3.68)	Hydroxydocosanedioic acid ($C_{22}H_{42}O_5$)	B13	222 (±16.8)
Total		1293 (±28.4)	Hydroxytetracosanedioic acid ($C_{24}H_{46}O_5$)	B15	25.3 (±3.08)
Hydroxyacids			Total		458 (±36.7)
Hydroxytetradecanoic acid ($C_{14}H_{28}O_3$)	C12	81.4 (±6.02)	Diacids		
Hydroxypentadecanoic acid ($C_{15}H_{30}O_3$)	C16	78.8 (±11.4)	Butanedioic acid ($C_4H_6O_4$)	B1	134 (±21.3)
Hydroxyhexadecanoic acid ($C_{16}H_{32}O_3$)	C20	389 (±41.0)	Pentanedioic acid ($C_5H_8O_4$)	B2	43.8 (±10.3)
Hydroxyheptadecanoic acid ($C_{17}H_{34}O_3$)	C23	79.5 (±26.4)	Nonanedioic acid ($C_9H_{16}O_4$)	B3	85.1 (±36.9)
Hydroxyoctadecanoic acid ($C_{18}H_{36}O_3$)	C26	65.0 (±10.2)	Tetradecanedioic acid ($C_{14}H_{26}O_4$)	B4	18.8 (±4.67)
Dihydroxyoctadecanoic acid ($C_{18}H_{36}O_4$)	C33	92.2 (±18.4)	Hexadecanedioic acid ($C_{16}H_{30}O_4$)	B5	1209 (±52.2)
Trihydroxyoctadecanoic acid ($C_{18}H_{36}O_5$)	C37	252 (±17.7)	Heptadecanedioic acid ($C_{17}H_{32}O_4$)	B6	74.3 (±25.6)
Hydroxynonadecanoic acid ($C_{19}H_{38}O_3$)	C32	29.8 (±4.35)	Octadecanedioic acid ($C_{18}H_{34}O_4$)	B8	363 (±75.8)
Hydroxyeicosanoic acid ($C_{20}H_{40}O_3$)	C36	15.6 (±8.81)	Eicosanedioic acid ($C_{20}H_{38}O_4$)	B10	237 (±27.0)
Hydroxydocosanoic acid ($C_{22}H_{44}O_3$)	C42	133 (±4.72)	Docosanedioic acid ($C_{22}H_{42}O_4$)	B12	337 (±21.3)
Hydroxytetracosanoic acid ($C_{24}H_{48}O_3$)	C44	236 (±7.68)	Tetracosanedioic acid ($C_{24}H_{46}O_4$)	B14	178 (±13.0)
Total		1452 (±59.0)	Total		2680 (±111)
Unsaturated acids			Other acids		
Tetradecenoic acid ($C_{14}H_{26}O_2$)	C8	93.1 (±39.1)	$C_{15}H_{22}O_4$	C17	18.2 (±8.00)
Hexadecenoic acid ($C_{16}H_{30}O_2$)	C13	976 (±53.4)	$C_{16}H_{35}O_4N_3$	C38	29.0 (±5.57)
Octadecenoic acid ($C_{18}H_{34}O_2$)	C21	1393 (±43.8)	$C_{17}H_{26}O_4$	C24	6.84 (±3.00)
Total		2462 (±69.2)	$C_{17}H_{26}O_5$	C28	8.32 (±2.90)
Cyclic acids			$C_{18}H_{26}O_4$	C27	439 (±322)
$C_6H_6O_4$	C4	20917 (±1020)	$C_{20}H_{24}O_3$	C29	31.4 (±9.41)
$C_6H_4O_5$	C5	238484 (±25812)	$C_{21}H_{26}O_3$	C34	44.2 (±11.2)
Benzoic acid ($C_7H_6O_2$)	C3	20448 (±2654)	$C_{22}H_{28}O_3$	C39	25.5 (±3.87)
$C_7H_6O_8$	A1	6050 (±502)	$C_{30}H_{48}O_3$	C46	104 (±12.7)
Total		285899 (±27885)	$C_{30}H_{48}O_4$	C47	109 (±9.08)
			Total		815 (±323)

Table S-2 Amount ($\mu g.g^{-1}$ of original HA weight) and standard deviation (in parentheses) of compounds detected in ORG1 by GC-MS. Peak numbers (PNo) refer to labelling in Figure 4.

Alkanoic acids	PNo		Di-,tri-hydroxyacids	PNo	
Dodecanoic acid	A2	89.5 (±12.9)	2,16-diOH-hexadecanoic acid	A24	933 (±132.4)
Tetradecanoic acid	A3	269 (±12.9)	2,9-diOH-octadecanoic acid	A30	1300 (±183.9)
iso-Pentadecanoic acid	A5	291 (±16.8)	2,18-diOH-octadecanoic acid	A29	1694 (±127.1)
anteiso-Pentadecanoic acid	A6	241 (±11.2)	9, 10, 18-triOH-octadecanoic acid	A36	527 (±74.4)
Pentadecanoic acid	A7	95.1 (±22.4)	Total		4454 (±67.2)
iso-Hexadecanoic acid	A10	313 (±28.0)	ω-hydroxyacids		
Hexadecenoic acid	A9	134 (±39.2)	16-OH-hexadecanoic acid	A18	2385 (±171.8)
Hexadecanoic acid	A11	481 (±36.9)	18-OH-octadecanoic acid	A25	227 (±156.5)
iso-Eptadecanoic acid	NL	274 (±13.4)	20-OH-eicosanoic acid	A31	1968 (±162)
anteiso-Eptadecanoic acid	NL	285 (±89.5)	22-OH-docosanoic acid	A35	2403 (±172.2)
Eptadecanoic acid	NL	190 (±12.9)	24-OH-tetracosanoic acid	A40	2102 (±162)
iso-Octadecanoic acid	A16	112 (±39.2)	26-OH-hexacosanoic acid	A45	1912 (±158.4)
anteiso-Octadecanoic acid	A15	83.9 (±11.2)	Total		10997 (±714)
Octadecadienoic acid	A21	67.1 (±8.95)	α, ω-diacids		
Octadecenoic acid	A14	414 (±83.9)	Nonadioic acid	A1	273 (±38.4)
Octadecanoic acid	A17,22	4695 (±50.4)	Hexadecandioic acid	A20	3507 (±116.2)
Nonadecanoic acid	NL	224 (±11.2)	Octadecandioic acid	A27	243 (±64.2)
Eicosanoic acid	A23	89.5 (±12.9)	Eicosandioic acid	A33	1845 (±116)
Henicosanoic acid	NL	134 (±50.4)	Heneicosandioic acid	NL	1405 (±116)
Docosanoic acid	A28	1371 (±252)	Docosandioic acid	A38	2608 (±113.7)
Tricosanoic acid	NL	442 (±134)	Tricosandioic acid	NL	1410 (±99.6)
Tetracosanoic acid	A34	1371 (±83.9)	Tetracosandioic acid	A43	177 (±98)
Pentacosanoc acid	NL	291 (±112)	Pentacosandioic acid	NL	344 (±48.5)
Hexacosanoic acid	A39	795 (±218)	Hexacosandioic acid	A47	1471 (±10.1)
Eptacosanoic acid	NL	241 (±39.2)	Total		13282 (±461)
Octacosanoic acid	A44	828 (±196)	Alkanols		
Nonacosanoic acid	NL	224 (±11.2)	Tetradecanol	A4	74.2 (±10.7)
Triacontanoic acid	A48	61.6 (±11.2)	Pentadecanol	A8	4.1 (±1.8)
Hentriacontanoic acid	NL	39.2 (±11.2)	Hexadecanol	A13	28.9 (±12.4)
Dotriacontanoic acid	A49	33.6 (±50.4)	Octadecanol	A19	635 (±223)
Total		14179 (±119)	Eicosanol	A26	86.6 (±33.0)
α,β-hydroxyacids			Docosanol	A32	528 (±297)
3-OH tetradecanoic acid	A12	1312 (±185.5)	Tetracosanol	A37	115 (±57.7)
2-OH-tetracosanoic acid	A41	1666 (±139.6)	Hexacosanol	A42	33.0 (±4.5)
Total		2978 (±577)	Octacosanol	A46	86.6 (±12.4)
			Total		1591 (±97.0)

NL: Not labelled in Figure 4

Table S-3 Amount ($\mu g.g^{-1}$ of original HA weight) and standard deviation (in parentheses) of compounds detected in ORG2 by GC-MS. Peak numbers (PNo) refer to labelling in Figure 4

Alkanoic acids	PNo	
Nonanoic acid	C1	5.10 (±0.5)
Dodecanoic acid	C7	10.2 (±1.2)
Tetradecanoic acid	C8	96.9 (±25.5)
iso-Pentadecanoic acid	C10	30.6 (±15.3)
anteiso-Pentadecanoic acid	C11	5.10 (±2.0)
Pentadecanoic acid	C12	35.7 (±5.1)
iso-Hexadecanoic acid	C18	35.7 (±5.10)
Hexadecanoic acid	C19	719 (±42.8)
iso-Eptadecanoic acid	NL	76.5 (±45.9)
anteiso-Eptadecanoic acid	NL	25.5 (±10.2)
Octadecadienoic acid	C23	96.9 (±15.3)
Octadecenoic acid	C25	133 (±66.3)
Octadecanoic acid	C26	56.1 (±38.8)
Docosanoic acid	C38	209 (±30.6)
Tricosanoic acid	NL	86.7 (±51.0)
Tetracosanoic acid	C47	357 (±189)
Pentacosanoc acid	NL	10.2 (±7.65)
Hexacosanoic acid	NL	86.7 (±35.7)
Eptacosanoic acid	NL	5.10 (±0.50)
Octacosanoic acid	NL	81.6 (±45.9)
Nonacosanoic acid	NL	40.8 (±10.2)
Total		2203 (±123)

Alkanols		
Dodecanol	B1	44.2 (±20.1)
Tetradecanol	B3	88.4 (±48.2)
Pentadecanol	B4	498 (±104)
Hexadecanol	B5	141 (±56.2)
Octadecanol	B6	309 (±80.3)
Eicosanol	B7	64.3 (±6.3)
Docosanol	B8	586 (±394)
Tetracosanol	B9	24.1 (±3.2)
Hexacosanol	B10	120 (±68.3)
Eptacosanol	B11	4.02 (±0.20)
Octacosanol	B12	84.3 (±40.2)
Total		1964 (±107)

Di-,tri-hydroxyacids	PNo	
2,9-diOH-hexadecanoic acid	C36	206 (±29.7)
2,16-diOH-hexadecanoic acid	C37	1006 (±68.4)
epoxy-hexadecanoic acid	C22	1177 (±92.5)
2,9-diOH-octadecanoic acid	C39	3138 (±362)
2,18-diOH-octadecanoic acid	C40	347 (±150.9)
epoxy-octadecanoic acid	C27	1368 (±121.2)
2,9-diOH-octadecandioic acid	C41	689 (±61.3)
9, 10, 18-triOH-octadecanoic acid	C48	2881 (±147.8)
Total		10811 (±557)

ω-hydroxyacids		
9-OH-nonanoic acid	C5	1039 (±88.3)
14-OH-tetradecanoic acid	C21	1028 (±87.7)
16-OH-hexadecenoic acid	C28	1481 (±141.9)
16-OH-hexadecanoic acid	C30	2017 (±117)
18-OH-octadecenoic acid	C33	1531 (±346)
18-OH-octadecanoic acid	C35	1039 (±90.5)
20-OH-eicosanoic acid	C43	134 (±89.4)
22-OH-docosanoic acid	C45	156 (±90.5)
24-OH-tetracosanoic acid	C52	162 (±86.6)
26-OH-hexacosanoic acid	C55	196 (±27.9)
Total		8784 (±472)

α,β-hydroxyacids		
3-OH-dodecanoic acid	C9	148 (±105)
3-OH-tridecanoic acid	C16	1112 (±98.9)
3-OH tetradecanoic acid	C20	198 (±28.4)
2-OH-pentadecanoic acid	NL	117 (±102.6)
3-OH-hexadecanoic acid	C29	1341 (±119.2)
2-OH-eptadecanoic acid	C31	1217 (±97.6)
3-OH-eptadecanoic acid	C32	117 (±98.2)
3-OH-octadecanoic acid	C34	148 (±106.9)
3-OH-eicosanoic acid	C42	1168 (±103.2)
2-OH-docosanoic acid	C44	1205 (±102.6)
2-OH-tricosanoic acid	C50	1131 (±98.9)
2-OH-tetracosanoic acid	C51	1013 (±143.3)
2-OH-pentacosanoic acid	C53	1118 (±98.2)
2-OH-hexacosanoic acid	C54	111 (±99.5)
Total		10145 (±623)

α, ω-diacids	PNo	
Octadioic acid	C2	103 (±14.7)
Nonadioic acid	C4	137 (±19.6)
Decandioic acid	NL	9.81 (±1.37)
Dodecandioic acid	NL	78.5 (±46.1)
Hexadecandioic acid	C24	907 (±56.9)
Docosandioic acid	C46	618 (±47.1)
Tricosandioic acid	C49	564 (±48.1)
Total		2418 (±211)

Aromatic compounds		
3-OH-Benzoic acid	C3	180 (±11.2)
3, 4-diOH-Benzoic acid	C6	256 (±17.6)
4-OH-Cinnamic acid	C13	140 (±10.8)
2-OH-Cinnamic acid	C14	160 (±14.8)
3-OH-Cinnamic acid	C15	152 (±72.1)
3, 4-diOH-Cinnamic acid	C17	148 (±68.1)
Benzald.-3,4-OH	B2	120 (±10.8)
Total		1157 (±78.5)

Steroids		
β-sitosterol	B13	147 (±79.6)
Stigmasterol	B14	35.8 (±5.2)
Total		183 (±23.9)

NL: Not Labelled in Figure 4

Table S-4. Amount ($\mu g.g^{-1}$ of original HA weight) and standard deviation (in parentheses) of compounds detected in ACQ2 by HPSEC-ESI-MS. Peak numbers (PNo) refer to labelling in Figure 5.

Even-numbered alkanoic acids	PNo	
Decanoic acid ($C_{10}H_{20}O_2$)	A6	0.71 (±0.19)
Dodecanoic acid ($C_{12}H_{24}O_2$)	A11	0.98 (±0.44)
Tetradecanoic acid ($C_{14}H_{28}O_2$)	A14	0.98 (±0.63)
Hexadecanoic acid ($C_{16}H_{32}O_2$)	A19	10.30 (±1.28)
Octadecanoic acid ($C_{18}H_{36}O_2$)	A23	0.54 (±0.05)
Total		13.51 (±1.28)
Odd-numbered alkanoic acids		
Pentadecanoic acid ($C_{15}H_{30}O_2$)	A16	7.24 (±0.73)
Unsaturated alkanoic acids		
Pentadecenoic acid ($C_{15}H_{28}O_2$)	A15	1.68 (±0.16)
Hexadecenoic acid ($C_{16}H_{30}O_2$)	A18	16.48 (±1.75)
Eptadecenoic acid ($C_{17}H_{32}O_2$)	A20	1.59 (±0.17)
Octadecenoic acid ($C_{18}H_{34}O_2$)	A22	3.86 (±0.41)
Total		23.61 (±1.81)
Hydroxyunsaturated acids		
Hydroxypentenoic acid ($C_5H_8O_3$)	A1	3.87 (±0.99)
Hydroxyhexenoic acid ($C_6H_{10}O_3$)	A2	3.62 (±0.64)
Hydroxydecenoic acid ($C_{10}H_{18}O_3$)	A7	1.70 (±1.15)
Hydroxyhexadecenoic acid ($C_{16}H_{30}O_3$)	A21	1.24 (±0.48)
Hydroxyoctadecadienoic acid ($C_{18}H_{32}O_3$)	A24	1.99 (±0.22)
Hydroxyoctadecenoic acid ($C_{18}H_{34}O_3$)	A25	2.18 (±0.18)
Total		14.62 (±1.80)

Diacids	PNo	
Pentanedioic acid ($C_5H_8O_4$)	A3	0.29 (±0.03)
Hexanedioic acid ($C_6H_{10}O_4$)	A5	0.68 (±0.07)
Nonanedioic acid ($C_9H_{16}O_4$)	A8	0.31 (±0.01)
Total		1.27 (±0.07)
Cyclic acids		
Benzoic acid ($C_7H_6O_2$)	C1	157 (±18.0)
$C_6H_6O_4$	A4	8.37 (±0.80)
$C_6H_4O_5$	C2	103 (±6.0)
$C_7H_6O_8$	A12	5.61 (±0.37)
$C_9H_8O_3N_2$	A9	19.65 (±0.21)
$C_8H_7O_3N_3$	A10	42.03 (±1.43)
$C_{10}H_{11}O_3N_3$	A13	7.29 (±0.21)
Total		343 (±24.0)
N-containing acids		
$C_7H_{14}O_4N_2$	B1	0.98 (±0.19)
$C_7H_{13}O_6N$	B2	4.27 (±0.52)
$C_{10}H_{18}O_5N_2$	A17	2.45 (±0.36)
$C_{12}H_{21}O_8N$	C3	1.32 (±0.32)
$C_{10}H_{29}O_7N_5$	B4	7.06 (±0.28)
$C_{12}H_{31}O_6N_5$	B5	0.96 (±0.23)
$C_{12}H_{33}O_6N_5$	B6	4.30 (±0.64)
$C_{16}H_{30}O_5N_2$	B3	4.01 (±0.62)
$C_{16}H_{29}O_6N_3$	A26	9.69 (±0.56)
$C_{18}H_{34}O_6N_4$	B7	1.64 (±0.21)
Total		36.68 (±1.35)

Table S-5. Amount ($\mu g.g^{-1}$ of original HA weight) and standard deviation (in parentheses) of compounds detected in ORG3 by GC-MS. Peak numbers (PNo) refer to labelling in Figure 4.

Alkanoic acids	PNo		Di-,tri-hydroxyacids	PNo	
Nonanoic acid	D1	3.93 (±0.9)	2,9-diOH-octadecanoic acid	D24	386 (±86)
Dodecanoic acid	D7	3.93 (±1.9)	2,18-diOH-octadecanoic acid	D25	398 (±59)
Tetradecanoic acid	D8	15.70 (±5.3)	9, 10, 18-triOH-octadecanoic acid	D31	205 (±88)
Pentadecanoic acid	D9	11.78 (±3.6)	Total		988 (±85)
Hexadecenoic acid	D14	7.85 (±1.00)	ω-hydroxyacids		
Hexadecanoic acid	D15	302 (±74.6)	16-OH-hexadecanoic acid	D13	132 (±4.0)
iso-Eptadecanoic acid	NL	3.93 (±1.8)	22-OH-docosanoic acid	D28	75.7 (±3.9)
anteiso-Eptadecanoic acid	NL	11.8 (±2.8)	24-OH-tetracosanoic acid	D34	63.8 (±16)
Octadecadienoic acid	D16	7.85 (±2.4)	Total		271 (±10)
Octadecenoic acid	D18	27.5 (±2.7)	α,β-hydroxyacids		
Octadecanoic acid	D19	141 (±20.4)	2-OH-docosanoic acid	D27	67.8 (±7.9)
Eicosanoic acid	D20	11.8 (±0.8)	2-OH-tricosanoic acid	NL	63.8 (±1.8)
Henicosanoic acid	D21	3.93 (±0.39)	2-OH-tetracosanoic acid	D33	71.8 (±8.7)
Docosanoic acid	D22	11.8 (±4.6)	2-OH-pentacosanoic acid	NL	63.8 (±4.9)
Tricosanoic acid	D26	3.93 (±2.11)	2-OH-hexacosanoic acid	D38	67.8 (±9.9)
Tetracosanoic acid	D29	7.85 (±1.4)	Total		335 (±7.5)
Pentacosanoc acid	NL	3.9 (±1.3)	α, ω-diacids		
Hexacosanoic acid	D32	3.9 (±0.5)	Hexadioic acid	NL	76.3 (±3.6)
Octacosanoic acid	D36	11.8 (±3.0)	Octadioic acid	D2	4.49 (±0.08)
Nonacosanoic acid	NL	3.9 (±0.9)	Nonadioic acid	D5	144 (±4.6)
Triacontanoic acid	D40	23.55 (±6.7)	Hexadecandioic acid	D17	98.7 (±7.7)
Dotriacontanoic acid	NL	7.85 (±0.67)	Total		323 (±5.5)
Total		632 (±55.4)	Aromatic compounds		
Alkanols			3-OH-Benzoic acid	D3	8.0 (±0.1)
Dodecanol	D4	3.96 (±0.2)	3, 4-diOH-Benzoic acid	D6	5.0 (±0.9)
Tetracosanol	D23	7.92 (±0.3)	4-OH-Cinnamic acid	D10	3.0 (±0.4)
Pentacosanol	D30	15.8 (±3.2)	2-OH-Cinnamic acid	D11	5.0 (±0.3)
Hexacosanol	D35	27.7 (±4.0)	3-OH-Cinnamic acid	D12	3.0 (±0.9)
Octacosanol	D37	35.6 (±4.8)	Total		24.0 (±2.8)
Eicosanol	D39	7.92 (±3.96)	Steroids		
Docosanol	NL	182 (±21.8)	β-sitosterol	D41	17.7 (±2.9)
Tricosanol	NL	0.1 (±0.1)	Friedelan-3-one	D42	47.3 (±5.3)
Tetracosanol	D43	4.0 (±1.6)	Total		65 (±4.0)
Hexacosanol	NL	3.9 (±0.7)			
Octacosanol	NL	15.8 (±1.2)			
Total		305 (±5.7)			

NL: Not Labelled in Figure 4.

Table S-6. $T_{1\rho}H$ values (ms) of solid RES0 and RES4 residues by Variable Spin Lock (VSL) experiments.

Spectral region (ppm)	185-150	150-95	95-60	60-0
RES0	2.4	3.4	2.6	4.4
RES4	2.7	5.0	NA	5.3

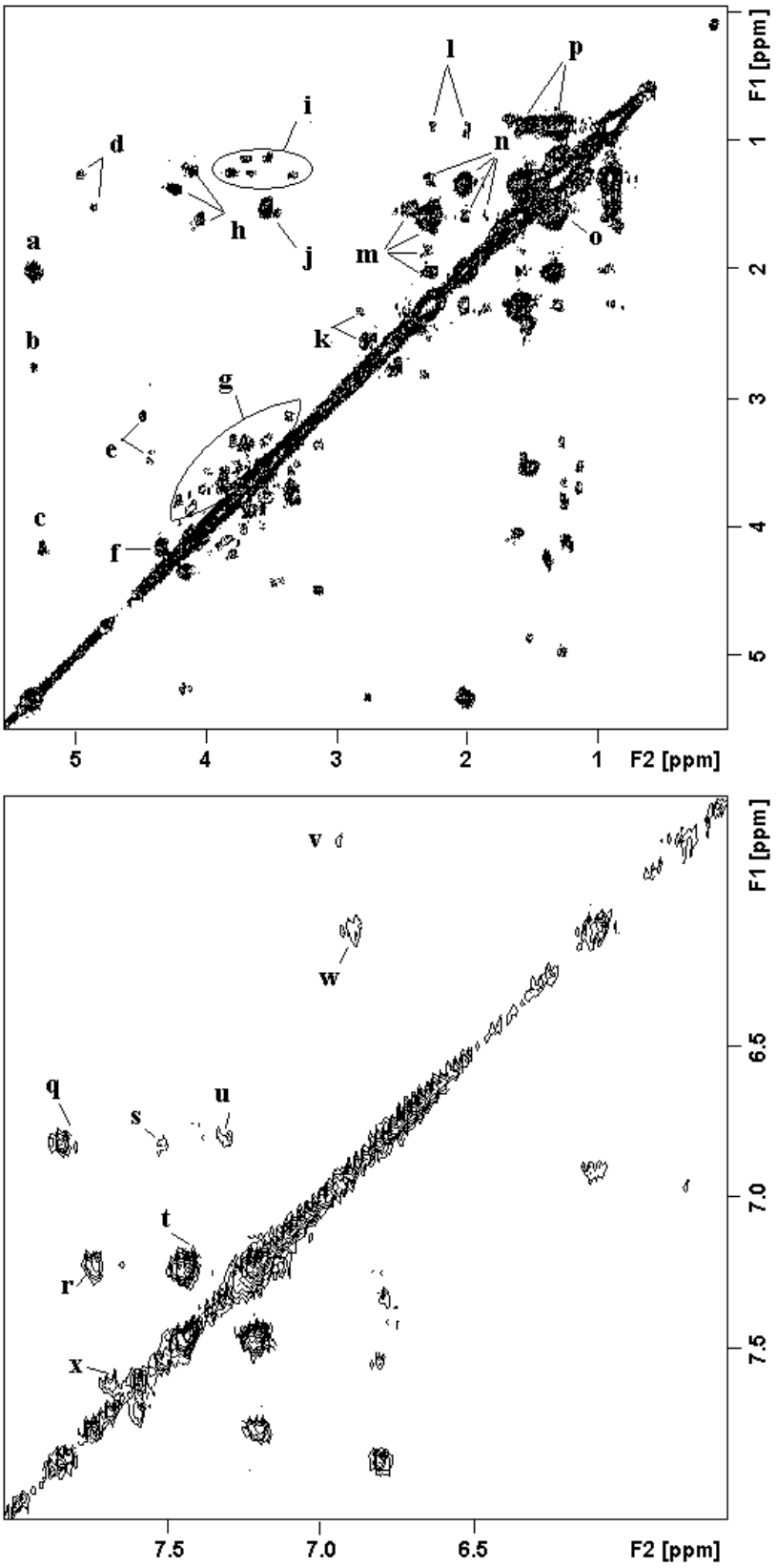
NA: Not Applicable

Table S-7 Amount ($\mu g.g^{-1}$ of original HA weight) and standard deviation (in parentheses) of compounds detected in RES4 by HPSEC-ESI-MS. Peak numbers (PNo) refer to signals in Figure 6.

Even-numbered alkanoic acids	PNo		Cyclic acids	PNo	
Decanoic acid ($C_{10}H_{20}O_2$)	B4	76.6 (±11.3)	Benzoic acid ($C_7H_6O_2$)	B1	4337 (±491)
Dodecanoic acid ($C_{12}H_{24}O_2$)	B5	47.7 (±21.6)	$C_6H_6O_4$	B2	238484 (±25812)
Tetradecanoic acid ($C_{14}H_{28}O_2$)	B7	179 (±39.3)	$C_6H_4O_5$	B3	7888 (±365)
Hexadecanoic acid ($C_{16}H_{32}O_2$)	B10	324 (±118)	$C_7H_6O_8$	A3	9158 (±560)
Octadecanoic acid ($C_{18}H_{36}O_2$)	B13	8.28 (±3.54)	Total		259866 (±25825)
Total		635 (±127)	Other acids		
Odd-numbered alkanoic acids			$C_{15}H_{22}O_4$	A4	38.9 (±14.6)
Pentadecanoic acid ($C_{15}H_{30}O_2$)	B8	191 (±1.09)	$C_{17}H_{26}O_4$	B14	13.3 (±5.33)
Eptadecanoic acid ($C_{17}H_{34}O_2$)	B11	24.9 (±8.74)	$C_{17}H_{26}O_5$	B16	18.9 (±6.97)
Total		216 (±8.81)	$C_{18}H_{34}O_3$	B15	22.5 (±5.57)
Unsaturated acids			$C_{18}H_{34}O_4$	B18	21.7 (±14.6)
Tetradecenoic acid ($C_{14}H_{26}O_2$)	B6	46.5 (±3.59)	$C_{18}H_{36}O_4$	B19	4.23 (±2.03)
Hexadecenoic acid ($C_{16}H_{30}O_2$)	B9	326 (±1.74)	$C_{18}H_{34}O_5$	B21	4.45 (±1.84)
Octadecenoic acid ($C_{18}H_{34}O_2$)	B12	59.0 (±19.1)	$C_{19}H_{30}O_5$	B22	1.58 (±0.40)
Total		431 (±19.5)	$C_{19}H_{30}O_6$	B23	7.90 (±3.66)
Hydroxyunsaturated acids			$C_{20}H_{24}O_3$	B17	9.42 (±3.87)
Ac. Idrossiesenoico ($C_6H_{10}O_3$)	A1	33.0 (±2.59)	$C_{21}H_{26}O_3$	B20	7.89 (±2.00)
Ac. Idrossieptenoico ($C_7H_{12}O_3$)	A2	22.8 (±0.65)			151 (±24.0)
Total		55.8 (±2.67)			

Figure S-1 A-D. Bidimensional enlarged spectra and peak labelling of organosoluble unbound fraction (**ORG1**) in $CDCl_3/CD_3OD$: homocorrelated ^1H-^1H COSY (**A**), and TOCSY (**B**); and heterocorrelated ^1H-^{13}C HSQC (**C**), and HMBC (**D**).

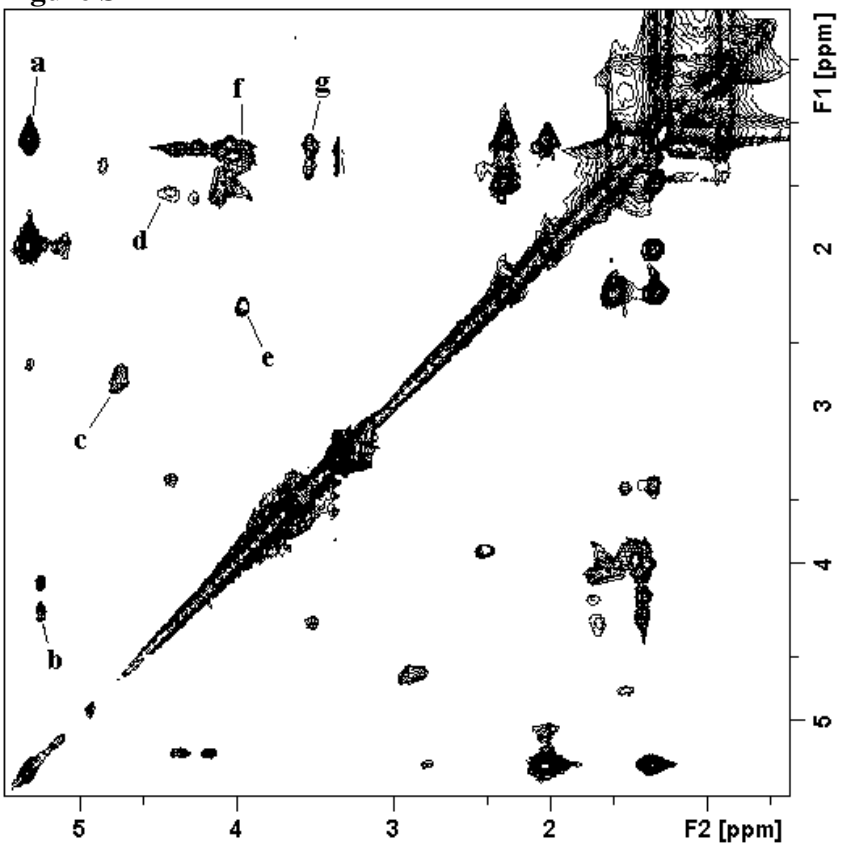
Figure S-1A



ORG1
COSY

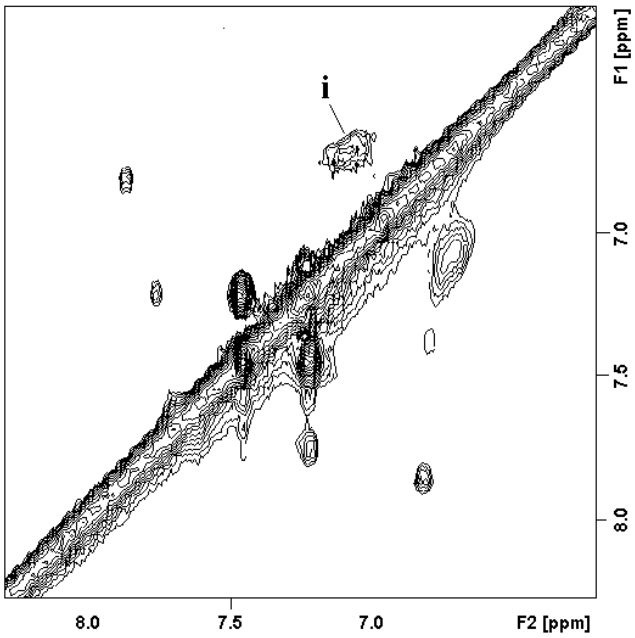
	F2, F1
a	5.30, 2.00
b	5.30, 2.77
c	5.28, 4.17
d	4.96, 1.26
d	4.88, 1.52
e	4.50, 3.15
e	4.44, 3.48
f	4.40, 4.20
g	3.36-4.22, 3.15-3.87
h	4.12, 1.25
h	4.28, 1.40
h	4.05, 1.60
i	3.81, 1.23
i	3.73, 1.14
i	3.67, 1.26
i	3.54, 1.13
i	3.35, 1.26
j	3.54, 1.51
k	2.81, 2.82
k	2.80, 2.55
l	2.27, 0.87
l	2.00, 0.94
m	2.44, 1.52
m	2.30, 1.60
m	2.30, 1.85
m	2.29, 2.02
n	2.28, 1.32
n	2.03, 1.33
n	2.03, 1.61
n	1.87, 1.56
o	1.59, 1.32
p	1.52, 0.87
p	1.31, 0.89
q	7.86, 6.82
r	7.76, 7.23
s	7.54, 6.82
t	7.46, 7.23
u	7.33, 6.79
v	6.95, 5.82
w	6.90, 6.10
x	7.62, 7.71

Figure S-1B



ORG1 TOCSY

	F2, F1
a	5.34, 1.31
b	5.28, 4.33
c	4.77, 2.85
d	4.45, 1.66
e	3.94, 2.38
f	4.07, 1.87
g	3.55, 1.38
h	2.30, 1.29
i	7.09, 6.71



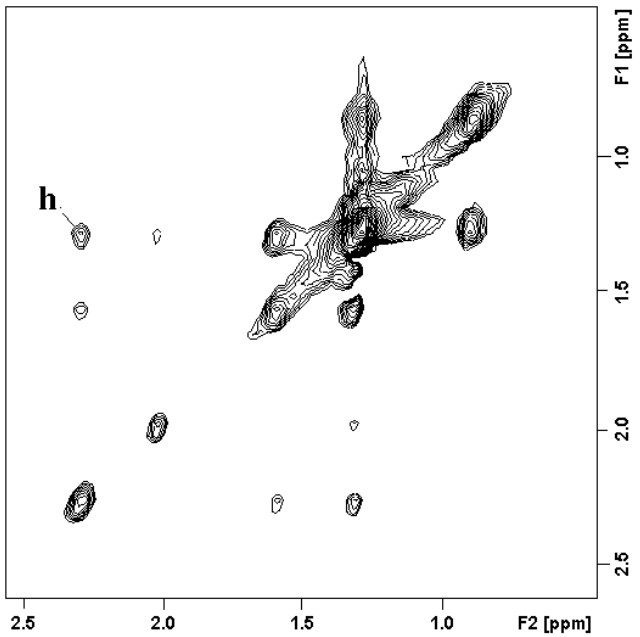
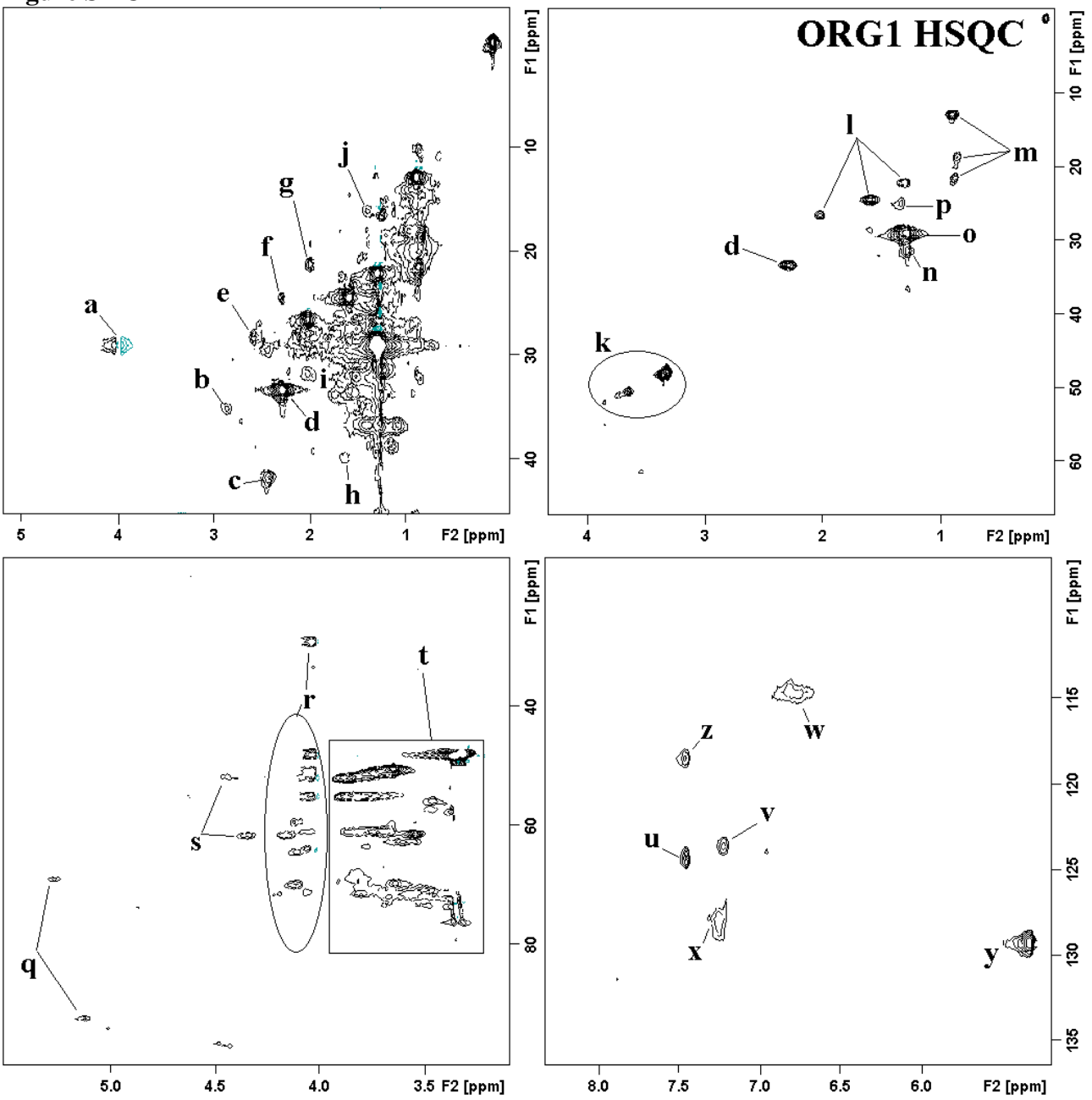
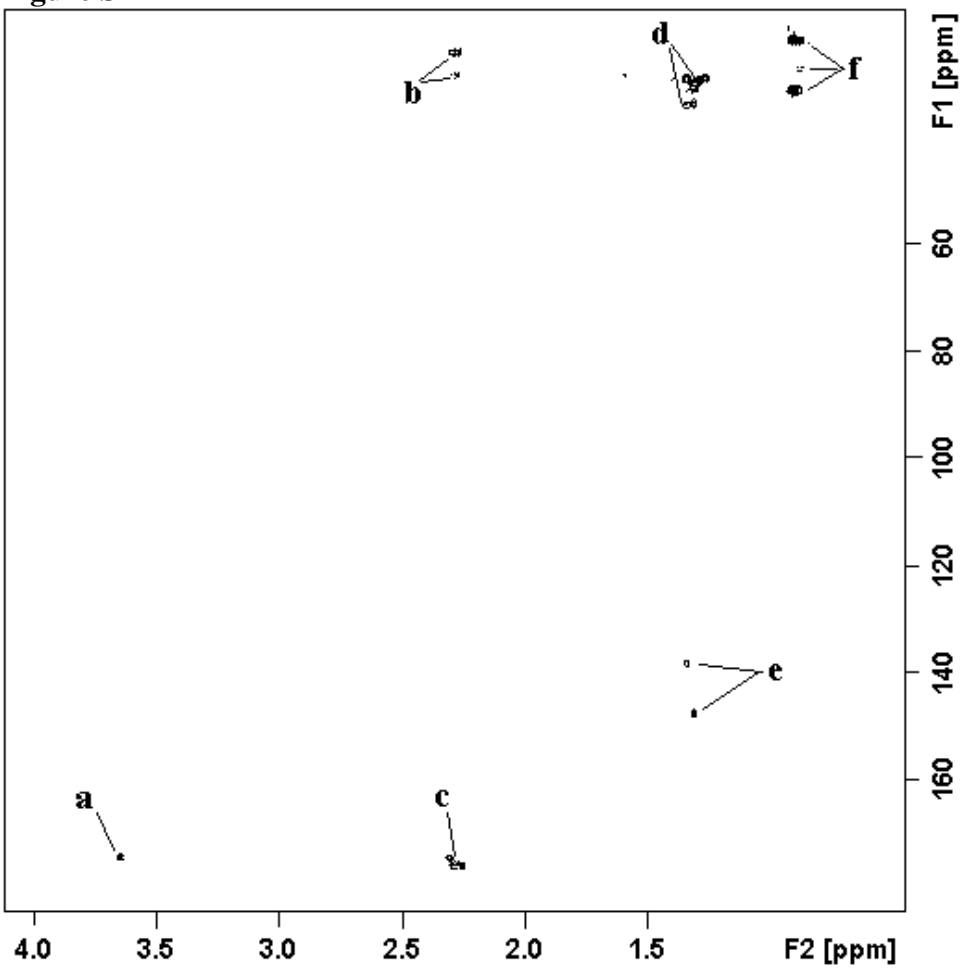


Figure S-1C



	F2, F1		F2, F1
a	4.00, 28.9	m	0.88, 21.5
b	2.87, 35.3	n	1.28, 31.5
c	2.45, 41.9	o	1.30, 29.1
d	2.29, 33.4	p	1.34, 25.1
e	2.59, 28.3	q	5.27, 69.0
f	2.30, 24.6	q	5.13, 92.4
g	2.01, 21.4	r	4.01, 29.1
h	1.98, 39.3	r	4.02-4.13, 48.2, 70.0
i	2.03, 32.0	s	4.44, 51.9
j	1.42, 16.3	s	4.36, 61.8
k	3.86, 52.0	t	3.38-3.88, 48.0-76.4
k	3.67, 50.6	u	7.46, 124.3
k	3.33, 48.0	v	7.23, 123.6
l	2.03, 26.6	w	6.81, 114.6
l	1.60, 24.5	x	7.25, 128.0
l	1.32, 22.3	y	5.35, 129.3
m	0.90, 13.1	z	7.47, 118.4
m	0.87, 18.7		

Figure S-1D

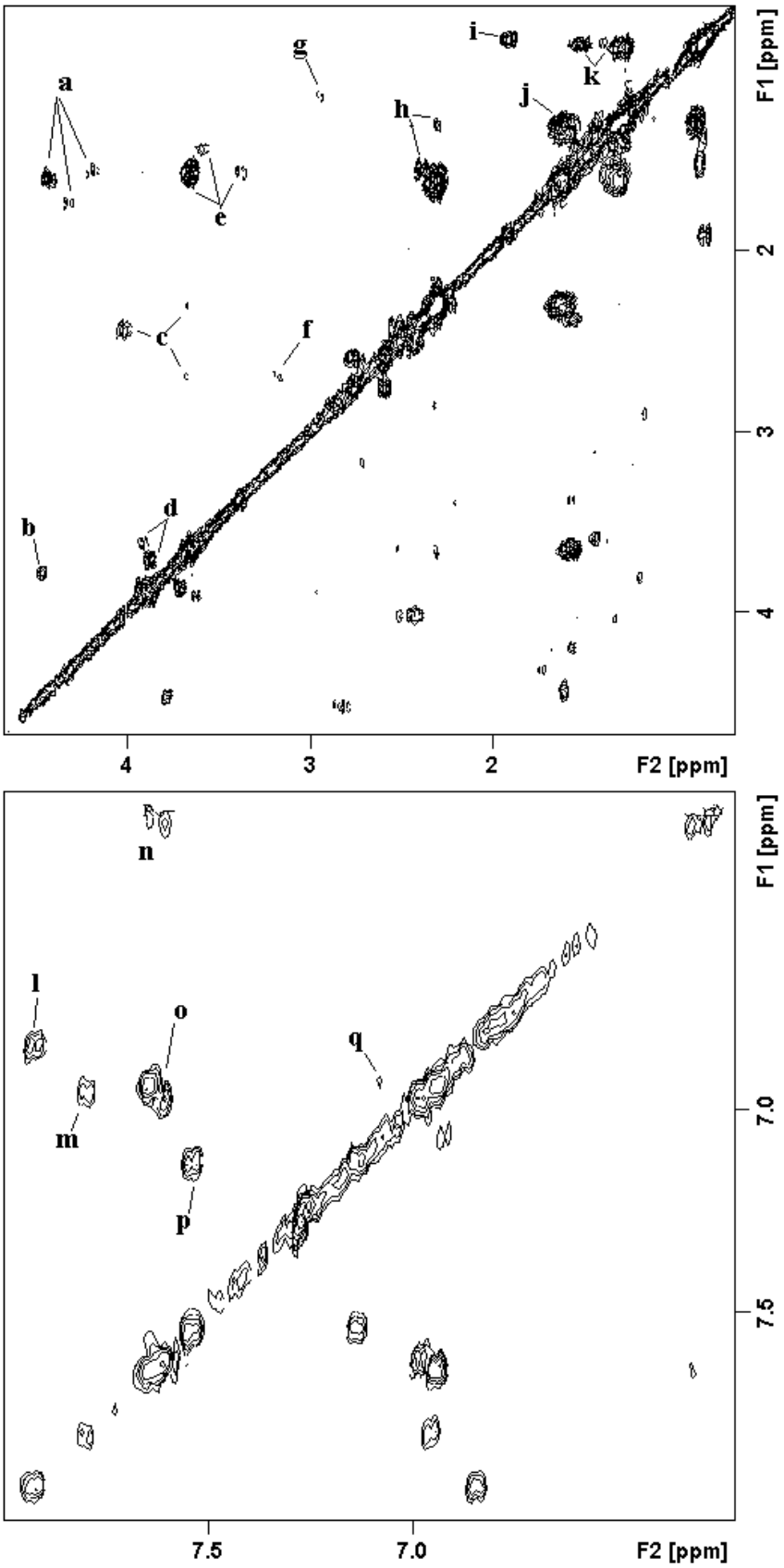


ORG1 HMBC

	F2, F1
a	3.66, 174.8
b	2.30, 24.1
b	2.29, 28.8
c	2.30, 175.6
d	1.32, 30.1
d	1.33, 34.0
e	1.33, 138.2
e	1.31, 147.8
f	0.90, 22.0
f	0.88, 27.2
f	0.91, 31.6

Figure S-2 A-D. Bidimensional enlarged spectra and peak labelling of organosoluble weakly-bound ester fraction (**ORG2**) in DMSO-d6: homocorrelated ^{1}H-^{1}H COSY (**A**), and TOCSY (**B**); and heterocorrelated ^{1}H-^{13}C HSQC (**C**), and HMBC (**D**).

Figure S-2A



	F2, F1
a	4.44, 1.61
a	4.32, 1.73
a	4.19, 1.56
b	4.47, 3.79
c	4.02, 2.44
c	3.68, 2.29
c	3.69, 2.70
d	3.92, 3.62
d	3.89, 3.71
e	3.66, 1.58
e	3.59, 1.44
e	3.38, 1.58
f	3.17, 2.71
g	2.95, 1.14
h	2.31, 1.31
h	2.31, 1.63
i	1.92, 0.83
j	1.62, 1.31
k	1.53, 0.86
k	1.28, 1.88
l	7.92, 6.84
m	7.80, 6.96
n	7.63, 6.29
o	7.64, 6.95
p	7.54, 7.13
q	7.08, 6.93

Figure S-2B

ORG2
TOCSY

	F2, F1		F2, F1
a	2.33, 1.31	l	4.96, 1.39
b	3.66, 1.34	m	4.96, 1.63
c	4.32, 1.35	n	5.07, 1.98
d	4.21, 1.35	o	4.52, 2.35
e	4.04, 1.32	p	4.50, 2.53
f	3.91, 1.32	q	4.53, 2.81
g	3.39, 1.32	r	4.24, 2.82
h	3.15, 1.30	s	7.47, 6.56
i	5.84, 2.19	t	7.17, 6.43
j	5.37, 1.32	u	6.99, 6.81
k	5.36, 2.01	v	7.13, 6.78

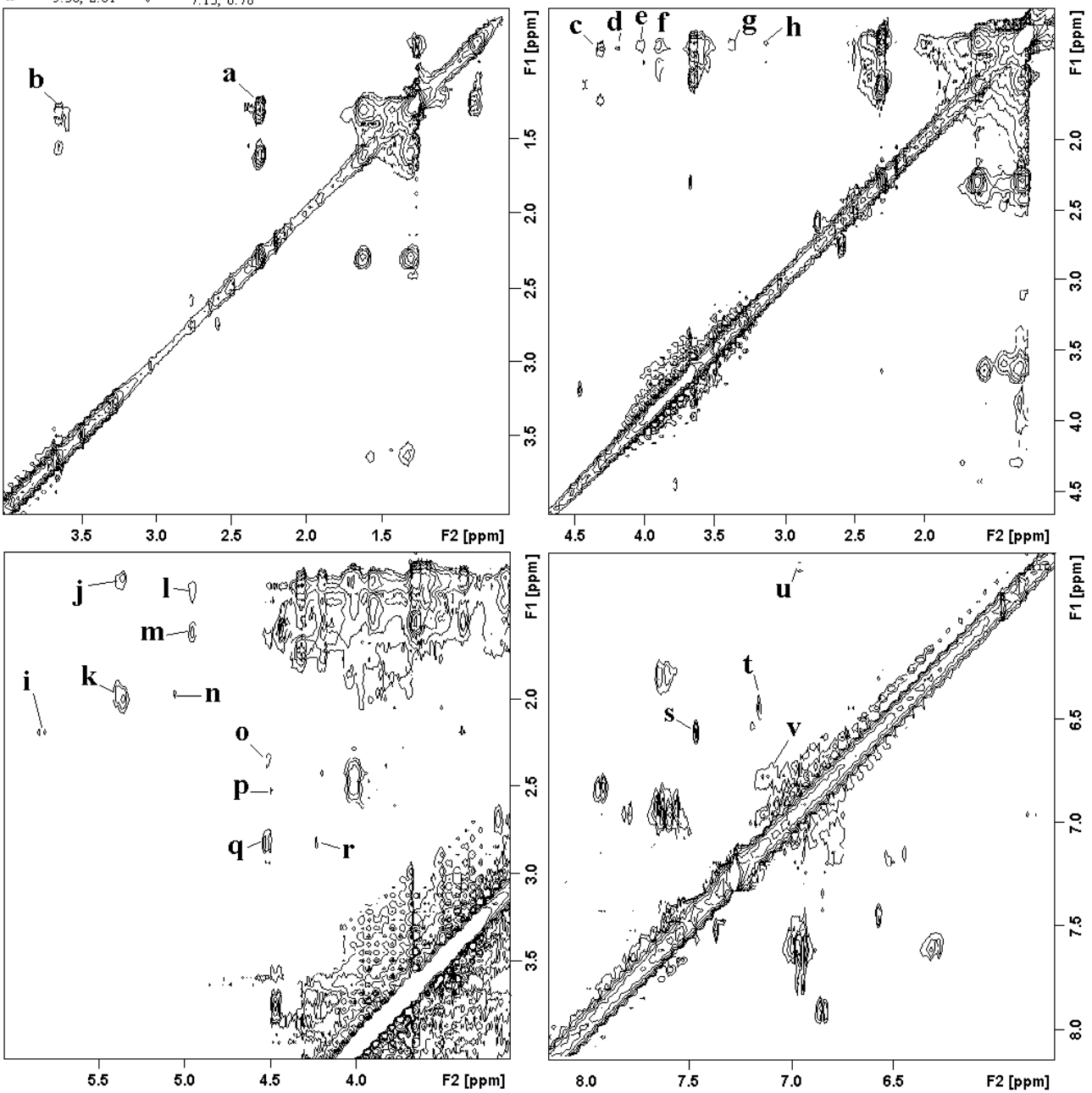
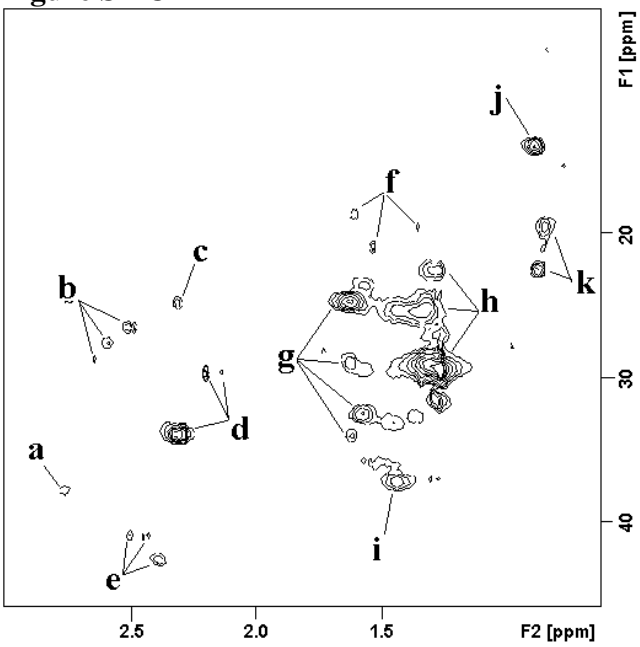


Figure S-2C



	F2, F1		F2, F1
a	2.77, 37.8	l	4.47, 70.6
b	2.65, 28.7	l	4.33, 72.2
b	2.60, 27.6	l	4.20, 70.3
b	2.52, 26.5	m	4.05, 52.4
c	2.32, 24.7	m	3.90, 52.0
d	2.21, 29.8	n	4.07, 56.2
d	2.15, 29.5	n	3.96, 56.3
d	2.32, 33.9	n	3.97, 57.8
e	2.51, 40.9	o	3.91, 81.6
e	2.44, 41.0	p	3.68, 51.3
e	2.40, 42.6	q	3.66, 62.8
f	1.61, 18.7	r	3.69, 70.4
f	1.54, 20.9	r	3.60, 71.8
f	1.36, 19.5	r	3.38, 72.7
g	1.63, 24.7	s	3.50, 50.7
g	1.63, 28.8	s	3.30, 49.9
g	1.59, 32.5	t	3.45, 53.7
g	1.63, 33.9	u	3.33, 56.2
h	1.30, 22.6	u	3.34, 58.3
h	1.35, 25.4	v	7.94, 131.6
h	1.27, 29.4	v	7.65, 124.1
i	1.45, 37.2	v	7.62, 126.6
j	0.89, 13.9	w	7.58, 111.3
k	0.86, 19.6	w	7.48, 107.0
k	0.88, 22.4		

ORG2 HSQC

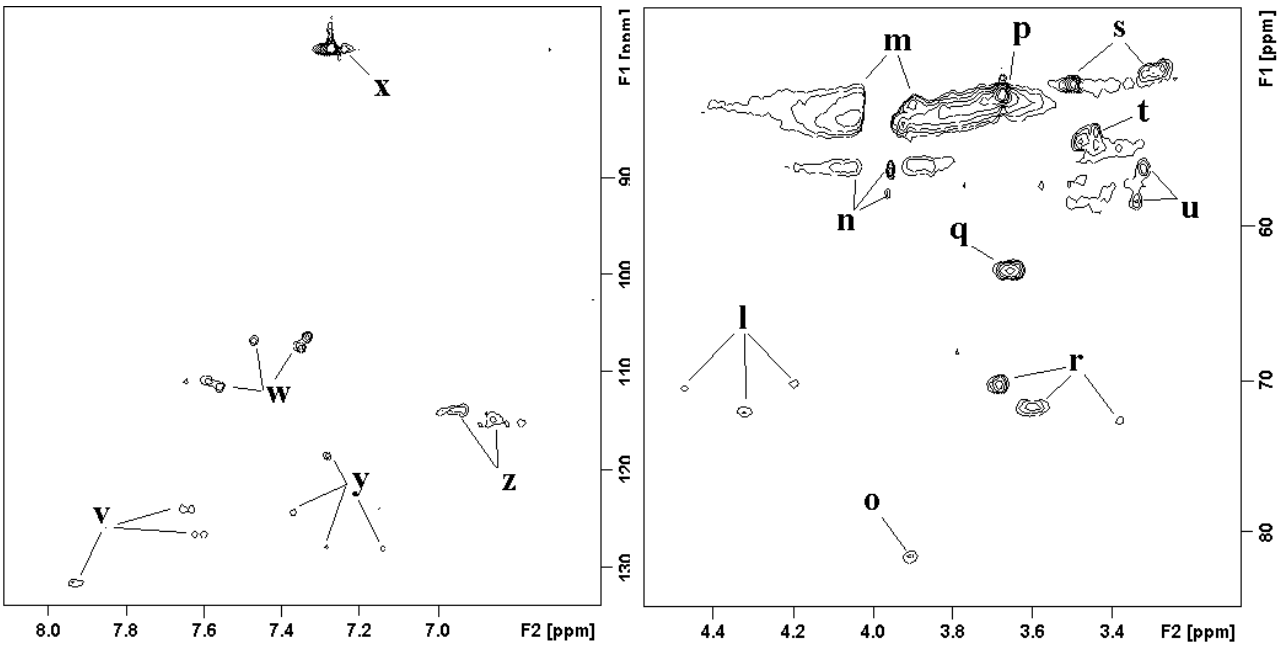
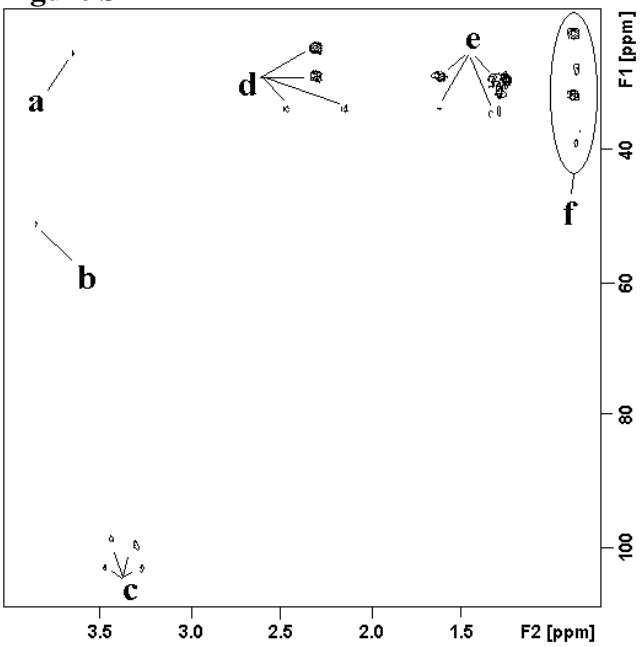


Figure S-2D



ORG2 HMBC

	F2, F1		F2, F1
a	3.65, 25.6	f	0.89, 31.8
b	3.66, 32.6	f	0.88, 39.0
c	3.49, 103.1	g	3.96, 146.6
c	3.44, 98.9	h	3.97, 151.2
c	3.31, 99.7	i	3.94, 157.5
c	3.28, 103.1	j	3.73, 173.3
d	2.48, 33.9	j	3.68, 173.8
d	2.31, 24.7	k	2.31, 174.2
d	2.31, 29.0	l	2.50, 191.5
d	2.16, 34.1	l	2.52, 201.0
e	1.63, 28.6	l	2.20, 206.6
e	1.63, 33.6	m	1.63, 174.3
e	1.26, 29.4	n	1.34, 138.3
e	1.34, 34.7	n	1.30, 146.9
f	0.88, 22.3	o	7.54, 77.1
f	0.87, 27.5	p	7.01, 77.1

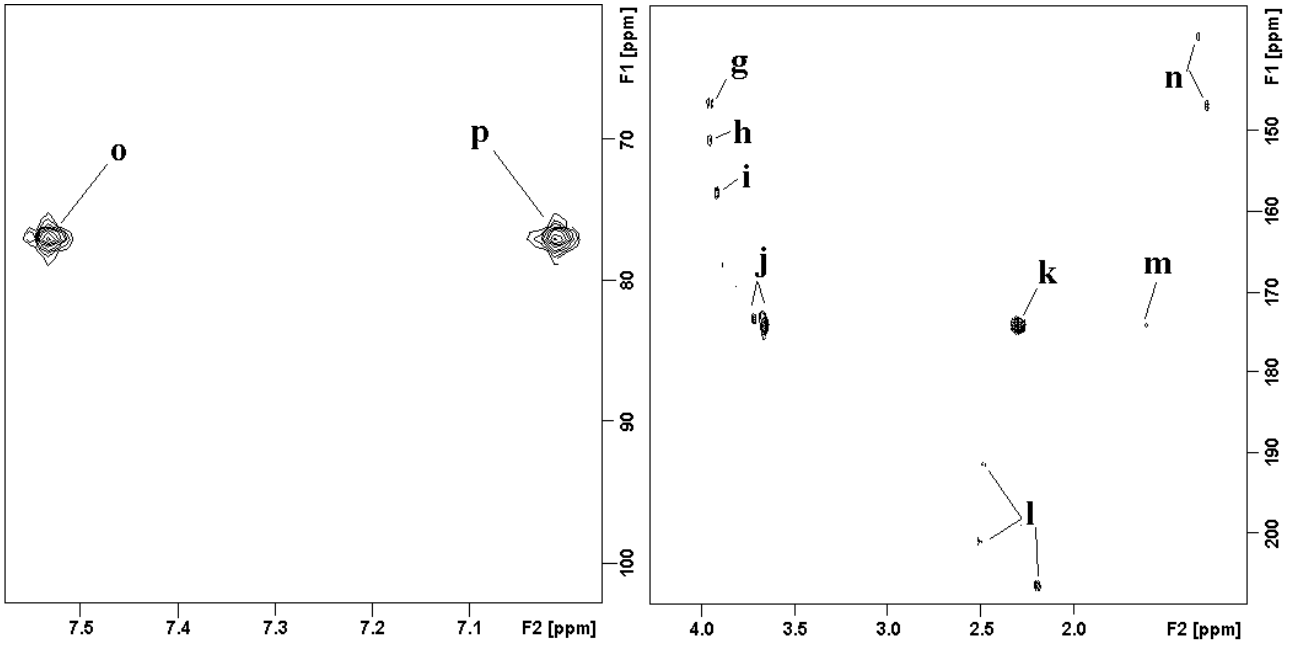
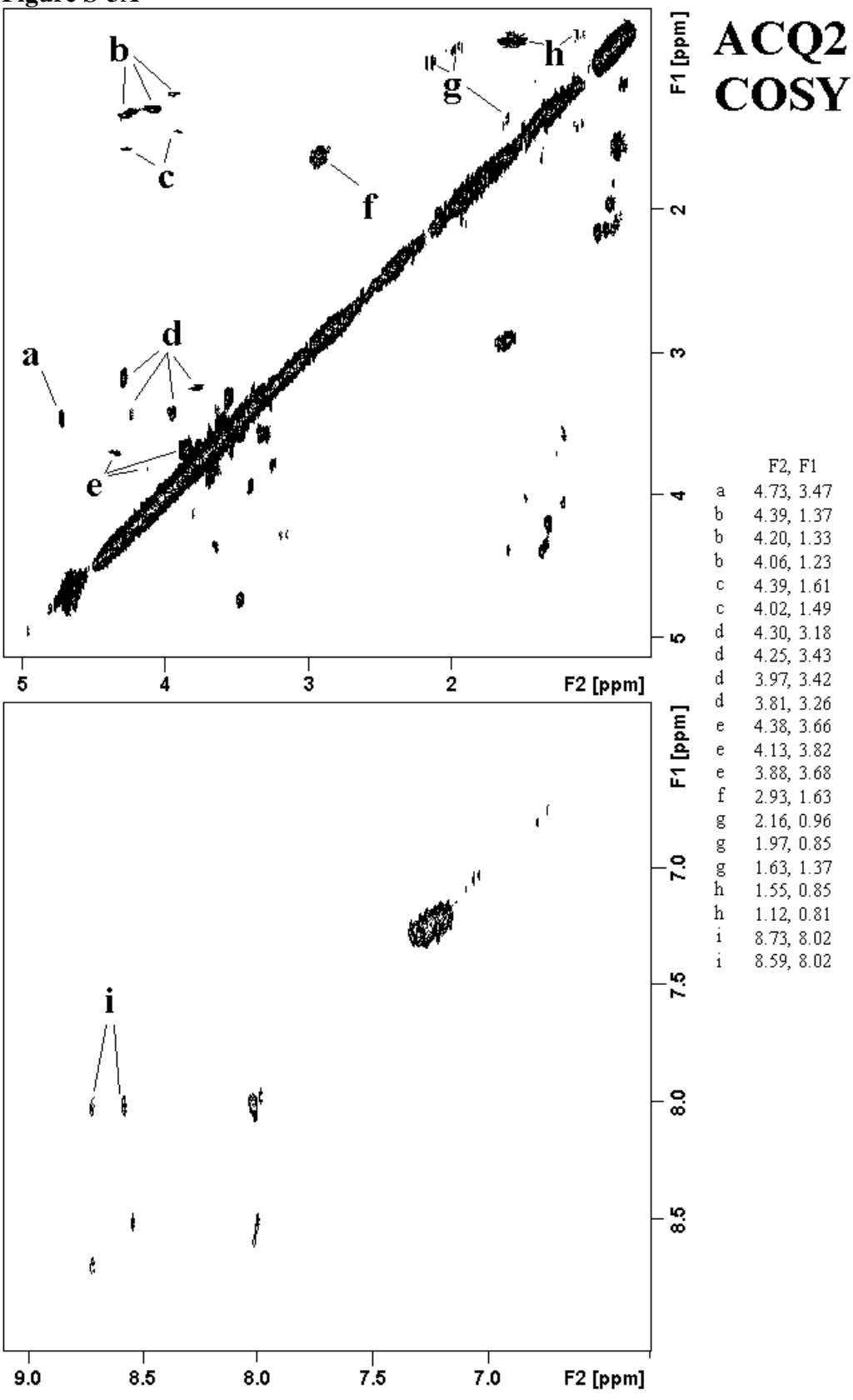


Figure S-3 A-C. Bidimensional enlarged spectra and peak labelling of hydrosoluble weakly-bound ester fraction (**ACQ2**) in DMSO-d6: homocorrelated ^{1}H-^{1}H COSY (**A**) and TOCSY (**B**): and heterocorrelated ^{1}H-^{13}C HSQC (**C**).

Figure S-3A



	F2, F1
a	4.73, 3.47
b	4.39, 1.37
b	4.20, 1.33
b	4.06, 1.23
c	4.39, 1.61
c	4.02, 1.49
d	4.30, 3.18
d	4.25, 3.43
d	3.97, 3.42
d	3.81, 3.26
e	4.38, 3.66
e	4.13, 3.82
e	3.88, 3.68
f	2.93, 1.63
g	2.16, 0.96
g	1.97, 0.85
g	1.63, 1.37
h	1.55, 0.85
h	1.12, 0.81
i	8.73, 8.02
i	8.59, 8.02

Figure S-3B

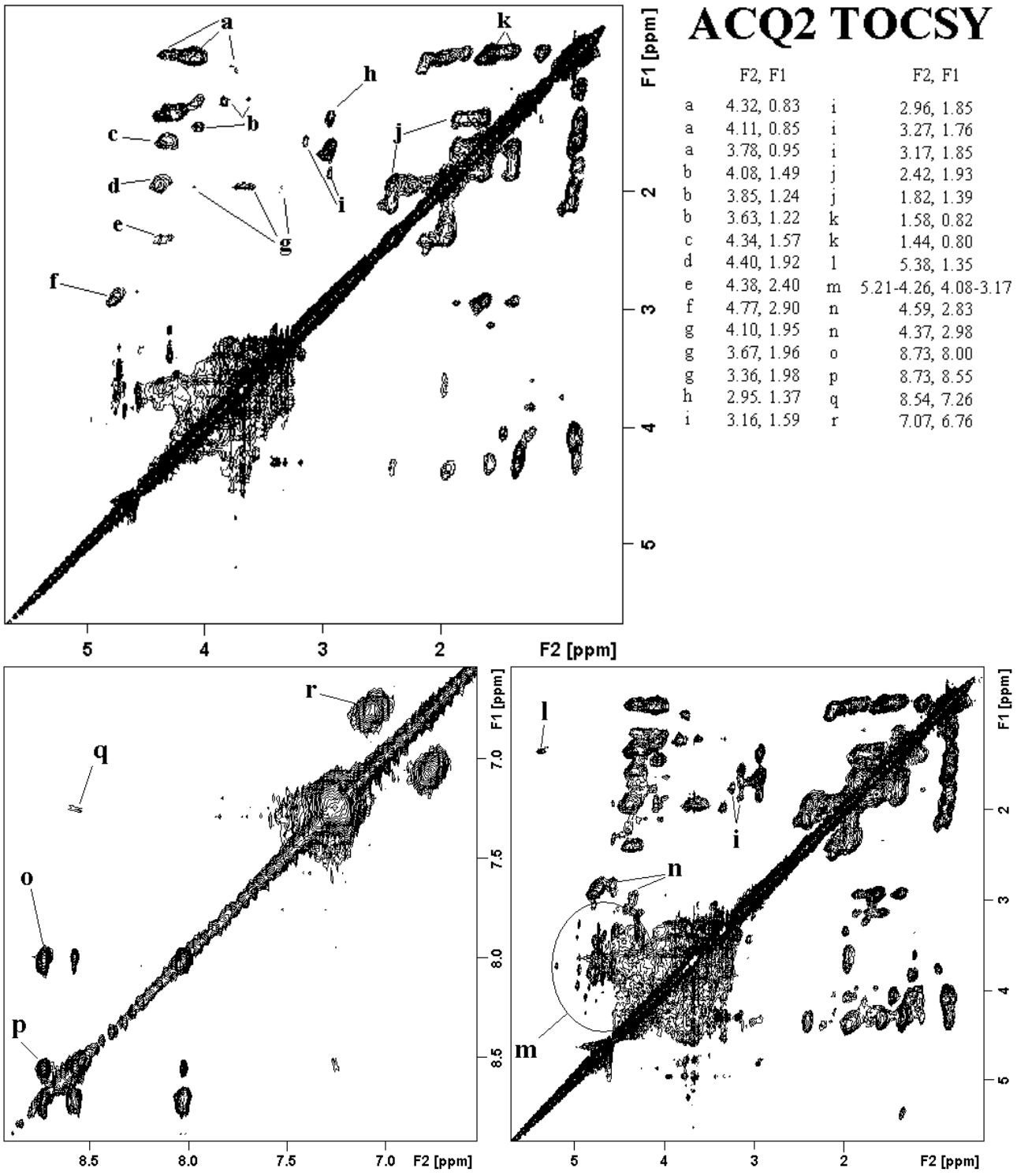
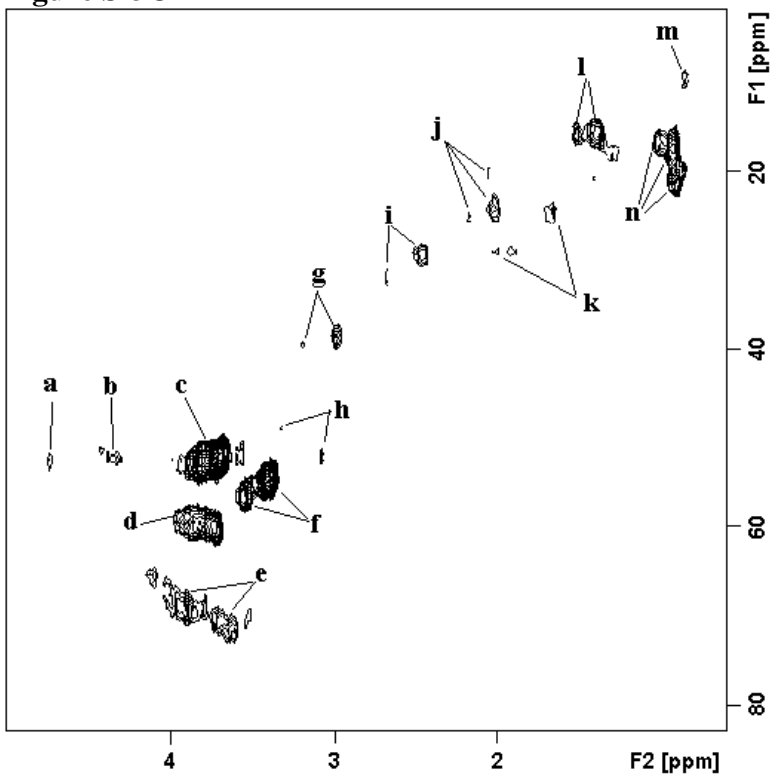
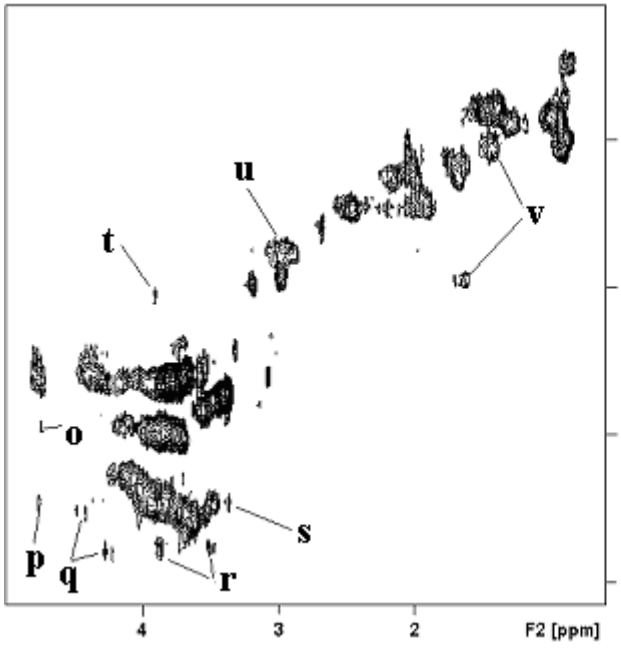

ACQ2 TOCSY
F2, F1
a 4.32, 0.83
a 4.11, 0.85
a 3.78, 0.95
b 4.08, 1.49
b 3.85, 1.24
b 3.63, 1.22
c 4.34, 1.57
d 4.40, 1.92
e 4.38, 2.40
f 4.77, 2.90
g 4.10, 1.95
g 3.67, 1.96
g 3.36, 1.98
h 2.95. 1.37
i 3.16, 1.59
i 2.96, 1.85
i 3.27, 1.76
i 3.17, 1.85
j 2.42, 1.93
j 1.82, 1.39
k 1.58, 0.82
k 1.44, 0.80
l 5.38, 1.35
m 5.21-4.26, 4.08-3.17
n 4.59, 2.83
n 4.37, 2.98
o 8.73, 8.00
p 8.73, 8.55
q 8.54, 7.26
r 7.07, 6.76
F1 [ppm]
F2 [ppm]

Figure S-3C



ACQ2 HSQC

	F2, F1		F2, F1
a	4.77, 52.3	m	0.87, 9.8
b	4.38, 52.2	n	1.03, 16.7
c	3.81, 53.0	n	0.94, 17.3
d	3.85, 59.5	n	0.92, 21.2
e	3.92, 69.2	o	4.75, 58.8
e	3.67, 71.3	p	4.77, 68.9
f	3.55, 56.5	q	4.45, 71.0
f	3.41, 54.7	q	4.25, 75.7
g	3.20, 39.5	r	3.87, 75.6
g	2.99, 38.7	r	3.49, 75.4
h	3.32, 48.5	s	3.37, 69.4
h	3.08, 52.1	t	3.91, 41.4
i	2.68, 32.1	u	3.00, 34.8
i	2.48, 29.5	v	1.45, 21.1
j	2.18, 25.0	v	1.64, 38.9
j	2.06, 20.2	w	7.28, 127.9
j	2.03, 24.4	x	6.80, 115.3
k	1.98, 29.0	y	5.32, 94.9
k	1.68, 24.7	y	5.02-4.78, 99.7-95.2
l	1.53, 16.0	z	4.59, 99.7
l	1.40, 15.9	z	4.35, 103.0



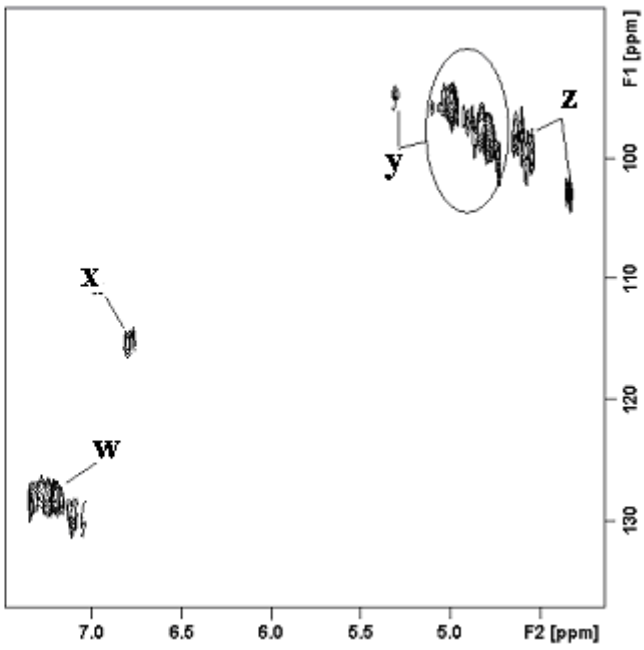
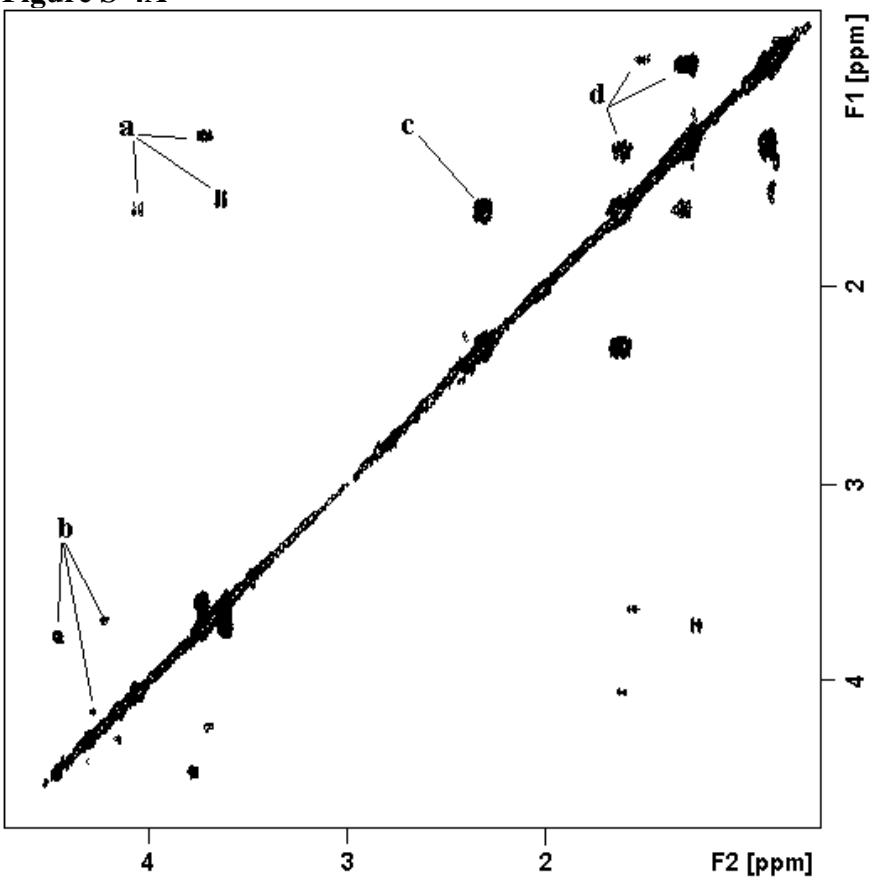


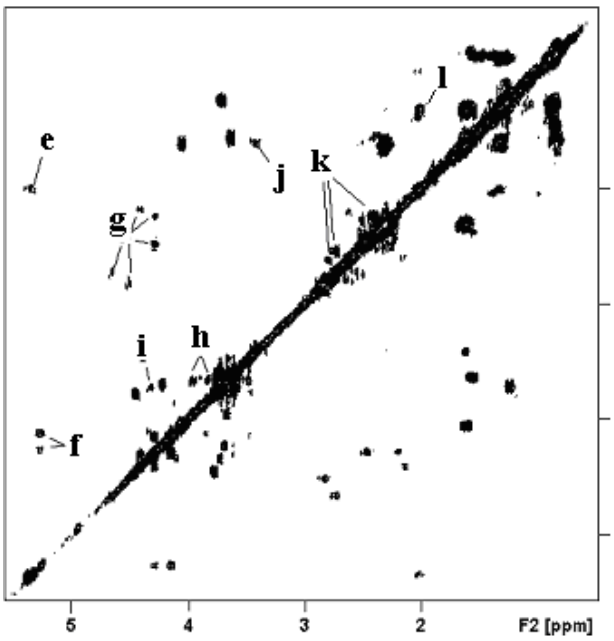
Figure S-4 A-D. Bidimensional enlarged spectra and peak labelling of organosoluble strongly-bound ester fraction (**ORG3**) in DMSO-d6: homocorrelated ^{1}H-^{1}H COSY (**A**), and TOCSY (**B**); and heterocorrelated ^{1}H-^{13}C HSQC (**C**), and HMBC (**D**).

Figure S-4A



ORG3 COSY

	F2, F1		F2, F1
a	4.06, 1.60	k	2.80, 2.63
a	3.64, 1.56	k	2.74, 2.54
a	3.72, 1.25	k	2.63, 2.20
b	4.47, 3.78	l	2.03, 1.34
b	4.31, 4.14	m	7.65, 9.00
b	4.24, 3.69	m	7.47, 8.93
c	2.33, 1.62	n	8.13, 6.89
d	1.62, 1.32	n	8.14, 7.00
d	1.51, 0.86	o	8.22, 7.53
d	1.30, 0.87	o	8.07, 7.46
e	5.37, 2.01	o	7.97, 7.52
f	5.26, 4.16	o	7.88, 7.51
f	5.25, 4.31	p	7.94, 6.91
g	4.67, 2.72	p	7.78, 6.94
g	4.52, 2.81	p	7.65, 6.94
g	4.42, 2.18	q	7.79, 7.18
g	4.30, 2.25	q	7.74, 7.54
g	4.30, 2.50	q	7.54, 7.14
h	3.96, 3.66	q	7.59, 7.46
h	3.84, 3.66	r	7.12, 6.82
i	4.34, 3.72	r	7.09, 6.59
j	3.42, 1.61	r	7.01, 6.46



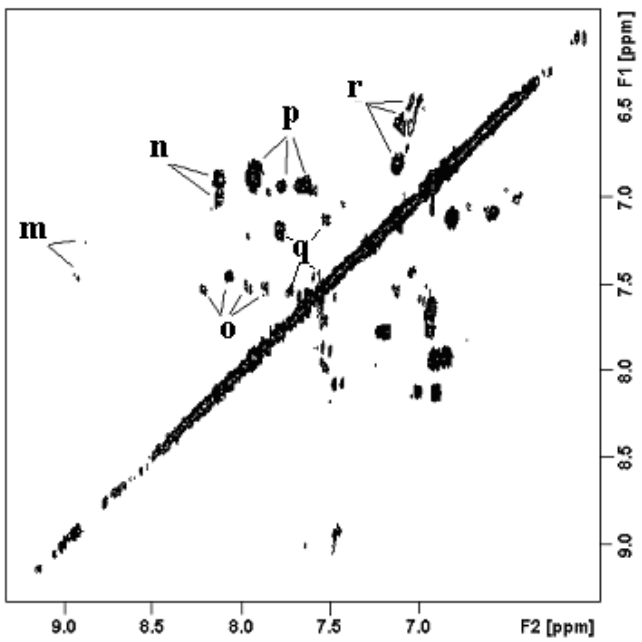


Figure S-4B

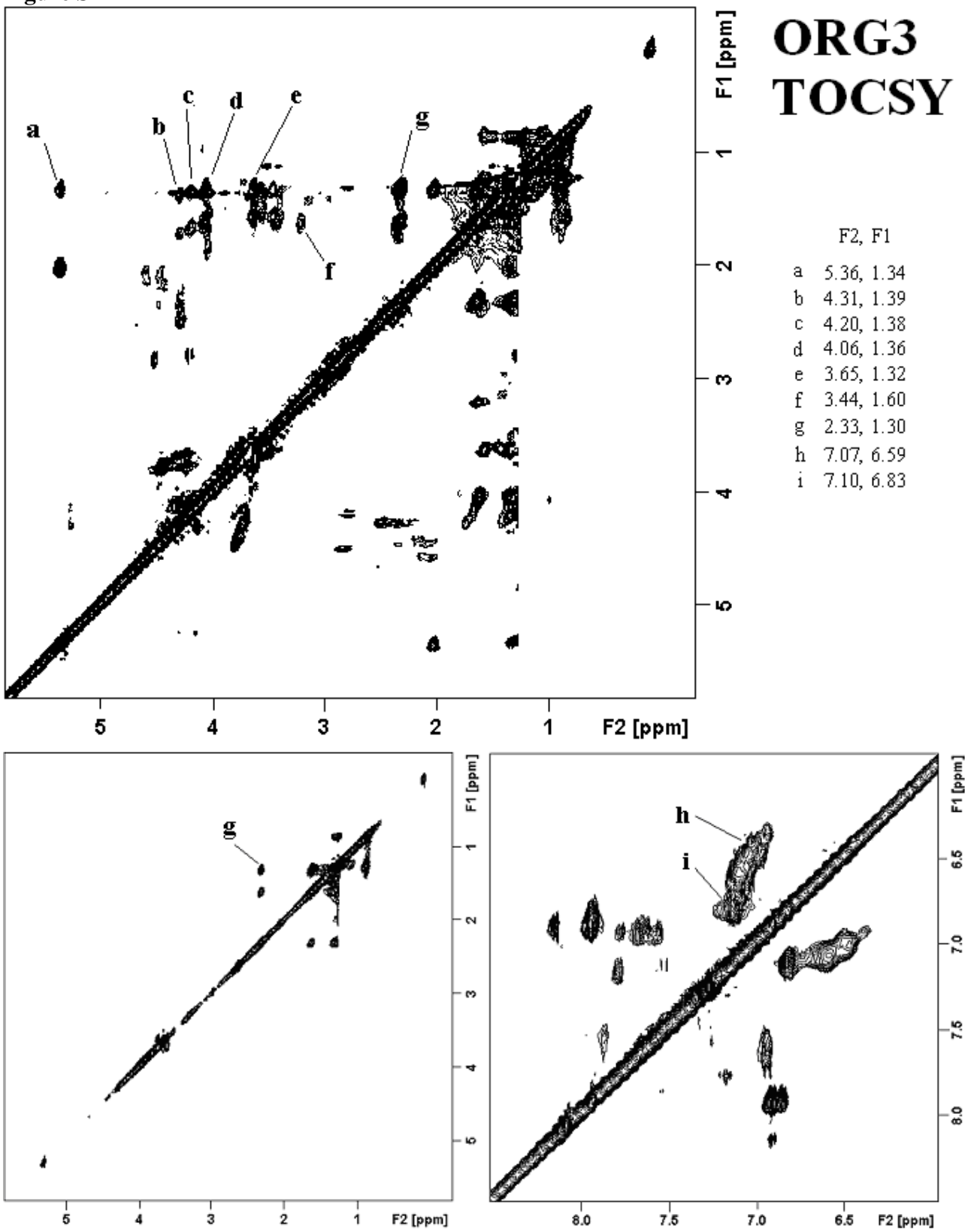

ORG3
TOCSY
F2, F1
a 5.36, 1.34
b 4.31, 1.39
c 4.20, 1.38
d 4.06, 1.36
e 3.65, 1.32
f 3.44, 1.60
g 2.33, 1.30
h 7.07, 6.59
i 7.10, 6.83
F1 [ppm]
F2 [ppm]

Figure S-4C

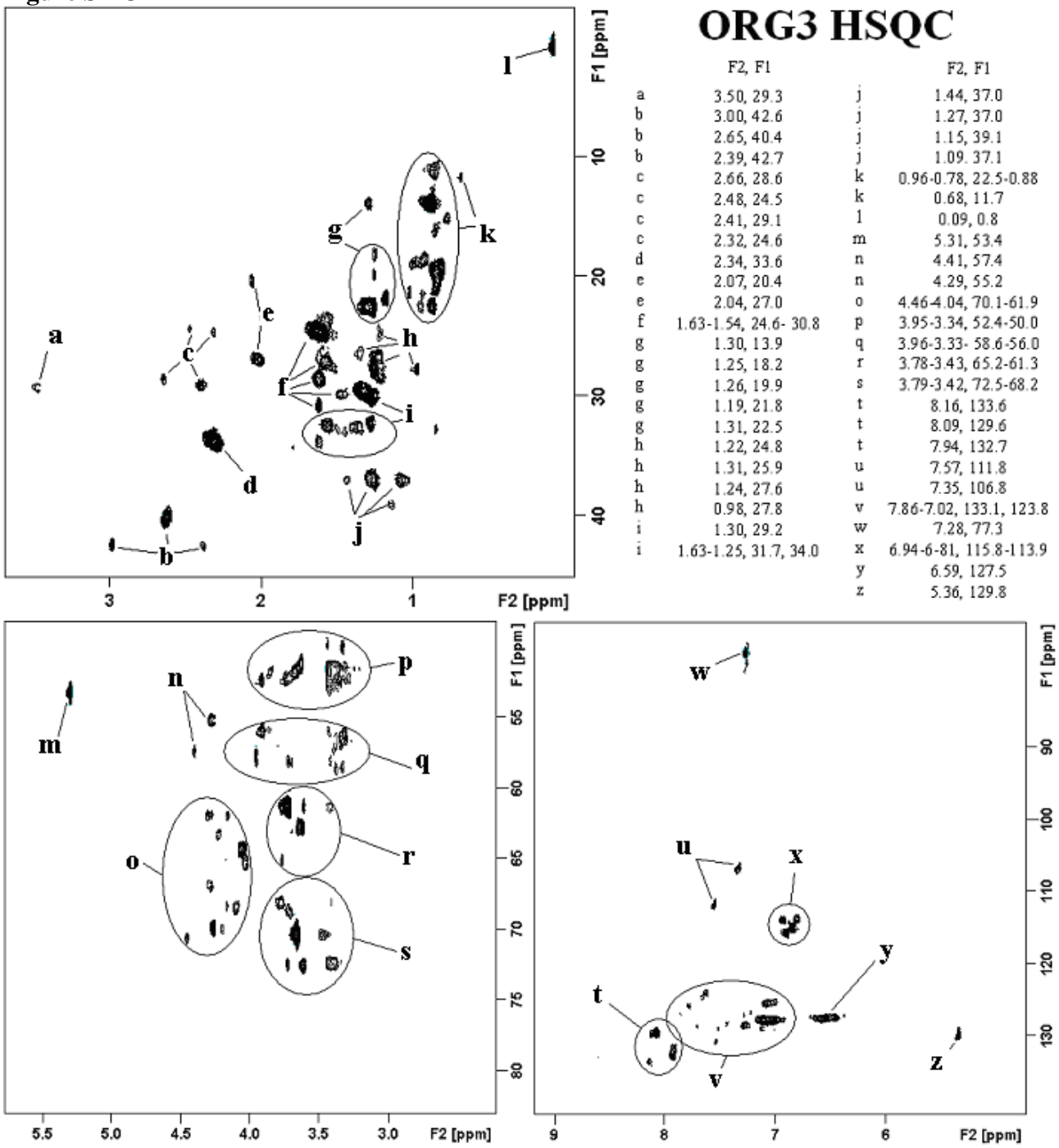
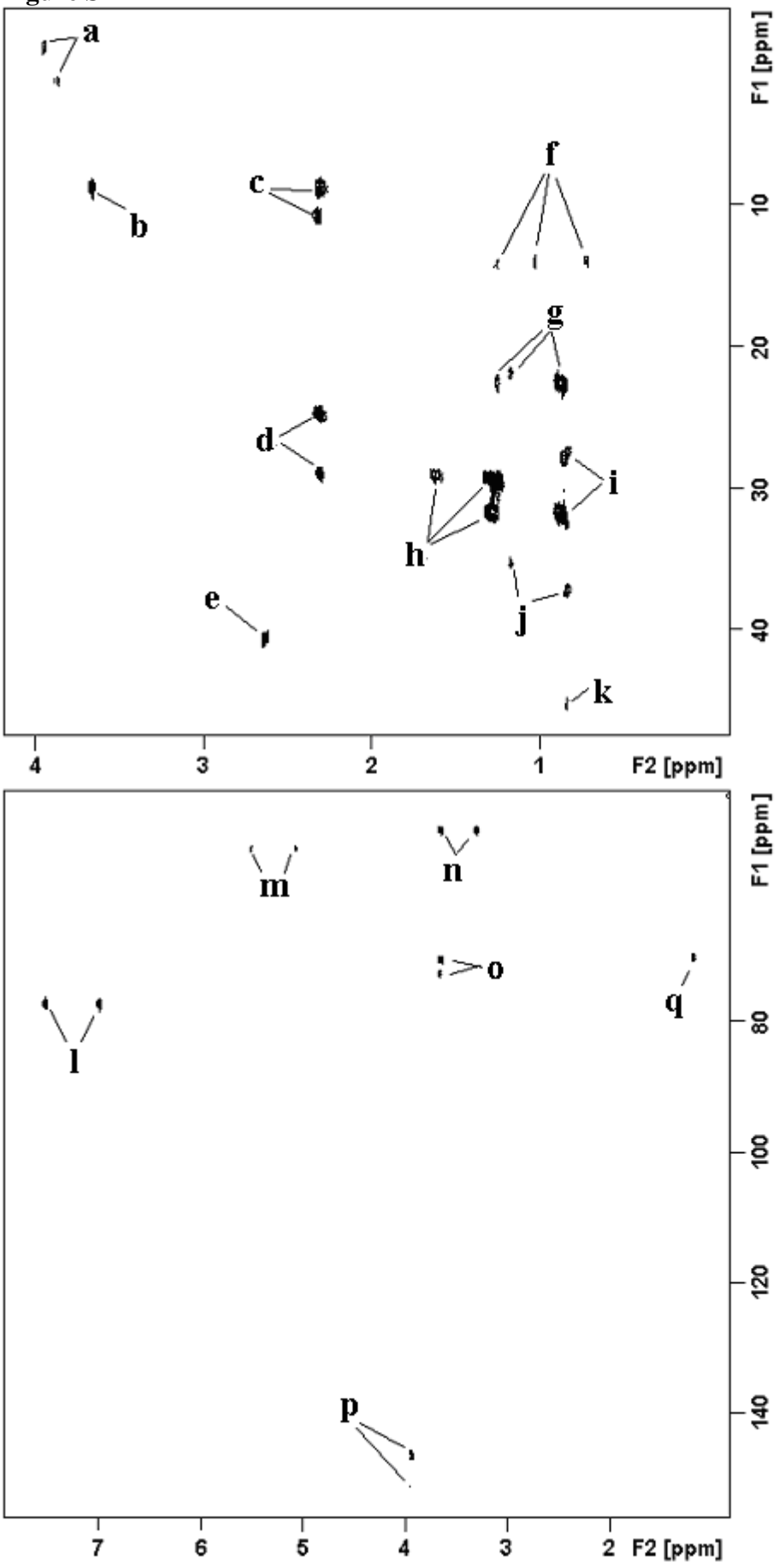
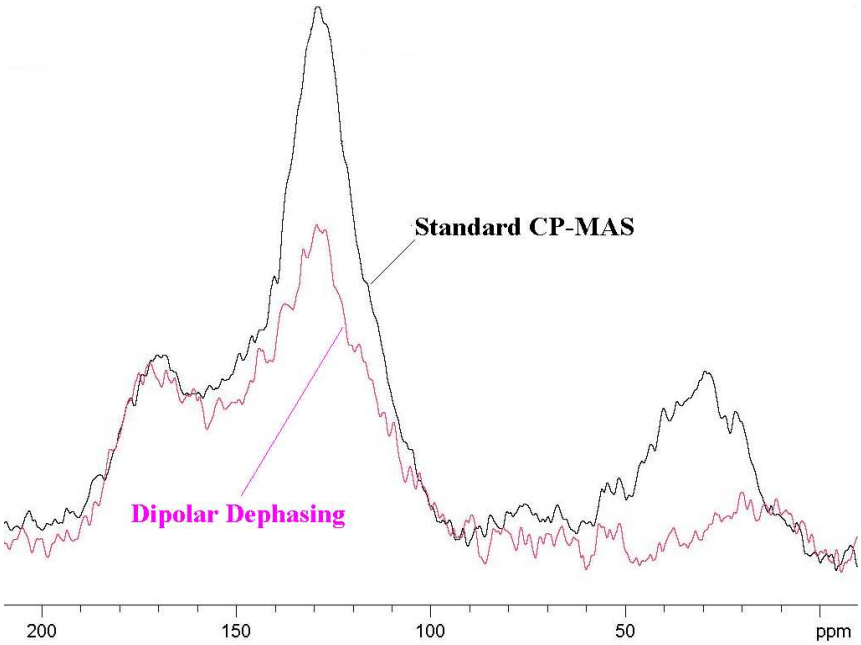

ORG3 HSQC
F2, F1
F2, F1
a 3.50, 29.3
b 3.00, 42.6
b 2.65, 40.4
b 2.39, 42.7
c 2.66, 28.6
c 2.48, 24.5
c 2.41, 29.1
c 2.32, 24.6
d 2.34, 33.6
e 2.07, 20.4
e 2.04, 27.0
f 1.63-1.54, 24.6- 30.8
g 1.30, 13.9
g 1.25, 18.2
g 1.26, 19.9
g 1.19, 21.8
g 1.31, 22.5
h 1.22, 24.8
h 1.31, 25.9
h 1.24, 27.6
h 0.98, 27.8
i 1.30, 29.2
i 1.63-1.25, 31.7, 34.0
j 1.44, 37.0
j 1.27, 37.0
j 1.15, 39.1
j 1.09. 37.1
k 0.96-0.78, 22.5-0.88
k 0.68, 11.7
l 0.09, 0.8
m 5.31, 53.4
n 4.41, 57.4
n 4.29, 55.2
o 4.46-4.04, 70.1-61.9
p 3.95-3.34, 52.4-50.0
q 3.96-3.33- 58.6-56.0
r 3.78-3.43, 65.2-61.3
s 3.79-3.42, 72.5-68.2
t 8.16, 133.6
t 8.09, 129.6
t 7.94, 132.7
u 7.57, 111.8
u 7.35, 106.8
v 7.86-7.02, 133.1, 123.8
w 7.28, 77.3
x 6.94-6-81, 115.8-113.9
y 6.59, 127.5
z 5.36, 129.8
F1 [ppm]
F2 [ppm]

Figure S-4D



	F2, F1
a	3.96, -1.3
a	3.80, 1.3
b	3.67, 8.7
c	2.31, 8.7
c	2.33, 10.7
d	2.32, 24.6
d	2.31, 28.9
e	2.64, 40.5
f	1.26, 14.1
f	1.04, 14.1
f	0.73, 14.1
g	1.26, 22.6
g	1.19, 21.9
g	0.89, 22.4
h	1.62, 29.1
h	1.27, 29.3
h	1.29, 31.6
i	0.85, 27.8
i	0.88, 31.8
j	1.18, 35.4
j	0.84, 37.2
k	0.84, 45.2
l	7.55, 77.3
l	7.02, 77.0
m	5.53, 53.3
m	5.08, 53.3
n	3.67, 50.6
n	3.31, 50.6
o	3.66, 70.3
o	3.66, 72.6
p	3.95, 142.4
p	3.95, 151.4
q	1.18, 70.1

Figure S-5. Dipolar Dephasing (DD) and ^{13}C-CPMAS spectra, and normalized area in spectral regions for RES4.



Spectral region (ppm)	150-185	95-150	0-60
CPMAS	1.00	3.42	1.30
DD	1.00	2.47	0.42

Figure S-6 A-C. Bidimensional enlarged spectra and peak labelling of final fractionation residue (**RES4**) in D_2O/NaOD: homocorrelated ^1H-^1H COSY (**A**), and TOCSY (**B**): and heterocorrelated ^1H-^{13}C HSQC (**C**).

Figure S-6A

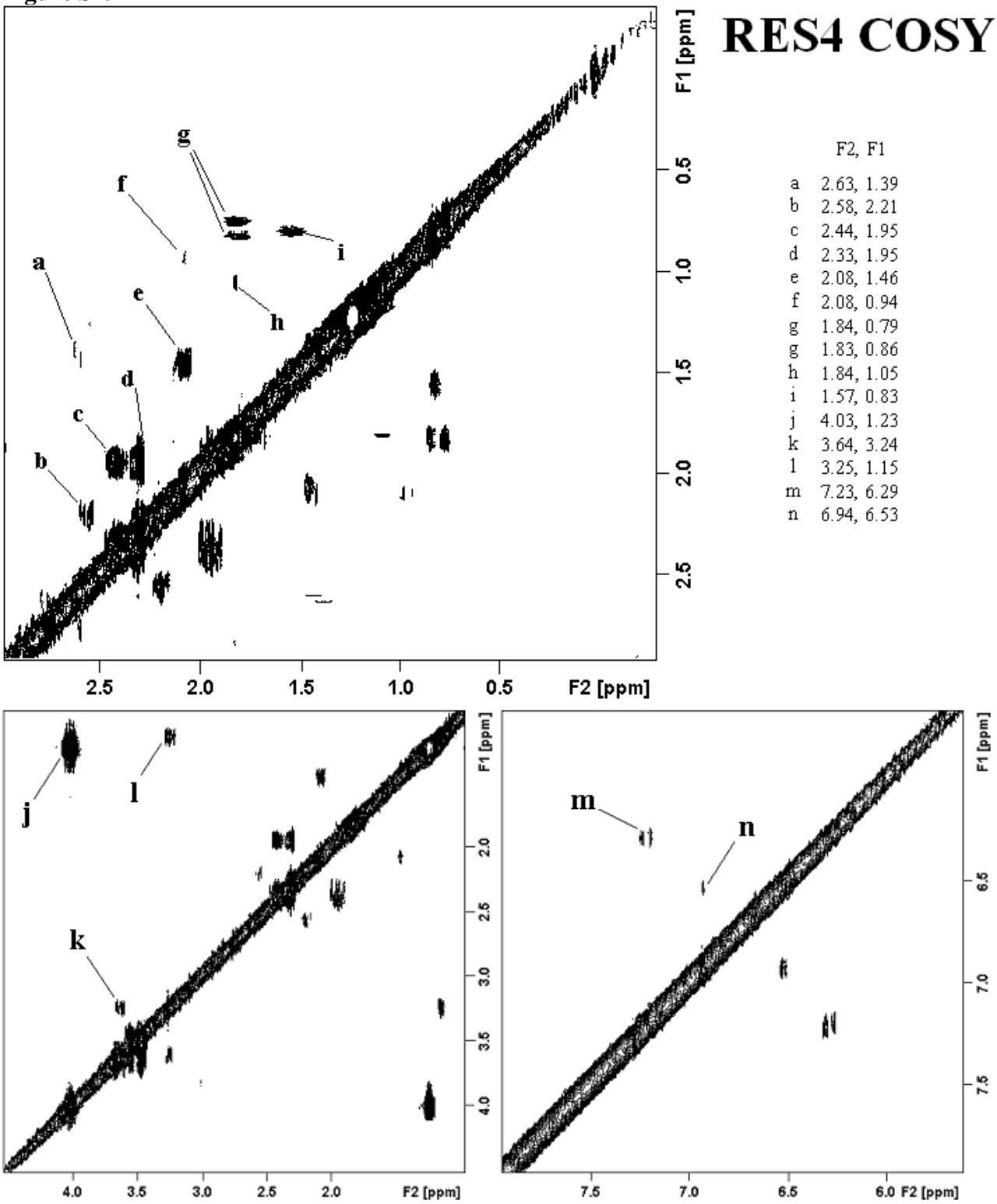


Figure S-6B

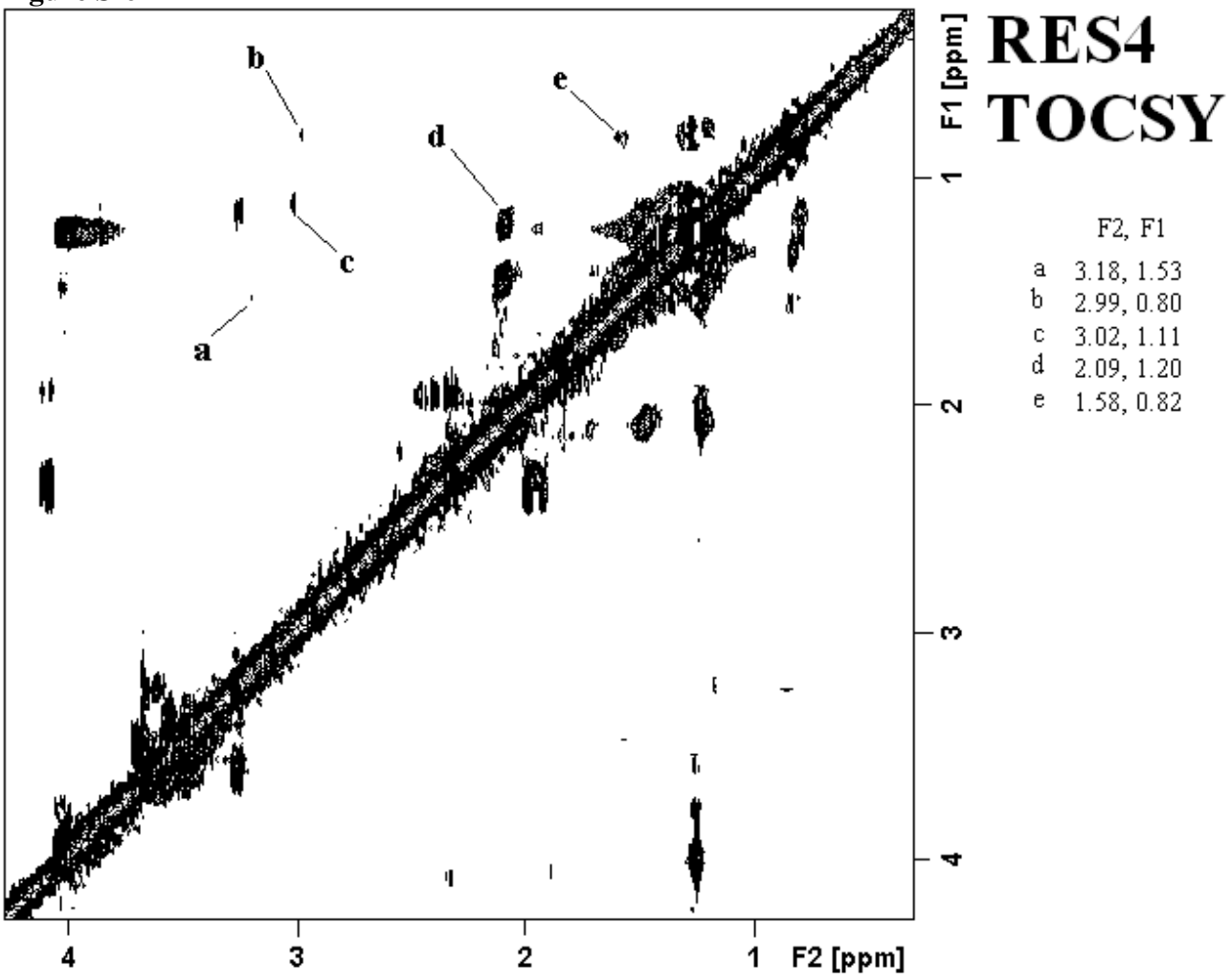
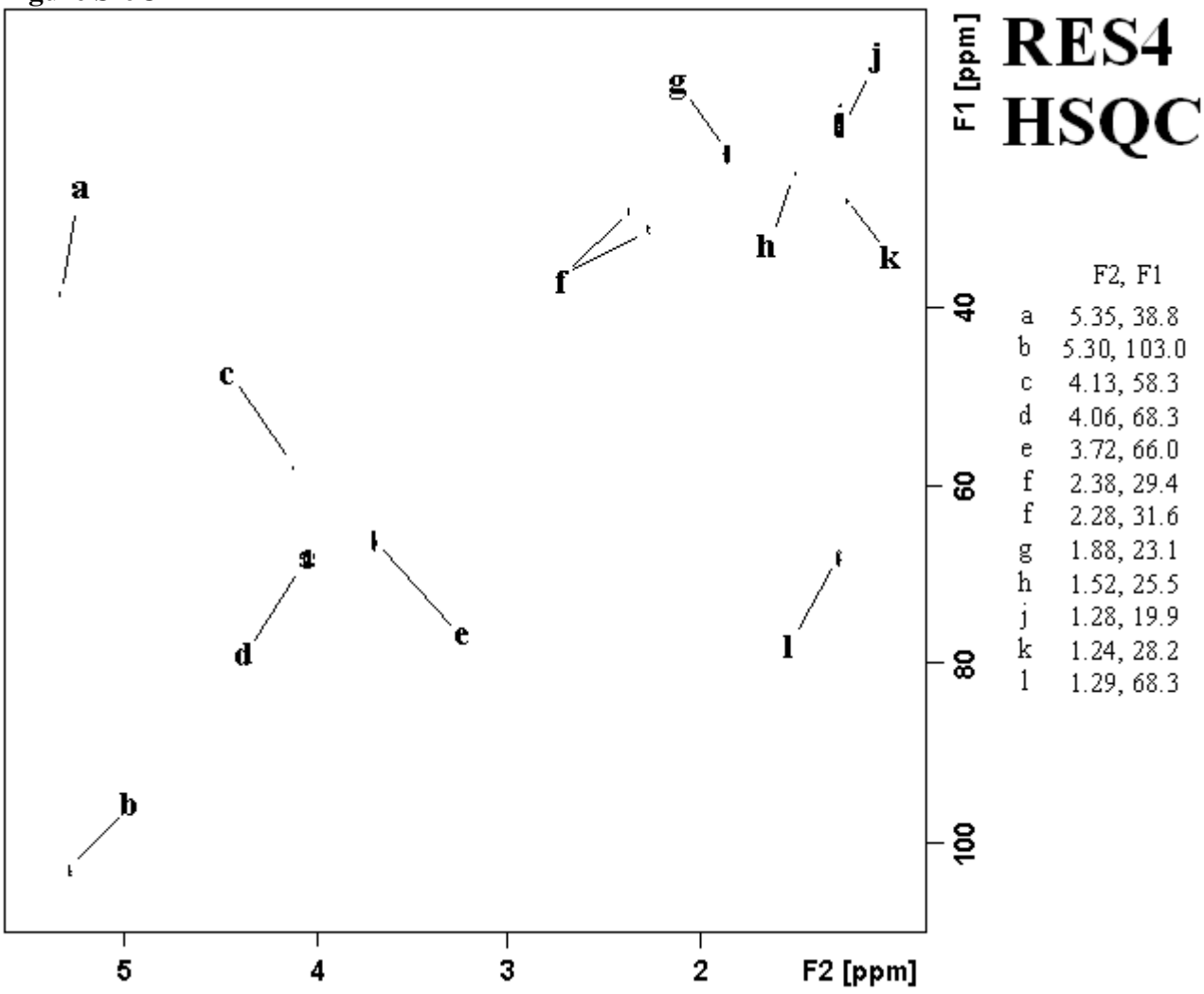

RES4
TOCSY
F2, F1
a 3.18, 1.53
b 2.99, 0.80
c 3.02, 1.11
d 2.09, 1.20
e 1.58, 0.82
F1 [ppm]
F2 [ppm]
a
b
c
d
e

Figure S-6C


RES4
HSQC
F1 [ppm]
F2 [ppm]
40
60
80
100
5
4
3
2
a
b
c
d
e
f
g
h
j
k
l
F2, F1
a 5.35, 38.8
b 5.30, 103.0
c 4.13, 58.3
d 4.06, 68.3
e 3.72, 66.0
f 2.38, 29.4
f 2.28, 31.6
g 1.88, 23.1
h 1.52, 25.5
j 1.28, 19.9
k 1.24, 28.2
l 1.29, 68.3