Electronic Supplementary Material (ESI) for Analytical Methods.

SUPPORTING INFORMATION

Exploring the complementarity of fast multipulse and multidimensional NMR methods for metabolomics: a chemical ecology case study

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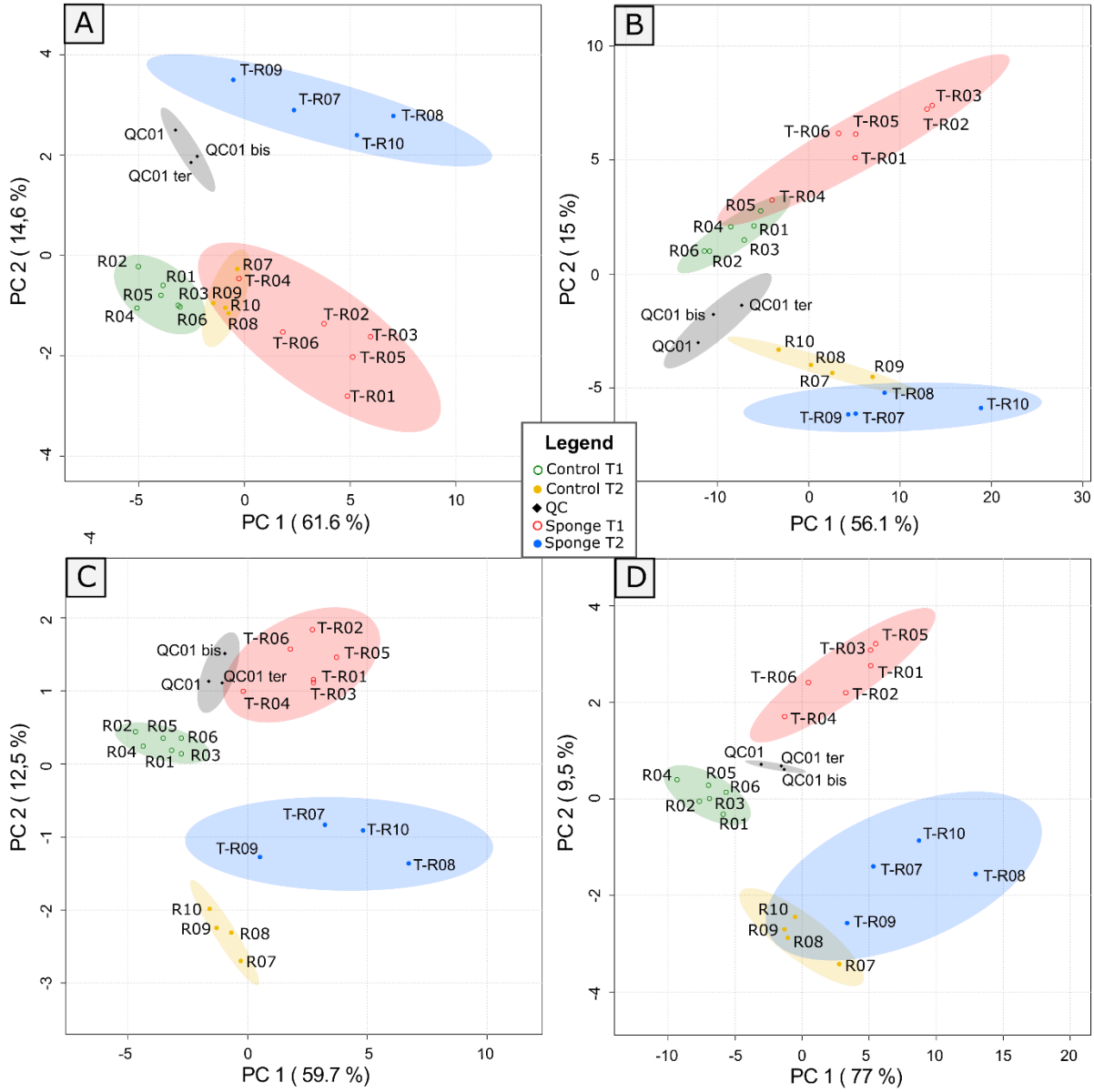

Figure SI 1 - 2D PCA score plots with QC: (A) ^{1}H; (B) ^{13}C; (C) UF COSY; (D) SYMAPS HSQC

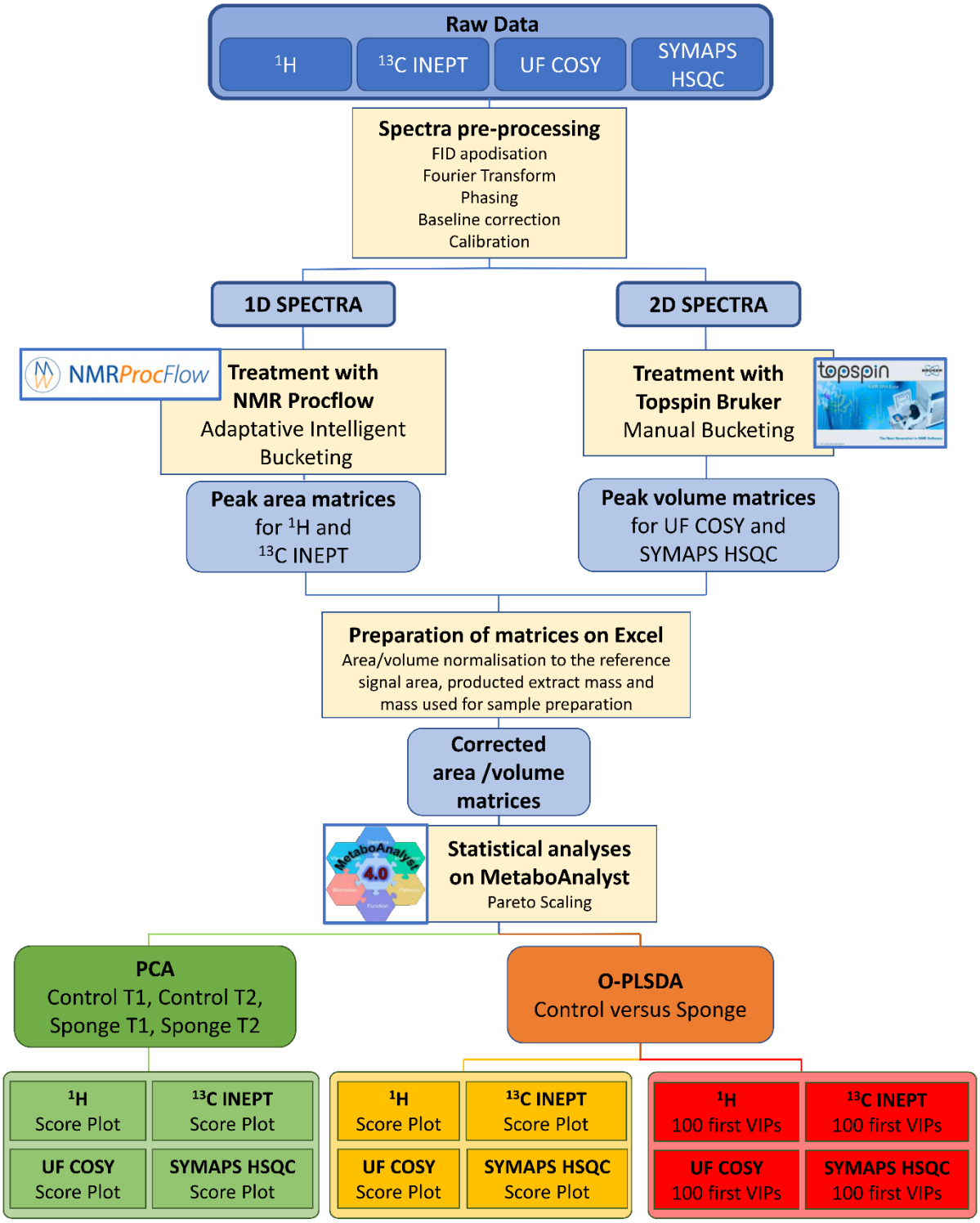

Figure SI 2 - NMR data processing workflow

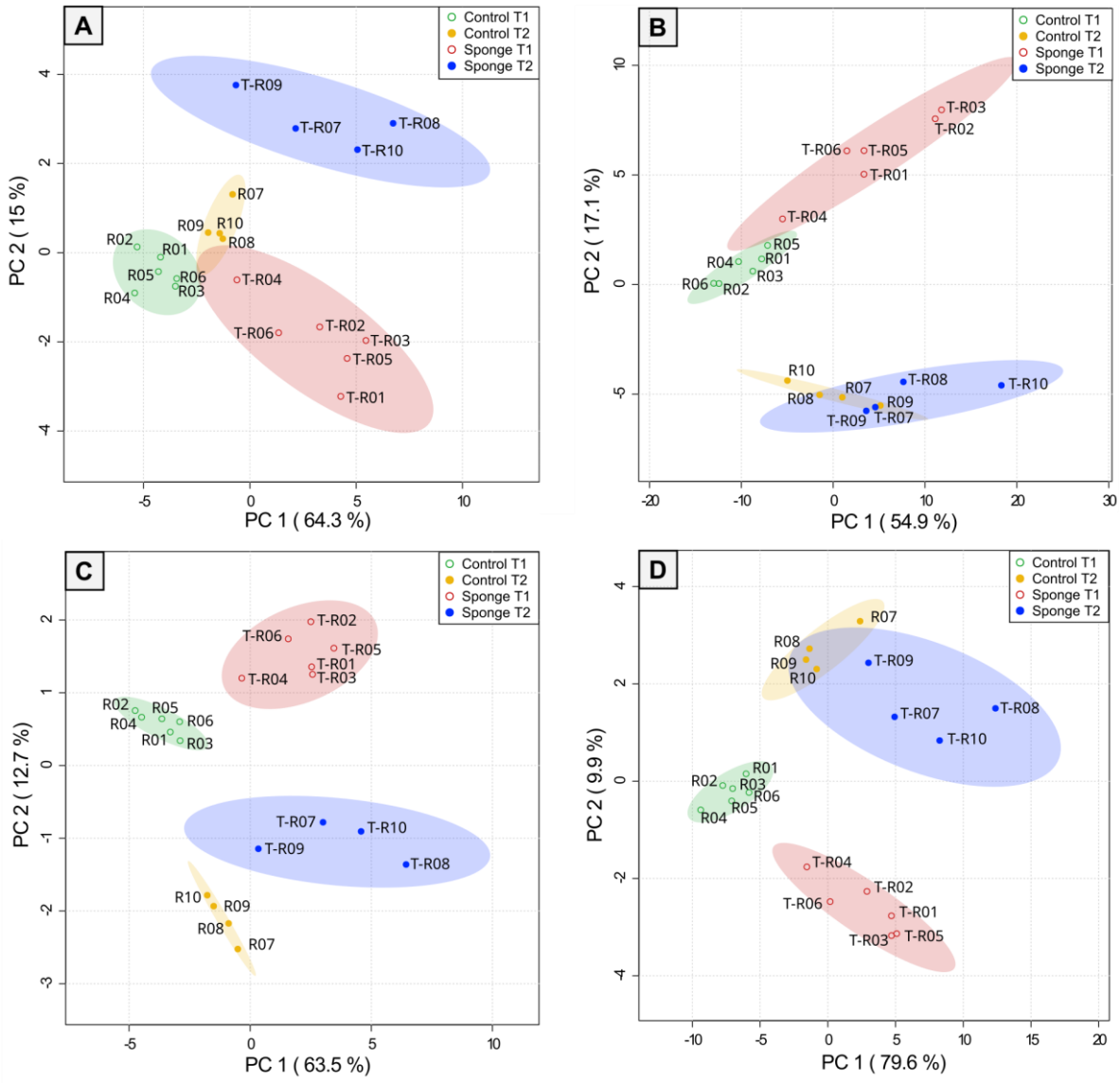

Figure SI 3 - 2D PCA score plots: (A) ^{1}H; (B) ^{13}C; (C) UF COSY; (D) SYMAPS HSQC

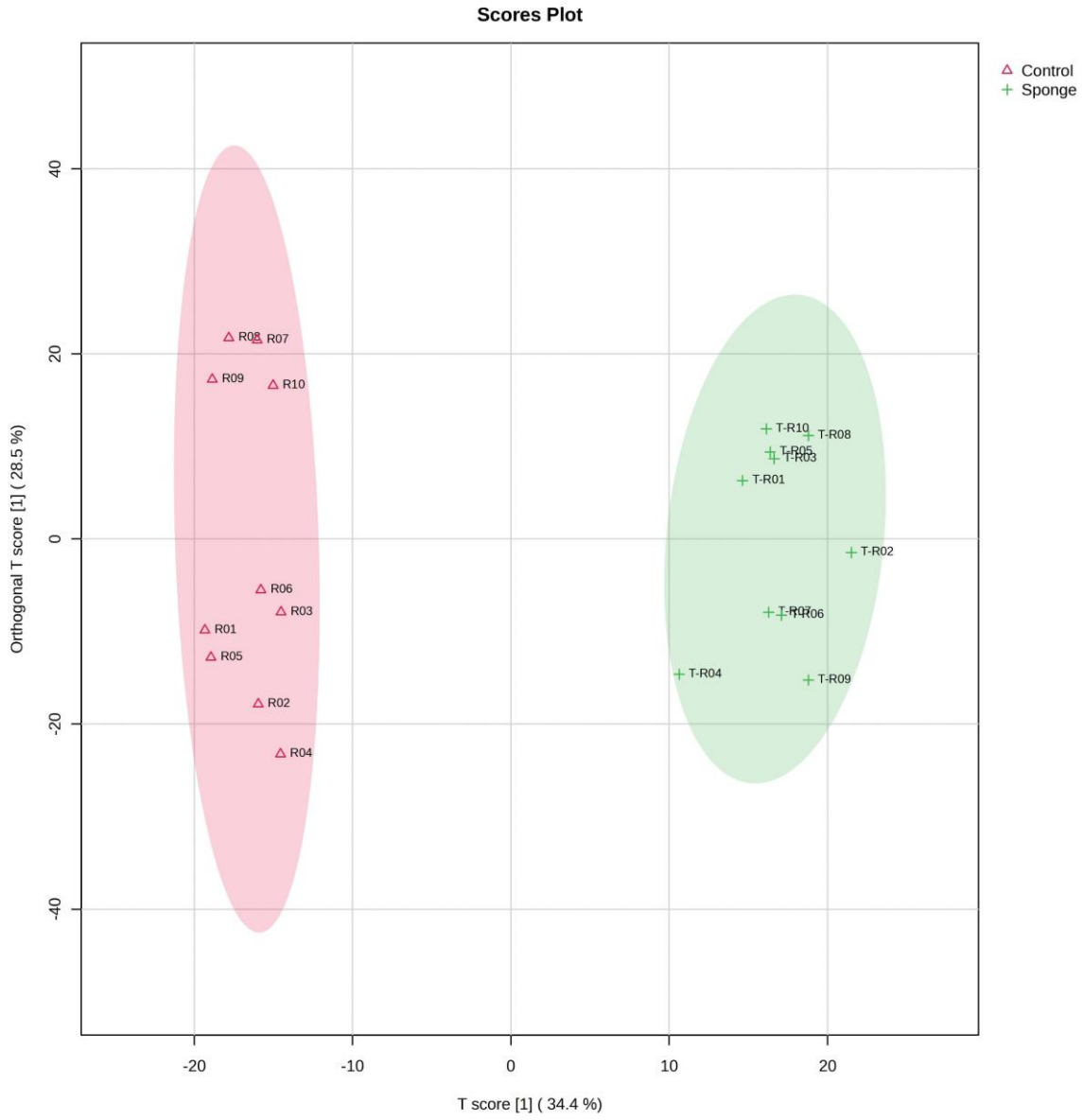

Figure SI 4 - ^{1}H OPLS-DA score plot: medium effect

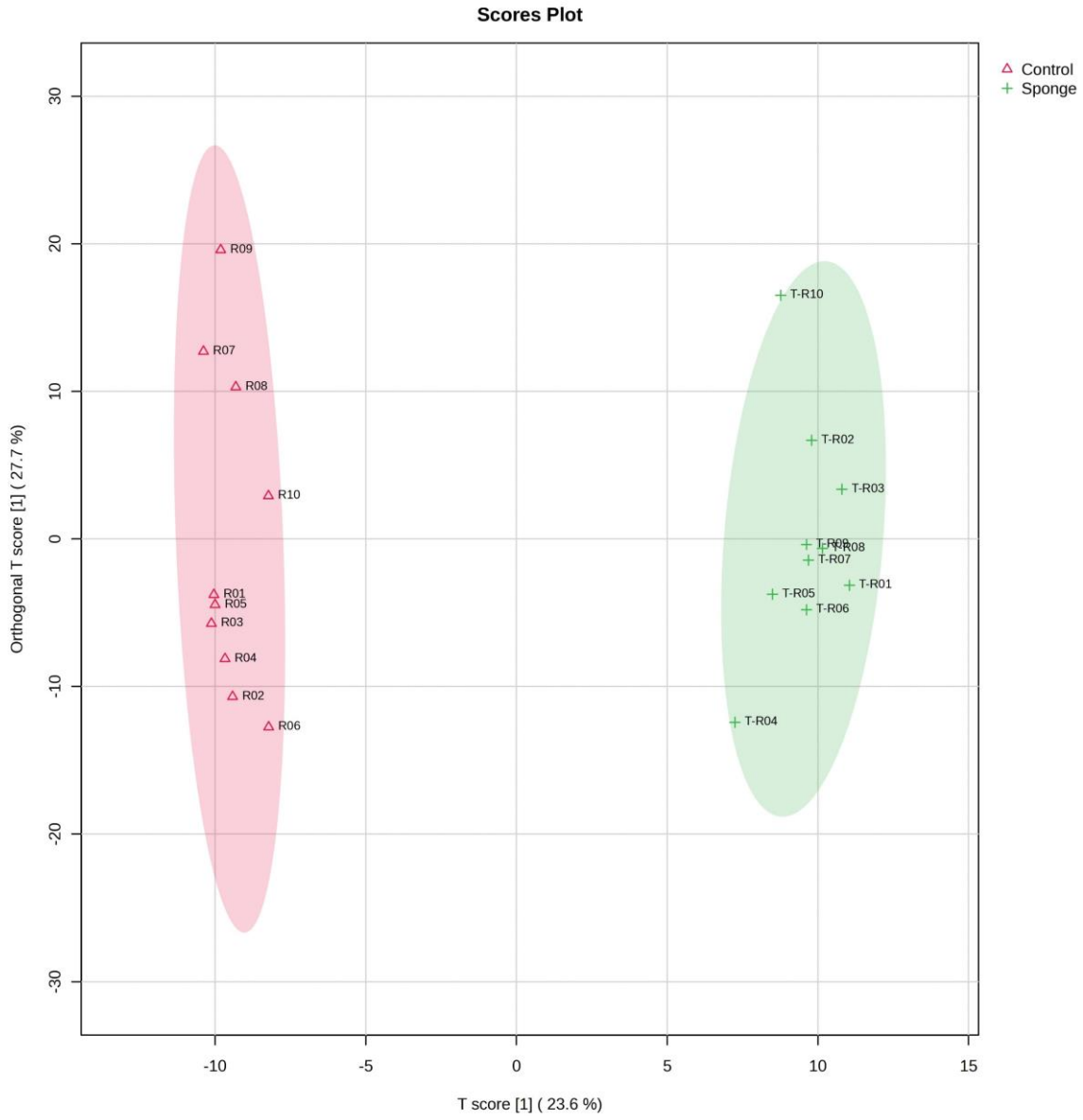

Figure SI 5 - ^{13}C INEPT OPLS-DA score plot: medium effect

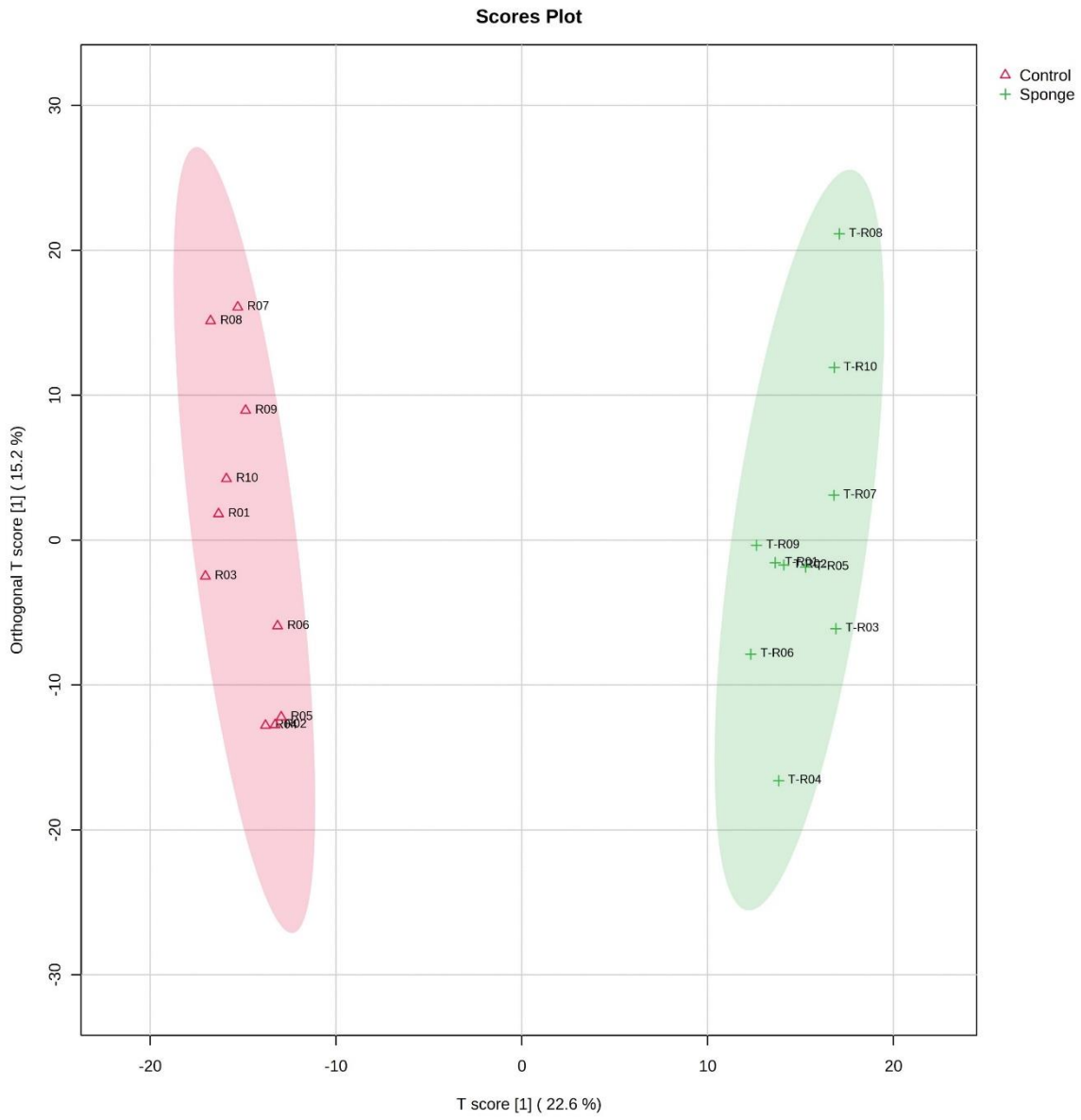

Figure SI 6 - UF COSY OPLS-DA score plot: medium effect

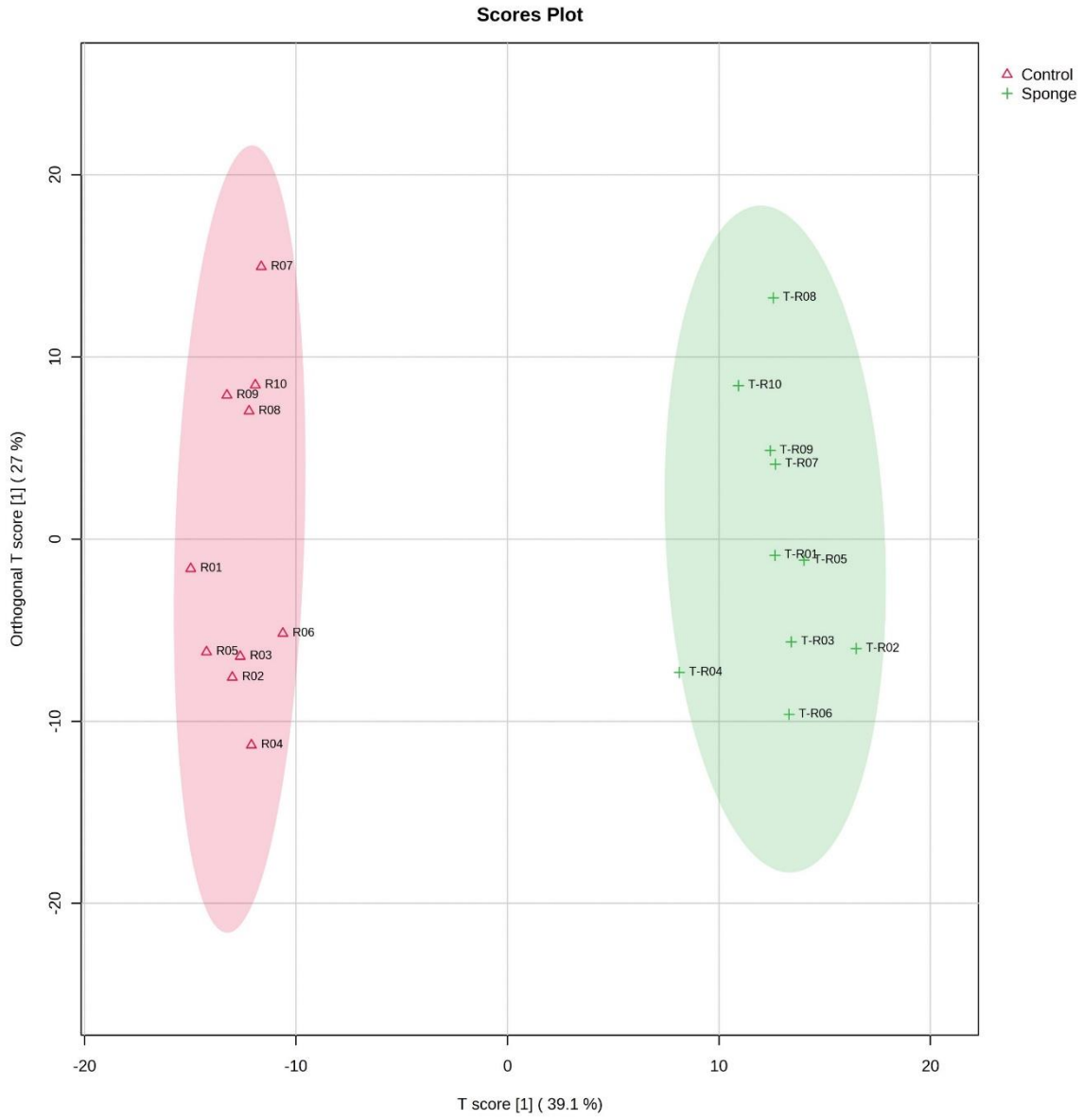

Figure SI 7 - SYMAPS HSQC OPLS-DA score plot : medium effect

100 first VIP buckets

15 to 16 ppm	14 to 15 ppm	13 to 14 ppm	12 to 13 ppm	11 to 12 ppm	10 to 11 ppm	9 to 10 ppm	8 to 9 ppm	7 to 8 ppm	6 to 7 ppm	5 to 6 ppm	4 to 5 ppm	3 to 4 ppm	2 to 3 ppm	1 to 2 ppm	0 to 1 ppm
0	0	0	0	2	3	0	0	12	16	26	2	4	3	14	1

All buckets

15 to 16 ppm	14 to 15 ppm	13 to 14 ppm	12 to 13 ppm	11 to 12 ppm	10 to 11 ppm	9 to 10 ppm	8 to 9 ppm	7 to 8 ppm	6 to 7 ppm	5 to 6 ppm	4 to 5 ppm	3 to 4 ppm	2 to 3 ppm	1 to 2 ppm	0 to 1 ppm
1	0	0	9	11	33	19	35	99	111	106	91	98	91	106	90

Min — Max

Figure SI 8 - Distribution of the 100 first VIPs and all ^{1}H buckets: the upper part reports the 100 first VIP buckets highlighted by OPLS-DA analysis; the lower part reports all ^{1}H buckets. Color code corresponds to bucket density in the spectrum range, from low amount (blue), to medium (white) and high (red).

100 first VIP buckets

140 to 150 ppm	130 to 140 ppm	120 to 130 ppm	110 to 120 ppm	100 to 110 ppm	90 to 100 ppm	80 to 90 ppm	70 to 80 ppm	60 to 70 ppm	50 to 60 ppm	40 to 50 ppm	30 to 40 ppm	20 to 30 ppm	10 to 20 ppm	0 to 10 ppm
6	3	14	23	3	0	0	1	0	4	1	9	28	8	0

All buckets

140 to 150 ppm	130 to 140 ppm	120 to 130 ppm	110 to 120 ppm	100 to 110 ppm	90 to 100 ppm	80 to 90 ppm	70 to 80 ppm	60 to 70 ppm	50 to 60 ppm	40 to 50 ppm	30 to 40 ppm	20 to 30 ppm	10 to 20 ppm	0 to 10 ppm	-10 to 0 ppm
18	19	82	62	7	1	0	3	6	23	20	39	83	40	0	5

Min — Max

Figure SI 9 - Distribution of the 100 first VIP and all ^{13}C INEPT buckets: the upper part reports the 100 first VIP buckets highlighted by OPLS-DA analysis; the lower part reports all ^{13}C buckets. Color code corresponds to bucket density in the spectrum range, from low amount (blue), to medium (white) and high (red).

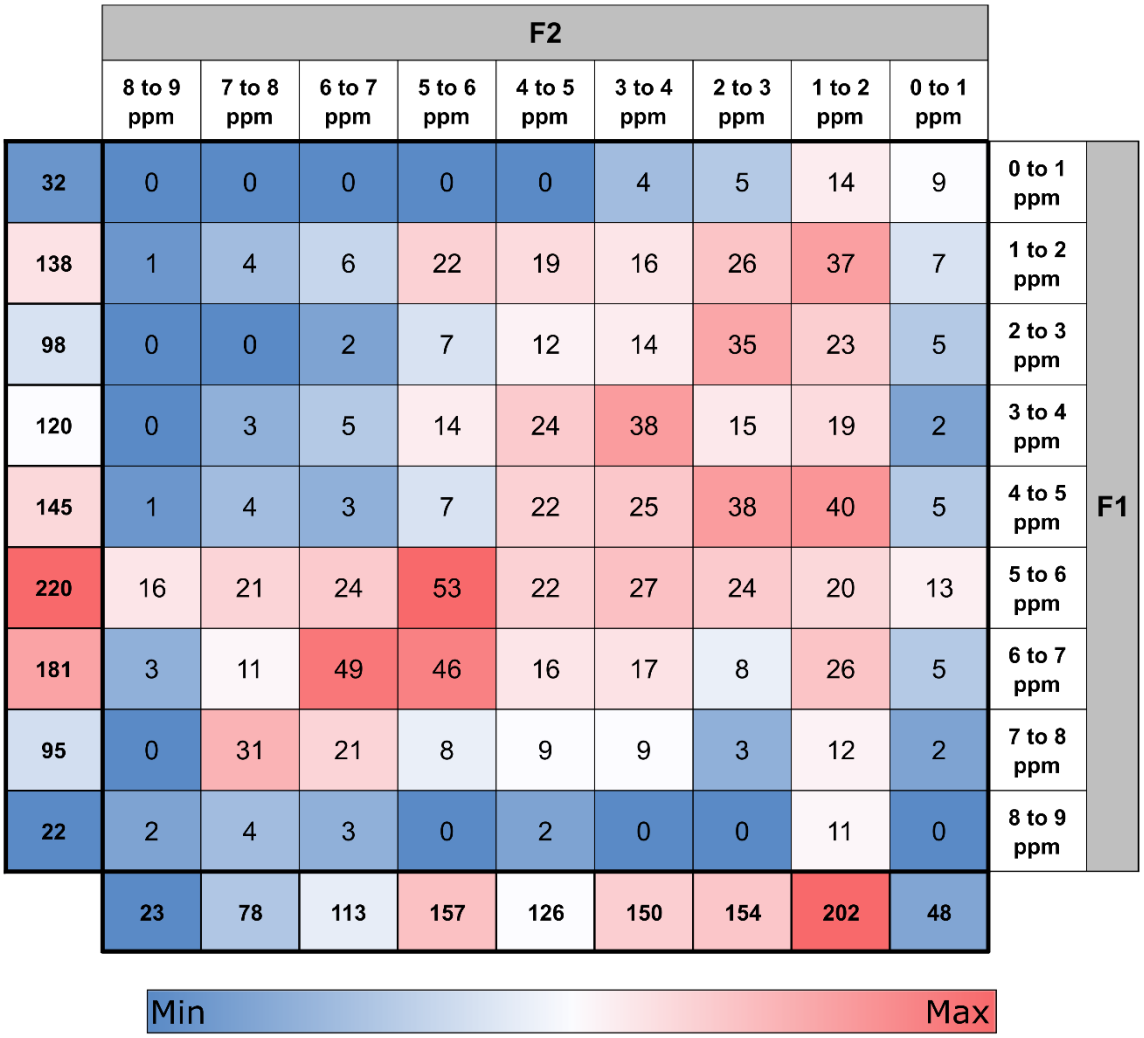

Figure SI 10 - Spectral distribution of UF COSY buckets. Color code corresponds to bucket density in the spectrum range, from low amount (blue), to medium (white) and high (red).

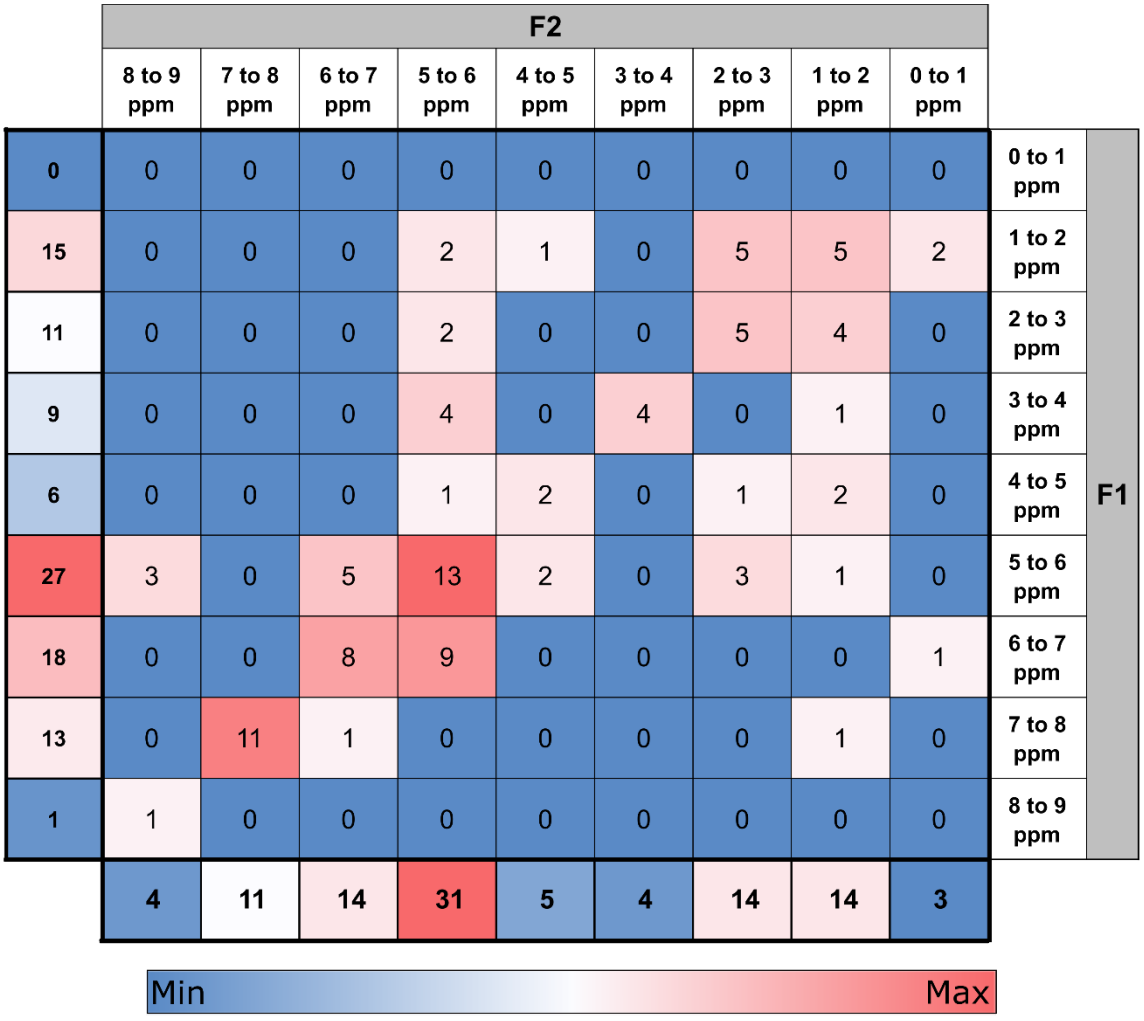

Figure SI 11 - Spectral distribution of the first 100 VIP in UF COSY. Color code corresponds to bucket density in the spectrum range, from low amount (blue), to medium (white) and high (red).

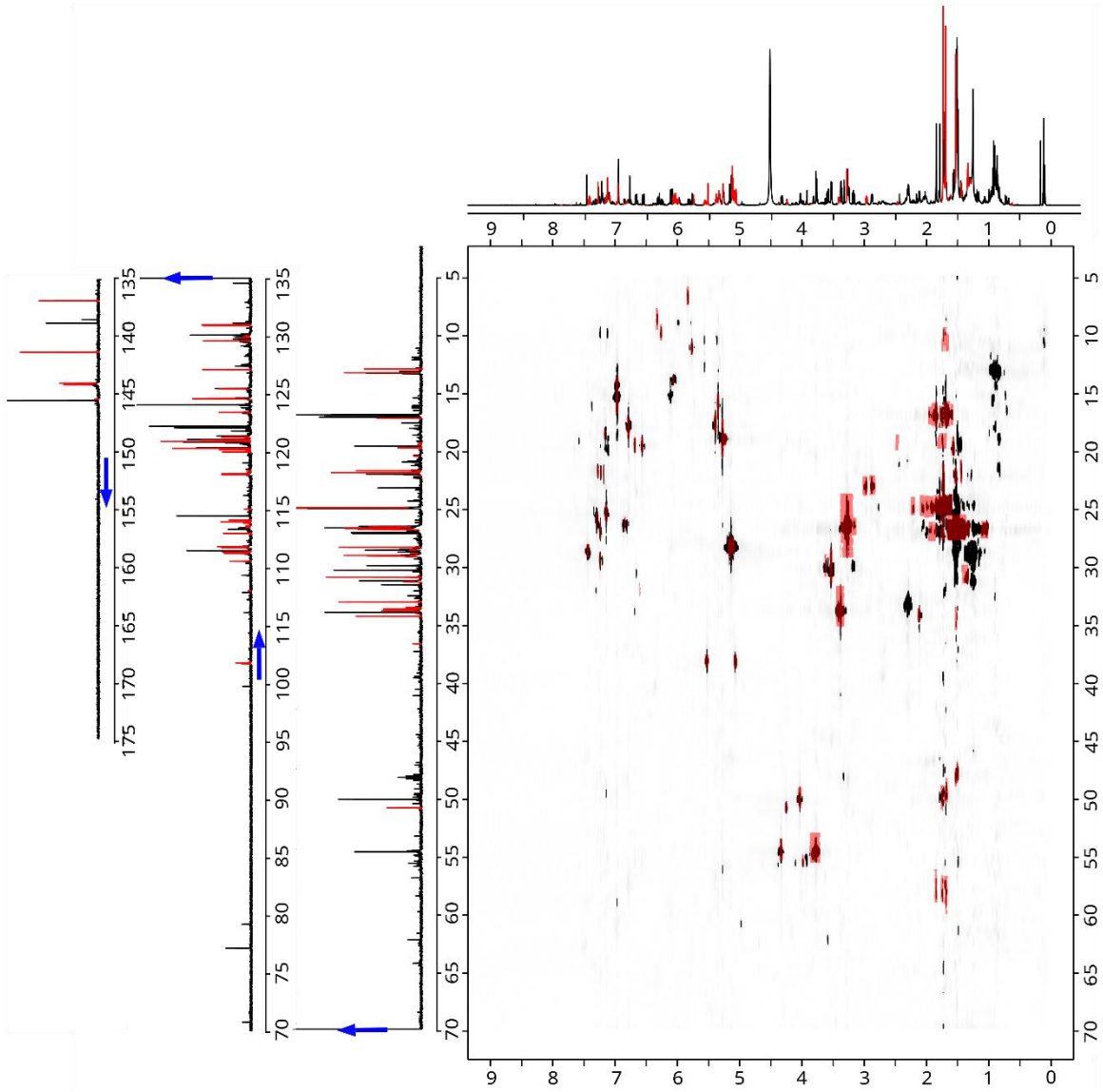

Figure SI 12 - SYMAPS HSQC with ^{1}H and numerically folded ^{13}C INEPT spectra as projections: on the top, 1D ^{1}H spectrum with their 100 first OPLS-DA VIP buckets in red, on the bottom left, 1D ^{13}C INEPT numerically folded spectrum with their 100 first OPLS-DA VIP buckets in red, on the bottom right, 2D SYMAPS HSQC spectrum with their 100 first OPLS-DA VIP buckets in red.

	F2											
	10 to 11 ppm	9 to 10 ppm	8 to 9 ppm	7 to 8 ppm	6 to 7 ppm	5 to 6 ppm	4 to 5 ppm	3 to 4 ppm	2 to 3 ppm	1 to 2 ppm	0 to 1 ppm	
13	0	0	0	2	6	4	0	0	0	1	0	0 to 10 ppm
79	0	0	0	4	19	11	0	0	6	29	10	10 to 20 ppm
103	0	0	0	11	6	8	0	31	13	28	6	20 to 30 ppm
75	0	0	0	0	4	2	0	10	7	45	7	30 to 40 ppm
54	0	0	0	1	0	0	1	4	0	43	5	40 to 50 ppm
56	0	0	0	0	0	1	4	3	0	43	5	50 to 60 ppm
61	3	0	0	2	0	0	1	1	0	54	0	60 to 70 ppm
	3	0	0	20	35	26	6	49	26	243	32	

F1

Min Max

Figure SI 13 - Spectral distribution of SYMAPS HSQC buckets. Color code corresponds to bucket density in the spectrum range, from low amount (blue), to medium (white) and high (red).

	F2											
	10 to 11 ppm	9 to 10 ppm	8 to 9 ppm	7 to 8 ppm	6 to 7 ppm	5 to 6 ppm	4 to 5 ppm	3 to 4 ppm	2 to 3 ppm	1 to 2 ppm	0 to 1 ppm	
3	0	0	0	0	3	2	0	0	0	0	0	0 to 10 ppm
26	0	0	0	2	10	8	0	0	3	7	0	10 to 20 ppm
47	0	0	0	7	1	4	0	3	5	10	2	20 to 30 ppm
9	0	0	0	0	1	2	0	0	2	9	0	30 to 40 ppm
6	0	0	0	0	0	0	0	1	0	4	2	40 to 50 ppm
7	0	0	0	0	0	0	0	0	0	4	2	50 to 60 ppm
2	1	0	0	0	0	0	0	0	0	5	0	60 to 70 ppm
	1	0	0	9	15	16	0	4	10	39	6	

F1

Min Max

Figure SI 14 - Spectral distribution of the first 100 VIP in SYMAPS HSQC. Color code corresponds to bucket density in the spectrum range, from low amount (blue), to medium (white) and high (red).

Table SI 1 - OPLS-DA model parameters for the four NMR methods according the tested parameter (medium, time effects): "Medium" lines are the OPLS-DA results for Control (T1 + T2) samples versus Sponge (T1 + T2). Other lines show the effect time on T1 or T2 samples only. "Time" lines are the OPLS-DA results for T1 (Control + Sponge) vs T2 (Control + Sponge). For this study, we concentrate only on the results obtained on Medium lines to compare NMR methods.

		OPLS-DA Models Parameters								
		p1			Permutation					
Analysis technique	**Studied factor**	**R2X**	**R2Y**	**Q2**	**n/2000**	**Q2**	**p**	**n/2000**	**R2Y**	**p**
^{1}H	**Medium**	**0.344**	**0.842**	**0.788**	**0**	**0.948**	**<5.10^{-4}**	**0**	**0.981**	**<5.10^{-4}**
^{1}H	Medium at T1	0.534	0.84	0.803	4	0.897	0.002	4	0.989	0.002
^{1}H	Medium at T2	0.533	0.973	0.936	47	0.984	0.0235	47	0.998	0.0235
^{1}H	**Time**	**0.192**	**0.838**	**0.74**	**0**	**0.954**	**<5.10^{-4}**	**0**	**0.983**	**<5.10^{-4}**
^{1}H	Time on Ctrl	0.583	0.91	0.877	10	0.958	0.005	10	0.995	0.005
^{1}H	Time on Sponge	0.339	0.98	0.885	6	0.964	0.003	6	0.991	0.003
UF COSY	**Medium**	**0.226**	**0.832**	**0.752**	**0**	**0.898**	**<5.10^{-4}**	**0**	**0.988**	**<5.10^{-4}**
UF COSY	Medium at T1	0.33	0.926	0.835	2	0.877	0.001	2	0.977	0.001
UF COSY	Medium at T2	0.349	0.898	0.783	60	0.874	0.03	60	0.997	0.03
UF COSY	**Time**	**0.147**	**0.844**	**0.722**	**0**	**0.909**	**<5.10^{-4}**	**0**	**0.984**	**<5.10^{-4}**
UF COSY	Time on Ctrl	0.278	0.947	0.812	8	0.854	0.004	13	0.99	0.0065
UF COSY	Time on Sponge	0.247	0.968	0.773	6	0.831	0.003	32	0.995	0.016
^{13}C	**Medium**	**0.236**	**0.757**	**0.644**	**0**	**0.877**	**<5.10^{-4}**	**12**	**0.991**	**0.006**
^{13}C	Medium at T1	0.357	0.76	0.676	7	0.802	0.0035	133	0.997	0.0665
^{13}C	Medium at T2	0.355	0.93	0.854	61	0.957	0.0305	61	1	0.0305
^{13}C	**Time**	**0.308**	**0.989**	**0.885**	**1**	**0.954**	**<5.10^{-4}**	**1**	**0.989**	**<5.10^{-4}**
^{13}C	Time on Ctrl	0.551	0.895	0.834	9	0.938	0.0045	9	0.998	0.0045
^{13}C	Time on Sponge	0.369	0.872	0.797	6	0.951	0.003	6	0.995	0.003
SYMAPS HSQC	**Medium**	**0.391**	**0.717**	**0.644**	**0**	**0.911**	**<5.10^{-4}**	**0**	**0.983**	**<5.10^{-4}**
SYMAPS HSQC	Medium at T1	0.6	0.871	0.839	7	0.885	0.0035	10	0.949	0.005
SYMAPS HSQC	Medium at T2	0.53	0.834	0.755	53	0.942	0.0265	53	0.998	0.0265
SYMAPS HSQC	**Time**	**0.225**	**0.582**	**0.5**	**0**	**0.962**	**<5.10^{-4}**	**0**	**0.995**	**<5.10^{-4}**
SYMAPS HSQC	Time on Ctrl	0.551	0.895	0.834	13	0.938	0.0065	13	0.998	0.0065
SYMAPS HSQC	Time on Sponge	0.369	0.872	0.797	4	0.967	0.002	4	0.992	0.002

Table SI 2 - 100 first ^{1}H VIP buckets from OPLS-DA (Part 1/3)

^{1}H VIP Ranking	Bucket	V1	V2	Center [ppm]	Min [ppm]	Max [ppm]	Potential correspondence in F1 in UF COSY	Potential correspondence in F2 in UF COSY	Potential correspondence in F2 in HSQC
1	B7_2997	1.651	0.239	7.300	7.293	7.306	Yes	Yes	Yes
2	B12_1959	1.626	0.182	12.196	12.187	12.205	No	No	No
3	B7_4380	1.611	0.439	7.438	7.431	7.445	Yes	Yes	Yes
4	B5_1073	1.593	0.467	5.107	5.103	5.112	Yes	Yes	Yes
5	B6_0605	1.592	0.467	6.060	6.058	6.063	Yes	Yes	Yes
6	B6_3701	1.591	0.391	6.370	6.368	6.372	No	Yes	No
7	B7_1593	1.587	0.497	7.159	7.154	7.165	Yes	Yes	Yes
8	B6_9771	1.585	0.479	6.977	6.973	6.981	Yes	Yes	No
9	B4_3958	1.579	0.377	4.396	4.393	4.399	No	No	No
10	B3_2907	1.577	0.506	3.291	3.279	3.302	Yes	Yes	Yes
11	B7_5404	1.572	0.352	7.540	7.536	7.545	No	No	No
12	B7_8954	1.569	0.406	7.895	7.889	7.902	No	No	No
13	B6_4305	1.566	0.438	6.430	6.429	6.432	No	Yes	No
14	B5_2933	1.561	0.522	5.293	5.285	5.301	Yes	Yes	Yes
15	B6_6041	1.559	0.419	6.604	6.602	6.606	Yes	Yes	Yes
16	B5_0751	1.559	0.515	5.075	5.071	5.079	Yes	Yes	Yes
17	B6_0410	1.557	0.556	6.041	6.034	6.048	Yes	Yes	Yes
18	B10_0666	1.555	0.526	10.067	10.058	10.075	No	No	Yes
19	B6_8944	1.554	0.450	6.894	6.889	6.900	No	Yes	No
20	B7_1698	1.553	0.560	7.170	7.165	7.175	Yes	Yes	Yes
21	B3_4237	1.551	0.537	3.424	3.414	3.433	Yes	Yes	No
22	B5_0607	1.551	0.512	5.061	5.050	5.071	Yes	Yes	Yes
23	B7_5636	1.550	0.046	7.564	7.559	7.569	No	No	No
24	B5_9981	1.546	0.488	5.998	5.995	6.001	Yes	Yes	Yes
25	B7_7377	1.540	0.406	7.738	7.736	7.739	No	No	No
26	B5_3480	1.538	0.587	5.348	5.342	5.354	Yes	Yes	No
27	B2_5900	1.537	0.446	2.590	2.584	2.596	No	Yes	No
28	B6_0304	1.535	0.550	6.030	6.027	6.034	Yes	Yes	Yes
29	B5_0959	1.535	0.593	5.096	5.089	5.103	Yes	Yes	Yes
30	B5_1211	1.532	0.595	5.121	5.115	5.127	Yes	Yes	Yes
31	B7_1798	1.529	0.609	7.180	7.175	7.185	Yes	Yes	Yes
32	B5_0812	1.528	0.449	5.081	5.079	5.083	Yes	Yes	Yes
33	B7_2179	1.528	0.510	7.218	7.216	7.220	Yes	Yes	No
34	B6_0852	1.526	0.592	6.085	6.082	6.088	Yes	Yes	Yes
35	B5_5211	1.525	0.562	5.521	5.511	5.531	Yes	Yes	Yes
36	B5_5333	1.524	0.529	5.533	5.531	5.535	Yes	Yes	Yes
37	B6_0683	1.519	0.621	6.068	6.063	6.073	Yes	Yes	Yes
38	B5_5422	1.519	0.596	5.542	5.540	5.545	Yes	Yes	Yes
39	B6_0061	1.517	0.605	6.006	6.001	6.011	Yes	Yes	Yes

40	B1_5327	1.515	0.596	1.533	1.527	1.538	Yes	Yes	Yes
41	B7_7332	1.515	0.548	7.733	7.730	7.736	No	No	No
42	B7_7459	1.513	0.596	7.746	7.743	7.748	No	No	No

Table SI 3 - 100 first ^{1}H VIP buckets from OPLS-DA (Part 2/3)

^{1}H VIP Ranking	Bucket	V1	V2	Center [ppm]	Min [ppm]	Max [ppm]	Potential correspondence in F1 in UF COSY	Potential correspondence in F2 in UF COSY	Potential correspondence in F2 in HSQC
43	B6_8274	1.512	0.466	6.827	6.822	6.833	No	Yes	Yes
44	B6_5057	1.511	0.439	6.506	6.504	6.507	Yes	Yes	No
45	B2_4699	1.505	0.626	2.470	2.459	2.481	Yes	Yes	Yes
46	B5_2790	1.501	0.607	5.279	5.273	5.285	Yes	Yes	Yes
47	B1_3410	1.500	0.642	1.341	1.338	1.344	Yes	Yes	Yes
48	B1_3349	1.500	0.635	1.335	1.332	1.338	Yes	Yes	Yes
49	B7_1063	1.499	0.651	7.106	7.101	7.112	No	Yes	Yes
50	B5_1325	1.499	0.643	5.132	5.127	5.138	Yes	Yes	Yes
51	B5_5699	1.499	0.631	5.570	5.564	5.576	Yes	Yes	No
52	B5_3620	1.498	0.642	5.362	5.354	5.370	Yes	Yes	No
53	B5_5806	1.497	0.629	5.581	5.576	5.586	Yes	Yes	No
54	B-0_3424	1.495	0.552	-0.342	-0.346	-0.339	No	No	No
55	B7_0661	1.491	0.608	7.066	7.061	7.072	No	Yes	No
56	B5_4040	1.490	0.664	5.404	5.398	5.410	Yes	Yes	No
57	B7_4522	1.487	0.648	7.452	7.445	7.459	Yes	Yes	Yes
58	B5_2158	1.486	0.668	5.216	5.213	5.219	Yes	Yes	No
59	B10_0452	1.486	0.642	10.045	10.039	10.051	No	No	Yes
60	B5_5595	1.485	0.611	5.560	5.555	5.564	Yes	Yes	No
61	B1_7432	1.485	0.662	1.743	1.727	1.759	Yes	Yes	Yes
62	B10_1628	1.481	0.634	10.163	10.136	10.189	No	No	No
63	B5_5498	1.479	0.624	5.550	5.545	5.555	Yes	Yes	Yes
64	B7_4231	1.477	0.689	7.423	7.415	7.431	Yes	Yes	No
65	B1_5253	1.471	0.673	1.525	1.523	1.527	Yes	Yes	Yes
66	B7_8832	1.471	0.433	7.883	7.878	7.889	No	No	No
67	B1_6921	1.470	0.663	1.692	1.685	1.700	Yes	Yes	Yes
68	B2_9812	1.469	0.673	2.981	2.975	2.987	Yes	Yes	Yes
69	B5_0862	1.468	0.670	5.086	5.083	5.089	Yes	Yes	Yes
70	B6_0546	1.468	0.703	6.055	6.051	6.058	Yes	Yes	Yes
71	B1_3268	1.467	0.677	1.327	1.322	1.332	Yes	Yes	Yes
72	B6_8366	1.465	0.485	6.837	6.833	6.840	No	No	Yes
73	B6_0793	1.461	0.698	6.079	6.076	6.082	Yes	Yes	Yes
74	B6_9982	1.460	0.667	6.998	6.989	7.008	Yes	Yes	No
75	B2_9691	1.459	0.677	2.969	2.963	2.975	Yes	Yes	Yes
76	B-0_3297	1.459	0.531	-0.330	-0.339	-0.320	No	No	No
77	B5_7574	1.458	0.674	5.757	5.752	5.762	Yes	Yes	Yes
78	B3_4054	1.456	0.663	3.405	3.397	3.414	Yes	Yes	No
79	B4_2482	1.455	0.696	4.248	4.243	4.254	Yes	Yes	No
80	B6_8452	1.455	0.523	6.845	6.840	6.850	No	No	Yes
81	B5_5068	1.454	0.646	5.507	5.503	5.511	Yes	Yes	No

82	B1_4388	1.453	0.703	1.439	1.435	1.442	Yes	Yes	Yes
83	B11_8978	1.453	0.391	11.898	11.886	11.909	No	No	No
84	B11_9453	1.453	0.398	11.945	11.935	11.955	No	No	No

Table SI 4 - 100 first ^{1}H VIP buckets from OPLS-DA (Part 3/3)

^{1}H VIP Ranking	Bucket	V1	V2	Center [ppm]	Min [ppm]	Max [ppm]	Potential correspondence in F1 in UF COSY	Potential correspondence in F2 in UF COSY	Potential correspondence in F2 in HSQC
85	B5_5376	1.452	0.619	5.538	5.535	5.540	Yes	Yes	Yes
86	B0_6249	1.452	0.677	0.625	0.614	0.636	No	No	No
87	B5_3913	1.451	0.727	5.391	5.385	5.398	Yes	Yes	No
88	B6_6852	1.451	0.595	6.685	6.682	6.689	Yes	Yes	Yes
89	B1_6594	1.450	0.670	1.659	1.653	1.666	Yes	Yes	Yes
90	B1_3470	1.448	0.716	1.347	1.344	1.350	Yes	Yes	Yes
91	B3_8614	1.448	0.617	3.861	3.857	3.865	No	Yes	No
92	B1_7036	1.447	0.722	1.704	1.700	1.708	Yes	Yes	Yes
93	B1_2969	1.445	0.711	1.297	1.292	1.302	No	Yes	Yes
94	B1_6109	1.445	0.734	1.611	1.605	1.617	No	Yes	Yes
95	B7_1467	1.443	0.724	7.147	7.140	7.154	Yes	Yes	No
96	B6_8849	1.443	0.640	6.885	6.881	6.889	No	No	No
97	B5_5910	1.442	0.709	5.591	5.586	5.596	Yes	Yes	No
98	B4_7440	1.441	0.650	4.744	4.740	4.748	No	No	No
99	B1_2871	1.439	0.722	1.287	1.282	1.292	No	Yes	Yes
100	B5_4208	1.439	0.737	5.421	5.417	5.425	Yes	Yes	No

Table SI 5 - 100 first ^{13}C VIP buckets from OPLS-DA (Part 1/3)

^{13}C VIP Ranking	Bucket	V1	V2	Center [ppm]	Min [ppm]	Max [ppm]	Potential Correspondence in F1
1	B113_7829	1.821	0.568	113.783	113.748	113.818	Yes
2	B119_9937	1.806	0.654	119.994	119.962	120.026	Yes
3	B143_7794	1.804	0.672	143.779	143.757	143.801	Yes
4	B107_8090	1.795	0.614	107.809	107.782	107.836	Yes
5	B111_5956	1.792	0.585	111.596	111.572	111.620	Yes
6	B28_9028	1.789	0.547	28.903	28.877	28.929	Yes
7	B129_6858	1.763	0.637	129.686	129.670	129.701	Yes
8	B117_8638	1.762	0.717	117.864	117.825	117.903	Yes
9	B111_1731	1.761	0.715	111.173	111.144	111.203	Yes
10	B28_8019	1.749	0.674	28.802	28.782	28.822	Yes
11	B129_8164	1.746	0.642	129.816	129.789	129.844	Yes
12	B118_1131	1.741	0.419	118.113	118.090	118.136	Yes
13	B113_7300	1.723	0.279	113.730	113.712	113.748	Yes
14	B143_8644	1.710	0.734	143.864	143.854	143.875	Yes
15	B27_5577	1.704	0.603	27.558	27.541	27.575	Yes
16	B117_9304	1.699	0.754	117.930	117.903	117.958	Yes
17	B20_3724	1.699	0.608	20.372	20.356	20.389	Yes
18	B101_6241	1.698	0.681	101.624	101.576	101.672	Yes
19	B113_6743	1.697	0.583	113.674	113.637	113.712	Yes
20	B13_2898	1.692	0.750	13.290	13.246	13.334	Yes
21	B26_6971	1.681	0.781	26.697	26.679	26.716	Yes
22	B28_8418	1.672	0.740	28.842	28.822	28.862	Yes
23	B36_5855	1.671	0.661	36.585	36.567	36.604	Yes
24	B112_7543	1.667	0.772	112.754	112.686	112.823	Yes
25	B33_5858	1.665	0.829	33.586	33.562	33.609	Yes
26	B70_5963	1.662	0.685	70.596	70.578	70.614	No
27	B111_6413	1.657	0.823	111.641	111.620	111.663	Yes
28	B32_9499	1.650	0.820	32.950	32.893	33.007	Yes
29	B28_5689	1.640	0.766	28.569	28.540	28.598	Yes
30	B113_4295	1.638	0.356	113.430	113.395	113.464	Yes
31	B30_8279	1.635	0.857	30.828	30.783	30.872	Yes
32	B31_2518	1.628	0.813	31.252	31.195	31.309	Yes
33	B123_2084	1.625	0.805	123.208	123.173	123.244	Yes
34	B19_2220	1.623	0.617	19.222	19.206	19.239	Yes
35	B26_6268	1.622	0.805	26.627	26.608	26.646	Yes
36	B34_1764	1.605	0.843	34.176	34.122	34.231	Yes
37	B26_7396	1.605	0.823	26.740	26.716	26.763	Yes
38	B28_6188	1.584	0.860	28.619	28.598	28.639	Yes
39	B129_3957	1.583	0.885	129.396	129.353	129.438	Yes
40	B113_8888	1.583	0.633	113.889	113.860	113.917	Yes
41	B120_8366	1.582	0.878	120.837	120.803	120.870	Yes
42	B136_6586	1.581	0.863	136.659	136.601	136.717	Yes

Table SI 6 - 100 first ^{13}C VIP buckets from OPLS-DA (Part 2/3)

^{13}C VIP Ranking	Bucket	V1	V2	Center [ppm]	Min [ppm]	Max [ppm]	Potential Correspondence in F1
43	B141_0771	1.572	0.905	141.077	141.021	141.134	Yes
44	B18_0579	1.566	0.707	18.058	18.017	18.099	Yes
45	B28_4757	1.566	0.888	28.476	28.461	28.490	Yes
46	B126_9110	1.566	0.825	126.911	126.863	126.959	No
47	B120_1204	1.562	0.899	120.120	120.088	120.152	Yes
48	B13_1803	1.561	0.909	13.180	13.155	13.206	No
49	B119_7879	1.561	0.867	119.788	119.730	119.846	Yes
50	B118_1616	1.560	0.588	118.162	118.136	118.187	Yes
51	B59_6992	1.559	0.289	59.699	59.677	59.721	Yes
52	B124_3739	1.555	0.891	124.374	124.324	124.424	No
53	B28_2444	1.552	0.914	28.244	28.208	28.280	Yes
54	B28_9483	1.550	0.804	28.948	28.929	28.968	Yes
55	B19_3796	1.527	0.715	19.380	19.354	19.405	Yes
56	B21_9970	1.526	0.889	21.997	21.967	22.027	Yes
57	B21_7945	1.525	0.928	21.794	21.758	21.831	Yes
58	B145_1500	1.522	0.520	145.150	145.109	145.191	No
59	B110_3815	1.522	0.878	110.381	110.352	110.411	Yes
60	B125_2562	1.519	0.781	125.256	125.231	125.282	Yes
61	B113_9521	1.510	0.756	113.952	113.917	113.987	Yes
62	B28_6892	1.509	0.857	28.689	28.658	28.721	Yes
63	B24_8313	1.508	0.920	24.831	24.808	24.855	Yes
64	B111_5450	1.505	0.965	111.545	111.518	111.572	Yes
65	B12_8547	1.491	0.926	12.855	12.809	12.901	No
66	B130_7097	1.489	0.888	130.710	130.674	130.746	Yes
67	B143_8842	1.488	0.974	143.884	143.875	143.894	Yes
68	B111_0725	1.479	0.958	111.073	111.043	111.102	Yes
69	B121_0437	1.472	0.930	121.044	120.999	121.088	Yes
70	B111_1227	1.470	0.786	111.123	111.102	111.144	Yes
71	B113_3233	1.467	0.737	113.323	113.291	113.355	Yes
72	B50_7373	1.466	0.947	50.737	50.675	50.799	Yes
73	B33_4582	1.464	0.991	33.458	33.432	33.485	Yes
74	B44_4838	1.457	0.263	44.484	44.467	44.501	No
75	B33_7496	1.456	0.968	33.750	33.719	33.780	Yes
76	B101_5378	1.446	0.600	101.538	101.500	101.576	Yes
77	B26_4658	1.446	0.869	26.466	26.455	26.477	Yes
78	B22_0804	1.440	0.735	22.080	22.056	22.105	Yes
79	B29_0139	1.435	1.001	29.014	28.968	29.060	Yes
80	B119_8680	1.429	0.687	119.868	119.846	119.890	Yes
81	B130_7794	1.426	0.956	130.779	130.746	130.813	Yes
82	B124_4408	1.424	1.018	124.441	124.424	124.458	No
83	B19_6859	1.421	0.998	19.686	19.622	19.750	Yes
84	B17_1152	1.420	0.957	17.115	17.085	17.146	Yes

Table SI 7 - 100 first ^{13}C VIP buckets from OPLS-DA (Part 3/3)

^{13}C VIP Ranking	Bucket	V1	V2	Center [ppm]	Min [ppm]	Max [ppm]	Potential Correspondence in F1
85	B21_6387	1.418	1.023	21.639	21.613	21.665	Yes
86	B143_8394	1.415	0.959	143.839	143.825	143.854	Yes
87	B28_6485	1.413	0.969	28.649	28.639	28.658	Yes
88	B120_7122	1.399	1.012	120.712	120.679	120.745	Yes
89	B121_1191	1.388	0.946	121.119	121.088	121.150	Yes
90	B29_0763	1.371	1.042	29.076	29.060	29.093	Yes
91	B114_8669	1.370	0.623	114.867	114.833	114.900	Yes
92	B58_1908	1.366	0.444	58.191	58.171	58.211	Yes
93	B26_6623	1.348	1.046	26.662	26.646	26.679	Yes
94	B125_2064	1.334	0.663	125.206	125.182	125.231	Yes
95	B27_2848	1.326	0.961	27.285	27.249	27.321	Yes
96	B38_7969	1.322	0.618	38.797	38.751	38.843	Yes
97	B26_5584	1.316	1.088	26.558	26.521	26.596	Yes
98	B55_6512	1.312	0.886	55.651	55.628	55.674	No
99	B26_4991	1.304	1.060	26.499	26.477	26.521	Yes
100	B29_2522	1.290	1.001	29.252	29.196	29.308	Yes

Table SI 8 - 100 first UF COSY VIP buckets from OPLS-DA (Part 1/3)

UF COSY VIP Ranking	Nom	V1	V2	Center F1 [ppm]	Start ^{1}H F1 [ppm]	End ^{1}H F1 [ppm]	Center F2 [ppm]	Start ^{1}H F2 [ppm]	End ^{1}H F2 [ppm]	Potential correspondence in F1	Potential correspondence in F2
1	B_1013	2.022	0.448	7.447	7.455	7.434	7.456	7.541	7.370	Yes	Yes
2	B_0998	1.987	0.550	7.145	7.184	7.114	7.434	7.526	7.334	Yes	Yes
3	B_1007	1.960	0.580	7.437	7.477	7.400	7.176	7.266	7.093	Yes	Yes
4	B_0016	1.944	0.728	1.338	1.368	1.307	1.335	1.405	1.261	Yes	Yes
5	B_1004	1.940	0.609	7.279	7.302	7.256	7.084	7.148	7.028	Yes	Yes
6	B_0461	1.934	0.666	5.061	5.095	5.035	5.088	5.176	4.996	Yes	Yes
7	B_0981	1.933	0.658	7.016	7.035	6.999	7.014	7.119	6.902	Yes	Yes
8	B_0627	1.919	0.700	1.705	1.733	1.675	5.644	5.793	5.493	Yes	Yes
9	B_0469	1.918	0.788	5.764	5.810	5.715	6.625	6.804	6.439	Yes	Yes
10	B_1003	1.899	0.738	7.224	7.249	7.201	7.084	7.151	7.014	Yes	Yes
11	B_0542	1.897	0.690	6.071	6.087	6.052	6.086	6.172	6.002	Yes	Yes
12	B_0468	1.894	0.848	5.561	5.609	5.514	5.973	6.126	5.818	Yes	Yes
13	B_0486	1.891	0.422	5.779	5.793	5.760	6.372	6.425	6.323	Yes	Yes
14	B_0526	1.888	0.780	6.051	6.065	6.037	5.132	5.261	4.993	Yes	Yes
15	B_0956	1.885	0.671	6.591	6.602	6.572	6.367	6.443	6.291	Yes	Yes
16	B_0224	1.883	0.398	1.081	1.107	1.054	2.532	2.617	2.448	No	Yes
17	B_0454	1.872	0.795	5.521	5.547	5.496	5.536	5.671	5.396	Yes	Yes
18	B_0148	1.872	0.917	3.383	3.445	3.323	5.358	5.490	5.218	Yes	Yes
19	B_0459	1.863	0.847	5.259	5.313	5.206	5.304	5.415	5.189	Yes	Yes
20	B_0214	1.856	0.867	1.556	1.589	1.529	4.269	4.377	4.152	Yes	Yes
21	B_0127	1.855	0.662	2.284	2.313	2.259	2.090	2.163	2.022	No	No
22	B_0980	1.854	0.868	6.967	6.992	6.938	6.992	7.104	6.880	Yes	Yes
23	B_0473	1.854	0.943	5.140	5.197	5.079	6.108	6.228	5.986	Yes	Yes
24	B_0477	1.853	0.816	5.086	5.099	5.073	5.822	5.871	5.773	Yes	No
25	B_0633	1.853	0.384	2.284	2.320	2.252	5.968	6.024	5.912	No	Yes
26	B_0223	1.851	0.352	1.150	1.166	1.127	2.575	2.667	2.485	No	Yes
27	B_0147	1.847	0.934	3.249	3.319	3.184	5.283	5.424	5.140	Yes	Yes
28	B_0962	1.847	0.919	6.556	6.601	6.503	5.768	5.894	5.643	Yes	Yes
29	B_0463	1.846	0.728	5.091	5.118	5.065	5.369	5.452	5.284	Yes	Yes
30	B_0502	1.844	0.965	5.987	6.017	5.956	5.558	5.658	5.465	Yes	Yes
31	B_1006	1.842	0.599	7.284	7.302	7.267	7.176	7.211	7.149	Yes	Yes
32	B_0991	1.839	0.725	7.180	7.201	7.156	6.976	7.046	6.901	Yes	Yes
33	B_0033	1.836	0.892	2.289	2.317	2.258	1.496	1.572	1.429	No	Yes
34	B_0263	1.833	0.967	4.249	4.294	4.200	1.540	1.636	1.446	Yes	Yes
35	B_0593	1.833	0.983	4.244	4.288	4.198	4.258	4.344	4.175	Yes	Yes
36	B_0226	1.825	0.387	1.046	1.082	1.008	2.235	2.324	2.154	No	No
37	B_0017	1.825	0.935	1.338	1.370	1.307	0.898	1.009	0.789	Yes	No
38	B_0023	1.812	1.001	1.437	1.478	1.398	0.909	0.989	0.838	Yes	No
39	B_0082	1.810	0.958	2.314	2.360	2.266	2.947	3.035	2.865	No	Yes
40	B_0476	1.802	0.676	5.130	5.145	5.109	5.800	5.841	5.763	Yes	No
41	B_1023	1.801	0.747	8.244	8.261	8.225	8.233	8.292	8.172	No	No
42	B_0549	1.800	0.945	6.284	6.306	6.255	5.854	5.972	5.743	No	Yes

Table SI 9- 100 first UF COSY VIP buckets from OPLS-DA (Part 2/3)

UF COSY VIP Ranking	Nom	V1	V2	Center F1 [ppm]	Start ^{1}H F1 [ppm]	End ^{1}H F1 [ppm]	Center F2 [ppm]	Start ^{1}H F2 [ppm]	End ^{1}H F2 [ppm]	Potential correspondence in F1	Potential correspondence in F2
43	B_0081	1.799	0.979	2.962	3.002	2.925	2.305	2.400	2.210	Yes	No
44	B_0528	1.796	0.760	5.987	5.992	5.979	5.110	5.225	4.996	No	Yes
45	B_0458	1.792	1.022	5.378	5.435	5.316	5.385	5.497	5.281	Yes	Yes
46	B_0442	1.790	0.782	5.650	5.673	5.630	5.094	5.147	5.042	No	Yes
47	B_0026	1.786	0.990	1.734	1.758	1.713	1.723	1.861	1.588	Yes	Yes
48	B_0781	1.785	0.975	5.784	5.808	5.762	2.300	2.396	2.199	Yes	No
49	B_0103	1.784	0.854	2.962	3.001	2.927	2.473	2.539	2.406	Yes	Yes
50	B_0748	1.784	0.486	7.289	7.311	7.271	1.049	1.122	0.974	Yes	No
51	B_0965	1.779	0.479	6.596	6.619	6.573	6.620	6.731	6.504	Yes	Yes
52	B_0783	1.777	0.572	5.804	5.826	5.788	2.090	2.180	1.997	No	No
53	B_0585	1.776	0.823	4.536	4.641	4.429	4.549	4.679	4.413	No	No
54	B_0957	1.770	1.047	6.680	6.721	6.637	6.340	6.482	6.197	No	Yes
55	B_0028	1.768	0.994	1.452	1.507	1.395	2.095	2.182	2.012	Yes	No
56	B_0699	1.768	0.722	6.957	6.985	6.937	0.725	0.804	0.652	Yes	Yes
57	B_0241	1.768	1.016	3.259	3.309	3.208	1.718	1.839	1.599	Yes	Yes
58	B_0175	1.765	0.671	3.279	3.296	3.255	3.783	3.862	3.701	Yes	Yes
59	B_0630	1.764	0.747	1.700	1.748	1.654	5.299	5.416	5.181	Yes	Yes
60	B_0771	1.764	1.018	5.269	5.310	5.224	1.718	1.833	1.595	Yes	Yes
61	B_0472	1.757	0.709	5.794	5.815	5.770	5.962	6.022	5.900	No	Yes
62	B_0894	1.753	0.588	5.650	5.678	5.627	8.346	8.415	8.277	No	No
63	B_0539	1.750	0.726	6.036	6.050	6.020	5.833	5.900	5.763	Yes	No
64	B_0516	1.748	0.738	6.041	6.052	6.028	5.385	5.468	5.298	Yes	Yes
65	B_0029	1.746	1.000	1.482	1.529	1.442	2.305	2.392	2.209	Yes	No
66	B_0138	1.745	0.641	2.531	2.564	2.498	1.076	1.141	1.009	No	No
67	B_1005	1.744	0.731	7.294	7.332	7.264	7.332	7.441	7.218	Yes	Yes
68	B_0200	1.735	0.956	3.279	3.303	3.256	3.271	3.392	3.149	Yes	Yes
69	B_0128	1.734	0.841	2.121	2.148	2.093	1.928	1.986	1.872	No	No
70	B_0481	1.733	0.495	5.051	5.072	5.033	6.038	6.133	5.946	Yes	Yes
71	B_0151	1.730	0.918	3.596	3.612	3.574	5.407	5.474	5.346	No	Yes
72	B_0525	1.728	1.137	6.091	6.112	6.068	5.148	5.261	5.029	Yes	Yes
73	B_0538	1.727	0.447	6.036	6.046	6.017	6.021	6.126	5.917	Yes	Yes
74	B_0953	1.727	0.924	6.333	6.352	6.318	6.162	6.229	6.097	No	No
75	B_0201	1.725	0.526	3.323	3.342	3.306	3.325	3.463	3.182	No	Yes
76	B_0555	1.721	0.757	6.393	6.411	6.378	6.307	6.343	6.267	No	No
77	B_0598	1.720	1.062	4.620	4.641	4.598	5.461	5.540	5.383	No	Yes
78	B_0004	1.720	1.150	1.502	1.560	1.447	1.523	1.658	1.389	Yes	Yes
79	B_0084	1.719	0.712	2.477	2.501	2.454	2.467	2.559	2.378	Yes	Yes
80	B_0518	1.716	1.005	6.140	6.156	6.117	5.412	5.510	5.311	No	Yes
81	B_0443	1.709	0.767	5.650	5.677	5.630	4.975	5.035	4.908	No	No
82	B_0487	1.703	1.099	5.843	5.884	5.802	6.307	6.408	6.208	No	Yes
83	B_0898	1.701	0.794	5.873	5.918	5.823	8.556	8.624	8.490	No	No
84	B_0434	1.697	1.071	5.432	5.458	5.409	4.630	4.727	4.534	Yes	No

Table SI 10 - 100 first UF COSY VIP buckets from OPLS-DA (Part 3/3)

UF COSY VIP Ranking	Nom	V1	V2	Center F1 [ppm]	Start ^{1}H F1 [ppm]	End ^{1}H F1 [ppm]	Center F2 [ppm]	Start ^{1}H F2 [ppm]	End ^{1}H F2 [ppm]	Potential correspondence in F1	Potential correspondence in F2
85	B_0268	1.691	0.632	4.422	4.444	4.400	2.063	2.127	1.995	Yes	No
86	B_0203	1.691	0.641	3.427	3.443	3.404	3.406	3.507	3.306	Yes	Yes
87	B_0503	1.688	0.849	6.036	6.050	6.019	5.558	5.622	5.488	Yes	Yes
88	B_0635	1.687	0.901	2.304	2.328	2.280	5.601	5.689	5.512	No	Yes
89	B_0982	1.687	0.422	7.036	7.049	7.026	7.165	7.197	7.129	No	Yes
90	B_0895	1.685	0.816	5.705	5.722	5.681	8.394	8.444	8.338	No	No
91	B_1002	1.682	1.136	7.234	7.262	7.212	7.257	7.357	7.156	Yes	Yes
92	B_0500	1.682	0.974	5.888	5.916	5.859	5.887	5.943	5.828	No	No
93	B_0043	1.681	0.809	1.507	1.537	1.475	1.324	1.368	1.279	Yes	Yes
94	B_0269	1.680	0.982	4.229	4.261	4.200	1.189	1.240	1.136	Yes	No
95	B_0035	1.679	1.163	2.116	2.147	2.081	1.448	1.518	1.372	No	Yes
96	B_0784	1.678	1.038	5.853	5.874	5.829	2.117	2.238	2.002	No	No
97	B_0018	1.677	1.004	1.338	1.356	1.316	1.486	1.533	1.437	Yes	Yes
98	B_1019	1.676	0.472	7.482	7.498	7.466	7.364	7.386	7.348	No	No
99	B_0149	1.676	1.231	3.536	3.568	3.504	5.401	5.546	5.257	No	Yes
100	B_0546	1.671	0.939	6.091	6.114	6.074	5.881	5.923	5.835	Yes	No

Table SI 11 - 100 first SYMAPS HSQC VIP buckets from OPLS-DA (Part 1/3)

HSQC VIP Ranking	Bucket	V1	V2	Center F1 [ppm]	Start ^{13}C F1 [ppm]	End ^{13}C F1 [ppm]	Center F2 [ppm]	Start ^{1}H F2 [ppm]	End ^{1}H F2 [ppm]	Potential correspondence in ^{13}C folded	Potential correspondence in ^{1}H
1	B338	1.527	0.355	19.360	19.762	18.978	6.605	6.607	6.601	Yes	Yes
2	B352	1.491	0.424	25.928	26.622	25.282	7.301	7.307	7.294	Yes	Yes
3	B411	1.488	0.429	21.708	22.239	21.215	7.276	7.285	7.267	Yes	Yes
4	B355	1.485	0.414	18.504	18.978	17.998	7.161	7.168	7.154	Yes	Yes
5	B290	1.481	0.439	38.111	38.737	37.520	5.073	5.092	5.055	Yes	Yes
6	B291	1.478	0.432	38.111	38.694	37.477	5.531	5.551	5.511	Yes	Yes
7	B353	1.475	0.398	25.864	26.524	25.217	7.287	7.293	7.282	Yes	Yes
8	B413	1.469	0.361	21.772	22.300	21.281	7.185	7.190	7.179	Yes	Yes
9	B35	1.458	0.434	19.202	19.885	18.517	2.472	2.495	2.448	Yes	Yes
10	B327	1.458	0.598	8.541	9.440	7.611	6.326	6.339	6.313	Yes	No
11	B20	1.454	0.622	33.130	34.352	31.932	2.304	2.362	2.246	Yes	No
12	B3	1.454	0.571	16.759	17.844	15.684	1.679	1.731	1.628	Yes	Yes
13	B326	1.454	0.568	9.715	10.387	9.015	6.263	6.276	6.250	Yes	No
14	B350	1.453	0.443	29.259	29.921	28.647	7.245	7.253	7.237	Yes	No
15	B65	1.453	0.528	16.790	17.709	15.846	1.598	1.619	1.576	Yes	Yes
16	B313	1.447	0.520	8.858	9.375	8.297	6.001	6.008	5.994	Yes	Yes
17	B314	1.446	0.601	8.890	9.407	8.362	5.979	5.988	5.970	Yes	No
18	B357	1.444	0.603	28.561	29.137	27.961	7.440	7.456	7.424	Yes	Yes
19	B296	1.440	0.551	28.022	28.774	27.319	5.129	5.135	5.123	Yes	Yes
20	B200	1.438	0.302	36.525	37.157	35.923	1.353	1.360	1.346	Yes	Yes
21	B312	1.437	0.557	13.840	14.503	13.196	6.019	6.031	6.008	Yes	Yes
22	B21	1.430	0.633	34.050	34.668	33.458	2.120	2.136	2.105	Yes	No
23	B6	1.426	0.603	19.075	19.745	18.415	1.752	1.823	1.681	Yes	Yes
24	B117	1.425	0.594	34.685	35.088	34.243	1.475	1.484	1.465	No	No
25	B421	1.424	0.201	53.118	53.881	52.340	0.831	0.836	0.826	No	No
26	B331	1.421	0.636	19.424	20.121	18.717	6.685	6.696	6.674	Yes	Yes
27	B337	1.421	0.631	19.424	19.860	18.978	6.590	6.595	6.583	Yes	Yes
28	B138	1.417	0.565	50.929	51.543	50.297	1.690	1.698	1.683	Yes	Yes
29	B142	1.417	0.651	57.750	58.960	56.529	1.752	1.770	1.733	Yes	Yes
30	B339	1.413	0.588	31.893	32.501	31.260	6.606	6.616	6.596	Yes	Yes
31	B38	1.412	0.705	24.659	25.567	23.725	2.070	2.106	2.033	Yes	No
32	B51	1.407	0.688	16.886	17.628	16.143	1.947	1.971	1.922	Yes	No
33	B214	1.400	0.471	66.190	66.720	65.680	1.536	1.538	1.533	No	Yes
34	B311	1.397	0.689	13.776	14.307	13.262	6.043	6.053	6.032	Yes	Yes
35	B134	1.394	0.653	49.882	51.045	48.739	1.754	1.772	1.735	Yes	Yes
36	B58	1.390	0.661	21.740	22.757	20.678	1.445	1.460	1.431	Yes	Yes
37	B15	1.386	0.721	30.560	31.353	29.775	1.378	1.433	1.323	Yes	Yes
38	B304	1.384	0.711	13.808	14.283	13.363	6.066	6.078	6.055	Yes	Yes
39	B317	1.383	0.714	11.111	11.563	10.649	5.805	5.811	5.799	Yes	No
40	B23	1.382	0.732	22.977	23.778	22.147	2.987	3.021	2.952	No	Yes
41	B423	1.382	0.609	59.146	59.904	58.409	0.913	0.918	0.908	Yes	No
42	B201	1.379	0.683	47.947	48.335	47.564	1.341	1.346	1.335	No	Yes

Table SI 12 - 100 first SYMAPS HSQC buckets from OPLS-DA (Part 2/3)

HSQC VIP Ranking	Bucket	V1	V2	Center F1 [ppm]	Start ^{13}C F1 [ppm]	End ^{13}C F1 [ppm]	Center F2 [ppm]	Start ^{1}H F2 [ppm]	End ^{1}H F2 [ppm]	Potential correspondence in ^{13}C folded	Potential correspondence in ^{1}H
43	B14	1.373	0.747	28.720	29.512	27.934	1.353	1.406	1.299	Yes	Yes
44	B365	1.372	0.757	66.317	67.517	65.100	10.053	10.115	9.989	No	Yes
45	B318	1.370	0.707	11.175	11.563	10.812	5.793	5.798	5.788	Yes	No
46	B433	1.369	0.612	20.661	21.431	19.890	0.947	0.955	0.938	Yes	No
47	B300	1.366	0.757	18.789	20.400	17.193	5.280	5.299	5.261	Yes	Yes
48	B224	1.364	0.742	37.572	38.082	37.041	1.275	1.286	1.265	Yes	Yes
49	B310	1.364	0.755	13.808	14.438	13.196	6.089	6.097	6.081	Yes	Yes
50	B330	1.361	0.764	19.392	20.056	18.717	6.567	6.582	6.551	Yes	No
51	B334	1.361	0.723	19.551	19.991	19.076	6.526	6.532	6.519	Yes	No
52	B42	1.360	0.723	24.722	25.514	23.936	2.221	2.252	2.189	Yes	No
53	B315	1.360	0.726	6.542	7.349	5.749	5.833	5.846	5.821	Yes	No
54	B111	1.359	0.729	38.587	39.097	38.105	1.508	1.513	1.503	Yes	No
55	B380	1.357	0.694	25.293	26.046	24.544	2.973	2.983	2.962	Yes	Yes
56	B238	1.355	0.643	39.793	40.510	39.084	1.368	1.377	1.359	No	No
57	B120	1.354	0.716	22.121	22.768	21.445	1.534	1.560	1.508	Yes	Yes
58	B275	1.354	0.320	30.592	31.311	29.923	1.733	1.737	1.729	Yes	Yes
59	B141	1.351	0.751	58.258	59.895	56.592	1.688	1.712	1.665	Yes	Yes
60	B54	1.348	0.784	16.981	17.979	16.008	1.760	1.787	1.733	Yes	Yes
61	B299	1.348	0.761	28.212	28.507	27.913	5.078	5.088	5.069	Yes	Yes
62	B356	1.345	0.772	19.836	20.285	19.337	7.108	7.113	7.103	Yes	Yes
63	B303	1.344	0.784	11.016	11.640	10.393	5.768	5.779	5.758	Yes	No
64	B60	1.339	0.780	24.722	25.349	24.107	1.321	1.333	1.309	Yes	Yes
65	B414	1.338	0.631	43.378	43.982	42.768	0.902	0.907	0.896	No	No
66	B267	1.336	0.770	53.784	54.360	53.199	1.705	1.713	1.696	No	Yes
67	B298	1.326	0.817	28.180	28.804	27.557	5.101	5.111	5.090	Yes	Yes
68	B39	1.325	0.691	26.975	27.724	26.198	2.086	2.120	2.052	Yes	No
69	B46	1.325	0.764	16.790	17.466	16.143	2.031	2.055	2.008	Yes	No
70	B13	1.323	0.849	24.595	25.429	23.746	1.735	1.875	1.594	Yes	Yes
71	B370	1.323	0.667	48.105	49.010	47.231	3.271	3.281	3.261	No	Yes
72	B336	1.321	0.627	19.614	20.056	19.141	6.544	6.549	6.538	Yes	No
73	B405	1.318	0.745	26.911	27.430	26.402	3.569	3.577	3.561	Yes	No
74	B297	1.316	0.800	28.117	28.745	27.468	5.117	5.122	5.112	Yes	Yes
75	B109	1.315	0.765	38.555	39.001	38.067	1.495	1.502	1.488	Yes	No
76	B320	1.313	0.684	11.016	11.465	10.551	5.729	5.734	5.724	Yes	No
77	B129	1.312	0.617	61.145	61.889	60.393	1.478	1.484	1.471	No	No
78	B319	1.311	0.608	11.111	11.498	10.747	5.745	5.749	5.741	Yes	Yes
79	B412	1.311	0.822	21.835	22.370	21.298	7.231	7.237	7.225	Yes	No
80	B345	1.310	0.781	26.277	26.883	25.642	6.842	6.859	6.825	Yes	Yes
81	B393	1.308	0.804	26.753	27.272	26.244	3.753	3.765	3.741	Yes	No
82	B43	1.305	0.625	16.790	17.360	16.255	2.177	2.197	2.157	Yes	No
83	B78	1.305	0.833	10.318	11.358	9.281	1.697	1.750	1.644	Yes	Yes
84	B37	1.300	0.874	24.722	25.672	23.778	1.996	2.031	1.961	Yes	No

Table SI 13 - 100 first SYMAPS HSQC VIP buckets from OPLS-DA (Part 3/3)

HSQC VIP Ranking	Bucket	V1	V2	Center F1 [ppm]	Start ^{13}C F1 [ppm]	End ^{13}C F1 [ppm]	Center F2 [ppm]	Start ^{1}H F2 [ppm]	End ^{1}H F2 [ppm]	Potential correspondence in ^{13}C folded	Potential correspondence in ^{1}H
OP85	B188	1.300	0.819	62.224	62.789	61.710	1.245	1.255	1.235	No	No
86	B63	1.300	0.824	22.152	23.000	21.299	1.342	1.358	1.326	Yes	Yes
87	B410	1.297	0.876	18.916	19.446	18.379	5.237	5.259	5.214	Yes	No
88	B61	1.293	0.880	24.786	25.457	24.134	1.258	1.290	1.225	Yes	Yes
89	B123	1.289	0.880	47.661	48.552	46.744	1.500	1.512	1.489	No	No
90	B280	1.288	0.827	29.608	30.052	29.213	1.641	1.658	1.624	Yes	Yes
91	B435	1.279	0.849	26.689	27.220	26.147	0.957	0.968	0.945	Yes	No
92	B102	1.278	0.607	35.287	35.720	34.861	1.741	1.750	1.731	No	Yes
93	B259	1.278	0.624	66.571	66.974	66.172	1.728	1.732	1.724	No	Yes
94	B199	1.277	0.837	45.884	46.485	45.290	1.250	1.261	1.239	No	No
95	B53	1.277	0.893	16.759	17.736	15.819	1.894	1.917	1.870	Yes	No
96	B32	1.276	0.819	26.626	27.303	25.935	3.457	3.485	3.430	Yes	No
97	B281	1.275	0.900	28.434	28.890	27.986	1.638	1.660	1.616	Yes	Yes
98	B409	1.273	0.853	18.853	19.327	18.379	5.312	5.326	5.299	Yes	No
99	B418	1.272	0.847	43.346	43.842	42.815	0.926	0.929	0.922	No	No
100	B258	1.271	0.764	66.634	67.227	66.088	1.735	1.737	1.732	No	Yes

Table SI 14 - Global data for all ^{1}H NMR buckets: Minimum (MIN), maximum (MAX) and mean SNR were measured for each sample group. To quantify the proportion of data under the limit of detection and quantification, number of buckets with a Signal to Noise Ratio (SNR) under 3 and 10 have been counted.

	Blank_Ctrl	Blank_Sponge	Control_T1	Control_T2	QC	Sponge_T1	Sponge_T2
MIN	0	0	0	0	0	0	1
MAX	11471	17332	20872	17698	24118	21244	21870
MEAN	117	195	768	706	775	769	802
Number of buckets with a SNR<3	256	167	10	16	12	15	12
% SNR<3	28%	19%	1%	2%	1%	2%	1%
Number of buckets with a SNR<10	400	340	63	65	63	78	65
% SNR<10	44%	38%	7%	7%	7%	9%	7%
Total buckets	901	901	901	900	900	901	901

Table SI 15 - Global data for all ^{13}C NMR buckets: Minimum (MIN), maximum (MAX) and mean SNR were measured for each sample group. To quantify the proportion of data under the limit of detection and quantification, number of buckets with a Signal to Noise Ratio (SNR) under 3 and 10 have been counted.

	Blank_Ctrl	**Blank_Sponge**	**Control_T1**	**Control_T2**	**QC**	**Sponge_T1**	**Sponge_T2**
MIN	0	0	0	0	0	1	1
MAX	27	24	253	228	304	272	270
MEAN	2	2	17	17	20	19	20
Number of buckets with a SNR<3	380	338	79	72	77	71	79
% SNR <3	92%	82%	19%	18%	19%	17%	19%
Number of buckets with a SNR<10	396	395	267	262	247	263	258
% SNR <10	96%	96%	65%	64%	60%	64%	63%
Total buckets	411	411	411	411	411	411	411