Supplementary Information

Limited impact of presaturation time on the accuracy of ^{1}H qNMR using a NOESY with high-power presaturation

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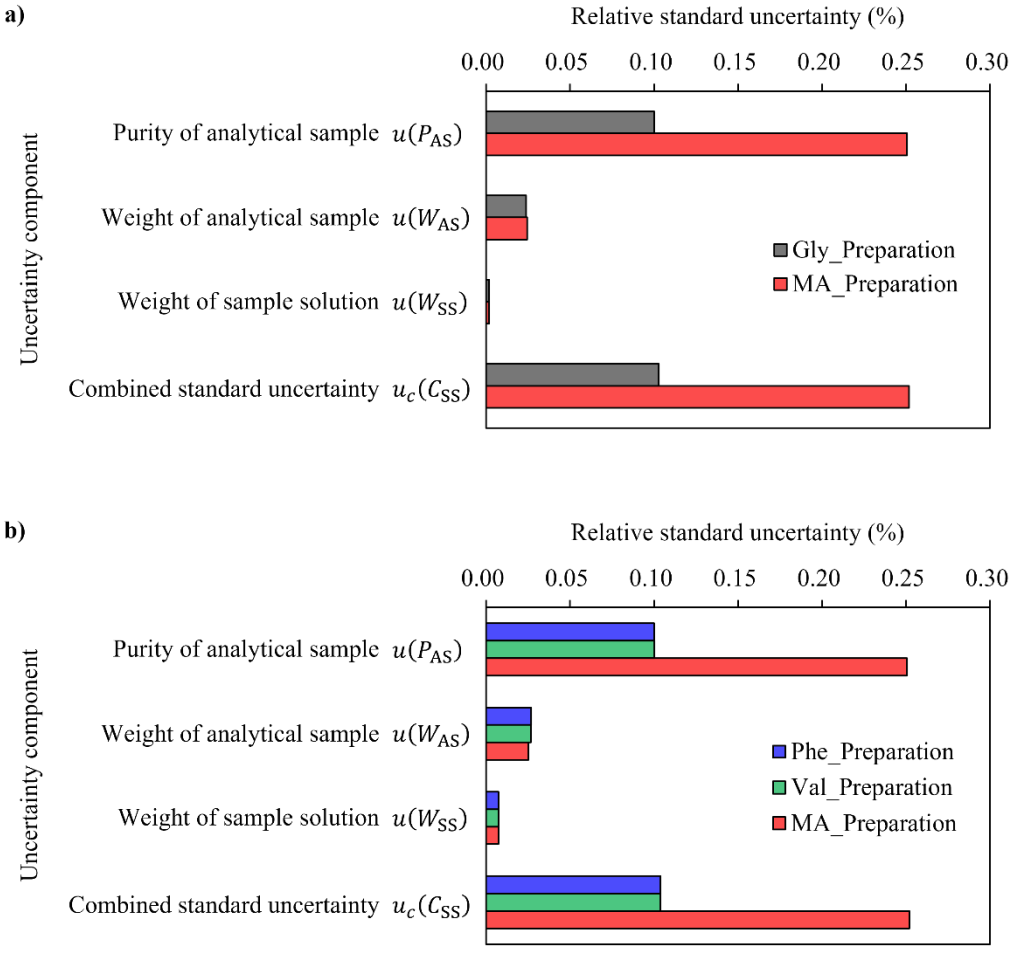

Fig. SI1 Total uncertainty budgets for prepared concentrations of sample solutions. a) A H_2O/D_2O (90/10 vol %) solution containing Gly, MA and DSS-d_6, b) another H_2O/D_2O (90/10 vol %) solution containing Phe, Val, MA and DSS-d_6. The prepared concentrations of the solutions were calculated by equation S1.

$$C_{SS} = (W_{AS}/W_{SS}) \cdot P_{AS} \cdot 10^6 \qquad \text{(S1)}$$

Here, C is concentration (mg kg^{-1}), W is weight (mg) and P is purity (kg kg^{-1}). Subscripts SS and AS refer to sample solution and analytical sample, respectively. Combined standard uncertainties of the prepared concentrations of the sample solutions were evaluated using each component uncertainty in equation S1.

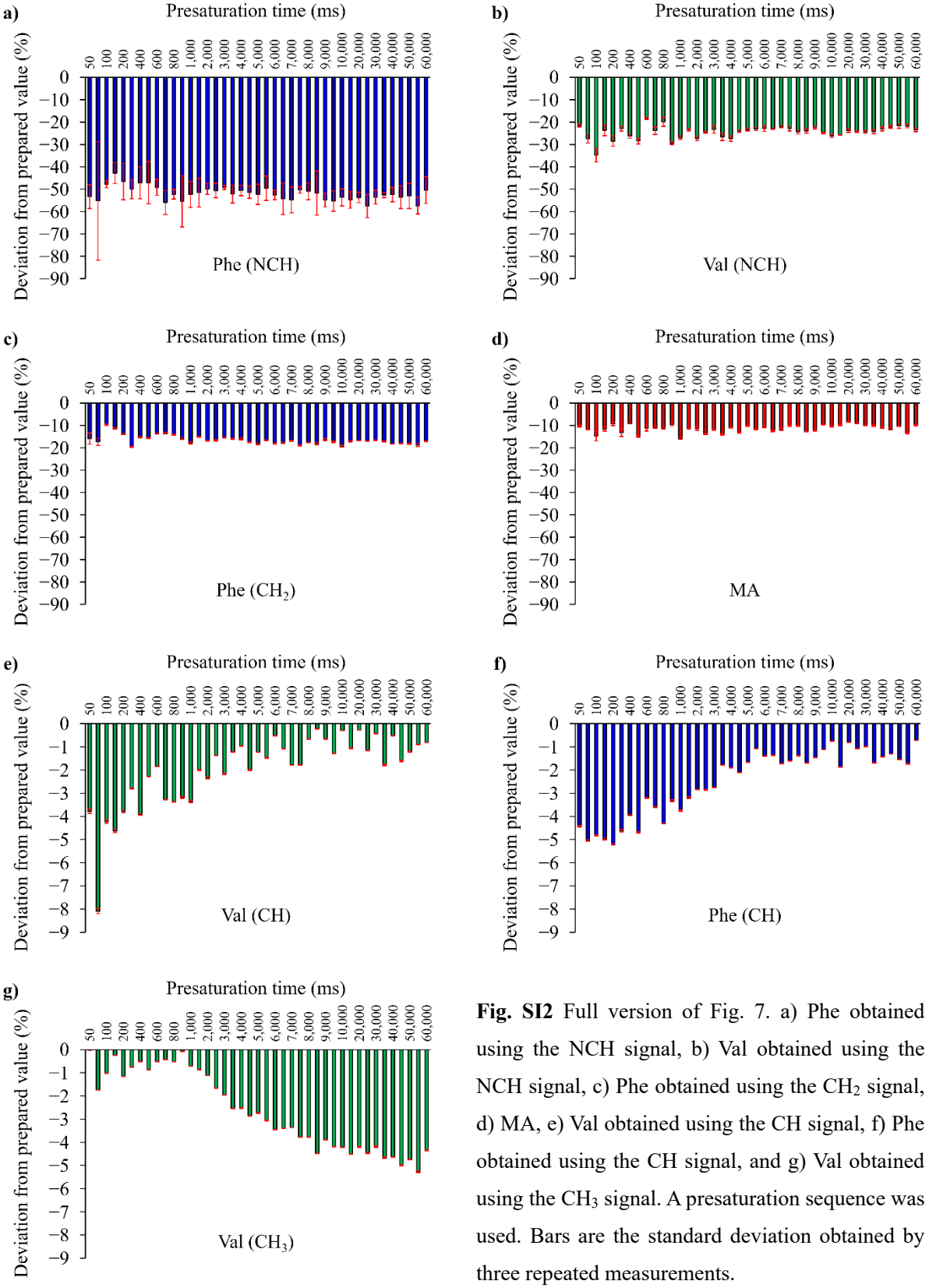

Fig. SI2 Full version of Fig. 7. a) Phe obtained using the NCH signal, b) Val obtained using the NCH signal, c) Phe obtained using the CH_2 signal, d) MA, e) Val obtained using the CH signal, f) Phe obtained using the CH signal, and g) Val obtained using the CH_3 signal. A presaturation sequence was used. Bars are the standard deviation obtained by three repeated measurements.

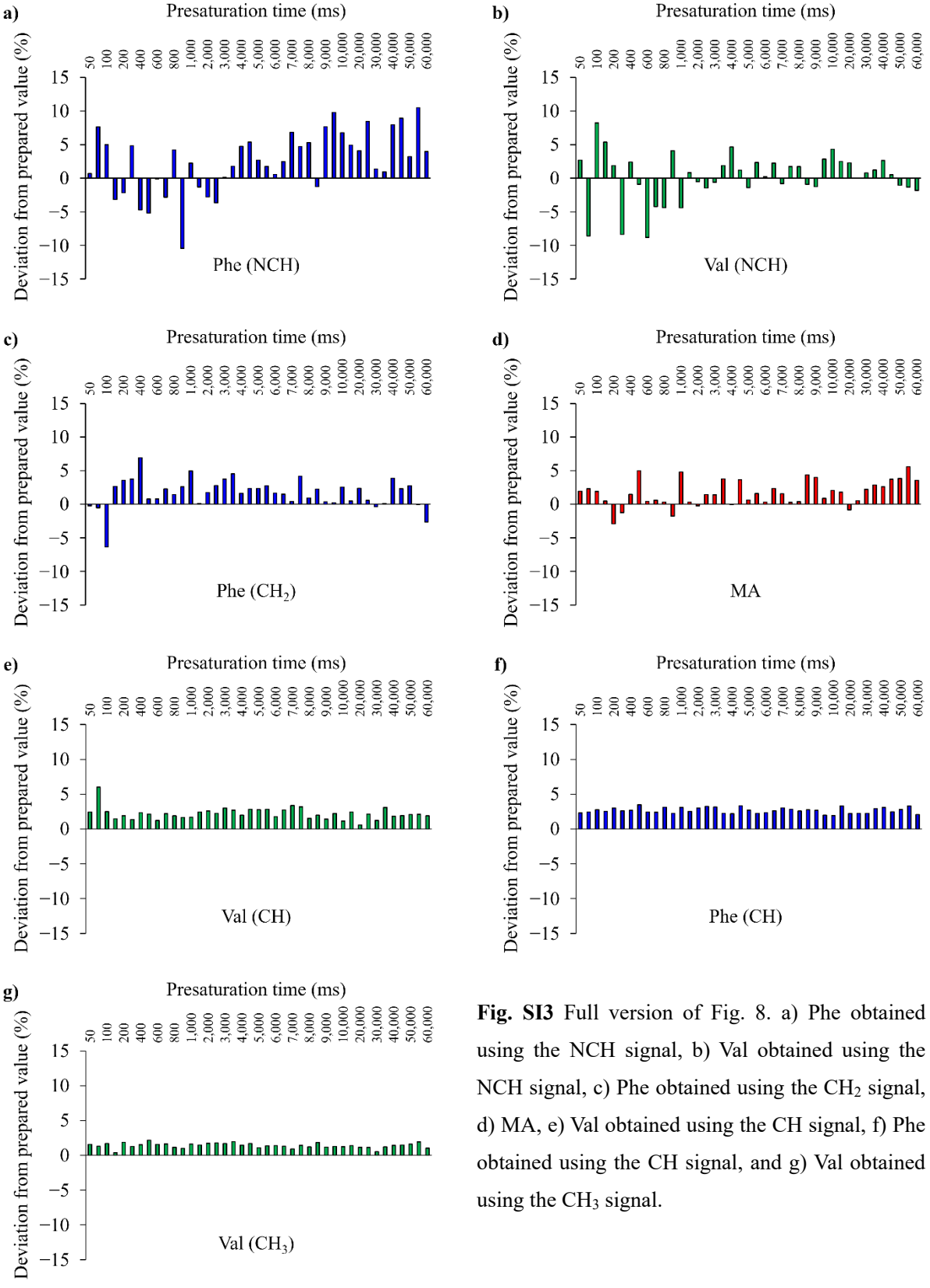

Fig. SI3 Full version of Fig. 8. a) Phe obtained using the NCH signal, b) Val obtained using the NCH signal, c) Phe obtained using the CH_2 signal, d) MA, e) Val obtained using the CH signal, f) Phe obtained using the CH signal, and g) Val obtained using the CH_3 signal.

Table SI1 Presaturation strengths for a H_2O/D_2O (90/10 vol %) solution containing Gly, MA and DSS-d_6.

Attenuation (dB)	90 degree pulse width [a] (μs)	Presaturation strength [b] ($\gamma B_1/2\pi$, Hz)
112	2793,419.0	0.1
106	1400,026.0	0.2
100	701,675.1	0.4
94	351,670.6	0.7
88	176,252.8	1.4
82	88,335.7	2.8
76	44,272.7	5.6
70	22,188.9	11.3
64	11,120.8	22.5
58	5,573.6	44.9
55	3,945.8	63.4
52	2,793.4	89.5
46	1,400.0	178.6
40	701.7	356.3
34	351.7	710.9
28	176.3	1,418.4
22	88.3	2,830.1

[a] The values were calculated using an attenuation of 5.9 dB and a pulse width of 13.84 μs for a 90 degree pulse.

[b] The values were calculated by reciprocals of 360 degree pulse widths.

Table SI2 Presaturation strengths for a H_2O/D_2O (90/10 vol %) solution containing Phe, Val, MA and DSS-d_6.

Attenuation (dB)	90 degree pulse width [a] (μs)	Presaturation strength [b] ($\gamma B_1/2\pi$, Hz)
112	2688,464.0	0.1
106	1347,423.8	0.2
100	675,311.6	0.4
94	338,457.6	0.7
88	169,630.6	1.5
82	85,016.7	2.9
76	42,609.3	5.9
70	21,355.2	11.7
64	10,703.0	23.4
58	5,364.2	46.6
55	3,797.6	65.8
52	2,688.5	93.0
46	1,347.4	185.5
40	675.3	370.2
34	338.5	738.6
28	169.6	1,473.8
22	85.0	2,940.6

[a] The values were calculated using an attenuation of 5.9 dB and a pulse width of 13.32 μs for a 90 degree pulse.

[b] The values were calculated by reciprocals of 360 degree pulse widths.

Table SI3 Unconverted concentration data corresponding to Figs. 3e and 3f.

Presaturation time (ms)	Measured concentration [a] (mg kg^{-1})	
	Gly	MA
1	138	291
5	210	268
10	219	267
25	226	253
50	210	242
100	217	245
200	210	243
300	218	239
400	212	244
500	204	235
700	213	239
900	210	239
1,000	210	240
1,500	206	229
2,000	205	244
2,500	223	242
3,000	217	233
3,500	219	253
4,000	220	247
4,500	218	237
5,000	214	242
5,500	225	253
6,000	217	243
6,500	216	239
7,000	219	260
7,500	225	233
8,000	224	245
8,500	214	256
9,000	223	239
9,500	219	247

10,000	221	262
15,000	214	237
20,000	214	242
25,000	217	243
30,000	220	250
35,000	222	262
40,000	219	260
45,000	215	250
50,000	219	247
55,000	219	255
60,000	214	263

Table SI4 Unconverted concentration data corresponding to Fig. 5.

Presaturation time (ms)	Measured concentration [a] (mg kg^{-1})													
	Phe NCH		Val NCH		Phe CH_2		MA		Val CH		Phe CH		Val CH_3	
50	460	(102)	792	(43)	813	(27)	943	(45)	962	(19)	950	(8)	991	(2)
80	509	(157)	625	(121)	797	(58)	932	(42)	955	(13)	946	(3)	971	(2)
100	556	(181)	716	(25)	814	(40)	898	(45)	959	(2)	951	(4)	982	(2)
150	524	(126)	797	(12)	886	(7)	916	(25)	945	(4)	947	(2)	977	(1)
200	497	(122)	716	(37)	870	(37)	904	(28)	957	(23)	949	(7)	982	(3)
300	532	(147)	671	(68)	815	(25)	881	(8)	961	(10)	951	(1)	981	(3)
400	467	(74)	743	(18)	890	(27)	953	(10)	960	(6)	959	(4)	985	(1)
500	463	(39)	689	(27)	826	(59)	928	(12)	974	(11)	959	(4)	988	(4)
600	493	(66)	711	(24)	846	(55)	919	(32)	970	(20)	963	(8)	986	(8)
700	400	(73)	701	(26)	861	(14)	924	(22)	965	(8)	959	(6)	987	(4)
800	505	(33)	739	(46)	847	(41)	918	(31)	961	(4)	959	(2)	982	(1)
900	331	(20)	728	(84)	841	(27)	914	(9)	961	(2)	960	(3)	985	(3)
1,000	485	(115)	670	(94)	846	(35)	917	(23)	959	(6)	965	(3)	984	(1)
1,500	459	(51)	753	(10)	824	(8)	914	(2)	980	(3)	964	(0)	981	(3)

2,000	460	(8)	704	(52)	824	(44)	910	(4)	978	(7)	972	(4)	981	(2)
2,500	444	(48)	721	(45)	835	(66)	902	(24)	984	(6)	975	(5)	977	(5)
3,000	495	(61)	743	(24)	857	(21)	921	(21)	984	(3)	975	(5)	973	(2)
3,500	482	(76)	734	(26)	858	(26)	925	(14)	990	(9)	975	(3)	970	(2)
4,000	526	(14)	754	(18)	831	(32)	916	(19)	985	(3)	974	(3)	965	(3)
4,500	524	(68)	752	(14)	824	(40)	932	(4)	984	(10)	983	(5)	964	(1)
5,000	489	(54)	734	(42)	817	(11)	931	(9)	991	(7)	981	(8)	959	(3)
5,500	507	(16)	773	(53)	835	(5)	925	(7)	989	(9)	982	(2)	959	(1)
6,000	466	(89)	755	(20)	810	(22)	924	(27)	988	(3)	980	(3)	955	(1)
6,500	468	(79)	774	(24)	812	(20)	926	(9)	992	(8)	983	(2)	955	(3)
7,000	505	(40)	754	(51)	811	(11)	925	(12)	991	(4)	984	(1)	952	(3)
7,500	528	(32)	770	(15)	827	(41)	928	(13)	989	(4)	983	(6)	953	(1)
8,000	527	(10)	754	(19)	808	(17)	930	(13)	984	(2)	982	(3)	950	(1)
8,500	456	(61)	733	(10)	815	(9)	943	(8)	993	(6)	981	(4)	950	(3)
9,000	513	(78)	745	(15)	815	(30)	944	(11)	983	(7)	983	(2)	949	(2)
9,500	529	(19)	761	(19)	804	(24)	941	(8)	985	(7)	979	(5)	947	(2)
10,000	516	(98)	762	(32)	808	(35)	943	(8)	984	(5)	982	(1)	947	(1)
15,000	487	(77)	749	(22)	808	(49)	947	(16)	989	(6)	985	(5)	945	(1)
20,000	489	(53)	766	(18)	830	(9)	935	(16)	978	(5)	984	(5)	946	(3)
25,000	494	(28)	739	(12)	810	(39)	943	(9)	985	(7)	982	(1)	943	(1)
30,000	464	(31)	744	(22)	805	(17)	950	(14)	983	(3)	983	(3)	940	(0)
35,000	469	(25)	751	(1)	805	(26)	956	(3)	988	(6)	983	(8)	942	(1)
40,000	539	(55)	776	(31)	830	(41)	943	(18)	988	(6)	987	(4)	944	(2)
45,000	537	(31)	764	(21)	817	(7)	949	(7)	979	(8)	982	(1)	941	(1)
50,000	487	(52)	754	(15)	820	(30)	962	(20)	984	(5)	983	(2)	945	(3)
55,000	516	(108)	750	(20)	788	(10)	949	(8)	987	(3)	986	(5)	943	(0)
60,000	520	(49)	728	(35)	778	(4)	963	(12)	986	(12)	984	(3)	943	(3)

[a] The measured concentrations are displayed in ascending order of absolute chemical shift difference between the analyte signal and presaturation offset. The number in parenthesis indicates the standard deviation obtained by three repeated measures at the same presaturation time.

Table SI5 Unconverted concentration data corresponding to Fig. SI2.

Presaturation time (ms)	Measured concentration [a] (mg kg^{-1})													
	Phe NCH		Val NCH		Phe CH_2		MA		Val CH		Phe CH		Val CH_3	
50	454	(45)	766	(22)	816	(127)	923	(29)	939	(22)	928	(6)	975	(3)
80	435	(209)	709	(52)	802	(75)	908	(9)	897	(12)	922	(7)	959	(2)
100	507	(18)	636	(56)	876	(24)	878	(125)	934	(17)	924	(5)	966	(4)
150	555	(61)	745	(78)	861	(29)	911	(72)	930	(15)	923	(9)	973	(5)
200	518	(91)	697	(56)	835	(18)	934	(79)	939	(14)	920	(5)	964	(3)
300	485	(41)	752	(37)	779	(7)	894	(108)	948	(9)	926	(11)	968	(2)
400	513	(78)	719	(26)	823	(19)	938	(30)	937	(8)	933	(6)	971	(5)
500	514	(102)	698	(34)	818	(11)	876	(24)	953	(3)	925	(6)	967	(2)
600	494	(36)	797	(22)	838	(22)	915	(105)	958	(3)	940	(5)	971	(7)
700	428	(41)	743	(52)	839	(18)	918	(34)	944	(4)	936	(9)	971	(3)
800	464	(19)	781	(79)	833	(14)	915	(33)	943	(6)	929	(1)	970	(5)
900	432	(90)	688	(16)	816	(24)	932	(59)	945	(13)	938	(11)	975	(4)
1,000	463	(51)	713	(20)	798	(23)	868	(25)	943	(13)	934	(7)	969	(3)
1,500	471	(59)	745	(17)	823	(14)	911	(11)	956	(7)	940	(12)	967	(6)
2,000	487	(25)	709	(27)	807	(7)	913	(53)	953	(17)	943	(4)	965	(3)
2,500	479	(31)	736	(12)	808	(13)	887	(9)	962	(5)	943	(7)	959	(4)
3,000	493	(10)	749	(57)	820	(15)	907	(11)	954	(11)	944	(2)	956	(2)
3,500	464	(35)	716	(43)	814	(13)	886	(17)	964	(9)	953	(4)	951	(6)
4,000	480	(22)	708	(32)	815	(26)	917	(12)	966	(7)	952	(4)	951	(3)
4,500	472	(25)	740	(16)	801	(10)	894	(14)	956	(9)	950	(2)	948	(0)
5,000	463	(40)	748	(19)	794	(17)	925	(18)	963	(3)	955	(4)	949	(5)
5,500	490	(54)	750	(23)	809	(7)	909	(16)	961	(10)	960	(6)	946	(5)
6,000	461	(19)	753	(44)	794	(9)	921	(31)	970	(8)	957	(3)	942	(4)
6,500	444	(58)	752	(10)	798	(16)	902	(21)	965	(10)	958	(7)	942	(4)
7,000	439	(46)	761	(18)	807	(10)	909	(34)	958	(5)	954	(3)	943	(2)
7,500	482	(14)	753	(38)	786	(8)	925	(17)	958	(10)	955	(4)	939	(0)
8,000	476	(35)	737	(26)	799	(1)	926	(18)	969	(6)	957	(5)	939	(2)
8,500	469	(89)	742	(30)	793	(24)	898	(12)	973	(14)	954	(4)	932	(1)
9,000	438	(24)	757	(23)	812	(25)	903	(13)	969	(6)	957	(4)	937	(1)
9,500	434	(36)	733	(8)	802	(12)	932	(20)	963	(5)	960	(2)	935	(3)

10,000	450	(32)	720	(18)	783	(17)	922	(18)	973	(10)	963	(9)	935	(2)
15,000	439	(30)	725	(2)	803	(8)	929	(25)	965	(11)	953	(2)	931	(2)
20,000	449	(20)	744	(26)	807	(4)	944	(14)	973	(13)	963	(6)	935	(4)
25,000	412	(37)	739	(9)	804	(6)	937	(28)	964	(5)	960	(4)	932	(5)
30,000	451	(26)	737	(18)	808	(6)	927	(14)	971	(10)	961	(2)	935	(7)
35,000	460	(9)	739	(27)	804	(12)	926	(27)	958	(14)	954	(5)	930	(7)
40,000	462	(28)	751	(29)	792	(4)	917	(27)	971	(1)	957	(5)	930	(4)
45,000	451	(43)	759	(20)	795	(4)	911	(30)	960	(16)	958	(1)	927	(4)
50,000	456	(48)	764	(42)	793	(11)	923	(7)	964	(12)	956	(4)	929	(1)
55,000	414	(27)	763	(31)	788	(27)	891	(13)	967	(3)	954	(6)	924	(7)
60,000	482	(57)	745	(24)	804	(8)	927	(19)	968	(5)	964	(2)	933	(3)

[a] The measured concentrations are displayed in ascending order of absolute chemical shift difference between the analyte signal and presaturation offset. The number in parenthesis indicates the standard deviation obtained by three repeated measures at the same presaturation time.

Table SI6 Unconverted concentration data corresponding to Fig. SI3.

Presaturation time (ms)	Measured concentration [a] ($mg\ kg^{-1}$)						
	Phe NCH	Val NCH	Phe CH_2	MA	Val CH	Phe CH	Val CH_3
50	6.8	26.2	−2.4	20.1	23.8	22.6	15.2
80	74.1	−83.9	−5.0	23.9	58.6	23.8	12.6
100	48.4	80.2	−61.8	19.6	24.4	26.9	16.3
150	−30.6	52.3	25.8	4.9	14.5	24.6	3.6
200	−21.1	18.3	34.6	−30.2	18.4	29.3	18.2
300	47.0	−81.6	36.2	−12.9	13.1	25.3	12.5
400	−45.8	23.4	67.1	15.1	22.9	26.0	14.8
500	−50.5	−8.6	7.7	51.3	20.8	33.7	21.2
600	−0.8	−86.0	7.6	4.1	12.1	23.7	15.4
700	−27.3	−41.4	22.1	6.1	21.4	23.4	15.9
800	41.0	−42.7	14.0	3.2	18.5	30.3	11.2

900	−101.3	39.8	25.2	−18.2	16.2	21.8	9.8
1,000	21.8	−43.0	48.0	49.5	16.5	30.2	15.6
1,500	−12.5	7.9	0.9	3.1	23.9	24.4	14.3
2,000	−27.3	−4.9	17.0	−2.6	25.1	29.1	16.7
2,500	−35.7	−14.3	26.9	14.7	22.0	31.6	17.4
3,000	1.5	−5.8	36.3	14.4	29.2	30.5	16.3
3,500	17.4	18.3	43.9	39.0	26.3	21.9	19.0
4,000	46.2	45.2	15.9	−0.8	19.2	21.4	14.0
4,500	52.1	11.6	22.7	37.6	27.5	32.4	16.4
5,000	26.0	−13.6	22.8	6.3	27.0	26.3	10.7
5,500	17.0	22.8	26.7	16.4	27.7	21.8	13.2
6,000	5.4	2.1	16.1	3.4	17.2	22.6	13.4
6,500	24.0	22.0	14.5	24.3	26.6	25.3	12.5
7,000	66.3	−7.8	4.1	16.0	32.9	29.5	8.7
7,500	45.9	16.9	40.6	2.9	31.0	27.4	13.9
8,000	51.4	16.7	9.2	3.7	15.0	25.1	11.6
8,500	−12.3	−9.1	21.7	45.0	19.4	26.9	18.0
9,000	74.4	−12.3	3.5	41.2	13.7	26.0	11.6
9,500	95.0	27.6	2.1	9.1	21.8	19.3	12.2
10,000	65.8	42.1	24.8	21.2	11.1	18.8	12.0
15,000	48.0	24.5	4.5	18.3	23.6	32.0	13.6
20,000	39.6	22.1	23.1	−8.8	5.4	21.5	11.0
25,000	81.9	0.1	5.5	5.4	20.8	21.8	10.8
30,000	13.3	7.7	−3.7	22.8	12.0	21.8	4.9
35,000	9.0	12.0	1.0	29.6	30.1	28.2	11.7
40,000	77.3	25.7	37.5	26.7	17.9	30.1	13.8
45,000	86.6	5.0	22.3	38.2	18.8	24.0	14.4
50,000	31.1	−10.2	26.6	39.5	20.4	27.0	15.5
55,000	102.1	−13.1	−0.1	57.5	20.5	32.2	18.8
60,000	38.6	−17.8	−25.8	36.5	18.5	20.0	10.1

[a] The measured concentrations are displayed in ascending order of absolute chemical shift difference between the analyte signal and presaturation offset.