

Interlaboratory study of ethanol usage as an internal standard in direct determination of volatile compounds in alcoholic products

Supplementary Materials

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$$u(C_i (B)) = \rho_{Eth} \cdot \sqrt{\left(\frac{\partial C_i (B)}{\partial m_i^B}\right)^2 + \left(\frac{\partial C_i (B)}{\partial C_i}\right)^2 + \left(\frac{\partial C_i (B)}{\partial m_{HS}^B}\right)^2 + \left(\frac{\partial C_i (B)}{\partial C_i (HS)}\right)^2 + \left(\frac{\partial C_i (B)}{\partial C_{m/m}^{Eth} (HS)}\right)^2}, \quad (S.1)$$

$$u(C_i (HS)) = \frac{\rho_{Eth}}{\rho_{HS}} \cdot \sqrt{\frac{\partial C_i (HS)}{\partial A_i^{HS}} + \frac{\partial C_i (HS)}{\partial RRF_i^{Eth}} + \frac{\partial C_i (HS)}{\partial C_{v/v}^{Eth} (HS)} + \frac{\partial C_i (HS)}{\partial A_{Eth}^{HS}}}, \quad (S.2)$$

$$u(RRF_i^{Eth}) = \sqrt{\left(\frac{\partial RRF_i^{Eth}}{\partial A_{HS}^C}\right)^2 + \left(\frac{\partial RRF_i^{Eth}}{\partial m_i (C)}\right)^2 + \left(\frac{\partial RRF_i^{Eth}}{\partial A_i^{st}}\right)^2 + \left(\frac{\partial RRF_i^{Eth}}{\partial m_{Eth} (C)}\right)^2}, \quad (S.3)$$

$$u(C_i (C, D, 1, 2)) = \rho_{Eth} \cdot \sqrt{\left(\frac{\partial C_i (C, D, 1, 2)}{\partial m_B^{C,D,1,2}}\right)^2 + \left(\frac{\partial C_i (C, D, 1, 2)}{\partial C_i (B)}\right)^2 + \left(\frac{\partial C_i (C, D, 1, 2)}{\partial C_i (HS)}\right)^2 + \left(\frac{\partial C_i (C, D, 1, 2)}{\partial m_{HS}^{C,D,1,2}}\right)^2 + \left(\frac{\partial C_i (C, D, 1, 2)}{\partial C_{Eth} (B)}\right)^2 + \left(\frac{\partial C_i (C, D, 1, 2)}{\partial C_{m/m}^{Eth} (HS)}\right)^2}, \quad (S.4)$$

Table S.1. Results of z-score determination for SS-B

acetaldehyde					methyl acetate				ethyl acetate			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	412	0.90	-0.38		416	0.33	-0.67		417	0.36	-0.35	
2	416	10.77	0.48		424	6.25	-0.17		442	10.65	1.34	
3	411	0.38	-0.67		415	0.28	-0.78		422	0.64	-0.01	
4	422	1.31	1.70		444	2.41	1.02		421	2.82	-0.12	
5	412	2.69	-0.53		459	0.28	1.99		441	1.64	1.26	
6	408	6.81	-1.19		420	9.66	-0.47		415	8.44	-0.52	
7	413	1.96	-0.22		421	3.33	-0.36		417	3.51	-0.37	
8	408	6.81	-1.19		420	9.66	-0.47		415	8.44	-0.52	
9	406	1.52	-1.60		447	1.10	1.23		393	1.36	-1.99	
methanol					2-propanol				1-propanol			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	425	0.72	0.99		405	0.34	-0.46		418	0.01	-0.28	
2	425	6.69	1.29		407	1.03	0.73		423	2.06	1.72	
3	425	0.23	0.84		404	0.07	-0.90		417	0.28	-0.64	
4	422	1.07	-1.07		406	1.14	0.18		417	0.56	-0.36	
5	425	1.32	0.98		420	0.79	9.33	Grubbs**	422	0.50	1.35	
6	423	1.68	-0.67		405	1.69	-0.57		416	0.70	-0.81	
7	425	0.67	1.34		409	1.92	1.95		419	0.32	0.14	
8	423	1.68	-0.67		405	1.69	-0.57		416	0.70	-0.81	
9	412	0.96	-9.44	Grubbs**					415	2.21	-1.15	
2-methyl-1-propanol					1-butanol				3-methyl-1-butanol			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	430	0.35	-0.13		417	0.57	-0.15		412	0.18	-0.23	
2	436	2.86	1.72		428	0.75	8.55	Grubbs**	421	3.37	5.58	Grubbs**
3	429	0.18	-0.57		417	0.19	0.40		412	0.58	-0.42	
4	429	1.10	-0.49		415	0.88	-1.37		410	0.86	-1.31	
5	432	0.99	0.55		416	0.47	-0.64		413	0.96	0.22	
6	428	0.10	-0.86		417	0.69	0.02		411	1.11	-0.84	
7	431	1.07	0.03		419	0.42	1.97		414	0.63	1.40	
8	428	0.10	-0.86		417	0.69	0.02		411	1.11	-0.84	
9	425	2.71	-1.83		416	0.49	-0.82		410	5.82	-1.88	

* Straggler, ** Statistical outlier

Table S.2. Results of z-score determination for SS-D

acetaldehyde					methyl acetate				ethyl acetate			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	186	0.38	1.46		179	0.89	-1.92		190	0.60	0.48	
2	179	6.14	-1.45		185	8.16	1.76		192	5.26	1.99	
3	182	0.38	-0.57		181	0.96	-0.37		187	0.88	-1.41	
4	184	0.94	0.55		182	1.32	-0.14		189	0.52	0.36	
5	179	1.57	-1.79		181	1.75	-0.67		188	1.30	-0.26	
6	182	6.85	-0.25		181	6.85	-0.39		189	7.61	0.25	
7	182	2.25	-0.24		181	2.50	-0.40		188	2.48	-0.58	
8	182	6.85	-0.25		181	6.85	-0.39		189	7.61	0.25	
9	184	0.58	0.48		183	0.79	0.71		191	0.46	1.33	
methanol					2-propanol				1-propanol			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	199	0.60	1.57		181	0.45	1.06		186	0.26	1.68	
2	186	0.26	-17.60	Grubbs**	178	2.96	-1.89		190	1.49	11.28	Grubbs**
3	197	0.45	-0.47		180	0.40	0.26		186	0.14	-0.23	
4	199	0.84	1.99		180	1.04	0.37		186	1.01	-0.32	
5	198	0.62	0.18		182	0.48	1.32		186	0.46	0.12	
6	197	0.85	-0.55		181	1.14	0.45		186	0.24	-0.36	
7	197	0.53	-0.28		179	0.64	-0.49		185	0.32	-0.67	
8	197	0.85	-0.55		181	1.14	0.45		186	0.24	-0.36	
9	197	0.15	-0.13						185	0.47	-1.96	
2-methyl-1-propanol					1-butanol				3-methyl-1-butanol			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	192	0.28	1.79		185	0.40	0.27		183	0.19	0.30	
2	189	7.48	-1.83		183	9.12	-1.79		188	0.02	2.54	Cochran*
3	191	0.24	0.93		185	0.17	0.85		183	0.09	0.34	
4	191	0.41	0.31		185	0.95	-0.35		182	0.84	-0.34	
5	191	0.36	0.51		186	1.28	1.46		184	0.40	0.37	
6	190	1.30	0.11		185	0.58	-0.11		183	0.68	0.02	
7	191	0.80	0.26		185	0.12	0.60		183	0.36	0.07	
8	190	1.30	0.11		185	0.58	-0.11		183	0.68	0.02	
9	190	0.96	-0.62		184	3.09	-1.17		180	3.48	-1.25	

* Straggler, ** Statistical outlier

Table S.3. Results of z-score determination for SS-1

acetaldehyde					methyl acetate				ethyl acetate			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	48.3	0.05	0.44		44.3	0.15	-0.37		49.5	0.08	1.45	
2	49.4	1.49	1.34		50.4	0.63	1.63	Cochran*	49.6	2.63	1.58	
3	47.4	0.09	-0.28		46.6	0.22	0.41		47.4	0.07	-0.37	
4	48.1	0.70	0.28		42.2	0.31	-1.04		48.3	0.76	0.40	
5	47.1	0.69	-0.57		42.0	0.22	-1.11		47.8	0.77	-0.05	
6	47.0	0.53	-0.61		46.7	0.45	0.41		48.0	0.56	0.16	
7	45.7	0.66	-1.72		46.3	0.73	0.29		46.1	0.58	-1.57	
8	47.0	0.53	-0.61		46.7	0.45	0.41		48.0	0.56	0.16	
9	49.4	1.20	1.36		40.7	0.66	-1.55		64.0	1.50	14.38	Grubbs**
methanol					2-propanol				1-propanol			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	61.4	0.09	0.80		47.1	0.34	0.99		47.7	0.29	-0.19	
2	57.1	3.15	-1.38		46.2	1.56	-0.16		48.9	1.50	1.83	Cochran*
3	59.9	0.07	0.03		47.3	0.09	1.21		48.0	0.09	0.37	
4	56.6	0.59	-1.62		46.8	0.16	0.61		46.9	0.50	-1.49	
5	59.0	1.58	-0.43		44.7	0.74	-1.99		47.5	0.43	-0.45	
6	60.8	0.18	0.51		46.7	0.33	0.52		47.1	0.27	-1.17	
7	59.1	0.30	-0.38		46.3	0.18	0.02		47.5	0.25	-0.55	
8	60.8	0.18	0.51		46.7	0.33	0.52		47.1	0.27	-1.17	
9	62.6	0.65	1.42						47.5	0.28	-0.43	
2-methyl-1-propanol					1-butanol				3-methyl-1-butanol			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	49.2	0.06	0.68		47.2	0.20	0.00		47.0	0.04	0.13	
2	49.7	1.54	1.96		48.3	1.66	1.81		47.8	1.51	1.98	
3	48.9	0.18	0.18		47.3	0.06	0.02		47.0	0.12	0.13	
4	48.4	0.42	-1.05		46.7	0.22	-0.81		46.4	0.08	-1.23	
5	49.1	0.19	0.55		47.0	0.62	-0.32		46.9	0.34	-0.04	
6	48.7	0.16	-0.33		46.5	0.33	-1.26		46.8	0.18	-0.38	
7	48.3	0.14	-1.18		46.9	0.10	-0.51		46.3	0.15	-1.53	
8	48.7	0.16	-0.33		46.5	0.33	-1.26		46.8	0.18	-0.38	
9	49.3	0.32	0.93		47.8	1.33	0.88		47.0	0.76	0.19	

* Straggler, ** Statistical outlier

Table S.4. Results of z-score determination for SS-2

acetaldehyde					methyl acetate				ethyl acetate			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	9.7	0.17	-1.02		8.6	0.13	-0.21		10.0	0.04	1.17	
2	10.5	0.21	-0.15		9.2	0.15	1.35		9.1	0.31	-0.68	
3	10.8	0.10	0.22		9.1	0.09	1.21		9.2	0.13	-0.46	
4	11.5	0.66	0.97		8.5	0.14	-0.35		9.7	0.11	0.64	
5	11.8	0.06	1.26		8.2	0.31	-1.13		9.7	0.08	0.63	
6	9.5	0.18	-1.26		8.2	0.09	-1.09		8.7	0.20	-1.51	
7	9.6	0.06	-1.17		8.8	0.14	0.25		9.4	0.06	-0.06	
8	9.5	0.18	-1.26		8.2	0.09	-1.09		8.7	0.20	-1.51	
9	11.1	0.32	0.46		5.4	0.31	-8.38	Grubbs**	30.1	0.57	42.62	Grubbs**
methanol					2-propanol				1-propanol			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	22.7	0.12	0.11		10.4	0.10	0.73		10.0	0.07	1.01	
2	24.7	0.64	1.65						10.0	0.05	0.79	
3	22.4	0.14	-0.12		10.4	0.05	0.72		10.1	0.09	1.27	
4	21.7	0.50	-0.71		10.3	0.15	0.49		10.0	0.30	0.77	
5	20.8	0.42	-1.40		9.3	0.14	-1.89		9.7	0.06	0.02	
6	22.4	0.30	-0.16		9.9	0.26	-0.59		9.0	0.48	-1.63	
7	22.2	0.03	-0.33		10.4	0.02	0.68		9.8	0.23	0.35	
8	22.4	0.30	-0.16		9.9	0.26	-0.59		9.3	0.13	-0.91	
9	24.7	1.24	1.62						9.4	0.18	-0.79	
2-methyl-1-propanol					1-butanol				3-methyl-1-butanol			
Lab	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier	Mean, mg/L AA	Standard deviation, mg/L AA	z-score	Outlier
1	9.9	0.08	0.52		9.5	0.16	1.14		9.4	0.04	0.58	
2	9.1	0.16	-1.36		9.3	0.32	0.12		9.0	0.32	-1.13	
3	9.8	0.05	0.17		9.4	0.02	0.65		9.2	0.13	-0.35	
4	9.4	0.14	-0.57		9.2	0.09	-0.07		9.2	0.17	-0.16	
5	9.9	0.08	0.58		9.2	0.12	0.03		9.8	0.14	1.95	
6	9.2	0.06	-1.06		8.8	0.10	-1.73		9.1	0.32	-0.83	
7	9.7	0.13	-0.06		9.3	0.15	0.25		9.1	0.03	-0.55	
8	9.2	0.06	-1.06		8.8	0.10	-1.73		9.1	0.32	-0.83	
9	10.4	0.35	1.76		9.4	0.18	0.52		10.7	0.27	5.53	Grubbs**

* Straggler, ** Statistical outlier

Table S.7. Summary of calculated statistical parameters for SS-1*

Statistical parameter	Compound																										
	acetaldehyde		methyl acetate		ethyl acetate		methanol		2-propanol		1-propanol		isobutanol		1-butanol		isoamylol										
Assigned value, mg/L AA	48		45		48		60		46		48		49		47		47										
Mean, mg/L AA	48		45		48		60		46		48		49		47		47										
n	9		9		9		9		8		9		9		9		9										
Outliers	0		0		1		0		0		0		0		0		0										
n ₁	9		9		8		9		8		9		9		9		9										
r, %	4.6		2.8		5.6		5.7		3.9		2.8		3.2		4.5		3.5										
s _r , mg/L AA	0.8		0.5		1.0		1.2		0.6		0.5		0.6		0.8		0.6										
RSD _r , %	1.7		1.0		2.0		2.0		1.4		1.0		1.1		1.6		1.2										
Ho _r	0.3		0.2		0.3		0.3		0.2		0.2		0.2		0.2		0.2										
R, %	6.2		3.8		7.8		7.6		5.2		3.9		4.2		6.0		4.6										
s _R , mg/L AA	1.1		0.6		1.3		1.6		0.9		0.7		0.7		1.0		0.8										
RSD _R , %	2.2		1.4		2.8		2.7		1.9		1.4		1.5		2.2		1.7										
Ho _R	0.5		0.3		0.6		0.7		0.4		0.3		0.4		0.5		0.4										
$\hat{\delta}$, mg/L AA	0.0		-0.3		0.2		-0.1		0.2		-0.2		0.1		-0.1		-0.1										
S _{$\hat{\delta}$} , mg/L AA	0.3		0.2		0.4		0.4		0.2		0.2		0.2		0.3		0.2										
$\hat{\delta}$, %	-0.1		-0.7		0.5		-0.2		0.4		-0.5		0.1		-0.2		-0.1										
Interval for the bias, mg/L AA	-0.6	< δ <	0.5	-0.6	< δ <	0.0	-0.5	< δ <	1.0	-1.0	< δ <	0.7	-0.3	< δ <	0.7	-0.6	< δ <	0.14	-0.3	< δ <	0.5	-0.6	< δ <	0.4	-0.5	< δ <	0.4
u, mg/L AA	1.8		1.5		2.0		2.5		1.7		1.6		1.7		1.8		1.6										
u, %	3.8		3.3		4.2		4.1		3.6		3.3		3.4		3.7		3.5										
U, % (P=0.95 k=2)	7.6		6.6		8.4		8.2		7.2		6.7		6.8		7.5		6.9										

Table S.8. Summary of calculated statistical parameters for SS-2*

Statistical parameter	Compound																										
	acetaldehyde			methyl acetate			ethyl acetate			methanol			2-propanol			1-propanol			isobutanol			1-butanol			isoamylol		
Assigned value, mg/L AA	10.7			8.7			9.4			22.6			10.1			9.7			9.7			9.2			9.3		
Mean, mg/L AA	10.5			8.6			9.3			22.7			10.1			9.7			9.6			9.2			9.2		
n	9			9			9			9			7			9			9			9			9		
Outliers	0			1			1			0			1			0			0			0			1		
n ₁	9			8			8			9			7			9			9			9			8		
r, %	7.5			4.6			4.6			6.7			4.8			6.5			4.4			4.8			6.1		
s _r , mg/L AA	0.3			0.1			0.2			0.6			0.2			0.2			0.2			0.2			0.2		
RSD _r , %	2.7			1.7			1.7			2.4			1.7			2.3			1.6			1.7			2.2		
Ho _r	0.3			0.2			0.2			0.3			0.2			0.3			0.2			0.2			0.3		
R, %	10.0			6.3			6.3			8.9			6.4			8.5			5.9			6.3			8.3		
s _R , mg/L AA	0.4			0.2			0.2			0.7			0.2			0.3			0.2			0.2			0.3		
RSD _R , %	3.6			2.3			2.3			3.2			2.3			3.1			2.1			2.3			3.0		
Ho _R	0.7			0.4			0.4			0.7			0.4			0.6			0.4			0.4			0.5		
$\hat{\delta}$, mg/L AA	-0.2			-0.1			-0.1			0.1			0.0			0.0			-0.1			0.0			0.0		
S _{$\hat{\delta}$} , mg/L AA	0.1			0.1			0.1			0.2			0.1			0.1			0.1			0.1			0.1		
$\hat{\delta}$, %	-1.8			-0.6			-1.2			0.3			-0.3			0.4			-0.5			-0.3			-0.5		
Interval for the bias, mg/L AA	-0.4	< δ <	0.002	-0.2	< δ <	0.1	-0.2	< δ <	0.0	-0.3	< δ <	0.5	-0.2	< δ <	0.1	-0.1	< δ <	0.2	-0.2	< δ <	0.1	-0.1	< δ <	0.1	-0.2	< δ <	0.1
u, mg/L AA	0.5			0.3			0.4			1.0			0.4			0.4			0.4			0.4			0.4		
u, %	4.7			3.8			3.8			4.5			3.8			4.4			3.7			3.8			4.3		
U, % (P=0.95 k=2)	9.5			7.6			7.6			9.0			7.7			8.8			7.4			7.6			8.6		

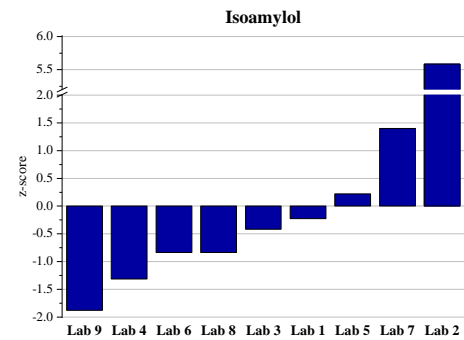
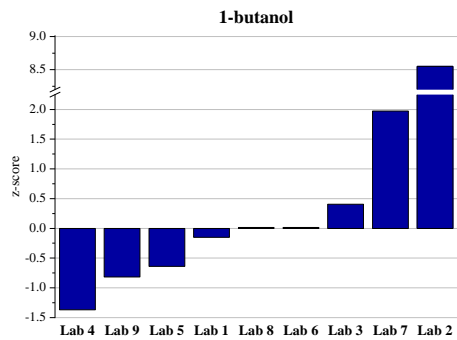
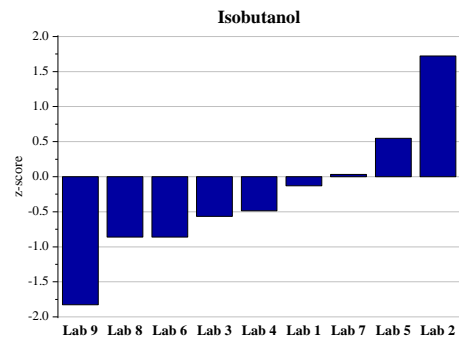
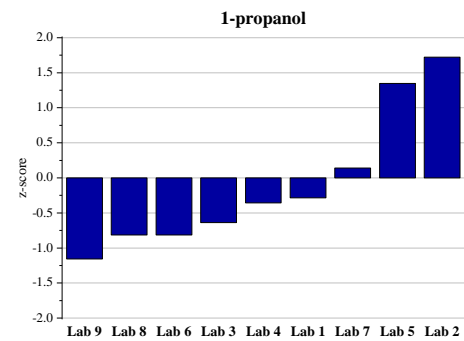
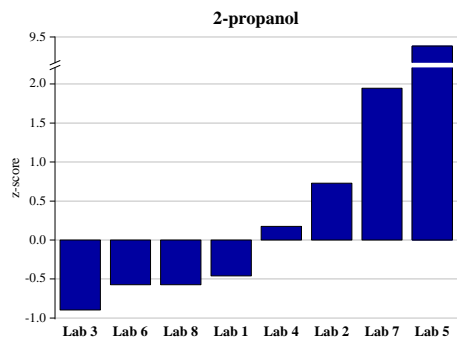
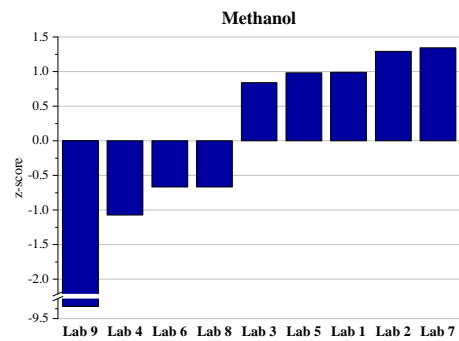
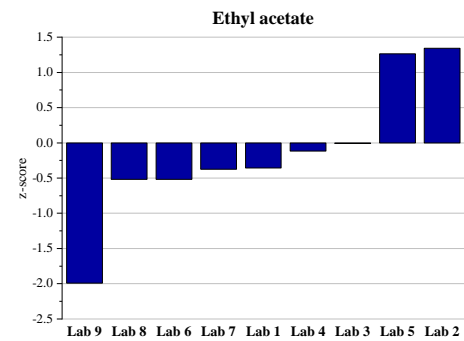
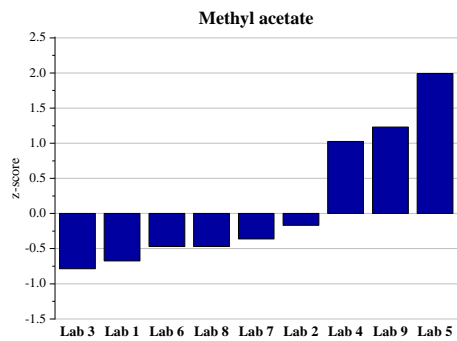
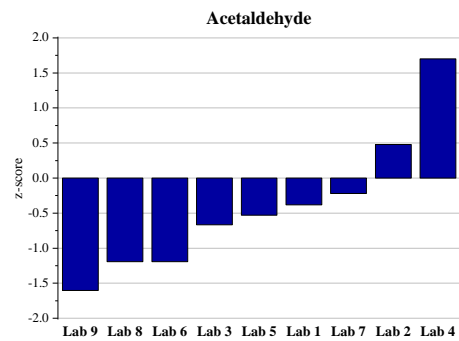


Figure S.1. Z-scores SS-B

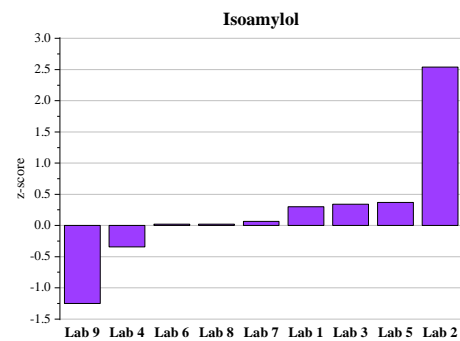
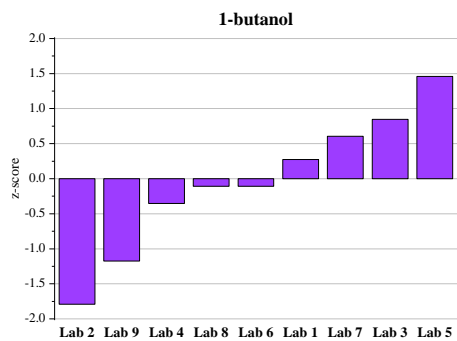
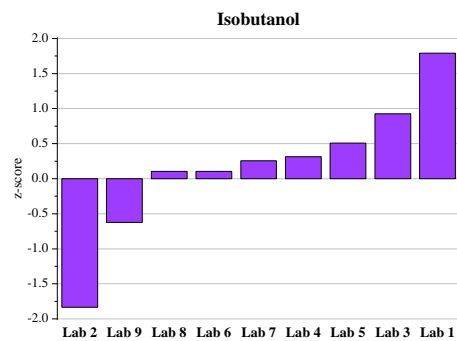
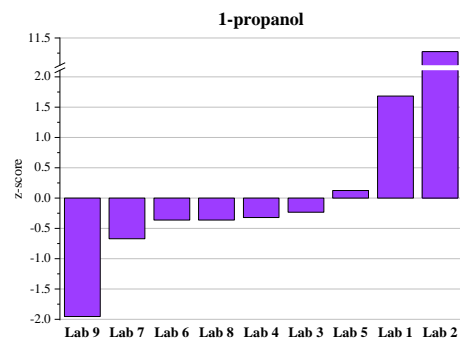
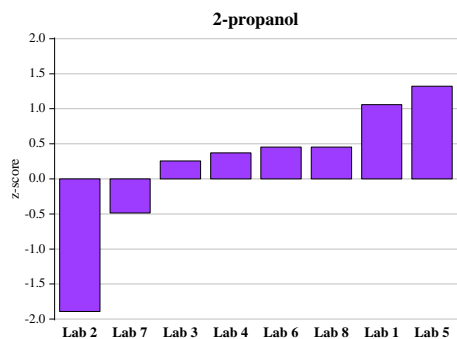
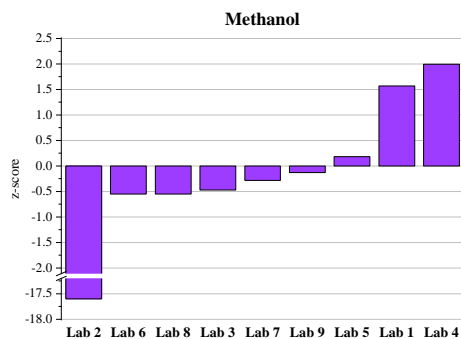
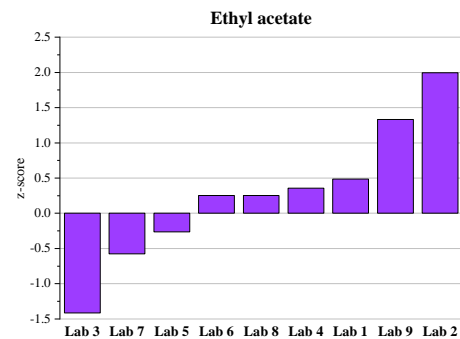
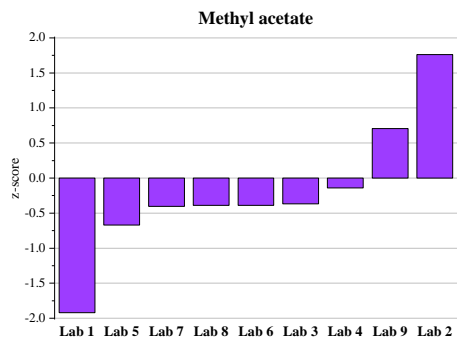
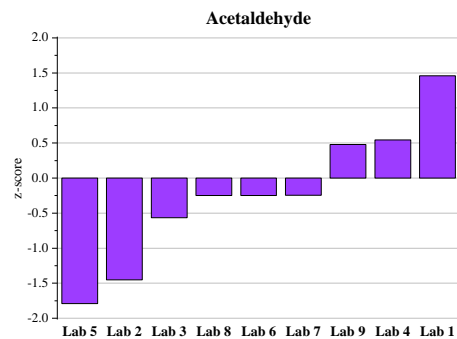


Figure S.2. Z-scores SS-D

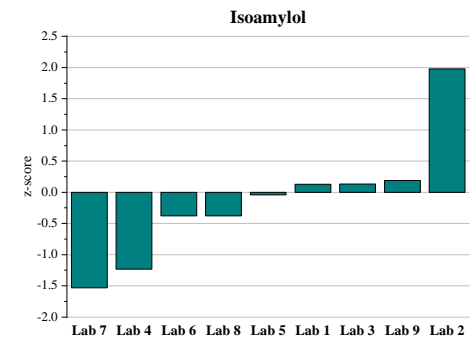
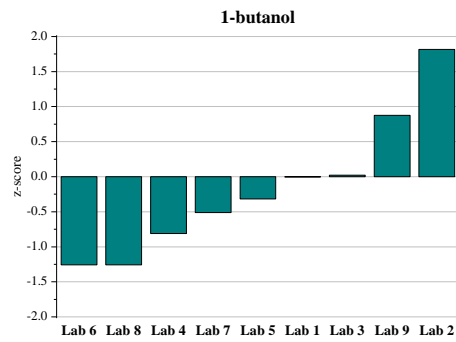
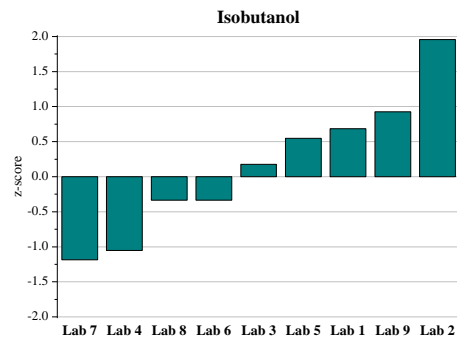
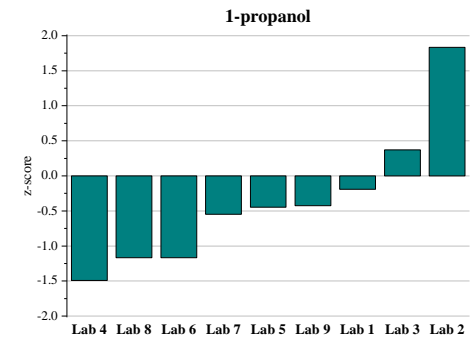
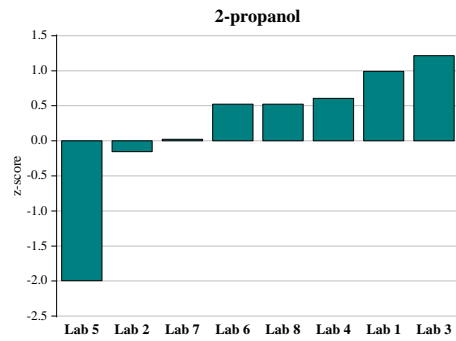
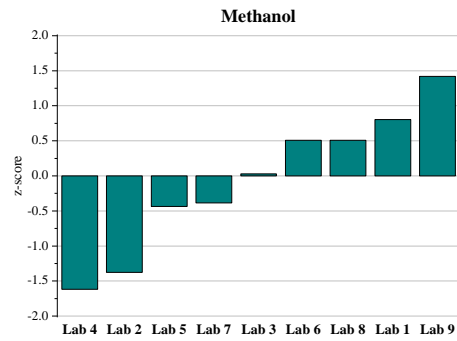
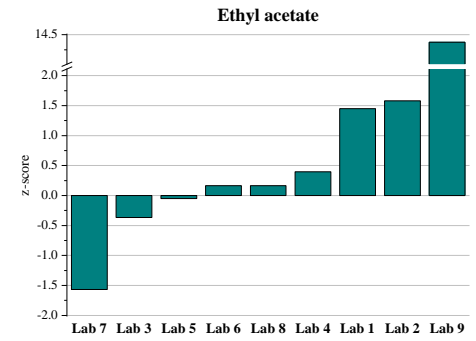
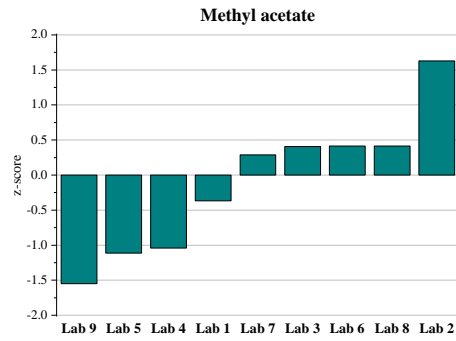
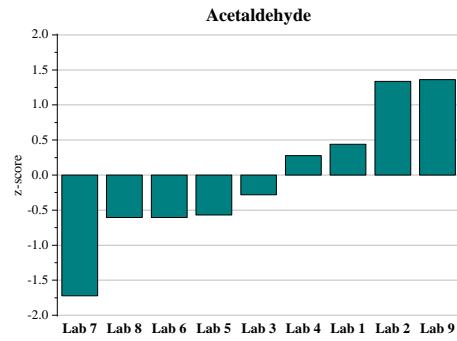


Figure S.3. Z-scores SS-1

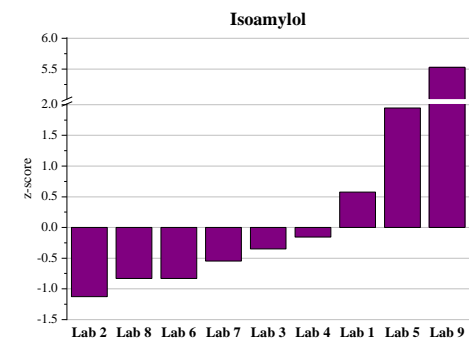
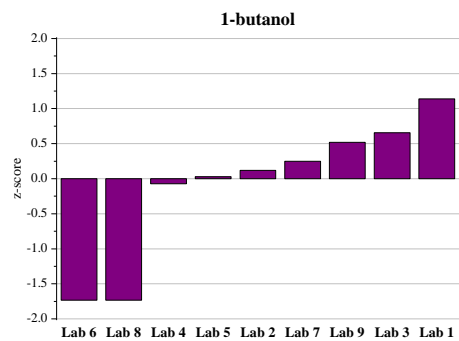
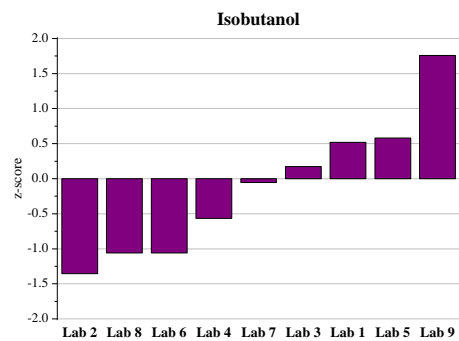
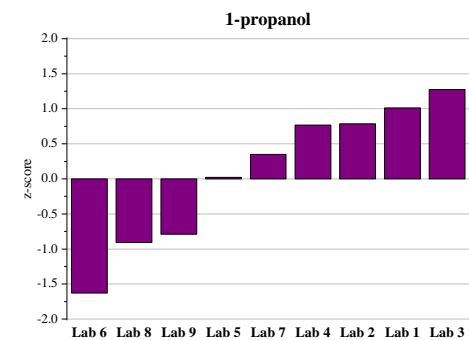
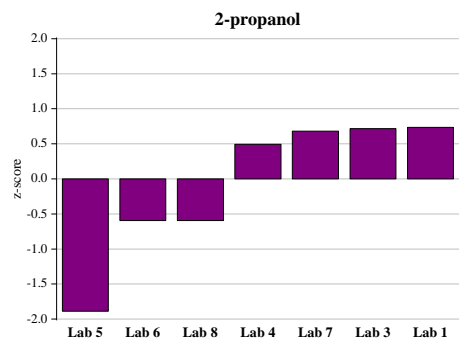
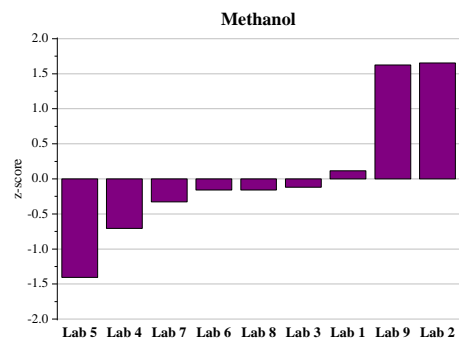
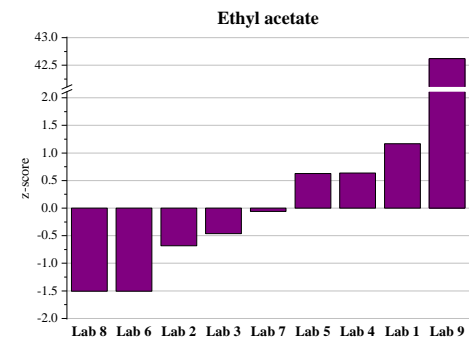
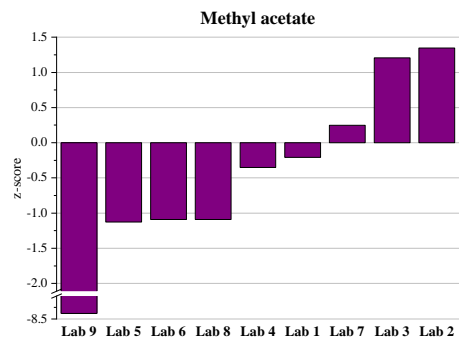
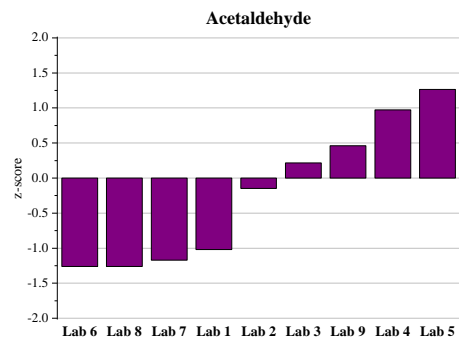


Figure S.4. Z-scores SS-2

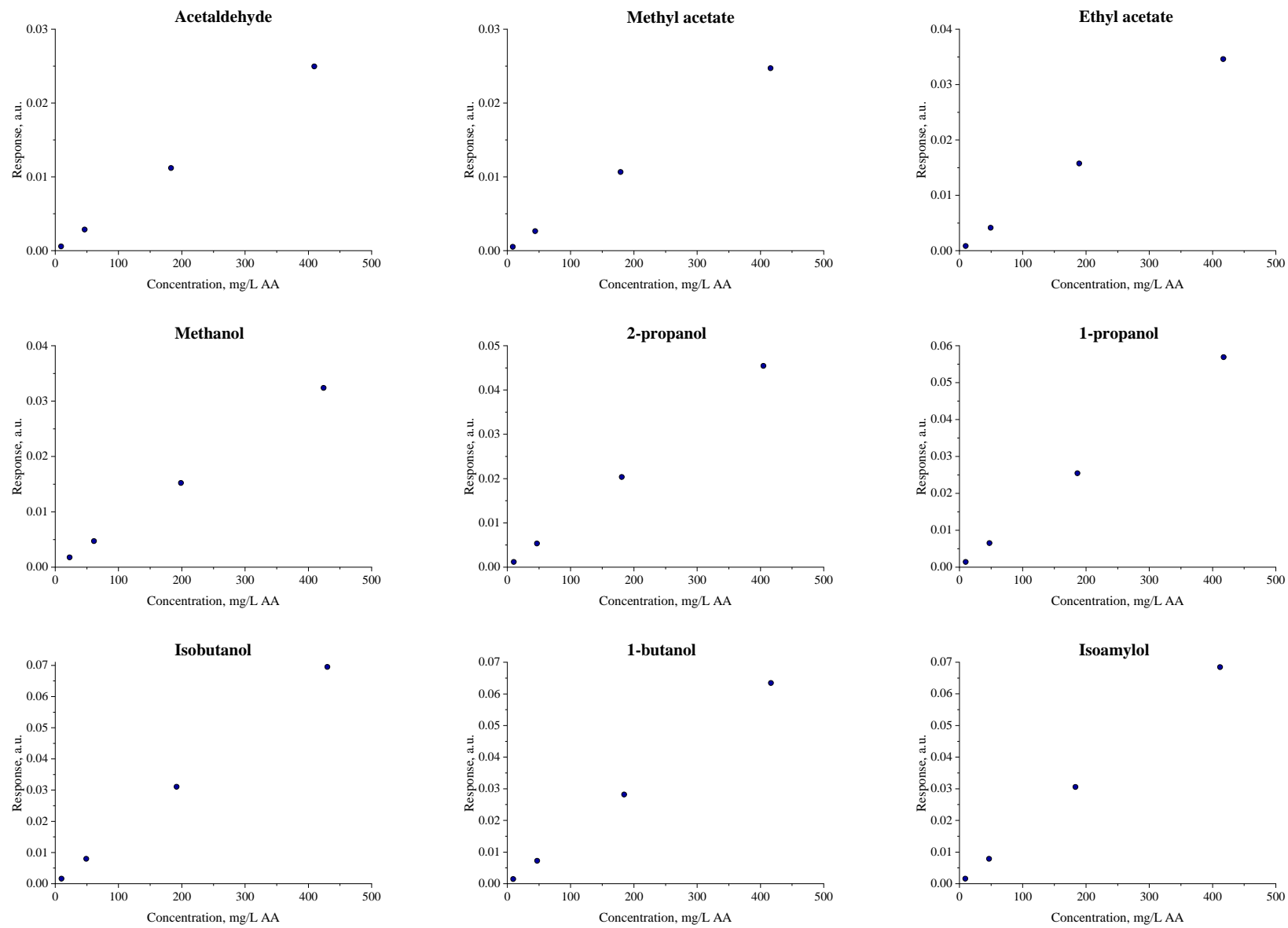


Figure S5. The linearity of the method in laboratory 1

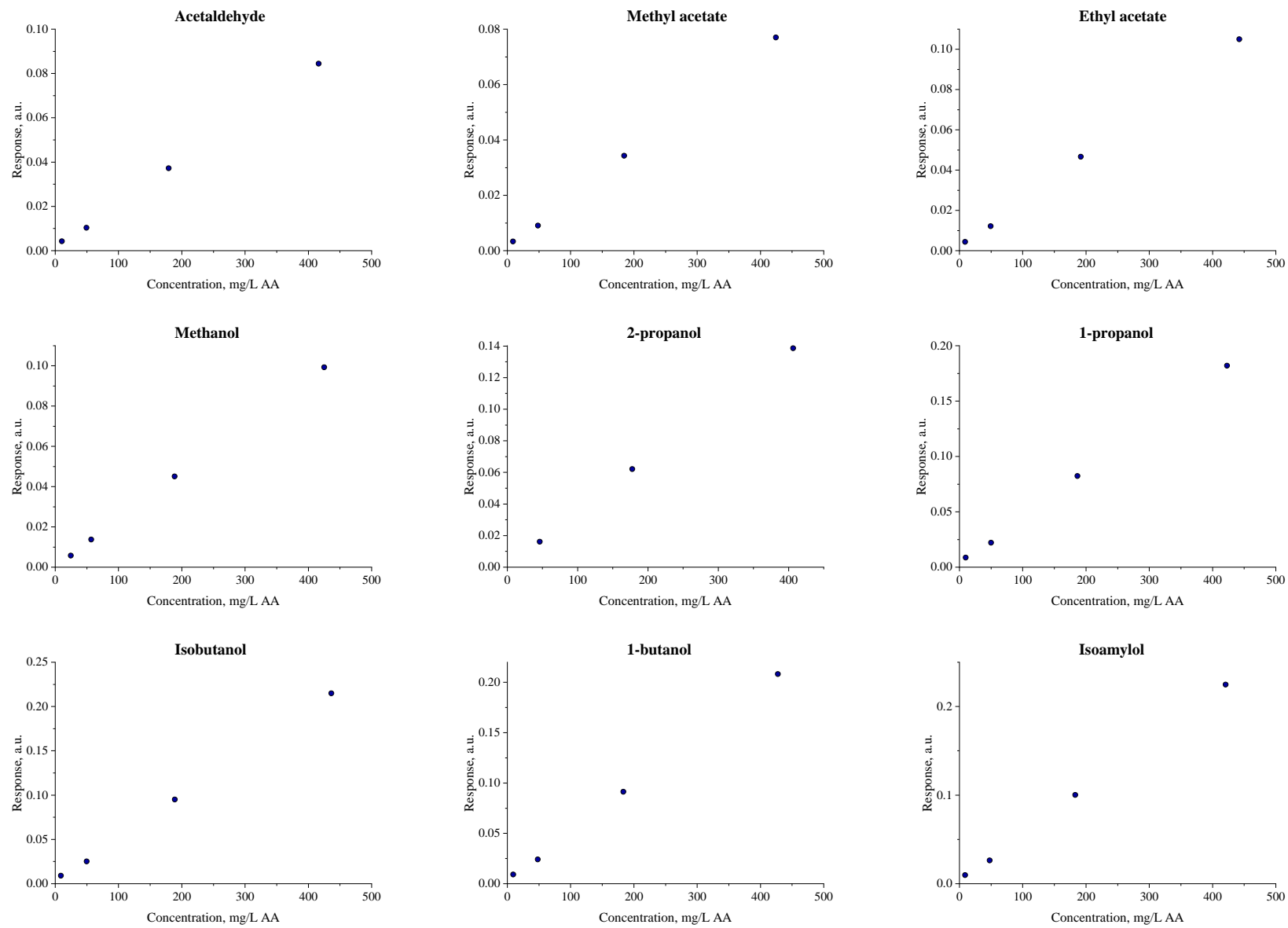


Figure S6. The linearity of the method in laboratory 2

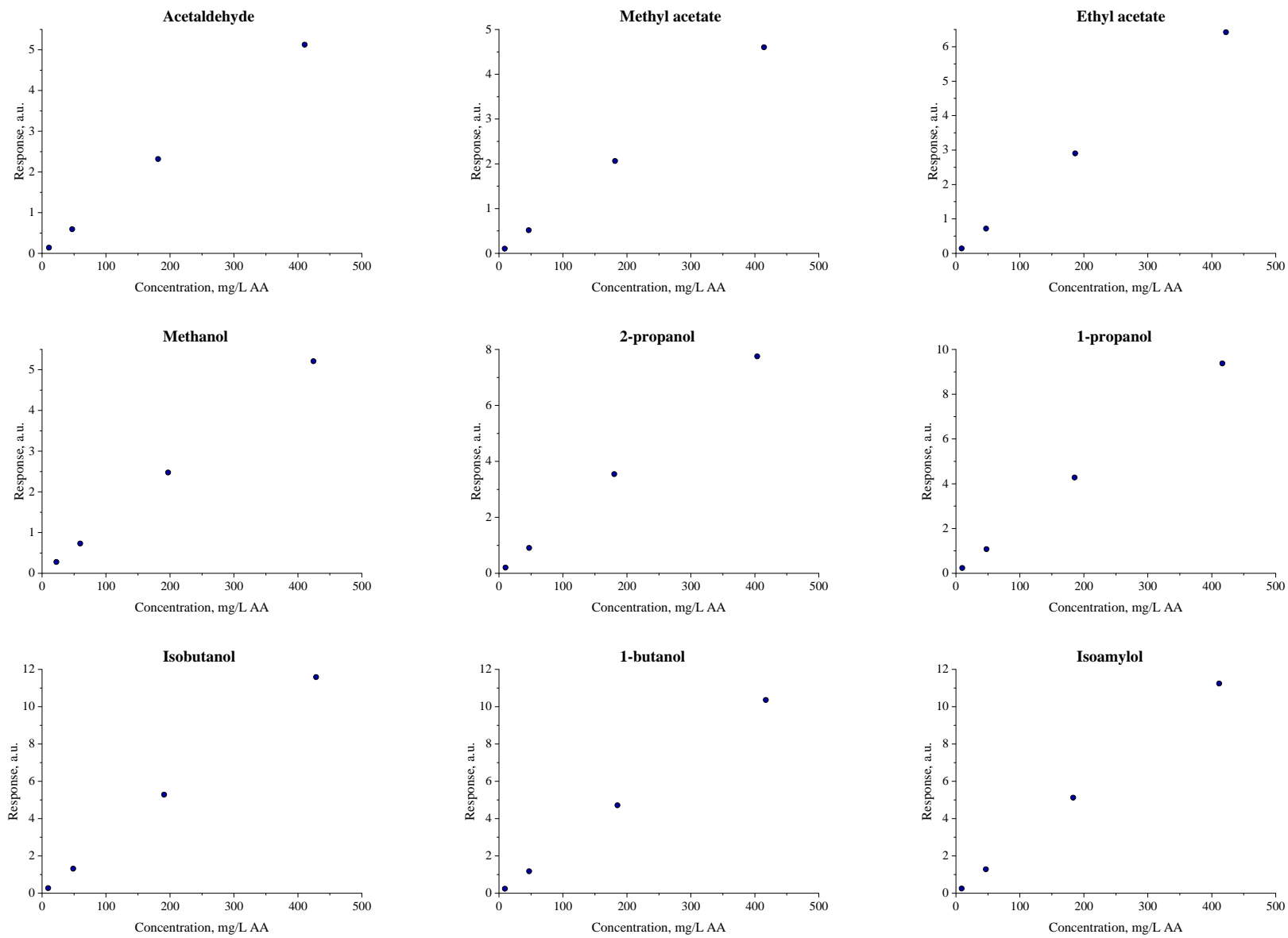


Figure S7. The linearity of the method in laboratory 3

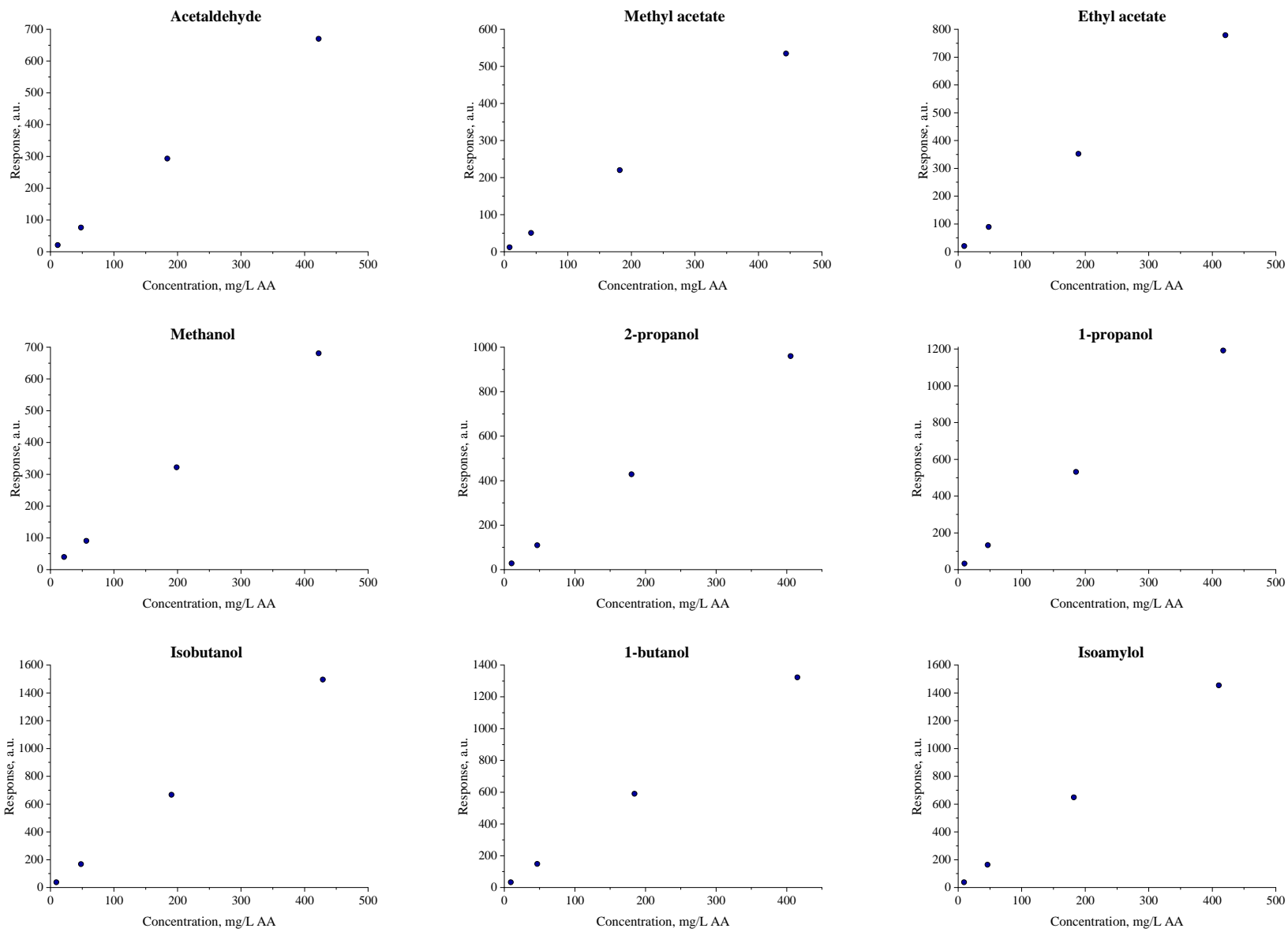


Figure S8. The linearity of the method in laboratory 4

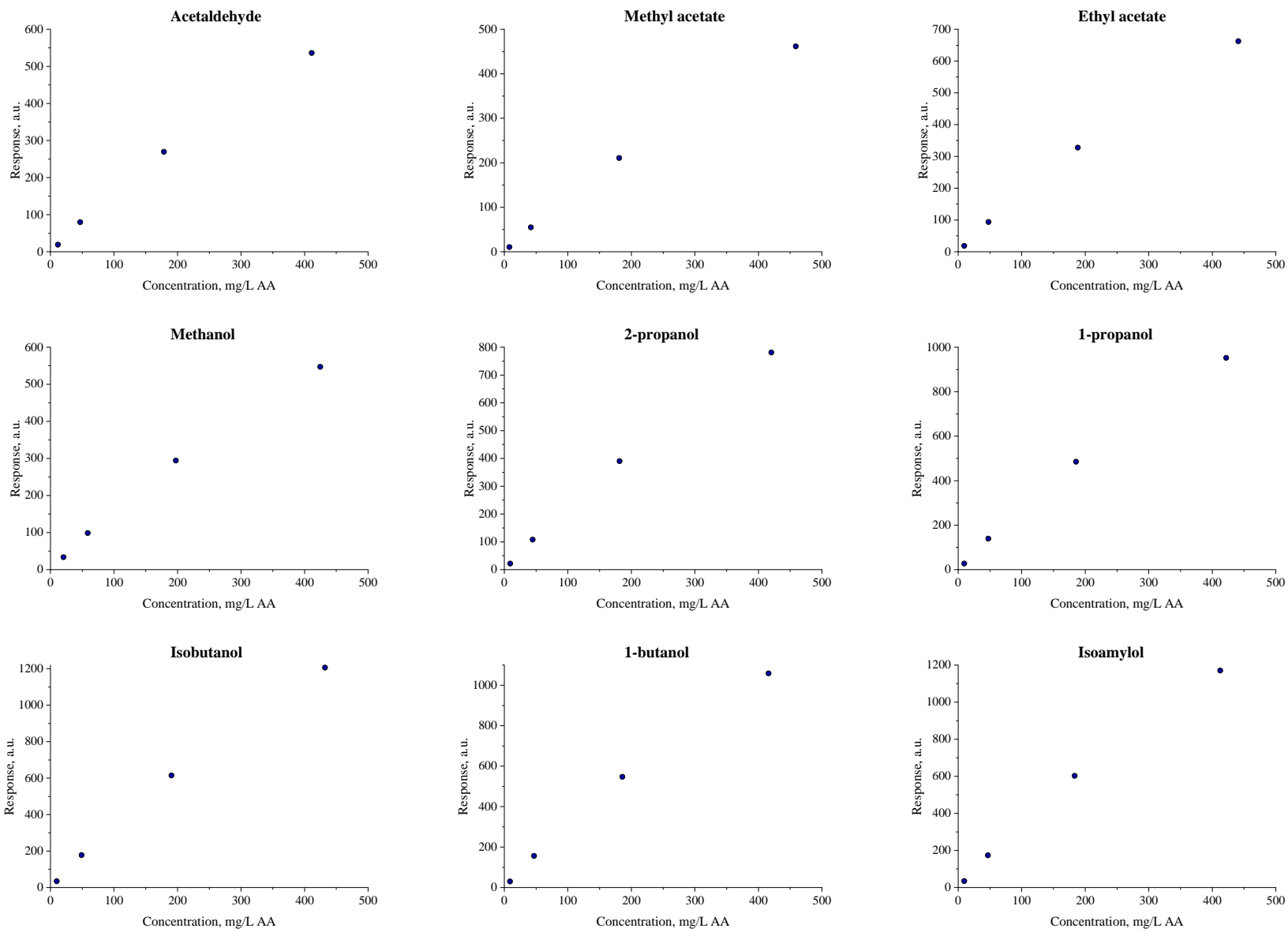


Figure S9. The linearity of the method in laboratory 5

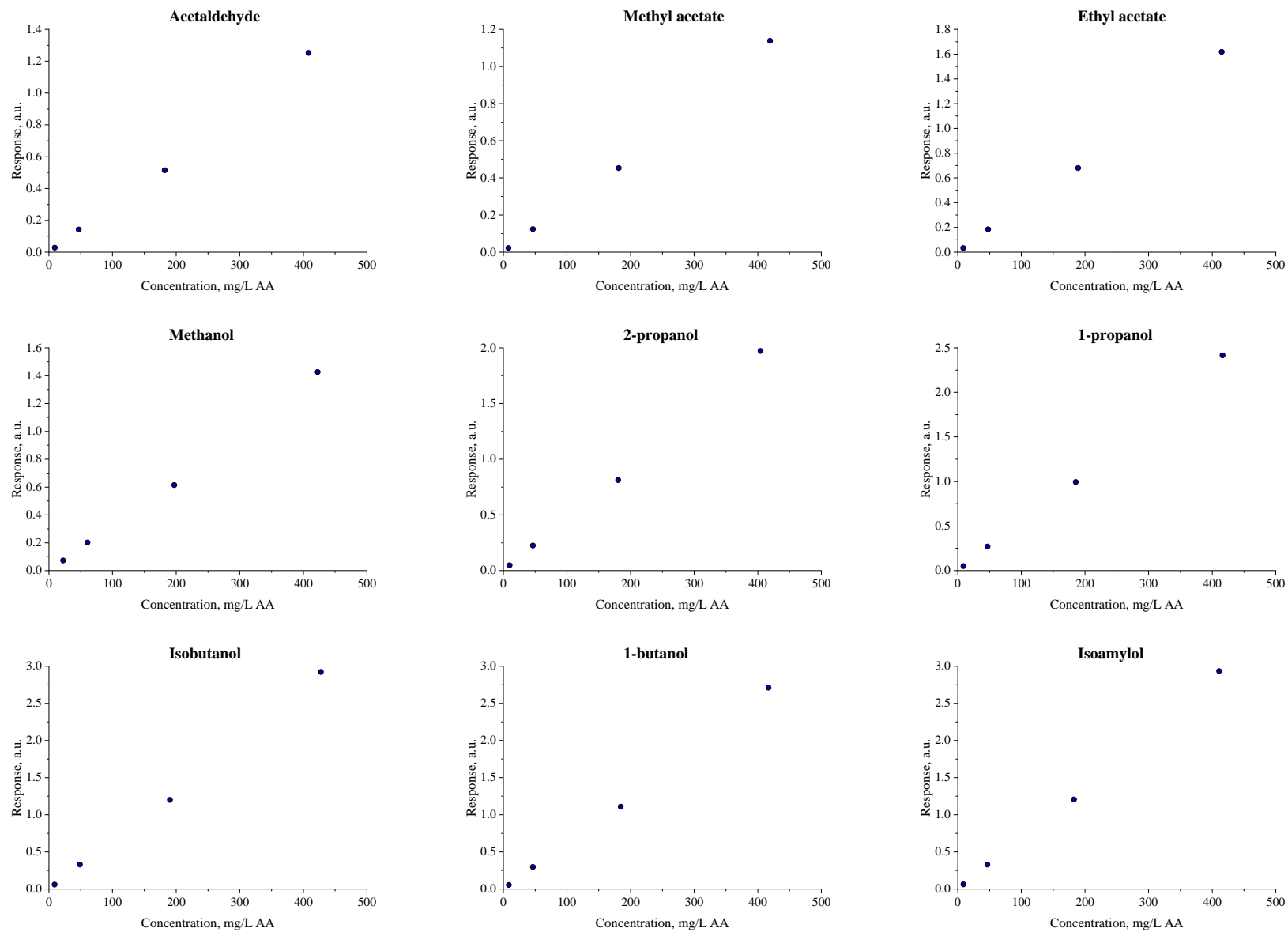


Figure S10. The linearity of the method in laboratory 6

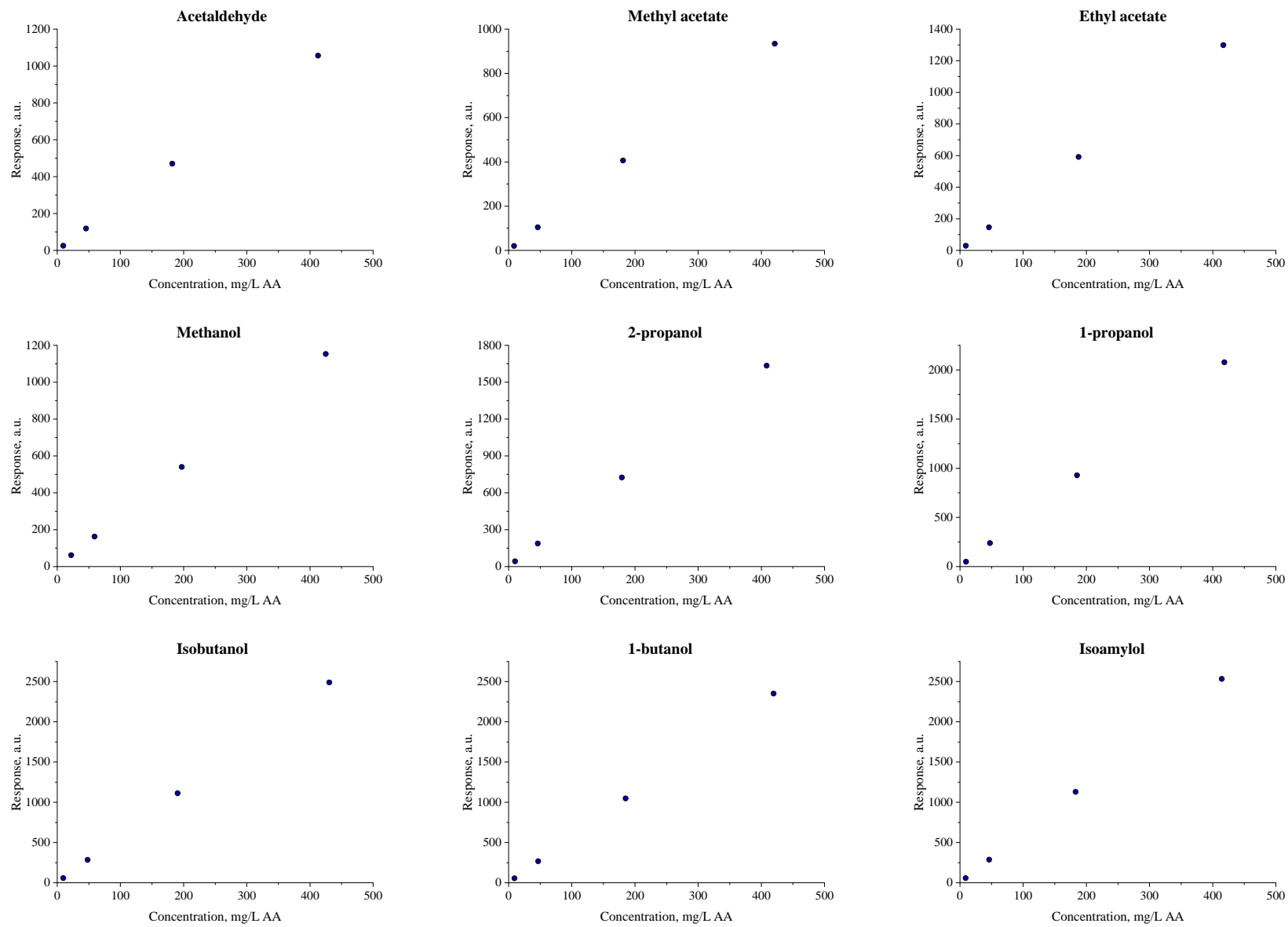


Figure S11. The linearity of the method in laboratory 7

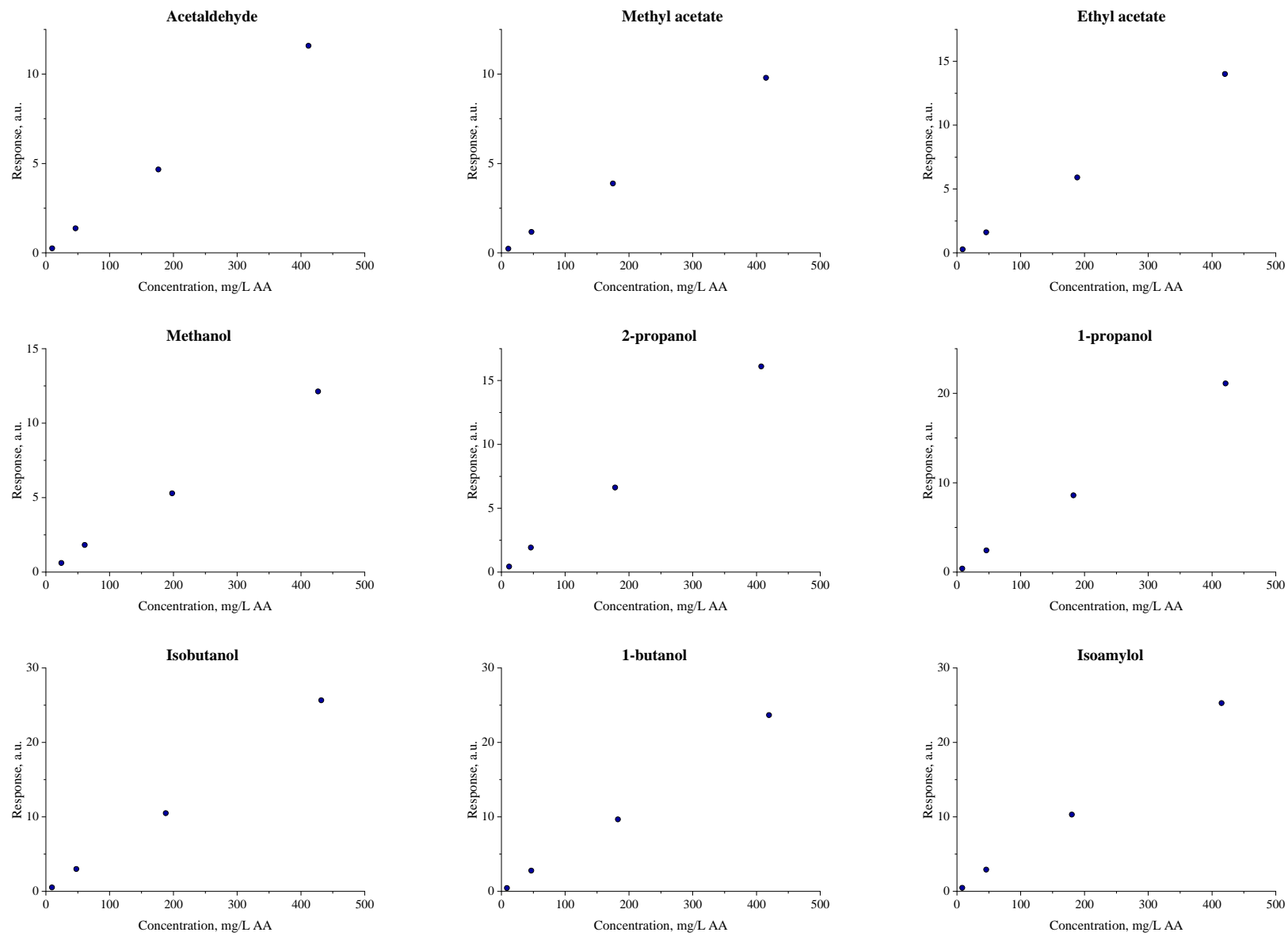


Figure S12. The linearity of the method in laboratory 8

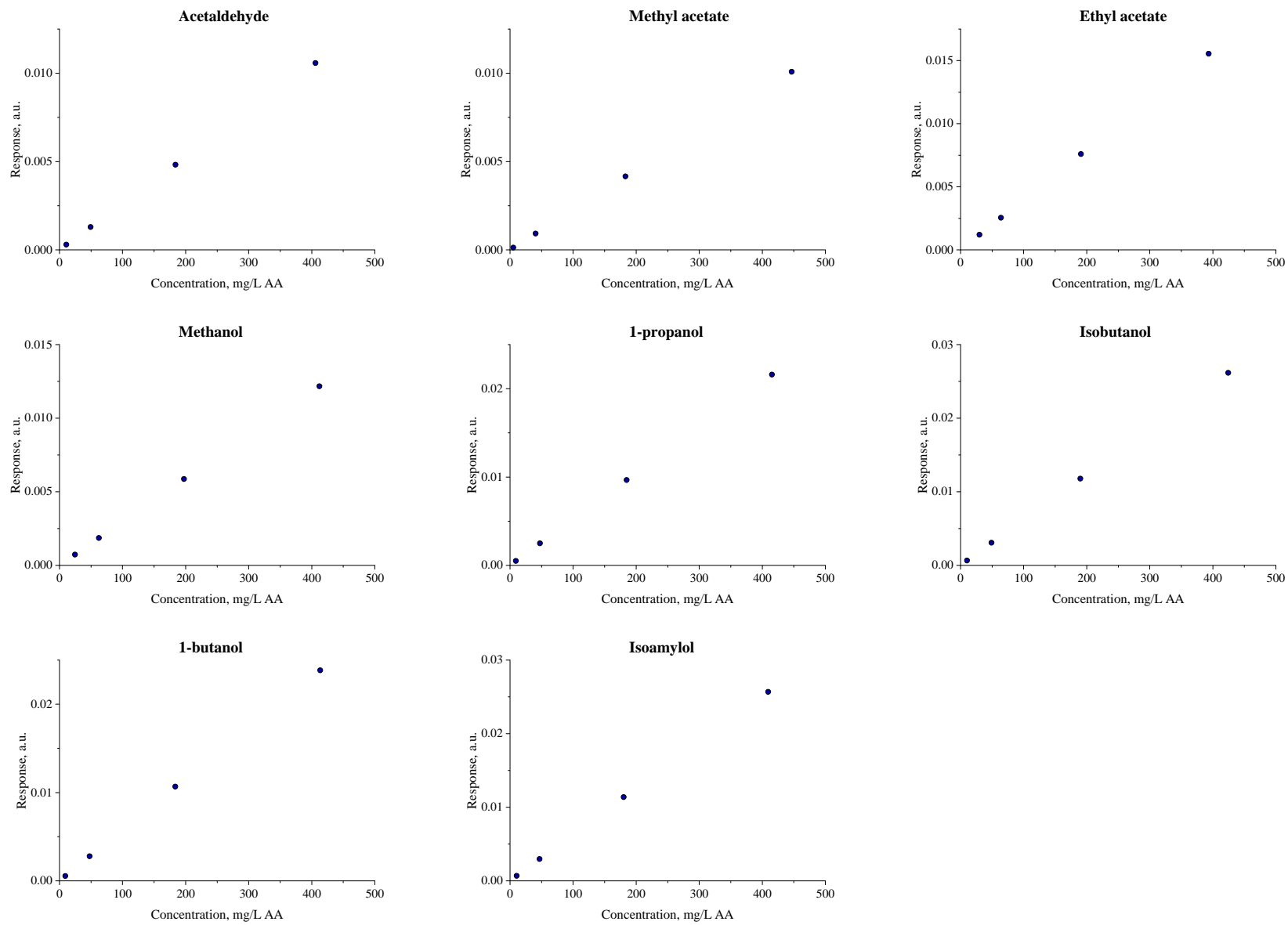


Figure S13. The linearity of the method in laboratory 9