Supplementary Information for:

Methane Emissions and Rumen Metabolite Concentrations in Cattle Fed Two Different Silages.

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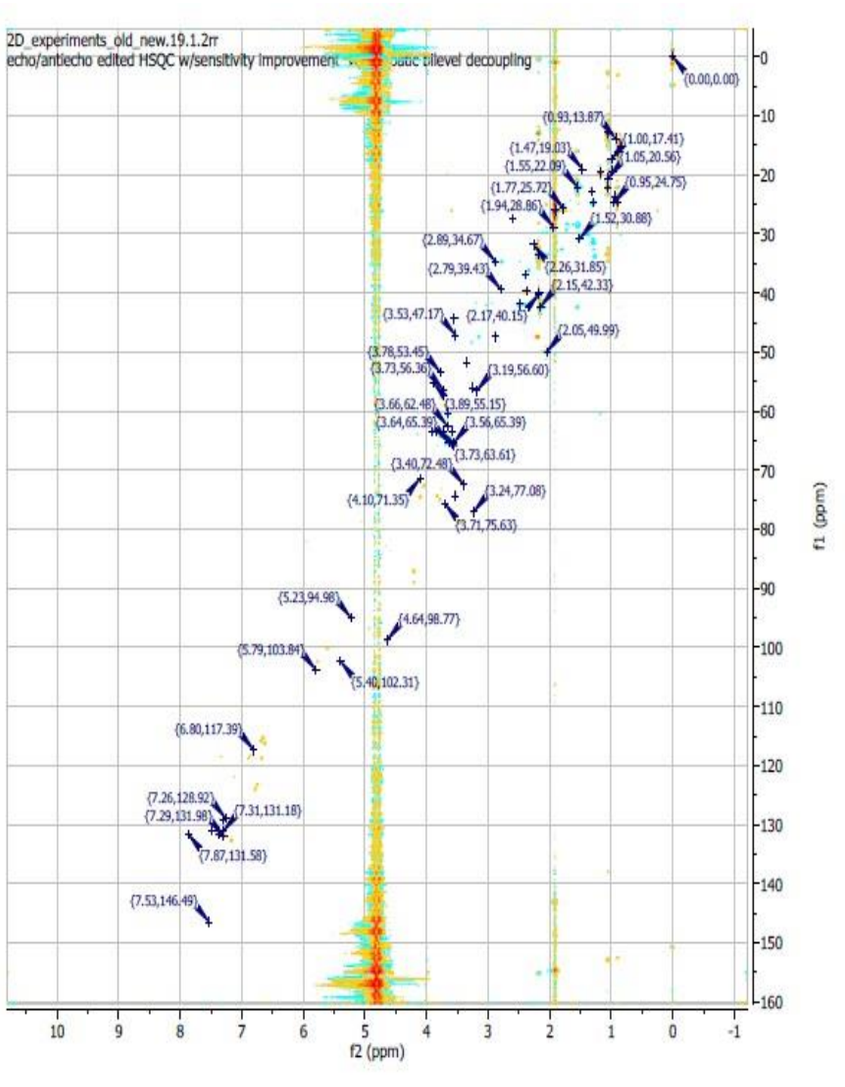
This PDF file includes:

	Parameter	Value
1	Data File Name	D:/ 2D_experiments_old_new/ 19/ pdata/ 1/ 2rr
2	Title	2D_experiments_old_new. 19.1.2rr
3	Comment	echo/ antiecho edited HSQC w/ sensitivity improvement w/ adiabatic bilevel decoupling
4	Origin	Bruker BioSpin GmbH
5	Owner	chemist
6	Site	
7	Instrument	Neo800
8	Author	
9	Solvent	H2O+D2O
10	Temperature	298.0
11	Pulse Sequence	hsqcedetgpsisp2.3
12	Experiment	HSQC-EDITED
13	Probe	Z168389_0002 (CP2.1 TCI 80056 H-C/ N-D-05 Z XT)
14	Number of Scans	48
15	Receiver Gain	64.0
16	Relaxation Delay	1.5000
17	Pulse Width	12.3200
18	Presaturation Frequency	
19	Acquisition Time	0.1065
20	Acquisition Date	2020-01-19T15:27:43
21	Modification Date	2020-01-19T15:27:32
22	Class	
23	Spectrometer Frequency	(799.57, 201.07)
24	Spectral Width	(9615.4, 33181.3)
25	Lowest Frequency	(-968.2, -956.4)
26	Nucleus	(1H, 13C)
27	Acquired Size	(1024, 1024)
28	Spectral Size	(4096, 2048)
29	Digital Resolution	(2.35, 16.20)



Supplementary Figure 1S. 2D HSQC spectra with the ^{1}H and ^{13}C chemical shifts of the identified metabolites

Supplementary Table 1S. Metabolites identified by 2D ^{1}H-^{13}C HSQC and ^{1}H-^{1}H TOSCY spectra and their respective ^{1}H and ^{13}C chemical shifts and their RANCM Rank (Joesten and Kennedy, 2019)[(1)]

Compound	HMDB ^{1}H and ^{13}C Chemical Shifts	2D HSQC/TOCSY ^{1}H and ^{13}C Chemical Shifts	COLMARm ^{1}H and ^{13}C Chemical Shifts	RANCM Rank
1. Acetate	1.90/26.08	1.91/26.04	1.90/25.99	3S
2. Butyrate	2.14/42.34, 1.54/22.07, 0.91/15.91	2.15/42.33, 1.55/22.09, 0.88/15.96	2.14/42.33, 1.55/22.08, 0.88/15.93	3
3. Propionate	2.16/33.48, 1.03/12.98	2.17/33.46, 1.04/12.90	2.17/33.41, 1.04/12.89	3
4. Methanol	3.37/51.43	3.35/51.76	3.34/51.57	3S
5. Isobutyrate	1.04/22.00, 2.37/39.57	1.05/22.09, 2.38/39.67	1.05/22.10, 2.37/39.67	3S
6. Ethanol	1.17/19.59, 3.64/60.30	1.17/19.59, 3.65/60.30	1.17/19.49, 3.64/60.13	3
7. Isovalerate	0.89/24.67, 1.93/28.84, 2.03/49.90	0.90/24.67, 1.94/28.86, 2.05/49.99	0.90/24.59, 1.94/28.89, 2.04/49.94	3
8. Leucine	0.94/23.57, 0.95/24.79, 1.70/26.77, 1.70/42.59, 3.74/56.21	0.95/23.62, 0.95/24.75, No peak, No peak, 3.73/56.36	0.94/23.59, 0.96/24.75, 1.70/26.87, 1.70/42.53, 3.72/56.11	3
9. Uracil	5.79/103.71, 7.48/146.25, 7.56/146.25	5.79/103.84, 7.53/146.49, No shift	5.79/103.80, 7.53/146.26, No shift	3
10. 3-PP	2.48/41.87, 2.87/34.56, 7.25/128.79, 7.30/131.04, 7.35/131.16	2.48/41.92, 2.89/34.67, 7.26/128.92, 7.31/131.18, 7.36/131.10	2.48/41.87, 2.88/34.56, 7.26/128.79, 7.30/131.04, 7.36/131.16	3
11. Alanine	1.49/19.02, 3.77/53.55	1.47/19.03, 3.78/53.45	1.47/18.87, 3.77/53.22	3
12. Phenylacetate	3.52/47.17, 7.29/131.85, 7.29/129.15, 7.37/131.51	3.53/47.17 7.29/131.98 7.30/129.24 7.36/131.50	3.52/47. 17 7.29/129.16 7.29/131.89 7.37/131.37	3
13. Glucose	3.23/76.95, 3.39/72.34, 3.52/74.19, 3.73/63.35, 3.89/63.47, 4.63/98.71, 5.22/94.93	3.24/77.08, 3.340/72.48, 3.53/74.34, 3.73/63.61, 3.90/63.53 4.64/98.77, 5.23/94.97	N.A, N.A, N.A, N.A, N.A 4.64/98.64, 5.22/94.83	3
14. Glutamate	1.77/25.65, 2.17/39.90	1.77/25.72, 2.17/39.91	1.78/25.72, 2.17/40.05	3
15. Valine	0.97/19.40, 1.03/20.75, 2.25/31.89, 3.59/ 63.35	0.98/19.35, 1.04/20.56, 2.26/31.85, 3.58/63.37	0.90/19.37, 1.03/20.70, 2.27/31.83, 3.60/63.08	3
16. Valerate	0.84/15.82, 0.90/15.82, 1.28/24.62, 1.51/30.77, 2.16/40.06	0.85/15.40, Peak overlap, 1.29/24.75, 1.52/30.88, 2.17/40.15	0.88/15.89, No peak, 1.29/24.75, 1.51/30.85, 2.17/40.12	3
17. Methylamine	2.59/27.60	2.59/27.49	N.A	3S
18. Trimethylamine	2.88/47.47	2.88/47.41	N.A	3S
19. Succinate	2.38/36.82	2.40/36.85	N.A	3S
20. Isoleucine	0.92/13.91, 0.99/17.36, 1.45/26.99, 1.96/38.68, 3.65/62.52	0.93/13.87, 1.00/17.41, No peak, No peak, 3.66/62.48	N.A	3
21. Lactate	1.31/22.90, 4.10/71.37	1.32/22.90, 4.10/71.35	N.A	3
22. Glycine	3.54/44.30	3.55/44.34	N.A	3S
23. Maltose	3.57/74.38, 3.58/77.25, 3.62/79.41, 3.67/75.67, 3.71/75.44,	No peak, No peak, No peak, No peak, 3.70/75.63,	N.A	1

	3.80/63.29, 3.83/63.23, 4.64/98.50, 5.22/94.64, 5.39/102.28	No peak, 3.83/63.45, Peak overlap, Peak overlap, 5.40/102.31		
24. Benzoate	7.47/130.97 7.86/131.44 7.64/133.95	7.47/131.10, 7.87/131.58, No peak	N.A	2
25. Aspartate	2.71/39.33, 2.80/39.47, 3.90/55.08	No peak, 2.79/39.43, 3.89/55.15	N.A	2
26. Dimethylamine	2.71/37.29	N.A	N.A	1S
27. 2-Hydroxyvalerate	N.A	N.A	N.A	N.A
28. Creatinine	3.03/32.97, 4.05/59.23	N.A	N.A	1
29. Xanthine	7.92/140.43	N.A	N.A	1S
30. Formate	8.39/172.41	N.A	N.A	1S
31. Acetoacetate	2.27/32.24, 3.43/56.18	N.A	N.A	1
32. 1,3-Dihydroxyacetone	4.40/67.49	N.A	N.A	1S
33. Alloisoleucine	0.88/16.00, 0.95/13.81, 0.96/16.05, 1.33/28.08, 1.42/28.18, 2.05/38.32, 3.73/61.39	N.A	N.A	1
34. Caffeine	3.41/27.99, 3.59/29.61, 3.99/33.73, 7.50/141.41	N.A	N.A	1
35. Malonate	3.11/50.23	N.A	N.A	1S
36. Choline	3.18/56.69, 3.50/70.14, 4.05/58.49	3.19/56.60, No peak, No peak,	N.A	1
37. Betaine	3.25/55.85, 3.88/68.64	3.25/56.03, No peak	N.A	1
38. Creatine	3.02/39.50, 3.91/56.43	N.A	N.A	1
39. Fumarate	6.50/138.00	N.A	N.A	1S
40. Acetamide	2.00/22.11	N.A	N.A	1S
41. 3-Hydroxyphenylacetate	3.46/47.00, 6.78/116.16, 6.78/118.81, 6.84/124.10, 7.24/132.64	No peak, 6.80/117.39, Peak overlap, No peak, No peak	N.A	1
42. Fructose	3.55/66.83, 3.54/65.41, 3.66/65.59, 3.78/70.42, 3.88/72.53, 3.98/72.14, 4.10/77.42	No peak, 3.56/65.39, 3.64/65.34, No peak, No peak, No peak, No peak	N.A	1

Extra References

1. Joesten, W. C. & Kennedy, M. A. RANCM: a new ranking scheme for assigning confidence levels to metabolite assignments in NMR-based metabolomics studies. *Metabolomics.* **15**(1) doi: 10.1007/s11306-018-1465-2 (2019).