

Table S1 – Variation results for both lysine (Lys) and internal standard (IS) of samples that were stored after derivatization, at 24 h, 48 h and 96 h of storage at room temperature (RT), 4 °C and -20 °C for Li₂CO₃ buffer, phosphate buffer solution (PBS) and rumen inoculum, n = 5. Recovery was determined by dividing the peak area at each time point by the initial peak area (before storage) and the values are shown as mean ± standard deviation.

Matrix	24 h		48 h		96 h	
	Lys	IS	Lys	IS	Lys	IS
Recovery (%) when stored at RT						
Li₂CO₃	98 ± 7	99 ± 6	69 ± 4	91 ± 4	70 ± 4	93 ± 6
PBS	84 ± 2	84 ± 2	88 ± 4	88 ± 4	23 ± 8	22 ± 8
Rumen	101 ± 5	101 ± 6	91 ± 8	92 ± 8	113 ± 6	115 ± 6
Recovery (%) when stored at 4 °C						
Li₂CO₃	101 ± 5	100 ± 5	83 ± 12	88 ± 26	84 ± 16	68 ± 40
PBS	84 ± 1	87 ± 2	83 ± 6	83 ± 6	87 ± 8	87 ± 8
Rumen	99 ± 2	100 ± 4	93 ± 7	92 ± 7	105 ± 13	78 ± 13
Recovery (%) when stored at -20 °C						
Li₂CO₃	51 ± 12	21 ± 20	37 ± 4	10 ± 3	38 ± 3	11 ± 2
PBS	94 ± 14	94 ± 6	36 ± 7	9 ± 6	34 ± 16	9 ± 2
Rumen	37 ± 6	8 ± 1	32 ± 8	12 ± 4	32 ± 14	9 ± 5

Table S2 – Variation results for both lysine (Lys) and internal standard (IS) of samples that were stored prior to derivatization, at 24 h, 48 h and 96 h of storage at room temperature (RT), 4 °C and -20 °C for Li₂CO₃ buffer, phosphate buffer solution (PBS) and rumen inoculum, n = 5. Recovery was determined by dividing the peak area at each time point by the initial peak area (before storage) and the values are shown as mean ± standard deviation.

Matrix	24 h		48 h		96 h	
	Lys	IS	Lys	IS	Lys	IS
Recovery (%) when stored at RT						
Li₂CO₃	227 ± 23	56 ± 6	80 ± 8	17 ± 2	144 ± 19	10 ± 10
PBS	82 ± 9	39 ± 4	106 ± 7	18 ± 1	78 ± 24	70 ± 19
Rumen	133 ± 24	2 ± 1	94 ± 8	1 ± 1	4 ± 5	5 ± 6
Recovery (%) when stored at 4 °C						
Li₂CO₃	177 ± 11	90 ± 6	82 ± 4	63 ± 3	118 ± 4	55 ± 2
PBS	93 ± 5	80 ± 3	104 ± 7	62 ± 4	74 ± 7	39 ± 4
Rumen	42 ± 4	31 ± 4	44 ± 3	59 ± 4	136 ± 31	2 ± 1
Recovery (%) when stored at -20 °C						
Li₂CO₃	150 ± 14	98 ± 7	95 ± 22	97 ± 6	116 ± 16	97 ± 7
PBS	96 ± 14	96 ± 7	111 ± 9	91 ± 7	78 ± 24	70 ± 19
Rumen	137 ± 28	94 ± 17	145 ± 26	103 ± 16	160 ± 31	97 ± 17