

Tables S1 and S2: Analytical results for all amino acids measured in the collagen samples of this study. Isotopic values are an average of triplicate measurements, the \pm values are the standard deviation of the triplicate measurements.

Table S1: Carbon isotope results for all samples

S-SFU #	2690	2692	2693	2694	2695	2696	2697	2698	2699	2701	2703	2704
species	pike	human	human	felis	felis	canis	bos	bos	cervus	panthera	equus	equus
Bulk $\delta^{13}\text{C}$ (‰)	-22.6	-20.3	-20.6	-20.9	-20.3	-18.8	-20.6	-19.9	-20.4	-18.2	-20.5	-20.4
alanine	-24.7 \pm 0.1	-23.9 \pm 0.1	-23.3 \pm 0.2	-22.2 \pm 0.1	-23.8 \pm 0.1	-22.9 \pm 0.1	-26.0 \pm 0.1	-25.6 \pm 0.1	-24.8 \pm 0.0	-21.3 \pm 0.1	-22.7 \pm 0.1	-22.6 \pm 0.0
valine	-30.1 \pm 0.3	-25.9 \pm 0.2	-26.4 \pm 0.2	-25.0 \pm 0.3	-25.7 \pm 0.2	-25.6 \pm 0.1	-29.2 \pm 0.2	-27.9 \pm 0.1	-28.2 \pm 0.2	-24.0 \pm 0.3	-26.8 \pm 0.1	-26.5 \pm 0.2
glycine	-11.5 \pm 0.2	-13.2 \pm 0.0	-12.0 \pm 0.0	-12.1 \pm 0.1	-13.9 \pm 0.1	-14.5 \pm 0.1	-17.5 \pm 0.1	-16.6 \pm 0.0	-15.6 \pm 0.1	-10.1 \pm 0.1	-14.9 \pm 0.1	-13.9 \pm 0.1
leucine	-33.1 \pm 0.5	-26.3 \pm 0.3	-26.7 \pm 0.2	-30.1 \pm 0.1	-29.8 \pm 0.3	-26.6 \pm 0.8	-28.3 \pm 0.0	-27.7 \pm 0.0	-31.1 \pm 0.1	-28.1 \pm 0.1	-30.1 \pm 0.2	-30.4 \pm 0.2
norleucine	-29.5 \pm 0.1	-29.5 \pm 0.1	-29.6 \pm 0.1	-29.0 \pm 0.0	-29.7 \pm 0.1	-29.7 \pm 0.0	-29.4 \pm 0.0	-30.1 \pm 0.0	-29.7 \pm 0.0	-29.2 \pm 0.1	-29.1 \pm 0.1	-29.0 \pm 0.0
proline	-22.2 \pm 0.1	-19.7 \pm 0.1	-19.8 \pm 0.1	-19.8 \pm 0.1	-20.4 \pm 0.1	-18.7 \pm 0.1	-18.9 \pm 0.1	-18.1 \pm 0.0	-20.8 \pm 0.1	-18.2 \pm 0.1	-19.9 \pm 0.1	-19.9 \pm 0.0
threonine	-18.7 \pm 0.8	-10.2 \pm 0.2	-13.6 \pm 1.8	-9.8 \pm 0.4	-8.2 \pm 0.2	-3.9 \pm 0.1	-7.8 \pm 0.1	-7.6 \pm 0.2	-14.1 \pm 0.2	-9.2 \pm 0.5	-12.9 \pm 0.3	-12.8 \pm 0.1
aspartic acid	-22.2 \pm 0.1	-21.1 \pm 0.1	-20.5 \pm 0.3	-20.4 \pm 0.1	-20.0 \pm 0.2	-20.3 \pm 0.1	-18.0 \pm 0.1	-15.8 \pm 0.0	-19.6 \pm 0.1	-18.0 \pm 0.1	-18.5 \pm 0.1	-18.9 \pm 0.1
serine	-8.9 \pm 0.1	-7.8 \pm 0.3	-5.5 \pm 0.1	-11.1 \pm 0.3	-9.2 \pm 0.2	-8.8 \pm 0.1	-9.7 \pm 0.3	-7.2 \pm 0.1	-12.3 \pm 0.0	-8.0 \pm 0.2	-15.0 \pm 0.2	-13.1 \pm 0.1
glutamic acid	-22.5 \pm 0.1	-21.0 \pm 0.1	-20.5 \pm 0.2	-19.7 \pm 0.1	-20.7 \pm 0.2	-18.8 \pm 0.1	-19.1 \pm 0.1	-18.5 \pm 0.1	-19.3 \pm 0.0	-18.5 \pm 0.0	-18.4 \pm 0.1	-18.7 \pm 0.1
phenylalanine	-33.3 \pm 0.6	-29.0 \pm 0.4	-29.9 \pm 0.2	-26.5 \pm 0.4	-26.6 \pm 0.2	-26.0 \pm 0.3	-27.4 \pm 0.2	-26.9 \pm 0.3	-26.6 \pm 0.4	-24.8 \pm 0.3	-25.3 \pm 0.2	-25.3 \pm 0.3
hydroxyproline	-22.9 \pm 0.2	-20.7 \pm 0.0	-20.4 \pm 0.1	-20.0 \pm 0.0	-20.4 \pm 0.1	-18.9 \pm 0.1	-19.8 \pm 0.1	-19.9 \pm 0.0	-21.2 \pm 0.1	-18.2 \pm 0.0	-19.7 \pm 0.2	-19.8 \pm 0.0
lysine	-21.3 \pm 1.4	-20.9 \pm 0.1	-20.2 \pm 0.4	-22.2 \pm 0.3	-19.1 \pm 0.3	-18.8 \pm 0.4	-18.8 \pm 0.1	-17.5 \pm 0.2	-22.3 \pm 0.1	-21.0 \pm 0.5	-22.7 \pm 0.5	-22.5 \pm 0.3

Table S2: Nitrogen isotope results for all samples

S-SFU #	2690	2692	2693	2694	2695	2696	2697	2698	2699	2701	2703	2704
species	pike	human	human	felis	felis	canis	bos	bos	cervus	panthera	equus	equus
Bulk $\delta^{15}\text{N}$ (‰)	9.2	13.6	14.0	8.6	6.5	12.2	8.0	7.8	4.6	10.1	6.7	4.9
alanine	23.7 \pm 0.6	18.6 \pm 0.1	20.5 \pm 0.4	12.7 \pm 0.2	9.7 \pm 0.1	15.7 \pm 0.2	8.8 \pm 0.6	8.7 \pm 0.3	5.6 \pm 0.7	13.2 \pm 0.2	6.4 \pm 0.1	4.3 \pm 0.4
glycine	1.7 \pm 0.1	13.3 \pm 0.7	12.6 \pm 0.4	5.4 \pm 0.7	3.1 \pm 0.0	8.8 \pm 0.1	6.6 \pm 0.1	5.6 \pm 0.2	2.4 \pm 0.3	6.2 \pm 0.3	2.6 \pm 0.3	2.0 \pm 0.0
valine	19.6 \pm 1.4	19.5 \pm 0.3	18.6 \pm 0.2	17.3 \pm 1.4	12.0 \pm 0.6	16.0 \pm 0.9	11.9 \pm 0.3	11.9 \pm 0.3	9.2 \pm 0.3	14.5 \pm 0.6	10.0 \pm 0.3	7.7 \pm 0.6
leucine	20.9 \pm 0.7	18.7 \pm 0.8	20.7 \pm 0.1	14.3 \pm 0.7	10.3 \pm 0.9	16.6 \pm 0.3	9.6 \pm 0.6	9.7 \pm 0.8	6.4 \pm 0.4	14.2 \pm 0.3	7.6 \pm 0.5	4.7 \pm 1.0
norleucine	-0.9 \pm 0.2	-1.0 \pm 0.1	-1.9 \pm 0.3	-0.5 \pm 0.1	-2.4 \pm 0.0	-2.0 \pm 0.1	-1.8 \pm 0.6	-2.1 \pm 0.2	-1.3 \pm 0.1	-1.6 \pm 0.2	-1.9 \pm 0.2	-2.0 \pm 0.2
threonine	-20.3 \pm 3.7	-22.5 \pm 0.4	-25.5 \pm 1.0	-14.6 \pm 2.2	-20.3 \pm 0.4	-11.6 \pm 1.3	-7.9 \pm 0.7	-7.2 \pm 1.5	-8.5 \pm 1.4	-18.5 \pm 0.6	-7.7 \pm 0.8	-8.7 \pm 0.8
serine	6.1 \pm 1.0	16.2 \pm 0.6	14.7 \pm 1.5	7.5 \pm 1.0	4.8 \pm 0.9	11.0 \pm 0.9	6.3 \pm 1.1	5.7 \pm 0.3	3.6 \pm 0.2	8.6 \pm 0.6	3.6 \pm 0.3	1.4 \pm 0.3
proline	16.6 \pm 0.5	17.0 \pm 0.3	17.7 \pm 0.3	13.5 \pm 0.6	8.9 \pm 0.4	13.8 \pm 0.1	9.7 \pm 0.7	10.5 \pm 0.2	7.8 \pm 0.3	13.8 \pm 0.4	10.4 \pm 0.5	8.5 \pm 0.6
aspartic acid	17.2 \pm 1.3	17.8 \pm 0.6	19.5 \pm 0.3	13.5 \pm 1.5	9.1 \pm 1.1	15.0 \pm 0.0	10.3 \pm 0.1	9.7 \pm 0.4	7.5 \pm 0.8	14.2 \pm 1.0	9.9 \pm 0.4	7.1 \pm 0.4
glutamic acid	18.7 \pm 1.2	19.5 \pm 0.5	21.1 \pm 0.7	14.3 \pm 0.4	10.1 \pm 0.1	17.2 \pm 0.3	10.5 \pm 0.6	10.9 \pm 0.1	7.8 \pm 0.3	15.3 \pm 1.3	9.0 \pm 0.1	6.1 \pm 0.3
hydroxyproline	16.9 \pm 0.6	16.3 \pm 0.7	17.7 \pm 0.3	13.8 \pm 0.4	8.6 \pm 0.2	13.0 \pm 0.2	9.2 \pm 0.4	9.7 \pm 0.1	7.0 \pm 0.2	13.7 \pm 0.8	10.4 \pm 0.3	8.6 \pm 1.5
phenylalanine	-0.9 \pm 4.0	2.1 \pm 3.8	1.9 \pm 2.4	9.4 \pm 3.6	6.2 \pm 3.9	3.7 \pm 1.7	5.6 \pm 2.0	10.8 \pm 7.5	3.3 \pm 2.4	6.7 \pm 2.0	10.2 \pm 1.5	7.7 \pm 2.4
lysine	2.1 \pm 1.4	5.7 \pm 2.2	5.2 \pm 1.0	4.9 \pm 0.6	1.1 \pm 0.2	5.4 \pm 0.9	1.9 \pm 2.7	3.8 \pm 0.4	2.4 \pm 0.5	6.0 \pm 0.8	3.3 \pm 1.1	1.6 \pm 0.8

Tables S3 and S4: Analytical results for QC collagen standards in comparison with longterm average values. Isotopic values are an average of triplicate measurements, the \pm values are the standard deviation of the triplicate measurements.

Table S3: Carbon isotope results for collagen QC standards (SRM-1, SRM-2, and SRM-3) from the two analytical sessions.

	SRM-1 (seal) $\delta^{13}\text{C}$ (‰)						SRM-2 (deer) $\delta^{13}\text{C}$ (‰)						SRM-3 (fish) $\delta^{13}\text{C}$ (‰)					
	21-Jun-21		29-Jun-21		longterm avg		21-Jun-21		29-Jun-21		longterm avg		21-Jun-21		29-Jun-21		longterm avg	
					(n=23)						(n=22)						(n=21)	
Ala	-17.4	\pm 0.1	-17.0	\pm 0.0	-16.5	\pm 1.3	-29.6	\pm 0.0	-28.2	\pm 0.1	-28.5	\pm 1.8	-20.1	\pm 0.1	-19.5	\pm 0.0	-18.9	\pm 2.0
Gly	-21.3	\pm 0.1	-20.9	\pm 0.2	-19.8	\pm 1.8	-31.0	\pm 0.2	-30.6	\pm 0.1	-30.3	\pm 1.6	-24.0	\pm 0.4		\pm	-22.2	\pm 1.8
Val	-6.1	\pm 0.0	-4.8	\pm 0.1	-6.0	\pm 2.4	-20.2	\pm 0.0	-18.2	\pm 0.0	-19.6	\pm 2.2	-13.1	\pm 0.0	-12.3	\pm 0.1	-12.2	\pm 2.3
Leu	-23.0	\pm 0.1	-24.1	\pm 0.1	-24.7	\pm 2.3	-33.3	\pm 0.1	-33.8	\pm 0.1	-35.2	\pm 1.7	-25.5	\pm 0.7	-27.6	\pm 0.2	-26.2	\pm 1.5
Nor	-29.5	\pm 0.1	-29.5	\pm 0.1	-29.7	\pm 0.8	-29.4	\pm 0.1	-29.1	\pm 0.1	-29.5	\pm 0.7	-29.7	\pm 0.0	-32.0	\pm 0.1	-29.5	\pm 0.6
Thr	-13.3	\pm 0.1	-14.6	\pm 0.0	-14.6	\pm 0.9	-23.1	\pm 0.1	-23.5	\pm 0.0	-23.6	\pm 1.1	-15.1	\pm 0.1	-15.6	\pm 0.1	-15.4	\pm 0.9
Ser	-4.2	\pm 0.2	-6.0	\pm 0.3	-5.4	\pm 2.0	-12.5	\pm 0.1	-17.8	\pm 0.1	-16.6	\pm 2.4	-1.8	\pm 0.3		\pm	-7.9	\pm 2.8
Pro	-13.4	\pm 0.3	-14.5	\pm 0.0	-14.8	\pm 2.0	-22.0	\pm 0.2	-23.2	\pm 0.0	-23.4	\pm 1.5	-15.4	\pm 0.0	-22.0	\pm 0.3	-15.9	\pm 1.3
Asx	-2.5	\pm 0.2	-4.7	\pm 0.1	-5.7	\pm 2.0	-11.7	\pm 0.2	-12.7	\pm 0.2	-14.1	\pm 2.2	-6.0	\pm 0.1	-12.0	\pm 0.0	-8.8	\pm 2.1
Glx	-14.4	\pm 0.2	-14.2	\pm 0.1	-15.1	\pm 1.8	-23.1	\pm 0.1	-22.3	\pm 0.1	-22.7	\pm 1.4	-15.0	\pm 0.0	-18.4	\pm 0.1	-14.6	\pm 1.5
Hyp	-24.3	\pm 0.2	-24.1	\pm 0.3	-24.0	\pm 2.0	-29.8	\pm 0.3	-29.1	\pm 0.2	-29.4	\pm 1.6	-23.6	\pm 0.4	-25.1	\pm 0.6	-22.8	\pm 2.0
Phe	-14.2	\pm 0.0	-14.4	\pm 0.1	-14.9	\pm 1.8	-23.5	\pm 0.1	-23.2	\pm 0.1	-23.5	\pm 1.5	-15.3	\pm 0.1	-15.7	\pm 0.1	-15.7	\pm 1.8
Lys	-14.4	\pm 0.3	-18.0	\pm 0.2	-15.2	\pm 2.9	-21.7	\pm 0.1	-25.5	\pm 0.3	-22.0	\pm 3.1	-13.8	\pm 0.1	-22.1	\pm 0.6	-14.5	\pm 2.9

Table S4: Nitrogen isotope results for collagen QC samples (SRM-1, SRM-2, and SRM-3) from the two analytical sessions.

	SRM-1 (seal) $\delta^{15}\text{N}$ (‰)						SRM-2 (deer) $\delta^{15}\text{N}$ (‰)						SRM-3 (fish) $\delta^{15}\text{N}$ (‰)														
	25-Oct-21			01-Nov-21			longterm avg			25-Oct-21			01-Nov-21			longterm avg			25-Oct-21			01-Nov-21			longterm avg		
Ala	24.6	±	0.2	25.3	±	0.3	25.5	±	1.5	2.1	±	0.3	2.2	±	0.2	2.5	±	0.8	9.8	±	0.2	10.1	±	0.1	9.8	±	1.0
Gly	11.4	±	0.1	11.6	±	0.2	12.2	±	1.1	-1.8	±	0.0	-2.8	±	0.2	-2.0	±	0.9	8.5	±	0.3	8.1	±	0.3	8.5	±	1.2
Val	23.9	±	0.5	25.5	±	0.3	25.0	±	1.7	6.0	±	0.6	5.9	±	0.4	7.0	±	1.3	10.5	±	*	12.4	±	0.2	12.4	±	1.1
Leu	26.9	±	0.5	26.9	±	0.4	27.1	±	1.5	1.9	±	0.3	1.4	±	0.3	1.9	±	1.0	9.7	±	1.1	10.4	±	0.2	9.9	±	1.0
Nle	-1.4	±	0.3	-1.4	±	0.0	-1.1	±	1.0	-2.1	±	0.3	-2.5	±	0.1	-1.8	±	1.0	-1.5	±	0.0	-2.0	±	0.2	-1.8	±	1.1
Thr	-26.4	±	0.7	-27.0	±	0.4	-22.8	±	4.3	-12.9	±	1.4	-16.0	±	0.6	-12.9	±	2.6				-2.5	±	6.1	-3.2	±	1.9
Ser	19.2	±	0.5	14.4	±	0.4	15.4	±	4.2	-2.3	±	0.8	-2.7	±	0.9	-3.9	±	3.1	7.5	±	0.1	7.2	±	2.7	6.5	±	2.5
Pro	25.1	±	0.7	26.0	±	0.6	26.6	±	1.6	3.7	±	0.3	3.3	±	0.3	4.5	±	0.8	10.9	±	0.4	11.1	±	0.2	11.9	±	1.0
Asx	22.5	±	1.9	22.5	±	0.4	22.9	±	1.4	2.9	±	0.3	2.9	±	0.2	3.8	±	0.8	9.7	±	1.2	9.2	±	0.7	10.3	±	1.1
Glx	26.4	±	0.9	26.7	±	0.0	27.5	±	1.7	3.9	±	0.3	3.9	±	0.2	4.7	±	1.0	10.0	±	0.8	11.2	±	0.2	11.6	±	1.3
Hyp	25.3	±	0.1	25.1	±	0.5	26.5	±	1.8	3.4	±	0.2	2.6	±	0.2	4.1	±	1.0	10.3	±	0.8	10.5	±	0.1	11.5	±	1.2
Phe	4.9	±	4.7	2.9	±	0.7	8.4	±	4.0	2.2	±	2.5	5.0	±	6.1	2.7	±	2.4	-0.2	±	*	4.7	±	8.6	7.6	±	2.8
Lys	9.0	±	0.7	8.4	±	0.7	8.4	±	1.1	-3.5	±	0.9	-4.1	±	1.0	-3.4	±	1.3	4.1	±	0.6	2.3	±	1.6	4.0	±	1.1

Tables S5 and S6: Analytical results for the QCmix standard in comparison with longterm average values. Isotopic values are an average of triplicate measurements, the \pm values are the standard deviation of the triplicate measurements.

Table S5: Carbon isotope results for QCmix from both analytical sessions.

	QCMix $\delta^{13}\text{C}$ (‰)									
	21-Jun-21			29-Jun-21			longterm avg (n=31)			Expected values
Gly	-9.8	\pm	0.5	-8.8	\pm	0.5	-9.3	\pm	1.3	-9.3
Val	-21.3	\pm	1.0	-18.2	\pm	2.1	-21.2	\pm	2.8	-20.3
Nle	-30.0	\pm	0.4	-29.0	\pm	0.5	-29.8	\pm	0.8	-29.6
Pro	-8.9	\pm	2.5	-10.3	\pm	1.1	-11.3	\pm	1.0	-11.4
Glx	-29.2	\pm	2.8	-25.6	\pm	0.8	-26.9	\pm	2.3	-26.4

Table S6: Nitrogen isotope results for QCmix from both analytical sessions.

	QCMix $\delta^{15}\text{N}$ (‰)												
	25-Oct-21			25-Oct-21			01-Nov-21			01-Nov-21			longterm avg (n=40)
Gly	20.7	\pm	0.1	21.3	\pm	0.3	21.7	\pm	0.2	21.6	\pm	0.3	21.4 \pm 1.4
Val	28.7	\pm	0.4	28.4	\pm	1.0	29.6	\pm	0.1	28.6	\pm	1.0	27.9 \pm 1.6
Nle	-1.2	\pm	0.4	-1.0	\pm	0.2	-1.5	\pm	0.0	-1.7	\pm	0.1	-1.5 \pm 0.7
Pro	-0.2	\pm	0.1	0.8	\pm	0.5	-0.4	\pm	0.2	0.1	\pm	0.5	0.5 \pm 0.7
Glx	-4.1	\pm	1.0	-3.8	\pm	0.2	-3.0	\pm	0.7	-3.1	\pm	0.6	-4.2 \pm 1.1

Supplementary Figures:

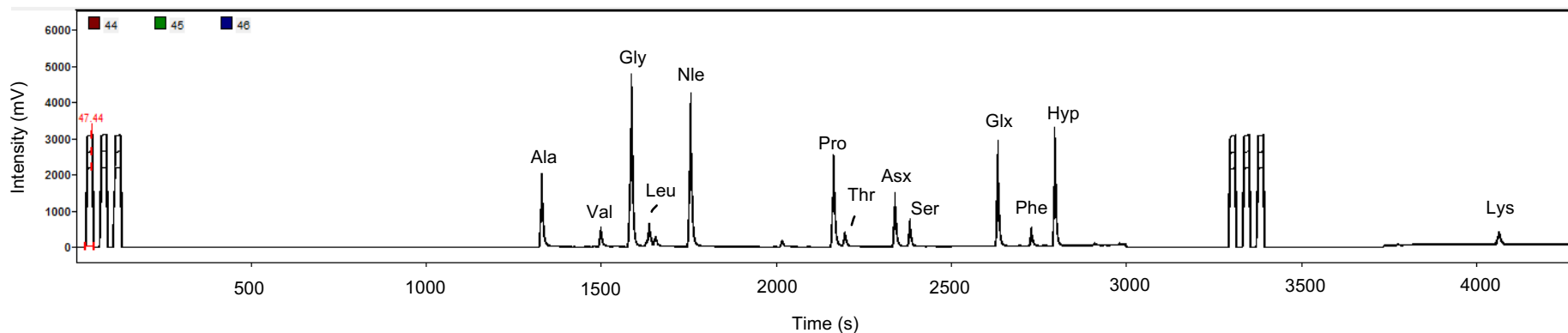


Figure S1: Typical CO₂ gas chromatogram of NAIP ester derivatized amino acids from bone collagen (S-SFU 2692). Ala = alanine, Val = valine, Gly = glycine, Leu = leucine, Nle = norleucine, Pro = proline, Thr = threonine, Asx = aspartic acid, Ser = serine, Glx = glutamic acid, Phe = phenylalanine, Hyp = hydroxyproline, Lys = lysine.

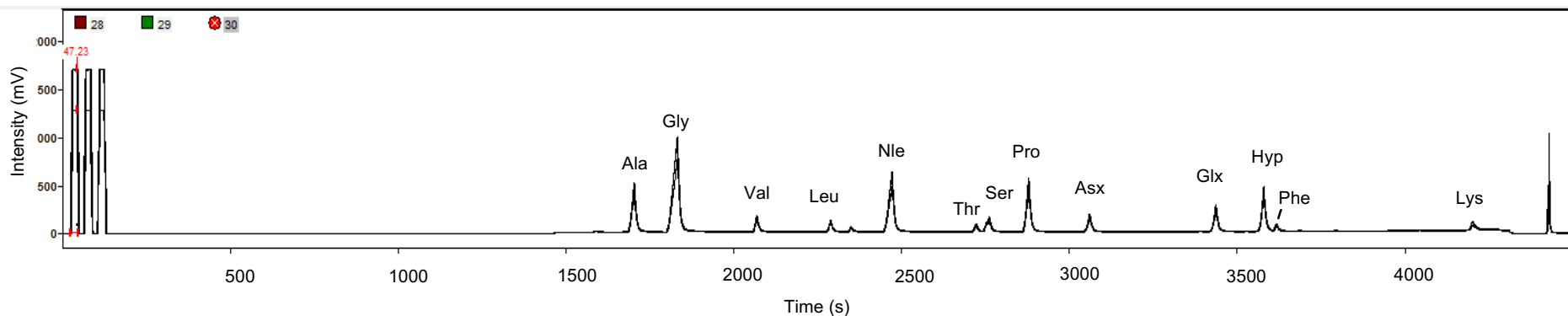


Figure S2: Typical N₂ gas chromatogram of NAIP ester derivatized amino acids from bone collagen (S-SFU 2692). Ala = alanine, Gly = glycine, Val = valine, Leu = leucine, Nle = norleucine, Thr = threonine, Ser = serine, Pro = proline, Asx = aspartic acid, Glx = glutamic acid, Hyp = hydroxyproline, Phe = phenylalanine, Lys = lysine.

Table S7: Analytical results for bulk tissue isotope analysis for the Šandalja samples considered in the study. In **green**, values previously obtained by Richards et al. (2015); in **black**, values obtained at SFU for this study.

S-SFU	Submitter No. (S-EVA)	Site	Species	$\delta^{13}\text{C}_{\text{VPDB}}$	$\delta^{15}\text{N}_{\text{AIR}}$	$\delta^{34}\text{S}_{\text{VCDT}}$	wt% C	wt% N	%S	C:N _{atomic}	C:S _{atomic}
2690	5253	Croatia-Sandalja II	pike	-22.6	9.2		36.3	12.2		3.5	
2692	5256	Croatia-Sandalja II	human	-20.3	13.6	5.2	41.0	15.0	0.1	3.2	759.1
2693	5257	Croatia-Sandalja II	human	-20.6	14.0		35.8	11.4		3.6	
2694	5259	Croatia-Sandalja II	felis	-20.9	8.6	11.6	46.6	16.0	0.2	3.4	584.2
2695	5262	Croatia-Sandalja II	felis	-20.3	6.5		40.6	14.3		3.3	
2696	5263	Croatia-Sandalja II	canis	-18.8	12.2	10.3	33.4	12.3	0.1	3.2	680.5
2697	5265	Croatia-Sandalja II	bos	-20.6	8.0	12.2	43.5	15.3	0.2	3.3	631.3
2698	5266	Croatia-Sandalja II	bos	-19.9	7.8	11.1	45.2	15.7	0.2	3.4	621.3
2699	5268	Croatia-Sandalja II	cervus	-20.4	4.6		41.6	14.7		3.3	
2701	5271	Croatia-Sandalja II	panthera	-18.2	10.1	9.4	41.7	15.4	0.2	3.2	607.1
2703	5275	Croatia-Sandalja II	equus	-20.5	6.7		40.7	14.5		3.3	
2704	5276	Croatia-Sandalja II	equus	-20.4	4.9		40.6	14.3		3.3	