Contribution to Mediterranean medieval dietary studies: stable carbon and nitrogen isotope data of marine and catadromous fish from Provence (9th–14th CE)

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Data Article

Contribution to Mediterranean medieval dietary studies: Stable carbon and nitrogen isotope data of marine and catadromous fish from Provence (9th–14th CE)

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ABSTRACT

Whilst marine resources are one of the pillars of the Mediterranean diet, their mode of acquisition and subsequent consumption by medieval populations in southern France are still not well known. Throughout Europe, bioarchaeological techniques, however, are beginning to reveal hitherto unknown aspects of these practices both dating to the medieval period as well as other periods of history and prehistory. This study involved the stable isotope analysis of five marine and catadromous taxa from three medieval sites in Provence, France: "rue Frédéric Mistral" at Fos-sur-Mer, "le Château" at Hyères and "Couvent des Dominicaines - Parking/Collège Mignet" at Aix-en-Provence. In total, 127 specimens, including *Anguilla anguilla, Dicentrarchus labrax, Sparus aurata, Diplodus sargus* and Mugilidae were subjected to car-

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bon and nitrogen stable isotope analysis. The study provides a crucial and unprecedented point of reference of the carbon and nitrogen isotopic variability of one of the main dietary resources in the Mediterranean world, fish.

Specifications Table

Subject	Archaeology
Specific subject area	Stable carbon and nitrogen isotopes analysis of bone collagen Bioarchaeology
Type of data	Table Figure
How data were acquired	Carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) stable isotope measurements were acquired via Elemental Analyser-Isotope Ratio-Mass Spectrometry (EA-IRMS), using a Europa Scientific 20–20 IRMS. Statistical tests were conducted using R version 4.0.4 (2021–02–15).
Data format	Raw
Parameters for data collection	Vertebrae from five taxa *Anguilla anguilla* ($n = 53$), *Dicentrarchus labrax* ($n = 29$), *Sparus aurata* ($n = 4$), *Diplodus sargus* ($n = 7$) and Mugilidae ($n = 34$) were sampled entirely for collagen extraction (total $n = 127$). Four samples did not provide enough material for EA-IRMS measurements. The remaining 123 samples were analysed by EA-IRMS at Iso-Analytical Limited (United Kingdom). One in five samples was measured in duplicate to check for analytical reliability when enough material was available (extracted weight >2 mg, $n = 23$).
Description of data collection	Samples were ultrasonicated in MilliQ water for 15 min until clean. Collagen was then extracted following a modified ABA protocol [1]. 0.13–1.1 mg of each extracted bone collagen sample were measured by EA-IRMS.
Data source location	All sampled skeletal collections are kept in the LA3M UMR 7298, MMSH at Aix Marseille University, Aix-en-Provence, France. The collection comes from the medieval archaeological sites of “rue Frédéric Mistral” at Fos-sur-Mer, “le Château” at Hyères and “Couvent des Dominicaines - Parking/Collège Mignet” at Aix-en-Provence, Provence, France.
Data accessibility	Repository: IsoArcH (www.isoarch.eu) [2] Data identification number: 10.48530/isoarch.2021.012 Direct URL: https://doi.isoarch.eu/doi/2021.012 Data is available under the Creative Commons BY-NC-SA 4.0 license.

Value of the Data

- This dataset can benefit archaeologists, anthropologists, and zooarchaeologists interested in palaeodietary reconstruction and past fish acquisition practices and palaeoecology.
- This dataset is useful to reconstruct the consumption and acquisition of marine resources by medieval communities of the Western Mediterranean Basin.
- The dataset provided can be used to explore the changes of those practices and their affiliated environments during medieval times.
- This dataset enlightens the interest and the necessity of building archaeologically documented datasets of the isotopic variability of this key dietary resource to better assess its importance in the Mediterranean diet through time.

1. Data Description

The data comprise of elemental and isotope values of 127 vertebrae of marine and catadromous fish specimens (53 *Anguilla anguilla*, 29 *Dicentrarchus labrax*, 4 *Sparus aurata*, 7 *Diplodus sargus* and 34 Mugilidae). This dataset is the result of a research project (Icht'Isomed[2] https://www.univ-amu.fr/fr/public/icthisomed2) which combined an archaeo-ichthyological approach and an analysis of the carbon and nitrogen isotope values of 5 key fish species of three medieval archaeological sites in Provence, South of France. The objective is to reconstruct the environments used for foraging marine resources Table 1. present sample data by specimen, providing site, taxon and sampled bone of the specimens analysed, collagen extraction data (Initial weight of the prepared sample, amount of extracted collagen and associated yield), elemental values (carbon and nitrogen contents expressed in percentage, atomic C/N ratio) and carbon and nitrogen isotope values (δ^{13}C and δ^{15}N) using delta notation (δ) in per mil increments (‰).

The samples come from three sites located along the Mediterranean seashore of Provence in South-East France: "rue Frédéric Mistral" at Fos-sur-Mer (Bouches-du-Rhône, France), "le Château" at Hyères (Var, France) and "Couvent des Dominicaines - Parking/Collège Mignet" at Aix-en-Provence (Bouches-du-Rhône, France) (Fig. 1). The samples from "le Château" at Hyères come from a dump layer dated between the end of the 8th and the middle of the 10th century AD [3]. The ones from the " rue Frédéric Mistral" habitat site in Fos-sur-Mer are from occupation levels dated between the 10th and the end of the 14th centuries AD [4]. The samples from "Couvent des Dominicaines - Parking/Collège Mignet" at Aix-en-Provence originate from a deposit pit used for consumption wastes and dated from the early 14th century AD [5].

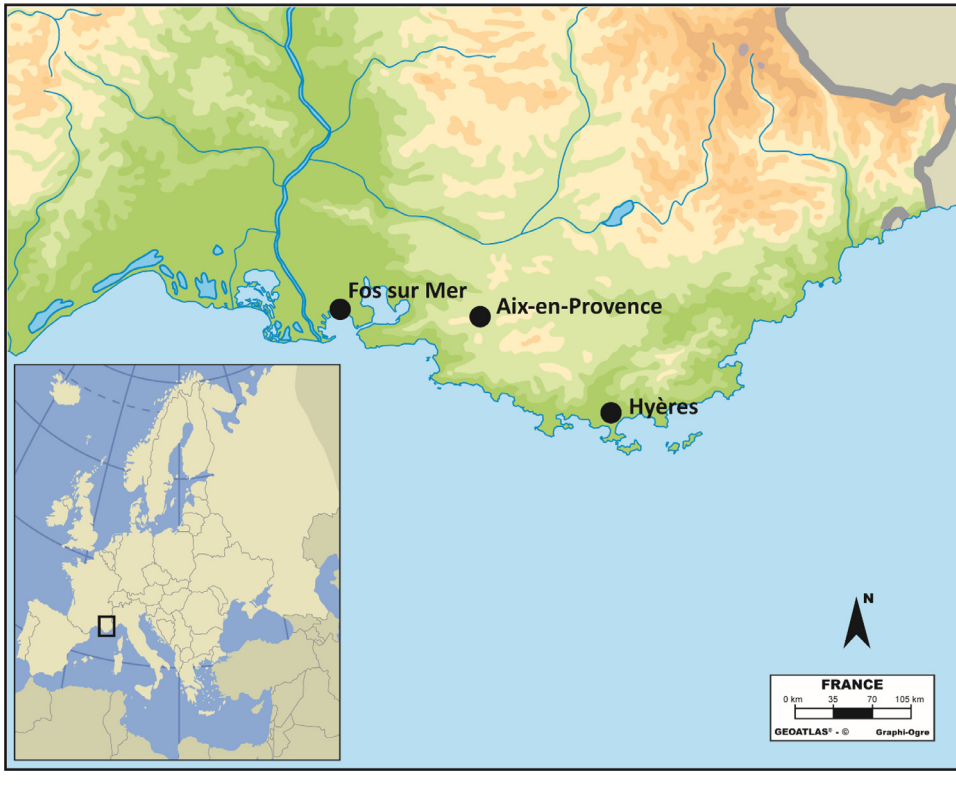

Fig. 1. Location of the three sites in southern France.

Table 1
Elemental and isotopic data (carbon and nitrogen) of the fish samples analysed with related skeletal and archaeological information. Abbreviations: FOS= "rue Frédéric Mistral", Fos-sur-Mer, Bouches-du-Rhône, France, HYERES="le Château", Hyères, Var, France, AIX="Couvent des Dominicaines – Parking/Collège Mignet", Aix-en-Provence, Bouches-du-Rhône, France, VTC= caudal vertebra, VTPC= precaudal vertebra, VTT= thoracic vertebra, UND undetermined vertebra,*samples did not provid enough collagen for EA-IRMS analyses or do not met the minimum thresholds for carbon and nitrogen contents (%C>13% and%N > 5% [10]) or atomic C:N ratio (between 2.9 and 3.6 [11]).

LabCode	Site	Taxon	Sampled bone	Initial weight (mg)	Extracted weight (mg)	Yield (mg/g)	Weight analysed (mg)	Carbon content (%)	Nitrogen content (%)	C:N	δ^{13}C (‰)	δ^{15}N (‰)
AIX_F01	AIX	*Anguilla anguilla*	VTC	16.11	2.9	180.0	0.92	24.43	9.40	3.03	−9.98	10.64
AIX_F02	AIX	*Anguilla anguilla*	VTPC	18.66	3.4	182.2	1.02	16.22	5.63	3.36	−12.79	14.19
AIX_F03*	AIX	*Anguilla anguilla*	VTPC	23.89	5.2	217.7	0.99	8.59	2.93	3.42	−11.81	10.66
		Duplicate values					0.96	8.85	2.99	3.45	−11.81	10.82
AIX_F04	AIX	*Anguilla anguilla*	VTPC	11.11	2.2	198.0	0.71	26.61	10.11	3.07	−11.57	11.06
AIX_F05*	AIX	*Anguilla anguilla*	VTPC	24.52	5.9	240.6	0.96	9.94	3.54	3.27	−11.05	10.98
AIX_F06	AIX	*Anguilla anguilla*	VTPC	16.37	3.0	183.2	1.02	30.51	11.75	3.03	−13.53	16.14
AIX_F07*	AIX	*Anguilla anguilla*	VTPC	12.16	1.7	139.8	0.14	22.84	7.21	3.70	−15.12	14.23
AIX_F08	AIX	*Anguilla anguilla*	VTPC	12.26	2.4	195.8	0.56	23.15	9.22	2.93	−9.16	10.71
AIX_F09*	AIX	*Anguilla anguilla*	UND	3.01	<1	>10						
AIX_F10*	AIX	Mugilidae	VTC	80.01	9.3	116.2	1.10	12.11	4.42	3.19	−11.99	9.10
AIX_F11*	AIX	Mugilidae	VTC	52.76	7.0	132.7	0.97	12.77	4.58	3.25	−16.65	13.06
		Duplicate values					1.00	12.75	4.54	3.28	−16.55	13.32
AIX_F12*	AIX	Mugilidae	VTC	28.68	8.9	310.4	0.97	6.90	2.33	3.46	−14.77	13.98
		Duplicate values					0.96	6.79	2.34	3.39	−14.55	13.90
FOS_F01	FOS	*Anguilla anguilla*	VTC	58.40	9.8	167.8	1.06	41.96	15.85	3.09	−10.62	14.15
		Duplicate values					0.92	40.11	15.22	3.08	−10.56	14.10
FOS_F02	FOS	*Anguilla anguilla*	VTC	15.50	3.0	193.5	1.01	38.76	14.52	3.11	−11.39	16.99
FOS_F03	FOS	*Anguilla anguilla*	VTC	26.30	4.2	159.7	1.03	41.96	15.48	3.16	−25.94	9.47
FOS_F04	FOS	*Anguilla anguilla*	VTC	33.20	4.4	132.5	0.99	41.77	15.56	3.13	−26.26	10.65
FOS_F05	FOS	*Anguilla anguilla*	VTC	51.40	7.4	144.0	0.96	43.56	16.33	3.11	−21.69	9.84
FOS_F06	FOS	*Anguilla anguilla*	VTC	72.40	10.6	146.4	0.95	40.58	15.32	3.09	−20.77	11.66
FOS_F07	FOS	*Anguilla anguilla*	VTPC	47.60	6.5	136.6	1.00	41.12	15.52	3.09	−9.62	13.41
		Duplicate values					1.02	41.57	15.59	3.11	−9.60	13.44
FOS_F08	FOS	*Anguilla anguilla*	VTPC	74.30	10.0	134.6	0.97	42.75	15.92	3.13	−9.94	13.48

(*continued on next page*)

Table 1 (*continued*)

LabCode	Site	Taxon	Sampled bone	Initial weight (mg)	Extracted weight (mg)	Yield (mg/g)	Weight analysed (mg)	Carbon content (%)	Nitrogen content (%)	C:N	δ^{13}C (‰)	δ^{15}N (‰)
FOS_F09	FOS	*Anguilla anguilla*	VTC	63.00	9.2	146.0	1.10	38.13	14.35	3.10	−9.80	12.99
FOS_F10	FOS	*Anguilla anguilla*	VTC	30.80	5.2	168.8	1.01	41.87	15.44	3.16	−9.54	13.24
FOS_F11	FOS	*Anguilla anguilla*	VTC	177.90	18.8	105.7	0.97	22.28	8.36	3.11	−21.79	9.07
FOS_F12	FOS	*Anguilla anguilla*	VTC	111.60	11.8	105.7	1.04	43.36	16.11	3.14	−9.93	17.32
		Duplicate values					1.05	43.39	16.03	3.16	−9.88	17.33
FOS_F13	FOS	*Anguilla anguilla*	VTC	68.40	8.0	117.0	1.10	42.48	15.86	3.12	−9.09	16.04
FOS_F14	FOS	*Anguilla anguilla*	VTC	18.90	3.7	195.8	1.02	39.17	14.46	3.16	−9.65	12.36
FOS_F15	FOS	*Anguilla anguilla*	VTC	109.00	9.5	87.2	1.00	43.81	16.06	3.18	−20.52	10.64
FOS_F16	FOS	*Anguilla anguilla*	VTC	134.20	20.1	149.8	0.99	44.84	16.89	3.10	−10.68	15.97
FOS_F17	FOS	*Anguilla anguilla*	VTC	59.30	3.9	65.8	1.07	43.02	16.20	3.10	−10.86	15.85
		Duplicate values					0.95	41.81	15.85	3.08	−10.82	15.90
FOS_F18	FOS	*Anguilla anguilla*	VTC	90.00	1.7	18.9	0.94	37.13	13.85	3.13	−27.74	9.95
FOS_F19	FOS	*Anguilla anguilla*	VTC	56.40	6.4	113.5	0.99	43.22	16.28	3.10	−9.10	11.99
FOS_F20	FOS	*Anguilla anguilla*	VTPC	12.86	14.8	115.1	0.98	42.39	16.07	3.08	−10.88	13.90
FOS_F21	FOS	*Anguilla anguilla*	VTPC	111.70	15.2	136.1	1.10	44.70	16.83	3.10	−19.58	11.25
FOS_F22	FOS	*Anguilla anguilla*	VTPC	49.10	6.3	128.3	1.07	44.32	16.58	3.12	−24.01	12.57
		Duplicate values					1.04	43.19	16.12	3.13	−24.05	12.61
FOS_F23	FOS	*Anguilla anguilla*	VTPC	60.60	9.8	161.7	0.96	45.87	17.34	3.09	−11.22	11.93
FOS_F24	FOS	*Anguilla anguilla*	VTPC	66.50	0.7	10.5	0.54	32.47	12.36	3.07	−9.35	12.39
FOS_F25	FOS	*Dicentrarchus labrax*	VTPC	285.00	26.7	93.7	1.00	44.79	17.01	3.07	−9.72	11.54
FOS_F26	FOS	*Dicentrarchus labrax*	VTPC	243.90	26.5	108.7	1.10	46.06	17.35	3.10	−9.75	11.46
FOS_F27	FOS	*Dicentrarchus labrax*	VTPC	197.30	19.8	100.4	1.08	45.87	17.18	3.12	−9.80	11.45
FOS_F28	FOS	*Dicentrarchus labrax*	VTPC	111.80	8.1	72.5	0.95	44.25	16.15	3.20	−11.05	12.58
		Duplicate values					0.98	44.56	16.19	3.21	−11.10	12.57
FOS_F29	FOS	*Dicentrarchus labrax*	VTPC	165.30	15.9	96.2	1.10	42.08	15.76	3.12	−8.78	11.32
FOS_F30	FOS	*Dicentrarchus labrax*	VTPC	239.30	17.1	71.5	0.96	45.09	17.08	3.08	−10.06	13.54

(*continued on next page*)

Table 1 (*continued*)

LabCode	Site	Taxon	Sampled bone	Initial weight (mg)	Extracted weight (mg)	Yield (mg/g)	Weight analysed (mg)	Carbon content (%)	Nitrogen content (%)	C:N	δ^{13}C (‰)	δ^{15}N (‰)
FOS_F31	FOS	*Dicentrarchus labrax*	VTC	161.40	26.1	161.7	1.05	49.45	18.48	3.12	−10.19	13.58
FOS_F32	FOS	*Dicentrarchus labrax*	VTPC	307.80	30.8	100.1	1.00	32.40	12.17	3.11	−10.75	10.15
FOS_F33	FOS	*Dicentrarchus labrax*	VTC	93.80	14.9	158.8	1.10	43.68	15.46	3.30	−11.03	12.39
		Duplicate values					0.99	42.84	15.33	3.26	−10.97	12.42
FOS_F34	FOS	*Dicentrarchus labrax*	VTC	43.30	6.3	145.5	0.96	40.04	15.29	3.06	−11.67	11.28
FOS_F35	FOS	*Dicentrarchus labrax*	VTPC	275.50	26.9	97.6	1.06	42.87	15.37	3.25	−11.54	13.39
FOS_F36	FOS	*Dicentrarchus labrax*	VTPC	87.40	9.7	111.0	1.07	38.82	14.67	3.09	−8.74	13.87
FOS_F37	FOS	*Dicentrarchus labrax*	VTPC	113.00	11.2	99.1	0.91	41.36	15.43	3.13	−8.80	13.92
FOS_F38	FOS	*Dicentrarchus labrax*	VTC	111.60	9.4	84.2	1.00	41.99	15.58	3.14	−13.10	11.28
		Duplicate values					1.05	42.07	15.63	3.14	−13.14	11.32
FOS_F39	FOS	*Dicentrarchus labrax*	VTC	290.20	36.1	124.4	1.04	35.42	13.14	3.15	−9.52	13.34
FOS_F40	FOS	*Dicentrarchus labrax*	VTC	122.60	15.1	123.2	1.00	41.99	15.92	3.08	−13.38	6.55
		Duplicate values					0.96	42.93	16.36	3.06	−13.42	6.51
FOS_F41	FOS	*Dicentrarchus labrax*	VTC	254.70	32.1	126.0	1.01	43.62	16.54	3.08	−12.57	14.24
FOS_F42	FOS	*Dicentrarchus labrax*	VTC	74.10	10.8	145.7	0.91	41.61	15.80	3.07	−9.33	11.36
FOS_F43	FOS	*Dicentrarchus labrax*	VTC	200.60	13.1	65.3	0.95	40.94	15.29	3.12	−10.46	10.03
		Duplicate values					0.98	39.92	15.09	3.09	−10.52	10.04
FOS_F44	FOS	*Dicentrarchus labrax*	VTPC	197.20	23.5	119.2	1.05	41.31	14.91	3.23	−10.49	13.56
FOS_F45	FOS	*Dicentrarchus labrax*	VTPC	204.70	9.3	45.4	0.99	39.41	14.25	3.23	−11.99	11.65
FOS_F46	FOS	*Dicentrarchus labrax*	VTC	286.60	28.8	100.5	0.91	40.60	15.34	3.09	−10.81	10.35
FOS_F47	FOS	*Dicentrarchus labrax*	VTC	132.70	17.3	130.4	0.93	43.43	16.23	3.12	−11.65	13.43
FOS_F48	FOS	*Dicentrarchus labrax*	VTC	199.40	24.0	120.4	0.93	41.95	15.82	3.09	−9.95	11.82
		Duplicate values					0.95	40.82	15.53	3.07	−9.91	11.82
FOS_F49	FOS	*Diplodus sargus*	VTC	26.60	4.3	161.7	0.96	38.96	14.74	3.08	−6.80	9.68
FOS_F50	FOS	*Diplodus sargus*	VTC	32.20	4.4	136.6	0.99	39.54	14.85	3.11	−8.32	10.43
FOS_F51	FOS	*Diplodus sargus*	VTC	27.20	3.2	117.6	1.08	40.69	15.40	3.08	−7.65	7.83

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Table 1 (*continued*)

LabCode	Site	Taxon	Sampled bone	Initial weight (mg)	Extracted weight (mg)	Yield (mg/g)	Weight analysed (mg)	Carbon content (%)	Nitrogen content (%)	C:N	δ^{13}C (‰)	δ^{15}N (‰)
FOS_F52	FOS	*Diplodus sargus*	VTPC	52.70	6.6	125.2	1.05	41.09	15.38	3.12	−6.84	8.82
FOS_F53	FOS	*Diplodus sargus*	VTPC	27.20	3.7	136.0	0.99	38.54	14.54	3.09	−9.44	12.56
FOS_F54	FOS	*Diplodus sargus*	VTPC	122.90	15.0	122.1	0.95	42.51	16.16	3.07	−6.59	9.15
FOS_F55	FOS	*Diplodus sargus*	VTC	38.50	7.3	189.6	1.10	30.08	11.43	3.07	−6.41	8.66
			Duplicate values				0.98	30.87	11.72	3.07	−6.36	8.71
FOS_F56	FOS	Mugilidae	VTPC	311.50	21.7	69.7	0.91	40.85	15.14	3.15	−15.93	6.59
			Duplicate values				1.01	42.15	15.57	3.16	−15.99	6.60
FOS_F57	FOS	Mugilidae	VTPC	247.40	20.8	84.1	1.08	42.07	15.63	3.14	−11.35	8.39
FOS_F58	FOS	Mugilidae	VTPC	179.90	17.5	97.3	1.07	43.86	16.55	3.09	−8.72	9.88
FOS_F59	FOS	Mugilidae	VTC	190.70	19.1	100.2	0.99	43.06	15.55	3.23	−5.87	7.90
FOS_F60	FOS	Mugilidae	VTC	190.10	13.4	70.5	1.00	41.91	15.70	3.11	−22.46	9.06
FOS_F61	FOS	Mugilidae	VTC	206.40	18.9	91.6	0.97	42.10	15.51	3.17	−12.96	12.82
			Duplicate values				0.95	41.73	15.39	3.16	−12.94	12.84
FOS_F62	FOS	Mugilidae	VTC	353.70	27.5	77.7	1.05	41.96	15.36	3.19	−6.60	9.14
FOS_F63	FOS	Mugilidae	VTPC	196.80	19.0	96.5	1.08	43.78	16.54	3.09	−9.88	11.45
FOS_F64	FOS	Mugilidae	VTPC	197.40	11.0	55.7	1.03	39.07	14.60	3.12	−10.52	11.23
FOS_F65	FOS	Mugilidae	VTC	110.00	9.4	85.5	1.05	42.87	15.87	3.15	−19.19	12.99
FOS_F66	FOS	Mugilidae	VTPC	303.80	35.7	117.5	1.03	44.75	16.48	3.17	−13.64	9.56
			Duplicate values				1.03	45.09	16.55	3.18	−13.58	9.55
FOS_F67	FOS	Mugilidae	VTPC	227.60	18.4	80.8	0.95	42.11	15.55	3.16	−12.55	12.69
FOS_F68	FOS	Mugilidae	VTPC	195.40	25.5	130.5	0.96	33.46	12.50	3.12	−6.54	9.18
FOS_F69	FOS	Mugilidae	VTPC	362.40	29.3	80.8	1.01	43.75	15.55	3.28	−6.01	7.85
FOS_F70	FOS	Mugilidae	VTPC	182.70	15.0	82.1	0.95	42.99	15.92	3.15	−6.45	9.01

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Table 1 (*continued*)

LabCode	Site	Taxon	Sampled bone	Initial weight (mg)	Extracted weight (mg)	Yield (mg/g)	Weight analysed (mg)	Carbon content (%)	Nitrogen content (%)	C:N	δ^{13}C (‰)	δ^{15}N (‰)
FOS_F71	FOS	Mugilidae	VTC	84.10	9.8	116.5	1.05	42.08	14.80	3.32	−6.73	6.95
		Duplicate values					0.99	42.94	15.47	3.24	−6.69	6.97
FOS_F72	FOS	Mugilidae	VTC	89.80	10.1	112.5	1.03	38.82	14.08	3.22	−6.97	6.99
FOS_F73	FOS	Mugilidae	VTC	162.40	9.3	57.3	0.93	39.50	14.61	3.15	−13.86	8.07
FOS_F74	FOS	Mugilidae	VTC	212.70	23.4	110.0	1.05	44.59	16.48	3.16	−10.29	11.02
FOS_F75	FOS	*Sparus aurata*	VTC	44.40	6.6	148.6	1.02	36.34	13.75	3.08	−6.59	8.78
FOS_F76	FOS	*Sparus aurata*	VTC	86.20	10.4	120.6	1.00	41.50	15.51	3.12	−12.00	14.50
		Duplicate values					1.03	41.97	15.72	3.11	−12.10	14.52
FOS_F77	FOS	*Sparus aurata*	VTPC	277.40	35.1	126.5	0.97	39.83	15.14	3.07	−8.65	10.13
FOS_F78	FOS	*Sparus aurata*	VTC	164.20	14.1	85.9	1.10	43.48	16.13	3.14	−7.22	9.86
Hy_F01	HYERES	*Anguilla anguilla*	VTC	12.40	1.3	104.8	0.30	25.60	9.34	3.20	−15.43	8.02
Hy_F02	HYERES	*Anguilla anguilla*	VTC	10.20	2.4	235.3	0.97	34.63	12.92	3.13	−11.41	11.43
Hy_F03	HYERES	*Dicentrarchus labrax*	VTPC	33.80	7.1	210.1	0.97	15.53	5.72	3.17	−13.01	7.26
		Duplicate values					1.09	15.12	5.46	3.23	−12.95	7.06
Hy_F04	HYERES	*Dicentrarchus labrax*	VTC	18.10	2.8	154.7	0.95	37.55	13.78	3.18	−12.82	7.13
Hy_F05	HYERES	*Dicentrarchus labrax*	VTC	18.90	2.7	142.9	0.93	35.22	12.76	3.22	−15.13	11.51
Hy_F06	HYERES	*Dicentrarchus labrax*	VTC	13.20	1.9	143.9	0.83	35.71	12.92	3.23	−12.96	6.00
Hy_F07	HYERES	Mugilidae	VTPC	22.40	5.2	232.1	1.03	21.85	8.04	3.17	−12.33	11.74
		Duplicate values					0.98	22.15	8.09	3.20	−12.38	11.79
Hy_F08	HYERES	Mugilidae	VTC	12.10	1.8	148.8	0.98	36.91	13.32	3.23	−12.24	9.64
Hy_F09	HYERES	Mugilidae	VTPC	19.10	2.4	125.7	1.01	35.22	12.76	3.22	−12.53	6.98
Hy_F10	HYERES	Mugilidae	VTPC	45.30	4.1	90.5	1.09	40.07	14.90	3.14	−12.35	13.02
Hy_F11	HYERES	Mugilidae	VTPC	10.00	1.2	120.0	0.41	31.86	11.54	3.22	−12.68	6.90

(*continued on next page*)

Table 1 (*continued*)

LabCode	Site	Taxon	Sampled bone	Initial weight (mg)	Extracted weight (mg)	Yield (mg/g)	Weight analysed (mg)	Carbon content (%)	Nitrogen content (%)	C:N	δ^{13}C (‰)	δ^{15}N (‰)
Hy_F12*	HYERES	Mugilidae	VTPC	25.70	5.1	198.4	1.10	8.03	2.57	3.65	−18.49	8.26
Duplicate values							1.03	7.45	2.42	3.60	−18.37	8.19
Hy_F13	HYERES	Mugilidae	VTC	19.30	2.6	134.7	1.09	37.43	13.85	3.15	−11.11	8.74
Hy_F14	HYERES	Mugilidae	VTPC	92.50	8.6	93.0	0.98	38.63	14.31	3.15	−14.60	14.90
Hy_F15	HYERES	*Anguilla anguilla*	VTC	7.80	1.3	166.7	0.49	34.66	12.47	3.24	−10.29	14.16
Hy_F16	HYERES	*Anguilla anguilla*	VTC	6.80	1.6	235.3	0.38	34.19	12.21	3.27	−18.34	10.10
Hy_F17	HYERES	*Anguilla anguilla*	VTPC	7.00	1.6	228.6	0.28	30.07	10.07	3.49	−12.61	12.85
Hy_F18	HYERES	*Anguilla anguilla*	VTT	7.50	2.2	293.3	0.57	36.35	12.79	3.32	−13.90	14.09
Hy_F19	HYERES	*Anguilla anguilla*	VTC	7.70	1.4	181.8	0.75	33.63	11.72	3.35	−13.31	13.94
Hy_F20	HYERES	*Anguilla anguilla*	VTC	5.50	1.4	254.5	0.24	28.01	9.48	3.45	−16.53	12.62
Hy_F21	HYERES	*Anguilla anguilla*	VTC	5.00	1.4	280.0	0.52	27.25	9.17	3.47	−13.64	12.14
Hy_F22	HYERES	*Dicentrarchus labrax*	VTC	6.80	1.6	235.3	0.66	31.52	11.41	3.22	−12.70	8.25
Hy_F23	HYERES	Mugilidae	VTPC	6.30	1.2	190.5	0.50	27.33	9.38	3.40	−12.87	8.70
Hy_F24	HYERES	Mugilidae	VTPC	6.10	1.1	180.3	0.36	28.47	10.00	3.32	−13.22	7.16
Hy_F25	HYERES	*Anguilla anguilla*	VTC	3.80	1.3	342.1	0.31	28.17	9.83	3.34	−15.61	13.98
Hy_F26	HYERES	*Anguilla anguilla*	VTC	4.10	1.2	292.7	0.24	29.56	9.86	3.50	−11.31	8.95
Hy_F27*	HYERES	*Anguilla anguilla*	VTC	4.10	0.5	122.0	0.44	30.60	9.41	3.79	−13.59	11.68
Hy_F28*	HYERES	*Anguilla anguilla*	VTC	3.00	<1	>10						
Hy_F29	HYERES	*Anguilla anguilla*	VTC	3.50	0.5	142.9	0.30	26.39	9.15	3.37	−10.20	12.90
Hy_F30*	HYERES	*Anguilla anguilla*	VTT	2.40	0.7	291.7	0.15	26.60	11.59	2.68	−14.06	6.33
Hy_F31*	HYERES	*Anguilla anguilla*	VTC	20.00	<1	>10						
Hy_F32*	HYERES	*Anguilla anguilla*	VTC	7.00	<1	>10						
Hy_F33*	HYERES	*Anguilla anguilla*	VTC	5.10	1.0	196.1	0.14	28.71	9.07	3.69	−15.50	11.16
Hy_F34*	HYERES	*Anguilla anguilla*	VTC	4.70	1.0	212.8	0.18	26.01	8.39	3.62	−13.87	11.85
Hy_F35*	HYERES	*Anguilla anguilla*	VTC	4.30	1.0	232.6	0.15	18.89	5.60	3.94	−12.54	11.32
Hy_F36*	HYERES	Mugilidae	VTPC	4.30	0.9	209.3	0.13	25.43	7.58	3.91	−14.04	7.03
Hy_F37*	HYERES	Mugilidae	UND	3.10	0.7	225.8	0.16	22.23	6.58	3.94	−13.81	7.42

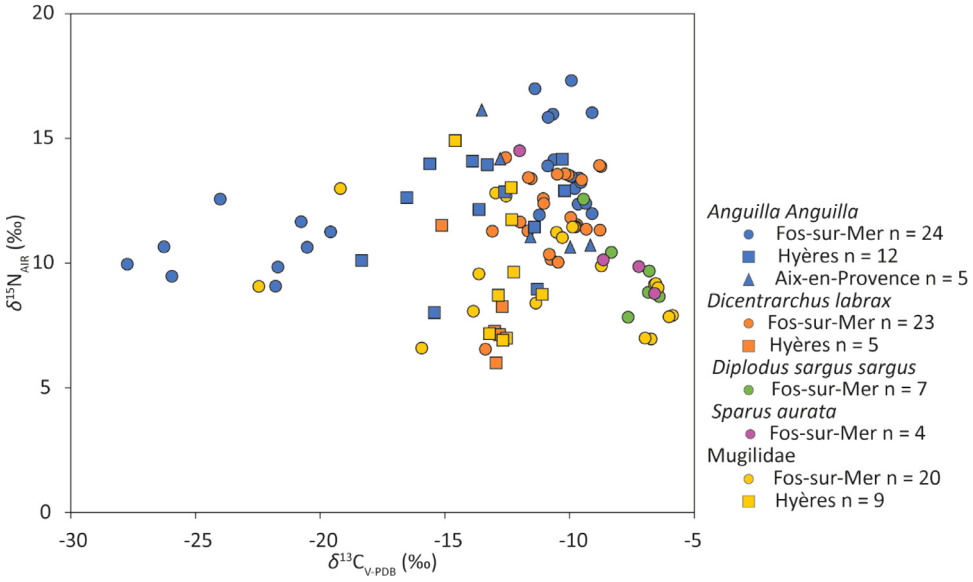

Fig. 2. $\delta^{13}C$ and $\delta^{15}N$ values obtained from the fish remains analysed in this study. The data have been disaggregated according to species and site.

The quality of the extracted collagen was measured according to their collagen yield (higher than 10 mg/g [9]), their carbon and nitrogen contents (%C > 13% and%N > 5% [10]) and their atomic C:N ratio (between 2.9 and 3.6 [11]). All samples met the minimum thresholds for collagen yield but 4 did not yield enough material to allowed EA analysis. 9 samples had atomic C:N outside the accepted values and 5 more did not reach the minimum amount of carbon and nitrogen contents. In total 109 samples (86%) met the thresholds for collagen preservation. 50 of them (39%) have carbon and nitrogen contents in collagen higher than 30 and 11%, respectively and C:N ratio lower than 3.2, more restrictive criteria pointed out by van Klinken and Guiry & Szpak for very good collagen conservation [9,12]. There is no strong correlation between the isotopic signatures of the remaining 109 samples and their yield (Spearman tests, $S = 262{,}024$, $p = 0.34$ rho -0.09 for C; $S = 210{,}146$, $p = 0.18$ rho 0.13 for N), their atomic C:N ($S = 310{,}344$, $p = 0.001$ rho -0.29 for C; $S = 264{,}148$, $p = 0.30$ rho -0.10 for N), their carbon contents ($S = 197{,}040$, $p = 0.06$ rho 0.18 for C; $S = 200{,}611$, $p = 0.08$ rho 0.17 for N) and their nitrogen content ($S = 199{,}024$, $p = 0.07$ rho 0.17 for C; $S = 194{,}772$, $p = 0.04$ rho 0.19 for N). A significant alteration of the isotopic information for the remaining samples can therefore be ruled out [11]. In total, 109 samples are then exploitable for isotopic interpretation. Isotopic data of those samples are summarized by site and taxa in Table 2. A scatterplot of $\delta^{13}C$ and $\delta^{15}N$ data according to site and taxa is presented in Fig. 2.

Table 2
Summary of the isotopic data (carbon and nitrogen) of the fish samples analysed by taxa and site.

				δ^{13}C (‰)			δ^{15}N (‰)		
Site	Datation	Taxon	n	Min	Max	Mean±SD	Min	Max	Mean±SD
Fos-sur-Mer *rue Frédéric Mistral*	10–14th	*Anguilla anguilla*	24	−27.7	−9.1	−15.0 ± 6.7	9.1	17.3	12.8 ± 2.4
		Dicentrarchus labrax	24	−13.4	−8.7	−10.6 ± 1.3	6.6	14.2	12.0 ± 1.7
		Diplodus sargus	7	−9.4	−6.4	−7.4 ± 1.1	7.8	12.6	9.6 ± 1.5
		Mugilidae	19	−22.5	−5.9	−10.9 ± 4.7	6.6	13.0	9.5 ± 2.0
		Sparus aurata	4	−12.0	−6.6	−8.6 ± 2.4	8.8	14.5	10.8 ± 2.5
Hyères *le Château*	8–10th	*Anguilla anguilla*	12	−18.3	−10.2	−13.5 ± 2.6	8.0	14.2	12.1 ± 2.1
		Dicentrarchus labrax	5	−15.1	−12.7	−13.3 ± 1.0	6.0	11.5	8.0 ± 2.1
		Mugilidae	9	−14.6	−11.1	−12.7 ± 0.9	6.9	14.9	9.8 ± 2.9
Aix-en-Provence *Couvent des Dominicaines - Parking/Collège Mignet*	14th	*Anguilla anguilla*	5	−13.5	−9.2	−11.4 ± 1.8	10.6	16.1	12.5 ± 2.5

2. Experimental Design, Materials and Methods

Prior to the extraction of collagen, the collected bone remains were subjected to a standard procedure: codified taxonomic nominations and standardised measurements were carried out with the help of the referential collections of the LA3M and CCJ osteological platforms [6–8,14–18]. Every vertebra sampled was then photographed using a 2D-3D digital microscope Hirox^{TM}.

Collagen extraction was performed at UMR 7269 LAMPEA (France), following a modified ABA method that acknowledges the fragility of the material [1]. Fish vertebrae were kept complete and first washed using successive bath of MilliQ water in an ultrasonic tank until the liquid remained clear. Samples were then demineralised at *ca.* 4 °C in 12 ml of either 0.1 M (samples weighing less than 100 mg) or 0.5 M HCl (samples weighing more than 100 mg) depending upon the weight of the sample. Solution was changed daily until bones were fully demineralised. Once demineralised, the samples were rinsed five times with MilliQ water. Demineralised bones were then placed in a 0.125 M NaOH solution at room temperature for successive 30 min sessions (until solution stopped changing colour). The samples were finally rinsed 5 times with MilliQ water and then gelatinised in a pH3 solution (10^{-}3 M HCl) at 75°C for 48 h. The resultant soluble collagen was filtered with Ezee filters^{TM}, frozen, and subsequently freeze-dryed.

After being weighed into tin capsules (between 0.13 and 1.1 mg), collagen samples were analysed by EA-IRMS on a Europa Scientific Elemental analyser coupled with a Europa Scientific 20-20 continuous flow isotope ratio Mass Spectrometer at Iso-Analytical Limited (United Kingdom). The analysis was conducted in a batch process by which a reference is analysed followed by several samples and then another reference. One in five samples were measured in duplicate when enough material was available to check analytical reliability.

The reference material used for $\delta^{13}C$ and $\delta^{15}N$ analysis was IA-R068 (soy protein, $\delta^{13}C_{V\text{-}PDB} = -25.22$ ‰, $\delta^{15}N_{AIR} = +0.99$ ‰). IA-R068, IA-R038 (L-alanine, $\delta^{13}C_{V\text{-}PDB} = -24.99$ ‰, $\delta^{15}N_{AIR} = -0.65$ ‰), IA-R069 (tuna protein, $\delta^{13}C_{V\text{-}PDB} = -18.88$ ‰, $\delta^{15}N_{AIR} = +11.60$ ‰) and a mixture of IAEA-C7 (oxalic acid, $\delta^{13}C_{V\text{-}PDB}$ = -+14.48 ‰) and IA-R046 (ammonium sulphate, $\delta^{15}N_{AIR} = +22.04$ ‰) were run as quality control check samples during analysis. IA-R068, IA-R038 and IA-R069 are calibrated against and traceable to IAEA-CH-6 (sucrose, $\delta^{13}C_{V\text{-}PDB} = -10.449$ ‰) and IAEA-N-1 (ammonium sulphate, $\delta^{15}N_{AIR} = +0.40$ ‰). IA-R046 is calibrated against and traceable to IAEA-N-1. IAEA-C7, IAEA-CH-6 and IAEA-N-1 are inter-laboratory comparison standards distributed by the International Atomic Energy Agency, Vienna (Austria).

Following recommendations by Szpak et al [13]., based on repeated measurements of calibration standards, check standards, and sample replicates, precision was determined to be ± 0.07 ‰, ± 0.06 ‰ and ± 0.06 ‰ for $\delta^{13}C$ and $\delta^{15}N$, respectively. Based on the difference between the observed and known δ values of the check standards and the long-term standard deviations of these check standards, accuracy or systematic error was determined to be ± 0.17 and ± 0.12 for $\delta^{13}C$ and $\delta^{15}N$, respectively. The total analytical uncertainties were estimated to be ± 0.17 for $\delta^{13}C$, ± 0.13 for $\delta^{15}N$.

Ethics Statement

Not applicable.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that have or could be perceived to have influenced the data reported in this article.

CRediT Author Statement

Leïa Mion: Conceptualization, Formal analysis, Investigation, Methodology, Writing – original draft, Writing – review & editing; **Tatiana André:** Conceptualization, Data curation, Investigation, Writing – review & editing; **Anne Mailloux:** Conceptualization, Funding acquisition, Supervision, Writing – review & editing; **Myriam Sternberg:** Conceptualization, Funding acquisition, Supervision, Writing – review & editing; **Arturo Morales Muniz:** Conceptualization, Writing – review & editing; **Eufrasia Rosello-Izquierdo:** Conceptualization, Writing – review & editing; **Laura Llorente Rodríguez:** Writing – review & editing; **Estelle Herrscher:** Conceptualization, Funding acquisition, Methodology, Supervision, Writing – review & editing.

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