

**[Supplementary material]**

**Something fishy in the Great Lakes? A reappraisal of early pottery use in north-eastern North America.**

Karine Taché<sup>1,\*</sup>, Manon Bondetti<sup>2,3</sup>, Alexandre Lucquin<sup>2</sup>, Marjolein Admiraal<sup>2,3</sup> & Oliver E. Craig<sup>2</sup>

<sup>1</sup> *Department of Anthropology, CUNY Queens College, 65–30 Kissena Boulevard, Queens, NY 11367, USA*

<sup>2</sup> *BioArCh, University of York, Environment Building, Wentworth Way, Heslington, York YO10 5DD, UK*

<sup>3</sup> *Arctic Centre, University of Groningen, Aweg 30, 9718CW Groningen, the Netherlands*

\* *Author for correspondence (Email: karine.tache@qc.cuny.edu)*

**Table S1. Fatty acid abundances and isotope values of Vinette 1 and Arctic residue samples. The last two columns contrast interpretation of archaeological vessel residues based on Malainey *et al.*'s (1999) criteria and the biomarker/isotope approach. Cn:x are fatty acids with carbon length n and number of unsaturations x. Full aquatic biomarkers: presence of C<sub>18</sub>, C<sub>20</sub> APAAs, isoprenoid fatty acids. Partial aquatic biomarkers: presence of C<sub>18</sub> APAAs, isoprenoid fatty acids.**

VESSEL PORTION	ID	FATTY ACIDS (REL %)												GCCIRMS (‰)		AQUATIC BIO	IDENT. BASED ON MALAINEY <i>et al.</i> 's (1999)
		C <sub>12:0</sub>	C <sub>14:0</sub>	C <sub>15:0</sub>	C <sub>16:1</sub>	C <sub>16:0</sub>	C <sub>17:0</sub>	C <sub>18:1</sub>	C <sub>18:0</sub>	C <sub>20:1</sub>	C <sub>20:0</sub>	C <sub>24:1</sub>	C <sub>24:0</sub>	C <sub>16:0</sub> δ <sup>13</sup> C	C <sub>18:0</sub> δ <sup>13</sup> C		
Rim	NAK8-12AEr	0.35	6.21	1.32	3.25	38.11	1.28	11.42	12.23	4.66	1.86	0.31	0.08	-23.91	-23.42	Full	Fish or corn
Body	AtGr9-1-I	0.63	5.90	7.06	0.05	37.20	0.97	0.69	46.02	0.00	1.32	0.00	0.15	-27.85	-29.22	Full	Large herbivore
Rim	PB01-I *	0.94	4.64	3.39	0.92	47.11	3.26	2.32	34.63	0.00	2.31	0.00	0.48			Full	Large herbivore
Rim	PB03-I *	0.82	3.67	2.60	0.00	28.28	7.80	0.42	53.47	0.00	2.49	0.00	0.46	-29.31	-30.88	Full	Large herbivore
Rim	BhF11-1-I	2.89	5.25	4.96	0.00	41.26	5.21	1.11	36.03	0.00	2.95	0.00	0.34	-27.09	-28.13	Full	Large herbivore
Body	BhF11-2-I	1.43	4.68	4.95	0.14	33.77	8.02	0.54	43.91	0.00	2.40	0.00	0.16	-27.05	-28.08	Full	Large herbivore
Body	BhF11-3-I	0.84	2.72	2.54	0.12	49.81	5.22	1.03	33.36	0.00	4.10	0.00	0.26	-25.65	-26.37	Full	Large herbivore
Body	BhF11-4-I	3.82	11.96	8.56	0.75	37.67	7.50	2.25	21.03	0.00	5.90	0.00	0.55	-26.82	-26.63	Full	Plant (except corn)
Rim	BhF11-6-I	4.62	7.07	2.12	0.08	53.18	2.90	0.66	26.86	0.00	2.36	0.00	0.13	-24.63	-25.69	Full	Large herbivore
Rim	BhF11-11-I	3.54	5.76	2.53	0.25	39.89	3.26	1.47	35.85	0.00	6.54	0.00	0.91	-26.55	-27.14	Full	Large herbivore
Rim	BhF11-13-I	0.01	3.60	4.03	0.08	35.02	7.63	0.88	44.72	0.00	3.52	0.00	0.51	-29.61	-31.93	Full	Large herbivore
Rim	BhF11-14-I	2.49	5.65	3.51	0.12	35.33	4.02	1.03	45.93	0.00	1.56	0.00	0.36	-27.27	-28.53	Full	Large herbivore
Rim	BhF11-15-I	0.47	1.89	1.14	0.11	42.46	4.09	1.00	41.09	0.00	6.33	0.00	1.42	-25.17	-25.54	Full	Large herbivore
Rim	BhF11-17-I	1.93	5.85	4.81	0.13	34.85	8.76	0.61	40.92	0.00	1.91	0.00	0.23	-27.78	-29.36	Full	Large herbivore
Rim	BhF11-18-I	3.35	8.69	8.48	0.00	47.44	5.25	1.23	23.13	0.00	2.12	0.00	0.31	-23.98	-24.51	Full	Plant (except corn)
Rim	BhF11-26-I	3.90	3.50	1.47	0.23	41.49	4.58	1.13	38.28	0.00	4.91	0.00	0.51	-24.40	-24.91	Full	Large herbivore
Body	CcFb1-1-I	0.57	1.30	0.88	0.00	59.50	2.45	0.45	33.62	0.00	1.09	0.00	0.14	-27.71	-27.92	Full	Large herbivore

Body	CcFb1-4-I	1.90	8.96	3.35	0.17	46.97	2.37	1.74	32.94	0.00	1.20	0.00	0.39	-27.09	-30.52	Full	Large herbivore
Body	CcFb1-6-I	0.50	3.70	2.21	0.10	41.48	4.31	1.76	39.68	0.00	5.41	0.00	0.86	-28.67	-29.93	Full	Large herbivore
Body	CcFb4-1-I	0.00	1.19	1.05	0.09	62.77	3.59	1.07	29.06	0.00	1.03	0.00	0.15	-26.98	-27.24	Full	Large herbivore
Body	CcFb4-2-I	0.00	0.57	0.17	0.10	52.94	1.66	0.78	42.57	0.00	1.10	0.00	0.11	-29.67	-30.07	Full	Large herbivore
Body	CeEu12-4-I	0.42	4.68	3.21	0.15	55.31	4.19	0.89	28.79	0.00	2.00	0.00	0.36	-24.74	-26.25	Full	Large herbivore
Rim	CeEu12-6-I	0.72	4.00	1.28	0.19	39.35	3.88	1.30	41.63	0.00	6.15	0.00	1.48	-26.84	-27.12	Full	Large herbivore
Rim	VT1-I	2.77	7.26	5.93	1.00	28.11	9.13	2.50	38.90	0.00	3.56	0.00	0.84			Full	Plant & large herbivore
Body	36DA11-2-I	1.50	6.49	2.44	0.00	53.26	2.06	0.57	29.14	0.00	3.98	0.00	0.56	-23.47	-25.18	Full	Large herbivore
Body	36Pi14-3-I	0.37	8.78	2.03	0.00	54.37	1.59	0.46	31.28	0.00	1.04	0.00	0.08	-25.35	-25.66	Full	Large herbivore
Rim	28Sx28-1-I	0.55	3.20	3.57	0.05	30.00	7.04	0.44	53.34	0.11	1.39	0.00	0.32	-26.67	-29.16	Full	Large herbivore
Body	28Sx28-3-I	0.58	2.34	3.06	0.00	40.42	4.23	0.00	46.93	0.00	1.85	0.00	0.59	-25.89	-26.51	Full	Large herbivore
Rim	27BK16-2-I	1.21	6.00	5.29	0.09	39.85	5.37	1.52	37.78	0.00	2.33	0.00	0.55			Full	Large herbivore
Rim	36La51-1-I	2.08	6.61	3.13	0.13	59.70	2.27	0.73	22.34	0.00	2.21	0.00	0.81			Full	Plant (except corn)
Rim	36La51-3-I	4.67	11.36	2.87	0.00	54.97	1.85	0.38	21.64	0.00	2.05	0.00	0.23			Full	Plant (except corn)
Body	6LF70-3-I	0.21	1.23	0.89	0.00	29.93	2.14	0.61	63.80	0.00	0.81	0.00	0.37			Full	Large herbivore
Rim	6LF70-6-I	1.31	12.63	2.71	0.17	58.63	1.43	0.97	15.13	1.83	2.53	2.04	0.63			Full	Plant (except corn)
Body	6LF70-7-I	1.86	6.58	1.50	0.00	46.12	1.62	2.11	33.46	0.00	5.17	0.00	1.58			Full	Large herbivore
Rim	6LF70-8-I	0.74	6.40	2.33	0.00	56.74	2.41	0.95	26.02	0.00	3.43	0.00	0.97			Full	NA
Body	6LF1-1-I	0.45	3.46	1.88	0.23	54.19	1.69	2.76	22.52	0.00	5.25	0.00	7.57			Full	NA
Body	NH38.6-3-I	1.01	10.75	1.73	0.16	51.38	2.29	0.77	25.70	0.70	4.21	0.00	1.31	-23.94	-23.53	Full	Plant (except corn)
Body	M12.43-1-I	2.17	6.47	3.09	0.00	62.81	3.02	0.63	19.97	0.00	1.24	0.00	0.61			Full	Plant (except corn)
Body	Mpt-2-I	0.45	7.03	2.71	0.19	51.11	2.77	0.34	34.31	0.00	0.82	0.00	0.28			Full	Large herbivore
Body	M49.2-5-I	5.20	9.61	4.09	0.44	42.46	3.35	1.31	31.67	0.00	1.54	0.00	0.34			Full	Plant & large herbivore
Rim	M49.2-14-I	2.88	6.78	3.02	0.51	35.52	4.19	1.27	40.25	0.00	4.16	0.00	1.42	-25.66	-25.78	Full	Large herbivore

Rim	M38.7-4-I	1.61	8.10	3.38	0.00	40.15	3.79	1.15	35.22	0.00	4.78	0.00	1.82	-23.74	-23.62	Full	Large herbivore
Body	RI1428-2-I	0.45	3.28	2.46	0.00	51.04	3.34	1.51	32.63	0.00	4.51	0.00	0.78	-22.50	-21.99	Full	Large herbivore
Rim	RI1428-4-I	0.78	7.05	2.55	0.99	63.31	1.82	3.26	14.81	0.00	3.61	0.00	1.81	-25.96	-24.18	Full	Plant (except corn)
Body	GDA2-I	0.62	5.96	4.86	0.09	40.51	4.75	1.26	38.95	0.00	2.48	0.00	0.52	-26.42	-28.87	Full	Large herbivore
Body	GDA-6a-I	1.73	12.02	3.88	0.44	58.70	2.22	1.00	18.28	0.00	1.31	0.00	0.42	-24.15	-24.01	Full	Plant (except corn)
Body	GDA-7-I	1.92	10.63	2.34	0.83	61.97	1.45	0.98	15.79	0.00	2.37	0.00	1.72	-23.81	-23.81	Full	Plant (except corn)
Body	GDA-10-I	1.89	10.49	4.81	0.15	46.49	3.33	0.85	29.60	0.00	1.90	0.00	0.48	-25.23	-27.93	Full	Plant & large herbivore
Body	AdHc4-1-I	36.99	19.83	1.64	0.32	23.42	1.22	1.20	12.22	0.00	2.05	0.00	1.11	-27.45	-27.93	Partial	Plant (except corn)
Rim	BB01-I	0.47	3.21	4.20	0.00	25.23	14.14	0.93	50.65	0.00	1.04	0.00	0.12			Partial	Large herbivore
Rim	BB08-I	0.81	9.31	2.83	0.00	50.77	1.25	1.10	32.94	0.00	0.52	0.00	0.47			Partial	Large herbivore
Body	Cda17.3-10-I	0.24	2.96	1.39	0.00	37.76	4.39	1.09	50.75	0.00	1.16	0.00	0.25	-31.11	-31.86	Partial	Large herbivore
Body	VineValley1-I	0.80	2.37	1.31	0.08	29.60	6.14	0.48	57.65	0.00	1.43	0.00	0.14	-27.34	-28.29	Partial	Large herbivore
Body	VineValley3-I	1.17	2.96	3.05	0.00	34.14	5.76	0.60	50.99	0.00	1.13	0.00	0.21			Partial	Large herbivore
Rim	BhFl1-20-I	0.76	1.97	1.38	0.10	17.83	4.18	0.71	66.93	0.00	5.16	0.00	0.97	-24.95	-25.20	Partial	Large herbivore
Body	CcFb1-5-I	0.00	2.30	0.99	0.00	46.70	1.02	1.44	46.73	0.00	0.69	0.00	0.13	-29.69	-30.54	Partial	Large herbivore
Rim	BgFg2-1-I	16.54	12.89	3.08	1.34	39.81	1.09	1.89	22.25	0.00	0.63	0.00	0.48			Partial	Plant (except corn)
Body	Sheldon-1-I	0.13	1.28	0.52	0.03	28.29	2.05	1.54	65.53	0.00	0.48	0.00	0.15	-28.89	-29.40	Partial	Large herbivore
Body	36Br58-3-I	2.65	6.31	6.87	0.00	34.81	6.85	0.00	41.59	0.00	0.69	0.00	0.24			Partial	Plant & large herbivore
Body	18AG8-2-I	0.59	2.71	1.05	0.25	36.34	1.96	1.06	52.54	0.00	2.47	0.00	1.03	-27.77	-29.25	Partial	Large herbivore
Body	28Sx28-4-I	0.29	0.68	0.38	0.00	14.41	1.59	0.22	77.19	0.00	4.08	0.00	1.15	-27.03	-28.37	Partial	Large herbivore
Body	6LF2-1-I	2.19	14.09	8.14	0.00	56.30	2.54	1.67	11.80	0.00	1.16	0.00	2.11			Partial	Plant (except corn)
Rim	NH38.6-1-I	2.76	34.05	3.59	1.51	37.17	1.77	10.22	8.43	0.00	0.36	0.00	0.14	-24.96	-24.22	Partial	Large herbivore
Body	M49.2-12-I	0.49	3.17	2.04	0.10	35.56	4.53	0.71	52.05	0.00	1.13	0.00	0.22			Partial	Large herbivore

Body	M38.7-2-I	5.29	11.86	3.45	0.00	49.48	2.09	1.05	25.10	0.00	1.04	0.00	0.65			Partial	Plant (except corn) OR plant & large herbivore
Rim	M38.11-1-I	1.49	13.02	2.37	0.28	50.20	2.39	1.39	26.03	0.00	1.85	0.00	0.97			Partial	Plant (except corn) OR plant & large herbivore
Body	RI1428-7-I	0.54	9.74	2.42	2.21	61.55	1.64	2.38	16.90	0.00	1.01	0.00	1.61	-21.69	-22.85	Partial	Plant (except corn)
Body	GDA-1-I	2.25	9.54	3.19	0.00	52.73	2.04	0.59	28.08	0.00	1.28	0.00	0.30			Partial	Plant & large herbivore
Body	GDA-5-I	1.20	5.66	2.02	0.00	46.40	1.66	0.81	39.81	0.00	2.07	0.00	0.37			Partial	Large herbivore
Rim	GDA-13-I	3.14	16.11	3.33	0.24	51.98	1.62	4.59	15.43	0.09	2.54	0.00	0.94	-26.69	-26.31	Partial	Plant (except corn)
Body	BB05-I	9.72	17.52	5.57	0.00	50.20	1.69	0.44	13.87	0.00	0.52	0.00	0.47			Absent	Plant (except corn)
Body	BB07-I	16.89	22.01	1.23	0.00	37.66	0.96	1.29	19.32	0.00	0.33	0.00	0.30			Absent	Plant (except corn)
Rim	BB09-I	17.92	30.37	3.52	0.00	34.90	1.35	0.84	10.18	0.00	0.48	0.00	0.44			Absent	Plant (except corn)
Body	BB10-I	0.92	11.07	6.57	0.00	56.11	2.21	1.68	19.82	0.00	0.87	0.00	0.76			Absent	Plant (except corn)
Rim	PB09-I	0.24	6.72	3.26	1.15	50.54	1.10	3.39	32.55	0.00	0.49	0.00	0.56			Absent	Large herbivore
Body	Cda17.3-1-I	2.70	4.04	1.53	0.00	44.84	0.85	0.85	43.92	0.00	0.82	0.00	0.45			Absent	Large herbivore
Body	Cda17.3-2-I	4.05	3.46	1.24	0.00	29.36	2.64	0.17	57.69	0.00	1.04	0.00	0.36			Absent	Large herbivore
Body	Cda17.3-3-I	0.92	11.01	5.14	0.00	54.54	1.63	2.24	22.54	0.00	0.96	0.00	1.04			Absent	Plant (except corn)
Rim	Cda17.3-4-I	2.74	11.16	6.23	0.00	54.33	2.40	1.11	19.21	0.00	1.25	0.00	1.56			Absent	Plant (except corn)
Body	Cda17.3-6-I	1.05	3.73	1.38	0.19	50.62	1.31	1.05	37.39	0.00	1.67	0.00	1.59			Absent	Large herbivore
Rim	Cda17.3-7-I	1.04	5.23	2.55	0.00	50.94	1.18	1.29	31.68	0.00	2.73	0.00	3.35			Absent	Large herbivore
Body	Cda17.3-9-I	7.23	11.13	4.91	0.00	49.67	1.69	1.59	21.61	0.00	1.11	0.00	1.07			Absent	Plant (except corn)
Body	BaGn16-1-I	19.97	9.46	2.14	0.13	58.45	0.63	0.98	7.71	0.00	0.27	0.00	0.28			Absent	Plant (except corn)
Body	BaGn16-2-I	12.19	13.69	2.97	0.24	50.05	0.99	1.57	17.55	0.00	0.42	0.00	0.34			Absent	Plant (except corn)
Body	BaGn16-3-I	12.19	10.19	3.00	0.37	51.65	1.71	0.69	19.15	0.00	0.62	0.00	0.43			Absent	Plant (except corn)
Body	BaGn16-4-I	0.00	8.98	3.62	0.00	60.69	1.54	1.08	22.84	0.00	0.68	0.00	0.57			Absent	Plant (except corn)

Rim	BhF11-12-I	5.29	9.73	2.95	0.00	40.54	2.19	0.95	35.12	0.00	2.70	0.00	0.54			Absent	Plant & large herbivore
Body	CcFb1-2-I	0.09	1.29	0.87	0.00	51.60	1.78	2.47	41.01	0.00	0.63	0.00	0.26	-28.56	-29.97	Absent	Large herbivore
Body	CcFb1-3-I	7.27	17.11	4.87	0.09	44.96	1.75	1.94	20.80	0.00	0.78	0.00	0.43	-26.90	-28.84	Absent	Plant (except corn)
Body	CcFb1-7-I	0.00	4.22	1.35	0.00	47.32	0.65	0.41	45.59	0.00	0.30	0.00	0.17	-29.12	-29.92	Absent	Large herbivore
Body	CcFb4-3-I	0.00	0.00	0.00	0.00	43.15	0.34	0.09	55.82	0.00	0.45	0.00	0.14			Absent	Large herbivore
Body	CcFb4-4-I	0.00	0.70	0.31	0.00	51.06	3.01	1.43	42.03	0.00	1.19	0.00	0.27	-28.82	-29.75	Absent	Large herbivore
Body	CcFb4-6-I	0.00	0.19	0.12	0.00	46.98	0.36	0.48	51.50	0.00	0.21	0.00	0.17			Absent	Large herbivore
Body	CcFb4-7-I	0.00	0.09	0.11	0.26	48.54	0.42	1.16	48.77	0.00	0.29	0.00	0.35			Absent	Large herbivore
Rim	CcFb4-9-I	0.00	0.00	0.13	0.27	50.87	1.10	3.32	43.83	0.00	0.26	0.00	0.22	-28.98	-29.40	Absent	Large herbivore
Body	36Br58-2-I	1.99	3.71	1.73	0.16	23.37	2.39	0.60	63.39	0.00	1.99	0.00	0.67			Absent	Large herbivore
Body	18AG3-1-I	2.32	13.51	5.32	2.96	47.42	1.45	2.67	22.35	0.00	0.94	0.00	1.08			Absent	Plant (except corn)
Body	18AG8-1-I	2.59	15.19	4.62	0.00	50.47	1.44	2.05	21.63	0.00	0.98	0.00	1.03			Absent	Plant (except corn)
Rim	28Sx28-2-I	4.19	18.04	6.23	0.00	55.35	2.70	1.77	10.13	0.00	0.93	0.00	0.65			Absent	Plant (except corn)
Body	27CA15-1	1.45	9.59	2.64	0.82	51.00	3.36	6.23	22.85	0.00	1.25	0.00	0.82			Absent	Plant (except corn)
Rim	36La51-2-I	10.00	16.72	4.95	0.00	55.38	1.70	0.00	10.09	0.00	0.83	0.00	0.32			Absent	Plant (except corn)
Body	6LF70-4-I	0.89	3.33	1.73	0.40	44.38	1.00	4.24	42.00	0.00	0.84	0.00	1.20			Absent	Large herbivore
Body	6LF70-5-I	5.04	12.26	2.82	0.00	43.72	1.62	1.77	30.53	0.96	0.00	0.00	1.27			Absent	Plant & large herbivore
Body	RI1428-5-I	1.11	8.31	2.94	2.97	60.77	1.94	2.39	17.74	0.00	0.98	0.00	0.84			Absent	Plant (except corn)
Body	Knox-1-I	0.00	2.51	1.55	0.13	51.26	0.70	1.02	40.97	0.00	1.00	0.00	0.85			Absent	Large herbivore
Body	GDA-4-I	2.81	8.32	2.81	0.00	47.34	1.00	0.44	36.32	0.00	0.60	0.00	0.37			Absent	Large herbivore
Body	GDA-8-I	0.18	14.13	5.82	0.00	49.29	2.62	2.75	21.56	0.00	1.85	0.00	1.80			Absent	Plant (except corn)
Body	GDA-9-I	3.07	16.36	2.83	0.13	47.65	1.22	2.27	24.75	0.00	0.92	0.00	0.81	-26.57	-26.40	Absent	Plant (except corn) OR plant & large herbivore
Body	GDA-11-I	3.21	19.99	7.08	0.00	51.01	2.04	1.76	12.40	0.00	1.05	0.00	1.47			Absent	Plant (except corn)

Rim	GDA-12-I	5.93	18.45	7.90	2.67	48.63	2.15	1.71	10.93	0.00	0.86	0.00	0.78			Absent	Plant (except corn)
Rim	GDA-14-I	3.73	21.95	5.28	0.51	46.04	1.80	4.80	14.15	0.00	0.97	0.00	0.78	-23.97	-24.35	Absent	Plant (except corn)

## Supplementary methods

Ceramic samples ( $\approx 1\text{--}2\text{g}$  drilled from the potsherd interior surface) were weighed and lipids were extracted and methylated according to established protocol by direct acid-catalysed transesterification to maximise recovery (Correa-Asciendo & Evershed 2014). To identify dihydroxy fatty acids, acid extracts were treated with BSTFA to obtain their TMS ethers following a protocol described in Lucquin *et al.* (2016b). GCMS analysis was performed using an Agilent 7890A series chromatograph assembled to an Agilent 5975C Inert XL mass selective detector with a quadrupole mass analyser (Agilent technologies, Cheshire, UK). A splitless injector was used and held at  $300^\circ\text{C}$ . The GC column was directly put in the ion source of the mass spectrometer. The ionisation energy of the MS was 70 eV and spectra were obtained by scanning between  $m/z$  50 and 800. In addition to scanning using the same conditions as previously established (Craig *et al.* 2012), a SIM method was performed to identify specific aquatic biomarkers (Cramp & Evershed 2014). The column used was DB23 (50%-Cyanopropyl)-methylpolysiloxane column (60m,  $250\mu\text{m}$  &  $0.25\mu\text{m}$ ; J & Scientific, Folsom, CA, USA). The temperature program was 2 minutes at  $50^\circ\text{C}$ ,  $10^\circ\text{C}/\text{min}$  to  $100^\circ\text{C}$ ,  $4^\circ\text{C}/\text{min}$  to  $140^\circ\text{C}$ ,  $0.5^\circ\text{C}/\text{min}$  to  $160^\circ\text{C}$  and  $20^\circ\text{C}/\text{min}$  to  $250^\circ\text{C}$  for 10 minutes. Helium was also used as the carrier gas at a rate flow of  $1.5\text{mL}/\text{min}$  (Shoda *et al.* 2017). Four groups of ions were selected:  $m/z$  74, 87, 213, 270 to identify 4,8,12-trimethyltridecanoic acid;  $m/z$  74, 88, 101, 312 for pristanic;  $m/z$  74, 101, 171, 326 for phytanic and  $m/z$  74, 105, 262, 290, 318, 346 for  $\omega$ -(*o*-alkylphenyl) alkanolic acids corresponding to the carbon length  $\text{C}_{16}$  to  $\text{C}_{22}$ , respectively. This method was also conducted to confirm the source of lipids by calculating the relative contribution of phytanic's SRR diastereomer (Lucquin *et al.* 2016a).

Analysis by GC-C-IRMS was undertaken on the carbon stable isotope ratios of methyl palmitate ( $\text{C}_{16:0}$ ) and methyl stearate ( $\text{C}_{18:0}$ ). The analysis was performed on a Delta V Advantage Isotope Ratio Mass Spectrometer (Thermo Fisher Scientific, Bremen, Germany) linked to a Trace 1310 Gas Chromatograph (Thermo Fisher) with a ConFlo IV interface. One microlitre of each sample was injected into a DB-5 fused-silica column (60m x  $0.25\text{mm}$  id x  $0.25\mu\text{m}$  film thickness). The temperature was set for 0.5 minutes at  $50^\circ\text{C}$ , which increased by  $25^\circ\text{C min}^{-1}$  to  $175^\circ\text{C}$ ,  $8^\circ\text{C min}^{-1}$  to  $325^\circ\text{C}$  and held for 20 minutes. The carrier gas was ultrahigh purity grade helium at a flow rate of  $2\text{ml min}^{-1}$ . The eluted products were combusted to  $\text{CO}_2$  and ionised in the source of the mass spectrometer by electron ionisation. The ion intensities of  $m/z$  44, 45, and 46 were



monitored in order to automatically compute the  $^{13}\text{C}/^{12}\text{C}$  ratio of each peak. Computations were performed with Isodat 3.0 Gas Isotope Ratio MS Software (version 3.0; Thermo Fisher), which were based on comparisons with a standard reference gas ( $\text{CO}_2$ ) of known isotopic composition that was repeatedly measured. Moreover, during each run, replicates of international standard (Indiana standard A6 and F8-3) were measured to produce a linear calibration curve (RSQ 0.996-0.998). The results from the analysis are reported in parts per mille (‰) relative to an international standard (V-PDB).

The accuracy and precision of the instrument was determined on *n*-alkanoic acid ester standards of known isotopic composition (Indiana standard F8-3). The mean  $\pm$  S.D. values of these were  $-30.00\pm 0.10\text{‰}$  and  $-23.26\pm 0.04\text{‰}$  for the methyl ester of  $\text{C}_{16:0}$  (reported mean value vs. VPDB  $-29.90\pm 0.03\text{‰}$ ) and  $\text{C}_{18:0}$  (reported mean value vs. VPDB  $-23.24\pm 0.01\text{‰}$ ) respectively. Each sample was measured in replicate (mean of S.D.  $0.13\text{‰}$  for  $\text{C}_{16:0}$  and  $0.09\text{‰}$  for  $\text{C}_{18:0}$ ). Values were also corrected subsequent to analysis to account for the methylation of the carboxyl group that occurs during acid extraction. Corrections were based on comparisons with a standard mixture of  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  fatty acids of known isotopic composition processed in each batch under identical conditions.

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