

Supplementary Table 1 - Complete oxygen isotope data on natural goethites

Sample	Mass (mg)	Blank (%)	Yield (%)	Furnace bulk $\delta^{18}\text{O}_{\text{VSMOW}}$ (‰)	+/- (‰)	Laser bulk $\delta^{18}\text{O}_{\text{VSMOW}}$ (‰)	\pm (‰)	Extracted $\delta^{18}\text{O}_{\text{VSMOW}}$ (‰)	+/- (‰)
Igarape Bahia (CIT-16404)	3.02	0.3	99	-3.291	0.6				
Para, Brazil	9.31	0.8	23					-22.429	0.6
Roy Hill (02-02-B3)	1.95	0.6	93			-1.961	0.020		
Western Australia	2.20	0.6	111	-1.859	0.6				
	2.37	0.4	108	-1.464	0.6				
	1.84	1.0	114	-2.004	0.6				
	1.53	1.2	117	-2.095	0.6				
	2.49	0.6	98	-1.851	0.6				
	2.12	3.1	110	-1.853	0.6				
	1.96	1.0	99	-2.092	0.6				
	2.86	1.5	114	-1.555	0.6				
			mean	-1.847	0.21				
	7.48	3.1	28					-20.643	0.600
	10.08	3.1	7					-19.556	0.600
			mean					-20.100	0.424
Roy Hill (02-02-Cy3)	2.26	0.4	91			-1.710	0.025		
Western Australia	1.95	0.4	110			-1.654	0.034		
			mean			-1.690	0.020		
	6.89	1.3	27					-20.040	0.600
Yandi (02-03)	2.07	0.3	107			0.976	0.028		
Western Australia	16.20	2.3	24					-20.879	0.600
Yandi (02-01-a)	1.78	2.3	90			0.677	0.026		
Western Australia	6.20	0.9	17					-20.679	0.600
Yandi (02-02)	1.83	0.3	94			0.703	0.022		
Western Australia	18.50	3.3	15					-20.806	0.600
	13.70	4.3	15					-19.999	0.600
			mean					-20.403	0.424
Pikes Peak (DMNH-14510)	2.62	0.2	117	2.720	0.46				
Colorado, USA	14.90	0.7	20					-8.535	0.600
Pikes Peak (DMNH-10029)	1.68	0.4	84			-0.363	0.041		
Colorado, USA	25.80	2.6	20					-11.761	0.600

Supplementary Table 2 - Intercomparison and reproducibility of bulk furnace extraction

Sample	Mass (mg)	Blank (%)	Yield (%)	$\delta^{18}\text{O}_{\text{laser bulk}}$ (‰)	\pm (‰)	$\delta^{18}\text{O}_{\text{furnace bulk}}$ (‰)	+/- (‰)	$\delta^{18}\text{O}_{\text{furnace-laser}}$ (‰)
Yandi (02-01-a)	1.78	2.3	90	0.677	0.026			
	1.95	1.2	101			0.166	0.6	-0.511
	1.95	1.1	109			0.303	0.6	-0.374
Roy Hill (02-02-Cy3)	2.26	0.4	91	-1.710	0.025			
	1.95	0.4	110	-1.654	0.034			
	2.24	not recorded	not recorded			-1.811	0.6	-0.129
	1.95	3.6	120			-1.721	0.6	-0.039
Roy Hill (02-02-B3)	1.95	0.6	93	-1.961	0.020			
	2.20	0.6	111			-1.859	0.6	0.102
	2.37	0.4	108			-1.464	0.6	0.497
	1.84	1.0	114			-2.004	0.6	-0.043

Supplementary Table 3 - Reproducibility of goethite reference material extracted $\delta^{18}\text{O}$

Sample	Mass	Blank	Yield	$\delta^{18}\text{O}_{\text{extracted}}$	
	(mg)	(%)	(%)	(‰)	(‰)
Roy Hill (02-02-2018)	15.3	2.8	22	-20.310	
	16.8	2.0	27	-19.294	
	18.6	1.9	26	-18.854	
	16.1	2.4	24	-19.134	
				Average	Standard Deviation
				-19.398	0.635

Supplementary Table 4 - Complete oxygen isotope data on synthetic goethites

Sample	Notes	Mass (mg)	Blank (%)	Yield (%)	Furnace bulk $\delta^{18}\text{O}_{\text{VSMOW}}$ (‰)	+/- (‰)	Extracted $\delta^{18}\text{O}_{\text{VSMOW}}$ (‰)	+/- (‰)
S05-GOE-H-2	5 °C, pH 2	1.30	2.5	110	2.95	0.6		
		6.94	3.6	12			-24.64	0.6
S22-GOE-H-5	22 °C, pH 2	1.63	1.9	112	3.10	0.6		
		10.39	1.1	8			-15.61	0.6
S40-GOE-H-1	40 °C, pH 2	1.68	1.6	95	1.68	0.6		
		7.11	6.6	12			-10.65	0.6
S22-GOA-H-2	22 °C, pH 2, no Al-uptake	2.41	0.5	89	3.93	0.6		
		7.65	8.9	7			-14.68	0.6
S40-GOA-H-3	40 °C, pH 2, no Al-uptake	2.08	1.2	98	3.18	0.6		
		1.87	1.6	98	2.43	0.6		
		2.19	1.4	98	2.33	0.6		
				mean	2.65	0.35		
		8.81	1.4	11			-9.91	0.6
S70-GOA-H-3	70 °C, pH 2, no Al-uptake	2.32	3.4	81	-0.70	0.6		
		2.03	0.8	82	-0.46	0.6		
				mean	-0.58	0.42		
		7.87	2.4	10			-2.49	0.6
S22-GOE-K-5	22 °C, pH 12	1.63	1.9	110	-0.18	0.6		
		1.50	2.7	84	-0.48	0.6		
				mean	-0.33	0.42		
		10.22	0.6	21			-16.86	0.6
S40-GOE-K-2	40 °C, pH 12	1.85	0.5	116	-13.79	0.6		
		5.91	1.4	16			-25.06	0.6
S70-GOE-K-5	70 °C, pH 12	1.87	2.3	103	-3.07	0.6		
		7.12	4.4	13			-5.61	0.6

Sample naming conventions:

GOE = pure goethite

GOA = precipitated from Al-rich solution (90:10 - Fe:Al)

K = precipitated in KOH base (pH 12)

H = precipitated in HNO₃ acid (pH 2)

Supplementary Table 5 - New (U-Th)/He dates

Sample	Corrected Age (Ma)	±	U (ppm)	Th (ppm)	He (nmol/g)	Mass (ug)
Roy Hill (02-02-B3)	56.2		0.88	0.28	0.29	93
Roy Hill (02-02-B3)	70.1		0.66	0.26	0.28	122
Roy Hill (02-02-B3)	70.1		0.66	0.32	0.28	91
Mean	65.5	3.3	0.73	0.29	0.28	
Bahia (CIT-16404)	11.2		93	0.22	5.63	91
Bahia (CIT-16404)	11.8		84	0.17	5.41	103
Bahia (CIT-16404)	10.5		91	0.18	5.20	95
Mean	11.2	0.6	89	0.19	5.41	

Measurements followed procedures described by Miller et al. (2017)