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The triple oxygen isotope composition of phytoliths as a proxy of continental atmospheric humidity: insights from climate chamber and climate transect calibrations

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Table S1: a) Comparison between IRMS (4 replicates, SD of 0.015‰, 0.010‰ and 5 per meg for $\delta^{17}O$, $\delta^{18}0$ and ^{17}O-excess respectively) and laser analyser (12 replicates, SD displayed) measurements of working water standards. SD for standard deviation; **b)** Measurements of soil water samples with the isotope laser analyzer (Picarro L2140i) operated in ^{17}O-excess mode with and without the Picarro micro combustion module (MCM); SD: standard deviation calculated on the replicates.

a)

	Laser analyzer Picarro L2140i (Ecotron)			IRMS MAT 253 (LSCE)			Difference laser analyzer/IRMS		
	$\delta^{18}O$	$\delta^{17}O$	^{17}O-excess	$\delta^{18}O$	$\delta^{17}O$	^{17}O-excess	$\delta^{18}O$	$\delta^{17}O$	^{17}O-excess
	‰	‰	per meg	‰	‰	per meg	‰	‰	per meg
GIENS-1	-0.13	-0.07	1.11	-0.26	-0.14	-5.30	0.13	0.08	6.40
ECO-1	-5.68	-2.97	29.13	-5.61	-2.94	28.10	-0.07	-0.03	1.04
ICEBERG-1	-26.88	-14.25	36.09	-27.13	-14.38	35.43	0.24	0.13	0.66

b)

	WITHOUT MCM (3replicates)				WITH MCM (3 replicates)											
Sample	$\delta^{18}O$				$\delta^{2}H$				$\delta^{17}O$				^{17}O-excess			
		SD		SD		SD		SD		SD		SD		SD		
	‰		‰		‰		‰		‰		‰		per meg		per meg	
B3-100-10-05-16	-2.643	0.029	-2.607	0.010	-18.704	0.187	-18.580	0.019	-1.392	0.014	-1.365	0.009	4.1	3.4	12.7	5.8
B2-60-10-05-16	-3.495	0.014	-3.469	0.023	-23.750	0.082	-23.541	0.073	-1.835	0.010	-1.814	0.019	12.0	6.4	18.7	11.1
B3-100-03-06-16	-2.799	0.018	-2.766	0.022	-18.868	0.105	-18.894	0.185	-1.462	0.022	-1.457	0.019	16.7	12.7	4.8	7.4
M1-40-03-06-16	-5.605	0.020	-5.584	0.005	-31.737	0.077	-31.684	0.155	-2.938	0.012	-2.929	0.004	25.7	3.0	23.5	1.7
B1-85-10-05-16	-2.945	0.038	-2.901	0.010	-20.987	0.018	-20.925	0.050	-1.551	0.045	-1.528	0.008	4.7	25.4	4.8	12.1
B10-40-10-05-16	-2.726	0.029	-2.697	0.022	-19.891	0.071	-19.594	0.097	-1.434	0.030	-1.416	0.015	6.8	16.2	8.5	10.3
B1-40-03-06-16	-3.903	0.011	-3.895	0.005	-25.017	0.187	-24.959	0.025	-2.041	0.012	-2.040	0.009	21.6	6.4	18.9	10.7
		0.023		0.014		0.104		0.086		0.021		0.012		10.5		8.4

Table S2. Growth chamber experiment : measured $\delta^{18}O$, $\delta^{17}O$ and ^{17}O-excess of irrigation water (IW), soil water, leaf water (LW) and phytoliths. Av : average ; n : number of replicates ; SD : standard deviation calculated on the replicates; n.v. : no value.

	Irrigation water							Soil water							Leaf water							Phytoliths							
Sample	$\delta^{18}O$	SD	$\delta^{17}O$	SD	n	$\delta'^{18}O$	^{17}O-excess	$\delta^{18}O$	SD	$\delta^{17}O$	SD	n	$\delta'^{18}O$	^{17}O-excess	$\delta^{18}O$	SD	$\delta^{17}O$	SD	n	$\delta'^{18}O$	^{17}O-excess	$\delta^{18}O$	SD	$\delta^{17}O$	SD	n	$\delta'^{18}O$	^{17}O-excess	SD
	‰		‰			‰	per meg	‰		‰			‰	per meg	‰		‰			‰	per meg	‰		‰			‰	per meg	
P1-40-29-04-16	-5.546	0.017	-2.912	0.013	3	-5.562	20	-2.562	0.026	-1.389	0.029	3	-2.565	-35	10.733	0.106	5.519	0.082	2	10.676	-133	45.454	0.212	23.361	0.152	2	44.451	-378	41
P10-40-10-05-16	-5.594	18.139	-2.933	16.016	3	-5.610	25	-2.697	0.022	-1.416	0.015	3	-2.701	9	7.590		3.870		1	7.561	-130	41.947	0.348	21.590	0.199	2	41.091	-336	15
P1-40-20-05-16	-5.580	0.019	-2.917	0.019	3	-5.596	33	-3.658	0.013	-1.913	0.013	3	-3.665	20	10.807		5.554		1	10.749	-137	41.150	0.592	21.161	0.291	2	40.326	-352	18
P1-40-03-06-16	n.v.		n.v.			n.v.	n.v.	n.v.		n.v.			n.v.	n.v.	8.530		4.360		1	8.494	-135	41.758		21.451		1	40.909	-376	
Av.						**-5.589**	**26**						**-2.977**	**-2**						**9.370**	**-134**						**41.694**	**-360**	
SD						**0.025**	**6**						**0.600**	**29**						**1.596**	**3**						**1.867**	**20**	
P10-60-29-04-16	-5.564	0.007	-2.929	0.008	3	-5.579	13	-2.504	0.067	-1.296	0.057	3	-2.507	27	9.581	0.015	4.942	0.008	2	9.535	-104	39.426	0.528	20.346	0.255	4	38.669	-275	23
P2-60-10-05-16	-5.563	0.001	-2.917	0.016	3	-5.579	24	-3.469	0.023	-1.814	0.019	3	-3.475	19	11.370		5.832		1	11.306	-154	37.883	0.340	19.579	0.184	4	37.183	-243	4
P10-60-20-05-16	-5.566	0.021	-2.920	0.027	3	-5.582	23	-3.260	0.028	-1.699	0.008	3	-3.266	23	6.453		3.323		1	6.432	-78	37.368	0.504	19.306	0.257	2	36.687	-249	4
P10-60-03-06-16	n.v.		n.v.			n.v.	n.v.	n.v.		n.v.					2.488		1.241		1	2.485	-72	36.034		18.597		1	35.400	-265	
Av.						**-5.580**	**20**						**-3.083**	**23**						**7.440**	**-102**						**36.985**	**-258**	
SD						**0.002**	**6**						**0.509**	**4**						**3.869**	**37**						**1.351**	**15**	
P2-85-29-04-16	-5.594	0.014	-2.937	0.001	3	-5.610	21	-1.667	0.016	-0.920	0.010	3	-1.668	-39	2.219	0.067	1.127	0.050	2	2.217	-44	30.718	0.385	15.920	0.212	3	30.255	-180	7
P1-85-10-05-16	-5.542	22.510	-2.898	22.807	3	-5.558	33	-2.901	0.010	-1.528	0.008	3	-2.905	5	2.402		1.238		1	2.399	-30	31.151	0.206	16.149	0.122	3	30.675	-176	1
P2-85-20-05-16	-5.561	0.014	-2.897	0.018	3	-5.577	43	-3.975	0.018	-2.082	0.010	3	-3.983	19	1.103		0.528		1	1.102	-54	30.218	0.070	15.642	0.036	2	29.770	-198	15
P2-85-03-06-16	n.v.		n.v.			n.v.	n.v.	n.v.				3			0.802		0.391		1	0.802	-32	30.134	0.252	15.552	0.090	2	29.689	-244	
Av.						**-5.581**	**32**						**-2.852**	**-5**						**1.630**	**-40**						**30.098**	**-199**	
SD						**0.026**	**11**						**1.158**	**30**						**0.796**	**11**						**0.459**	**31**	
P3-100-10-05-16	-5.582	0.034	-2.930	0.028	3	-5.597	21	-2.607	0.010	-1.365	0.009	3	-2.611	13	9.125	1.955	4.707	0.986	2	9.084	-100	30.876	0.027	15.992	0.003	2	30.409	-190	17
P3-100-20-05-16	-5.572	10.963	-2.916	6.038	3	-5.588	29	-2.677	0.015	-1.409	0.007	3	-2.680	6	2.121		1.094		1	2.119	-25	29.901	0.148	15.497	0.071	3	29.463	-178	6
P3-100-03-06-16	n.v.		n.v.			n.v.	n.v.	n.v.		n.v.			n.v.	n.v.	-5.382		-2.844		1	-5.396	1	30.286		15.676		1	29.837	-199	
Av.						**-5.593**	**25**						**-2.646**	**9**						**1.935**	**-41**						**29.903**	**-189**	
SD						**0.007**	**5**						**0.049**	**5**						**7.242**	**53**						**0.477**	**11**	
Av.(a)						**-5.586**	**26**						**-2.889**	**16**															
SD (a)						**0.006**	**5**						**0.188**	**8**															

(a) Calculated on the raw values.

Table S3. Growth chamber experiment: predicted isotopic enrichment in ^{18}O from irrigation water to leaf water ($\Delta^{18}_{LW\text{-}IW}$) after Cernusak et al. (2016 ; Additional Supporting information). Refer to Cernusak et al. (1996) for symbol and calculations used in the table. Added calculations are displayed in grey columns: $\Delta^{17}_{LW\text{-}IW}$ and $^{17}O\text{-excess}_e$ were calculated using $^{17}\alpha_{eq} = {}^{18}\alpha_{eq}{}^{0.529}$ and $^{17}\alpha_k = {}^{18}\alpha_{eq}{}^{0.518}$, for the equilibrium fractionation and kinetic fractionation, respectively. $\lambda_{LW\text{-}IW} = \Delta'^{17} / \Delta'^{18}$. IW: irrigation water; LW : leaf water (LW).

	Sampling details			Physiological data		Isotopic data						Calculations																				
																					Predicted							Observed				
Sample	Air tem.	Leaf temp.	Air RH	Stomatal cond.	Boundary layer cond.	Atm. vapor $\delta^{18}O$	Atm. vapor $\delta^{17}O$	IW $\delta^{18}O$	IW $\delta^{17}O$	LW $\delta^{18}O$	LW $\delta^{17}O$	air vapor pressure- e_a	leaf vapor pressure- e_i	w_a/w_i	ε_k for $\delta^{18}O$	ε_k for $\delta^{17}O$	ε^* for $\delta^{18}O$ at leaf temp	ε^* for $\delta^{17}O$ at leaf temp	Δ_v for $\delta^{18}O$	Δ_v for $\delta^{17}O$	$\Delta'^{18}_{LW\text{-}IW}$	$\Delta'^{17}_{LW\text{-}IW}$	$\Delta'^{18}_{LW\text{-}IW}$	$\Delta'^{17}_{LW\text{-}IW}$	$^{17}O\text{-excess}_e$ LW-IW	$\lambda_{LW\text{-}IW}$	$\Delta'^{18}_{LW\text{-}IW}$	$\Delta'^{18}_{LW\text{-}}$ IW	$\Delta'^{17}O_{LW\text{-}}$ IW	$^{17}O\text{-excess}_e$ LW-IW	$\lambda_{LW\text{-}IW}$
	°C	°C	%	mol m^{-2} s^{-1}	mol m^{-2} s^{-1}	‰	‰	‰	‰	‰	‰	kPa	kPa		‰	‰	‰	‰	‰	‰	‰		‰		per meg		‰	‰	‰	per meg	
P1-40-29-04-16	25.0	25.0	41.2	0.031	2	-5.55	-2.91	-5.55	-2.91	10.73	5.52	1.31	3.18	0.41	27.860	14.336	9.386	4.954	0.000	0.000	25.922	13.426	25.591	13.336	-176	0.521	16.370	16.238	8.420	-154	0.519
P10-40-10-05-16	25.0	25.0	41.3	0.032	2	-5.59	-2.93	-5.59	-2.93	7.59	3.87	1.31	3.18	0.41	27.860	14.336	9.386	4.954	0.000	0.000	25.893	13.411	25.564	13.322	-176	0.521	13.259	13.171	6.799	-155	0.516
P1-40-20-05-16	25.0	25.0	41.9	0.032	2	-5.58	-2.92	-5.58	-2.92	10.81	5.55	1.33	3.18	0.42	27.857	14.334	9.386	4.954	0.000	0.000	25.723	13.324	25.398	13.236	-174	0.521	16.479	16.345	8.460	-170	0.518
P1-40-03-06-16	25.0	25.0	41.4	0.032	2	n.v.	n.v.	n.v.	n.v.	8.53	4.36	1.32	3.18	0.41	27.860	14.336	9.386	4.954	n.v.	n.v.											
P10-60-29-04-16	25.0	25.0	60.5	0.052	2	-5.56	-2.93	-5.56	-2.93	9.58	4.94	1.92	3.18	0.61	27.770	14.290	9.386	4.954	0.000	0.000	20.458	10.627	20.252	10.571	-122	0.522	15.230	15.115	7.864	-117	0.520
P2-60-10-05-16	25.0	25.0	60.2	0.052	2	-5.56	-2.92	-5.56	-2.92	11.37	5.83	1.91	3.18	0.60	27.772	14.291	9.386	4.954	0.000	0.000	20.543	10.670	20.335	10.614	-123	0.522	17.028	16.885	8.737	-178	0.517
P10-60-20-05-16	25.0	25.0	60.5	0.052	2	-5.57	-2.92	-5.57	-2.92	6.45	3.32	1.92	3.18	0.61	27.770	14.290	9.386	4.954	0.000	0.000	20.458	10.627	20.252	10.571	-122	0.522	12.087	12.014	6.242	-101	0.520
P10-60-03-06-16	25.0	25.0	60.3	0.052	2	n.v.	n.v.	n.v.	n.v.	2.49	1.24	1.92	3.18	0.60	27.771	14.291	9.386	4.954	n.v.	n.v.											
P2-85-29-04-16	25.0	25.0	80.2	0.074	2	-5.59	-2.94	-5.59	-2.94	2.22	1.13	2.55	3.18	0.80	27.680	14.244	9.386	4.954	0.000	0.000	14.918	7.789	14.808	7.758	-60	0.524	7.857	7.826	4.067	-65	0.520
P1-85-10-05-16	25.0	25.0	76.6	0.070	2	-5.54	-2.90	-5.54	-2.90	2.40	1.24	2.44	3.18	0.77	27.697	14.252	9.386	4.954	0.000	0.000	15.928	8.306	15.802	8.272	-72	0.523	7.989	7.957	4.139	-62	0.520
P2-85-20-05-16	25.0	25.0	81.5	0.075	2	-5.56	-2.90	-5.56	-2.90	1.10	0.53	2.59	3.18	0.82	27.675	14.241	9.386	4.954	0.000	0.000	14.554	7.602	14.449	7.573	-56	0.524	6.702	6.679	3.429	-97	0.513
P2-85-03-06-16	25.0	25.0	82.5	0.076	2	n.v.	n.v.	n.v.	n.v.	0.80	0.39	2.62	3.18	0.83	27.670	14.239	9.386	4.954	n.v.	n.v.											
P3-100-10-05-16	25.0	25.0	100.0	0.095	2	*-5.58*	*-2.93*	*-5.58*	*-2.93*	9.13	4.71	3.18	3.18	1.00	27.592	14.199	9.386	4.954	0.000	0.000	9.386	4.954	9.342	4.942	9	0.529	14.789	14.681	7.630	-122	0.520
P3-100-20-05-16	25.0	25.0	100.0	0.095	2	-5.57	-2.92	-5.57	-2.92	2.12	1.09	3.18	3.18	1.00	27.592	14.199	9.386	4.954	0.000	0.000	9.386	4.954	9.342	4.942	9	0.529	7.736	7.706	4.014	-54	0.521
P3-100-03-06-16	25.0	25.0	100.0	0.095	2	n.v.	n.v.	n.v.	n.v.	-5.38	-2.84	3.18	3.18	1.00	27.592	14.199	9.386	4.954	n.v.	n.v.											

Stomatal conductance: gs ranges from 0.1 to 0.5 in investigated C3 grasses is lower than 0.2 in C4 grasses. Cf Ocheltree et al., 2012. Here gs is calculated according to Liet al., 2017.

Boundary layer cond: 0.2 to 3 in Li et al., 2017