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*Supplement of*

## **Evidence of old soil carbon in grass biosilica particles**

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Supplement

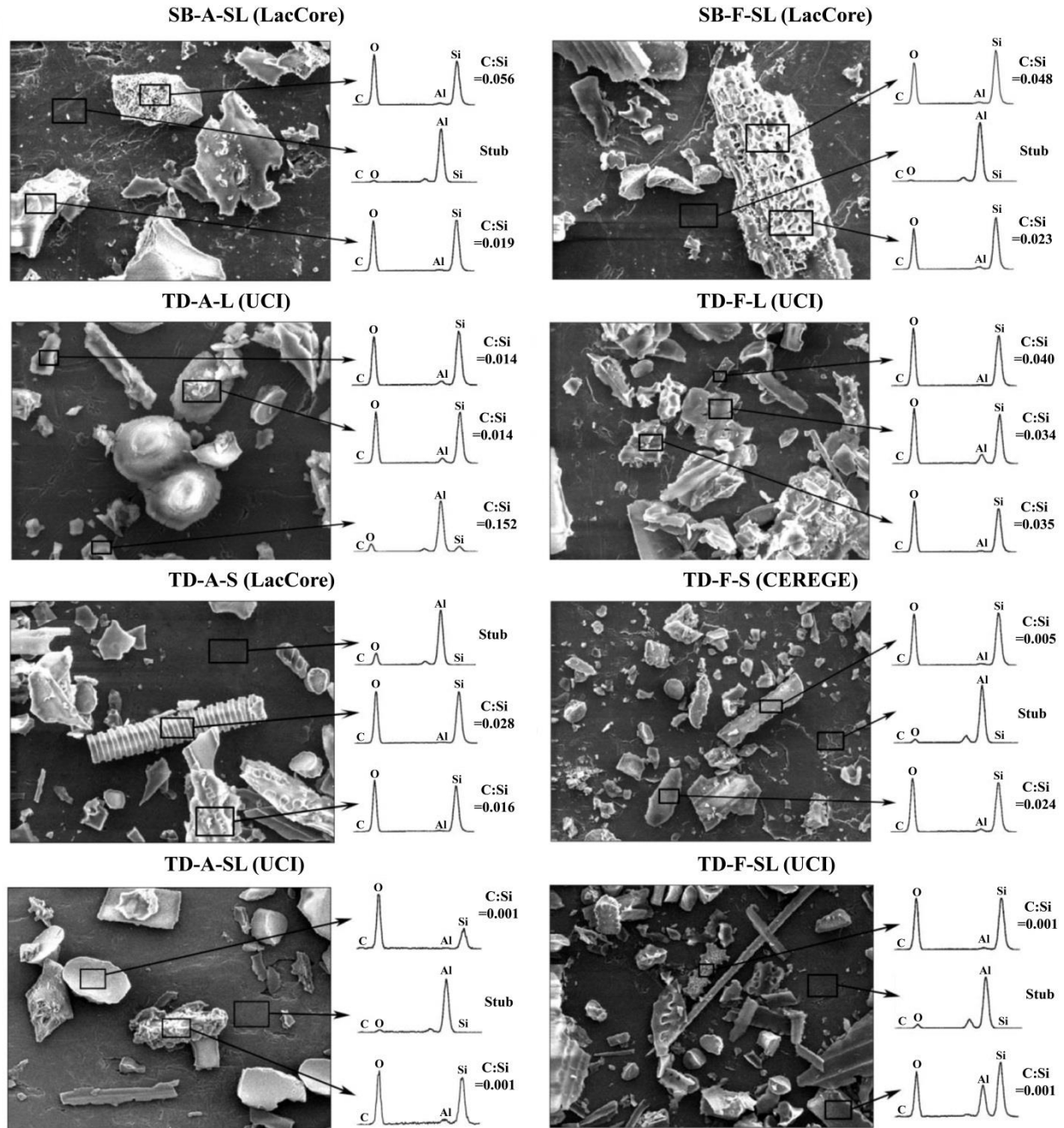


Fig. S1: Above ground C manipulation experiment. Examples of SEM images and EDS spectra of phytolith concentrates from sorghum and durum wheat experiments. The laboratory performing the extractions is shown in parenthesis. Although SEM-EDS data are semi-quantitative and somewhat inaccurate for C determinations alone, C:Si % mass ratios were distinct enough to allow detection of organic remains and silica particles. The C:Si values shown here are lower than 0.1, within the uncertainties obtained when measuring the SiC standard.

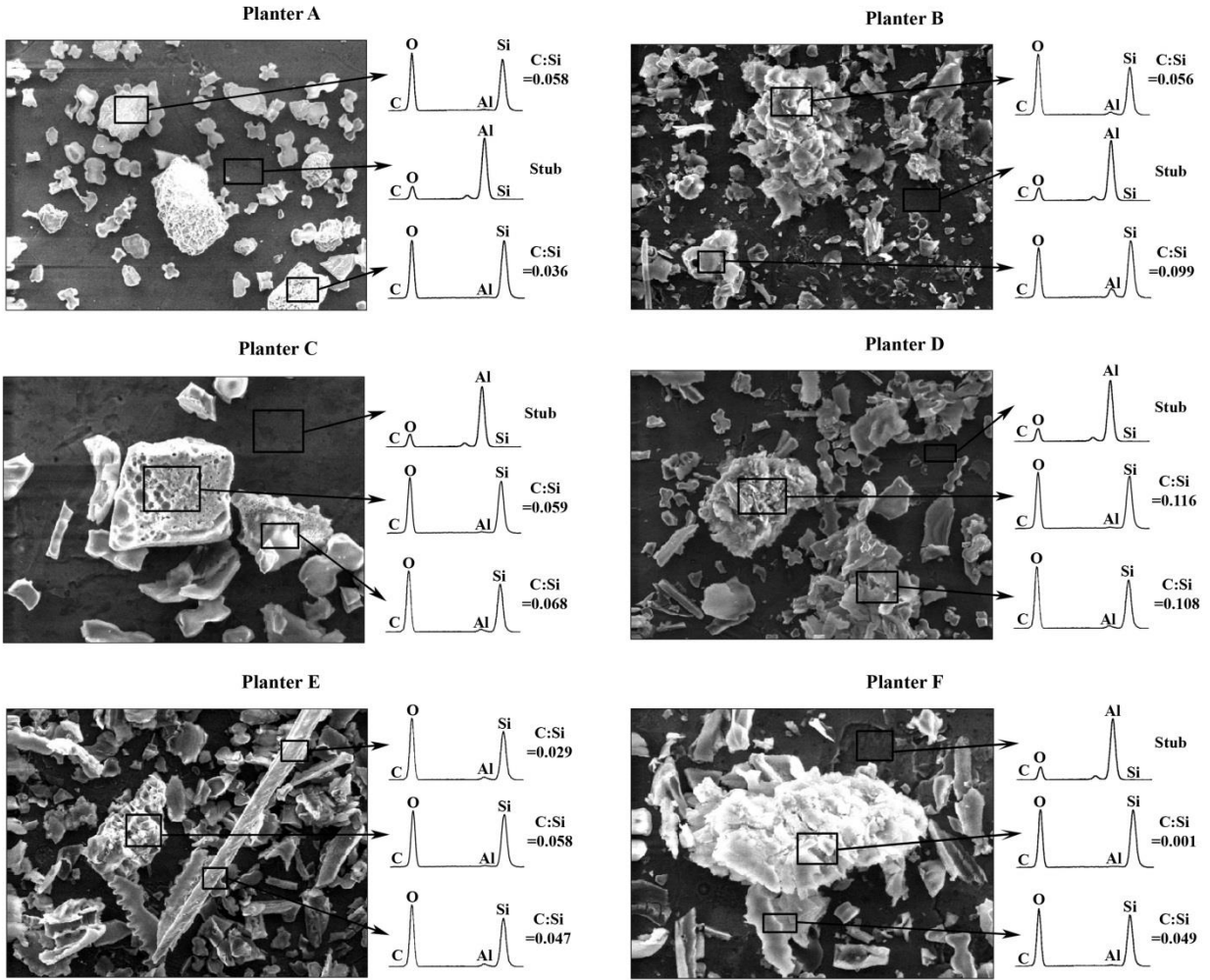


Fig. S2. Below-ground C manipulation experiment. Examples of SEM-EDS performed on phytolith concentrates from planters A-F. The C:Si values are indicative of an absence of organic residues in the concentrates and on the Al holder.

Table S1. Above-ground sorghum experiment. Complete  $^{14}\text{C}$  and  $\delta^{13}\text{C}$  results obtained for stems and leaves, phytoliths and SOM extracted fractions. Sample code is "species – ambient (A)/enriched (F)  $\text{CO}_2$  – stem and leaves (SL) of origin". Phytolith extraction protocols are described in Figure 2 of the main text. (HCl), acid carbonate removal; (A/A/A), acid/alkaline/acid treatment - carbonate and labile SOM removal. Laboratories of extraction are presented in the main text.

Series	Sample	Description	UCIAMS	Protocol	Lab.	Size (mgC)	Yield (%)	FmC	$\pm 1\sigma$	$^{14}\text{C}$ age <sup>a</sup> (years BP)	$\pm 1\sigma$	$\delta^{13}\text{C}$ -IRMS Av. $\pm$ Stdev. (‰)	Yield Av. $\pm$ Stdev. (‰)	FmC Av. $\pm$ Stdev. (‰)	$^{14}\text{C}$ offset <sup>c</sup> (years)	Oldest SOM-C contribution
Ambient $\text{CO}_2$	SB-A-SL	Stems and leaves	53275	NONE	UCI	1.01	40.8	1.0962	0.0023	-735	20	$-11.2 \pm 0.1$ (n=2)	$41.85 \pm 1.485$ (n=2)	$1.097 \pm 0.001$ (n=2)		0.00
			53276	NONE		0.86	42.9	1.0976	0.0023	-745	20					
		PhytC	123579	1a	LacCore	0.16	0.08	1.1108	0.0048	-840	35	$-16.7 \pm 0.5$ (n=2)	$0.08 \pm 0.000$ (n=2)	$1.109 \pm 0.000$ (n=2)	-85	-0.04
				123580		1a	0.13	0.08	1.1067	0.0059	-810					
			95335	2b	CEREGE	0.087	0.05	0.8291	0.0180	1510	180	$-21.6 \pm 0.1$ (n=2)	$0.05 \pm 0.000$ (n=3)	$0.05 \pm 0.031$ (n=3)	1990	0.77
				95336		2b	0.052	0.05	0.8902	0.0348	930					
			95337	2b	0.034	0.05	0.8499	0.0539	1310	510						
			100753	2a (2X)	SSAL / CEREGE	0.0043	0.01	0.8637	0.4654	1180	4330	n.a.	0.01	n.a.	1920	0.75
	SB-A-SOIL <sup>b</sup>	SOM Fraction (0-15cm)	138214	HCl	UCI	0.760	0.60	1.0323	0.0027	-250	25	$-20.8 \pm 0.1$	0.6	n.a.		
			138215	HCl		0.800	0.41	0.9884	0.0029	95	25	$-20.6 \pm 0.1$	0.41	n.a.		
			138216	HCl		0.810	0.35	0.9619	0.0026	310	25	$-20.0 \pm 0.1$	0.35	n.a.		
			138217	HCl		0.710	0.30	0.7849	0.002	1945	25	$-18.2 \pm 0.1$	0.3	n.a.		1.00
		Refractory Fraction (0-15cm)	138220	A/A/A	UCI	0.780	0.70	1.0502	0.0026	-390	20	$-22.7 \pm 0.1$ (n=2)	$0.53 \pm 0.247$ (n=2)	$1.055 \pm 0.006$ (n=2)		
			138222	A/A/A		0.740	0.35	1.0591	0.0026	-455	20					
Refractory Fraction (30-60cm)			138221	A/A/A		0.800	0.60	0.9815	0.0024	150	20	$-21.3 \pm 0.9$ (n=2)	$0.45 \pm 0.212$ (n=2)	$0.981 \pm 0.001$ (n=2)		
			138223	A/A/A		0.770	0.30	0.9803	0.0024	160	20					
Enriched $\text{CO}_2$	SB-F-SL	Stems and leaves	53273	NONE	UCI	0.82	23.5	0.6399	0.0014	3585	20	$-9.7 \pm 0.1$ (n=2)	$23.30 \pm 0.283$ (n=2)	$0.640 \pm 0.001$ (n=2)		0.00
			53274	NONE		0.73	23.1	0.6403	0.0014	3580	20					
		PhytC	123577	1a	LacCore	0.16	0.08	0.6975	0.0040	2895	50	$-16.6 \pm 0.5$ (n=2)	$0.08 \pm 0.000$ (n=2)	$0.691 \pm 0.010$ (n=2)	-610	0.18
				123578		1a	0.14	0.08	0.6840	0.0045	3050					
			95338	1b	CEREGE	0.031	0.10	0.8884	0.0630	950	570	n.a.	0.1	n.a.	-2633	0.88
	SB-F-SOIL	SOM Fraction (0-15cm)	114884	HCl	UCI	0.76	0.64	0.9776	0.0025	180	25	$-19.2 \pm 0.1$	0.64	n.a.		
			114885	HCl		0.63	0.23	1.0399	0.0028	-310	25	$-21.1 \pm 0.1$	0.23	n.a.		
			114886	HCl		0.83	0.31	1.0544	0.0025	-420	20	$-20.9 \pm 0.1$	0.31	n.a.		
			114887	HCl		0.57	0.22	0.9474	0.0023	435	20	$-19.0 \pm 0.1$	0.22	n.a.		
		Refractory Fraction (0-15cm)	114876	A/A/A	UCI	0.74	0.45	1.0477	0.0024	-370	20	$-22.2 \pm 1.4$ (n=2)	$0.32 \pm 0.184$ (n=2)	$1.046 \pm 0.002$ (n=2)		1.44
			114878	A/A/A		0.48	0.19	1.0446	0.0026	-345	25					
			Refractory Fraction (30-60cm)	114877		A/A/A	0.63	0.23	0.9378	0.0021	515	20	$-19.8 \pm 0.6$ (n=2)	$0.16 \pm 0.099$ (n=2)	$0.921 \pm 0.024$ (n=2)	
	114879	A/A/A	0.23	0.09	0.9045	0.0022	805	20								

<sup>a</sup> Negative  $^{14}\text{C}$  ages are associated with material that fixed C during the post-nuclear testing period (e.g. Post-AD 1950 to present); <sup>b</sup> C% - approximately 0.6 between 0 to 15cm, and 0.3 beyond 15cm; <sup>c</sup> relatively to  $^{14}\text{C}$  age of the bulk tissue.

Table S2. Above-ground durum wheat experiment. Complete  $^{14}\text{C}$  and  $\delta^{13}\text{C}$  results from stems and leaves, phytoliths and SOM fractions. Description (same as per Table S1).

Series	Sample	Description	UCIAMs	Protocol	Lab.	Size (mgC)	Yield (%)	FmC	$\pm 1\sigma$	$^{14}\text{C}$ age <sup>a</sup> (BP)	$\pm 1\sigma$	$\delta^{13}\text{C}$ -IRMS Av. $\pm$ Stdev. (‰)	Yield Av. $\pm$ Stdev. (‰)	FmC Av. $\pm$ Stdev. (‰)	$^{14}\text{C}$ offset <sup>e</sup> (years)	Oldest SOM-C contribution	
Ambient CO <sub>2</sub>	TD-A-L	Leaves	108998	NONE	UCI	0.89	43.70	1.0174	0.0013	-135	15	-26.9 $\pm$ 0.2 (n=16)	43.70	n.a.		0	
		PhytC	115598	1a	CEREGE	0.62	0.31	0.9788	0.0014	170	15	-33.7 $\pm$ 0.1 (n=4)	0.24 $\pm$ 0.090 (n=6)	0.9767 $\pm$ 0.0083 (n=6)	324	0.15	
			115599	1a		0.65	0.32	0.9832	0.0014	135	15						
			115600	1a		0.54	0.27	0.9846	0.0015	125	15						
			123564	1a		0.28	0.14	0.9644	0.0024	290	25						
			123565	1a		0.28	0.14	0.9725	0.0032	225	30						
			123568	1a		0.18	0.09	0.9923	0.0037	60	35						
		123569	1a	0.19	0.06	0.9823	0.0036	145	30								
		125984	2b	CEREGE	0.008	0.004	0.8428	0.0261	1370	250		0.004	n.a.	1505	0.64		
		125986	2b	UCI	0.005	0.002	0.7998	0.0463	1790	470		0.002	n.a.	1925	0.8		
		TD-A-S	Stems	108999	NONE	UCI	0.75	41.80	1.0222	0.0015	-170	15	-27.2 $\pm$ 0.1		n.a.		0
			PhytC	123572	1a	UCI	0.22	0.14	1.0089	0.0033	-65	30	-32.3 $\pm$ 0.1 (n=2)	0.14	n.a.	105	0.05
	130336			1a	LacCore	0.15	0.10	0.9904	0.0054	80	45	-34.3 $\pm$ 0.4 (n=2)	0.10 $\pm$ 0.007 (n=2)	0.9944 $\pm$ 0.0057 (n=2)	215	0.1	
	130337			1a		0.17	0.09	0.9984	0.0049	10	40						
	130340			2a	LacCore	0.077	0.032	0.9574	0.0105	350	90	n.a.	0.032	n.a.	520	0.23	
	TD-A-SL		PhytC	123575	1a	UCI	0.10	0.05	0.9858	0.0079	110	70	-33.0 $\pm$ 0.1 (n=2)	0.06 $\pm$ 0.007 (n=2)	0.989 $\pm$ 0.005 (n=2)	255	0.12
		123576		1a	0.11		0.06	0.9931	0.0064	60	60						
	TD-A-SOIL	SOM Fraction (0-15cm)	130190	HCl	UCI	0.76	1.4	0.9107	0.0013	750	15	-22.0 $\pm$ 4.2 (n=2)	1.45 $\pm$ 0.071 (n=2)	0.850 $\pm$ 0.085 (n=2)			
130193		HCl	0.72	1.5		0.7903	0.0016	1890	20								
	Refractory Fraction (0-15cm)	130206	A/A/A	UCI	0.76	1.1	0.8477	0.0012	1325	15	-19.9 $\pm$ 3.4 (n=2)	1.20 $\pm$ 0.141 (n=2)	0.746 $\pm$ 0.144 (n=2)		1		
		130200	A/A/A		0.79	1.3	0.6444	0.0010	3530	15							
Enriched CO <sub>2</sub>	TD-F-L	Leaves	109000	NONE	UCI	0.81	48.4	0.5808	0.0008	4365	15	-26.9 $\pm$ 0.2 (n=16)	48.4	n.a.		0	
		PhytC	115601	1a	CEREGE	0.25	0.13	0.5647	0.0032	4590	50	-31.6 $\pm$ 0.1 (n=2)	0.12 $\pm$ 0.010 (n=4)	0.5648 $\pm$ 0.002 (n=4)	224	0.75	
			115602	1a		0.25	0.13	0.5651	0.0031	4585	45						
			123562	1a		0.24	0.11	0.5647	0.0029	4590	45						
			123563	1a		0.26	0.12	0.5646	0.0031	4590	45						
			123566	1a		0.20	0.09	0.5575	0.0034	4695	50						
			123567	1a		0.19	0.09	0.5605	0.0035	4650	50						
		125983	2b	CEREGE	0.009	0.004	0.4057	0.0176	7250	350	n.a.	0.004	n.a.	2885	1		
		125985	2b	UCI	0.007	0.003	0.5329	0.0237	5060	360	n.a.	0.003	n.a.	695	1		
		TD-F-S	Stems	109001	NONE	UCI	0.80	43.5	0.5315	0.0007	5075	15	-27.1 $\pm$ 0.1		n.a.		0
			PhytC	123570	1a	UCI	0.11	0.09	0.5497	0.0059	4810	90	-31.5 $\pm$ 0.1 (n=2)	0.08 $\pm$ 0.021 (n=2)	0.5526 $\pm$ 0.0040 (n=2)	-310	0.76
				123571	1a		0.090	0.06	0.5554	0.0074	4720	110					
	130334			1a	LacCore		0.18	0.10	0.5627	0.0027	4620	40					
	130335			1a			0.13	0.100	0.5572	0.0039	4700	60					
	130339		2a	LacCore	0.060	0.03	0.5201	0.0081	5250	130	n.a.	0.03	n.a.	175	0		
	TD-F-SL		PhytC	123573	1a	UCI	0.14	0.07	0.5571	0.0072	4700	110	-32.1 $\pm$ 0.1 (n=2)	0.06 $\pm$ 0.021 (n=2)	0.550 $\pm$ 0.009 (n=2)	-280	0.68
				123574	1a		0.063	0.04	0.5438	0.0166	4890	250					
	TD-F-SOIL	SOM Fraction (0-15cm)	130194	HCl	UCI	0.81	1.5	0.7329	0.0011	2495	15	-18.2 $\pm$ 2.4 (n=2)	1.50 $\pm$ 0.000 (n=2)	0.676 $\pm$ 0.081 (n=2)			
			130195	HCl		0.79	1.5	0.6188	0.0009	3855	15						
		Refractory Fraction (0-15cm)	130201	A/A/A	UCI	0.80	1.5	0.5983	0.0010	4125	15	-16.0 $\pm$ 1.9 (n=2)	1.60 $\pm$ 0.141 (n=2)	0.559 $\pm$ 0.055 (n=2)	320	1	
			130202	A/A/A		0.81	1.7	0.5204	0.0009	5245	15						
	Std.	MSG70	PhytC	100754	Kelly's	CEREGE	0.081	0.1	0.6962	0.0074	2910	90	-30.4 $\pm$ 0.1 (n=2)	0.1 $\pm$ 0.000 (n=2)	0.697 $\pm$ 0.001 (n=2)		
				123581	Kelly's		0.24	0.1	0.6968	0.0026	2900	35					

<sup>a</sup> Negative  $^{14}\text{C}$  ages are associated with material that fixed C during the post-nuclear testing period (e.g. Post-AD 1950 to present); <sup>b</sup> C‰ - approximately 1.6 between 0 to 15cm, and 1.4 beyond 15cm; <sup>c</sup> relatively to  $^{14}\text{C}$  age of the bulk tissue; <sup>d</sup> Assigned value (cf main text); Kelly's - soil phytoliths extracted using a conventional protocol (Kelly et al. 1991) adapted to soil and sediment materials (see Crespin et al. 2008).

Table S3. Below-ground experiment. Complete  $^{14}\text{C}$  and  $\delta^{13}\text{C}$  results from seeds, stems and leaves, phytoliths, and amendments. Monthly integrated  $^{14}\text{CO}_2$  of ambient-air (collected in 60 L cylinders) and efflux, plus efflux  $\text{CO}_2$  concentrations are also shown.

Sample	Description	UCIAMS	Protocol	Size (mg C)	Yield (%)	FmC	$\pm 1\sigma$	$^{14}\text{C}$ age <sup>a</sup> (years BP)	$\pm 1\sigma$	$\delta^{13}\text{C}$ (‰)	$\pm 1\sigma$	$^{14}\text{C}$ offset <sup>c</sup> (years)	Oldest amendment C contribution
Single seed	Sorghum bicolor	83120	N/A	1.04	41.8	1.0621	0.0025	-480	20	-	-	-	
		83121		0.83	37.4	1.0667	0.0025	-515	20	-	-	-	
Ambient air	Period 5/31/11-7/7/11	119788	CO <sub>2</sub> purged from air (60L)	0.85	N/A	1.0258	0.0016	-200	15	-8.9	0.1	-	
	Period 5/9/12-6/6/12 (1)	119791		0.86	N/A	1.0217	0.0017	-165	15	-	-	-	
	Period 5/9/12-6/6/12 (2)	119790		0.85	N/A	1.0231	0.0021	-180	20	-9.1	0.1	-	
	Period 7/24/12-8/21/12	119792		0.86	N/A	1.0220	0.0021	-170	20	-	-	-	
	Period 8/4/11-9/21/11	119789		0.83	N/A	1.0283	0.0018	-220	15	-8.7	0.1	-	
	Same day collection - single	83847		CO <sub>2</sub> purged from air (1L)	1.18	N/A	1.0334	0.0019	-260	15	-8.6	0.1	-
		83846	0.84		N/A	1.0224	0.0019	-175	15	-9.4	0.1	-	
Planter A	Stems	119796	N/A	0.75	43.2	1.0215	0.0017	-165	15	-13	0.1	-	
	Leaves	119795		0.82	45.7	1.0258	0.0019	-200	15	-13	0.1	-	0.00
	PhytC	130348	1a-CEREGE	0.11	0.08	1.0635	0.0078	-480	60	-18.7	0.1	320	0.64
		104361	2b-CEREGE	0.051	0.03	1.0038	0.0274	-20	220	-15.3	0.1	-140	-
		104899		0.022	0.04	1.0109	0.0780	-80	620	-	-	-	-80
		104365	2b-SSAL	0.0081	0.01	0.9925	0.3419	60	2770	-	-	-	-220
	Miracle Gro® (MG)	136938	soil amendment	0.82	49.5 (n=3)	1.0849	0.0028	-650	25	-26.1	0.1	-	1.00
		136939		0.75		1.0123	0.0028	-95	25	-25	0.1	-	-
		136940		0.4		1.0348	0.0030	-270	25	-	-	-	-
	CO <sub>2</sub> respired/chamber	83842	1.72 ppm/sec <sup>b</sup>	1.2	N/A	1.1089	0.0020	-825	15	-21.4	0.1	-	
Stems	119798	N/A	0.85	43.3	1.0162	0.0019	-125	15	-12.7	0.1	-		
Leaves	119797		0.75	43	1.0223	0.0019	-170	20	-13	0.1	-	0.00	
PhytC	130345	1a-UCI	0.12	0.1	0.9444	0.0066	460	60	-19.1	0.1	-620	0.09	
	104360	2b-CEREGE	0.022	0.02	0.8768	0.0629	1060	580	-21.4	0.1	-1220	0.17	
	104366	2b-SSAL	0.0078	0.01	0.6509	0.3065	3450	3790	-	-	-	-3610	
	Greensand (GS)	60658;59	soil amendment	0.22	0.06(OC;n=4); 0.1 (bulk)	0.1591 (n=2)	0.0016	14765	78	-24.3(OC;n=4); -12.6 (bulk)	0.1	-	1.00
	Ionic Grow (IG)	58255;74887		0.04-0.60	0.8	0.0374 (n=2)	0.0101	26550	2192	-26.4	0.1	-	
CO <sub>2</sub> respired/chamber	83843	1.28 ppm/sec <sup>b</sup>	1.2	N/A	0.8370	0.0015	1430	15	-9.9	0.1	-		
Stems	119800	N/A	0.8	42.7	1.0244	0.0016	-190	15	-12.5	0.1	-		
Leaves	119799		0.85	46.9	1.0228	0.0017	-175	15	-13	0.1	-	0.00	
PhytC	130346	1a-UCI	0.11	0.1	1.0016	0.0073	0	60	-20.4	0.1	-160	0.02	
	104362	2b-CEREGE	0.027	0.02	0.8845	0.0499	990	460	-17.5	0.1	-1150	0.14	
	104900	2b-CEREGE	0.012	0.02	0.8196	0.1540	1600	1510	-	-	-	-1760	
Ionic Grow (IG)	58255; 74887	soil amendment	0.04-0.60	0.8	0.0374 (n=2)	0.0101	26550	2192	-26.4	0.1	-	1.00	
CO <sub>2</sub> respired/chamber	-	-	-	-	-	-	-	-	-	-	-	-	
Planter D	Stems	119802	N/A	0.83	42.3	1.0174	0.0016	-135	15	-12.5	0.1	-	
	Leaves	119801		0.84	43.1	1.0208	0.0018	-160	15	-13	0.1	-	0.00
	PhytC	130347	1a-UCI	0.13	0.1	1.0002	0.0070	0	60	-20	0.1	-160	0.04
	Earth juice (EJ)	58253;54 & 60660	soil amendment	0.06-0.89	15.4 (n=2)	0.4991 (n=3)	0.0013	5583	24	-24.1 (n=2)	0.2	-	1.00
	CO <sub>2</sub> respired/chamber	83844	0.45 ppm/sec <sup>b</sup>	0.94	N/A	0.9805	0.0019	160	20	-9.4	0.1	-	
Planter E	Stems	119804	N/A	0.82	41.4	1.020	0.0017	-150	15	-12.6	0.1	-	
	Leaves	119803		0.81	46.1	1.021	0.0018	-160	15	-13.6	0.1	-	0.00
	PhytC	130344	1a-UCI	0.1	0.1	0.9371	0.0075	520	70	-17.6	0.1	-680	0.08
	Fossil Fuel (FF)	136937; 153690 & 91	soil amendment	0.7 - 0.9	33.04(n=3)	0.0055 (n=3)	0.0003	43340	1700	-26.2 (n=2)	0.2	-	1.00
	CO <sub>2</sub> respired/chamber	83845	0.34 ppm/sec <sup>b</sup>	1.04	N/A	1.0048	0.0022	-35	20	-11	0.1	-	
Planter F (control)	Stems	119794	N/A	0.72	44.2	1.023	0.0019	-180	20	-12.7	0.1	-	
	Leaves	119793		0.83	41.2	1.015	0.0019	-115	20	-13.3	0.1	-	
	PhytC	130343	1a-UCI	0.13	0.1	1.0074	0.0065	-50	60	-20.8	0.1	-110	N/A

<sup>a</sup> Negative  $^{14}\text{C}$  ages are associated with material that fixed C during the post-nuclear testing period (e.g. Post-AD 1950 to present); <sup>b</sup> Substrate amendment CO<sub>2</sub> efflux - measured from closed chambers (cf txt); <sup>c</sup>Relatively to the average  $^{14}\text{C}$  age of the bulk plant tissue from all planter