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# Soil carbon dioxide emissions from the Mojave Desert: isotopic evidence for a carbonate source

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## RESEARCH LETTER

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## Key Points:

- $\delta^{13}\text{C}_2$  Keeling plots were used to determine  $\text{CO}_2$  sources from an alkaline calcareous desert soil *in situ*
- Isotopic values show that carbonates serve as a substrate for daytime  $\text{CO}_2$  emissions from dry soils
- Carbonate-driven emissions increase strongly with surface temperatures up to  $>60^\circ\text{C}$

## Supporting Information:

- Supporting Information S1
- Table S2

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## Soil carbon dioxide emissions from the Mojave desert: Isotopic evidence for a carbonate source

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**Abstract** Arid soils represent a substantial carbonate pool and may participate in surface-atmosphere  $\text{CO}_2$  exchange via a diel cycle of carbonate dissolution and exsolution. We used a Keeling plot approach to determine the substrate  $\delta^{13}\text{C}$  of  $\text{CO}_2$  emitted from carbonate-dominated soils in the Mojave desert and found evidence for a nonrespiratory source that increased with surface temperature. In dry soils at  $25\text{--}30^\circ\text{C}$ , the  $\text{CO}_2$  substrate had  $\delta^{13}\text{C}$  values of  $-19.4 \pm 4.2\text{‰}$ , indicative of respiration of organic material (soil organic matter =  $-23.1 \pm 0.8\text{‰}$ ).  $\text{CO}_2$  flux increased with temperature; maximum fluxes occurred above  $60^\circ\text{C}$ , where  $\delta^{13}\text{C}_2$  substrate ( $-7.2\text{‰} \pm 2.8\text{‰}$ ) approached soil carbonate values ( $0.2 \pm 0.2\text{‰}$ ). In wet soils,  $\text{CO}_2$  emissions were not temperature dependent, and  $\delta^{13}\text{C}_2$  substrate was lower in vegetated soils with higher flux rates, higher organic C content, and potential root respiration. These data provide the first direct evidence of  $\text{CO}_2$  emissions from alkaline desert soils derived from an abiotic source and that diurnal emission patterns are strongly driven by surface temperature.

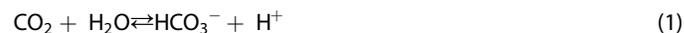
### 1. Introduction

Arid and semiarid landscapes comprise  $\sim 40\%$  of the Earth's terrestrial surface and are expected to be particularly susceptible to global change. As a result, there is considerable interest in the mechanisms controlling surface-atmosphere carbon dioxide ( $\text{CO}_2$ ) exchange in arid lands. Recent research has demonstrated that in arid ecosystems with alkaline soils derived from calcareous parent material, abiotic (nonrespiratory)  $\text{CO}_2$  exchange may be an overlooked aspect of the C cycle [Serrano-Ortiz *et al.*, 2010]. Although ignoring this flux may lead to overestimates of biological respiration, whether or not this process actually represents a meaningful C sink is currently debated [Schlesinger, 2016]. Several lines of evidence suggest that ecosystems including the saline Gubantonggut desert in western China [Xie *et al.*, 2009], Chihuahuan desert shrubland [Hamerlynck *et al.*, 2013], and unvegetated playa in Nevada [Yates *et al.*, 2012] experience  $\text{CO}_2$  uptake and loss apparently mediated by dissolution and exsolution from the soil carbonate pool. However, thus far no *in situ* measurements have directly identified that carbonates are the source of these emissions. Arid soils are also known to represent a major global carbonate reservoir [Eswaran *et al.*, 2000], and carbonate concentrations in soil can exceed organic C by an order of magnitude or more [Stevenson and Verburg, 2006]. Given that this flux can apparently be moderated by environmental variables such as atmospheric  $\text{CO}_2$  concentrations, temperature, and rainfall distribution [Hamerlynck *et al.*, 2013] and that these variables are highly susceptible to change under future climate, detailed description of the process and mechanisms is a pressing priority.

One line of evidence for an abiotic C flux comes from eddy covariance and chamber methods [Xie *et al.*, 2009; Yates *et al.*, 2012; Hamerlynck *et al.*, 2013; Ma *et al.*, 2013], indicating that several arid ecosystems appear to take up  $\text{CO}_2$  at night and exhibit positive fluxes from dry soils during the day. These observations are supported by lab experiments showing uptake, and some subsequent release, of isotopically labeled  $\text{CO}_2$  [Liu *et al.*, 2015a; Fa *et al.*, 2016a], as well as diel changes in  $\text{CO}_2$  concentration [Yates *et al.*, 2012; Liu *et al.*, 2015b; Fa *et al.*, 2016b].

Under dry conditions, the proposed mechanism for these fluxes has some uncertainties [Fa *et al.*, 2016b] but seems to be driven by a diel cycle in which changes in soil temperature shift the  $\text{CO}_2$ -carbonate-bicarbonate equilibrium, with added effects of turbulence and gas expansion driving convection [Hamerlynck *et al.*, 2013; Fa *et al.*, 2016b]. Dissolution of  $\text{CO}_2$  in the soil solution follows Henry's law, being temperature dependent and proportional to the partial pressure of  $\text{CO}_2$  in the soil [Serrano-Ortiz *et al.*, 2010]. At night, cooling of dry soils drives upward air migration and deposition of water vapor, along with

possible dew deposition, that causes CO<sub>2</sub> uptake and subsequent CaCO<sub>3</sub> dissolution [Hamerlynck *et al.*, 2013; Fa *et al.*, 2016b], according to



CO<sub>2</sub> solubility is greater at lower nighttime temperatures, although the extent to which dissolution occurs is dependent on the availability of soil water [Fa *et al.*, 2016b]. During the day as surface temperatures increase, carbonate solubility in water films decreases and the expansion of gases plus surface turbulence decreases the concentration of CO<sub>2</sub> in the soil pore spaces, driving outgassing of CO<sub>2</sub> from soil water films and causing CaCO<sub>3</sub> to again precipitate [Roland *et al.*, 2013; Fa *et al.*, 2016b]. Lab experiments confirm that this process is highly dependent on temperature [Chevallier *et al.*, 2016] as well as the rate of change of temperature [Liu *et al.*, 2015b]. Under field conditions, surface CO<sub>2</sub> fluxes show a positive correlation with temperature up to 55°C in the Chihuahuan desert [Hamerlynck *et al.*, 2013].

Carbon stable isotopes ( $\delta^{13}\text{C}$ ) offer a useful way to resolve CO<sub>2</sub> sources, using Keeling plots to identify the  $\delta^{13}\text{C}$  of the evolved fluxes, and are most effective when the potential sources have well-separated isotopic values [Pataki *et al.*, 2003]. Soil CO<sub>2</sub> fluxes derived from biological respiration (plant roots or microbes) would be expected to closely reflect the highly depleted  $\delta^{13}\text{C}$  values generated by photosynthesis (e.g.,  $-22$  to  $-24.5\text{‰}$  for the arid woody shrub *Larrea tridentata* [Rundel and Sharifi, 1993]) or present in soil organic carbon, with comparatively little fractionation [ŠantRůčková *et al.*, 2000; Werth and Kuzyakov, 2010]. Solid soil carbonates in the Mojave desert, conversely, have much more enriched  $\delta^{13}\text{C}$  values, close to  $-1\text{‰}$  [Stevenson and Verburg, 2006], and CO<sub>2</sub> in ambient air is close to  $-8\text{‰}$ . This significant separation, combined with estimates of fractionation during carbonate dissolution and exsolution, can be used to partition the sources of soil CO<sub>2</sub> fluxes. While lab incubations suggest that CO<sub>2</sub> evolved from these soils can have enriched values close to that of carbonates [Stevenson and Verburg, 2006], no evidence from the Mojave (or any arid lands, to our knowledge) exists to identify carbonates as a source *in situ*.

The Mojave desert is an arid ecosystem occupying almost 124,000 km<sup>2</sup>, characterized by alkaline, carbonate-dominated soils, and strong diel variations in temperature. Previous observations from Mojave show that that CO<sub>2</sub> fluxes from dry soils are highest in summer during the middle of the day, where high soil surface temperatures (up to 80°C) are unlikely to support microbial activity (personal observation). In this study, we used *in situ* measurements of the C isotope composition of the soil surface CO<sub>2</sub> flux to test the hypothesis that CO<sub>2</sub> emissions from dry soils originate from soil carbonates rather than biological respiration. We measured soil CO<sub>2</sub> fluxes at a range of surface temperatures, under both dry and wet conditions, and used Keeling plots to compare the  $\delta^{13}\text{C}$  with the isotopic composition of potential substrates.

## 2. Materials and Methods

### 2.1. Site

Sampling was conducted at the Nevada Desert Free-Air Carbon dioxide Enrichment (FACE) Facility (Nye County, Nevada; 36°49'N, 115°55'W; elevation 970 m), 90 km northwest of Las Vegas, Nevada, in June 2009. The site is contained within the Nevada test site (U.S. Department of Energy) and has been protected from recreation and grazing disturbances at least 55 years [Jordan *et al.*, 1999]. Mean annual rainfall is 140 mm, occurring primarily as winter storms and also as brief, localized summer events. Air temperatures at the site range from  $-10^\circ\text{C}$  (winter minimum) to  $47^\circ\text{C}$  (summer maximum). Diel temperature variation is large, and in summer, midday soil surface temperatures can reach  $>75^\circ\text{C}$  [McCalley and Sparks, 2009]. Vegetation at the site is classified as *L. tridentata*-*Ambrosia dumosa*-*Lycium* spp. desert scrub, with vegetation cover of  $\sim 20\%$ . Soils are classed as Aridsols, derived from calcareous alluvium, with carbonate values reported in the range of  $\sim 0.025 \text{ g C cm}^{-3}$  at 0–10 cm [Marion *et al.*, 2008]. Surface soils (A1 horizon to 16 cm) are loamy sands (89% sand, 6% silt, and 5% clay), with heterogeneous gravel content to 1 m and a pH of 8–9 [Jordan *et al.*, 1999]. Subsoils are well drained and lack a caliche layer.

### 2.2. Soil and Gas Sampling

Ten soil cores were taken to a depth of 5 cm beneath *L. tridentata* and plant interspaces. All soil samples were passed through a 2 mm screen to remove large rock fragments and dried at  $60^\circ\text{C}$  for 72 h. Soils were

**Table 1.** The  $\delta^{13}\text{C}$  of Soil (Organic and Carbonate Fractions) Beneath *Larrea tridentata* or Plant Interspace (0–5 cm Depth)<sup>a</sup>

	Soil %C (Carbonate)	Soil %C (Organic)	$\delta^{13}\text{C}$ Organic	$\delta^{13}\text{C}$ Carbonate
Interspace	2.15 ± 0.36	0.19 ± 0.04	−22.8 ± 1.1	0.3 ± 0.1
<i>Larrea tridentata</i>	2.48 ± 0.60	0.55 ± 0.06	−23.4 ± 0.2	0.1 ± 0.2

<sup>a</sup>Values are average ± 1 SD ( $n = 5$  per cover type).

homogenized using a mortar and pestle. Samples were analyzed for  $\delta^{13}\text{C}$  and %C using a continuous flow isotope ratio mass spectrometer (IR-MS; Model V Advantage; Thermo Scientific, Bremen, Germany). A replicate set of samples was analyzed for carbonate  $\delta^{13}\text{C}$  using a Thermo Finnigan GasBench II (Thermo, San Jose, USA) coupled to the IR-MS using the phosphoric acid-calcium carbonate reaction method outlined in Revez *et al.* [2001]. All isotope analyses were conducted at the Cornell University Stable Isotope Laboratory.

Field  $\text{CO}_2$  sampling was conducted using static chambers [McCalley *et al.*, 2011]. Soil collars (25.5 cm in diameter and 15 cm tall) were installed approximately 7.5 cm into the soil profile in June 2004. Collars were sealed with gastight transparent lids fitted with rubber septa and vents. Temperature at the soil surface was monitored over the course of each incubation using a fine-wire thermocouple inserted 1 cm into the soil, with values recorded every 1 min. Time of day when measurements were taken was staggered to provide a range of soil surface temperatures (from ~08:00 A.M. to 07:00 P.M.).

Headspace  $\text{CO}_2$  samples were measured under either dry (ambient) or postwetting conditions (18 h after the application of 10 mm water). A precipitation event of 2 mm occurred 18 days prior to the beginning of sampling (Western Regional Climate Centre). Soil water content for this site in June averages 2–3% volumetric water content [Nowak, 2012]. Headspace samples were taken every 30 min for 2 h (postwetting) or 3 h (dry soils). Samples were withdrawn using a gastight syringe fitted with a 21-gauge needle (BD, Franklin Lakes, USA). This needle gauge is large enough to avoid isotopic fractionation of  $\text{CO}_2$  during filling (Sparks and McCalley, unpublished data). Samples were injected into evacuated 20 mL glass vials fitted with butyl rubber septa and transported to Cornell University for analysis. Travel standards were employed to check for any vial leakage. The  $\delta^{13}\text{C}$ - $\text{CO}_2$  and  $\text{CO}_2$  concentrations were measured on a GasBench II coupled to the IR-MS described above.

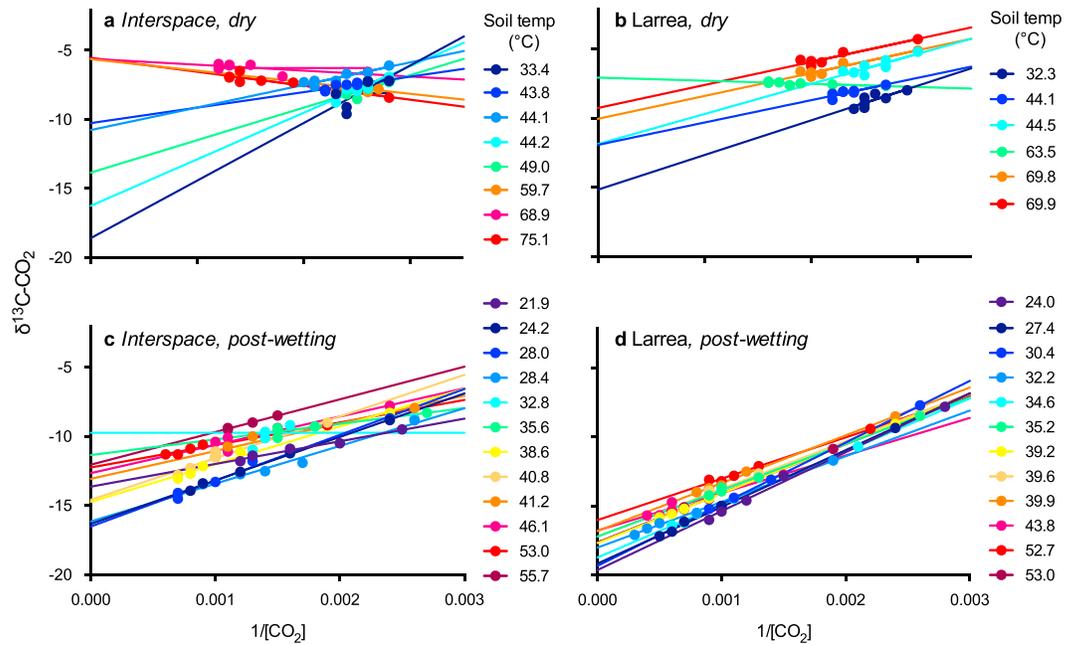
### 2.3. Statistical Analysis

Keeling plot intercepts for sample  $\text{CO}_2$  concentration and  $\delta^{13}\text{C}$  were solved using Model II (standard major axis) regression, which has been determined to be more suitable for Keeling data than simple linear regression in reducing errors when extrapolating far from the data [Pataki *et al.*, 2003]. This analysis was performed using the “lmodel2” package in R [Legendre, 2014]. Several samples (all from dry sampling) were excluded from analyses if Keeling plot linearity was poor (generally  $r^2$  less than 0.35) and slopes were nonsignificant.  $\text{CO}_2$  flux rates were determined using linear regression applied to concentration increase over time for the first 2 h of measurement. Linear regression was used to analyze the relationship between temperature and  $\text{CO}_2$  flux and source composition. Student's  $t$  test was used to compare  $\text{CO}_2$  fluxes between cover types.

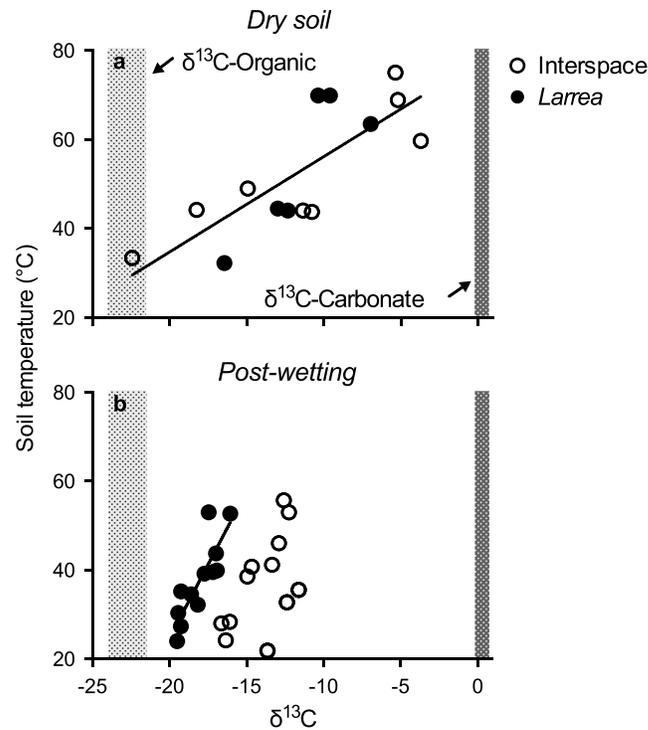
## 3. Results

The  $\delta^{13}\text{C}$  values of soil organic C and soil carbonate C at the Mojave site were well separated, averaging −22.8–−23.4‰ and 0.1–0.3‰, respectively (Table 1).

In dry soils,  $\delta^{13}\text{C}$  of the  $\text{CO}_2$  source (Keeling plot solution) moved away from the organic C value and toward the carbonate C value as soil temperature increased up from 33°C to an average of 75°C (Figures 1 and 2). This linear relationship was significant for both interspace and *Larrea* soils ( $P < 0.008$  and  $P < 0.0287$ , respectively; slope of the two lines not significantly different) and the two combined ( $\delta^{13}\text{C} = 2.14 \times \text{temp} + 77.6$ ,  $r^2 = 0.63$ ,  $P < 0.0001$ ; Figure 2). This strong relationship was not seen in postwetting soils, where the  $\delta^{13}\text{C}$  of the  $\text{CO}_2$  source varied by less than 5‰ within each vegetation type (Figures 1 and 2), although there was a significant relationship between source  $\delta^{13}\text{C}$  and temperature for *Larrea* soils ( $P < 0.0005$ ). Temperatures in postwetting soils did not exceed 55°C. In wet soils, the  $\text{CO}_2$  source value was on average 4.4‰ higher for interspace than *Larrea* soils, which contained a greater percentage of depleted organic C (Table 1).



**Figure 1.** Keeling plot for  $\delta^{13}\text{C-CO}_2$  of static chamber measurements, taken from (a and b) interspace or (c and d) *Larrea tridentata* soils at varying soil surface temperatures, under dry conditions or 3 h after a 10 mm water addition. Soil temperatures are mean values for 2 h (wet) or 3 h (dry) incubation, measured at 1 cm depth.

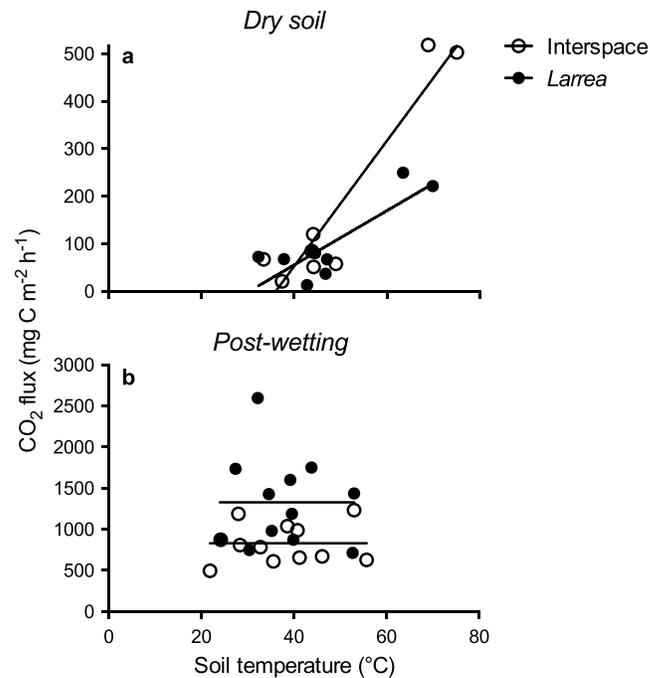


**Figure 2.** Keeling plot ( $\delta^{13}\text{C-CO}_2$  value versus inverse mixing ratio) intercepts, representing the  $\delta^{13}\text{C}$  value of the  $\text{CO}_2$  source, for interspace or *Larrea tridentata* soils under (a) dry conditions or (b) 18 h after a 10 mm water addition. The shaded gray areas represent the range of  $\delta^{13}\text{C}$  of soil carbonate and organic C (0–5 cm) at the site ( $n = \text{each}$ ). The line indicates the simple linear regression.

There was a positive linear relationship between soil  $\text{CO}_2$  flux from dry soils and soil temperature ( $^{\circ}\text{C}$ ) for both interspace and *Larrea* soils ( $P < 0.0007$  and  $P < 0.0017$ , respectively; slope of the two lines not significantly different) and for the combined cover types ( $P < 0.0001$ , flux =  $9.74 \times \text{temp} - 342.9$ ,  $r^2 = 0.71$ ; Figure 3). Postwetting fluxes were significantly greater ( $495\text{--}2595 \text{ mg C m}^{-2} \text{ h}^{-1}$  wet versus  $13\text{--}518 \text{ mg C m}^{-2} \text{ h}^{-1}$  dry) but did not show a significant relationship with temperature (Figure 3).  $\text{CO}_2$  fluxes from postwetting *Larrea* soils were significantly greater than from interspace soils ( $P < 0.0088$ ).

#### 4. Discussion

In this study, we found isotopic evidence for a shift toward a carbonate-derived  $\text{CO}_2$  source under high daytime surface soil temperatures in the carbonate-dominated soils of the Mojave desert. We also found that high surface temperatures ( $>69^{\circ}\text{C}$ ) support the greatest rates of  $\text{CO}_2$  efflux from dry soils under summer conditions.



**Figure 3.** Soil CO<sub>2</sub> flux (mg C m<sup>-2</sup> h<sup>-1</sup>) from soils beneath *Larrea tridentata* or interspace at varying soil temperatures, under (a) dry conditions or (b) 18 h after a 10 mm water addition.

Under wet conditions, it appears that microbial and/or autotrophic respiration increases significantly, leading to greater CO<sub>2</sub> fluxes and average  $\delta^{13}\text{C}_{\text{CO}_2}$  source values closer to that of soil organic C.

While natural abundance isotopic methods have been applied to show abiotic sources for CO<sub>2</sub> emissions in Antarctic dry valleys [Shanhun *et al.*, 2012], they have not to our knowledge been applied in situ to characterize CO<sub>2</sub> sources. This system is characterized by having two soil C pools (organic C and carbonate C) with distinct and nonoverlapping  $\delta^{13}\text{C}$  values, of  $\sim -23\text{‰}$  and  $\sim 0\text{‰}$ , respectively. In both *Larrea* and interspace soils under dry conditions, we found that the  $\delta^{13}\text{C}$ -CO<sub>2</sub> source (derived from Keeling plots) increased linearly with surface temperature, from  $-22\text{‰}$  at 33°C to a maximum of  $-5\text{‰}$  at 69°C. The high end of this range represents some of the most isotopically enriched C source values

reported for CO<sub>2</sub> sources from untreated soils [Pataki *et al.*, 2003]. CO<sub>2</sub> flux rate also increased linearly over this temperature range. Thus, this pattern is consistent with low fluxes of respiration-derived CO<sub>2</sub> (from soil organic matter (SOM) or root respiration) from the soil surface at lower temperatures and an increasing flux of isotopically heavy CO<sub>2</sub> derived from carbonate precipitation as temperatures increase. Although this mechanism has been reported thus far only for calcium carbonate, it is possible that nocturnal dissolution of sodium carbonate (also present in Mojave soils at this pH [Wood *et al.*, 2005]) and subsequent diurnal decomposition of sodium bicarbonate at high surface temperatures [Whittig and Janitzky, 1963] also contribute to the abiotic flux.

Although microbial respiration is often thought to increase with temperature, this relationship would be expected to reverse as soils become very hot (especially in very dry soils). Cable *et al.* [2011], for example, found that soil respiration decreased substantially between 20 and 50°C in the Sonoran desert. This, combined with isotopic evidence, suggests that the high CO<sub>2</sub> efflux we see at high daytime soil temperatures does not represent biological respiration but rather an increase in the processes thought to drive carbonate precipitation and thus CO<sub>2</sub> release. Specifically, higher soil temperatures reduce the solubility of CO<sub>2</sub> and drive convection-assisted CO<sub>2</sub> outgassing.

In contrast, rates of CO<sub>2</sub> efflux in wet soils were not temperature dependent between 24 and 55°C (wet soils do not typically reach the same high temperatures as dry soils) but were greater in *Larrea* soils than in interspaces. Water addition is routinely observed to increase biological respiration in arid (noncarbonate) soils [Austin *et al.*, 2004], and thus, this pattern fits with an interpretation of biological respiration being the primary source of CO<sub>2</sub> from these soils under wet conditions. The  $\delta^{13}\text{C}_{\text{CO}_2}$  values are offset from that of SOM (on average 5‰ enriched for *Larrea* soils and 9‰ enriched for interspace soils) and are consistent with lab incubations of Mojave soils under comparable temperature and moisture conditions [Stevenson and Verburg, 2006]. Because biological respiration is expected to have a comparatively small fractionation effect (up to 2.1‰ [Werth and Kuzyakov, 2010]), this offset suggests that in wet soils some diurnally evolved CO<sub>2</sub> is still derived from the carbonate source. Alternatively, exchange of atmospheric CO<sub>2</sub> (at  $\sim -7\text{‰}$ ) with CO<sub>2</sub> in the soil pore space (in equilibrium with soil carbonate) as soils dry during the incubation [Schlesinger, 1985] might also contribute to the apparent isotopic enrichment of the

headspace CO<sub>2</sub>. The smaller offset for *Larrea* is consistent with *Larrea* having greater rates of CO<sub>2</sub> flux, most of which is probably biological in origin and consistent with its larger soil organic C pool and possible autotrophic root respiration.

There is currently a knowledge gap in our understanding of carbon cycling in arid, carbonate-dominated systems. The data presented here add an independent line of isotopic verification to the growing body of evidence that abiotic processes are important element of the C cycle in desert soils. We show that diurnally evolved CO<sub>2</sub> arises in part from carbonate sources, providing a source to balance the nighttime CO<sub>2</sub> uptake observed in arid systems [Hamerlynck *et al.*, 2013; Liu *et al.*, 2015b; Yates *et al.*, 2012; Xie *et al.*, 2009] and likely keeping the system at (or close to) a state of C equilibrium [Schlesinger, 2016].

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