Observations of the anomalous oxygen isotopic composition of carbon dioxide in the lower stratosphere and the flux of the anomaly to the troposphere


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Measurements of the triple oxygen isotopic composition of stratospheric CO2 in whole air samples from the NASA ER-2 aircraft show anomalous enrichments in 17O and 18O. The compact correlation of the isotope anomaly (defined as $\Delta^{17}O = \delta^{17}O - 0.516 \times \delta^{18}O$) with simultaneous N2O measurements demonstrates that $\Delta^{17}O_{CO2}$ is a long-lived tracer with a stratospheric source. These characteristics, and an isotopic link to O3 production, make $\Delta^{17}O_{CO2}$ potentially useful as a tracer of integrated stratospheric chemistry and transport. The $\Delta^{17}O_{CO2}$-N2O correlation is also used to estimate a net $\Delta^{17}O_{CO2}$ flux to the troposphere of $3.6 \pm 0.9 \times 10^{15}$ mol CO2 yr$^{-1}$. This flux is required to predict and understand the CO2 and O2 isotope anomalies in the troposphere and their use as tracers of gross carbon exchanges between the atmosphere and biosphere on interannual to glacial-interglacial time scales.


1. Introduction

[2] Oxygen has 3 stable isotopes, 16O, 17O, and 18O. Most kinetic and equilibrium isotope effects alter 17O/16O ratios by half the degree to which the 18O/16O ratios change [e.g., Thiemens, 1999]. However, anomalous or “mass-independent” isotope effects, which result in 17O isotopic compositions that are not related to the 18O isotopic composition by a factor of ~0.5, are evident in many atmospheric species, including O3 [e.g., Lammerzahl et al., 2002] and stratospheric CO2 [Thiemens et al., 1991, 1995a, 1995b; Alexander et al., 2001; Lammerzahl et al., 2002]. The CO2 isotope anomaly is of interest for its potential as a tracer of stratospheric processes [e.g., Yung et al., 1997; Alexander et al., 2001; Lammerzahl et al., 2002] and of gross carbon exchanges between the atmosphere and biosphere and their sensitivity to climate change on annual [Hoag et al., 2002] to millennial [Luz et al., 1999; Blunier et al., 2002] time scales.

[3] The CO2 isotope anomaly originates in whole or in part from transfer from O3 via Equations (1)–(3) [e.g., Yung et al., 1991].

\[
\begin{align*}
O + O_2 + M &\rightarrow O_3 + M \quad (M = N_2, O_2, etc.) \quad (1) \\
O_3 + hv &\rightarrow O_2 + O(1D) \quad (2) \\
O(1D) + CO_2 &\rightarrow CO_2^+ \rightarrow CO_2 + O(3P) \quad (3)
\end{align*}
\]

Unusual isotope effects in Equation (1) result in large, anomalous enrichments in 17O and 18O in O3. While the underlying theory is still under investigation [e.g., Gao and Marcus, 2001; Babikov et al., 2003], the isotope effects themselves are now well-quantified by laboratory measurements [e.g., Mauersberger et al., 1999]. O(1D) from (2) can exchange isotopes with CO2 in (3). Whether an additional anomalous isotope effect occurs in (3) remains controversial [e.g., Wen and Thiemens, 1993; Yung et al., 1997; Barth and Zahn, 1997]. For full application of the CO2 isotope anomaly as a tracer of stratospheric and biogeochemical processes, additional information on the source and production rate of the anomaly is required.

[4] In this study, we report 29 measurements of the anomalous isotopic composition of stratospheric CO2 from whole air samples collected by the NASA ER-2 aircraft. These measurements complement the existing 52 published measurements on samples collected from a C-141 aircraft [Thiemens et al., 1995a], balloons [Thiemens et al., 1991; Alexander et al., 2001; Lammerzahl et al., 2002] and rockets [Thiemens et al., 1995b]. The correlation of the CO2 isotope anomaly with N2O mixing ratios provides new insight into the source of the anomaly, its distribution in the stratosphere, and its potential as a tracer of integrated stratospheric chemistry and transport. It also allows the flux of the anomaly to the troposphere to be estimated from the global N2O loss rate. An
Table 1. ER-2 Measurements

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*Average range: 0.08 ± 0.08 km (max = 0.27, typical <0.05).

Range: 0.01 to 0.44.

Average range: 3.7 ± 3.9 ppbv.

Accurate flux is required for using CO2 and O2 isotope anomalies as new constraints on gross carbon exchanges.

2. Measurement Description

[5] Whole air samples were collected during the POLARIS campaign [Newman et al., 1999] by an automated sampler with 29 electropolished 1.6-liter stainless steel canisters and a 4-stage metal bellows pump [Flocke et al., 1999]. The pump pressurized the canisters to 40 psia, resulting in collection times of 15–180 s at altitudes of 9–21 km, respectively, and a ~4.5 ℓ STP sample volume. After trace gas mixing ratios were measured by GC/MS, 2 to 4 ℓ of sample remained, half of which was transferred to a glass flask for isotopic analyses of N2O, CH4, H2, and N2. CO2 was cryogenically separated from the other half and immediately analyzed or stored in a glass ampoule.

[6] Due to isotopic interference between $^{17}$O-$^{12}$C-$^{16}$O and $^{16}$O-$^{17}$C-$^{16}$O, the purified CO2 samples were converted to O2 by reaction with BrF5 at 800°C for 45 hours [Thiemens et al., 1991] prior to analysis on a Finnigan MAT 251 dual inlet isotope ratio mass spectrometer at UCSD in 1997–8. The isotope compositions are reported as $\delta^{17}$O and $\delta^{18}$O on the V-SMOW scale where $\delta^{18}$O = 1000[$(^{18}O/^{16}O)_{sample}/(^{18}O/^{16}O)_{VSMOW}]−1$. Measurement precision for the isotope anomaly (defined in Equation (4) below) is estimated to be ±0.1‰. Measurements of $\delta^{15}$N of N2 at Princeton (M. Bender, personal communication) yield an upper limit of 0.1‰ for fractionation of $^{18}$O in CO2 due to sample collection and handling. Tests of standards in the canisters suggest that $\delta^{18}$O is altered by <0.1‰ after short- or long-term storage in dry canisters under laboratory conditions. Measurements of $\delta^{13}$C are not available for these samples since CO2 was converted to O2 prior to isotopic analysis.

[7] Mixing ratios of N2O and O3 were measured in situ by the Airborne Tunable Diode Laser Spectrometer [Podolske and Loewenstein, 1993] and the O3 UV spectrophotometer [Proffitt and McLaughlin, 1983]. The in situ N2O and O3 data are averaged over the WAS collection period weighted by the canister fill rate.

3. Results and Discussion

[8] Measurements of $\delta^{17}$O and $\delta^{18}$O of CO2 from the ER-2 (Table 1) are plotted in Figure 1 with previous measurements. Clearly, CO2 is anomalously fractionated since the $\delta^{17}$O-$\delta^{18}$O slope is significantly larger than slopes of 0.50 to 0.529 for mass-dependent fractionation [e.g., Thiemens, 1999]. Assuming errors of 0.5‰ for both $\delta^{17}$O and $\delta^{18}$O, a weighted least squares regression [Williamson, 1968] of the ER-2 data yields a slope of 1.47 ± 0.28 (2σ), or 1.64 ± 0.38 excluding the lowest $\delta^{18}$O datum. For comparison, the same regression and assumed errors yield slopes of 1.21 ± 0.16 (2σ) or 1.64 ± 0.38 excluding the lowest $\delta^{18}$O datum. In comparison with previous analyses, the regression and errors yield a slope of 1.21 ± 0.16 for the Thiemens et al. [1995b] rocket data, 1.72 ± 0.22 for the Lammerzahl et al. [2002] balloon data, 2.06 ± 1.16 for the Alexander et al. [2001] balloon data, and 1.37 ± 0.12 for the combined rocket data from Thiemens et al. [1995b] and Zipf and Erdman [1994]. Scatter in the ER-2 data is discussed below.
To examine the distribution of the CO$_2$ isotope anomaly in the stratosphere, we define its magnitude as

$$\Delta^{17}O_{CO_2} = \frac{\delta^{17}O}{\delta^{18}O} = \frac{\delta^{17}O}{\delta^{18}O} - 0.516 \times \delta^{18}O$$

where 0.516 was selected empirically to best represent $\Delta^{17}O_{CO_2}$ in air entering the stratosphere ($\sim 0.1\%$ for N$_2$O $\sim 315$ ppbv). In Figure 2, $\Delta^{17}O_{CO_2}$ is plotted against altitude. $\Delta^{17}O_{CO_2}$ generally increases with altitude but is highly variable, ranging from 1.9 to 9$\%$ at 20 km. Such variability is typical of long-lived tracers measured with high spatial resolution in the extratropics, a result of transport of air filaments between different regions, reversible air displacements, and the stratospheric circulation bringing older air down from higher altitudes [e.g., Boering et al., 1996]. If the local chemical time scales controlling $\Delta^{17}O_{CO_2}$ are long compared to transport times, such variability can be removed by plotting $\Delta^{17}O_{CO_2}$ against another long-lived tracer [e.g., Plumb and Ko, 1992] such as N$_2$O, which is slowly photolyzed and photo-oxidized by O(1D) with time and altitude. Figure 3 shows $\Delta^{17}O_{CO_2}$ versus N$_2$O from the ER-2 and Thiemens et al. [1995b] rocket samples, the only datasets for which N$_2$O is available. N$_2$O and $\Delta^{17}O_{CO_2}$ from the ER-2 are tightly correlated, with a nearly linear increase in $\Delta^{17}O_{CO_2}$ as N$_2$O decreases. Interestingly, the ER-2 and rocket observations overlap despite gaps of 9 km and 7 years in sample collection. In contrast, Figure 4 demonstrates that $\Delta^{17}O_{CO_2}$ does not correlate with O$_3$, a tracer with a 1–3 month lifetime at 20 km.

$\Delta^{17}O_{CO_2}$:N$_2$O correlation provides new insight into the production rate and variation of $\Delta^{17}O_{CO_2}$ in the stratosphere. First, the tight correlation for N$_2$O $> 80$ ppb demonstrates that $\Delta^{17}O_{CO_2}$ is a long-lived tracer. Thus, while it has been suggested [Thiemens et al., 1995b; Thiemens, 1999] that the negative $\Delta^{17}O_{CO_2}$:N$_2$O correlation results from a common connection with O(1D), the correlation would result from transport even in the absence of this commonality. This conclusion is further supported by the tight correlation of $\Delta^{17}O_{CO_2}$ with mean ages derived from ER-2 CO$_2$ mixing ratios [not shown; see Andrews et al., 2001], which are not influenced by O(1D), and by the larger scatter in the correlation in the upper stratosphere and mesosphere (i.e., for N$_2$O $< 50$ ppb), where the N$_2$O lifetime is comparable to the transport time scales.

Second, the $\Delta^{17}O_{CO_2}$:N$_2$O correlation is consistent with Yung’s chemical mechanism (1)–(3) coupled with stratospheric transport. Production of $\Delta^{17}O_{CO_2}$ is probably most rapid in the upper tropical stratosphere where production rates of O$_3$ and O(1D) are highest. When air descends via the stratospheric circulation, the large upper stratospheric signal in $\Delta^{17}O_{CO_2}$ is long-lived even far from the main source region. Thus, large values of $\Delta^{17}O_{CO_2}$ in the lower stratosphere result from the stratospheric circulation and not from “particle precipitation” (cosmic ray) events in the polar regions which could generate large amounts of O(1D)
in the lower stratosphere, an alternative hypothesis of Lammerzahl et al. [2002] to explain the large $\Delta^{17}O_{CO_2}$ values observed near 22 km. [12] Third, the negative $\Delta^{17}O_{CO_2}$:N$_2$O correlation demonstrates that $\Delta^{17}O_{CO_2}$ has a stratospheric source but weak, if any, sink. The primary sink for $\Delta^{17}O_{CO_2}$ (and (1)–(3) in general) should be mass-dependent isotope exchange with liquid water in plant leaves, soils, and the ocean [e.g., Farquhar et al., 1993, based on $\delta^{18}O$]. While Yang et al. [1991] and Alexander et al. [2001] have suggested that $\Delta^{17}O_{CO_2}$ could be a tracer of $O(1D)$ abundances and of transport in regions where tracers like N$_2$O and CH$_4$ are losing their dynamic range, the applications of a tracer with a stratospheric source but weak to no sink may be even broader ranging. For example, when the molecular-level details of the isotope chemistry producing $\Delta^{17}O_{CO_2}$ are known, a tracer with such a stratospheric source may serve as a new diagnostic for transport errors in 2D and 3D models and in predicting the build-up of pollutants from stratospheric aircraft [e.g., Hall et al., 1999]. Furthermore, if $\Delta^{17}O_{CO_2}$ arises solely from transfer from O$_3$ (i.e., no anomalous isotope effect in (3)), then $\Delta^{17}O_{CO_2}$ is a tracer with production and transport terms that are proportional to those of odd oxygen but with no free radical loss term. Thus, $\Delta^{17}O_{CO_2}$ could be used to account for integrated O$_3$ production and transport while leaving integrated O$_3$ chemical loss as the free parameter to compare with model results and instantaneous loss rates from free radical measurements. In the meantime, simply the unusual curvilinear $\Delta^{17}O_{CO_2}$:N$_2$O correlation may provide additional constraints on transport and mixing in the stratosphere to mesosphere that are complementary to those from other tracer:tracer combinations.

[13] Fourth, the $\Delta^{17}O_{CO_2}$:N$_2$O correlation can be used to quantify the flux of $\Delta^{17}O_{CO_2}$ to the troposphere. The slope of two long-lived tracers is equal to the ratio of their net vertical fluxes [Plumb and Ko, 1992]. For N$_2$O, the net vertical flux is the global N$_2$O loss rate, or 13 TgN yr$^{-1}$ + 25% [Prather and Ehhalt, 2001]. Fluxes to the troposphere have been estimated in this manner for NO$_x$ and O$_3$ [Mishchenko and Fahey, 1994; McLinden et al., 2000] and, using a small, unpublished subset of the ER-2 data reported here, for $\Delta^{17}O_{CO_2}$ [Luz et al., 1999]. Here, we apply the Plumb and Ko relationship to the ER-2 data for N$_2$O > 195 ppb, restricting N$_2$O for several reasons. First, mixing in the vortex, confirmed by CH$_4$ and N$_2$O isotope measurements from POLARIS [McCarthy et al., 2003; Park et al., 2003], results in a slightly larger variability in the $\Delta^{17}O_{CO_2}$:N$_2$O correlation for N$_2$O between 80 and 150 ppbv. Second, higher N$_2$O values are most relevant for air returning to the troposphere [Olsen et al., 2001]. The resulting net isotope flux of $\Delta^{17}O_{CO_2}$ is 3.6 ± 0.9‰ Pm1oCO$_2$ yr$^{-1}$. The net flux is independent of the mass flux of air and in general is the quantity needed for biogeochemistry applications. A stratosphere-to-troposphere flux can be derived using air mass flux calculations from, e.g., Holton [1990] or Appenzeller et al. [1996], yielding fluxes for $\Delta^{17}O_{CO_2}$ of 3.9 and 4.4‰ Pm1oCO$_2$ yr$^{-1}$, respectively. Overall, the largest uncertainty for both the downward and net $\Delta^{17}O_{CO_2}$ fluxes is the N$_2$O loss rate. Uncertainty in the $\Delta^{17}O_{CO_2}$:N$_2$O fit is 3%, and the slope is expected to be robust with respect to both season and latitude based on the ER-2 CH$_4$ and N$_2$O isotope analyses noted above. Thus, these fluxes should be accurate to 25% as well as precise. Finally we note that, using the ER-2 data reported here, estimates of the stratospheric contribution to the current O$_3$ isotope anomaly in LUZ99 do not change.

[14] Several significant uncertainties remain. While the slope and 2σ uncertainty of the ER-2 $\delta^{17}O$:δ$^{18}O$ relationship is within the estimated 2σ uncertainties of slopes from the other datasets, there is significantly more scatter in δ$^{17}O$ and δ$^{18}O$ than in Lammerzahl et al. [2002] and Thiemens et al. [1995b]. The scatter could perhaps be explained by mixing in the lower stratosphere or a seasonal variation in tropospheric CO$_2$ isotope compositions [Troller et al., 1996] entering the stratosphere. However, seasonal variations should damp out for N$_2$O < ~275 ppbv [e.g., Andrews et al., 2001] and the scatter does not, and mixing alone cannot explain how $\delta^{18}O$ lighter than tropospheric CO$_2$ could result. The tight $\Delta^{17}O_{CO_2}$:N$_2$O correlation suggests that the $\delta^{17}O$:δ$^{18}O$ scatter is mass-dependent. Measurements of δ$^{17}N$ of N$_2$ indicate this mass-dependent fractionation cannot be due to sample handling. One explanation for the scatter, if not real atmospheric variability, is fractionation in the BrF$_5$ CO$_2$-O$_2$ conversion (e.g., see Figure 2) of Brenninkmeijer et al. [1995]: δ$^{18}O_{CO_2}$ varied by 0.3‰ in a set of samples before BrF$_5$ conversion to O$_2$ and by 1.4‰ after, although laboratory controls suggest this is not a problem. Another explanation is isotopic exchange with water on the surface of ER-2 canisters. During cryogenic separation, excess residual water was found in several samples; excluding these samples reduces but does not eliminate the scatter in Figure 1. It also cannot be ruled out that exchange with collected stratospheric H$_2$O can occur; we calculate that 360 ppmv of CO$_2$ exchanging with 6 ppmv of highly isotopically-depleted H$_2$O in the lower stratosphere [Johnson et al., 2001] could alter δ$^{18}O$ by 2‰ while changing $\Delta^{17}O_{CO_2}$ by only 0.1‰. Future analyses using different CO$_2$-O$_2$ conversion techniques and on samples which were rapidly removed from dry canisters should help to address this uncertainty. Finally, questions remain as to whether there is an additional anomalous isotope effect in (3), and what molecular details determine the magnitude of $\Delta^{17}O_{CO_2}$. Further stratospheric observations, combined with continuing laboratory experiments and modeling, should help to resolve these remaining questions and provide an understanding sufficient to use $\Delta^{17}O_{CO_2}$ as a tracer of stratospheric processes and as the basis for new proxies of gross carbon exchanges between the atmosphere and biosphere.

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