



**Johnson, Gareth and Raistrick, Mark and Mayer, Bernhard and Shevalier, Maurice and Taylor, Steve and Nightingale, Michael and Hutcheon, Ian (2009) The use of stable isotope measurements for monitoring and verification of CO2 storage. Energy Procedia, 1 (1). pp. 2315-2322. ISSN 1876-6102 , <http://dx.doi.org/10.1016/j.egypro.2009.01.301>**

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GHGT-9

## The use of stable isotope measurements for monitoring and verification of CO<sub>2</sub> storage

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### Abstract

Stable isotope data can assist in successful monitoring of the fate of injected CO<sub>2</sub> in enhanced oil recovery and geological storage projects. This is demonstrated for the International Energy Agency Greenhouse Gas Weyburn-Midale CO<sub>2</sub> Monitoring and Storage Project (Saskatchewan) and the Pembina Cardium CO<sub>2</sub> Monitoring Project (Alberta) where fluid and gas samples from multiple wells were collected and analyzed for geochemical and isotopic compositions. In both projects, C and O isotope values of injected CO<sub>2</sub> were sufficiently distinct from those of background CO<sub>2</sub> in the reservoir. Consequently C and O isotope ratios constitute a suitable ‘fingerprint’ for tracing the fate of injected CO<sub>2</sub> in the respective reservoirs.

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**Keywords:** CO<sub>2</sub> Storage; Monitoring; Geochemistry; Stable isotopes; Carbon; Oxygen

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### 1. Introduction

Global atmospheric concentrations of carbon dioxide (CO<sub>2</sub>) have increased markedly as a result of human activities since 1750 and now far exceed pre-industrial values determined from ice cores spanning several hundred thousands of years [1]. The recent global increase in atmospheric carbon dioxide concentration is due primarily to fossil fuel combustion [2]. At the current rate of increase in atmospheric CO<sub>2</sub> concentrations (~2ppm/year) further warming and changes in the global climate system will very likely be larger than those already observed during the 20<sup>th</sup> century [2]. It is thus likely that anthropogenic CO<sub>2</sub> emissions must be reduced to stabilize the climate “to preserve a planet on which civilization developed and to which life on Earth is adapted” [3].

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Carbon Capture and Storage (CCS) is a promising component of any serious plan to reduce anthropogenic CO<sub>2</sub> emissions to the atmosphere and thereby mitigate future impacts of human-induced climate change [4]. CCS associated with the combustion of fossil fuels allows for near carbon neutrality (typically capture is 90% of CO<sub>2</sub> emissions) whilst still exploiting the energy gain from carbon based fuels. A successful CCS project must demonstrate that the injected CO<sub>2</sub> is safely and securely sequestered in the chosen reservoir. Combined geophysical and geochemical monitoring programs have the potential to determine the fate of the injected CO<sub>2</sub> in the reservoir and to assess the nature and effectiveness of the storage mechanisms [5].

As CO<sub>2</sub> is injected into reservoirs it will undergo a number of physical and chemical reactions [6]. CO<sub>2</sub> density will increase with increasing depth and when the CO<sub>2</sub> reaches its critical point (31°C and 74bar) at depths of approximately 800m [7] in the subsurface it will become a supercritical fluid. Interaction between the CO<sub>2</sub> and the reservoir fluids will lead to a variety of dissolution processes and partitioning of the CO<sub>2</sub> into different phases. A portion of the CO<sub>2</sub> will stay as free phase supercritical fluid, some will dissolve in residual hydrocarbons in the reservoir and some will dissolve in the reservoir fluids. In the latter case CO<sub>2</sub> will undergo a number of chemical reactions that include the following [8,9]:



After commencement of CO<sub>2</sub> injection, geochemical patterns in reservoir fluids and gases will reveal the above-mentioned processes. For example, the production of hydrogen ions (equation 2) will decrease pH while the bicarbonate ions produced in the same equation will increase alkalinity when the hydrogen ions are buffered by water-rock reactions. Geochemical data obtained for reservoir fluids and gases can therefore be used to monitor the fate of CO<sub>2</sub> in the subsurface. This is particularly true if injected CO<sub>2</sub> can be distinguished from carbon compounds in the reservoir by tracer techniques. Stable isotope ratios of carbon and oxygen (expressed as δ<sup>13</sup>C and δ<sup>18</sup>O values) can be easily determined for injected CO<sub>2</sub> and carbon compounds in reservoir fluids and gases by a suitable geochemical monitoring and measurement program. It is essential to determine the isotopic composition of CO<sub>2</sub> (and DIC) in the reservoir prior to CO<sub>2</sub> injection. If the injected CO<sub>2</sub> is isotopically distinct with respect to baseline CO<sub>2</sub>, stable isotope ratios can be used as ‘fingerprints’ and facilitate monitoring of the migration and behavior of the CO<sub>2</sub> in the reservoir after commencement of CO<sub>2</sub> injection. The objective of this paper is to demonstrate the usefulness of isotopic tracers for geochemical monitoring of injected CO<sub>2</sub>.

## 2. Study areas

Data from two CO<sub>2</sub>-Enhanced Oil Recovery (EOR) projects in western Canada are used to demonstrate the feasibility of using geochemical and isotopic data to trace the fate of injected CO<sub>2</sub> in the subsurface. The sites are the International Energy Agency Greenhouse Gas Weyburn - Midale CO<sub>2</sub> Monitoring and Storage Project in southern Saskatchewan and the Pembina Cardium CO<sub>2</sub> Monitoring Project in central Alberta (Fig. 1). The Mississippian Midale beds of the carbonate reservoir at Weyburn consist of an upper dolostone ‘Marly’ unit and a lower limestone ‘Vuggy’ unit into which CO<sub>2</sub> has been injected since 2000 [10]. In contrast, CO<sub>2</sub> at the Pembina project is injected into the siliciclastic Upper Cretaceous Cardium formation that comprises a series of stacked sandstone reservoirs (see companion paper by Nightingale et al. for full mineralogy). This provides an excellent opportunity to study and compare the interaction of CO<sub>2</sub> with two contrasting sedimentary reservoir lithologies: siliciclastics and carbonates.

## 3. Methods

To monitor changes in the CO<sub>2</sub> distribution in the reservoir, the geochemical and isotopic composition of the fluids and gases must be determined at regular intervals throughout the duration of the project. One critical aspect is the establishment of a baseline pre-injection study. The baseline study will reveal both the ‘natural’ state of the reservoir fluids before CO<sub>2</sub> injection and should also aim at elucidating natural variability in the reservoir.

Sample collection differs slightly at the two sites due to the operation of the reservoir and chemical composition of the fluids (e.g. high H<sub>2</sub>S values at Weyburn), however generalities can be drawn. Emberley et al. [11] described

the procedures in detail, that, in summary, are the collection of multiple gas, oil and water samples at each well monitored in the project and the subsequent analyses. At Pembina 8 wells were sampled and at Weyburn 43 were sampled. Analysis of the fluid and gas samples revealed up to 40 individual parameters for each well on each of 3 baseline and 28 monitoring trips at Pembina and 1 baseline and 10 monitor trips at Weyburn. These included: alkalinity, pH, gas composition ( $\text{CO}_2$ ,  $\text{H}_2\text{S}$ , C1-4), major ionic species (Na, K, Cl, Ca, Mg, Fe etc) and C, O and S isotopic compositions of various compounds.

$\text{CO}_2$  in the casing gas from the wells was collected in sealed containers and the chemical composition was analyzed in the field using a Varian CP4900 Micro GC. In the laboratory, the samples were analyzed for carbon isotope ratios of  $\text{CO}_2$  ( $\delta^{13}\text{C}_{\text{CO}_2}$ ) using a GC coupled to an isotope ratio mass spectrometer (IRMS).  $\delta^{13}\text{C}$  values are recorded in per mil (‰) relative to V-PDB. Accuracy and precision for the  $\delta^{13}\text{C}_{\text{CO}_2}$  values is better than  $\pm 0.5$  ‰. Oxygen isotope ratios of  $\text{CO}_2$  ( $\delta^{18}\text{O}_{\text{CO}_2}$ ) were also determined by comparison against oxygen isotope ratios of  $\text{CO}_2$  generated from internationally accepted reference materials and are reported versus V-SMOW. Samples for oxygen isotope ratio determinations on reservoir fluids were collected in 15 mL plastic vacu-tubes and  $\delta^{18}\text{O}$  values were determined by using equilibration techniques coupled with dual inlet IRMS. Oxygen isotope ratios are recorded using the usual delta ( $\delta$ ) notation ( $\delta^{18}\text{O}_{\text{H}_2\text{O}}$ ) in per mil (‰) deviation relative to V-SMOW with an analytical uncertainty of  $< \pm 0.2$  ‰.

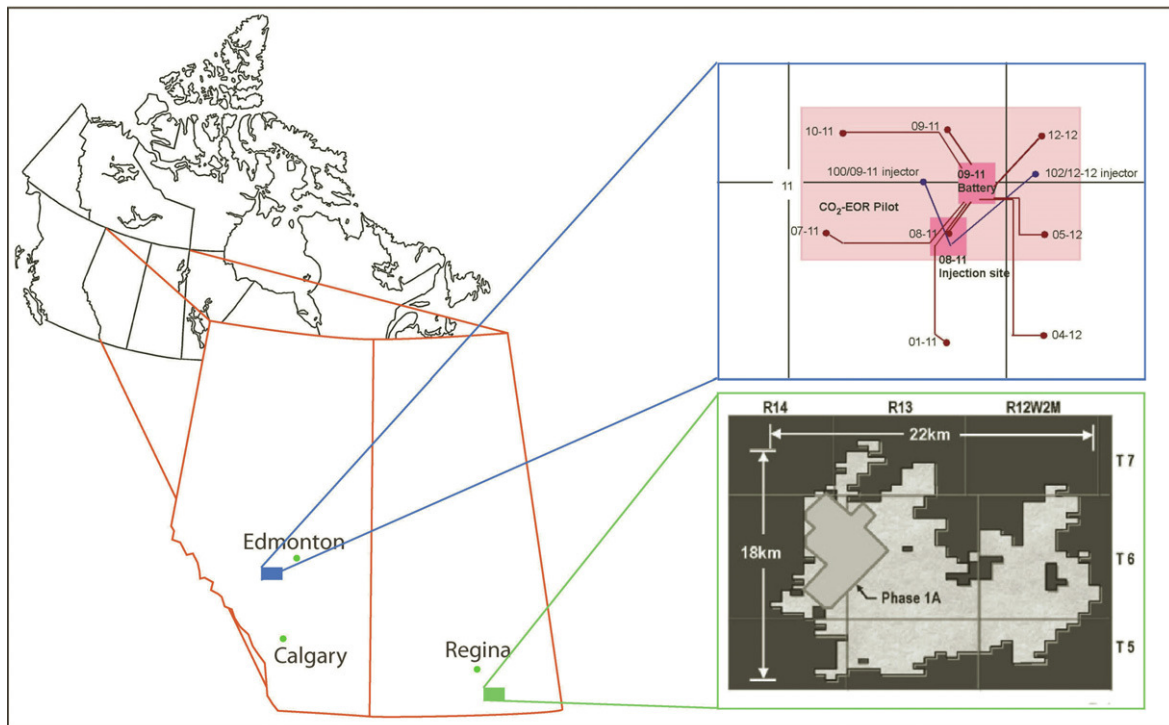


Figure 1: Locations of study sites in western Canada. Blue box Pembina Cardium  $\text{CO}_2$  Monitoring Project. Green Box: International Energy Agency Greenhouse Gas Weyburn-Midale  $\text{CO}_2$  Monitoring and Storage Project.

#### 4. Results

$\text{CO}_2$  contents (in mole %) and  $\delta^{13}\text{C}$  of  $\text{CO}_2$  and  $\delta^{18}\text{O}$  of  $\text{H}_2\text{O}$  values were determined for both projects and are displayed for selected observation wells in Figures 2 and 3.

#### 4.1. Carbon isotope ratios

Figure 2 shows both the mole % CO<sub>2</sub> and the  $\delta^{13}\text{C}_{\text{CO}_2}$  values against time lapsed in the monitoring project for two selected wells at Weyburn. Baseline  $\delta^{13}\text{C}$  values of CO<sub>2</sub> gas ( $\delta^{13}\text{C}_{\text{CO}_2}$ ) were between -12‰ and -14‰ while baseline CO<sub>2</sub> concentrations were < 10 mole %. The injected CO<sub>2</sub> at Weyburn comes from a coal gasification plant in North Dakota and has a  $\delta^{13}\text{C}_{\text{CO}_2}$  value of -20.4 ‰. After CO<sub>2</sub> injection commenced, geochemical data for Well 1 revealed that CO<sub>2</sub> contents increased to reach values of 70-90 mole % (Fig. 2a) after over a year of injection whilst  $\delta^{13}\text{C}_{\text{CO}_2}$  values steadily decreased to values near -20 ‰ (Fig. 2b). In contrast, geochemical data for Well 2 displayed only a minor increase in CO<sub>2</sub> concentration with time (Fig. 2d) while the  $\delta^{13}\text{C}_{\text{CO}_2}$  values trended towards values near -20 ‰ circa one year after CO<sub>2</sub> injection began (Fig. 2e).

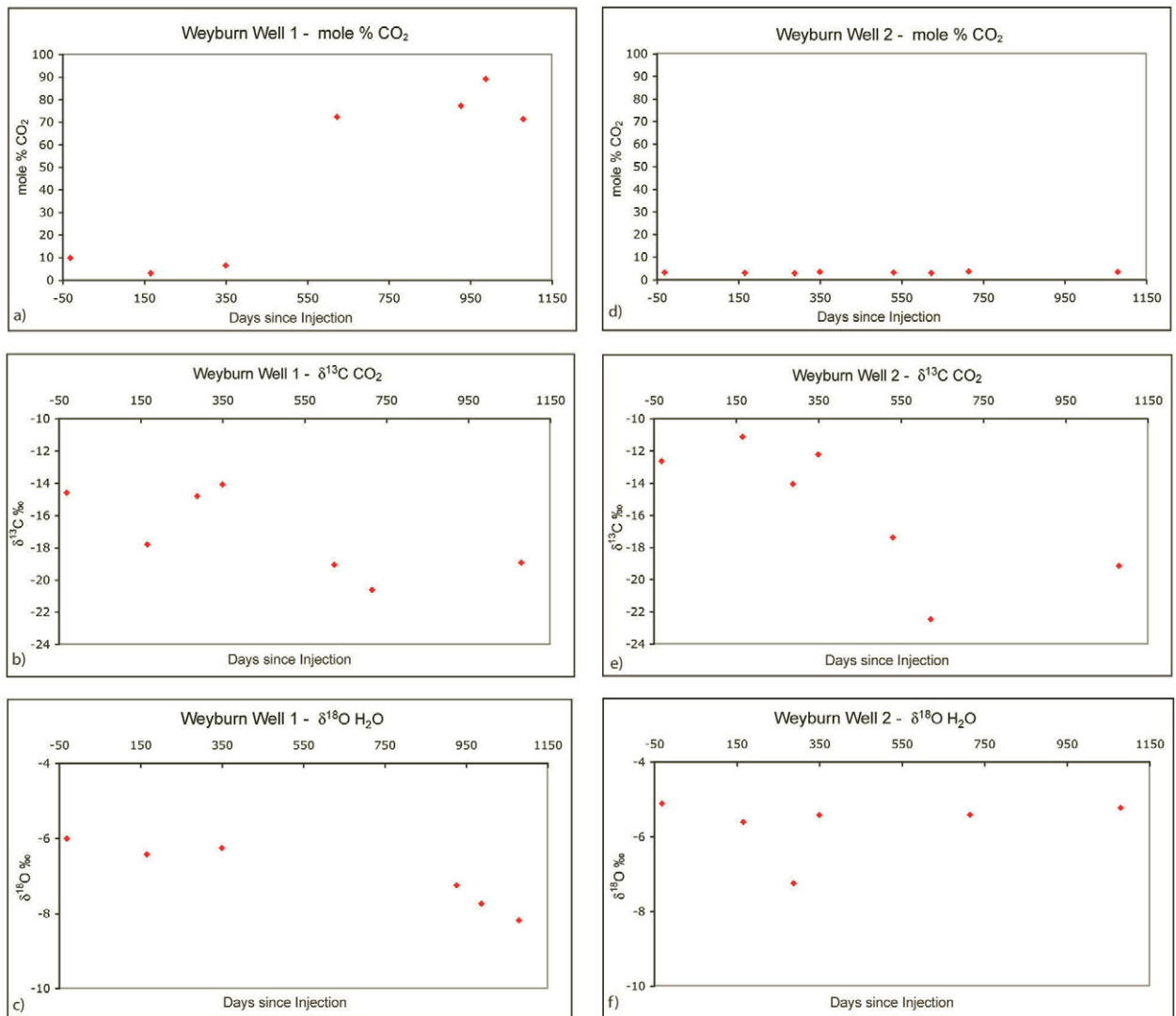


Figure 2: Weyburn isotope ratios. a) Mole % CO<sub>2</sub> vs. Days since Injection, Weyburn Well 1. b)  $\delta^{13}\text{C}_{\text{CO}_2}$  vs. Days since Injection, Weyburn Well 1. c)  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  vs. Days since Injection, Weyburn Well 1. d) Mole % CO<sub>2</sub> vs. Days since Injection, Weyburn Well 2. e)  $\delta^{13}\text{C}_{\text{CO}_2}$  vs. Days since Injection, Weyburn Well 2. f)  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  vs. Days since Injection, Weyburn Well 2.

Figure 3 summarizes both the mole % CO<sub>2</sub> and the  $\delta^{13}\text{C}_{\text{CO}_2}$  values against time lapsed in the monitoring project for two selected wells at Pembina. Baseline  $\delta^{13}\text{C}$  values for CO<sub>2</sub> gas were between -16 ‰ and -18 ‰ and CO<sub>2</sub>

concentrations at baseline were  $\sim 1$  mole %. CO<sub>2</sub> injected at Pembina is trucked in daily and has a mean  $\delta^{13}\text{C}$  value of  $-4.5 \pm 2$  ‰ depending on the source of the CO<sub>2</sub>. Geochemical data for Well 1 revealed that CO<sub>2</sub> concentrations increased rapidly as CO<sub>2</sub> injection began to maximum values around 90% (Fig. 3a) within 6 months of injection. At the same time  $\delta^{13}\text{C}_{\text{CO}_2}$  values increased rapidly towards values of  $-5$  ‰ (Fig. 3b). Geochemical data for Well 2 showed negligible changes in CO<sub>2</sub> concentration with time (Fig. 3d) while  $\delta^{13}\text{C}$  values trended towards  $-8$  ‰ over a period of 6 months after injection began (Fig. 3e).

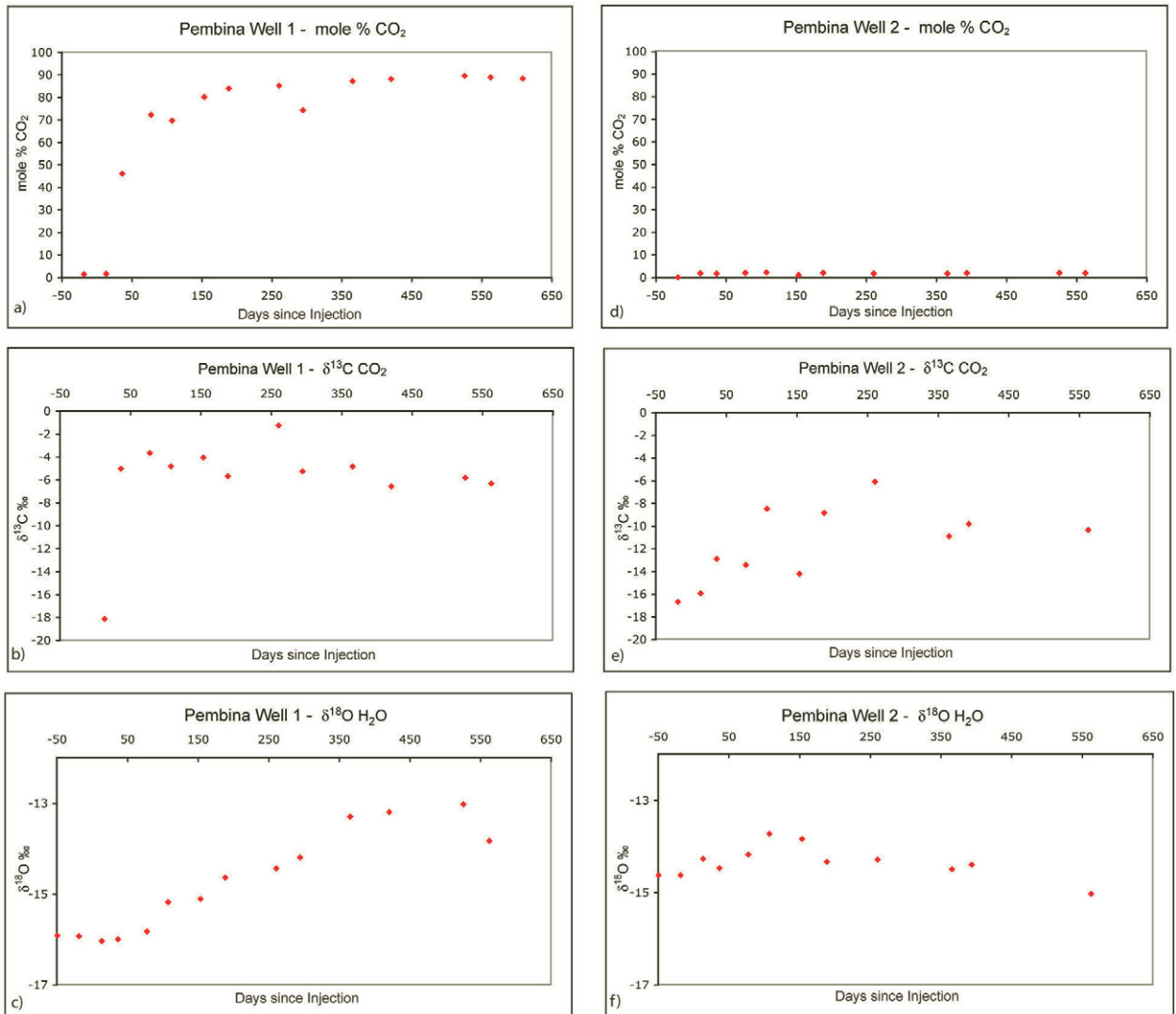


Figure 3: Pembina isotope ratios. a) Mole % CO<sub>2</sub> vs. Days since Injection, Pembina Well 1. b)  $\delta^{13}\text{C}_{\text{CO}_2}$  vs. Days since Injection, Pembina Well 1. c)  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  vs. Days since Injection, Pembina Well 1. d) Mole % CO<sub>2</sub> vs. Days since Injection, Pembina Well 2. e)  $\delta^{13}\text{C}_{\text{CO}_2}$  vs. Days since Injection, Pembina Well 2. f)  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  vs. Days since Injection, Pembina Well 2.

#### 4.2. Oxygen isotope ratios

Oxygen isotope ratios ( $\delta^{18}\text{O}$ ) of injected CO<sub>2</sub> and of fluids ( $\delta^{18}\text{O}_{\text{H}_2\text{O}}$ ) obtained from the same observation wells described above were determined. Baseline  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  values for brines at Weyburn were  $\sim -6$  ‰. Injected CO<sub>2</sub> had a  $\delta^{18}\text{O}$  value of  $+4.9$  ‰ vs. V-SMOW. As CO<sub>2</sub> content increased at Well 1 (Fig. 2a) the  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  values of the



reservoir brine decreased by approximately 2 ‰ (Fig. 2c). In contrast, the  $\delta^{18}\text{O}$  value of the reservoir brine remained unchanged at  $\sim -6$  ‰ at Well 2 (Fig. 2f) where no significant change in  $\text{CO}_2$  concentration was observed (Fig. 2d).

At Pembina the baseline  $\delta^{18}\text{O}$  values for the reservoir fluids were  $-15$  to  $-16$  ‰. Injected  $\text{CO}_2$  had a  $\delta^{18}\text{O}$  value of  $+28.7$  ‰ vs. V-SMOW. As  $\text{CO}_2$  concentrations increased in Well 1 (Fig. 3a)  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  values increased by  $\sim 3$  ‰ (Fig. 3c). In contrast the  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  values remained unchanged ( $-15$  ‰) in Well 2 (Fig. 3f) that experienced no significant increase in  $\text{CO}_2$  contents (Fig. 3d).

## 5. Discussion

In assessing trends of  $\delta^{13}\text{C}_{\text{CO}_2}$ , the detection of  $\text{CO}_2$  across the spatial extent of the reservoir is addressed. Figures 2 and 3 show that as  $\text{CO}_2$  concentrations increase, the  $\delta^{13}\text{C}_{\text{CO}_2}$  values trend towards that of the injected  $\text{CO}_2$ . Note that this trend away from baseline  $\delta^{13}\text{C}$  values towards that of the injected  $\text{CO}_2$ , in opposite senses of direction (towards positive or negative), is simply as a result of the Weyburn  $\text{CO}_2$  having a more negative isotope signature than the baseline  $\text{CO}_2$ , and conversely, the Pembina  $\text{CO}_2$  having a more positive  $\delta^{13}\text{C}$  value. This trend is indicative of injected  $\text{CO}_2$  migrating towards the wells where the  $\text{CO}_2$  is sampled.

Well 2 at both Weyburn (Fig. 2d,e) and Pembina (Fig. 3d,e) show that even with very small increases in  $\text{CO}_2$  concentration at the wells, the  $\delta^{13}\text{C}_{\text{CO}_2}$  values trend toward the value of the injected  $\text{CO}_2$ . Figure 4 explains these trends further. The data plotted as crosses on Figure 4 are the data represented in Figures 2 and 3. The solid lines represent the theoretical two endmember mixing curve between background and injected  $\text{CO}_2$ . Where initial concentrations of  $\text{CO}_2$  are low, as at both wells at Pembina, the addition of small amounts of  $\text{CO}_2$  rapidly changes the  $\delta^{13}\text{C}$  value of the sample. At 10 mole %  $\text{CO}_2$ , more than 90% of the  $\text{CO}_2$  will be from the injection source and hence the  $\delta^{13}\text{C}$  values approach those of the injected  $\text{CO}_2$ . At Weyburn, where initial  $\text{CO}_2$  concentrations are markedly higher, additional injected  $\text{CO}_2$  will change the carbon isotope value at different rates. At Well 1, with 9%  $\text{CO}_2$  at baseline, the carbon isotope response will be slower than at Well 2 (3%  $\text{CO}_2$  at baseline) assuming a similar rate of injected  $\text{CO}_2$  reaching each well. At the point when injected  $\text{CO}_2$  comprises 90% of the sample at Weyburn, mole %  $\text{CO}_2$  has to be significantly higher than at Pembina (90 and 40% for Well 1 and 2 respectively). Thus, Figure 4 indicates also that  $\delta^{13}\text{C}_{\text{CO}_2}$  can be used as an early identifier of injected  $\text{CO}_2$  breakthrough at individual wells. It also indicates that significant attention must be paid to both the distinction between background and injected  $\text{CO}_2$  isotope values and to the host lithology (and hence initial  $\text{CO}_2$  concentrations in the reservoir) when monitoring  $\text{CO}_2$  projects.

The  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  trends reveal the extent to which oxygen in the  $\text{CO}_2$  has been exchanged with the oxygen in the reservoir brines. Kharaka et al. [12] showed that in a  $\text{CO}_2$  storage setting the volume of  $\text{CO}_2$  sourced oxygen in the system can increase to the point where it influences the  $\delta^{18}\text{O}$  values of the reservoir fluids. Due to the relatively low abundance of oxygen that could be liberated by water-rock interactions, the only other reaction that could change the value of the  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  would be water injection into the reservoir. Water injection would also change the hydrogen isotope values ( $\delta^2\text{H}$ ). The wells that showed change in  $\delta^2\text{H}$  were omitted from this study so that changes in  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  were due to  $\text{CO}_2$  interaction only. In Figure 2 and 3 it can be seen that when  $\text{CO}_2$  concentration increased significantly the  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  value is influenced whereas when  $\text{CO}_2$  concentration does not change significantly the  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  value remains constant. This indicates that in Well 1 at both Weyburn and Pembina there is enough  $\text{CO}_2$  in contact with the water to influence its  $\delta^{18}\text{O}$  value. Conversely, at Well 2 at both sites there is too little  $\text{CO}_2$  to change the  $\delta^{18}\text{O}_{\text{H}_2\text{O}}$  value.

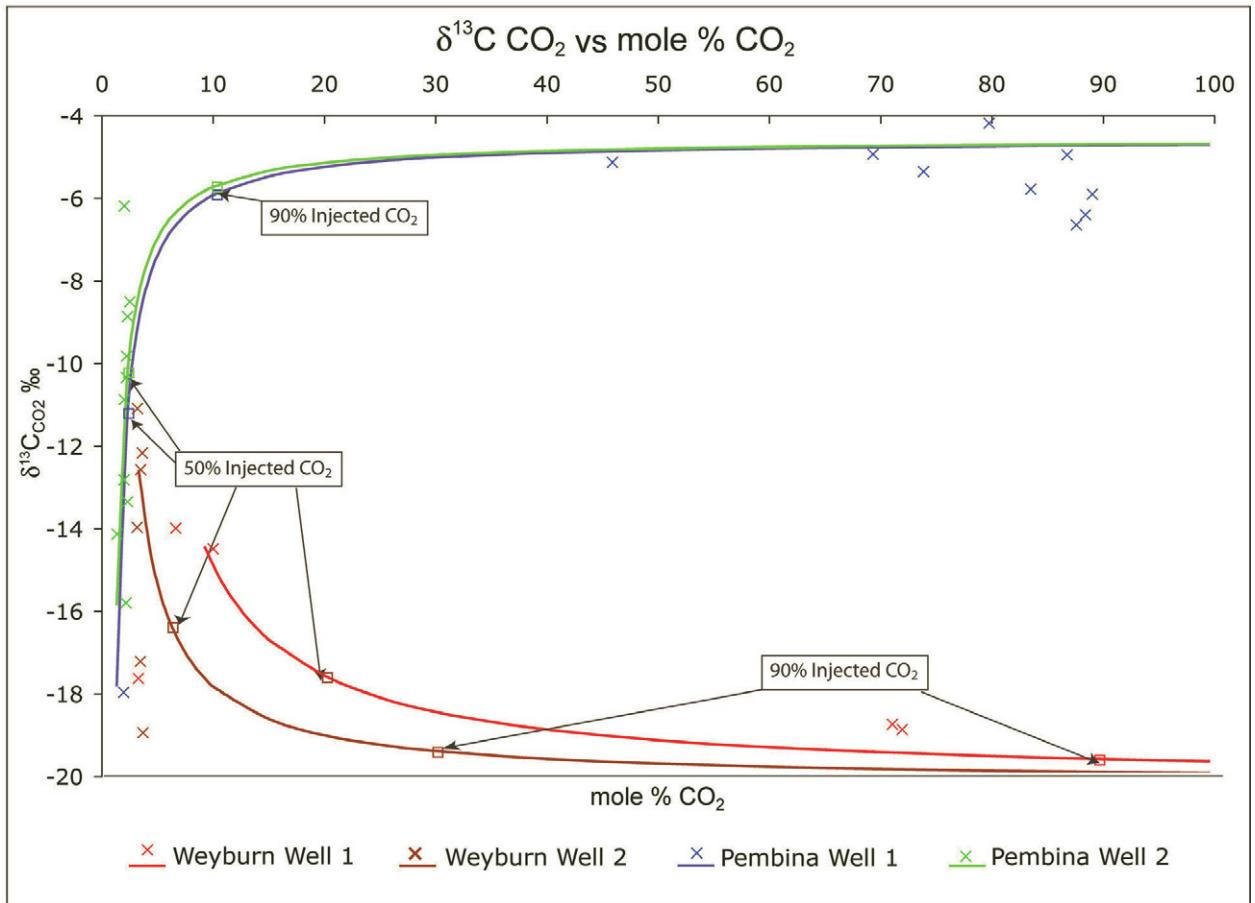


Figure 4.  $\delta^{13}\text{C}_{\text{CO}_2}$  versus mole %  $\text{CO}_2$  for Wells 1 and 2 at both Weyburn and Pembina. Solid lines show the theoretical two endmember mixing curve between background and injected  $\text{CO}_2$  for each well. Crosses represent measurements taken at all four wells. The squares and labels indicate the theoretical points at which injected  $\text{CO}_2$  comprises 50% and 90% of the  $\text{CO}_2$  sample at each well.

## 6. Conclusion

Regulatory and safety issues dictate that successful CCS projects will require the ability to trace the fate of  $\text{CO}_2$  in the reservoir. Carbon isotope ratios are an effective tool to trace the movement and reaction of injected  $\text{CO}_2$  in mature oil fields, provided that the injection  $\text{CO}_2$  is isotopically distinct. In addition, trends observed in the  $\delta^{18}\text{O}$  values of produced water at several wells at Pembina and Weyburn point towards an increasing amount of  $\text{CO}_2$  sourced oxygen in the produced waters. Geochemical data taken obtained for fluids from the same wells as this study at Pembina (see companion paper by Shevalier et al.) show further evidence that  $\text{CO}_2$  is causing water-rock reactions in the reservoir and provides additional means to detect  $\text{CO}_2$  presence in the reservoir.

Further work to quantify the amount of  $\text{CO}_2$  sourced oxygen and thus to quantify the amount of  $\text{CO}_2$  dissolved in the produced waters using the  $\delta^{18}\text{O}$  values is currently being pursued.

## Acknowledgements

Funding for the Pembina Cardium  $\text{CO}_2$  Monitoring Project was received from Penn West Energy Trust, Alberta Energy Research Institute, Western Economic Diversification Canada, Natural Resources Canada and the Alberta



Government. Funding for the IEA GHG Weyburn-Midale CO<sub>2</sub> Monitoring and Storage Project is provided by the Petroleum Technology Research Centre in collaboration with EnCana Corporation (the operator of the Weyburn oil field).

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