**Principals of Isotope Geochemistry and Applications to Groundwater Investigations**

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**ABSTRACT**

Isotope geochemistry has emerged as a formidable geochemical tool in groundwater investigations for coal combustion residuals (CCR). Specifically, isotopic "fingerprinting" of dissolved metals such as boron (δ^{11}B), lithium (δ^{7}Li), and strontium (87Sr/86Sr), or of the water molecules themselves (i.e., δ^{2}H, δ^{18}O, or tritium), serves as a proxy for common contaminants of concern and provides powerful insights into several important topics, including (1) identification of alternate sources of contamination, (2) plume delineation, (3) contaminant migration over time, (4) site assessment, (5) attenuation predications, and (6) seepage rates.

As water flows through a matrix (e.g., coal ash, soil, or rock), metals leach from particle surfaces and dissolve into the water. As a result, the water acquires the same diagnostic isotopic composition as the matrix that it flowed through or interacted with, regardless of the concentration, pH, or redox potential. Furthermore, inputs of less than 1% from a given source are often discernable using isotopic fingerprinting. A distinct advantage of using hydrogen and oxygen isotopes of water is that these isotopes are a property of the water itself and not subject to adsorption or retardation. Isotopes of the water molecule are sensitive indicators of both physical and chemical changes in water. This information can be used to evaluate groundwater age, flow paths, and relative proportions of groundwater, precipitation, and surface water in a groundwater sample.

**1.0 Introduction**

CCR are one of the largest industrial waste streams generated in the United States. In 2012, coal-fired electric utilities in the U.S. and Puerto Rico burned over 800 million tons of coal and generated approximately 110 million tons of CCR (EPA, 2017). Power companies managing CCR impoundments have recently been faced with new challenges. CCR can exhibit elevated levels of regulated elements, and the 2015 Federal “CCR Rule” requires power companies to ensure that these elements are not leaching from disposal areas and causing impacts to groundwater quality. If groundwater monitoring or assessment studies identify statistically significant increases of these regulated elements in groundwater, corrective actions may be required.
Given the significant financial and environmental considerations, it is imperative to accurately identify the sources, delineate contaminant plumes and their migration (flow paths), and predict the behavior or attenuation of contamination in groundwater (fate and transport models). Beginning with a classic paper by Urey (1947), use of isotope variations in natural materials has become commonplace for a variety of Earth and cosmic sciences (e.g., Friedman and O'Neil, 1977). Davidson and Basset (1993) pioneered the use of isotope geochemistry as a tool in CCR-related groundwater investigations. Over the past several decades, isotopic "finger-printing" of dissolved metals such as boron ($\delta^{11}$B), lithium ($\delta^{7}$Li), and strontium ($^{87}$Sr/$^{86}$Sr), or of the water molecules themselves (i.e., $\delta^{2}$H, $\delta^{18}$O or tritium), has proven to be a successful aid in CCR-related groundwater investigations. This approach provides powerful insights into several important topics, including (1) identification of alternate sources of contamination, (2) plume delineation, (3) contaminant migration over time, (4) site assessment, (5) attenuation predications, and (6) seepage rates.

This paper provides an overview of the basic principles of isotope geochemistry and a review of several isotope investigations that have recently proven effective for CCR-related projects (e.g., Davidson and Basset, 1993; Basset et al., 1995; Williams and Hervig, 2004; Buszka et al., 2007; and Harkness et al., 2015).

2.0 Isotope Fundamentals
2.1 Basic Principals

An atom is composed of two primary components: a positively charged central nucleus, which contains most of the mass, and a "cloud" of negatively charged orbiting electrons (Allegre, 2008). In general, the nucleus of any element (i.e., atom) is made up of protons and neutrons, and a given element is defined by the number of protons ($Z$) it contains in its nucleus. Protons and neutrons are approximately equal in mass (neutrons are slightly heavier than protons) but differ in their electrical charges; protons carry a positive charge (+e) while neutrons are electrically neutral.

The mass number ($A$) of a given element is defined by summing the number of protons ($Z$) and neutrons ($N$) such that $A = N + Z$. For a given element on the periodic table, there are atoms with different mass numbers ($A$) that are the result of differences in the number of neutrons present in the nuclei. These nuclides are known as isotopes of an element. The various isotopes of an element are denoted by their different mass numbers. For example, as shown in Figure 1, oxygen has three isotopes, each defined by the number of neutrons. Because an element is defined by the number of protons, all three oxygen isotopes have eight protons but eight, nine, or ten neutrons, giving oxygen isotope mass numbers of 16 (i.e., $^{16}$O = 8 protons + 8 neutrons), 17 (i.e., $^{17}$O = 8 protons + 9 neutrons) and 18 (i.e., $^{18}$O = 8 protons + 10 neutrons).
There are both stable and unstable (radioactive) isotopes that occur naturally. Stable isotopes have nuclei that do not decay to other isotopes but may themselves be produced by the decay of radioactive isotopes (i.e., radiogenic isotopes). Strontium-86 (\(^{86}\text{Sr}\)) is an example of a stable, radiogenic isotope; it is formed as the result of the radioactive decay of rubidium-87 (\(^{87}\text{Rb}\)). Radioactive (unstable) isotopes have nuclei that spontaneously decay over time to form other isotopes. There are more than 300 stable isotopes and over 1,000 known radioactive isotopes. The isotopic composition of all the naturally occurring chemical elements has been well established, both in terms of the number of isotopes and their relative proportions, or natural abundances. For instance, of all the naturally occurring oxygen on the Earth, 99.76\% is \(^{16}\text{O}\), 0.04\% is \(^{17}\text{O}\), and 0.20\% is \(^{18}\text{O}\) (Figure 1).

### 2.2 Delta Notation

The stable isotopic composition of a sample is generally evaluated using the delta (\(\delta\)) notation according to the following equation:

\[
\delta_x(\permil) = \left(\frac{R_{\text{sample}}}{R_{\text{standard}}} - 1\right) \times 1,000; \quad (1)
\]

where \(R_{\text{sample}}\) and \(R_{\text{standard}}\) are the ratio of the heavy isotope to the light isotope (e.g., \(^{11}\text{B}/^{10}\text{B}\)) in the sample and in a standard of known composition, respectively. Stable isotope values are most commonly reported in units of permil (\(\permil\)), which are equivalent to parts per thousand. This form of reporting can be considered roughly analogous to a percentage and is an expression of how enriched (or depleted) the sample is in a given isotope relative to the standard. As an example, if a sample is depleted in \(^{11}\text{B}\) (i.e., the
heavy isotope) relative to the standard, then $R_{\text{sample}}$ is less than $R_{\text{standard}}$ and according to equation 1, the $\delta^{11}\text{B}$ of the sample will be negative. The more depleted in $^{11}\text{B}$, the smaller $R_{\text{sample}}$ becomes relative to the standard and the more negative the $\delta^{11}\text{B}$. If, however, the sample is enriched in $^{11}\text{B}$ relative to the standard, then $R_{\text{sample}}$ will be greater than $R_{\text{standard}}$ and the $\delta^{11}\text{B}$ of the sample will be positive.

It should be noted that not all isotope systems are evaluated using the delta notation; several, including the strontium $87$-$86$ ratio ($^{87}\text{Sr}/^{86}\text{Sr}$), are reported simply as the ratio value. Other isotopes systems that are not considered in this report use the epsilon notation ($\varepsilon$), which captures even smaller scale variations in isotope fractionations.

### 2.3 Solute and Water Isotope Tracers

Environmental isotopes are generally naturally occurring isotopes whose distributions in the hydrosphere can aid in hydrological and geochemical investigations. Their utility derives from the fact that isotopes have distinctive “fingerprints,” and isotopic studies can integrate trace and small-scale variability in these fingerprints to give an effective indication of area-wide processes (e.g., Kendall and Doctor, 2003). Unlike most chemical tracers, environmental isotopes are conservative, meaning the composition is not altered by changing environmental conditions such as redox potential, pH, or sorption processes.

For the lighter elements (i.e., H, Li, B, C, N, O), the mass differences between the stable isotopes of an element are large enough that various physical, chemical, and biological processes can alter (fractionate) the relative proportions of heavy and light isotope(s) in predictable and observable ways; these fractionations allow isotopes to serve as fingerprints and tracers for a variety of investigations. The applications of environmental isotopes as hydrologic tracers in groundwater systems generally fall into two main categories: (1) tracers of the solutes in the water (solute isotope geochemistry) and (2) tracers of the water itself (water isotope hydrology) (Kendall and Doctor, 2003). Table 1 provides a summary of the most relevant environmental isotope tracers in CCR-related investigations.
Solute Isotope Geochemistry

Because solute isotope geochemistry considers the isotopic composition of constituents that have dissolved in groundwater, it is important to understand how the dissolved constituents have acquired their isotopic signature. As water moves through a given matrix (e.g., coal, salt, limestone, sandstone, or shale), pieces of the matrix dissolve into the water and the isotopic signature of the dissolved component is captured and transported within the groundwater. The isotopic composition of the natural geologic matrix was established during the initial deposition of the sediments that ultimately formed the rock unit. The different sedimentary lithologies are each exposed to unique biogeochemical conditions (e.g., redox potential, pH, temperature, salinity, etc.) during sedimentation that resulted in unique isotopic fractionations. These unique fractionations are what give different geologic materials unique isotopic compositions, or fingerprints. Similarly, different anthropogenic materials, such as fertilizer, CCR, road salt, or municipal wastewater, each have a unique isotopic composition that is a function of the materials used to generate them. For instance, the isotopic signature of CCR is a function of the source coal, combustion temperature, and the chemical treatments it may have been subjected to (e.g., Ruhl et al., 2015). When the groundwater flows through or mixes with water sources that have a significantly different isotopic composition, the resulting mixture will have an altered isotopic composition that will reflect the relative contributions from each source.

Boron, lithium, and strontium isotopic fingerprints ($\delta^{11}$B, $\delta^7$Li, and $^{87}$Sr/$^{86}$Sr, respectively) have emerged as the most effective stable solute-isotope tracers in CCR-related investigations (e.g., Harkness et al., 2016, 2018; Ruhl et al., 2003; Buska et al., 2007). While these constituents do not have Federal maximum contaminant levels and are not likely to drive enforceable regulatory actions, they can serve as a proxy capable of

| Table 1: Summary of current environmental isotope tracers in CCR-related investigations |
|-------------------------------------------|-----------------|------------------|
| **Element** | **Notation** | **Unit** |
| Solute Stable Isotopes |  |  |
| Boron | $\delta^{11}$B | permil (‰) |
| Lithium | $\delta^7$Li | permil (‰) |
| Strontium | $^{87}$Sr/$^{86}$Sr | unitless |
| Hydrocarbon Gas (CH$_4$) Stable Isotopes |  |  |
| Carbon | $\delta^{13}$C | permil (‰) |
| Hydrogen | $\delta^2$H (δD) | permil (‰) |
| Stable Water (H$_2$O) Isotopes |  |  |
| Hydrogen | $\delta^2$H (δD) | permil (‰) |
| Oxygen | $\delta^{18}$O | permil (‰) |
| Unstable Water Isotopes |  |  |
| Hydrogen (tritium) | $^3$H | Tritium Units (TU) |
identifying the presence of groundwater from an alternate source.

**Water Isotope Hydrology: δ¹⁸O, δ²H (δD) and ³H (tritium)**

Water isotope hydrology considers the isotopic composition of the water molecules (H₂O) themselves. The δ¹⁸O and δ²H are stable isotopes that provide information regarding the chemical and physical processes that have altered the water composition, and tritium (³H) is a radioactive hydrogen isotope that can give qualitative information regarding the age of the water (greater than or less than ~ 80 years old).

The δ¹⁸O and δ²H of surface waters are generally a function of the isotopic composition of the precipitation. The main factors that control the δ¹⁸O and δ²H of precipitation at a given location are altitude, distance inland, latitude, temperature, and humidity. Figure 2 is a plot of δ²H vs δ¹⁸O and demonstrates that globally, the stable isotope composition of rainwater falls along a linear trend-line known as the Global Meteoric Water Line (GMWL) (Craig, 1961a). Natural surface and subsurface processes such as evaporation, or exchange reactions with minerals, can generate large isotopic fractionations away from the GMWL. Additionally, industrial processes, such as use of cooling water, will also produce a distinctive isotopic signature of the water. These fractionations provide the opportunity to fingerprint water from different sources and develop mixing or models between leachate and fresh water and to quantify seepage rates.

![Figure 2: Plot of δ²H vs δ¹⁸O of water. The main linear trendline depicts the GMWL. Natural waters generally fall along this line as a function of temperature, altitude, humidity, latitude, and distance inland. Various chemical and physical processes will fractionate the water in predicable ways. Shown here is the evaporation trend in lake water (from Kendall et al., 1995).](image-url)
Tritium ($^3$H) is a radiogenic and radioactive isotope of hydrogen that decays to $^3$He (Helium-3) with a half-life of approximately 12.43 years. Tritium content is expressed in tritium units (TU), where 1 TU equals one $^3$H atom in $10^{18}$ atoms of hydrogen. Tritium is produced naturally by cosmic ray spallation and occurs at a background concentration of approximately 2-8 TU (Kendall and Doctor, 2003). However, with the advent of above-ground thermonuclear testing in the 1950s, the levels of tritium in the atmosphere began to rise with maximum atmospheric concentrations recorded at several thousand TUs in 1963. In 1963, the atmospheric test ban treaty was signed, and, as a result, the tritium levels in the atmosphere and precipitation began to decline. Figure 3 provides the atmospheric tritium concentrations as a function of time.

![Tritium concentration in precipitation vs precipitation sample date. This figure depicts the changes in atmospheric tritium concentrations as a function of time. The higher concentrations in the 1950s and 1960s are due to above-ground thermonuclear weapons testing. Current tritium concentrations are approximately 4-8 TU.](image)

Tritium gas in the atmosphere is incorporated into water molecules (i.e., tritiated water) due to equilibrium exchange processes such that increased tritium concentrations in the atmosphere result in increased tritium concentrations in precipitation. Once tritiated water is beneath the water table, it is isolated from the atmosphere and no longer able to equilibrate. At this point, the tritium concentration measured in the water is strictly a function of time and the tritium decay constant. The simplest use of tritium is to
determine whether detectable concentrations are present in the water; waters derived from precipitation that entered the groundwater system before 1953 would have maximum tritium concentrations of approximately 0.05–0.2 TU by 2015. For waters with higher tritium contents, some fraction of the water must have been derived since 1953.

Tritium can also be used more quantitatively by incorporating $^3$He measurements ($^3$H decay product). The $^3$He/$^3$H age-dating method can provide accurate groundwater ages for “young” groundwater according to the following equation:

$$ t = \lambda^{-1} \ln \left( \frac{^3\text{He}}{^3\text{H}} + 1 \right) $$

where $\lambda^{-1}$ is the tritium decay constant, $^3$He* is the measured concentration of $^3$He derived from the decay of tritium, and $^3$H is the measured tritium concentration.

### 3.0 Case Studies

The following sections review several case studies that have used one or more isotope systems to aid in CCR-related groundwater investigations.

#### 3.1 Davidson and Bassett (1993)

In the paper titled *Application of Boron Isotopes for Identifying Contaminants Such As Fly Ash Leachate In Groundwater*, Davidson and Bassett (1993) proposed that the use of boron stable isotopes ($\delta^{11}$B) is a sensitive method for determining the existence and extent of a CCR leachate plume in groundwater. The usefulness of the method is predicated on establishing that the boron isotopic ratio of a CCR leachate is sufficiently unique to be distinguished from the boron isotopic ratio of uncontaminated (background) groundwater.

Figure 4 demonstrates that the $\delta^{11}$B of groundwater can be used to identify CCR leachate inputs even at concentrations as low as 1% leachate by volume. This is significant because impacted groundwater with leachate concentrations as high as 50% by volume can sometimes have boron concentrations that are indistinguishable from non-impacted groundwater. Given that plume boundaries are generally typified by relatively low contaminant concentrations, accurate delineation of the plume extent can be problematic using boron concentrations alone. However, combining the $\delta^{11}$B in the groundwater with boron concentrations can allow for much more precise modeling of the nature and extent of the leachate plume, and can aid in the development of flow and transport models by characterizing flow paths and groundwater flow reversals.
3.2 Williams and Hervig (2004)

To set precedent and context for the use of boron isotopes as organic source-tracers, Williams and Hervig (2004) measured the natural variability of $\delta^{11}$B from 26 different coals from across the U.S. In their paper titled Boron Isotope Composition of Coals: A Potential Tracer of Organic Contaminated Fluids, coal samples (and related CCR) were consistently found to have negative $\delta^{11}$B values (ranging from $-1\%$ to $-70\%$), regardless of the coal rank, type, or thermal maturity. Importantly, these values were distinctive from the positive isotopic composition of typical groundwater (i.e., $\delta^{11}$B groundwater = $+15\%$ to $+35\%$). In fact, the $\delta^{11}$B of coal was significantly more negative than most waters and crustal rocks (Figure 5). For example, meteoric water (e.g., surface freshwater) and domestic wastewater have a $\delta^{11}$B of $+10\%$ to $+40\%$, and $0\%$ to $+10\%$ respectively, and seawater or brines both have a $\delta^{11}$B of $+39\%$. The authors concluded that the isotopically light boron composition of coal makes $\delta^{11}$B a useful organic tracer.
In the United States Geological Survey (USGS) Scientific Investigations Report titled Evaluation of Ground-Water And Boron Sources By Use of Boron Stable-Isotope Ratios, Tritium and Selected Water-Chemistry Constituents Near Beverly Shores Northeastern Indiana, 2004, Busza et al. (USGS, 2007) conducted a study to determine if the source(s) of boron in domestic water wells could be determined using boron stable isotope data. Twenty-four domestic water-supply wells were sampled and classified based on 1) screened interval (i.e., the uppermost, surficial aquifer (SA) or the confined basal sand aquifer) and 2) the proximity of the well to a potential source of boron. Putative boron sources in the study area consisted of CCR leachate, domestic wastewater, and naturally occurring sources. Fifteen of the 24 samples were classified into four representative groups based on the above criteria. These classifications included 1) SA-water potentially affected by CCR leachate, 2) SA-water potentially affected by domestic wastewater, 3) SA-water with no identified anthropogenic boron inputs and, 4) basal sand aquifer groundwater that contains naturally occurring sources of boron. The nine remaining samples were collected from wells that lacked sufficient information to pre-determine the groundwater source.

Figure 6 provides a summary of the boron concentration and isotope data from the Busza et al. (2007) study, as well as results from additional publications. Results of their analyses demonstrated that the highest boron concentrations (15,700 to 24,400 μg/L) and isotopically lightest δ¹¹B values (+0.1 to +6.6‰) were present in the four wells completed in the SA located proximal to a CCR landfill (Table 1). Conversely, boron

Figure 5: Published ranges of δ¹¹B from crustal rocks, minerals and waters. The δ¹¹B for coal and kerogen is a distinctively more negative (i.e., depleted in the heavy isotope (¹¹B) relative to the light isotope (¹⁰B)). Not shown here is the range of values for domestic wastewater which has a positive δ¹¹B with an average of +20‰ (Eppich et al., 2012) (from Williams and Hervig, 2004).
concentrations in the five samples from the deeper, basal sand aquifer ranged from 656 to 1,800 µg/L and had the isotopically heaviest δ^{11}B values that ranged from +24.6 to +34.0‰. Intermediate isotopic values were measured in the SA domestic-wastewater affected wells (+8.7 to +11.7‰). SA samples with no anthropogenic boron sources had significantly lower boron concentrations than all other representative sample groups, but the δ^{11}B was similar to that of the basal sand aquifer. By characterizing the boron composition in the representative samples (Table 1), the authors were then able to determine the source of boron in the unclassified well samples. The majority of these samples contained naturally-sourced boron; however, two contained boron that was likely sourced from wastewater-affected SA water and one contained boron likely sourced from CCR-affected SA water.

<table>
<thead>
<tr>
<th>Potential boron source</th>
<th>Range of δ^{11}B, this investigation (per mil)</th>
<th>Ranges of δ^{11}B, published values (per mil)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal-combustion product affected water</td>
<td>0.1 to 6.6, four samples from four wells in the surficial aquifer</td>
<td>Laboratory-prepared leachate samples, by material</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-17 to -16, two fly-ash samples, bituminous coal (1)</td>
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<td></td>
<td></td>
<td>15.8, fly ash, sub-bituminous coal, front-fire furnace combustion, one sample (2)</td>
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<td></td>
<td></td>
<td>-19.2, fly ash, bituminous coal, front-fired furnace combustion, one sample (2)</td>
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<td></td>
<td></td>
<td>-4.1, fly ash, bituminous coal, cyclone-furnace combustion, one sample (2)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-7.9, fly ash, lignite, tangential-furnace combustion, one sample (2)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-13, flue-gas desulfurization, one sample (1)</td>
</tr>
<tr>
<td>Domestic-wastewater-affected surficial-aquifer water</td>
<td>8.7 to 11.7, four samples from three wells in the surficial aquifer</td>
<td>6.0 to 10.6, treated wastewater, four samples, Texas (3)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>7.6 to 12.9, raw sewage, three samples, Israel (4)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5.3 to 11.0, treated sewage, three samples, Israel (4)</td>
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<td></td>
<td></td>
<td>-2.7, treatment-plant effluent, one sample, Nevada (5)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.2, treatment-plant effluent, one sample, Arizona (6)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-0.2 and 0.7 per mil, two samples suspected to be affected by septic waste, Nebraska (7)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-5.7, wash water from a domestic washing machine, one sample, Nevada (5)</td>
</tr>
</tbody>
</table>

Boron (δ^{11}B) isotope data were reported in per mil or the deviation of the \( {^{11}B}/^{12}B \) ratio of the sample from that of a standard material, using the following formula:

\[
\delta^{11}B = \left(\frac{\left({^{11}B}/^{12}B\right)_{\text{sample}} - \left({^{11}B}/^{12}B\right)_{\text{standard}}}{\left({^{11}B}/^{12}B\right)_{\text{standard}}}\right) \times 1000, \text{ where the standard is a boric acid sample, NIST-951}
\]


**Figure 6**: Ranges of boron isotope ratios in samples from CCR-impacted water and domestic wastewater from this study compared to select published data from other studies (Modified from Buszka et al., 2007).

Tritium (³H) age-dating was also applied in this study to provide an independent check on the interpretations derived from the use of the δ^{11}B. As previously mentioned, the radioactive nature of tritium allows it to be used to age-date water. Water from the SA-aquifers in this study was expected to be young and contain tritium concentrations consistent with modern-day values. In contrast, the water in the deeper basal sand aquifer was expected to be older and therefore contain no or very low concentrations of tritium. Results of the tritium analyses confirmed that waters from the surficial aquifer were young and therefore the isotopically light δ^{11}B is consistent with recent
anthropogenic inputs from CCR or wastewater. In addition, the basal sand aquifer was “tritium-dead” and indicated an older groundwater (> ~80 years), which is consistent with dissolution of natural boron sources with an isotopically heavy δ¹¹B. The authors concluded that boron isotope ratios and tritium concentrations could be used to distinguish naturally-occurring boron from boron leaching from CCR.

4.0 Summary

Isotopes have been used for decades for a variety of Earth and cosmic sciences. The utility of isotopes is derived largely from their conservative nature and their well-defined and predictable behavior in a variety of settings. Isotope geochemistry has provided powerful insights in numerous groundwater investigations. In CCR-related investigations, isotope geochemistry can aid in meeting a variety of objectives including identifying alternate sources of contamination, developing mixing models, refining plume delineation, defining flow paths, and quantifying seepage rates. Environmental isotope tracers are generally categorized into two groups: (1) Solute isotopes which consider the isotopic composition of dissolved constituents and (2) water hydrology isotopes which consider the isotopic composition of the water molecules themselves. Recent solute isotope systems that have proven successful in CCR-related investigations include boron (δ¹¹B), strontium (⁸⁷Sr/⁸⁶Sr) and lithium (δ⁷Li). As the need to differentiate alternate sources of groundwater contamination continues to grow, additional isotope systems will likely emerge in CCR-related groundwater investigations.

REFERENCES


