



## Temporal characterization of flowback and produced water quality from a hydraulically fractured oil and gas well



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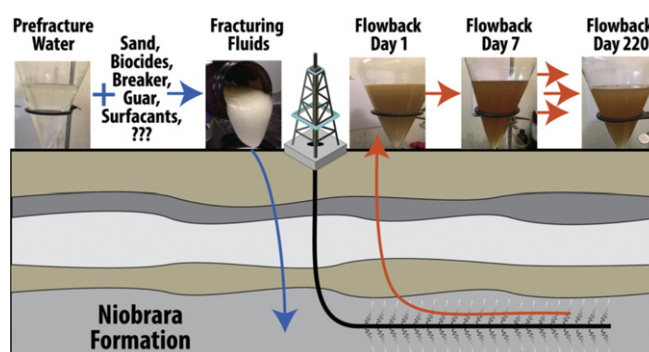
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### HIGHLIGHTS

- Comparison of ground water, fracturing fluid, flowback, and produced water over time
- TDS, metals, and anion data minimally changed over time in this DJ-Basin well.
- COD declined overtime and only ~30% of the volume injected returned over the study.
- Changing water quality impacts upon treatment and management were described.

### GRAPHICAL ABSTRACT



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### ABSTRACT

This study examined water quality, naturally-occurring radioactive materials (NORM), major ions, trace metals, and well flow data for water used and produced from start-up to operation of an oil and gas producing hydraulically-fractured well (horizontal) in the Denver-Julesburg (DJ) Basin in northeastern Colorado. Analysis was conducted on the groundwater used to make the fracturing fluid, the fracturing fluid itself, and nine flowback/produced water samples over 220 days of operation. The chemical oxygen demand of the wastewater produced during operation decreased from 8200 to 2500 mg/L, while the total dissolved solids (TDS) increased in this same period from 14,200 to roughly 19,000 mg/L. NORM, trace metals, and major ion levels were generally correlated with TDS, and were lower than other shale basins (e.g. Marcellus and Bakken). Although at lower levels, the salinity and its origin appear to be the result of a similar mechanism to that of other shale basins when comparing Cl/Br, Na/Br, and Mg/Br ratios. Volumes of returned wastewater were low, with only 3% of the volume injected (11 million liters) returning as flowback by day 15 and 30% returning by day 220. Low levels of TDS indicate a potentially treatment-amenable wastewater, but low volumes of flowback could limit onsite reuse in the DJ Basin. These results offer insight into the temporal water quality changes in the days and months following flowback, along with considerations and implications for water reuse in future hydraulic fracturing or for environmental discharge.

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## 1. Introduction

The use of unconventional drilling technologies (hydraulic fracturing and horizontal drilling) has rapidly increased over the past 15 years. This is especially the case in Colorado, where the number of active wells (both vertical and horizontal) increased from 22,228 to 53,228 between 2000 and 2015 (COGCC, 2015). The majority of the unconventional drilling in Colorado currently takes place within the Wattenberg Field, which has around 24,500 active wells in 2017 (COGCC, 2017). The Wattenberg Field is the most productive area of the Denver-Julesburg Basin (DJ Basin), and contains several formations, including the Niobrara Formation and the Codell Sandstone Member. The benefit this intense activity has had on the U.S. economy and the region is tempered by its potential impact on the local environment (Adgate et al., 2014) and, more pressingly, on regional water quantity and quality (Goodwin et al., 2014; Vidic et al., 2013). Concerns about water quantity arise from the significant volumes of water used in the fracturing fluids during hydraulic fracturing, which range from 7 to 33 million liters per well (Kondash and Vengosh, 2015; Malakoff, 2014). Following the hydraulic fracturing process, the well is flowed back, returning large volumes of water to the surface along with the oil and gas. This process generates a stream of wastewater throughout the life of the well that could negatively impact the regional water quality in the event of a spill or release (Torres et al., 2016; Vidic et al., 2013).

Over 95% of this wastewater in the US is disposed in Class II wells via deep well injection (Clark and Veil, 2009; McCurdy, 2011). While this method is considered to be the most economic and best practice for these wastewaters, it has recently been linked to seismic activity and potential environmental contamination (Ellsworth, 2013; Kassotis et al., 2016). These environmental impacts, coupled with the location of many shale basins in semiarid regions, magnifies the importance of understanding these wastewaters (e.g. water quality, origins of salinity, to volumes returned) and increasing their treatment and reuse.

Oil and gas wastewaters generated by hydraulically-fractured wells are commonly called flowback and produced water. Flowback is typically defined by the oil and gas industry as the point when the well is flowed back to allow the flow of oil, gas, and water to the surface. Once this mixture is directed towards separators for on-site production facilities (i.e., oil/gas/water separators), the resulting water is termed produced water. Flowback and produced waters consist of a variety of constituents that are either present within the fracturing fluid used during the fracturing process, or contributed from the formation itself (Barbot et al., 2013; Gregory et al., 2011; Thurman et al., 2017). Additives used within fracturing fluids vary by operator and formation, but typically consist of surfactants, biocides, friction reducers, gelling agents, and gel breakers, each of which serves a role during the hydraulic fracturing process (Rogers et al., 2015; Stringfellow et al., 2014; Thurman et al., 2014). These components, especially the gel-based fracturing fluids, increase the organic chemical load of the returning wastewaters. This organic chemical load has been shown to decrease with time (Esmaeilirad et al., 2015) as the returning wastewater begins to resemble formation water (i.e., produced water).

Formation-based constituents consist of total dissolved solids (TDS), hydrocarbons, naturally occurring radioactive material (NORM), metals, and other elements (Alley et al., 2011; Zhang et al., 2014). These parameters are known to increase to levels present in the native formation water, particularly TDS. Since TDS is generally considered the greatest challenge in the treatment and reuse of produced water, understanding the rate at which mixing of the formation and injected waters occurs, along with insights into the origin of the TDS (i.e., salinity), is important when considering reuse strategies. Furthermore, most research on NORM has focused on environmental monitoring, wastewater treatment, and radioanalytical chemistry of uranium (U), radium (Ra), and Ra decay products arising from flowback and produced waters, particularly from the Marcellus Shale (Nelson et al., 2015b, 2014; USGS, 2011; Zhang et al., 2014). However, very little is known about the concentration of

NORM in flowback and produced waters in other formations of Devonian age, such as the Niobrara formation of the DJ Basin in northeastern Colorado.

A more detailed view of the water quality characteristics of returning wastewaters and their volumes over time is needed to guide treatment and re-use efforts, and catalog indicator constituents in the event of a spill or release. Accordingly, the objectives of this study were to examine a single well in the Denver-Julesburg Basin over >7 months to (1) determine basic water quality, elemental composition, the origin and nature of salinity, and NORM concentrations of waters used and produced, (2) measure the volume of the returning flowback and produced waters over time, and (3) suggest reuse strategies and implications for the oil and gas wastewaters based on the findings.

## 2. Methods

### 2.1. Site and sampling description

Water samples were collected from a single horizontally-fractured well on a multi-well pad in the Wattenberg Field (Weld County, CO). The oil- and gas-producing well (wet) had 28 stages (~1800 m) targeting the Niobrara formation, and occurred at a depth of 2100 m. During the hydraulic fracturing process, a water sample was collected from an onsite storage tank prior to the generation of a gel-based fracturing fluid. The water sample originated from shallow groundwater from a nearby well. In addition to the groundwater, a sample of gelled fracturing fluid was also collected. The gelled fracturing fluid was broken using concentrated tert-butyl peroxide at 10%, and was heated for 1 h at 110 °C to return this complex mixture (semi-solid) to a liquid state that could be investigated using standard wastewater analysis techniques. This method was selected since 10% tert-butyl peroxide was used in this well as the breaker, and since the operator noted that high-temperatures were required for breaking. This resulted in several preliminary experiments at 10% tert-butyl peroxide under different temperatures, with 110 °C being chosen, since it yielded a completely dissolved mixture.

Flowback of the well was initiated after hydraulic fracturing and a one-month shut-in following the wells completion (shut-in: well closed after hydraulic fracturing to allow for other wells onsite to be fractured or to finish the construction of production facilities prior to flowing back a well). The first water sample was collected on the first day of flowback (D1), while the second water sample was collected on the fourth day of flowback (D4). After day four, the well was again shut in to set up for onsite production (oil/water/gas separator), which lasted three days. Following this period, another water sample was collected on the seventh day (D7) of flowback (not counting the three-day shut in). Water samples were also taken on the 15th (D15), 22nd (D22), 55th (D55), 80th (D80), 130th (D130), and 220th (D220) day following the flowing back of the well. D1 and D4 were taken from a flowback tank (emptied every 4–6 h) for our individual well, whereas D7 to D220 were collected from an oil/water/gas separator (sampling port) that isolated the individual well from the other hydraulically fractured wells on-site. After collection in burned glass amber bottles (1 L, headspace free), the water samples were immediately transported to the lab (~2 h after collection) and stored at 4 °C for up to 5 days prior to analysis. The well's water production was collected from the separators onsite meter (i.e., daily water production), while the total volume used for fracturing fluid was obtained from the well's FracFocus report (FracFocus, 2016) and was verified by the operator.

### 2.2. General water quality, inorganic ions, and metals analysis

Total dissolved solids (TDS), total suspended solids (TSS), and volatile suspended solids (VSS) were determined by Standard Methods (Eaton and Franson, 2005). The pH was determined using a ThermoSci Orion meter (Thermo Scientific). Turbidity was measured with a

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