

The U-tube Sampling Methodology and Real-Time Analysis of Geofluids

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ABSTRACT: The U-tube geochemical sampling methodology, an extension of the porous cup technique proposed by Wood [1973], provides minimally contaminated aliquots of multiphase fluids from deep reservoirs and allows for accurate determination of dissolved gas composition. The initial deployment of the U-tube during the Frio Brine Pilot CO₂ storage experiment, Liberty County, Texas, obtained representative samples of brine and supercritical CO₂ from a depth of 1.5 km. A quadrupole mass spectrometer provided real-time analysis of dissolved gas composition. Since the initial demonstration, the U-tube has been deployed for (1) sampling of fluids down gradient of the proposed Yucca Mountain High-Level Waste Repository, Argus Valley, Nevada (2) acquiring fluid samples beneath permafrost in Nunavut Territory, Canada, and (3) at a CO₂ storage demonstration project within a depleted gas reservoir, Otway Basin, Victoria, Australia. The addition of in-line high-pressure pH and EC sensors allows for continuous monitoring of fluid during sample collection. Difficulties have arisen during U-tube sampling, such as blockage of sample lines from naturally occurring waxes or from freezing conditions; however, workarounds such as solvent flushing or heating have been used to address these problems. The U-tube methodology has proven to be robust, and with careful consideration of the constraints and limitations, can provide high quality geochemical samples.

KEYWORDS: *geochemical sampling, carbon sequestration, borehole sampling, fluid sampling*

INTRODUCTION

Obtaining uncontaminated fluid samples from deep geologic aquifers is challenging because of the elevated pressures and temperatures encountered. Entering wellbores that are under pressure and which may contain explosive or sour gas is a complex task that requires the deployment of well-head pressure control equipment along with cranes or workover rigs to utilise wireline conveyed samplers. The technology to maintain fluid sample integrity during recovery has been developed primarily within the oilfield and

geothermal service industries, with examples of commercially available instruments being: Schlumberger's MDT fluid sampler; Halliburton's Armada Sampling System; and Kuster Corporation's flow through sampler. All three of these sampling devices are mechanically complex and need to be conveyed into and out of a wellbore to facilitate each sample recovery.

The U-tube was developed to simplify the recovery of fluids from deep boreholes and allow flexibility for post-sampling analysis [Freifeld *et al.*, 2005][Freifeld &

Trautz, 2006]. In particular, the ability to repeatedly collect large volume multiphase samples into high pressure cylinders facilitates both real-time field analysis as well as acquisition of sample splits for future laboratory based analysis.

U-TUBE OPERATION

The U-tube is permanently or semi-permanently deployed in a wellbore to provide the ability to periodically (or continuously) acquire samples. The U-tube is extremely simple, consisting of a loop of tubing, which has a tee at its base, terminating at a check valve that permits fluid to enter the loop of tubing (Figure 1). A filter at the inlet removes particulates that can interfere with the operation of the check valve. At the surface, the U-tube drive leg is connected to a supply of high pressure N₂ that closes the check valve and forces the sample that collects within the loop of tubing up the sample leg to the surface.

In its most basic form the surface sampling equipment can consist of an open container at which the sample leg terminates. However, for higher purity sampling or accurate dissolved gas chemistry analysis, the sample is directed into N₂ purged and/or evacuated high pressure sample cylinders. After a sample has been acquired, by venting both the sample and drive legs to atmospheric pressure, the check valve is allowed to open and fresh sample enters the U-tube sampling loop.

U-TUBE DEPLOYMENT

To date, we have deployed the U-tube using several distinct modalities depending on the wellbore completion requirements. As part of Nye County's Nuclear Waste Repository Early Warning groundwater monitoring program, a borehole in the Armagosa Valley, Nevada, USA, was completed with four U-tubes within two sand packs. At depths of 265 m and 350 m, two U-tubes were located within each sandpack and isolated using a bentonite backfill.

At the High Lake Site in Nunuvut Territory, Canada, a 7.5 cm diameter mineral exploration boring was completed with a U-tube to sample sub-permafrost brines [Freifeld *et al.*, 2008]. Small gage (1/4") stainless steel tubing served as the drive and sample legs of the U-tube as well as functioned as the strength member for lowering the U-tube and pneumatic packer to a depth of ~400 m. To prevent freezing within the sampling lines a heat trace providing 20 W/m was run along the entire length of the borehole completion.



Figure 1. Schematic layout of the downhole equipment for deploying a U-tube geochemical sampler

As part of geologic CO₂ sequestration demonstration projects conducted at the Frio Site, Liberty County, TX, USA, and

the Otway Project, Nirranda South, Victoria, Australia, U-tubes were deployed for sampling multi-phase mixtures of gas, water and supercritical CO₂. At both these sites, CO₂ was injected in the subsurface to understand the subsurface movement of the CO₂ plume and geochemical sampling was one of many technologies employed to understand the fate of the CO₂.

SAMPLE ACQUISITION AND ANALYSIS

The method for U-tube sample retrieval is determined by the end use of the collected fluid. For the relatively shallow fluid collection at Nye County's Amargosa Valley Site and at the High Lake Site, samples were driven to the surface using compressed N₂ gas supplied in standard 'G' size (~8000 l) industrial cylinders. At both the Amargosa and High Lake site, samples were collected at 1 bar and there was no attempt to maintain sterile sampling conditions.

For the CO₂ sequestration programs at the Frio Field Site and the Otway Project, samples were collected in high pressure, large volume (13 l) cylinders for surface processing. To maintain sample integrity the pressure cylinders at the Frio Site were flushed with N₂ and then evacuated using a rotary vane pump prior to filling to formation pressure with fluid from the U-tubes.

Because of difficulty in keeping vacuum pumps operating when they are exposed to significant volumes of moisture laden gas, it was decided that after flushing the sample cylinders with N₂, they would be allowed to bleed down to 1 bar pressure but not evacuated. The residual 13 l of N₂ in the sample cylinders acted as an internal standard, enabling an accurate estimation of dissolved gas concentration in the sample fluid [Freifeld & Trautz, 2006].

FRIO FIELD SITE

At the Frio Field Site, because of the need for real-time feedback to guide operational decision making and the

ephemeral nature of some of the monitored parameters, significant effort was given toward field analysis. To measure the pH before degassing, a high pressure pH electrode (Model TB567, ABB Inc. Reno, Nevada, USA) was installed on the high pressure sample line. In addition, benchtop measurements were acquired of electrical conductance, pH and alkalinity soon after sample collection [Kharaka et al, 2006].

A field deployable quadrupole mass spectrometer (Omnistar, Pfeiffer Vacuum GmbH, Asslar, Germany) was used to detect the arrival of the CO₂ plume in addition to determine CH₄, O₂, Ar and N₂ concentrations. Various tracers including Kr, SF₆ and perfluorocarbons were periodically co-injected with CO₂ to aid in determining rates of subsurface transport and changes in CO₂ saturation (Freifeld *et al.*, 2005). The gas tracers can be determined to a precision of ±100 ppb or better using the mass spectrometer, although frequent calibration is required to obtain this level of accuracy.

OTWAY PROJECT

The Otway Project is using a multilevel completion with three U-tubes to sample above and below the gas-water contact of a depleted gas reservoir, while CO₂ is injected in a well located 300 meters down dip. The uppermost U-tube was installed just below the mudstone cap rock and is sampling predominantly supercritical CH₄, while the lower two U-tubes initially produced water, but transitioned to predominantly CO₂ and CH₄ as the gas-water contact was pushed down by the increasing volumes of CO₂.

Early in the sampling program it was observed that there was a decrease in gas flow from the sample leg for the U-tube accessing the residual gas column. This flow restriction was found to be caused by a progressive build-up of wax within the ¼" sample line at the near surface. The wax composition was dominated by a homologous series of *n*-alkanes, maximizing at *n*-C₂₇, with a melting point of ~41°C; a temperature that would cause

it to solidify during surface sampling. Following a solubility study of different commercially available solvents, a product sold by ExxonMobil under the trade name Solvesso-100, composed primarily of C₉₋₁₀ dialkyl and trialkylbenzenes, provided the best solution for periodic flushing of wax that collects within the U-tube sampling lines.

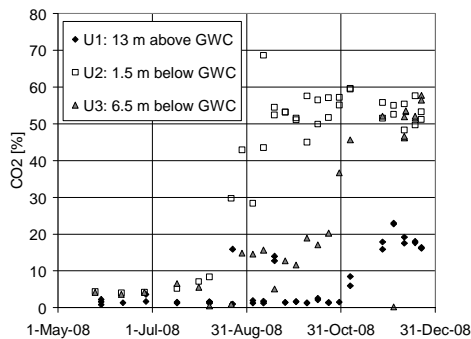


Figure 2. Molar fraction of CO₂ in gas samples collected in U-tubes at the Otway Project.

Figure 2 shows the molar fraction of CO₂ in the samples for all three U-tubes. Initial small increases in CO₂ appear in July for U-tube 2 (3 ½ months after the start of CO₂ injection), and August for U-tube 3. After the initial appearance as a dissolved phase, the CO₂ molar fraction rapidly increases as a free-phase gas component arrives. U-tube 2 became self lifting in September and no further water samples were obtained. Similarly for U-tube 3 the transition to gas occurred at the very end of December 2008, with the samples collected in 2009 containing only small amounts of liquid.

CONCLUSIONS

The U-tube geochemical sampling system has been demonstrated to operate in varied environmental conditions, from direct burial in sand/bentonite backfill to deep oil and gas reservoirs. As with all borehole sampling methods, careful attention needs to be paid to the type of fluid being sampled, the purpose of the sampling and the interaction of the borehole with the surrounding formation.

By addressing potential sampling difficulties and carefully controlling wellbore conditions, U-tubes can provide high purity samples for real-time and laboratory analysis.

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