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Evaluation of Fluorocarbon Tracer Retention in Dry and Wet Sand Column Tests

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Keywords

Tracers, hydrofluorocarbons, perfluorocarbons, freons

ABSTRACT

Fluorocarbon compounds were evaluated as geothermal tracers in sand column tests using damp, dry, and untreated 'washed sea sand'. Fluorocarbons evaluated in these tests included two hydrofluorocarbon freons, trifluoromethane (R23), 1,1,1,2-tetrafluoroethane (R134a), and four perfluorocarbons: perfluorodimethylcyclobutane, perfluoromethylcyclopentane, perfluoromethylcyclohexane, and perfluorodimethylcyclohexane. The retention of the fluorocarbon compounds was explored in flowing helium at 23°C. The effect of sand moisture content on fluorocarbon retention was examined. Fluorocarbon retention was strongly effected by sand moisture content. The fluorocarbon compounds flowed with the bulk fluid when the sand was saturated with water, but were significantly retained by the solid phase when the sand was thoroughly dried. The data suggests that the fluorocarbons may or may not be conservative at geothermal conditions and the compounds may not be suitable as tracers in reservoirs with a high degree of superheat.

Introduction

Geothermal tracers are used to provide information on flow paths between injection and production wells as well and to optimize efficiency of operation of geothermal fields. Tracers can be used to provide information on the flow of both liquid and vapor phases. Currently, tracer tests are the only method available to determine vapor and liquid mass flows in geothermal applications. Tracer test data was used by Rose, *et al.*, (1999) to estimate reservoir size and recharge rates and by Shook (1998) to calculate reservoir volume.

Tracer compounds must meet several criteria to be useful in field applications. The tracer compounds must not be present at appreciable levels in the field under study. They should flow

with the bulk fluid being monitored, be stable at reservoir temperatures, be detectable at low concentrations and have minimal environmental impact. Further, tracers must be economical at the quantities required. A more thorough discussion of tracer selection was recently provided by Hirtz, *et al.*, (2001).

Although a number of tracer compounds have been explored for geothermal applications, many fail to meet all of the desired selection criteria. For example, chlorofluorocarbons, which were used in The Geysers from 1900 to 1997 (Adams, *et al.*, 1991; Beall, *et al.*, 1994) have been phased out of production due to their negative environmental impact (Adams, *et al.*, 2001).

Stable isotopes, such as deuterium, tritium, and ¹⁸O, can be prohibitively expensive. Propane, helium and bromide may have background levels that are too high for practical use in certain locations (Hirtz, *et al.*, 2001), and alcohols can partition into both the vapor and liquid phases and are not detectable at low concentration levels. Consequently, new tracers, which meet all of the selection criteria, are needed.

Fluorocarbons have many properties that suggest they could be suitable for use as geothermal tracers. Fluorocarbons are environmentally acceptable, thermally stable, detectable at low concentration levels, and have insignificant background levels. Hydrofluorocarbons and perfluorocarbons (PFCs) have been explored as tracers for a wide variety of applications including motoring air pollutant transport (Pitchford, *et al.*, 2000), leak detection (Hassoun, *et al.*, 2000), indoor air quality study (Demokritou, *et al.*, 2002), ocean transport study (Ledwell and Bratkovich, 1995) and medical applications (Lee, *et al.*, 2001). In subsurface environments, fluorocarbon tracers have been used in microbiological characterization in subsurface basalts and sediments (Colwell, *et al.*, 1992), the characterization of natural gas reservoirs (Dugstad, *et al.*, 1993; Ljosland, *et al.*, 1993) and as partitioning tracers to estimated NAPL contamination (Deeds, *et al.*, 1999a; Deeds, *et al.*, 1999b; Mariner, *et al.*, 1999; and Deeds, *et al.*, 2000).

Only one study has reported the use of fluorocarbons for geothermal applications (Adams, *et al.*, 2001). Adams, *et al.*, (2001) explored two hydrofluorocarbon freons, R-134a and R-23, as vapor-phase tracers. The freons were found to be ther-

mally stable and provided a qualitative description of the distribution of injected water in a vapor-dominated system with low to moderate superheat.

This paper presents a preliminary analysis of the transport of fluorocarbon compounds in the gaseous phase. Fluorocarbons examined include two hydrofluorocarbon freons (trifluoromethane [R-23] and 1,1,1,2-tetrafluoroethane [R-134a]), and four perfluorocarbons (perfluorodimethylcyclobutane [DMCB], perfluoromethylcyclopentane [MCP], perfluoromethylcyclohexane [MCH], and perfluorodimethylcyclohexane [DMCH]). This study characterizes the retention of the specified compounds on a fixed bed of washed sea sand with helium as the mobile phase at 23°C. The effect of sand moisture content is examined. This work found that fluorocarbon retention is strongly effected by sand moisture content. The fluorocarbon compounds flow with the bulk fluid when the sand is saturated with water, but are significantly retained by the solid phase when the sand is thoroughly dried.

Method

Several fluorocarbon compounds were evaluated as tracer candidates by passing them through a packed column of washed sea sand. The mobilities of the fluorocarbon tracers through the sand column were compared to argon, a known conservative tracer. Flowing helium at 23°C was employed as the mobile phase.

Washed sea sand, obtained from Fischer Scientific, was sieved to remove grains larger than #20 and smaller than #120 US standard testing sieves. The sieved sand was used either: 1) without additional treatment, 2) dried overnight at 100°C or 3) saturated with nano-pure water. Ultra high purity helium, obtained from US Welding, was used as the carrier gas. The two hydrofluorocarbon tracer candidates were R-23, obtained from Advanced Specialty Gases, and R-134a, obtained from Praxair. Of the 4 perfluorocarbons evaluated as tracers, DMCB and DMCH were obtained from F2 Chemical Ltd. of England, while MCP and MCH were obtained from Lancaster Chemicals. All fluorocarbons were used as received. Ultra high purity argon, obtained from US Welding, was employed as a known conservative tracer. Physical properties of the tracers are provided in Table 1.

Table 1. Physical Properties of Candidate Tracer Compounds.

| Compound | Formula | M.W. | B.P. (°C) | Te (°C) |
|--------------------|--|------|-----------|--------------------|
| Argon ¹ | Ar | 40 | -186 | -122.3 |
| R23 ¹ | CF ₃ H | 70 | -82 | 26.2 |
| R134a ² | C ₂ F ₄ H ₂ | 102 | -26 | 101.1 ³ |
| DMCB ⁴ | C ₆ F ₁₂ | 300 | 45 | 171.7 |
| MCP ⁴ | C ₆ F ₁₂ | 300 | 48 | 185 |
| MCH ⁴ | C ₇ F ₁₄ | 350 | 76 | 212.8 |
| DMCH ⁴ | C ₈ F ₁₆ | 400 | 102 | 241 |

Sources: (1) Reid, *et. al.*, (1987); (2) CRC Handbook of Chemistry and Physics (3rd Electronic Edition); (3) McLinden, *et. al.*, (1998); (4) PCR, Inc. Manufacturer's catalog (1994-1995).

A schematic of the experimental apparatus is shown in Figure 1. The system consists of a mass-flow controller, an HPLC injection valve, a water jacketed sand column and a gas chromatograph (GC). The GC was equipped with two on-line sample injectors and two detectors. Sand column dimensions were 1 cm I.D. by 120 cm long and held approximately 150 g of washed sea sand. The temperature of the sand column was maintained by recirculating water from a constant temperature water bath through the column jacket. Ultra high purity helium was used as the mobile phase for the sand column system and was maintained at a constant flow rate of 1 cc/min by the mass-flow controller.

The jacketed column, available from Kontes Glass Co., was filled with sand using the following procedure. The column end fittings were initially installed on the bottom of to the column. A 1.0-cm diameter fiberglass filter (Whatman, GF/D style) was placed inside the column against the end fitting. The column was secured in the vertical position to a rod attached to the frame of a vortex mixer. The vibration of the mixer was used to settle the sand. The sand was placed in a 125 ml separatory funnel with a teflon valve. The small valve orifice of the separatory funnel provided constant slow flow of sand during column filling. The column was vibrated during the entire filling process, which took approximately 30 minutes. The column was vibrated an additional five minutes after loading. The vibration ensured that the column was evenly packed. A 1.0 cm diameter fiberglass filter was placed on the top of the sand column and a flow adapter end fitting was inserted into the column and positioned snugly against the filter. The packed column was maintained in a vertical orientation throughout tracer testing.

Three types of treatment were applied to the sand prior to loading into the column. As a base case untreated sand was loaded directly into the column after sieving. Dry sand was prepared by sieving the sand, drying in a furnace overnight at 100°C and loading into the column as outlined above. The sand lost less than 0.1 wt.% on drying. Damp sand was prepared by sieving, loading into the column, as outlined above, and slowly wetting the sand by pumping water into the top of the column at 1 cc/min. The column was drained, and helium was flowed over the sand, top down, at 1 cc/min overnight to remove any free water droplets.

The column void volume was determined by weighing the dry sand packed column, filling it with water, weighing the water flooded sand column and calculating the difference between the wet and dry column weights. Precautions were taken to eliminate air pockets or gas bubbles in the flooded sand column. After initially obtaining the total weight of the column, sand, end fittings and the first section of associated tubing, the column was evacuated. The column was back filled with carbon dioxide, evacuated a second time and refilled with carbon dioxide. Water was drawn into the column as the carbon dioxide was evacuated. The gas free flooded column assembly was then weighed. The weight of the water added to the column was used to determine the column void volume. Carbon dioxide was used in this process to displace small quantities of residual gas from the void space in the sand. Upon flooding with water, the CO₂ is solubilized in the water leaving the voids free of bubbles of residual gas and CO₂.

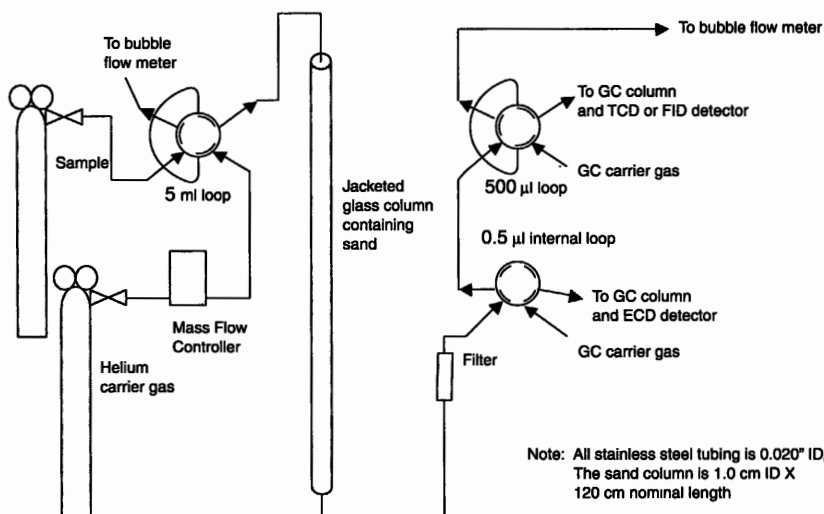


Figure 1. Schematic of Sand Column System.

Three series of gas phase tracers were used. Mix #1 included R23, DMCB and MCH in helium. Mix #2 included R134a, MCP, and DMCH in helium. The third series was argon. The concentrations of the perfluorocarbons ranged from 2780 – 3450 ppmv in the mixtures. The concentration of the freons ranged from 5120 – 8530 ppmv. Argon was injected as a pure gas. The high argon concentration was required due to low detector response. Tracers were verified to be in the gas phase by confirming the gas phase concentration by GC analysis.

Tracer retention times were evaluated by passing one of the three mixtures through the sand column in flowing helium. The column, valves and fittings were checked for leaks and the GC calibration checked against calibration standards prior to each run. The temperature of the sand column was then set using the recirculating water bath. The flow rate of the helium carrier gas was initiated at 1.0 cc/min. and allowed to equilibrate. The tracer mixture was injected into the helium flow using the HPLC valve equipped with a 5 ml sample loop. The gas was directed to the inlet of the vertically oriented column with top down flow. Effluent from the sand column was sampled on-line through the automated GC valve system. GC samples were obtained at either 4.0 or 4.3 minutes, depending on GC analysis time requirements.

Analysis of the sand column effluent concentrations were performed on-line using a Hewlett Packard 6890 gas chromatograph equipped with two sample valves, two capillary columns and two detectors. Detectors included a thermal conductivity detector (TCD), a flame ionization detector (FID), and an electron capture detector (ECD). The two sample valves were equipped with 0.5 µl and 500 µl sample loops. Argon analysis was performed using a 500 µl sample separated on a 0.32mm x 25 m Chrompack molsieve capillary column using the TCD. The GC was operated with a split ratio of 5:1 and a column flow rate of 5.0 cc/min. Freon and perfluorocarbon analysis was performed using a 500 µl sample separated on a 0.53mm x 60 m Supelco SPB-1 poly(dimethylsiloxane) capillary column,

quantified with the FID detector. The GC inlet was operated with a split ratio of 5:1 and a column flow rate of 10.0 cc/min. The ECD was available to analyze the PFCs but was not used since the FID was able to detect the PFCs and the freons. All analyses were run isothermally at 40°C for 4.0 minutes except those involving DMCH which took 4.3 minutes to elute.

The degree of tracer interaction with the stationary sand matrix was determined by calculating the first temporal moment of volume of the tracer and comparing it to the first temporal moment of volume of a known conservative tracer, argon.

The first temporal moment of volume of the tracer (i) is given by Dwarakanath, et. al., (1999):

$$\bar{V}_i = \frac{\int_0^{\infty} C_i V dV}{\int_0^{\infty} C_i dV} - 0.5V_s$$

Where \bar{V}_i is the first temporal moment of volume of tracer (i), C_i the concentration of tracer (i), V is the volume of carrier gas flowing into the system since the injection of the tracer, and V_s the volume of the tracer sample injected. This equation is only valid for constant flow systems with plug type (square wave) injection of the tracer. Since the carrier gas flow rate through the sand is 1.0 cc/min the first temporal moment can be expressed as either volume (cc) or time (minutes). Calculated first moments are referred to as the retention times of each compound. At constant flow rate, single phase conditions, the first temporal moment of a conservative tracer is equal to the pore volume.

The retardation factor for a given tracer (2) relative to a conservative tracer (1) is given by:

$$R = \frac{\bar{V}_2}{\bar{V}_1}$$

An ideal tracer would have a retardation factor of 1.0 indicating that it flows with the mobile phase with no interaction with the stationary matrix. In reality most compounds have some affinity for the stationary phase. Due to experimental error, system error, etc, a tracer is considered to be conservative if it has a retardation factor of no more than 1.20 (Whitley, 1997).

Results and Discussion

Three sets of tracer retention time analyses were performed using the freons and perfluorocarbons. The tracers were evaluated using the untreated sand, the damp sand and the dry sand. The experimental results demonstrate a strong influence of sand moisture content on tracer retention.

The untreated sand was found to contain 0.06 wt. % water. The elution of tracers from the column packed with untreated sand at ambient (23°C) temperature is shown in Figures 2 and 3. Effluent concentrations are shown as a fraction of the con-

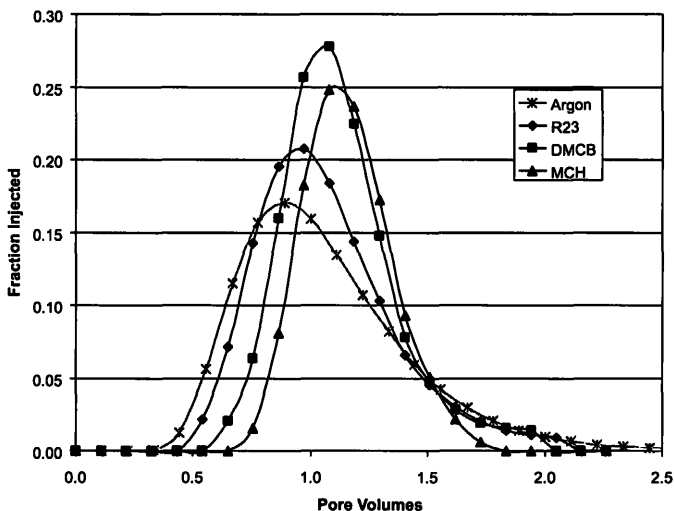


Figure 2. Elution of Argon and FC Mix #1 (R23, DMCB, MCH) Through Untreated Sand.

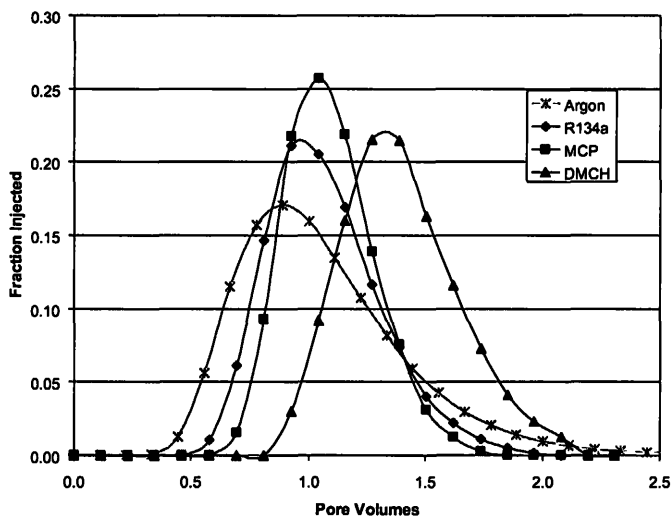


Figure 3. Elution of Argon and FC Mix #2 (R134a, MCP, DMCH) Through Untreated Sand.

centration injected. Average retention times, retardation factors – compared to Argon, and mass balances are provided in Table 2. The sand column residence time was approximately 36 minutes. The average retention time for argon, as determined by first moment analysis, was approximately 40 minutes. The average retention time for argon was in good agreement with the column pore volume, suggesting that the argon transports with the bulk fluid and is not significantly retained on the solid matrix. Mass balance closure for argon was calculated to be within 5% demonstrating that argon was recovered and not retained in the sand column.

Both freons had very similar retentions to argon, deviating by only 1%. In contrast, perfluorocarbon retention deviated somewhat from argon, resulting in retardation factors ranging from 1.02 to 1.34. Both freons and three PFCs (DMCB, MCP, and MCH) were found to be conservative with retardation fac-

Table 2. Fluorocarbon Tracer Transport Characteristics through Untreated Sand.

| Compound | First Moment (V) (cc or minutes) | Ave. R_f | (% dev) | Mass Bal. (%) |
|----------|----------------------------------|------------|---------|---------------|
| Argon | 39.8 | 1.00 | | 105 |
| R23 | 40.1 | 1.01 | (1.2) | 108 |
| R134a | 40.2 | 1.01 | (1.0) | 98 |
| DMCB | 42.0 | 1.05 | (0.6) | 114 |
| MCP | 41.2 | 1.02 | (1.5) | 99 |
| MCH | 43.6 | 1.09 | (0.6) | 95 |
| DMCH | 53.4 | 1.34 | (2.6) | 102 |

tors ranging from 1.01 to 1.09. The highest molecular weight PFC, DMCH, was retarded by 34% showing that it does not represent the flow of the bulk fluid and would not be a conservative tracer. DMCH is of special interest since it has a normal boiling point very close to that of water.

In general retention increases with the compounds' molecular weight or boiling point. Although the molecular weight of DMCB and MCP are the same, the different retention time for the two compounds suggest that compound structure also plays a role in retention.

All mass balances were reasonable, ranging from 95-114%. A mass balance of 100% is not expected because the value is calculated from the sum of concentrations over time and is limited by sample frequency (limited by GC sample run time) and analytical accuracy. Despite these limitations, the mass balance data demonstrates that all tracer compounds were recovered.

The damp sand contained 9.4 g water, corresponding to 6.4% by weight of dry sand, decreasing the void volume by 23%. The elution of tracers from the column packed with the damp sand at ambient (23°C) temperature is shown in Figures 4 and 5. Effluent concentrations are shown as a fraction of the concentration injected. Average retention times, retardation factors (compared to an assumed argon retention time), and mass balances are provided in Table 3. In the damp sand argon was not run due to

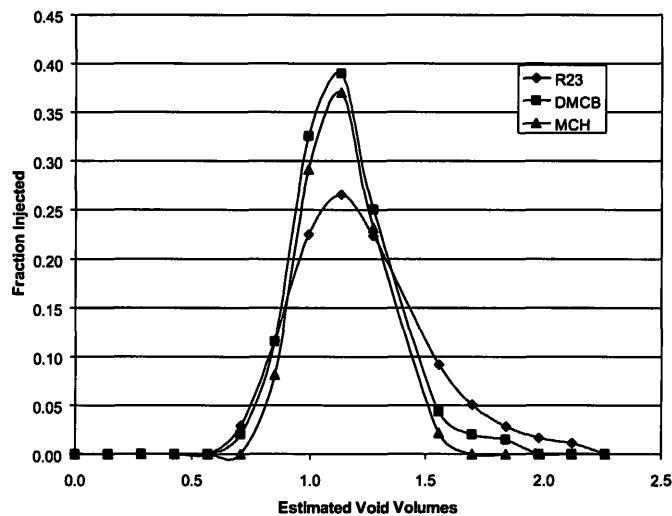


Figure 4. Elution of Fluorocarbon Mix #1 (R23, DMCB, MCH) Through Wet Sand.

analytical difficulties. Relative retention times are compared to an assumed first temporal moment of argon. The assumed first moment was calculated by subtracting the water volume used to saturate the sand from the argon first moment measured in the untreated sand experiments. Tracer recovery was good, with mass balances ranging from 93 to 110 wt.%.

Retardation factors were all within the conservative range, ranging from 1.05 to 1.17. In contrast to the results obtained with the untreated sand, the lower molecular weight freons had higher retardation factors than the PFCs. Freon retardation factors ranged from 1.14 to 1.17, while the PFC retardation factors were clustered about an average value of 1.07.

The damp sand was fully hydrated and had a film of water on the surface of the sand. From the larger retardation factor and the slight tailing of the peak, it is evident that the freons have slightly more affinity for the water film than do the PFC's.

The results using dry sand were expected to be very similar to those using untreated sand. The only difference between the two sands was that the dry sand was heated overnight at 100°C which caused the untreated sand to lose less than 0.1 wt.% through drying. Following drying, none of the compounds were conservative. The best performing freon tracer, R23, had a retardation factor of 2, while R134a had an average retardation factor greater than 5. The two lower molecular weight PFCs, DMCB and MCP, had average retardation factors greater than 10. The two higher molecular weight PFCs, MCH and DMCH, did not elute even after 25 pore volumes of carrier gas were passed through the column. The mild heat treatment caused a very large change in the tracer / sand surface affinity. These results are summarized in Table 4.

In order to verify the results obtained on the dry sand, a follow up experiment was performed. The column was repacked with untreated sand and the column heated to 60°C via the water jacket. This allowed the sand to slowly dry during the course of the experiments. The first injection of the tracer mixture

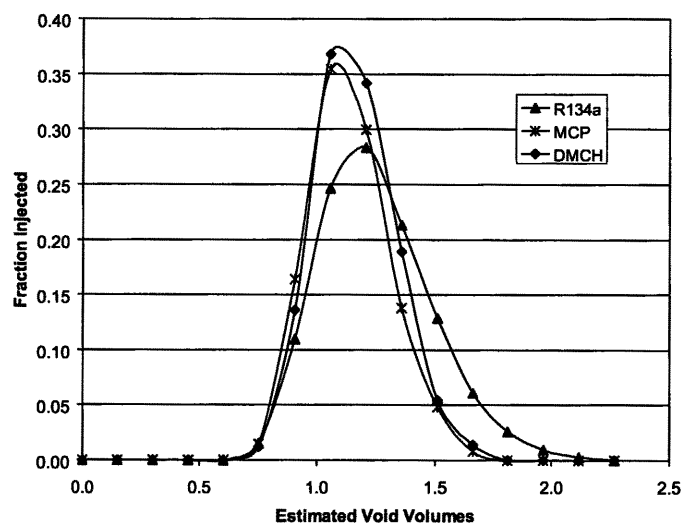


Figure 5. Elution of Fluorocarbon Mix #2 (R134a, MCP, DMCH) Through Wet Sand.

Table 3. Fluorocarbon Tracer Transport Characteristics through Sand after Flooding with Water and Draining.

| Compound | First Moment (V) (cc or minutes) | Ave. R_f | (% dev) | Mass Bal.(%) |
|----------|---|------------|---------|--------------|
| R23 | 34.8 | 1.14 | (0.2) | 104 |
| R134a | 35.9 | 1.17 | (2.1) | 97 |
| DMCB | 32.5 | 1.06 | (0.7) | 110 |
| MCP | 32.6 | 1.07 | (2.1) | 93 |
| MCH | 32.1 | 1.05 | (0.0) | 95 |
| DMCH | 33.2 | 1.09 | (1.7) | 97 |

Table 4. Retardation of Fluorocarbon Tracers in Dried Sand.

| Compound | First Moment (V) (cc or minutes) | Ave. R_f |
|----------|---|------------|
| R23 | 78 | 2 |
| R134a | 400 | > 5 |
| DMCB | 1800 | >10 |
| MCP | 1400 | >10 |
| MCH | Did Not Elute | >30 |
| DMCH | Did Not Elute | >30 |

occurred after the column had equilibrated for approximately one hour. The retardation of all of the tracers increased with each succeeding injection that occurred 2-3 hours apart. By the third injection most of the tracer compounds were retarded past the point of being conservative. An example of this behavior is given in Figure 6 for MCP. These experiments qualitatively demonstrate that the retardation of the tracers increases as the sand dries, supporting the results obtained with the dried sand experiments discussed above. This has significant implications for the utility of these tracers in superheated regions and requires further investigation.

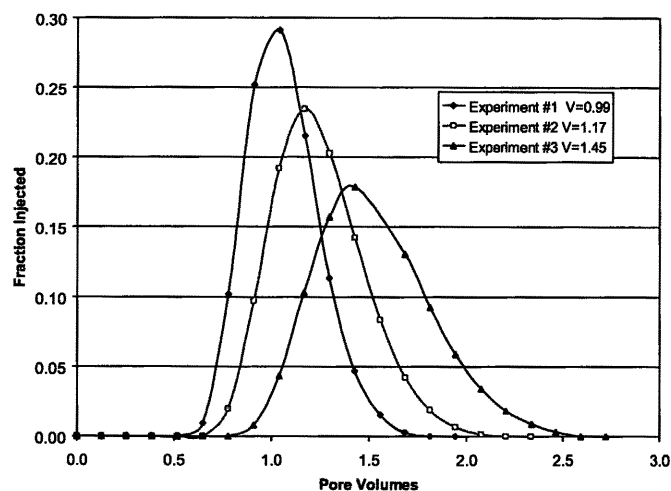


Figure 6. The Effect of Drying on the Elution of MCP as the Untreated Sand Dries at 60°C.

Conclusions

The experimental results demonstrate that sand moisture content has a strong influence on tracer retention. Untreated sand has only a minimal retarding effect on all of the fluorocarbons except DMCH. DMCH was slowed enough to make it non-conservative, and hence, not useful as a tracer under this condition. All tracers tested were conservative, including DMCH, through damp sand. The PFCs had less interaction with the damp sand than did the freons. Dry sand caused significant retardation for all fluorocarbons tested.

The data suggests that fluorocarbons may or may not be conservative at geothermal conditions. The area of the reservoir where the behavior of the tracer is suspect is after boiling, in the superheated steam region. As the steam becomes superheated, the rock becomes dry and the tracers could transition to non-conservative behavior. Further research needs to be performed at geothermal conditions with a focus on the moisture content of the solid matrix.

Acknowledgments

This work was supported by the U.S. Department of Energy, Office of Geothermal and Wind Technologies, under contract number DE-AC07-99ID13727. The authors gratefully acknowledge Michael Shook and Joel Renner of the Idaho National Engineering and Environmental Laboratory, Michael Adams of the University of Utah and Bruce Rouse of the University of Texas for their assistance and helpful discussions.

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