Permeability, Geochemical, and Water Quality Tests in Support of an Aquifer Thermal Energy Storage Site in Minnesota

April 1985

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PERMEABILITY, GEOCHEMICAL, AND WATER
QUALITY TESTS IN SUPPORT OF AN
AQUIFER THERMAL ENERGY STORAGE SITE
IN MINNESOTA

S. C. Blair
W. J. Deutsch
P. J. Mitchell

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Pacific Northwest Laboratory
Richland, Washington 99352

FOREWORD

Seasonal thermal energy storage (STES) involves storing thermal energy, such as winter chill, summer heat, and industrial waste heat, for future use in heating and cooling buildings or for industrial processes. Widespread development and implementation of STES would significantly reduce the need to generate primary energy in the U.S. In fact, 1980 data indicate that STES is suitable for providing 5 to 10% of the nation's energy with major contributions in the commercial, industrial, and residential sectors.

Aquifer thermal energy storage (ATES) is predicted to be the most cost-effective technology for seasonal storage of low-grade thermal energy. Approximately 60% of the U.S. is underlain with aquifers potentially suitable for underground energy storage. Under sponsorship of the U.S. Department of Energy, Pacific Northwest Laboratory (operated by Battelle Memorial Institute) has managed numerical modeling, laboratory studies, evaluation of environmental and institutional issues, and field testing of ATES at several sites.

This report describes the Underground Energy Storage Program's efforts to characterize physicochemical processes at DOE's ATES Field Test Facility (FTF) located on the University of Minnesota campus at St. Paul, Minnesota. Experimental efforts include: field tests at the St. Paul FTF to characterize fluid injectability and to evaluate the effectiveness of fluid-conditioning equipment, geochemical studies to investigate chemical reactions resulting from alterations to the aquifer's thermal regime, and laboratory tests on sandstone core from the site. Each experimental area is discussed and results obtained thus far are reported.

Landis D. Kannberg, Manager
Underground Energy Storage Program

SUMMARY

This report describes the Underground Energy Storage Program's efforts to characterize physicochemical processes at DOE's ATES FTF located on the University of Minnesota campus at St. Paul, Minnesota. Experimental efforts include: field tests at the St. Paul FTF to characterize fluid injectability and to evaluate the effectiveness of fluid-conditioning equipment, geochemical studies to investigate chemical reactions resulting from alterations to the aquifer's thermal regime, and laboratory tests on sandstone core from the site. Each experimental area is discussed and results obtained thus far are reported.

Field studies conducted at the St. Paul site included membrane filter tests to anticipate well impairment by particle plugging. These tests monitored suspended solids content and other injectability parameters of the thermal fluids. Onsite core flooding tests were also conducted to aid in determining response of the aquifer formation to the injected fluid. Results of membrane filter tests indicate that fluids injected during heat injection for four short-term storage cycles had very low suspended solids. Further, heat recovery fluids reinjected during the first two cycles had significant suspended solids content, while fluids reinjected during the heat recovery phase of later cycles had very low suspended solids. Metal filings also collected on filters provided evidence of bearing failure in downhole pumps. Overall, the filter test results indicate that, at temperatures to 100°C, well impairment due to suspended solids is not a problem. Results of core flooding tests demonstrate that several thousand pore volumes of fluid in the 50 to 90°C temperature range can be passed through representative core samples with no significant loss in permeability. However, examination of one core sample after flow of approximately 25,000 pore volumes showed that a substantial portion of the cementing agents in the sandstone was dissolved.

Geochemical studies included development of a computerized chemical model of an ATES system and use of the model to determine the effect of

heating the ground water at the St. Paul FTF on solution chemistry. In addition, the proposed filtration technique for removing precipitated carbonate minerals from solution was evaluated. It was found that the aquifer water is in equilibrium with the mineral calcite. Heating the solution will precipitate this mineral, and given the large volumes of water which will be heated, may cause plugging of the plant equipment and/or the aquifer. Gravel filters downstream from the heat exchanger will remove particulates from solution and enhance precipitation on the disposable gravel. The rate of precipitation of the calcite will be a function of the surface area of the material in the filters. Calculations based on empirical rate laws of calcite precipitation showed that the particles in the filter should be no larger than 0.6 mm in diameter (coarse sand size) to enable these filters to remove enough calcium and carbonate from solution to allow the water to be reinjected into the aquifer without causing potentially large changes in permeability.

Laboratory studies of permeability at elevated pressure and temperature were conducted on core samples from the Franconia-Ironton-Galesville (FIG) aquifer. The purpose of these studies was to provide baseline data on dominant physicochemical processes active in aquifers used for ATEs. These laboratory tests were performed in a state-of-the-art rock mechanics apparatus constructed specifically for this project and is described in this report.

Laboratory tests produced two results that are significant to ATEs. The first is that on the time scale of these tests (days), substantial changes in petrofabric were observed in the samples. These changes include loss of cementing agents, an increase in intergranular porosity, and loss of mechanical strength. Available results on changes in pore fluid chemistry indicate increased silica content after residence of pore fluid in the core for a few hours (overnight) at elevated temperature. This result is important to ATEs as it shows that the time scale of chemical reactions occurring in aquifers used for ATEs is on the order a few days or less and possibly a few hours. This result also indicates that cementing agents are removed first, dispersing fine particles and increasing porosity.

A second result is that initial heating of samples caused significant reduction in permeability in all laboratory tests and the amount of reduction was proportional to the rise in temperature. This is important to heat injection operations because it indicates that permeability reduction may occur in the receiving formation when the temperature of the injection stream is increased.

These results then indicate that the fabric of the FIG aquifer rocks that form the host aquifer for the St. Paul FTF is probably being changed by operation of the ATEs facility. However, it is apparent from the success of field operations to date that for short-term tests, these changes in the rock fabric do not impact the hydrologic performance of the aquifer formation. Field data from longer storage tests, along with downhole logs, will be necessary to confirm that cement dissolution is occurring and to assess the impact on site performance.

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PERMEABILITY, GEOCHEMICAL, AND WATER QUALITY TESTS IN SUPPORT OF AN AQUIFER THERMAL ENERGY STORAGE SITE IN MINNESOTA

1.0 INTRODUCTION

Aquifer thermal energy storage (ATES) is a method of subsurface heat storage that could be used in regions with suitable aquifer conditions to provide or supplement district heating systems. The U.S. Department of Energy (DOE) is currently sponsoring research into the ATES concept as part of the Underground Energy Storage (UES) Program at the Pacific Northwest Laboratory (PNL). One study area involves determinations of physicochemical processes occurring in storage aquifers that may affect ATES systems. Such processes could include clay dispersion, mineral dissolution and precipitation, and mobilization of internal fines as a result of thermal cycling and/or increased flow velocity. These processes are important to ATES because they can impair operation of injection/withdrawal wells, cause loss of permeability in the aquifer formation, and create problems in operation of surface equipment.

Since late 1981, PNL researchers have been conducting experiments to characterize physicochemical processes at the DOE's ATES Field Test Facility (FTF) located on the University of Minnesota campus at St. Paul, Minnesota. These experiments include 1) field tests at the St. Paul FTF to characterize fluid injectability and to evaluate the effectiveness of fluid-conditioning equipment, 2) geochemical studies to investigate chemical reactions resulting from alterations to the aquifer's thermal regime and 3) laboratory tests on sandstone core from the site. This report describes these studies and presents the results obtained.

2.0 BACKGROUND

The basic concept of ATEs is the storage of large amounts of hot or cold water in subsurface aquifer formations by heating or cooling portions of the formation. Successful development and operation of an ATEs facility requires cyclic heating/cooling of an aquifer as well as changes in flow rate and chemistry of the ground water. Experience in the oil and geothermal industries has shown that subsurface formations can be sensitive to these changes and the resulting problems can be significant. Such problems include well plugging, loss of reservoir stability, and various environmental effects. Previous experience has also shown that many of these problems can be anticipated and/or avoided if physicochemical studies are conducted before or in conjunction with fluid injection/withdrawal activities. Data obtained through physicochemical studies are used to characterize the interrelated changes in physical and geochemical properties of an aquifer system.

The work reported herein was conducted under the Laboratory/Field Studies Task of the UES Program. This task sought to identify and investigate important physicochemical processes at DOE's ATEs site in St. Paul, Minnesota, by developing and applying appropriate field, geochemical, and laboratory capabilities.

The St. Paul ATEs site is on the campus of the University of Minnesota-St. Paul. The system utilizes a doublet well configuration to inject and recover heated water from the subsurface. An array of monitoring wells surrounds the two pumping wells. All wells are completed in the Franconia-Ironton-Galesville (FIG) confined aquifer. The top of this aquifer is at a depth of approximately 180 m, and it is 63 m thick; it has an artesian head of 125 m and consists of horizontal, evenly stratified, fine- to coarse-grained, friable to compact sandstone with silty, clayey, and glauconitic zones and weak dolomitic cementation with numerous shale interbeds (Walton, 1981).

The system, depicted in Figure 2.1, is designed to use one well of the doublet as the supply/disposal or "cold" well and the other well as the heat storage or "hot" well. During heat storage, water is pumped from the cold well, heated, passed through a fixed-bed reactor to reduce the oversaturation of calcium carbonate, and injected into the hot well. During heat recovery, water is pumped from the hot well, heat is extracted from the water, and the water is reinjected into the cold well. The hot well design temperature is 150°C; temperatures up to 80°C are expected in the cold well. Pumping rates are approximately 19 ℓ /s with a pressure of approximately 0.83 MPa at the injection well head and 1.2 MPa at the top of the storage formation. Hydraulic properties of the FIG aquifer and results of preliminary heat storage testing are described by Walton (1981) and Miller (1983).

As of December 1983, four short-term heat storage cycles had been performed at the St. Paul site. These are summarized in Table 2.1. The heat injection portion of the first test was terminated due to well impairment by calcium carbonate precipitation after 50 hours of injection. Heat recovery portions of the first and second storage tests were each delayed by pump bearing failures caused by heated fluids. The third and fourth test cycles were completed without incident.

2.1 FIELD STUDIES

The field studies have involved characterizing suspended solids in injection/withdrawal fluids to assess potential impairment mechanisms at the St. Paul FTF. This is important, as injection well plugging often occurs shortly after injection is initiated. An example is the injection well impairment experienced at the Stony Brook, New York, ATEs site. A laboratory study in support of the Stony Brook site demonstrated that formation fines were highly susceptible to mobilization by relatively low pumping or injection fluid stream velocities (Owen and Peterson 1982). Corrosion products from the carbon steel completion liner may also have contributed to the well failure. Another example of injection well plugging is the clogging problem encountered during injection of 55°C water into the aquifer of

FIGURE 2.1 St. Paul Aquifer Thermal Energy Storage System

TABLE 2.1. Key Parameters for the Short-Term Test Cycles at the University of Minnesota ATEs Field Test Facility

	Flow Rate (ℓ/s)	Mean Temperature ($^{\circ}C$)	Volume Pumped (10^3 m^3)	Duration (Days)	Energy Recovery Factor ^(a)
Cycle 1					
Injection	18.4	89.4	8.3	5.2	
Storage				13	0.59
Recovery	18.1	59.3	8.1	5.2/6	
Cycle 2					
Injection	17.6	97.4	12.2	8 ^(b)	
Storage				90	0.46
Recovery	17.8	55.2	12.3	8	
Cycle 3					
Injection	18.3	106.1	12.2	7.7 ^(b)	
Storage				9.7	0.62
Recovery	17.8	81.1	11.8	7.7	
Cycle 4					
Injection	17.9	114.8	11.9	7.7 ^(b)	
Storage				10.1	0.58
Recovery	17.8	89.1	11.9	7.7	

(a) Thermal energy recovered above supply well temperatures divided by heat added during injection.

(b) Net injection time (i.e., periods of maintenance not included).

a thermal energy storage experiment performed by Auburn University near Mobile, Alabama (Molz, Warman and Jones 1978). In this case, the well impairment was apparently due to plugging of connective porosity by dispersed clay particles (Molz et al. 1979). These particles were derived from the aquifer formation. Laboratory tests with unheated water showed that the reaction was more likely the result of pore velocity increase rather than the increased temperature of the injected ground water.

In general, injection impairment can arise from either or both of two phenomena: 1) particulate plugging and 2) geochemical incompatibility. To diagnose injection problems in both areas, it is important to have detailed knowledge of the injection formation and the composition and suspended solids content of the fluid. Field studies have concentrated on characterizing suspended solids in injection/withdrawal fluids at the St. Paul FTF. These efforts are complemented by geochemical studies. To aid in determining response of the aquifer formation to injected fluid, onsite core flooding tests were conducted on core samples from the FIG aquifer and on samples of Massillon sandstone.

2.2 GEOCHEMICAL STUDIES

Potential geochemical effects that may influence ATEs system operation include mineral precipitation and dissolution, corrosion, clay dispersion, oxidation of reduced minerals, and ion exchange between aquifer minerals and the injected fluid. Mineral precipitation may result from the heating and cooling of ground water. The mineral mass formed may reduce the efficiency of the heat exchanger, plug pipes throughout the system, and impair well performance. Also, by using water that is incompatible with the chemical state of the clay minerals in the aquifer, clay minerals may disperse or swell, blocking the pore spaces. Oxidation, ion exchange, and mineral dissolution in response to water/rock interactions induced by fluid injection may lead to degradation of the ground-water quality.

Our capability to perform computer simulations of these chemical effects on the natural and engineered system has enabled detailed study of calcite precipitation from the injection fluid at the St. Paul FTF. In particular, the geochemical modeling codes MINTEQ and EQ3NR/EQ6 have been installed on PNL computers and are available to simulate various water/rock interactions. These codes determine the aqueous speciation of an analyzed water sample and calculate the relative saturation of minerals and other solids with respect to the solution composition. In addition, the codes can simulate the dissolution and precipitation of minerals and generate a

resultant water composition and mass of solid destroyed or produced. MINTEQ was used to simulate the effect on water composition of heating ground water at the St. Paul FTF. Modeling results confirm field evidence that calcium carbonate would precipitate extensively from solution.

2.3 LABORATORY STUDIES

Laboratory studies were conducted to provide baseline data on dominant physicochemical processes active in aquifers used for ATEs. To conduct these studies, a laboratory rock mechanics facility was developed. A state-of-the-art rock mechanics apparatus was specially designed to provide data on permeability, pore fluid chemistry, and rock material properties at pressures, temperatures, and flow rates equivalent to those imposed on a storage aquifer by an ATEs facility.

Physicochemical tests utilizing the laboratory apparatus were initiated in May 1982. Since then, several tests on core samples from the FIG aquifer have been completed. These tests measured permeability as a function of temperature and monitored geochemistry of pore fluid passed through core samples at various temperatures. The test results provide baseline data on the response of aquifer rock when invaded by water that is not in thermal or chemical equilibrium with the formation.

3.0 FIELD STUDIES

This section documents the field studies conducted at the St. Paul FTF to characterize the suspended solids in injection/withdrawal fluids.

3.1 INSTRUMENTATION AND METHODOLOGY

A portable Field Injectivity Test Stand (FITS) shown in Figure 3.1 was used to characterize suspended solids and injectability parameters of fluid streams. The FITS was used to divert a portion of the fluid stream through a reservoir analog such as a membrane filter or a core sample and was configured to enable sampling at several locations in the ATES facility. The FITS provides the capability to measure permeability of a reservoir analog under conditions of constant differential pressure or constant flow rate. Also, the reservoir analog can be maintained at process stream temperature during flow tests. All components of the FITS are constructed of Inconel or 316 stainless steel to reduce the possibility of chemical or physical contamination due to degradation of the apparatus by heated fluids. The apparatus is shown schematically in Figure 3.2; sampling points for heat injection and recovery fluids are shown in Figure 3.3.

Filters of various pore sizes were employed during testing of the process fluids. Total suspended solids in the fluid stream were determined from flow through either 0.4- μm or 0.45- μm membrane filters. The pore size of the injection formation was estimated to be 12 μm using Champlin's methods (1976); 10- μm filters were used along with core samples to simulate the receiving formation. Filtration data from the filters were used with methods presented by Barkman and Davidson (1972) to estimate injection well performance. Following Barkman and Davidson, characteristics of the injection well and formation were used to calculate water quality ratios, W/K_c where W equals suspended solids content and K_c equals permeability of the filter cake on 0.45- μm filters. It was determined that, for the injection half-life to be approximately 1 year, the water quality ratio had to be ≤ 0.25 . Filtration data were plotted as cumulative filter throughput

FIGURE 3.1. Field Injectivity Test Stand Flow System Panel and Control Panel

FIGURE 3.2. Field Injectivity Test Stand Schematic

FIGURE 3.3. Field Injectivity Test Stand Location Within St. Paul ATEs System

(volume) versus time from start of test, after the methods of Barkman and Davidson. All filtration and core flooding tests were conducted at the temperature of the injection stream and with differential pressure held constant. Differential pressure for most filters was held at 0.14 MPa.

Core samples of both the Franconia-Ironton-Galesville (FIG) aquifer and Massillon sandstone were also used as reservoir analogs. Permeability (K) of the core samples was calculated using Darcy's equation:

$$K = \frac{Q\mu l}{A\Delta P} \quad (3.1)$$

where Q = flow rate (ml/s)

μ = viscosity (centipoise)

l = sample length (cm)

A = cross-sectional area of sample (cm²)

ΔP = differential pressure (atm).

Several filter samples and one core sample were examined using standard scanning electron microscopy (SEM) techniques. Specific procedures for testing using the FITS are presented in Appendix A.

3.2 MEMBRANE FILTER TESTS

Representative data for flow of ambient temperature ground water pumped from the FIG aquifer (Well B) through a 10- μ m membrane filter are plotted in Figure 3.4. These data indicate the high quality of water present in the aquifer and that water quality improved as the well was developed. Suspended solids, determined from filter tests using 0.45- μ m filters, ranged from 0.01 to 0.13 ppm. Examination of filters using SEM indicated particles to be calcium-based minerals, sand grains, and some colloidal iron particles. Values of W/K_c calculated from 0.45- μ m filters for this water were in the range 0.126 to 0.013, indicating an injection half-life of more than 2 years.

Membrane filter tests were also used to characterize the heated ground-water injection fluid passed through the precipitator. Cumulative flow data

FIGURE 3.4. Injectability Test Data for Ambient Ground Water Pumped from Well B

for 10- μ m membrane filter tests, which sampled these injection fluids at the wellhead, are plotted in Figure 3.5. These data indicate that the overall injectability of this fluid is quite good. However, two distinct ranges of water quality are observed. Comparison of times of the filter testing with the operations log showed that these two ranges of water quality can be attributed to "age" of the precipitating agent; i.e., fresh precipitating agent produces higher water quality than does spent agent.

Water quality ratios (W/K_c) for these fluids ranged from 0.1 for fluids passed through fresh precipitating agent to 10 for fluids from spent agent. These water quality ratios indicate injection half-lives of from 0.025 to 2.5 years, respectively. However, most of the water injected has a water quality ratio of approximately 0.1, which indicates that the longer half-life estimate is more accurate. Concentrations of suspended solids in precipitator effluent were determined to be in the 0.2- to 0.7-ppm range

FIGURE 3.5. Cumulative Throughput Versus $\sqrt{\text{Time}}$ for 10- μm Filter Tests of Fluids During Heat Injection

(Table 3.1) and consisted of mostly colloidal iron and calcium-based minerals including aragonite. Numerous metallic filings were also collected on the filters. These were traced to malfunctioning pump bearings. Qualitative size determination from SEM indicates that colloidal iron and calcium-based mineral particles ranged in size from less than 1 μm to 100 μm . Typical suspended solids filtered from precipitator effluent with a 0.45- μm filter are shown in Figure 3.6, which was prepared from SEM photomicrographs.

Membrane filter tests were also conducted to monitor fluids during the heat recovery/reinjection phases of four heat storage cycles. Figure 3.7 is a summary plot of cumulative volume versus time data for 10- μm filters used to characterize heat recovery fluids. Suspended solids are summarized in Table 3.1. Water quality ratios (W/K_c) calculated for recovery/reinjection fluids ranged from 0.26 to 5, indicating an injection half-life of 0.05 to 1 year for reinjection into the cold well. However, as indicated in Figure

TABLE 3.1. Summary of Suspended Solids Data

Water Type	Suspended Solids (0.4 μm) ppm	Particle Size, μm	Filtration Properties
In situ ground water	0.01 - 0.13	< 5	Excellent
Heat injection	0.2 - 0.7	10 - 100	Very good
Heat recovery			
Cycle 1, 2	0.2 - 0.9	10 - 100	Good
Cycle 3, 4		< 10	Very good

FIGURE 3.6. Typical Suspended Solids Filtered from Precipitator Effluent During Heat Injection

3.7 and Table 3.1, the highest suspended solids content occurred in fluids pumped during cycles 1 and 2 (recovery/reinjection). These solids comprised mostly colloidal iron and lenticular aragonite particles along with metal filings. The filings were again indicative of malfunctioning bearings. Colloidal iron and calcium particles ranged in size from 20 to 100 μm . Typical particles filtered from heat recovery fluids during cycles 1 and 3 are shown in Figure 3.8.

The results shown in Figures 3.7 and 3.8 indicate that fluids reinjected during the recovery/reinjection phases of cycles 3 and 4 had substantially lower particulate content. This finding is further borne out by the summary data in Table 3.1. These results indicate that, to date, fluids injected during heat injection and recovery phases of ATEs operations at St. Paul have low suspended solids and favorable injectability characteristics.

FIGURE 3.7. Cumulative Throughput Through 10- μm Filters Versus $\sqrt{\text{Time}}$ for Fluids Reinjected During Heat Recovery

FIGURE 3.8. Typical Suspended Solids Filtered From Heat Recovery Fluids During Cycles 1 (a) and 3 (b)

3.3 CORE FLOODING TESTS

Three core samples from the injection formation and two samples of Massillon sandstone were tested in the FITS during heat injection. The Massillon samples (M-1, M-2) were tested using water sampled directly from the heat exchanger. As evidenced by the data in Figure 3.9, these cores show no significant loss of permeability after a few thousand pore volumes were passed through. The three samples (A-742, A-752, and A-761) from the injection formation (FIG aquifer) were tested during heat injection cycles after the precipitator was added to the system. Two of these cores showed no loss of permeability after passing several thousand pore volumes.

FIGURE 3.9. Permeability of Core Samples Plotted Versus Number of Pore Volumes for Core Flood Tests Conducted During Heat Injection

However, data for sample A-761 show a slight increase in permeability after passing approximately 2,500 pore volumes; permeability then decreased substantially after 15,000 pore volumes. This decrease was caused by formation of an iron-rich filter cake on the core surface.

Photomicrographs of sample A-761 showing microstructure before and after the core flooding test are presented in Figure 3.10. Interpretation of these photomicrographs indicates that flow of heated ground water causes much of the cement to be dissolved from the rock. This result is consistent with results of laboratory permeability tests at elevated pressure and temperature, which also indicate cement dissolution from St. Paul sandstones

FIGURE 3.10. Results of Core Flooding Tests Showing Surface Filtration and Cement Dissolution

(Blair and Deutsch 1983; Blair, Deutsch and Kannberg 1984). Further support for this interpretation is found in Tewhey et al. (1978), who reported that acidified high-temperature geothermal brine dissolved calcium carbonate from Kayenta sandstone.

3.4 SUMMARY OF FIELD TESTS

Membrane filter test results indicate that fluids injected during heat injection for all four storage cycles had very low suspended solids. Further, heat recovery fluids reinjected during cycles 1 and 2 had significant suspended solids content, while fluids reinjected during the heat recovery phase of cycles 3 and 4 had very low suspended solids. Metal

filings collected on filters provided evidence of pump bearing failure during cycles 1 and 2. Considering that fluids are alternately injected and withdrawn from each well of the doublet, the filter test results indicate that, at temperatures to 100°C, well impairment resulting from suspended solids is not a problem.

Results of core flooding tests demonstrate that several thousand pore volumes of fluid in the 50 to 90°C temperature range can be passed through representative core samples with no significant loss in permeability. Examination of one core sample after flow of approximately 25,000 pore volumes showed that a substantial portion of the cementing agents was dissolved.

4.0 GEOCHEMICAL STUDIES

In this section the development of the geochemical modeling codes, their use in ATEs applications, and an analysis of calcite precipitation kinetics and filter design are discussed in detail. In addition, guidelines for collecting and analyzing solutions for ATEs experimental studies are presented.

4.1 CODE DEVELOPMENT

At the beginning of FY82 the principal geochemical modeling code in use at PNL was WATEQ3 (Ball, Jenne and Cantrell 1981). This code is an advanced version of the original U.S. Geological Survey WATEQ code written by Truesdell and Jones (1973) to perform ion speciation and solubility calculations for aqueous solutions. The resulting description of relative saturation of the solution with respect to minerals in the code data base provides information on the types of water/rock interactions occurring in the system and the minerals that may be controlling the solution composition. For ATEs applications, it was decided that a useful feature to add to the code would be the ability to model mass transfers (mineral precipitation and dissolution) between the solution and solid phases. This advancement in modeling capability was achieved in FY82 by combining the thermodynamic data base and temperature and ionic strength correction routines of WATEQ3 with the mathematical structure of MINEQL (Westall, Zachary and Morel 1976). The new code is named MINTEQ (Felmy, Girvin and Jenne 1984).

With input data on solution composition and temperature, MINTEQ develops an equilibrium thermodynamic model of the system and calculates the activities of dissolved constituents in the solution and saturation indices of potential controlling minerals. By making use of a variety of model options, the investigator can choose to precipitate or dissolve minerals and note the resultant change in solution composition and the mass of mineral dissolved or precipitated to bring the solution into equilibrium with the

mineral(s) of interest. Simulations can be performed of raising the temperature of a solution as in a heat exchanger (see Section 4.2), oxidizing minerals in the reducing zone of an aquifer with oxygenated injection water, and dissolving or precipitating aquifer minerals as a result of solubility changes induced by heating the ground water.

The temperature correction routines for equilibrium constants in MINTEQ are limited to the range of 0 to 100°C. This range is below that expected for some ATEs applications; consequently, additional development of MINTEQ would be desirable to produce a code adequate to simulate all STES operations. In the interim, the EQ3NR/6 code (Wolery 1979) will be used for the higher temperature simulations. This code was installed on a PNL computer during FY82. EQ3NR/6 does not have the full capabilities of MINTEQ; consequently, it will serve simply as a backup to MINTEQ until MINTEQ can be improved.

4.2 MODEL APPLICATIONS

The MINTEQ geochemical modeling code was used to simulate the chemical response of the production water at the St. Paul FTF as it is heated. Analyses of Well B water sampled on May 15, 16, and 17, 1982, and injection water (heat exchanger effluent) sampled on May 18 were supplied by researchers at the St. Paul FTF. Table 4.1 lists a composite data set for the three Well B analyses and the analysis of the heated injection water.

With the input data from Table 4.1, MINTEQ was used to calculate the change in mineral solubilities and the amount of solid that might be expected to precipitate as the Well B water is heated. Heating was simulated by increasing the input temperature from 10°C to 90°C in increments of 10°C.

The results of the simulation for calcite (a mineral known to precipitate under these conditions) are given in Figure 4.1. A saturation index greater than zero indicates a solution is supersaturated with respect to a solid, and the solid may precipitate. An index less than zero means

TABLE 4.1. Chemical Analysis of Ground Water from Well B and Heated Injection Water (Concentration Units: mM)

Parameter	Well B	Injection Water
Temperature, °C	11	85
Alkalinity, mM	4.60	4.35
pH	7.46	7.29
Eh, mv	-100 ^(a)	
Ca, mM	1.36	1.21
Mg, mM	0.80	0.76
Fe, mM	0.015	0.001
H ₄ SiO ₄ , mM	0.104	0.104
SO ₄ , mM	0.1 ^(b)	0.1 ^(b)

- (a) Eh was estimated to be -100 mv (mildly reducing) for the Well B water to calculate ion speciation and mineral saturation indices.
(b) Sulfate analyses were not available at the time of the modeling study and had to be estimated.

FIGURE 4.1. Calcite Precipitation Simulation Results

undersaturation, and the equilibrium condition is represented by a saturation index of zero. Figure 4.1 shows that the ground water pumped from Well B (temperature = 11°C) is in approximate equilibrium with calcite. As the water is heated, the solution becomes increasingly supersaturated with calcite. The maximum amount of calcite that could precipitate from the solution at temperatures from 10 to 90°C is given in Figure 4.2. If the ground water is heated to 80°C as it flows through the heat exchanger at a rate of 1135 ℓ/min , a total of approximately 65,500 cm^3 of calcite could theoretically precipitate from the system per day. The analysis of the heat exchanger effluent water samples at 85°C (injection water, Table 4.1) shows that calcium carbonate (calcite) is being removed from the water; but, at the point sampled, calcite remains oversaturated with respect to the water (* in Figure 4.1). This suggests that calcite precipitation is not instantaneous, and that it will occur in the heat exchanger, piping, wellbore, and possibly in the aquifer itself.

FIGURE 4.2. Effect of Temperature on Calcite Precipitation

The saturation indices (SI) of iron minerals were also calculated for Well B ground water. An iron analysis was available for this water, but an Eh measurement was not. The Eh was estimated at -100 mv, which is representative of a confined aquifer under reducing conditions. Hematite (Fe_2O_3) is highly oversaturated (SI = 7.55) in this solution. Because of its large saturation index and slow rate of formation, hematite is not expected to be limiting iron concentration in this solution. Amorphous ferric hydroxide [$\text{Fe}(\text{OH})_3$] is undersaturated (SI = -2.56) in the ground water, but could influence iron concentration if the solution becomes oxidized. Siderite (FeCO_3) is near equilibrium (SI = -0.039) at the ground-water temperature and may be limiting iron concentration in the solution. Siderite solubility decreases with increasing temperature and would be expected to precipitate as the water is heated. However, iron concentration is approximately two orders of magnitude less than calcium in the ground water; consequently, the precipitation of iron minerals would cause much less plugging than the expected and observed amount of calcite formation.

4.3 IMPLICATIONS OF CALCITE PRECIPITATION RATE FOR ST. PAUL ATF

The precipitation of calcite in the wellbore of the St. Paul ATES facility inhibits the injectability of the heated ground water. One method for removing supersaturated calcite from solution is the addition of gravel filters downstream from the heat exchanger. The design of these filters must take into account the large amount of calcite that must be removed, the residence time of the flowing water in the filters, and the rate of calcite precipitation. Precipitation kinetic data for calcite and a variety of other minerals were reviewed and compiled in 1981 for the ATES Project by Don Langmuir and his associates^(a). Two different rate expressions for separate pH ranges were accepted by Langmuir as the closest fit to the available data. For pHs of 7 and below, the accepted rate law was developed by Plummer, Wigley and Parkhurst (1978). It has the form

^(a) Catts, J. G., M. J. Pavelich and D. Langmuir. 1981. Dissolution and Precipitation Kinetics of Calcite and Gypsum, Some Ferric Oxyhydroxides and Silica Polymorphs: A Literature Survey and Critique. Informal report HCS 81-9A, Hydrochem Systems Corporation, Golden, Colorado.

$$-\frac{d \frac{\text{MgCaCO}_3}{\text{m}^2 \text{ calcite}}}{dt} = k a_{\text{Ca}^{2+}} a_{\text{HCO}_3^-} \quad (4.1)$$

where k is the rate constant

a refers to activity of species.

For pHs in the range 8.4 to 9.0, Reddy's (1977) rate law best fits the data. This equation is

$$-\frac{d \frac{\text{MgCaCO}_3}{\text{m}^2 \text{ calcite}}}{dt} = kN^2 \quad (4.2)$$

where k is the rate constant

N is the molar concentration of calcite to be precipitated.

Langmuir believes that the difference in the rate laws reflects different, competing mechanisms at work in the separate ranges, which is reasonable considering the trend in dominant speciation of carbonate/bicarbonate between the two ranges.

The pH of the ground water at the St. Paul FTF is 7.46, which is outside the range of either of the rate laws, but is closest to the range in which Plummer's equation is believed to be valid. Using Plummer's rate constant and the activities of calcium and bicarbonate derived from a MINTEQ run using ground-water chemistry data, the rate of calcite precipitation is $205 \text{ mg/m}^2\text{hr}$. The surface area term (m^2) refers to the amount of calcite originally present on which precipitation can occur. If the dolomite gravel being considered for use in the filter pipes serves as an adequate substrate for precipitation and consists of 1-cm cubes, the approximate surface area exposed to 1 L of solution is 1.1 m^2 . At 80°C each liter of water would have to precipitate 100 mg of calcite to reach equilibrium with calcite (MINTEQ calculation). Using the derived precipitation rate, the amount of time required to precipitate this mass of calcite would be

$$\frac{100 \text{ g/l}}{\left(205 \frac{\text{mg}}{\text{m}^2 \text{hr}}\right) \times \left(1.1 \frac{\text{m}^2}{\text{l}}\right)} = 0.44 \text{ hr} = 26 \text{ min.} \quad (4.3)$$

The expected residence time in the filters is on the order of 10 s; consequently, if the rate expression is correct, there will not be sufficient time in the filters to remove all the necessary calcite. Using Reddy's rate expression, the precipitation rate would be 20 times faster, but his rate law may not be applicable in the pH range under consideration and the rate derived would still be much slower than necessary for the expected residence time in the filter pipes.

These calculations were performed assuming the filter particles consist of 1-cm (0.4-in.) cubes of calcite. The precipitation rate increases proportionally as the size of the particles decrease (surface area increases). Using Plummer's rate expression, the particles in the filter would have to be approximately 0.6 mm in diameter (coarse sand size) to have sufficient surface area to precipitate all the required calcite in 10 s.

The data Plummer used to derive his equation were obtained from experiments performed at 25°C. It is reasonable to assume that the precipitation will occur faster at the ATEs temperatures but, although no data are available to estimate the amount of this effect, it is doubtful that it would make a difference of two orders of magnitude as would be required if 1-cm gravel were used in the filters. Langmuir also reports that the precipitation rate increases as the partial pressure of CO₂ gas decreases below 0.3 atmospheres; however, at the expected CO₂ pressure of 0.01 atmospheres in the ATEs solution, the precipitation rate of calcite would be expected to increase only by a factor of 2 or 3.

In summary, the use of dolomite filters in the ATEs flow loop at the St. Paul FTF should enhance the precipitation of calcite; however, using Plummer's experimental rate data, gravel size sediment may not have a large enough surface area to remove a sufficient amount of calcite. Either the

residence time in the filters must be increased or the sediment size must be decreased significantly to make this method an effective means of equilibrating the water with calcite before injection of the water into the aquifer.

4.4 GUIDELINES FOR SAMPLING AND SOLUTION ANALYSIS

The successful aquifer storage and retrieval of energy in the form of heated or chilled water requires that the engineered system be compatible with the natural ground-water system. The composition of the ground water must be well known to predict and avoid potential problems that heating or cooling the water may create for operation of the plant. During FY82, guidelines were written for sampling and analyzing solutions from simulated or real ground-water energy storage systems (Deutsch 1982).

Sampling guidelines include methods for flushing wells of stagnant water and monitoring selective solution parameters (pH, Eh, temperature, or conductivity) as indicators of the efficiency of flushing. Certain unstable ground-water parameters (temperature, pH, Eh, dissolved oxygen, and conductivity) should be measured onsite. It is recommended that alkalinity, sulfide, and ammonia determinations be done within 24 hr of sampling. In addition to these field measurements, samples of the ground water should be filtered, preserved, and stored for laboratory analysis of major cations, anions, trace metals, organic and inorganic carbon and certain redox couples ($\text{Fe}^{2+}/\text{Fe}^{3+}$ and $\text{As}^{3+}/\text{As}^{5+}$). The final results of the analysis should be verified by computing the cation-anion balance and comparing measured conductivity with the solution analysis.

5.0 LABORATORY STUDIES

The laboratory studies consisted of two major activities. First, a rock mechanics facility was developed and constructed that would provide capability to measure permeability, pore fluid chemistry, and material properties of reservoir rocks at conditions equivalent to those imposed on a storage aquifer by an ATEs facility. The second effort comprised actual physicochemical testing of core samples from the FIG aquifer at the St. Paul FTF. The rock mechanics facility was used to measure permeability and monitor fluid geochemistry. This section describes the facility and documents the physicochemical test procedures and results.

5.1 APPARATUS

Development of a laboratory facility for testing reservoir materials at pressures, temperatures, and flow rates characteristic of ATEs was initiated in FY81 with the design and fabrication of the Aquifer Properties Test Facility (APTF), a state-of-the-art rock mechanics device. This apparatus was designed specifically for testing rock samples from ATEs sites and is shown in Figure 5.1. Table 5.1 summarizes capabilities of the apparatus, which was designed to accommodate not only physical properties research, but also geochemical research on pore fluid. The APTF provides capabilities to monitor up to 20 parameters simultaneously.

A microcomputer-based data acquisition system (DAS) has been developed and incorporated into the APTF. This DAS system converts analog signals to digital equivalents, averages these data, and writes data to disk and line printer. This DAS is illustrated schematically in Figure 5.2. Data output from the DAS are then preprocessed on the microcomputer and transmitted via phone lines to a VAX computer. Figure 5.3 shows the hardware configuration used to process data collected by the APTF. The names of computer programs used to process the data are listed in Table 5.2, along with program functions and computer location.

FIGURE 5.1. Aquifer Properties Test Facility

TABLE 5.1. Aquifer Properties Test Facility Capabilities

Parameter	Symbol	Measurement Range	Accuracy
Confining pressure	P_c	0 to 15,000 psi (0 to 1 kbar)	± 10 psi
Pore pressure	P_c	0 to 15,000 psi	± 10 psi
Sample temperature	T_s	0 to 200°C	$\pm 1^\circ\text{C}$
Fluid temperature (outside vessel)	T_F	0 to 95°C	$\pm 1^\circ\text{C}$
Flow	Q	0 to 0.15 $\ell/\text{min.}$	± 0.001 ℓ/min
Pressure difference across sample	ΔP	0 to 12,000 psi	N/A
Sample load		0 to 100,000 lb	10 lb

FIGURE 5.2. Schematic of Data Acquisition System for the Aquifer Properties Test Facility

FIGURE 5.3. Hardware Configuration for Data Acquisition and Processing System

5.2 SAMPLE PREPARATION AND TESTING PROCEDURES

Core samples tested in this study were Ironton-Galesville sandstone, which forms the lower section of the storage aquifer at the St. Paul site (see Figure 2.1). These samples represent the most permeable zone of the storage aquifer and consist of a friable, medium-grained, quartz sand with weak dolomitic cement (Walton et al. 1984). Samples for laboratory testing were subcored parallel to bedding and consisted of right circular cylinders 5.4 cm in diameter and 5 to 7 cm in length; the lengths of the cores were oriented parallel to bedding. Samples were oven-dried for a minimum of

TABLE 5.2. Computer Programs Associated with the Aquifer Properties Test Facility

<u>Program Name</u>	<u>Function</u>	<u>Machine</u>
'DAS'	Monitors analog Performs A/D conversion Averages data Writes floppy disk Logs on line printer	Apple II +
TRANSEND	Sends data file over modem to VAX	Apple II
T.	Runs on VAX to receive data transmittal via modem	VAX
LOOPDAT	Converts files sent from Apple II into binary files	VAX
PARASEL	Reads binary files and writes selected parameters to a formatted file	VAX
RS1	Table data processing routine	VAX

48 hr at approximately 80°C. For each test a core sample assembly was prepared as shown in Figure 5.4a or 5.4b. This assembly was incorporated into the sample stack assembly as shown in Figure 5.5. The sample stack assembly was evacuated to remove air from the sample before introducing pore fluid and then vacuum-saturated with pore fluid and placed in the pressure vessel.

The configuration of the APTF for these tests is shown schematically in Figure 5.6. Hydrostatic confining pressure of 6.2 MPa (900 psi) was

a

b

FIGURE 5.4. Sandstone Core Sample Assembly: a) used in Tests 1 and 2; b) Used in Tests 3 Through 8.

FIGURE 5.5. Sample Stack Assembly

introduced and held constant. Pore fluid was provided to the sample at a pressure of approximately 2.4 MPa (350 psi) and a flow rate of 0.35 ml/s; these conditions simulate in-situ conditions imposed by the ATEs facility. Flow was measured by an inline paddlewheel flow meter and was calculated from pump displacement. Pressure drop across the sample was measured with a differential pressure transducer. Temperature was controlled by an internal immersion heater within the pressure vessel and by heaters in the pore fluid system. To minimize pore fluid contamination from the apparatus (Stottlemire, Cooley and Banik 1981), all wetted parts were constructed of Inconel, Hastelloy, titanium, or 316 stainless steel. Additional detail on the sample setup and test apparatus is presented by Blair and Stottlemire (1981).

The general procedures for each test were as follows. The sample was vacuum-saturated with pore fluid. The confining stress was introduced. Pore pressure was increased to the desired level; flow was started through the sample and adjusted until it stabilized at the desired rate. Flow was maintained only during daytime hours; however, confining pressure, pore pressure, and temperature were maintained at appropriate levels throughout the testing. Fluids were sampled throughout the test duration. Scanning

FIGURE 5.6. Schematic of Permeability Measurement Apparatus

electron microscopy was used to characterize rocks and filters before and after testing.

Data were collected and reduced as follows:

1. The 'DAS' program was started and the signal sampling rate was set to 5 s.
2. The 5-s data were averaged over 2-min intervals and converted to scientific units. Appropriate corrections were applied and a permeability was calculated, based on averaged and corrected data. These computed corrected values were written to disk. Thus, each disk record represented an average of 24 5-s samples.

Permeability of the core was calculated as follows. The basic equation for permeability (k), assuming one-dimensional, laminar flow in a cylindrical plug, is given by Darcy's Law as

$$-k = \frac{Q \mu l}{A \Delta P} \quad (5.1)$$

where k = intrinsic permeability, darcies

Q = volume flow rate, ml/s

l = sample length, cm

A = sample cross-sectional area, cm^2

ΔP = pressure difference across the sample, bars

μ = pore fluid viscosity, centipoise.

A pore fluid density correction is applied to the flow rate because the flow rate may be measured at a point where fluid is at a different temperature than when in the core. The correction is given by

$$Q_c = Q_m \frac{V_t}{V_m} \quad (5.2)$$

where V_t = specific volume of the pore fluid at the sample, cm^3/g

V_m = specific volume of the pore fluid at the flow meter, cm^3/g

Q_m = volume flow rate at the flow meters, ml/s

V_t and V_m are calculated using the relationship^(a)

$$\frac{V_t}{V_m} = \frac{CT + 1}{C_0 + C_1T + C_2T^2 + C_3T^3 + C_4T^4 + C_5T^5}$$

where $C = 0.01688$

$$C_0 = 999.8400$$

$$C_1 = 16.9452$$

$$C_2 = -7.98704 \times 10^{-3}$$

$$C_3 = -4.61705 \times 10^{-5}$$

$$C_4 = 1.05563 \times 10^{-7}$$

$$C_5 = -2.8054 \times 10^{-10}$$

Fluid viscosity is also temperature-dependent and is calculated for flow at temperature from the relationship for temperature < 100°C

$$\mu = 10^x$$

$$\text{where } x = - \frac{a(T-20) + b(T-20)^2}{T + 105} + c$$

$$a = 1.3272$$

$$b = 1.053 \times 10^{-3}$$

$$c = 8.68 \times 10^{-4}$$

For temperature > 100°C

$$\mu = 39.3822 T^{-1.0728}$$

T = temperature (°C)

This is derived by fitting data from the CRC Handbook.

3. After completion of the testing, the 2-min data were examined and compared with the operations log and analog data records to identify

(a) CRC Handbook of Chemistry and Physics

intervals of steady-state flow and to verify sensor integrity. A reduced data set was formed by averaging the 2-min data over intervals where flow and temperature were judged to be steady-state. These reduced data are presented and discussed in this report.

5.3 EXPERIMENTS AND RESULTS

Eight laboratory experiments are reported in this section. These tests monitored permeability in core samples from the FIG aquifer at St. Paul at a variety of temperature and flow conditions and using different fluid chemistries. These tests are summarized in Table 5.3 and are discussed individually in the following subsections.

5.3.1 Test 1

To determine the extent to which high flow rate might affect core permeability, a permeability test was performed at a flow rate equivalent to that in the near-well region at ambient temperature. The resulting permeability data for the core sample tested are presented in Figure 5.7. As the data indicate, core sample permeability decreased steadily with time during the first 9 hr of flow, and then stabilized at approximately 0.6 of the original value. Permeability increased somewhat when flow direction was reversed (hour 13 in Figure 5.7), which may indicate some migration of internal fines (Jorda 1980; Khilar and Folger 1983). These data also suggest that before core samples of Ironton-Galesville sandstone are tested at high temperature, they should be flow-tested at ambient temperature for 6 to 8 hr so that a stable permeability is achieved.

5.3.2 Test 2

One high-temperature permeability test was performed on sample AC-1-786H to monitor the effect of increasing temperature on permeability. For this test a synthetic ground-water solution, which simulated actual fluid injected at the site, was used as the pore fluid. Composition of this fluid is listed as synthetic Fluid 1 in Table 5.4. Flow rate, confining pressure, and pore pressure were maintained constant at values cited

TABLE 5.3. Summary of Laboratory Tests

<u>Test No.</u>	<u>Test Name</u>	<u>Core ID</u>	<u>Temperature (°C)</u>	<u>Fluid Composition</u>	<u>Duration (Days)</u>	<u>Reverse Flow</u>
1	MN786B	AC-1-786	Ambient	Deionized water	3	Yes
2	MN786HC	AC-1-786	50 90 150	Fluid 1	4	No
3	MN785HA	AC-1-785	90	Fluid 1	4	Yes
4	MN785HB	AC-1-785	125	Fluid 1	4	Yes
5	MN785HC	AC-1-785	150	Fluid 1	4	Yes
6	MN775HA	AC-1-775	90	Fluid 2	4	Yes
7	MN775HB	AC-1-775	125	Fluid 2	4	Yes
8	MN775HC	AC-1-775	150	Fluid 2	4	Yes

FIGURE 5.7. Normalized Permeability Versus Time for Test 1

TABLE 5.4. Fluid Compositions for Tests on Samples AC-1-786H, AC-1-785H and AC-1-775H

Parameter	Injected Fluid (4/83)	Synthetic Fluid 1	Synthetic Fluid 2 (Ion Exchange)
Cations			
Ca	32.32	31.00	-
Fe (Ferrous)	1.06	0.95	-
K	7.82	7.21	-
Mg	17.86	15.14	-
Na	5.22	5.94	60.30
Cl	1.45	< 5.0	< 5.0
F	0.27	< 1.0	< 1.0
SO ₄	7.39	9.86	10.20
SiO ₂	10.56	11.72	11.56
Alkalinity (mg/l CO ₃ ⁻)	203.4	96.32	81.45
pH	6.8 to 6.9	6.8	6.9

All units mg/l except alkalinity and pH

previously. Permeability was measured during periods of flow while temperature was increased as shown in Figure 5.8.

Laboratory data for this test are summarized in Appendix B, Table B-1 and normalized permeability is plotted against temperature in Figure 5.9.

FIGURE 5.8. Flow and Temperature History for Test 2

FIGURE 5.9. Normalized Permeability Versus Temperature for Test 2

These data (summarized in Table 5.5) indicate that permeability decreased with increasing temperature. Chemical analyses of pore fluid samples taken during this test are shown in Table 5.6. These data show that the

TABLE 5.5. Summary Data for Test 2

Test Date	Temperature (°C)	K/K _o	No. of Points	Sigma
March 24	22	0.89	48	0.051
March 28	51	0.51	67	0.057
March 29	91	0.30	65	0.016
March 30	151	0.18	79	0.011

TABLE 5.6. Chemical Analysis (in mg/l) of Pore Fluid for Test 2

Chemical Constituents	Synthetic Ground Water	Temperature of Flow Experiment		
		22°C	90°C	150°C
Ca	21.0	42	50	37
K	< 0.3	0.58	3.10	4.6
Mg	19	21	22	10
Na	5	6.6	7.2	6.4
S	< 0.1	32	36	34
Si	6.5	9.9	38	19
Carbonate (mg/l CO ₃ ⁻)	68.0	57.6	28.8	26.2

concentrations of dissolved Ca, Mg, S, and Si all increased as the temperature was raised to 90°C, then decreased as the temperature was raised to 150°C. Although the sample was somewhat friable before the test, it lost all structural integrity during testing, as shown in Figure 5.10.

Scanning electron microscopy (SEM) photomicrographs showing the core sample before and after testing are presented in Figure 5.11. As can be seen, the rock initially was a relatively clean, weakly cemented sandstone. However, after the high-temperature test with synthetic injection fluid, substantial quantities of internal fines were generated and deposited evenly across the end of the rock. These fines range in size from submicron to 50 micron. Composition of these fines is that of the general rock matrix. Also, some of the matrix sand grains appear to be developing networks of fractures. This result indicates that much of the final permeability

FIGURE 5.10. Sample AC-1-786H After Testing at 150°C, Test 2

FIGURE 5.11. Scanning Electron Microscopy Evidence of Core Microstructure Changes with Temperature for Test 2: a) Pre-Test Core Microstructure; b) Post-Test Core Microstructure

reduction observed during this test can be attributed to particle plugging of pore throats (Khilar and Folger 1983). Examination of the geochemical data indicates that silica and calcium concentrations in the pore fluid increased substantially. This dissolution from the core sample may have dissolved cementing agents and liberated particles.

5.3.3 Tests 3, 4, and 5

After reviewing results of Test 2 along with other laboratory permeability data collected on similar samples, a series of three experiments were performed on sample AC-1-785H to provide further data on the observed loss of permeability and disintegration of rock fabric. Each experiment ran for 4 days. The generalized temperature and flow history for these tests is shown in Figure 5.12.

FIGURE 5.12. Generalized Description of Temperature, Flow and Fluid Sampling History for Tests 3 Through 8. Tests 3, 4, and 5 used Fluid 1 (Table 5.4) while Tests 6, 7, and 8 used Fluid 2. For Tests 3 and 6, $T_i = 90^\circ\text{C}$, for Tests 4 and 7, $T_i = 125^\circ\text{C}$ and for Tests 5 and 8, $T_i = 150^\circ\text{C}$.

The sample setup for these tests also was changed from that of previous tests as a series of three sandstone wafers was incorporated into the sample stack. These wafers could be easily removed after each test and then analyzed for changes in porosity, pore structure, and composition. The sample stack design used in Tests 3, 4, and 5 is shown schematically in Figure 5.4b.

During day 1 of each test of the series, the sample was kept at elevated pressure and ambient temperature. Flow was introduced at approximately 8 a.m. and was maintained in the normal direction for approximately 8 hr. Elevated confining and pore pressure was maintained overnight. On the morning of the second day, flow was reintroduced and permeability monitored for approximately 1 hr. Sample temperature was then

raised: for Test 3 to 90°C; for Test 4 to 125°C; for Test 5 to 150°C, and permeability was monitored continuously. Flow was stopped after approximately 8 hr; however, elevated pressure and temperature were maintained overnight. On the morning of the third day, flow was introduced in the normal direction for approximately 1 hr. Then flow direction was reversed and flow maintained for approximately 8 hr. The sample was again maintained overnight at elevated pressure and temperature without flow. On the fourth day of each test, flow was initiated and permeability monitored for approximately 1 hr before temperature was lowered to ambient. As on previous days, flow was maintained for approximately 8 hr. Pore fluid with composition of Fluid 1 in Table 5.4 was used in all three tests. As mentioned earlier, this fluid composition simulated fluid injected at the St. Paul site.

Laboratory data for Test 3 are presented in summary form in Table B.2. Data for temperature and normalized permeability (K/K_0) versus time from start of the test are plotted in Figure 5.13. Permeability has been normalized to the highest value of permeability at the start of each test to allow comparison between tests. Figure 5.13 shows that when temperature was raised to 90°C, permeability dropped to half its initial value and remained stable, even when flow was reversed. Some permeability was recovered when temperature was reduced.

Laboratory data for Test 4 are summarized in Table B.3, and data for temperature and normalized permeability versus time are plotted in Figure 5.14. These data show that during this test, permeability dropped when the sample was heated, and remained stable. Some permeability was recovered when the temperature was lowered to ambient.

A much different result was obtained for Test 5 at 150°C. Summary data for this test are listed in Table B.4 and temperature and normalized permeability versus time from start of the test are shown in Figure 5.15. Permeability data for the first 2 days of this test are similar to data collected in early parts of Tests 3 and 4; however, on the third day of the 150°C test the permeability decreased dramatically when flow was initiated and showed sensitivity to minor perturbations in the flow conditions. (The

FIGURE 5.13. Temperature and Normalized Permeability Versus Time for Test 3
Sample AC-1-785H

drastic fluctuations in permeability shown in Figure 5.15 are associated with pore fluid sampling activity.) This behavior is consistent with permeability impairment by migration of internal fines.

Data for normalized permeability of sample AC-1-785H (Tests 3, 4, and 5) as a function of temperature are illustrated in Figure 5.16 and

FIGURE 5.14. Temperature and Normalized Permeability Versus Time for Test 4, Sample AC-1-785H

summarized in Table 5.7. These data indicate that, although permeability was reduced 50% to 60% in Tests 3 and 4 (90 and 125°C), testing at 150°C caused loss of 80% of original permeability.

FIGURE 5.15. Temperature and Normalized Permeability Versus Time for Test 5 Sample AC-1-785H

As mentioned earlier, a wafer was removed from the sample stack after each test. These wafers for sample AC-1-785H are shown in Figure 5.17 along with a wafer of untested sample. Note that while wafers B (90°C test) and C (90°C and 125°C tests) appear much like the untested sample, wafer D from the 150°C test is very friable and has fine particles on the surface.

FIGURE 5.16. Normalized Permeability Versus Temperature for Sample AC-1-785H (Tests 3, 4, and 5). Error bars show 95% confidence limits.

TABLE 5.7. Summary Data for Tests 3, 4, and 5

Test Name	Temperature (°C)	K/K ₀ - Mean N ^o Flow	K/K ₀ - Mean R ^o Flow	Sigma
MN785HA	20	0.91	0.00	0.058
MN785HA	21	0.00	0.62	0.021
MN785HA	91	0.50	0.00	0.027
MN785HA	92	0.00	0.54	0.049
MN785HB	20	0.90	0.00	0.043
MN785HB	20	0.00	0.49	0.017
MN785HB	136	0.45	0.00	0.039
MN785HB	127	0.00	0.36	0.048
MN785HC	20	0.85	0.00	0.060
MN785HC	22	0.00	0.20	0.023
MN785HC	150	0.51	0.00	0.070
MN785HC	151	0.00	0.30	0.160
MN785HC	22	0.44	0.00	0.064

Samples From Tests Using Fluid #1

Sample	Comment
A.	Untested Sample
B.	Tested at 90°C
C.	Tested at 90° and 125°C
D.	Tested at 90, 125, and 150°C

FIGURE 5.17. Samples From Tests 3, 4, and 5 Using Fluid 1

Figure 5.18 presents SEM photomicrographs of both untested rock and a wafer that has been tested at 90, 125, and 150°C. This figure shows that small (1- to 10- μm) particles of feldspar and other minerals present in the untested sample are essentially absent from the tested sample. It is likely that the migration of these particles causes the permeability impairment.

An optical photomicrograph of a wafer tested during tests 3, 4, and 5 is presented in Figure 5.19. This photomicrograph was produced from a thin section made from the wafer. In this photomicrograph the porosity of the sample appears dark as the sample was impregnated with a dyed epoxy before

FIGURE 5.18. SEM Photomicrographs of Sample AC-1-785H Before and After High Temperature Flow Tests: a) Untested Sample; b) Sample Flow Tested at 90°, 125° and 150°C

sectioning. Digital comparison of porosity in sections prepared from untested and tested samples indicates that testing increased porosity from ~10% to ~16%.

5.3.4 Tests 6, 7, and 8

Tests 6, 7, and 8 were conducted on sample AC-1-775H in a manner similar to testing of sample AC-1-785H discussed above, except that the composition of pore fluid used in these tests simulated composition of injection fluid softened by sodium ion exchange. The composition of this fluid is shown as Fluid 2 in Table 5.4. Temperature, flow, and fluid sampling history for these tests are similar to that for Tests 3, 4, and 5 (see Figure 5.12). The sample was again constructed with wafers incorporated into the assembly as shown in Figure 5.4b.

Laboratory data for Test 6 (90°C) are summarized in Table B.5 and temperature and normalized permeability versus time are shown in Figure 5.20. These data show that increasing temperature had very little effect on permeability in this test.

FIGURE 5.19. Optical Photomicrograph of a Wafer Tested During Tests 3, 4, and 5

FIGURE 5.20. Temperature and Normalized Permeability Versus Time for Test 6, Sample AC-1-775H

Summary laboratory data for Test 7 are presented in Table B.6 and temperature and normalized permeability are shown as a function of time in Figure 5.21. These data show a dramatic loss of sample permeability as temperature is increased from ambient to 125°C. Permeability was erratic

FIGURE 5.21. Temperature and Normalized Permeability Versus Time for Test 7, Sample AC-1-775H

after flow direction was reversed on the third day (hour 50) and was stable on the fourth day, but dropped again to less than one-tenth the original value when temperature was lowered.

Laboratory data for Test 8 (150°C) are presented in Table B.7. Temperature and normalized permeability data versus time are shown in Figure 5.22. This figure shows that permeability decreased from to three-tenths of the original value as temperature was raised to 150°C. Dramatic

FIGURE 5.22. Temperature and Normalized Permeability Versus Time for Test 8, Sample AC-1-775H

fluctuations in permeability are associated with flow reversal and fluid sampling again indicating migration of fines. Permeability remained stable as temperature was lowered to ambient.

Normalized permeability data versus temperature for Tests 6, 7, and 8 are illustrated in Figure 5.23 and summarized in Table 5.8. The sensitivity of permeability to flow direction and the size of the 95% confidence limits for reversed flow indicate migration of fines to be the cause of impairment.

The effect of high temperature flow on this core sample is clearly shown in Figures 5.24a and b. Figure 5.24a is a photograph of untested core while the sample tested at 90°, 125° and 150°C using Fluid 2 is shown in Figure 5.24b.

Additional evidence of fines migration is shown in Figure 5.25. Photomicrographs 5.25a and b show the detail of cementing agents present in untested samples. Photomicrograph 5.25c shows the sample after testing at 90, 125, and 150°C. Note the broken nature of the grains. Figure 5.25d shows particles filtered from the pore fluid solution. The feldspar particles deposited on the filter are interpreted to be derived from the rock.

FIGURE 5.23. Normalized Permeability Versus Temperature for Sample AC-1-775H (Tests 6, 7, and 8)

TABLE 5.8. Summary Data for Tests 6, 7, and 8

Test Name	Temperature (°C)	$K/K_0 -$ Mean N^0 Flow	$K/K_0 -$ Mean R^0 Flow	Sigma
MN775HA	20	0.98	0.000	0.0390
MN775HA	22	0.00	0.890	0.0730
MN775HA	92	0.63	0.00	0.0260
MN775HA	87	0.00	0.930	0.0500
MN775HB	20	0.90	0.000	0.0480
MN775HB	19	0.00	0.036	0.0003
MN775HB	124	0.40	0.000	0.0370
MN775HB	127	0.00	0.540	0.1070
MN775HC	21	0.83	0.000	0.1100
MN775HC	22	0.00	0.230	0.0050
MN775HC	145	0.24	0.000	0.0790
MN775HC	152	0.00	0.290	0.0760

5.3.5 Observations from High Temperature Tests

Analysis of permeability data from Tests 3 through 8 yields the following observations.

1. During the first day of each test (ambient temperature flow), sample permeability decreased between 10 and 20%. This is consistent with the loss of permeability observed during the first 8 hours of flow in Test 1, and is attributed to migration of internal fines dispersed by the imposed flow and pressure conditions.
2. Initial heating caused substantial reduction of permeability in all tests and heating to higher temperatures generally caused a greater drop in permeability. Summary data for loss of permeability versus initial heating are summarized in Table 5.9 and shown graphically in Figure 5.26.

FIGURE 5.24. Effect of High Temperature Flow on Sample AC-1-775H: a) Untested; b) Tested at 90°, 125°, and 150°C Using Fluid 2

A, B - Untested Sample

C - Sample Flow Tested at 150°C

D - Particles Filtered From Pore Fluid (150°C Test)

FIGURE 5.25. SEM Photomicrographs Showing Effect of Flow Test on Sample AC-1-775H: A,B) Untested Sample; C) Sample Flow Tested at 150°C; D) Particles Filtered from Pore Fluid (150°C) (Test 8)

TABLE 5.9. Summary Data for Permeability Loss During Initial Heating for Tests 2 through 8

Test	ΔT (°C)	ΔK (% K_0)
2	27	-21
3	70	-30
4	116	-37
5	130	-36
6	70	-30
7	105	-(50 to 60)
8	125	-(40 to 45)

FIGURE 5.26. Percent Permeability Loss During Initial Heating Versus Change in Temperature (Heating Gradients Were 0.5 to 1°C/min)

3. Permeability data for these tests generally show large fluctuations in permeability on day 3 of testing, after overnight residence of fluid in the sample at elevated temperature. This may be due to increased dispersion of fines caused by dissolution of the cementing agents.
4. Flow testing at elevated temperature caused permanent reduction of permeability in all samples and higher temperatures generally caused a greater reduction. However, it is interesting to note that when temperature was lowered to ambient, samples tested using Fluid 2 behaved much differently than those tested using Fluid 1. In particular, as the temperature was lowered, samples tested using Fluid 1 gained significant permeability (10 to 20% of K_0) and then showed no change or a small increase in permeability during the remaining few hours of flow at ambient temperature. In contrast, samples tested at 90°C and 125°C using Fluid 2 showed very little change in permeability during actual cooling but then lost from 20 to 35% of their initial permeability during flow at ambient temperature.

Examination of the tested and untested samples using techniques of scanning electron microscopy (SEM) and optical microscopy coupled with image processing indicates that in these flow experiments much of the cementing material was removed (dissolved). Microscopic studies also indicate that porosity of the rock matrix increased a few percent due to the cement dissolution. This dissolution caused dispersion of fine particles bonded by the cement. These particles then migrated to the core-end faces and impaired sample permeability (see Figure 5.11b). Microscopic study also indicates some degradation of quartz grains in the samples.

5.4 DISCUSSION

Laboratory tests produced two results that are significant to ATEs. The first is that on the time scale of these tests, substantial changes in petrofabric were observed in the samples. These included loss of cementing agents, an increase in intergranular porosity, and loss of mechanical strength. Available results on changes in pore fluid chemistry indicate increased silica content after residence of pore fluid in the core for a few hours (overnight) at elevated temperature. This result is important to ATEs because it shows that the time scale of chemical reactions occurring in aquifers used for ATEs is on the order of a few days or less and possibly a few hours. This result also indicates that cementing agents are removed first, dispersing fine particles and increasing porosity.

A second result is that initial heating of samples caused significant reduction in permeability in all laboratory tests and the amount of reduction was proportional to the rise in temperature. This is important to heat injection operations because it indicates that permeability reduction may occur in the receiving formation when temperature of the injection stream is increased.

These results then indicate that the fabric of the FIG aquifer rocks that form the host aquifer for the St. Paul FTF is probably being changed by operation of the ATEs facility. However, it is apparent from the success of field operations to date that for short-term tests, these changes in the

rock fabric do not impact the hydrologic performance of the aquifer formation. Field data from longer storage tests, along with downhole logs will be necessary to confirm that cement dissolution is increasing porosity and to assess the impact on site performance.

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APPENDIX A

DETAILED FITS PROCEDURE

APPENDIX A

DETAILED FITS PROCEDURE

Filter Preparation

1. Prepare membrane filters by labeling outside edge, drying at 80°C for at least 2 hours, and weighing to the nearest 0.001 gram.
2. Enter filter number, filter pore size, type, and weight into a laboratory notebook table which has entries for filter holder used, dry weight of filter after test, and suspended solids (difference in dry weights divided by cumulative flow volume).
3. Secure stainless steel Millipore filter holder in vise.
4. Rinse holder with distilled water.
5. Place drain disc and then filter membrane in center of downstream side of holder.
6. Center upstream half of holder over filter; tighten bolts in a star pattern.
7. Cover upstream opening of holder with a piece of tape marked with filter number and pore size.

Test Stand Preparation

1. Flush exterior tubing or pipes and connect to desired sampling point of main system fluid stream.
2. Install filter holder into flow system panel, tightening all fittings.
3. Attach heater jacket around holder and set temperature to that expected of the fluid stream.
4. Deaerate flow panel with vacuum pump.
5. Slowly let water into flow panel lines by opening all valves except drainage valves.

Testing

1. Prepare data sheet by entering filter number, date, time, filter pore size and type, fluid stream source description and source temperature. Source pressure is recorded just before testing begins.
2. Begin stopwatch and digital printout (with data inputs selected on patch panel for data logger) at the instant flow is allowed through filter.
3. Adjust flow (using drain valves) such that the differential pressure across the filter is kept at a constant value.
4. At 1-minute intervals, record flow rate and cumulative flow volume. Also, note initial flow rate and time elapsed until desired differential pressure is established and maintained.
5. Record input temperature and pressure every few minutes throughout the test, and note any changes in differential pressure from the desired value.
6. Stop flow, stopwatch, and digital printout after desired testing time (usually 20 minutes for membrane filters).
7. Turn off and remove filter heater jackets from holder.
8. Pull last bit of flow through flow meter with vacuum pump and record total cumulative flow volume on data sheet.
9. Remove filter from holder and dry to allow final weighing for suspended solids calculation.

APPENDIX B

LABORATORY TEST DATA

TABLE B.1. Laboratory Data for Test 2

4N766C 2590 x 70

1	2 TIME (HR)	3 FLOW (ml/min)	4 OF (PSI)	5 K (+1)	6 K/K ₀	7 P-P	8 TEMP C
1	13.1	22.8	1.2	1040	0.98	508	22
2	13.2	22.2	1.2	1060	1.00	505	22
3	13.3	19.8	1.1	1040	0.98	486	22
4	13.7	21.0	1.3	1060	1.00	418	22
5	13.4	24.0	1.4	1030	0.97	434	22
6	13.4	22.2	1.3	1020	0.96	396	22
7	13.9	21.0	1.2	1000	0.94	389	22
8	14.0	20.4	1.2	980	0.92	318	22
9	14.0	16.8	1.0	1000	0.94	287	22
10	14.1	19.8	1.1	1030	0.97	374	22
11	14.2	19.8	1.2	990	0.93	389	22
12	14.2	17.4	1.1	970	0.92	332	22
13	14.3	21.0	1.2	940	0.92	436	22
14	14.4	22.2	1.3	980	0.92	521	22
15	14.4	21.0	1.3	940	0.89	493	22
16	14.5	19.8	1.3	930	0.88	479	22
17	14.6	20.4	1.3	920	0.87	472	22
18	14.6	17.4	1.1	910	0.86	370	22
19	14.7	16.8	1.1	940	0.89	356	22
20	14.8	17.4	1.1	930	0.88	369	22
21	14.8	17.4	1.1	940	0.89	366	22
22	14.9	19.8	1.2	940	0.89	316	22
23	15.0	14.0	1.1	960	0.91	304	22
24	15.0	22.8	1.4	930	0.88	462	22
25	15.1	21.0	1.4	910	0.86	461	22
26	15.2	20.4	1.3	900	0.85	484	22
27	15.2	19.2	1.3	910	0.86	458	22
28	15.3	18.6	1.2	910	0.86	429	22
29	15.4	22.8	1.4	950	0.90	323	22
30	15.4	21.6	1.4	930	0.88	359	22
31	15.5	21.6	1.4	930	0.88	351	22
32	15.6	19.8	1.3	920	0.87	328	22
33	15.6	24.0	1.5	940	0.89	445	22
34	15.7	24.0	1.6	900	0.85	496	22
35	15.4	22.8	1.5	900	0.85	456	22
36	15.5	22.8	1.5	910	0.86	463	22
37	15.9	21.6	1.4	910	0.86	414	22
38	16.0	14.0	0.8	870	0.82	217	22
39	16.4	15.8	1.1	970	0.92	411	21
40	16.5	20.4	1.3	950	0.91	463	21
41	16.5	16.8	1.1	890	0.84	498	21
42	16.6	17.4	1.1	910	0.86	531	21
43	16.7	21.0	1.4	920	0.87	398	21
44	16.7	16.2	1.1	860	0.81	377	21
45	16.8	12.6	0.9	840	0.79	261	22
46	16.9	22.2	1.5	890	0.84	480	22
47	16.9	16.2	1.0	890	0.84	376	22
48	16.1	17.4	1.1	860	0.81	348	23
49	16.7	19.2	1.0	640	0.60	271	50
50	16.7	21.0	1.1	650	0.61	437	51
51	16.8	24.6	1.3	640	0.60	503	51
52	16.9	23.4	1.2	650	0.61	481	51
53	16.9	22.8	1.2	630	0.59	515	51
54	16.9	21.0	1.1	620	0.58	464	51

TABLE B.1. Continued

MV786C 2500 x 70

0	1 TIME (HR)	2 FLOW (ml/min)	3 CP (PST)	4 K (ml)	5 K/K ₀	6 P-P	7 TEMP C
55	106.1	18.6	1.0	630	0.59	430	51
56	106.1	21.6	1.3	640	0.60	531	51
57	106.2	21.0	1.3	630	0.59	532	50
58	106.3	21.0	1.2	610	0.58	477	50
59	106.3	21.0	1.2	600	0.57	446	51
60	106.4	18.8	0.9	620	0.58	324	51
61	106.5	19.0	1.0	620	0.58	359	51
62	106.5	19.0	1.1	610	0.58	422	52
63	106.6	22.2	1.2	620	0.58	480	52
64	106.7	24.0	1.3	590	0.56	431	51
65	106.7	21.0	1.2	580	0.55	409	50
66	106.8	18.0	1.0	580	0.55	247	50
67	106.9	22.2	1.2	600	0.57	476	50
68	106.9	21.0	1.2	590	0.56	520	50
69	107.0	22.2	1.2	590	0.56	479	50
70	107.1	21.0	1.2	590	0.56	465	50
71	107.1	18.0	1.1	560	0.53	375	50
72	107.2	25.2	1.4	580	0.55	436	51
73	107.3	23.4	1.3	580	0.55	478	51
74	107.3	22.2	1.3	570	0.54	454	51
75	107.4	22.8	1.3	590	0.55	481	51
76	107.5	22.2	1.3	570	0.54	442	51
77	107.5	19.8	1.1	570	0.54	385	51
78	107.6	21.6	1.4	570	0.54	560	51
79	107.7	24.0	1.4	560	0.53	555	51
80	107.7	23.4	1.4	550	0.52	494	51
81	107.8	22.2	1.4	540	0.51	467	51
82	109.0	31.8	1.7	600	0.57	554	54
83	109.1	21.0	1.2	550	0.52	504	53
84	109.1	21.6	1.3	540	0.51	479	52
85	109.2	19.2	1.2	520	0.49	446	52
86	109.3	19.2	1.2	520	0.49	452	52
87	109.4	18.6	1.2	500	0.47	437	52
88	109.4	15.0	1.0	480	0.45	319	52
89	109.5	24.6	1.5	530	0.50	518	52
90	109.6	23.4	1.5	520	0.49	510	51
91	109.6	23.4	1.5	510	0.48	520	51
92	109.7	22.2	1.5	500	0.47	519	51
93	109.8	17.4	1.2	480	0.45	361	51
94	109.8	25.8	1.6	520	0.49	531	51
95	109.9	24.6	1.6	500	0.47	516	51
96	110.0	23.4	1.5	500	0.47	491	51
97	110.0	23.4	1.5	490	0.46	486	51
98	110.1	22.2	1.5	490	0.46	467	52
99	110.2	22.2	1.4	500	0.47	466	52
100	110.2	22.8	1.5	480	0.45	499	52
101	110.3	25.4	1.7	510	0.48	494	52
102	110.4	23.4	1.6	480	0.45	460	52
103	110.4	22.2	1.5	470	0.44	421	52
104	110.5	22.8	1.5	480	0.45	456	52
105	110.6	24.6	1.7	480	0.45	534	51
106	110.6	22.8	1.6	470	0.44	484	51
107	110.7	22.2	1.6	470	0.44	471	51
108	110.8	21.6	1.6	460	0.43	473	51

TABLE B.1. Continued

44.7661 252 x 71

6	1 TIT (°C)	2 FLOW (ml/min)	3 DP (PSI)	4 S (mm)	5 X/K ₀	6 P-P	7 TEMP °C
109	110.8	18.6	1.3	460	0.43	340	51
110	110.9	25.2	1.7	500	0.47	532	51
111	111.0	22.8	1.6	470	0.44	505	50
112	111.0	24.0	1.7	470	0.44	449	50
113	111.1	22.2	1.6	460	0.43	417	50
114	111.2	20.4	1.5	450	0.43	373	50
115	111.2	20.0	1.7	470	0.44	502	50
116	130.7	19.8	1.2	330	0.31	455	88
117	130.7	22.2	1.3	340	0.32	437	89
118	130.8	22.8	1.4	330	0.31	503	89
119	130.9	22.8	1.4	320	0.30	485	89
120	131.0	21.6	1.4	310	0.29	491	90
121	131.0	22.8	1.4	320	0.30	469	91
122	131.1	21.0	1.3	320	0.30	451	89
123	131.2	25.2	1.5	340	0.32	525	89
124	131.2	22.2	1.4	330	0.31	491	89
125	131.3	23.4	1.4	320	0.30	489	88
126	131.4	22.2	1.4	320	0.30	470	88
127	131.4	24.0	1.5	320	0.30	431	88
128	131.5	26.4	1.6	330	0.31	535	88
129	131.6	23.4	1.4	310	0.29	514	88
130	131.6	23.4	1.5	310	0.29	512	89
131	131.7	22.8	1.4	310	0.29	523	89
132	131.8	24.0	1.5	300	0.28	454	89
133	131.8	21.0	1.3	300	0.28	379	90
134	133.1	17.4	0.9	350	0.33	354	94
135	133.1	28.2	1.4	370	0.35	467	92
136	133.2	25.4	1.4	360	0.34	524	90
137	133.3	25.2	1.4	350	0.33	533	90
138	133.3	23.4	1.4	330	0.31	491	90
139	133.4	20.4	1.2	330	0.31	408	90
140	133.5	22.2	1.3	330	0.31	500	91
141	133.5	23.4	1.4	330	0.31	501	91
142	133.6	26.4	1.5	330	0.31	420	91
143	133.7	24.0	1.4	330	0.31	403	91
144	133.7	22.8	1.4	320	0.30	384	91
145	133.8	24.0	1.4	330	0.31	454	91
146	133.9	24.0	1.4	330	0.31	517	91
147	133.9	23.4	1.4	320	0.30	505	91
148	134.0	22.8	1.4	320	0.30	472	91
149	134.1	21.6	1.3	320	0.30	450	91
150	134.1	21.6	1.3	320	0.30	458	91
151	134.2	22.2	1.3	320	0.30	479	91
152	134.3	22.8	1.4	320	0.30	513	91
153	134.3	22.2	1.4	310	0.29	504	91
154	134.4	22.2	1.4	310	0.29	506	91
155	134.5	21.0	1.3	310	0.29	478	91
156	134.5	18.0	1.1	320	0.30	381	91
157	134.6	19.2	1.2	310	0.29	502	91
158	134.7	25.2	1.5	320	0.30	412	91
159	134.7	23.4	1.4	310	0.29	389	91
160	134.8	22.8	1.4	310	0.29	339	91
161	134.9	23.4	1.4	310	0.29	416	91
162	134.9	24.6	1.5	310	0.29	473	91

TABLE B.1. Continued

M57806 242 x 70

0	1 TIME (min)	2 FLOW (ml/min)	3 PC (PSI)	4 K (mm)	5 K/Ko	6 P-P	7 TEMP C
163	135.0	25.2	1.5	310	0.29	506	90
164	135.1	21.6	1.6	310	0.29	516	90
165	135.1	22.2	1.4	300	0.28	425	90
166	135.2	22.2	1.5	310	0.29	476	90
167	135.3	22.8	1.4	310	0.29	459	90
168	135.3	22.8	1.5	300	0.28	478	90
169	135.4	22.2	1.4	300	0.28	462	90
170	135.5	20.4	1.4	290	0.27	406	90
171	135.5	25.2	1.6	310	0.29	411	90
172	135.6	25.2	1.6	300	0.28	440	90
173	135.7	25.2	1.6	300	0.28	461	90
174	135.7	25.2	1.6	300	0.28	470	90
175	135.8	23.4	1.6	290	0.27	429	90
176	135.9	17.4	1.2	290	0.27	271	90
177	135.9	21.6	1.4	290	0.27	374	89
178	136.0	24.0	1.5	300	0.28	513	89
179	136.1	22.8	1.6	290	0.27	478	89
180	136.1	21.6	1.5	280	0.26	447	88
181	154.1	22.2	1.5	180	0.17	413	141
182	154.2	24.6	1.6	180	0.17	490	145
183	154.3	25.8	1.6	180	0.17	561	148
184	154.3	25.8	1.6	180	0.17	557	150
185	154.4	22.2	1.4	170	0.16	463	152
186	154.5	21.0	1.3	180	0.17	408	154
187	154.5	22.2	1.4	180	0.17	435	155
188	154.6	23.4	1.4	180	0.17	479	155
189	154.7	23.4	1.4	180	0.17	488	152
190	154.7	22.8	1.4	180	0.17	476	150
191	154.8	21.6	1.4	180	0.17	433	149
192	154.9	25.2	1.4	200	0.19	463	149
193	154.9	24.0	1.4	200	0.19	436	149
194	155.0	23.4	1.4	200	0.19	445	149
195	155.1	23.4	1.3	190	0.18	411	150
196	155.1	20.4	1.2	190	0.18	351	151
197	155.2	21.6	1.3	190	0.18	400	152
198	155.3	22.8	1.4	190	0.18	432	152
199	155.3	23.4	1.4	190	0.18	455	152
200	155.4	23.4	1.4	190	0.18	458	152
201	155.5	22.2	1.3	180	0.17	407	152
202	155.5	17.4	1.0	200	0.19	222	152
203	155.6	26.4	1.4	210	0.20	481	152
204	155.7	25.2	1.4	200	0.19	482	151
205	155.7	24.0	1.4	200	0.19	430	151
206	155.8	22.2	1.3	190	0.18	387	152
207	155.9	22.8	1.3	190	0.18	435	152
208	155.9	25.2	1.4	190	0.18	493	152
209	156.0	25.2	1.5	190	0.18	494	152
210	156.1	24.6	1.5	190	0.18	480	152
211	156.1	21.4	1.3	180	0.17	377	152
212	156.2	24.0	1.3	200	0.19	495	152
213	156.3	23.4	1.3	200	0.19	462	152
214	156.3	23.4	1.3	190	0.18	455	152
215	156.4	21.6	1.3	190	0.18	422	152
216	156.5	20.4	1.2	190	0.18	378	153

TABLE B.1. Continued

IN7840 259 2 70

0	1 TIME (HR)	2 FLOW (ml/min)	3 ΔP (PSI)	4 η (cP)	5 η/η_0	6 P-P	7 TEMP C
217	156.6	21.0	1.2	190	0.18	406	152
218	156.6	22.2	1.3	190	0.18	452	152
219	156.7	22.8	1.4	190	0.18	459	152
220	156.8	22.2	1.4	190	0.17	450	152
221	156.8	21.6	1.3	180	0.17	428	152
222	156.9	19.4	0.9	190	0.17	212	154
223	157.0	27.0	1.4	210	0.20	488	152
224	157.0	24.6	1.4	200	0.19	513	150
225	157.1	22.6	1.3	190	0.18	455	150
226	157.1	21.6	1.2	190	0.18	429	150
227	157.2	24.0	1.4	190	0.18	511	150
228	157.3	24.6	1.4	190	0.18	543	149
229	157.3	22.8	1.4	180	0.17	486	149
230	157.4	22.8	1.4	180	0.17	493	149
231	157.5	22.2	1.4	180	0.17	461	149
232	157.6	20.4	1.1	200	0.19	520	149
233	157.6	25.2	1.4	210	0.20	451	149
234	157.7	25.8	1.4	200	0.19	476	148
235	157.8	24.6	1.4	200	0.19	455	148
236	157.8	23.4	1.3	200	0.19	414	148
237	157.9	24.0	1.4	200	0.19	441	148
238	157.9	25.8	1.5	200	0.19	500	148
239	158.0	25.8	1.5	200	0.19	505	148
240	158.1	25.6	1.5	190	0.18	511	148
241	158.1	22.2	1.4	180	0.17	410	150
242	158.2	24.0	1.3	200	0.19	518	151
243	158.3	24.0	1.3	200	0.19	517	151
244	158.3	22.8	1.3	190	0.18	488	151
245	158.4	22.2	1.3	190	0.18	470	151
246	158.5	21.0	1.2	190	0.18	420	152
247	158.6	23.4	1.3	190	0.18	523	152
248	158.6	25.8	1.4	200	0.19	486	152
249	158.7	24.6	1.4	190	0.18	457	152
250	158.8	24.0	1.4	190	0.18	448	152
251	158.8	23.4	1.4	180	0.17	426	152
252	158.9	22.8	1.2	200	0.19	426	152
253	159.0	25.2	1.3	210	0.20	523	152
254	159.0	23.4	1.3	200	0.19	486	152
255	159.1	22.6	1.3	190	0.19	466	152
256	159.2	19.8	1.2	190	0.18	372	152
257	159.2	15.6	1.5	120	0.11	256	152
258	159.3	17.4	1.2	190	0.18	302	152
259	159.3	20.4	1.2	190	0.18	396	152

TABLE B.2. Laboratory Data for Test 3

MN785HAR 96R x 9C

0	1 TIME (HR)	2 SCANS	3 P-C (BAR)	4 P-E (BAR)	5 FLOW (ml/min)	6 DP (psf)	7 K (md)	8 K/Ko	9 TEMP C
1	0.4	54	66	40	24.5	2.8	753	0.92	20
2	1.4	65	61	36	24.6	2.6	803	0.98	20
3	2.1	40	62	35	24.6	2.8	760	0.93	20
4	2.6	65	60	34	24.6	2.7	763	0.93	20
5	3.1	66	59	34	24.6	2.6	800	0.97	20
6	3.7	53	62	37	24.6	3.0	689	0.84	20
7	4.3	59	63	40	24.6	2.6	815	0.99	20
8	4.9	65	62	36	24.6	2.9	723	0.88	20
9	5.2	5	56	33	24.6	2.7	785	0.96	20
10	5.5	57	62	37	24.6	2.9	724	0.88	20
11	6.1	67	65	39	24.6	2.9	715	0.87	20
12	6.4	6	62	39	24.7	2.5	821	1.00	20
13	6.7	60	63	38	24.6	2.9	709	0.86	20
14	7.2	66	65	41	24.6	3.0	699	0.85	20
15	7.7	25	65	42	24.7	2.8	742	0.90	20
16	11.0	1	60	36	0.0	0.0	0	0.00	20
17	12.6	1	63	40	0.0	0.0	0	0.00	20
18	13.0	1	61	38	0.0	0.0	0	0.00	20
19	14.0	1	64	41	0.0	0.0	0	0.00	20
20	15.0	1	67	45	0.0	0.0	0	0.00	20
21	16.0	1	67	44	0.0	0.0	0	0.00	20
22	17.0	1	61	38	0.0	0.0	0	0.00	20
23	18.0	1	60	37	0.0	0.0	0	0.00	20
24	19.0	1	63	41	0.0	0.0	0	0.00	20
25	20.0	1	57	35	0.0	0.0	0	0.00	20
26	21.0	1	67	45	0.0	0.0	0	0.00	20
27	22.0	1	64	45	0.0	0.0	0	0.00	20
28	23.0	1	59	38	0.0	0.0	0	0.00	20
29	23.8	7	67	41	24.6	3.1	663	0.81	21
30	26.1	6	62	37	25.0	1.8	376	0.46	91
31	26.6	14	65	40	25.1	1.6	417	0.51	92
32	27.2	14	61	35	25.0	1.5	437	0.53	92
33	27.9	15	62	36	25.0	1.5	427	0.52	91
34	28.0	9	67	41	25.0	1.7	390	0.47	91
35	29.0	7	73	46	25.0	1.5	424	0.52	91
36	29.6	9	66	40	25.1	1.6	400	0.49	92
37	30.2	7	66	38	25.1	1.6	399	0.49	92
38	30.7	9	60	32	25.0	1.6	398	0.49	92
39	31.3	9	62	35	25.0	1.7	397	0.48	91
40	31.6	1	59	35	0.0	0.0	0	0.00	90
41	32.6	1	71	48	0.0	0.0	0	0.00	91
42	33.6	1	79	57	0.0	0.0	0	0.00	91
43	34.6	1	72	51	0.0	0.0	0	0.00	91
44	35.6	1	67	46	0.0	0.0	0	0.00	91
45	36.6	1	62	41	0.0	0.0	0	0.00	91
46	37.6	1	69	48	0.0	0.0	0	0.00	91
47	38.6	1	65	43	0.0	0.0	0	0.00	91
48	39.6	1	73	52	0.0	0.0	0	0.00	91
49	40.6	1	69	48	0.0	0.0	0	0.00	91
50	41.6	1	76	55	0.0	0.0	0	0.00	91
51	42.6	1	72	51	0.0	0.0	0	0.00	91
52	43.6	1	76	55	0.0	0.0	0	0.00	91
53	44.6	1	77	57	0.0	0.0	0	0.00	91
54	45.6	1	64	44	0.0	0.0	0	0.00	91

TABLE B.2. Continued

MN765HAP 96R x 9C

0	1 TIME (HR)	2 SCANS	3 P-C (BAR)	4 P-E (BAR)	5 FLOW (ml/min)	6 DP (psi)	7 K (ml)	8 K/Ko	9 TEMP C
55	46.6	1	62	42	0.0	0.0	0	0.00	91
56	47.6	1	64	36	0.0	0.0	0	0.00	91
57	48.5	11	64	37	25.0	-1.5	450	0.55	91
58	49.1	6	67	42	25.0	-1.7	390	0.47	91
59	49.6	9	65	41	25.0	-1.5	451	0.55	91
60	50.2	11	71	44	25.0	-1.4	472	0.58	91
61	50.8	9	70	44	25.0	-1.6	412	0.50	91
62	51.3	9	73	48	25.0	-1.4	461	0.56	92
63	52.0	9	74	45	25.1	-1.4	479	0.58	92
64	52.5	10	66	38	25.1	-1.5	425	0.52	91
65	53.1	8	65	38	25.1	-1.4	465	0.57	92
66	53.7	9	67	40	25.1	-1.5	437	0.53	92
67	54.3	9	67	41	25.1	-1.4	463	0.56	92
68	54.9	11	68	39	25.0	-1.4	475	0.58	92
69	55.5	10	68	44	25.0	-1.5	442	0.54	92
70	55.9	1	43	20	0.0	0.0	0	0.00	91
71	56.8	1	94	61	0.0	0.0	0	0.00	92
72	57.8	1	90	69	0.0	0.0	0	0.00	91
73	58.8	1	81	59	0.0	0.0	0	0.00	91
74	59.8	1	95	73	0.0	0.0	0	0.00	91
75	60.8	1	90	68	0.0	0.0	0	0.00	91
76	61.8	1	95	74	0.0	0.0	0	0.00	91
77	62.8	1	77	56	0.0	0.0	0	0.00	91
78	63.8	1	36	65	0.0	0.0	0	0.00	91
79	64.8	1	77	56	0.0	0.0	0	0.00	91
80	65.8	1	75	54	0.0	0.0	0	0.00	91
81	66.8	1	64	44	0.0	0.0	0	0.00	91
82	67.8	1	64	43	0.0	0.0	0	0.00	91
83	68.8	1	68	47	0.0	0.0	0	0.00	91
84	69.8	1	70	50	0.0	0.0	0	0.00	91
85	70.8	1	79	59	0.0	0.0	0	0.00	91
86	71.8	3	65	41	25.1	-1.9	338	0.41	91
87	74.2	17	69	42	24.6	-4.3	492	0.60	19
88	74.8	18	66	41	24.6	-4.0	533	0.65	20
89	75.4	17	68	43	24.6	-4.2	500	0.61	20
90	76.0	15	69	44	24.6	-4.2	497	0.61	20
91	76.6	18	66	40	24.6	-4.0	514	0.63	21
92	77.2	17	67	41	24.6	-3.8	534	0.65	21
93	77.7	17	69	43	24.6	-4.1	497	0.61	21
94	78.3	18	70	48	24.6	-4.1	491	0.60	21
95	78.9	17	63	40	24.6	-4.1	489	0.60	21
96	79.5	17	65	42	24.6	-3.9	520	0.63	22

TABLE B.3. Laboratory Data for Test 4

MN785H8F 90R x 9C

0	1 TIME (HR)	2 SCANS	3 P-C (bar)	4 P-C (bar)	5 FLOW (ml/min)	6 DP (psi)	7 K (psi)	8 K/Ko	9 TEMP C
1	0.2	14	65	37	24.6	2.7	704	1.00	20
2	0.8	11	64	34	24.6	2.9	665	0.95	20
3	1.4	13	62	33	24.6	2.9	651	0.92	20
4	1.9	16	64	35	24.6	3.0	645	0.92	20
5	2.6	9	61	33	24.6	3.0	630	0.89	20
6	3.1	17	64	36	24.6	3.0	629	0.89	20
7	3.7	14	64	36	24.6	3.1	615	0.87	20
8	4.4	11	64	36	24.6	3.0	632	0.90	20
9	4.9	13	63	36	24.6	3.0	623	0.89	20
10	5.4	13	66	40	24.6	3.0	632	0.90	20
11	6.1	13	68	41	24.6	3.1	611	0.87	20
12	6.6	17	66	39	24.6	3.1	613	0.87	20
13	7.1	1	66	39	24.6	3.2	597	0.85	20
14	11.4	1	70	46	0.0	0.0	0	0.00	20
15	12.4	1	71	47	0.0	0.0	0	0.00	21
16	13.4	1	68	45	0.0	0.0	0	0.00	21
17	14.4	1	65	41	0.0	0.0	0	0.00	21
18	15.4	1	70	47	0.0	0.0	0	0.00	20
19	16.4	1	65	42	0.0	0.0	0	0.00	20
20	17.4	1	64	42	0.0	0.0	0	0.00	20
21	18.4	1	64	42	0.0	0.0	0	0.00	20
22	19.4	1	67	46	0.0	0.0	0	0.00	20
23	20.4	1	65	44	0.0	0.0	0	0.00	20
24	21.4	1	65	44	0.0	0.0	0	0.00	20
25	22.4	1	64	44	0.0	0.0	0	0.00	20
26	24.1	1	61	41	0.0	0.0	0	0.00	20
27	23.6	10	64	36	24.6	3.3	579	0.82	20
28	27.6	13	63	38	25.8	1.5	280	0.40	134
29	28.4	5	64	39	25.9	1.3	316	0.45	136
30	28.9	14	66	41	25.9	1.4	300	0.43	137
31	29.5	10	64	38	25.8	1.4	295	0.42	134
32	30.1	15	60	38	25.8	1.2	340	0.48	135
33	30.7	10	61	40	25.9	1.2	336	0.48	135
34	33.6	1	69	48	0.0	0.0	0	0.00	126
35	34.6	1	70	48	0.0	0.0	0	0.00	127
36	35.6	1	64	42	0.0	0.0	0	0.00	127
37	36.6	1	64	42	0.0	0.0	0	0.00	127
38	37.6	1	68	47	0.0	0.0	0	0.00	127
39	38.6	1	73	51	0.0	0.0	0	0.00	128
40	39.6	1	63	42	0.0	0.0	0	0.00	128
41	40.6	1	65	44	0.0	0.0	0	0.00	128
42	41.6	1	65	44	0.0	0.0	0	0.00	128
43	42.6	1	61	40	0.0	0.0	0	0.00	128
44	43.6	1	76	55	0.0	0.0	0	0.00	128
45	44.6	1	60	40	0.0	0.0	0	0.00	128
46	45.6	1	67	46	0.0	0.0	0	0.00	128
47	46.6	1	68	48	0.0	0.0	0	0.00	128
48	47.2	1	68	48	0.0	0.0	0	0.00	128
49	48.0	4	64	40	25.9	1.1	355	0.50	138
50	48.3	4	65	43	25.8	-1.4	293	0.42	132
51	48.7	13	73	48	25.6	-1.6	273	0.39	125
52	49.3	13	75	51	25.7	-1.6	264	0.38	127
53	49.9	13	74	50	25.7	-1.6	275	0.39	127
54	50.5	11	76	52	25.7	-1.6	267	0.38	127

TABLE B.3. Continued

MN785HR 90R x 90

0	1 TIME (HR)	2 SCANS	3 P=C (bar)	4 P=C (bar)	5 FLOW (ml/min)	6 DP (psi)	7 K (md)	8 K/Ko	9 TEMP C
55	51.1	14	65	41	25.7	-1.6	279	0.40	127
56	51.7	11	65	41	25.7	-1.5	281	0.40	127
57	52.3	10	71	47	25.7	-1.5	284	0.40	127
58	52.6	11	64	39	25.7	-1.5	287	0.41	127
59	53.4	11	61	36	25.7	-2.0	221	0.31	127
60	54.0	13	66	42	25.7	-2.1	211	0.30	127
61	54.6	14	69	45	25.7	-2.0	221	0.31	127
62	55.1	9	69	46	25.7	-1.8	243	0.34	128
63	55.5	1	66	33	0.0	0.0	0	0.00	128
64	56.5	1	69	43	0.0	0.0	0	0.00	128
65	57.5	1	64	39	0.0	0.0	0	0.00	128
66	58.5	1	62	38	0.0	0.0	0	0.00	128
67	59.5	1	60	37	0.0	0.0	0	0.00	128
68	60.5	1	69	46	0.0	0.0	0	0.00	128
69	61.5	1	71	49	0.0	0.0	0	0.00	129
70	62.5	1	68	46	0.0	0.0	0	0.00	128
71	63.5	1	69	48	0.0	0.0	0	0.00	128
72	64.5	1	71	50	0.0	0.0	0	0.00	128
73	65.5	1	64	44	0.0	0.0	0	0.00	128
74	66.5	1	70	49	0.0	0.0	0	0.00	128
75	67.5	1	64	44	0.0	0.0	0	0.00	128
76	68.5	1	71	51	0.0	0.0	0	0.00	128
77	69.5	1	70	51	0.0	0.0	0	0.00	128
78	70.5	1	72	50	0.0	0.0	0	0.00	130
79	71.1	1	63	43	0.0	0.0	0	0.00	129
80	72.2	12	63	37	25.6	-2.5	183	0.26	123
81	75.2	8	70	40	24.6	-5.4	345	0.49	21
82	75.4	7	69	40	24.6	-5.2	360	0.51	21
83	75.7	7	61	37	24.6	-5.7	329	0.47	21
84	76.3	11	70	46	24.6	-5.6	333	0.47	21
85	76.8	14	70	45	24.6	-5.6	330	0.47	22
86	77.5	9	62	37	24.6	-5.2	358	0.51	21
87	78.0	12	70	44	24.6	-5.5	334	0.48	21
88	78.6	10	71	44	24.6	-5.5	332	0.47	22
89	78.9	7	72	47	24.6	-5.2	351	0.50	22
90	79.2	4	71	43	24.6	-5.2	351	0.50	22

TABLE B.4. Laboratory Data for Test 5

MN785HCR 92R x 9C

0	1 TIME (HR)	2 SCANS	3 P-C (BAR)	4 P-E (BAR)	5 FLOW (ml/min)	6 DP (psi)	7 K (md)	8 K/Ko	9 TEMP C
1	0.3	17	75	49	24.6	3.0	583	0.86	20
2	0.8	17	67	40	23.6	3.1	546	0.81	20
3	1.5	18	66	38	24.6	2.6	674	1.00	20
4	2.1	17	67	35	24.6	3.0	591	0.88	20
5	2.6	17	65	34	24.6	3.0	581	0.86	20
6	3.2	18	66	34	24.6	3.1	554	0.82	20
7	3.8	17	70	38	24.6	3.1	554	0.82	20
8	4.4	18	71	38	24.6	3.3	532	0.79	20
9	4.9	11	72	41	24.6	3.2	545	0.81	20
10	10.3	1	66	38	0.0	0.0	0	0.00	20
11	11.3	1	73	46	0.0	0.0	0	0.00	20
12	12.3	1	72	45	0.0	0.0	0	0.00	20
13	13.3	1	73	46	0.0	0.0	0	0.00	20
14	14.3	1	74	48	0.0	0.0	0	0.00	20
15	15.3	1	69	43	0.0	0.0	0	0.00	20
16	16.3	1	71	44	0.0	0.0	0	0.00	20
17	17.3	1	71	44	0.0	0.0	0	0.00	20
18	18.3	1	69	43	0.0	0.0	0	0.00	20
19	19.3	1	65	39	0.0	0.0	0	0.00	20
20	20.3	1	64	38	0.0	0.0	0	0.00	20
21	21.1	1	71	45	0.0	0.0	0	0.00	20
22	21.5	6	66	39	24.6	2.9	589	0.87	21
23	25.0	11	74	45	26.1	1.2	290	0.43	146
24	25.3	4	72	44	26.2	1.1	309	0.46	148
25	25.7	18	68	41	26.2	1.0	325	0.48	150
26	26.3	17	70	41	26.2	1.1	313	0.46	150
27	26.9	18	68	40	26.2	1.0	325	0.48	150
28	27.2	5	72	44	26.2	1.0	332	0.49	150
29	27.6	10	70	42	26.2	1.0	337	0.50	150
30	29.0	17	68	40	26.2	1.0	343	0.51	151
31	29.6	18	69	40	26.2	1.0	328	0.49	151
32	29.0	9	74	46	26.3	1.0	342	0.51	151
33	29.3	6	72	45	26.2	1.0	345	0.51	151
34	29.6	1	73	41	0.0	0.0	0	0.00	154
35	30.6	1	64	36	0.0	0.0	0	0.00	151
36	31.6	1	59	31	0.0	0.0	0	0.00	151
37	32.6	1	64	36	0.0	0.0	0	0.00	151
38	33.6	1	68	40	0.0	0.0	0	0.00	151
39	34.6	1	63	35	0.0	0.0	0	0.00	151
40	35.6	1	54	27	0.0	0.0	0	0.00	151
41	36.6	1	70	42	0.0	0.0	0	0.00	151
42	37.6	1	56	29	0.0	0.0	0	0.00	151
43	38.6	1	58	32	0.0	0.0	0	0.00	151
44	39.6	1	65	38	0.0	0.0	0	0.00	151
45	40.6	1	66	39	0.0	0.0	0	0.00	151
46	41.6	1	65	39	0.0	0.0	0	0.00	151
47	42.6	1	71	45	0.0	0.0	0	0.00	151
48	43.6	1	74	48	0.0	0.0	0	0.00	151
49	44.6	1	71	45	0.0	0.0	0	0.00	152
50	45.6	9	72	44	26.1	0.7	485	0.72	147
51	45.9	7	74	46	26.2	0.9	369	0.55	150
52	46.2	4	71	43	26.3	-0.7	465	0.69	154
53	46.4	8	66	37	26.3	-0.9	387	0.57	152
54	46.9	18	63	32	26.2	-1.1	311	0.46	151

TABLE B.4. Continued

MN785HCR 92R x 9C

0	1 TIME (HR)	2 SCANS	3 P-C (BAR)	4 P-E (BAR)	5 FLOW (ml/min)	6 DP (psi)	7 K (mg)	8 K/Ko	9 TEMP C
55	47.5	17	64	34	26.2	-2.1	158	0.24	150
56	48.0	17	64	33	26.2	-3.7	92	0.14	150
57	48.6	18	63	32	26.2	-4.4	76	0.11	151
58	49.1	10	66	35	26.2	-4.0	85	0.13	151
59	49.4	5	65	35	26.2	-1.4	248	0.37	150
60	49.8	17	67	36	26.2	-1.5	223	0.33	151
61	50.4	18	69	36	26.2	-1.7	200	0.30	151
62	51.0	17	67	35	26.2	-1.8	186	0.28	151
63	51.5	17	72	39	26.2	-1.9	179	0.26	151
64	52.1	18	71	39	26.2	-1.9	174	0.26	151
65	52.7	16	72	39	26.2	-2.0	171	0.25	152
66	53.3	14	69	39	26.2	-1.5	226	0.34	151
67	53.5	1	72	39	0.0	0.0	0	0.00	152
68	54.5	1	61	34	0.0	0.0	0	0.00	153
69	55.5	1	66	39	0.0	0.0	0	0.00	153
70	56.5	1	64	37	0.0	0.0	0	0.00	153
71	57.5	1	65	39	0.0	0.0	0	0.00	153
72	58.5	1	68	41	0.0	0.0	0	0.00	153
73	59.5	1	69	43	0.0	0.0	0	0.00	153
74	60.5	1	68	42	0.0	0.0	0	0.00	153
75	61.5	1	67	41	0.0	0.0	0	0.00	153
76	62.5	1	70	44	0.0	0.0	0	0.00	153
77	63.5	1	64	44	0.0	0.0	0	0.00	153
78	64.5	1	68	43	0.0	0.0	0	0.00	153
79	65.5	1	69	43	0.0	0.0	0	0.00	153
80	66.5	1	70	45	0.0	0.0	0	0.00	153
81	67.5	1	68	43	0.0	0.0	0	0.00	153
82	68.5	1	69	45	0.0	0.0	0	0.00	153
83	69.9	11	66	37	26.2	-5.3	63	0.09	151
84	73.3	15	60	27	24.6	-12.6	130	0.19	23
85	73.6	17	50	12	24.6	-12.0	148	0.22	19
86	74.5	13	72	44	24.6	-14.2	122	0.18	20
87	74.9	10	61	35	24.6	-13.2	128	0.19	21
88	75.5	8	65	39	24.6	-10.6	158	0.23	22
89	75.8	8	69	38	24.6	5.9	281	0.42	22
90	76.2	17	71	39	24.6	6.0	274	0.41	22
91	76.8	17	64	33	24.6	5.9	276	0.41	23
92	77.3	6	61	31	24.6	4.4	363	0.54	23

TABLE B.5. Laboratory Data for Test 6

MN775HAR 46R x 9C

0	1 TIME (HR)	2 SCANS	3 P-C (BAR)	4 P-E (BAR)	5 FLOW (ml/min)	6 DP (psi)	7 K (md)	8 K/K0	9 TEMP C
1	0.1	6	74	47	23.7	9.1	267	1.00	20
2	0.3	5	76	54	23.7	9.0	270	1.00	20
3	0.7	17	76	54	23.7	9.5	255	0.89	20
4	1.3	17	76	54	23.7	9.9	245	0.83	20
5	1.8	17	77	56	23.7	9.7	248	0.85	20
6	2.2	7	76	56	22.9	9.4	247	0.84	20
7	4.5	10	78	50	23.7	9.2	261	0.91	20
8	5.1	9	78	54	23.7	9.0	267	0.90	20
9	5.5	17	79	59	23.7	8.7	274	0.86	20
10	5.1	17	82	61	23.7	8.9	269	0.85	20
11	5.6	13	83	61	23.7	8.9	268	0.84	20
12	7.2	11	84	60	23.3	8.4	278	0.84	21
13	24.0	17	80	59	23.7	8.8	271	0.86	20
14	24.5	17	80	59	23.7	9.0	266	0.83	20
15	30.3	14	79	57	23.7	8.7	165	0.52	85
16	31.1	8	75	55	23.7	4.3	175	0.55	88
17	33.6	12	77	55	23.7	-3.2	242	0.80	89
18	48.1	17	76	55	23.7	-3.2	238	0.80	93
19	48.7	13	74	53	23.7	-3.2	237	0.79	92
20	49.3	17	70	48	23.7	-3.1	239	0.80	92
21	49.9	17	71	49	23.7	-3.2	236	0.79	93
22	50.5	15	70	49	23.7	-2.9	263	0.78	93
23	51.0	11	69	47	23.7	-2.9	269	0.83	93
24	51.9	9	65	43	23.7	-3.3	243	0.86	94
25	52.4	17	67	46	23.7	-3.2	252	0.84	93
26	53.0	17	70	47	23.7	-3.1	259	0.83	94
27	53.5	18	67	45	23.7	-3.4	239	0.81	93
28	54.1	17	66	42	23.7	-3.1	262	0.83	94
29	54.5	7	63	36	23.7	-3.0	271	0.85	94
30	54.9	8	66	36	23.7	-3.1	275	0.87	94
31	55.1	7	66	43	23.7	-3.4	249	0.84	93
32	71.6	6	78	55	23.7	-8.0	279	0.90	23
33	72.0	17	77	55	23.7	-8.8	257	0.87	23
34	72.6	18	75	53	23.7	-8.8	257	0.84	23
35	73.2	17	75	51	23.7	-9.0	253	0.84	22
36	73.7	15	72	46	23.7	-8.9	256	0.83	22
37	74.3	17	73	54	23.7	-9.7	235	0.82	22
38	75.0	16	73	53	23.7	-8.7	261	0.82	22
39	75.4	12	71	50	23.7	-9.2	249	0.82	22
40	75.8	5	73	53	23.7	9.2	249	0.75	22
41	76.1	17	71	51	23.7	10.2	224	0.74	22
42	76.7	17	72	50	23.7	10.6	216	0.72	22
43	77.3	17	72	50	23.7	10.5	216	0.71	22
44	77.9	18	71	49	23.7	10.7	213	0.71	22
45	78.5	17	72	51	23.7	10.0	228	0.71	22
46	78.8	5	72	51	23.7	9.9	230	0.71	22

TABLE B.6. Laboratory Test Data for Test 7

M4775H3R 48R x 9C

0	1 TIME (HR)	2 SCANS	3 P-C (R/R)	4 P-E (R/R)	5 FLOW (ml/min)	6 DP (psi)	7 K (md)	8 K/Ks	9 TEMP C
1	0.2	12	63	31	23.7	5.3	404	0.97	20
2	0.5	4	71	39	23.7	5.1	417	1.00	20
3	0.4	3	71	40	23.7	5.9	362	0.87	20
4	1.3	17	70	41	23.7	5.8	372	0.89	20
5	1.9	14	69	37	23.7	6.0	359	0.86	20
6	2.5	15	67	35	23.7	6.0	354	0.85	20
7	2.8	5	71	29	23.7	6.0	356	0.85	20
8	3.1	10	63	36	23.7	5.6	382	0.91	20
9	7.0	14	64	37	23.7	5.9	359	0.86	21
10	7.3	5	66	39	23.7	5.8	363	0.87	21
11	7.6	10	66	39	23.7	5.6	374	0.90	21
12	23.8	4	64	39	23.7	5.3	394	0.94	21
13	24.1	11	73	46	23.7	5.3	387	0.93	22
14	27.7	5	62	44	23.9	3.6	140	0.33	126
15	28.1	17	67	43	23.7	3.1	163	0.39	126
16	28.4	13	70	45	23.7	2.9	177	0.42	122
17	29.3	17	71	45	23.7	2.9	175	0.42	123
18	30.0	11	64	37	23.7	3.1	160	0.38	124
19	30.5	17	67	43	23.7	2.9	170	0.41	129
20	31.1	17	70	44	23.7	3.1	163	0.39	125
21	31.4	5	64	39	23.7	3.2	163	0.39	122
22	31.6	4	62	37	23.7	2.8	186	0.44	121
23	47.9	3	53	57	23.7	2.7	193	0.46	120
24	48.1	3	70	44	23.7	3.0	176	0.42	121
25	48.4	3	73	45	23.7	3.6	143	0.34	124
26	48.6	5	73	46	23.7	-2.0	242	0.58	131
27	49.0	5	65	42	23.7	-2.3	207	0.50	133
28	49.4	17	64	43	23.7	-2.0	241	0.58	129
29	50.0	10	68	47	23.7	-2.0	248	0.59	125
30	50.7	11	70	45	23.7	-2.3	210	0.50	129
31	51.2	18	64	44	23.7	-2.1	231	0.50	127
32	51.7	17	67	42	23.7	-2.1	232	0.55	128
33	52.3	15	65	40	23.7	-2.2	226	0.56	128
34	53.1	7	64	36	23.7	-1.8	269	0.54	124
35	53.5	12	69	39	23.7	-1.9	262	0.64	127
36	53.7	5	67	42	23.7	-2.5	204	0.63	126
37	54.1	17	66	44	23.7	-2.2	225	0.49	126
38	54.7	17	66	43	23.7	-2.3	218	0.54	126
39	55.2	13	65	43	23.7	-2.4	207	0.52	126
40	71.7	11	73	42	23.7	-2.8	188	0.45	126
41	71.9	7	75	48	23.7	-2.9	179	0.45	123
42	72.2	6	73	50	23.8	-2.8	182	0.43	125
43	72.4	11	71	45	23.7	-3.4	148	0.44	127
44	76.8	6	71	45	23.7	-97.0	24	0.36	18
45	77.4	10	63	50	11.6	-83.5	13	0.06	18
46	78.3	30	79	55	5.9	-63.8	8	0.03	20
47	79.2	21	77	52	5.9	-66.6	8	0.02	21

TABLE B.7. Laboratory Test Data for Test 8

MN775MCF 45R x 9C

0	1 TIME (hr)	2 SCANS	3 P-C (bar)	4 P-E (bar)	5 FLOW (ml/min)	6 DP (psf)	7 K (md)	8 K/Ko	9 TEMP C
1	0.3	13	62	37	23.7	4.1	415	1.00	21
2	0.9	17	70	55	23.7	4.8	356	0.86	21
3	1.4	17	71	39	23.7	5.0	342	0.82	21
4	2.0	17	74	51	23.7	5.4	320	0.77	21
5	2.5	10	76	53	23.7	5.5	315	0.76	21
6	2.8	5	70	44	23.7	4.4	389	0.94	21
7	19.2	10	65	43	23.7	6.0	281	0.68	22
8	23.1	17	71	43	23.7	3.1	112	0.27	141
9	23.7	17	67	43	23.7	3.1	109	0.26	143
10	24.3	19	67	44	23.7	2.3	151	0.36	143
11	24.8	11	70	44	23.7	2.3	146	0.35	145
12	25.3	5	59	47	23.7	4.5	75	0.18	143
13	25.6	7	63	49	23.8	2.9	117	0.28	145
14	26.1	13	63	39	23.7	2.8	119	0.29	146
15	26.4	7	62	33	23.7	2.7	123	0.30	146
16	26.6	6	69	41	23.9	2.6	130	0.31	146
17	43.6	8	67	41	23.9	3.6	95	0.23	143
18	44.1	3	77	51	23.8	6.2	55	0.13	144
19	44.6	14	64	42	23.7	6.1	55	0.13	145
20	45.0	4	65	32	23.7	5.0	67	0.16	146
21	45.3	6	66	40	23.8	5.7	59	0.14	145
22	45.7	17	68	43	23.7	5.3	63	0.15	145
23	46.3	9	71	32	23.7	2.8	121	0.29	148
24	46.9	17	64	40	23.7	-2.4	142	0.34	149
25	47.4	9	69	45	23.7	-2.1	158	0.38	149
26	48.1	18	73	46	23.7	-2.1	163	0.39	147
27	48.7	17	74	48	23.7	-2.0	169	0.41	148
28	49.3	17	75	48	23.7	-2.1	162	0.39	147
29	49.9	17	78	53	23.7	-2.1	163	0.39	147
30	50.4	15	73	48	23.7	-2.1	162	0.39	148
31	57.6	7	66	43	23.9	-3.6	96	0.23	151
32	58.0	16	66	45	23.7	-3.8	90	0.22	151
33	68.6	17	69	48	23.7	-3.2	104	0.25	152
34	69.2	16	67	44	23.7	-3.2	106	0.26	153
35	69.8	18	67	43	23.7	-3.1	107	0.26	154
36	70.5	12	65	41	23.7	-2.9	115	0.28	154
37	71.0	17	66	42	23.7	-2.9	114	0.28	154
38	71.5	17	64	39	23.7	-3.0	110	0.26	154
39	72.1	17	67	40	23.7	-3.1	108	0.26	154
40	72.7	17	63	35	23.7	-3.2	104	0.28	154
41	73.3	17	60	31	23.7	-3.1	107	0.25	155
42	73.9	17	62	30	23.7	-3.5	97	0.26	154
43	74.4	14	64	37	23.7	-3.3	101	0.23	155
44	97.9	17	68	43	23.7	-16.9	95	0.24	25
45	98.4	17	68	42	23.7	-16.1	98	0.23	20

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