Iceland Deep Drilling Project: Testing of Reykjanes Brine and Steam in High Pressure Vessel - Experimental Plan and Vessel Specification

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Keywords: Reykjanes, hot deep brine system, hp-vessel, at site experiments.

ABSTRACT

Drilling of a 5 km well in the geothermal field in Reykjanes, Iceland is under preparation. The brine at this depth is believed to be of seawater salinity as in the system above. By testing the steam and brine tapped directly from the Reykjanes wells in a high-pressure vessel heated to expected deep reservoir temperature, information will be gained about chemical properties of the fluids in the deep system and also about corrosiveness of the fluids. The vessel will be charged with brine and steam from the Reykjanes power plant in predetermined ratios and also with solids representing the rock composition in the deep system. Then the vessel will be heated to reach experimental conditions of 400 to 500 °C at a pressure of 300 bar. The phases will be liquid brine, dry superheated steam or a mixture of brine and steam, depending on vessel temperature. Samples will be collected of liquid and vapour phase for analysis. By controlling the pressure and temperature in the vessel, the chemical equilibrium between gas, steam, brine and rock can be studied as well as the solution and dissolution of minerals in discharging well. The high experimental pressure and temperature will possess great demands on the vessel design. Special sampling procedures must be adopted in order to get representative samples of both steam and brine. The design and building of the experimental equipment is described as well the experimental procedures.

1. INTRODUCTION

Drilling of deep well in the geothermal field in Reykjanes, Iceland is under preparation. The aim is to drill to the depth of 5 km. The first deep well in the Icelandic Deep Drilling (Fröblöffsson et al., 2014) was drilled in Krafla in 2009 (Elders et al., 2014). It produced superheated steam at wellhead pressure of 140 bar and temperature of 450 °C. The superheated steam contained acid gas and was highly corrosive when it condensed. It also contained gaseous sulfur and dissolved silica making it unsuitable for utilization without scrubbing (Hauksson et al., 2014).

A 100 MW power plant is operated in the Reykjanes field tapping steam and brine from wells drilled to depth of 2500 to 3000 m. The brine in the Reykjanes field is of seawater salinity (3.3 wt%) and reservoir temperature is in the range of 300 to 350 °C. The brine at 5 km depth is believed to be of seawater salinity as in the system above. The temperature is very uncertain and temperature in the range of 380 to 640 °C can be expected.

The fluid property at the expected temperature is not well known. Some experiments have been done at very high temperature using artificial fluids. Based on these experiments solubility of minerals such as silica in hot water and steam can be predicted (Fournier and Potter, 1982). The volatility of HCl from brine to steam has also been studied in autoclave experiments by several scientists (Fournier and Thompson, 1993, Simonson and Palmer, 1993, Simonson et al., 1994, Bischoff et al., 1996 to name few). Despite of this research some uncertainty still exists about the behavior of geothermal brine and steam at the very high temperatures in the deep Reykjanes reservoir and in the discharging IDDP-2 well. It was therefore proposed that high temperature experiments using brine and steam tapped directly from the Reykjanes wells would give valuable information on what to expect when the IDDP-2 well will discharge. Similar experiments were done in 2002 and 2003 with brine from the exploratory wells although at lower pressure and temperature than can be expected from the IDDP-2 well. Precipitation of silica and metal silicates were studied in a high-pressure autoclave (Hauksson and Jörhällsson, 2003a and b). These experiments gave valuable information for the design of the Reykjanes power plant.

Brine and steam from the Reykjanes power plant will be heated in a vessel to the pressure and temperature to be expected in the reservoir and well at 5 km. The vessel will also be charged with solids representing the rock composition in the deep system. By controlling the pressure and temperature in the vessel the chemical properties of three different phase relations possible in the system can be studied. By this approach important information will be gained about chemical equilibrium between steam, brine and rock and also about corrosiveness of the fluids.

By building and operating such a vessel experience will be gained that may be useful for the design of the IDDP-2 wellbeing and fluid handling equipment and in developing the best approach for sampling of steam and brine from the discharging well.

To help us understand better the properties of steam and brine at the high temperature in the deep Reykjanes system several experiments are of interest. These include the study of the dissolution of minerals in brine and the partitioning of chemical constituents between brine and steam and also the corrosion of metals in brine and steam. The possible experimental combinations are endless and an effort must be made to focus on the conditions that are most interesting for the technical completion of the IDDP-2 project.

At high pressure and temperature the chemical constituents dissolved in brine will also dissolve to some extent in the steam. For silica this has been well demonstrated by experiments and correlations are available to predict the solubility of silica in brine and steam at high pressure and temperature (Fournier and Potter, 1982). Also the formation of HCl by reaction of silica with sodium
chloride at high pressure and temperature has been investigated (Fournier and Thompson, 1993) as well as the partitioning of some chlorides between brine and steam (Simonson et al., 1994, Simonson et al., 1997). For most chemicals the partition equilibrium is not well known.

It is very important for the IDDP-2 project to be able to predict whether the steam entering the well is likely to contain high concentration of silica or corrosive acid and how other constituents in the brine will affect this. Equilibrating unaltered basalt or other rock types with the Reykjanes brine in the liquid phase zone and then withdrawing samples can simulate the composition of fluids supposed to feed the IDDP-2 well. By subsequent lowering of vessel pressure the solution and dissolution of minerals in a discharging well can be studied.

2. CHARGING OF VESSEL.

At depth of 5 km the predicted pressure is in the range of 300 to 320 bar and the temperature in the range of 380 to 640 °C (Ómar Sigurðsson comm. pers.) At this pressure the phases will be liquid brine, dry superheated steam or mixture of brine and steam, depending on reservoir temperature. This is shown in the following PTx diagram showing the phase-properties of brine and steam as function of pressure and temperature. The diagram is based on correlations for pressure-temperature relationship of concentrated brine (Atkinson, 2002).

Three experimental points (A, B and C) are shown on the diagram representing the three principally different phase conditions. In point A (300 bar, 400°C) the pressure is sufficient to keep the fluid at liquid phase conditions. In point B (300 bar, 450°C) the vessel will contain superheated steam in equilibrium with brine of 33% salinity. In point C (300 bar, 500 °C) the vessel will contain superheated steam only. The charging of the vessel will be different for each condition. The conditions in Reykjanes wells at 2500 to 3000 m depth (REY) and in the Krafla IDDP-1 well are shown in the diagram. The critical point for water and for 3.3% brine is shown as CP and CP 3.3% respectively.

The Reykjanes plant separators are operated at 210 °C flashing temperature. The salinity is 3.3% in the reservoir brine but at flashing temperature the salinity is 4.1%.

The concept is to capture the brine and steam from the plant and heat up to reservoir temperature in a high-pressure vessel. There will be no pumps or flexible bags used and the pressure will be generated by expansion of the brine and steam in a constant volume vessel. By charging the vessel with brine and steam in the correct ratio and heating the vessel the target pressure and temperature conditions will be reached. Additionally the vessel will be charged with pieces of rock or specimens for material testing. The following figure shows the connections for charging the vessel with brine and steam from the power plant.
The bottom valve will be connected to the brine line from the high-pressure separator. The Bottom inlet line will also be connected to a condensate line so that dilution of brine inflow will be possible if required. The top valve will be connected to the steam line from the high-pressure separator. It will be possible to condense out the steam and get steam with enriched gas ratio if required. Flush lines will be connected to both bottom and top inlets allowing flushing with brine from below and steam from above. The flush lines will be connected to discharge. The flush lines will also be connected to a cooler allowing sampling of the filling fluids and measuring of filling volume.

The connection lines and valves will be outside the high pressure and temperature zone during the experiments and special high temperature and pressure valves are not needed for these lines.

For liquid phase experiments (zone A in figure 1) the vessel will first be charged with brine mixed with condensate so that in the end the vessel will contain brine close to the deep reservoir composition (3.3% salinity). When the vessel has been filled and sufficiently flushed it will be pressurized through the top connection with gas enriched steam and a small but exactly measured amount of brine tapped off until the vessel contains the correct steam-brine ratio. The steam sampling port will be flushed briefly with condensate to avoid contamination of sampling line.

The vessel will then be closed and heated slowly to experimental temperature. Upon heating, the brine will evaporate and expand, causing a pressure increase. At the same time the salinity of the brine will increase reducing the corresponding steam pressure.

At approximately 400 °C the vessel will be almost full of brine with very little headspace left. The pressure will be that of the steam pressure for 3.3% brine at the vessel temperature of 400 °C. The heating curve for zone A experiments is shown on figure 3 as a broken line.
The experimental conditions will be kept at 400 °C until equilibrium has been reached. Samples of steam and brine will be collected and the temperature lowered until the steam pressure becomes 250 bar. After new equilibrium time another sample of brine and steam can be withdrawn. By stepwise lowering of temperature different experimental conditions can be obtained and the minerals solubility and partitioning between brine and steam in the discharging well can be investigated in one experiment.

To reach experimental condition in zone B the vessel will be first filled with plant brine (4.1%) and then heated with top flush valve open until 75% of the brine has evaporated and the brine salinity has increased to 15%. Then the vessel will be pressurized with gas-enriched steam and closed. By heating to 450 °C the target pressure of 300 bar can be reached. This is shown in figure 4 (for zone B experiments) as a broken line.

The pressure and temperature in the vessel was calculated using correlations of Driesner for specific volume of steam and brine and Atkinson for the pressure and temperature relationship. The calculations are uncertain and exact target pressure may be difficult to reach and final adjusting must be left to experimentation.

Figure 3: Charging and heating to 400 °C and 282 bar (zone A).

Figure 4: Charging and heating to 450 °C and 300 bar (zone B).
When samples of steam are collected from the vessel the pressure will decrease. The vessel will then be cooled until the pressure reaches 250, 200 and 150 bar and new samples collected.

To reach condition in zone C all the brine in the vessel must be evaporated by heating. First the vessel will be filled with brine and then the brine will be driven out as steam until only 15% of the brine remains in the vessel. By this steam-brine ratio the target pressure of 300 bar can be reached at 500 °C. This is shown in the figure 5 (for zone C experiments) as a broken line. At this pressure and temperature the vessel will contain only superheated steam and salt (zone C).

**Figure 5: Charging and heating to 500 °C and 300 bar (zone C).**

When samples of steam are collected from the vessel the pressure will decrease and new equilibrium between salt and steam will be reached and solubility of various minerals in superheated steam can be studied at different pressures.

### 2. EXPERIMENTAL PLAN

The experiments will be time consuming and put strain on the equipment. Therefore a limited number of experiments can be performed. Following is a tentative experimental plan showing experiments, which are of most interests for the IDDP-2 project.

First partitioning between steam and brine will be studied. For these studies the vessel need not to be opened between experiments. Also equilibrium time should be relatively short (hours). When the partitioning chemistry has been mapped the solubility of minerals will be studied. Then the vessel will be charged with rock specimens and equilibrium of minerals examined. These experiments will take a longer time (days) and the vessel must be opened between experiments to renew the rock specimens. Finally specimens of different metals will be kept in the vessel for studying the corrosiveness of brine and steam. These experiments will take a relatively longer time (weeks).

#### 2.1 Partitioning

Partitioning in flashing brine will first be studied without any minerals in vessel. The brine will be equilibrated at 400 °C and corresponding steam pressure, starting from liquid phase (zone A). While controlling temperature, samples will be repeatedly collected to study the rate of equilibrium. Then the vessel will be recharged and the experiment repeated but with changing pressure.

Partitioning between concentrated brine and steam, starting from steam and concentrated brine (zone B) will be done without any minerals in vessel. The brine will be equilibrated and brine and steam sample collected at equilibrium conditions. After sampling the vessel will be cooled until the pressure reaches lower pressure and new samples collected after new equilibrium.

Partitioning between salt and superheated steam (zone C). The brine will be evaporated and the steam will be equilibrated with salt and minerals precipitated from the brine. Steam samples will be collected at equilibrium conditions and then steam released until pressure equilibrates to the lower pressure for new sampling.

#### 2.2 Mineral equilibrium

The vessel will be charged with rock specimen (e.g. unaltered basalt) and filled with brine and steam and heated to 400 °C (liquid zone A). While controlling temperature at 400 °C samples will be repeatedly collected to study the rate of equilibrium. The vessel
will be charged again with rock specimen and filled with brine and steam to reach the desired conditions. Brine and steam samples will be collected at different pressure and corresponding steam temperature.

Concentrated brine and steam, starting from steam and concentrated brine (zone B) with minerals specimens in vessel. The brine will be equilibrated and brine and steam sample collected at equilibrium conditions. After sampling the vessel will be cooled until the pressure reaches lower pressure and new samples collected after new equilibrium.

Equilibrium between salt and superheated steam (zone C). The brine will be evaporated and the steam will be equilibrated with salt and mineral specimens in the vessel. Steam sample will be collected at equilibrium conditions and then steam released until pressure equilibrates lower pressure for new sampling.

2.3 Metal corrosion
The vessel will be charged with metal specimens and filled with brine and steam and kept at high pressure and temperature in zone A, B or C for some time period. A sample of steam and brine will be collected in the end. The metal specimen will be examined for corrosion.

3. SAMPLING AND ANALYSIS
Sampling brine and steam from the vessel at 300 bar and 500 °C is not a trivial task. Upon depressing and cooling the dissolved minerals in the sample will precipitate and may accumulate in the sample line leading to wrong results. High volume vessels are very expensive and the vessel size will therefore be limited. The samples must be small to minimize the effect of sampling on the equilibrium conditions in the vessel and only small amounts can be used for flushing of the sample lines.

If samples are too small contamination of the vapor samples due to even a small amount of entrained liquid in the vapor phase will greatly affect the results.

It is proposed that the sampling will be done using evacuated container that can withstand the high pressure and temperature in the experimental vessel. The sample will be cooled and depressurized after collection in the sample vessel. Minerals that precipitate in the sample container will be dissolved and separately analyzed to get correct information about chemical composition of the brine and steam at experimental conditions, see figure 6.

Figure 6: Sampling from HP vessel.
At 450 °C and 300 bar approximately 45 g condensate can be collected in a 250 ml sample container and 87 g of brine can be collected in a 125 ml container. The samples will be analyzed by standard methods according to table 1.

### Table 1 Sample conservation and analysis.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Conservation</th>
<th>Components</th>
<th>Method</th>
<th>Size (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Headspace gas</td>
<td>Gigenbach + NaOH</td>
<td>CO₂, H₂S, H₂</td>
<td>Potentiometry and Gas Chromatography</td>
<td>250</td>
</tr>
<tr>
<td>pH, acidity, alkalinity</td>
<td>At site</td>
<td>pH, CO₂, H₂S</td>
<td>Potentiometry</td>
<td>25</td>
</tr>
<tr>
<td>Anions</td>
<td>Degassed</td>
<td>Cl, F, SO₄</td>
<td>Ion Chromatography</td>
<td>20</td>
</tr>
<tr>
<td>Cations</td>
<td>Acidified</td>
<td>Na, K, Ca, Mg, Fe, Mn, Cu, Pb, etc</td>
<td>ICP, ICP-MS</td>
<td>20</td>
</tr>
<tr>
<td>Silica</td>
<td>Diluted + NaOH</td>
<td>Si</td>
<td>ICP, ICP-MS</td>
<td>5</td>
</tr>
<tr>
<td>Rinse solution</td>
<td>Acidified</td>
<td></td>
<td>ICP, ICP-MS</td>
<td></td>
</tr>
</tbody>
</table>

4. VESSEL REQUIREMENTS

The high experimental pressure and temperature will present great demands on material property and vessel design. Figure 7 gives a PTx diagram showing the expected phase-properties of brine and steam at planned experimental conditions A, B and C and the pressure-temperature limits of the ASME class 2500 and 4500. If the vessel will be made to ASME class 2500 standard the experimental pressure must be reduced at higher temperature. Class 4500 standard is sufficient but then all parts of the vessel must comply with this standard.

![PTx diagram with pressure classes.](image)

The conditions in the vessel will be very corrosive and the vessel must be made of corrosive resistance material such as HASTELLOY® C-276 or C-2000 which are nickel-base alloys having good resistance to corrosion in acids and chloride solutions, over a wide temperature ranges (Haynes International). Other alloys such as Inconel 625 and Super Duplex and lining with exotic materials such as zirconium, tantalum or platinum may also be considered. Inlet valves and sampling valves etc must be of same material and pressure rating as the vessel.

The vessel will be a bolted closure vessel similar to the one shown in figure 8. For collecting samples high pressure and high temperature valves are needed e.g. available from HIP (High Pressure Equipment Company). The high temperature extension provides a means to move the packing area (stuffing box) away from the hot zone of a valve. Fins are provided around the packing area to dissipate heat and, with proper cooling of the extension, these valves can be used at 538°C. For safety the vessel will be provided with both a pressure relief valve and a rupture disk.
Figure 8: Bolted closure vessel and high temperature valves, HIP.

5. FUNDING AND PROJECT STATUS

The vessel size must be sufficiently large to allow a sufficient number of samples to be taken in each experiment without upsetting the steam-brine equilibrium in the vessel. The size will have great affect on the price and a size of 5 liters is suggested as a compromise.

Total budget is 345.000 USD and budget breakdown is shown in table 2.

Table 2 Budget breakdowns.

<table>
<thead>
<tr>
<th>Budget estimate</th>
<th>USD</th>
</tr>
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<tbody>
<tr>
<td>HP Vessel Hastelloy 5 liter</td>
<td>65.000</td>
</tr>
<tr>
<td>Heater + controls</td>
<td>10.000</td>
</tr>
<tr>
<td>Sampling vessels</td>
<td>20.000</td>
</tr>
<tr>
<td>Connections to plant</td>
<td>10.000</td>
</tr>
<tr>
<td>Facilities + vacuum pump etc</td>
<td>15.000</td>
</tr>
<tr>
<td>Total</td>
<td>120.000</td>
</tr>
<tr>
<td>Experiments</td>
<td></td>
</tr>
<tr>
<td>Partition experiments</td>
<td>89.000</td>
</tr>
<tr>
<td>Equilibrium experiments</td>
<td>84.500</td>
</tr>
<tr>
<td>Metal corrosion</td>
<td>52.000</td>
</tr>
<tr>
<td>Total Budget</td>
<td>345.500</td>
</tr>
</tbody>
</table>

6. REFERENCES


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