APPENDIX A

DIELECTRIC PROPERTIES AND MEASUREMENT

A.1 BASIC THEORY

A material is classified as dielectric if it has the ability to store energy when an external electric field is applied. For instance, if a DC voltage source is placed across a parallel plate capacitor as shown in Figure A.1, more charge is stored when a dielectric material is between the plates than if no material (a vacuum) is between the plates. The dielectric material increases the storage capacity of the capacitor by neutralizing charges at the electrodes which ordinarily would contribute to the external fields. The capacitance with the dielectric material is related to dielectric constant as follows:

\[ \kappa' = \varepsilon_r' = \frac{C}{C_0} \]  

where:

- \( \kappa' \), \( \varepsilon_r' \) = dielectric constant of the material
- \( C \) = capacitance with material = \( \frac{q}{V} \)
- \( C_0 \) = capacitance without material (vacuum) = \( \frac{A}{t} \)
- \( A \) = area of the capacitor plates
- \( t \) = distance between plates

![Figure A.1 Parallel plate capacitor DC case](image)

If an AC sinusoidal voltage source (V) is placed across the same capacitor (Figure A.2), the resulting current will be made up of a charging current \( (I_c) \) and a loss current \( (I_l) \)
which is related to dielectric constant. The losses in the material can be represented as a conductance (G) in parallel with a capacitor (C) as follows:

\[ I = I_{\text{charge}} + I_{\text{loss}} \]  
\[ I = V(j\omega C + G) \]

From equation A.1, \( C = C_0 \kappa' \), therefore

\[ I = V(j\omega C_0 \kappa' + G) \]

If \( G = \omega C_0 \kappa'' \), then

\[ I = V(j\omega C_0 \kappa' + \omega C_0 \kappa'') \]  
\[ I = V(j\omega C_0)(\kappa' - j\kappa'') \]  
\[ I = V(j\omega C_0)\kappa^* \]

Therefore, the complex dielectric constant (\( \kappa^* \)) given by

\[ \kappa^* = \kappa' - j\kappa'' \]

consists of a real part (\( \kappa' \)) which represents the storage and an imaginary part (\( \kappa'' \)) which represents the loss. The following notations are used for the complex dielectric constant interchangeably \( \kappa = \kappa^* = \varepsilon_r = \varepsilon_r^* \).

\[ \varepsilon_r^* = \kappa^* = \varepsilon^* / \varepsilon_0 = (\varepsilon' / \varepsilon_0) - j(\varepsilon'' / \varepsilon_0) \]  
where:

\( \varepsilon_r^* = \) complex relative permittivity
Appendix

$\varepsilon_0 = \text{permittivity in free space} \approx 8.854 \times 10^{-12} \text{ Farad/m}$

The real part of permittivity ($\varepsilon_r'$) is a measure of how much energy from an external electric field is stored in a material. $\varepsilon_r'$ is > 1 for most solids and liquids. The imaginary part of permittivity ($\varepsilon_r''$) is called the loss factor. It is a measure of how dissipative or lossy a material is to an external electric field.

A.2 ORIENTATION (DIPOLAR) POLARIZATION

A molecule is formed when atoms combine to share one or more of theirs electrons. This arrangement of electrons may cause an imbalance in charge distribution creating a permanent dipole moment. These moments are oriented in a random manner in the absence of an electric field so that no polarization exists. The electric field $E$ will exercise torque $T$ on the electric dipole, and the dipole will rotate to align with the electric field causing orientation polarization to occur (Figure A.3). If the field changes the direction, the torque will also change.

The friction accompanying the orientation of the dipole will contribute to the dielectric losses. The dipole rotation causes a variation in both $\varepsilon_r'$ and $\varepsilon_r''$ at the relaxation frequency which usually occurs in the microwave region.

![Figure A.3 Dipole rotation in electrical field](image-url)
A.3 MEASUREMENT

The schematic layout of the PNA system for measurement is shown in Figure A.4. The dimensions and pictures of dielectric probes used for this study are shown in Figure A.5 for a Slim Probe and Figure A.6 for a New Performance Probe.

Figure A.4. Layout of Measurement system

Figure A.5. Slim Probe
Appendix

A.4 COMPARISON BETWEEN SLIM PROBE AND NEW PERFORMANCE PROBE

During initial studies using slim probe, we demonstrated that the dielectric constant measurement has a potential application to detect hydrate water history. Further investigation was done during this phase of work to investigate similar application using a new Performance Probe. The experiment was designed to test water samples after hydrate formation and dissociation (HDW).

The result indicated that the water memory was a combined effect of changes in water structure and gas solubility. Water samples saturated with natural gas outside hydrate phase boundary (SDW) was analyzed to investigate the effect of gas solubility. Deionised water was used in all experiment as a reference data for determining changes of dielectric constant with respect to various types of water samples. The measurement was made for DW, HDW and SDW at atmospheric pressure and 4 °C.

The result shows that the new performance probe has higher sensitivity to detect changes in different type of water samples. The improvement in sensitivity was significant even with the worst case scenario where the sample prepared did not have continuous mixing as compared the sample preparation with continuous mixing. The comparison between Slim probe and New Performance probe results are shown in Figure A.6 New Performance Probe.
A.7 after 0.5 hour and Figure A.8 after 1 hour. As can be seen in Figure A.7, the difference in dielectric constant with DW for HDW at 4 GHz is increased by 10 times using New Performance Probe. The difference for HDW and SDW is very significant in the case of New Performance Probe as opposed to Slim Probe (old) at 1 hour as shown in Figure A.8.

Figure A.7. Comparison between Slim probe (old) and New Performance probe after 0.5 -0.6 hours.

Figure A.8. Comparison between Slim probe (old) and New Performance probe after 1 hour.
APPENDIX B

MONITORING COMPOSITIONAL CHANGES

B.1 INTRODUCTION

The objective of this work is to investigate techniques for monitoring changes in the composition of vapour and liquid phases to detect the hydrate formation and the hydrate ‘memory’ effect for use in early warning systems. Hydrate ‘memory’ has been found to remain in the aqueous phase for some time after hydrate dissociation. The studies based on dielectric constant technique shows that the hydrate memory was influenced by both gas solubility and changes in water structure. A very preliminary analysis on water samples taken from hydrate dissociation which was used for dielectric constant indicated that significant traces of $i$- and $n$-butane were present in water with a history of hydrate formation. Therefore, a series of tests have been designed to investigate this observation for natural gas-water systems. The main idea was to study changes in the composition of liquid and vapour phases.

The study was focused into three parts. The first part was compositional changes during, after hydrate formation and dissociation at atmospheric pressure. The second part was the analysis of changes in liquid samples after hydrate dissociation from all samples prepared for dielectric constant studies described in Section 3.3 of Chapter 3. The third part was online analysis of vapour samples for two systems; natural gas – water samples at low pressure and three components (95% C1, 4.5% C2 and 0.5% i-C4) system at constant pressure. These results are included in this appendix except the second part which are included in Section 3.3 of Chapter 3.

B.2. CHANGES DURING, AFTER FORMATION AND DISSOCIATION

B.2.1. Experimental set-up and procedure

Kinetic Rig 1 (shown in Figure B.1) with a total cell volume of 2400 ml was used. The rig was charged with 1000 ml of distilled water then vacuumed to remove any air. Natural gas (supplied by Air Products with composition given in Table B.1) was injected into the rig to a pressure of 103 bar (1500 psia) at 20 °C. Fluids were mixed by
means of a magnetic motor stirrer at 400 - 600 rpm. The fluid was allowed to equilibrate for 24 hours before sampling vapour and liquid phases for analyses. The system temperature was then reduced to 8 °C to observe changes in phase composition outside the hydrate structure-I region (predicted based on methane), before the temperature was reduced to 4°C to form hydrate. The fluid phases were then sampled again. Hydrates were then left in the system for several days before a further sample was taken to monitor any changes in the composition of the vapour phase. Hydrate was subsequently dissociated to outside the structure-I region (based on the predicted hydrate phase boundary using in-house HWHYD software given in Figure B.2) and vapour sample was analysed again before the temperature was increased to 20 °C to dissociate all hydrate. The fluid phase was then sampled at several stages to monitor the effect of hydrate water memory with time. The system was heated to 35°C for 24 hours, and then the temperature was reduced to 20°C to study the effect of heating on hydrate water memory.

![Figure B.1 Schematic diagram of Kinetic Rig-1](image)

**Table B.1 Natural Gas Composition**

<table>
<thead>
<tr>
<th>Component</th>
<th>N₂</th>
<th>C₁</th>
<th>CO₂</th>
<th>C₂</th>
<th>C₃</th>
<th>iC₄</th>
<th>nC₄</th>
<th>iC₅</th>
<th>nC₅+C₆</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mole (%)</td>
<td>2.16</td>
<td>89.69</td>
<td>1.69</td>
<td>4.62</td>
<td>1.26</td>
<td>0.17</td>
<td>0.30</td>
<td>0.06</td>
<td>0.05</td>
<td>100</td>
</tr>
</tbody>
</table>
Figure B.2. Hydrate phase boundaries for s-I (methane) and s-II hydrates.

The vapour and liquid phases were analysed using a Varian 3800 Gas Chromatograph (GC). Helium was used as the carrier gas with the flow rate adjusted to obtain a maximum sensitivity of the detector. Pure grade helium (3 ppm water trace and 0.5 ppm hydrocarbon) from Air Products was used. The carrier gas was cleaned by a molecular sieve positioned at the outlet of the helium bottle. Two detectors were used to perform the analyses: a Thermal Conductivity Detector (TCD) and a Flame Ionization Detector (FID). The TCD measures continuously the variation of the thermal conductivity of the carrier gas. The TCD is a non-destructive detector. The FID measures the compound capabilities to form ions when passing through a flame. The FID is a destructive detector, thus it is connected in series, at the outlet of the TCD. The analytical column used was a Hayesep R 100/120 Mesh column (silcosteel tube, length: 1.5 m, diameter: 1/8 inch). The hydrocarbon components were detected by FID while water, nitrogen and carbon dioxide were detected by TCD. 400µl of vapour sample and 0.5 µl of liquid sample were injected in the chromatograph via the injector with syringes.

B.2.2 Results and discussions

Two tests were performed for natural gas-water systems based on the procedure described in previous section. For the first test, hydrate started to form as the temperature was reduced to 8 °C based on the increase in torque and pressure drop (approximately 4 bar) as shown in Figures B.3 and B.4. Significant hydrate formation
was detected as the system temperature was further reduced to 4°C. At this condition, the stirrer appeared to stop functioning, most likely due to massive hydrate formation in the rig at this condition. The stirrer was subsequently turned off to avoid over heating of the motor. The system was left at this condition to form hydrates for 3 days before proceeding to the next step. The system temperature was increased to 13°C to dissociate s-I hydrate and the stirrer was then turned on to observe any reduction in torque following this partial hydrate dissociation. This step was followed by hydrate dissociation to outside phase boundary and heating to 35°C. The experimental path within hydrate phase boundary is shown in Figure B.5.

Figure B.3 Gas hydrate formation and dissociation for Test 1.

Figure B.4 Gas hydrate formation and dissociation for Test 1. The numbers represent samples taken for compositional analysis.
Figure B.5 The path followed during experiment with predicted hydrate phase boundary for Test 1. The numbers represent positions where samples were taken for compositional analysis.

<table>
<thead>
<tr>
<th>Sample no</th>
<th>Description</th>
<th>Temperature (°C)</th>
<th>Pressure (bar / psia)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Before formation</td>
<td>20</td>
<td>105 / 1530</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>After formation</td>
<td>8</td>
<td>71 / 1030</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>After formation</td>
<td>4</td>
<td>67 / 970</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>After formation</td>
<td>4</td>
<td>59 / 860</td>
<td>After 3 days, vapour only</td>
</tr>
<tr>
<td>5</td>
<td>After dissociation outside SI region</td>
<td>13</td>
<td>75 / 1090</td>
<td>vapour only</td>
</tr>
<tr>
<td>6</td>
<td>After dissociation</td>
<td>19.5</td>
<td>100 / 1457</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>After dissociation</td>
<td>20</td>
<td>100 / 1457</td>
<td>After 1 day</td>
</tr>
<tr>
<td>8</td>
<td>After dissociation</td>
<td>20</td>
<td>100 / 1456</td>
<td>After 1.2 days</td>
</tr>
<tr>
<td>9</td>
<td>After heating</td>
<td>20</td>
<td>99.8 / 1448</td>
<td></td>
</tr>
</tbody>
</table>

The liquid and vapour phase samples taken at various stages for compositional analysis are detailed in Table B.2. For the vapour phase, the compositional changes with respect to methane are presented in the form of percentage ratio of each component as shown in Figure B.6 and also the percentage ratio of before to that of after hydrate formation shown in Figure B.7. As seen from Figure B.7, significant reduction of the ratio of ethane to methane (C2/C1), propane to methane (C3/C1) and iso-butane/methane (iC4/C1) was observed due to hydrate formation. The ratio of carbon dioxide/methane...
(CO2/C1) was observed to remain relatively unchanged in the vapour phase. This result suggests that CO2 did not take part in hydrate formation to the same extent as C2 and C3. Other components in the system were not significantly affected. After hydrate dissociation, it was observed that the final liquid composition is very close to the initial composition before hydrate formation after about 1.5 days. Similarly, the vapour composition after subjected to heating at 35°C was close to that before hydrate formation. On the other hand, there is no clear trend of compositional changes in liquid phase as shown in Figure B.8 and B.9. This could be contributed to limited amount of liquid withdrawn during sampling. The CO2 component was clearly detected due to its high solubility in water phase. C1 and some traces of C2, C3 and C4 components were also detected. During hydrate formation there were no liquid samples available for analysis possibly due to conversion of most water to hydrate.

Figure B.6 Compositional analysis of vapour phase based on percentage ratio of component (mole %) /methane (mole %) for test 1.
Figure B.7 Compositional analysis of vapour phase based on percentage ratio of \( \frac{R_{\text{after}}}{R_{\text{before}}} \). \( R \) represent component (mole %) / methane (mole %) for test 1.

Figure B.8 Compositional analysis of liquid phase for test 1.
A further test was conducted using a similar gas composition and distilled water. The main difference from the first test was that the system was allowed to equilibrate for about 90 hours (more than 3 days) prior to reducing temperature to form hydrate. In the first test the system was left to equilibrate for only about 10 hours.

As shown in Figure B.11 and B.12, hydrates started to form as soon as the temperature reached 4 °C based on the increase in torque (Figure B.11) with a pressure drop of approximately 20 bars within 40 hours. This was followed by a very slow growth of hydrate for about 4 days before heating the system to dissociate hydrate. The path followed within the hydrate phase boundary during this experiment is plotted in Figure B.13.
Figure B.11 Gas hydrate formation and dissociation for Test 2.

Figure B.12 Gas hydrate formation and dissociation for Test 2. The numbers represent positions where samples were taken for compositional analysis.
Figure B.13 The path followed during experiment with predicted hydrate phase boundary for Test 2. The numbers represent samples taken for compositional analysis.

Table B.3 Samples collected for compositional analysis (Test 2).

<table>
<thead>
<tr>
<th>Sample no</th>
<th>Description</th>
<th>Temperature (°C)</th>
<th>Pressure (bar / psia)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Before formation</td>
<td>20</td>
<td>105 / 1530</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>Before formation</td>
<td>14</td>
<td>97 / 1400</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>After formation</td>
<td>4</td>
<td>55 / 800</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>After formation</td>
<td>4</td>
<td>41 / 590</td>
<td>After 1 days</td>
</tr>
<tr>
<td>5</td>
<td>After formation</td>
<td>4</td>
<td>39 / 570</td>
<td>After 5 days</td>
</tr>
<tr>
<td>6</td>
<td>After dissociation outside SI region</td>
<td>12</td>
<td>60 / 866</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>After dissociation outside SI region</td>
<td>20</td>
<td>87 / 1260</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>After dissociation</td>
<td>20</td>
<td>85 / 1230</td>
<td>After 1 day (limited liquid phase)</td>
</tr>
</tbody>
</table>

The samples collected during this test are detailed in the Table B.3. Vapour phase compositional data are presented in the form of percentage ratio, as described in the first experiment, and are plotted in Figures B.14 and B.15. Similar trends in the vapour compositional changes were observed in this experiment as compared to the first experiment. Significant reduction of ethane/methane (C2/C1), propane/methane (C3/C1) and iso-butane/methane (iC4/C1) was observed due to hydrate formation, while the ratio of carbon dioxide to methane (CO2/C1) remained unchanged.
For the liquid phase, again there is no clear trend of compositional changes as shown in Figure B.9 and B.10. As expected, CO$_2$ component was clearly detected due to its high solubility in water phase. C1, N2 and some traces of C2 and C4 components were also detected. It is interesting to point out that small traces of iC4 and nC4 were present in the sample after dissociation. It is important to point out that the analysis of liquid samples may not very representative of the liquid present in the system due to the current limitation on the configuration of the liquid sampling point at the rig. Further modifications of sampling point will be required for future tests to improve the sampling method.

![Bar chart showing compositional analysis of vapour phase based on percentage ratio of component (mole %)/methane (mole %) for test 2.](image)

Figure B.14 Compositional analysis of vapour phase based on percentage ratio of component (mole %)/methane (mole %) for test 2.
Figure B.15 Compositional analysis of vapour phase based on percentage ratio of \( \frac{R_{\text{after}}}{R_{\text{before}}} \). \( R \) represent component (mole %) / methane (mole %) for test 2.

Figure B.16 Compositional analysis of liquid phase for test 2.
B.2.3. Summary

Preliminary studies on fluid compositional analysis, particularly of the vapour phase, suggest that this technique has a potential to be used for monitoring the changes in composition due to hydrate formation. Significant reduction of the ratio of ethane to methane (C₂/C₁), propane to methane (C₃/C₁) and iso-butane/methane (iC₄/C₁) was observed due to hydrate formation. The ratio of carbon dioxide/methane (CO₂/C₁) was observed to remain relatively unchanged in the vapour phase. This result suggests that CO₂ did not take part in hydrate formation to the same extent as C₂ and C₃. Other components in the system were not significantly affected. However, monitoring water memory based on soluble gas present in the liquid phase required improvement on sampling technique.

B.3. ON-LINE COMPOSITIONAL CHANGES IN VAPOUR PHASE

B.3.1. Experimental set-up and procedure

The online compositional analysis set-up is shown in Figure B.18. Gas hydrate formation and dissociation was prepared using a high-pressure kinetic rig. This rig is equipped with 525 ml high-pressure cell (4500 psia), a magnetic bar for stirring, cooling jacket and temperature/pressure recording equipment controlled by computer. It has an operating temperature range of −30 to 75 °C with temperature being controlled within +/-0.05 °C. Temperature is measured by PRTs and pressure by Quartzdyne pressure.
transducers. Natural gas supplied by Air Products with the composition listed in Table B.1 was used in this study.

Experimental was setup by charging approximately 200 ml of deionized water into the kinetic rig at 20 °C. The kinetic rig was pressurised to 350 psia by injecting natural gas. The water-natural gas system was step by step cooled to 4 °C (inside the hydrate phase boundary) to form hydrates, while stirring at 240 rpm. The hydrate formation is detected based on the sudden temperature rise due to the release of latent heat of hydrate formation and the pressure reduction. After gas hydrate formation the system temperature was increased step by step to dissociate the hydrates. The composition of vapour phase was measure by directly sampled into gas chromatography to observe the changes in SII composition during the process.

The sampling is carried out using a capillary sampler injector, which is connected to the top of the cell through 0.1mm internal diameter capillary tube. The withdrawn samples are swept to a Varian 3800 gas chromatograph for analysis.

Figure B.18. Flow diagram of apparatus for on-line compositional analysis

A three-component gas mixture was used for this study which consists of 95 mole% of methane, 4.5 mole% of ethane and 0.5 mole% of iso-butane. Similar procedure and set-up describe above was followed. The main difference was that this analysis was conducted at constant pressure of 1500 psia and a HPLC pump was attached to the system to maintain the pressure via make-gas gas. The vapour samples was analysed at
several equilibrium temperatures which are 17 °C, 3.2 °C during hydrate formation, 12.3 °C, 13.6 °C, 17 °C, and 21 °C during hydrate dissociation.

B.3.2. Results and Discussions

On-line compositional changes in vapour phase at low pressure

The online analysis of vapour phase was made during hydrate formation and dissociation. The main purpose was to analyse if this set-up is workable for simulating on-line measurement. The analysis was done at low pressure (350 psia). Based on preliminary results shown in Figure B.19, it was observed that there is significant reduction (from 2.2 mole % to 0.2 mole %) in the concentrations of SII hydrate forming compounds during hydrate formation. The SII composition is the sum of all the hydrocarbon components that are known to form structure II hydrate, which are C₃, i-C₄, and n-C₄. A gradual increase in the concentrations of SII hydrate forming components was observed during hydrate dissociation as more gas was released from the aqueous phase. Similar trend was observed in the case of only monitoring i-C₄ concentration as shown in Figure B.20. This concentration of i-C₄ was reduced from 0.2 mole% to 0.02 mole %.

![Figure B.19 Online compositional analysis of vapour phase for SII hydrate forming components concentrations](image-url)

Figure B.19 Online compositional analysis of vapour phase for SII hydrate forming components concentrations
On-line compositional changes in vapour phase at constant pressure

To simulate the pipeline condition the compositional changes of vapour phase for simple system were investigated at constant pressure. As can be seen from Figure B.21, the composition of iso-butane in the vapour phase started to reduce at about 10 °C. This observation may indicate that hydrate started to form in the system. Further reduction in temperature leads to significant reduction in iso-butane concentration. At equilibrium temperature of about 3 °C, the composition reduced down to 60 % of its original feed value. During hydrate dissociation, the composition remains almost constant until reaching closer to the hydrate dissociation temperature predicted by in-house HWHYD software. The composition increased significantly as hydrate dissociated. This preliminary result may suggest that there is potential of monitoring compositional changes in vapour phase at constant pressure for hydrate early warning system. However, further investigation is required to further refine the technique and to find the suitable component for detecting the changes. Possibility of incorporating hydrate former as a tracer to detect hydrate formation in the system will also be considered.

Figure B.20. Online compositional analysis of vapour phase for i-C₄ concentration
Appendix

Figure B.21 Online compositional analysis of vapour phase for i-butane with make-up gas at 1500 psia

B.3.3. Summary

The compositional changes of vapour and liquid phase for various systems have been investigated. This preliminary result suggests that there is potential of monitoring compositional changes in vapour phase at constant pressure for hydrate early warning system. However, further investigation is required to further refine the technique and to find the suitable component for detecting the changes. Possibility of incorporating hydrate former as a tracer to detect hydrate formation in the system should also be considered.

B.4. COMPOSITIONAL ANALYSIS OF LIQUID PHASE FOR HDW AND SDW

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Concentration / mole</th>
<th>$C_2/C_1$ (%)</th>
<th>Average (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$N_2$</td>
<td>$CO_2$</td>
<td>$C_1$</td>
</tr>
<tr>
<td>HDW (inside)</td>
<td>1.15E-08</td>
<td>1.36E-08</td>
<td>9.05E-10</td>
</tr>
<tr>
<td></td>
<td>2.67E-08</td>
<td>1.32E-08</td>
<td>1.13E-09</td>
</tr>
<tr>
<td></td>
<td>1.15E-08</td>
<td>1.38E-08</td>
<td>8.3E-10</td>
</tr>
<tr>
<td></td>
<td>3.41E-09</td>
<td>1.15E-08</td>
<td>8.06E-10</td>
</tr>
<tr>
<td></td>
<td>SDW (inside)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>----------------</td>
<td>--------------</td>
<td>-----------</td>
<td>-----------</td>
</tr>
<tr>
<td></td>
<td>2.21E-09</td>
<td>5.8E-09</td>
<td>3.14E-10</td>
</tr>
<tr>
<td></td>
<td>2.55E-09</td>
<td>6.01E-09</td>
<td>2.46E-10</td>
</tr>
<tr>
<td>HDW (outside)</td>
<td>1.45E-09</td>
<td>5.95E-08</td>
<td>4.01E-08</td>
</tr>
<tr>
<td></td>
<td>1.71E-09</td>
<td>3.78E-08</td>
<td>2.29E-08</td>
</tr>
<tr>
<td></td>
<td>2.92E-09</td>
<td>6.62E-09</td>
<td>1.17E-08</td>
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<tr>
<td>SDW (outside)</td>
<td>4.95E-08</td>
<td>7.18E-08</td>
<td>9.32E-08</td>
</tr>
<tr>
<td></td>
<td>*</td>
<td>*</td>
<td>3.72E-09</td>
</tr>
<tr>
<td></td>
<td>*</td>
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<td>3.8E-09</td>
</tr>
<tr>
<td>HDWV</td>
<td>1.75E-09</td>
<td>2.31E-09</td>
<td>2.4E-09</td>
</tr>
<tr>
<td></td>
<td>2E-09</td>
<td>2E-09</td>
<td>1.86E-09</td>
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<td>*</td>
<td>2.94E-10</td>
</tr>
<tr>
<td></td>
<td>*</td>
<td>*</td>
<td>9.56E-11</td>
</tr>
<tr>
<td>SDWV</td>
<td>*</td>
<td>1.9E-08</td>
<td>3.01E-09</td>
</tr>
</tbody>
</table>
APPENDIX C

SCHEMATIC OF HIGH PRESSURE VISUAL CELL AND ULTRASONIC RIGS

HP VISUAL CELL

The experimental set-up is comprised of a high pressure cylinder fitted with sapphire windows at both ends. The cell can be used at pressures up to 517 bar and at temperatures between –30 and +50°C. The cell temperature is controlled by circulating water from a heater/chiller through an inbuilt jacket. A platinum resistance temperature probe monitors the cell temperature. The pressure of the cell is monitored by a strain gauge pressure transducer connected to the cell via a high pressure line. The accuracy of the temperature measurement is ±0.1°C and the pressure ±0.3 bar. A computer is used to log the cell pressure and temperature. A schematic of the experimental rig is shown in Figure C1. The cell is mounted on a rocking mechanism in order to give mixing when required.

ULTRASONIC RIG

The schematic of the ultrasonic set-up is shown in Figure D2. Its key part is the test cell. Two ultrasonic transducers are fitted at the two ends of the cell, respectively. One works as a transmitting transducer that generates acoustic signals, driven by the Pulser/Receiver (P/R), another one as a receiver that receives the signals through the fluids inside the cell.
The received signals are pre-amplified by the Pulser/Receiver and pre-processed by the Digital Storage Oscilloscope (DSO) and then sent to a Personal Computer (PC) to display the waveforms and save the data for further analysis. One of the two ends like a piston is movable, which is useful to adjust the volume of the test cell. A dial indicator is fitted to the tail bar of the piston for determination of the piston’s position. A hand pump and a piston vessel are used to inject test fluids. The test temperature is controlled by means of the cooling system that consists of a cooling bath and a jacket. In the tests the system temperature and pressure, and the waveforms can be automatically recorded by logging on the computer.

Figure C2. Schematic of Ultrasonic Rig
REFERENCES


