

CONSIDERATION OF THERMAL EFFECTS DURING RESERVOIR CONDITION SPECIAL CORE ANALYSIS TESTS

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ABSTRACT

When determining fluid contents of rock samples during high-temperature tests, particular attention should be focused on evaluating how temperature changes affect the fluid properties and desired measurements. This is especially true when considering results from high-temperature spontaneous imbibition, capillary pressure, and USBM wettability tests when samples are subjected to several cycles of ambient and elevated temperatures. Rock saturations calculated from effluent fluid volumes can be considerably different than true saturations unless the data analyses account for volume changes that result from temperature variations. For example, brine saturations calculated from 152° C drainage and imbibition oil-brine centrifuge tests can differ from actual saturations by over 10 saturation percent units if calculations do not account for changes in fluid volumes that occur as the sample is heated and cooled. Fluid volume corrections depend upon the initial saturation condition of the rock before it is heated, the type of fluid in contact with the sample as it is heated or cooled, and directions of temperature changes between test measurements.

Another important consideration is the amount of time given to achieve stable fluid saturations before measurements are recorded. Stabilization times determined during high temperature tests are often inappropriate for similar measurements at lower temperatures. This is because stabilization times are influenced by fluid viscosities, which in turn are temperature dependent. Stabilization times while centrifuging a 39 mD brine-saturated sandstone plug with mineral oil at 23° C were six times greater than those necessary during a similar 152° C test. This was because the viscosity of the oil at the 23° C temperature was 35 times higher than its viscosity at 152° C. Applying the same stabilization time criteria from the 152° C test to measurements at 23° C yielded capillary pressure versus saturation results and wettability indices that were considerably in error. Some of the saturation measurements from the 23° C test differed by over 10 saturation percent units from those that would have been achieved had sufficient stabilization time been allowed.

Corrections for fluid volumes produced or imbibed into samples because of thermal changes are fairly simple and straightforward as long as the densities of the fluids are well defined at both test temperature and ambient laboratory temperatures. Likewise, procedures for allowing sufficient stabilization times during centrifuge tests are fairly straightforward. Examples of these effects and corrections are provided from measurements on reservoir rock samples and fluids.

BACKGROUND

Core analysts recognize advantages of testing core plugs at reservoir-like temperatures when the measured characteristics are significantly influenced by temperature. Examples include measurements of relative permeability, capillary pressure and fluid saturation relationships, spontaneous imbibition and wettability determinations.

Comparisons of 25 reported relative permeability and capillary pressure tests conducted at elevated temperatures using relatively inert fluids show disagreement in effects of temperature on test results.¹ However, temperature is thought to have a definite effect on end-point saturations and hysteresis.²

Just how much of an effect does temperature have on rock fluid saturations? Temperature changes have definite effects on volumes of fluids imbibed or expelled from rock as it is heated and cooled. Temperature changes also influence test times necessary to achieve stable rock fluid saturations during various special core analyses. Importance of these considerations is described and illustrated in this paper through simple examples and descriptions of results for reservoir-condition measurements.

TEMPERATURE EFFECTS ON END-POINT SATURATIONS

Figure 1 is a diagram depicting saturation changes that may occur as a sample is heated and cooled during an oil-brine test. Throughout the test, the sample is driven to different end-point saturations. This is typically the case for several commonly used techniques for measuring spontaneous imbibition, centrifuge capillary pressure, and USBM wettability indices.

The o (oil) and w (brine) letters in the top left hand corner of each frame represent the fluid which surrounds or contacts the plug surfaces. Displaced fluid volumes are assumed to lose contact with the plug. Residual saturations are depicted on core plug icons and are assumed to be immobile at the end of each measurement cycle. Below each figure, the + or - ΔT caption indicates whether the temperature is raised or lowered at the start or end of the measurement set. Between each set, the plug is cooled to the ambient temperature and is transferred to a different fluid-filled container, such as from a drainage to an imbibition centrifuge tube.

In frame A, the brine-saturated plug is heated to the test temperature. The rock and fluids expand as the temperature increases. The rock expansion is often negligible compared to expansion of the fluids, so the volume of brine within the rock and container exceeds the pore volume of the rock. This excess volume can be calculated knowing the mass of brine originally contained in the rock pore volume and the initial and final densities of the brine for the test temperature and pressure conditions. This volume should be subtracted from the produced brine data when calculating rock saturations from volumetric measurements because it represents an excess volume rather than a saturation change. Otherwise, brine saturations calculated from the first oil-displacing brine cycle will be lower than actual plug saturations. Compared to actual saturations, this effect can make the residual brine saturation within the rock appear to decrease with increasing temperature.

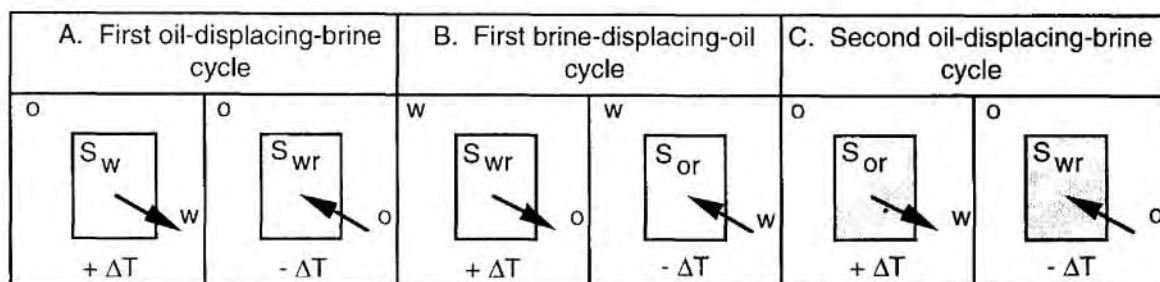


FIGURE 1. - Thermally induced fluid volume changes during elevated temperature centrifuge tests with a water-wet rock. Adapted from ref. 3.

Mobile brine is displaced from the plug in the second half of frame A. At the end of this cycle, the plug and pore fluids are cooled to the laboratory temperature. As the pore fluids cool, their volumes decrease. Because the sample is surrounded by oil, additional oil is imbibed into the rock as the temperature falls to the laboratory temperature. The brine volume within the rock decreases as the temperature drops. For this reason, the volumetric brine saturation within the plug decreases as the temperature is reduced. The final brine saturation of the plug at the laboratory temperature is less than what it was at the end of the higher temperature test even though the mass of brine within the plug is the same at both temperatures. The mass of oil imbibed into the rock can be calculated if the mass of the brine contained within the rock pore volume and fluid densities at the high and low temperatures and pressure conditions are known. Each time the sample is cooled to the ambient laboratory temperature, it is very useful to measure plug saturations using another method to double-check saturation calculations. The plug is transferred to a different container for the next set of measurements and is surrounded with brine.

In frame B, the plug and fluids are heated. Oil and brine within the plug increase in volume as the temperature increases. Their combined volumes at the test temperature exceed the pore volume of the plug, so fluid is expelled from the rock. Because the rock is at a residual brine saturation condition, one expects oil to be expelled from the rock because of this thermal expansion, as depicted in frame B. If this is the case, the brine saturation condition of the rock is the same as that which was present at the end of the previous oil-displacing-brine cycle when the plug and fluids were at the same elevated temperature condition. Continuing with this immobile brine saturation assumption, the volume of oil expelled from the rock because of thermal effects equals the volume expelled from expansion of the brine plus the volume expelled from expansion of the oil. These volumes can be calculated knowing the fluid densities, temperatures, and masses of brine and oil that were in the plug before it was heated. The assumption that only oil is expelled at this stage because of thermal expansion is not always true, as described later in the discussion section. Other complications are that the excess fluid which expands out of the rock as the temperature changes may cling to the sides of the rock until forced to break away. Depending upon the configuration of the container, this fluid volume may not be immediately visible. Spontaneous brine imbibition may also occur while the temperature is changing, which complicates the evaluation.

In the second half of frame B, the plug is cooled to room temperature. Again, as the rock cools, the volumes of the pore fluids decrease and additional brine (the surrounding fluid) is imbibed into the rock. For this reason, the oil saturation decreases as the temperature drops.

In frame C, the plug is placed in an oil-filled container for final oil-displacing brine measurements and is heated to the test temperature. One might expect the same mass of brine that enters the rock as it cooled after the previous measurement cycle to be expelled as the rock is again heated, and this is often the case. Finally, upon completing displaced-fluid volume measurements, the rock is cooled to the laboratory temperature. Additional oil is imbibed into the rock as it cools, again because the volumes of the pore fluids decrease as the temperature changes.

Numerical Example

Effects of temperature changes on volumes of fluids produced during centrifuge tests are illustrated with a simple example. Assume that a rock sample is used for oil-brine centrifuge capillary pressure and wettability measurements at temperatures of 66°, 93°, and 152° C. The temperature of the laboratory is 23° C, and the sample is cooled to the laboratory temperature between first oil-displacing-brine, first brine-displacing-oil, and second oil-displacing-brine saturation cycles. Further assume that the plug achieves residual brine and oil saturation fractions (S_{wr} and S_{or}) of 0.25 pore volumes at the end of each centrifuge cycle at each elevated temperature condition. Fluid densities and an example set of end-point saturations calculated for 152° C tests are shown in appendix tables 1 and 2. "True" end-point brine saturation results are compared with those inferred from produced fluid volumes in figure 2 a-c for the three temperature conditions.

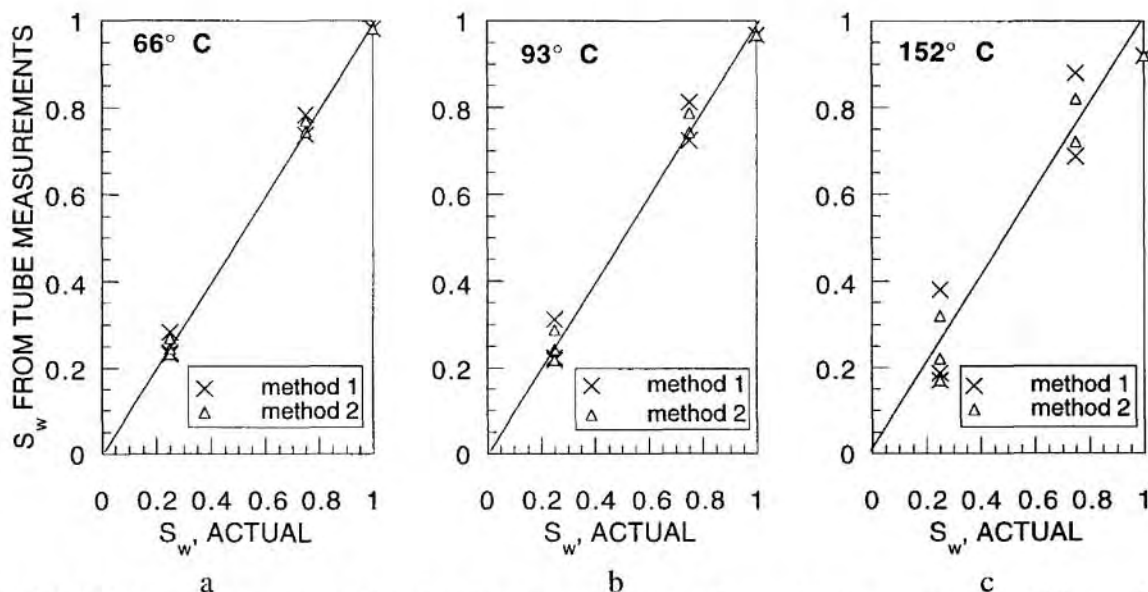


FIGURE 2. - Comparison of end-point saturations for elevated temperature conditions. In method 1, saturations are based on the volume of displaced fluid in the centrifuge tube at the respective temperature. Saturations at 23° C are correct but those at elevated temperatures are not because fluid expansion and contraction are not accounted for in the analysis. In method 2, saturations are inferred from high-temperature measurements only. Lines on the graphs show where results would fall if inferred and actual saturations were equal.

Inferred saturations of figure 2 were calculated using two incorrect data analysis techniques as described on the figure caption. Neither of the two techniques accounted for fluid volume changes because of thermal effects. For this reason, saturations inferred from both techniques are in error.

As shown by figure 2, potential errors in saturation measurements increase with increasing temperature. Although the first measurement technique infers correct fluid saturations when the sample and fluids cool to 23° C, neglecting to consider changes in fluid volumes with increasing temperature causes this method to differ from actual results to a greater extent than results which infer saturation changes from measurements only at elevated temperatures. Results for this method differ from "true" results for 152° C measurements by over 0.10 saturation units (10 saturation percent units) for brine-displacing oil cycle results.

Example From Tests on North Sea Sandstone Samples

Figures 3 and 4 show brine-displacing-oil capillary pressure results from tests on a sandstone plug sample from a North-Sea reservoir. Synthetic brine and n-decane were used for the tests. Figure 3 shows differences in results from 93° C measurements before and after correcting saturations for pore fluid volume changes that occurred because of temperature changes. Corrected results of figure 4 indicate that, for this plug and fluid system with temperatures to 93° C, capillary pressure versus average saturation results were relatively insensitive to changes in temperature.

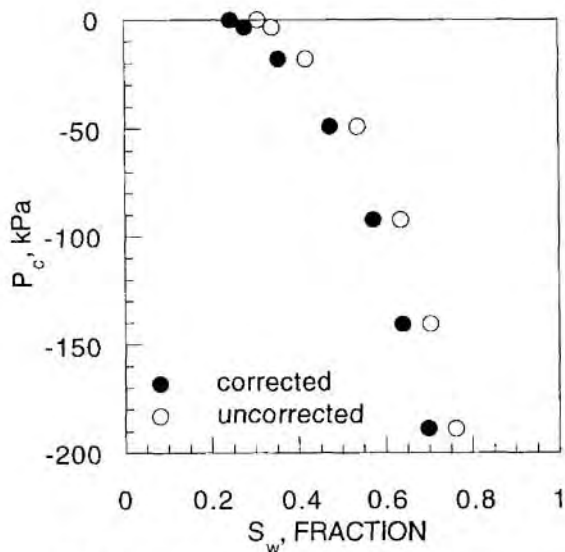


FIGURE 3. - Imbibition capillary pressure versus average saturation results from tests using n-decane and brine at 93° C. Corrected and uncorrected for fluid volume changes from thermal effects.

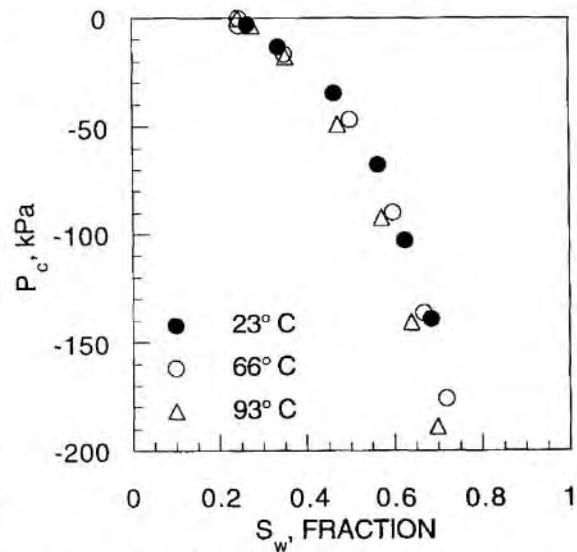


FIGURE 4. - Imbibition capillary versus average saturation results from tests using n-decane and brine at various temperatures. All results were corrected for thermal fluid volume changes.

TEMPERATURE AND TIMES FOR ACHIEVING SATURATION STABILITY

Another important consideration during a fluid displacement process is the amount of time given to achieve stable fluid saturations before recording measurements. Errors result when test times are too brief to yield stable saturations.^{4,5} Stabilization times determined during high temperature tests are often inappropriate for similar measurements at lower temperatures. This is because stabilization times are influenced by fluid viscosities, which in turn change with temperature.

Figure 5 shows centrifuge brine-displacing-oil capillary pressure versus average saturation results from USBM wettability tests conducted on a North-Sea sandstone plug. The permeability of the plug to air was 39 mD and its pore volume was 1.4 cm³. The plug was tested with mineral oil and n-decane at several temperatures. Tests were conducted using 2 and 12 hour per point measurement times.

Measurement times of 2 hours per point were considered satisfactory for tests with the mineral oil at 152° C. Testing the plug with mineral oil at 23° C using the same 2 hour per point time criteria as that of the 152° C test produced capillary pressure versus average saturation results that were in error by up to 10 saturation percent units (0.10 saturation fraction units). Other 23° C tests on lower permeability plugs using the same mineral oil and 2 hour measurement times showed results that differed from equilibrium saturations by over 15 saturation percent units and pressure curves which bowed in the wrong direction compared to what one would expect. The abnormal shapes of the curves significantly affected wettability indices calculated using the USBM method. Considering that the viscosity of the mineral oil at 23° C was 35 times that at 152° C, it makes sense that additional stabilization time was required during the 23° C tests.

When insufficient stabilization time is allowed, plug saturations are not indicative of equilibrium saturations. For example, figure 6 shows average plug saturations at various times as the plug was first driven to a residual brine saturation condition with a 3,000 rpm centrifuge speed. From the 23° C mineral oil data, after 2 hours, the average plug saturation was twice that of its final, stable saturation condition. Given enough time, the end-point saturation with the 70 cP mineral oil was similar to that from the test with 0.95 cP decane. Mineral oil and n-decane results on figure 5 were similar from tests at 23° C using 12 hour per point stabilization times. In this case, allowing sufficient time for equilibrium saturations to develop provided comparable results for the two fluid systems.

From this example, it is apparent that times required to achieve stable saturations during centrifuge tests are affected by the viscosities of the fluids, which in turn are affected by the test temperature. Plug permeabilities also influence stabilization times. Omoregie⁴ recommends minimum times at each centrifuge speed of 28 hours for samples of 1 mD or greater permeability and 56 hours for samples of lower permeability. For these and other reasons, do not apply one time criteria for all tests. Instead, allow sufficient time during each test interval for saturations to become stable before recording final data and changing experimental conditions.

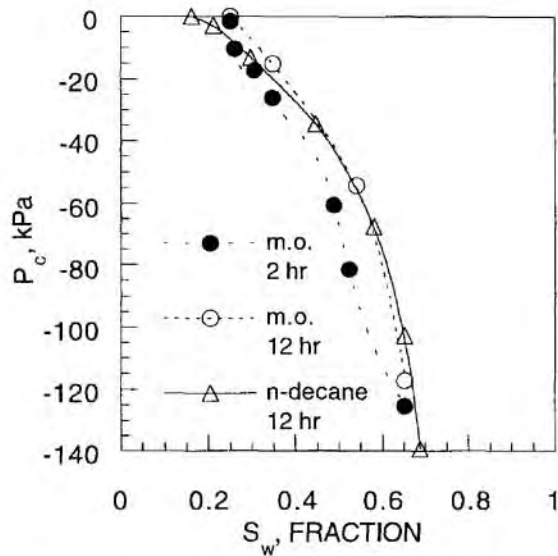


FIGURE 5. - Imbibition capillary pressure versus average saturation results for a plug using mineral oil (m.o.) and n-decane. Two and twelve hour per point measurement times. Viscosities for this 23° C test were 70 cP for mineral oil and 0.95 cP for n-decane.

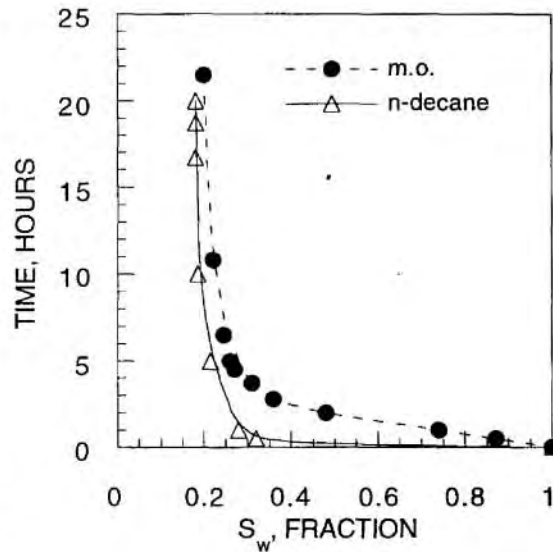


FIGURE 6. - Saturation changes with time as the brine-saturated plug was centrifuged at 3,000 rpm to residual brine saturation. The temperature was 23° C.

DISCUSSION

During centrifuge tests, as the rock is heated from room temperature to the test temperature, often there isn't any volume of the thermally expelled fluid in the graduated portion of the centrifuge tube until the spinning process begins. One explanation is that some or all of the fluid expelled from the rock by thermal expansion clings to the rock surface until the centrifuge rotation gives it an incentive to move into the graduated portion of the tube. With many centrifuges, the rock isn't visible when the tubes are in the tube holders and centrifuge buckets. After the tubes begin to spin, it may be impossible to visibly distinguish whether fluids were expelled because of the temperature change, spontaneous imbibition, or centrifuge rotation, so corrections must be applied considering fluid density changes with temperature and knowing which fluid is most likely expelled or imbibed as the result of a change in temperature. The volume expelled by thermal expansion that is calculated using the densities of the fluids at room and test temperatures is subtracted from all displaced volumes measured for that particular cycle.

Saturation changes that occur as the brine-saturated rock is heated and as the rock cools at the end of centrifuge cycles are highly predictable since these saturation changes are governed by the thermodynamics of the system. Saturation changes that occur when the partially saturated rock is reheated are also influenced by the rock wettability characteristics. Sometimes, a volume of the fluid which is considered to be at a residual saturation condition is expelled as the rock is heated. Between centrifuge cycles, it is advantageous to measure plug end-point saturations by another technique, such as mass

balance, X-ray absorption, or MRI to double-check end-point saturation results and insure that corrections for thermally induced saturation changes are properly applied.

The best approach toward minimizing temperature-related fluid saturation effects is to minimize the number of times samples are heated and cooled during a test. When this is not possible, then at least allow samples to fully cool to a known temperature between temperature cycles. Keep in mind that as a sample is cooled after an elevated temperature test, volumes of the pore fluids decrease, causing fluid surrounding the plug to be imbibed into the plug. If the plug is removed to air before it has cooled to the ambient laboratory temperature, it will imbibe air. Likewise, if the plug is transferred to a different container for the next drainage or imbibition measurement cycle before it has completely cooled, it will imbibe the surrounding fluid as it continues to cool. The amounts of imbibed fluids cannot be predicted when the process temperatures are unknown.

Lowering fluid viscosity by increasing operating temperature typically decreases the time required to reach end-point saturations. For a given system, fluid viscosities play an important role. However, temperature can also effect rock-fluid and fluid-fluid interactions through wettability, interfacial tension, contact angle, and solubility changes.^{6,7,8} Discussions of these changes are beyond the design of this paper.

CONCLUSIONS

To have meaningful data for comparisons of fluid contents of rock samples at various temperatures, particular attention should be focused on evaluating how temperature changes affect the fluid properties and measurements. This is especially true when considering results from high-temperature spontaneous imbibition, capillary pressure, and USBM wettability tests when samples are subjected to several cycles of ambient and elevated temperatures. Rock saturations calculated from effluent fluid volumes can be considerably different than true saturations unless the data analyses account for fluid volume changes that result from temperature changes.

Comparing results from displacement experiments with high and low viscosity fluids, tests with lower viscosity fluids generally require shorter time periods to reach final end-point saturations. As viscosity is affected by temperature, times to achieve stable saturations are also influenced by temperature. It is important to allow enough time during the experiment for fluid saturations to reach equilibrium values.

Significant conclusions from this work include the following:

1. Volumes of fluids contained in rock pores vary with changes in temperatures. It is important to account for these temperature-related volume changes when calculating rock saturations from volumetric measurements. Fluid volume corrections for thermal effects depend upon the initial saturation condition of the rock before it is heated, the type of fluid in contact with the sample as it is heated or cooled, and directions of temperature changes between test measurements.
2. Potential errors in fluid saturations calculated from volumetric techniques increase with increasing test temperature.

3. The time required to achieve stable saturations during centrifuge capillary pressure or wettability determinations is influenced by the viscosities of the test fluids, which in turn are influenced by the test temperature. Significant errors may result from applying the same saturation stabilization time criteria for both high and low temperature tests. Instead of using a single measurement time criteria, allow sufficient time during each test measurement interval for saturations to become stable before recording final data and changing experimental conditions. Rock-fluid and fluid-fluid interactions may also be influenced by temperature and should be considered important in an experimental program.
4. It is useful to double-check rock fluid saturations determined by volumetric measurements during high temperature tests by a second measurement technique, such as mass balance, X-ray absorption, or MRI measurements.

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APPENDIX - EXAMPLE PROBLEM

T, ° C	23	66	93	152
pw, g/mL	1.080	1.060	1.045	1.000
po, g/mL	0.725	0.695	0.670	0.620

	T, ° C	23	152			
	pw, g/mL	1.080	1.000			
	po, g/mL	0.725	0.620			
	PV, mL	1.000				
Test	Pore System (actual)					
Sequence	Vw, mL	Ww, g	Vo, mL	Wo, g	Sw	
1. Place plug in oil tube	1.000	1.080			1.000	
2. Heat to 152° C	1.000	1.000			1.000	
3. Centrifuge to Swr	0.250	0.250	0.750	0.465	0.250	
4. Cool to 23° C	0.231	0.250	0.769	0.557	0.231	
5. Place in brine tube	0.231	0.250	0.769	0.557	0.231	
6. Heat to 152° C	0.250	0.250	0.750	0.465	0.250	
7. Centrifuge to Sor	0.750	0.750	0.250	0.155	0.750	
8. Cool to 23° C	0.786	0.849	0.214	0.155	0.786	
9. Place in oil tube	0.786	0.849	0.214	0.155	0.786	
10. Heat to 152° C	0.750	0.750	0.250	0.155	0.750	
11. Centrifuge to Swr	0.250	0.250	0.750	0.465	0.250	
12. Cool to 23° C	0.231	0.250	0.769	0.557	0.231	
Test	Centrifuge tube measurements					
Sequence	Vw, mL	Ww, g	Vo, mL	Wo, g	Sw(1)*	Sw(2)**
1. Place plug in oil tube					1.000	1.000
2. Heat to 152° C	0.080	0.080			0.920	0.920
3. Centrifuge to Swr	0.830	0.830			0.170	0.170
4. Cool to 23° C	0.769	0.830			0.231	0.170
5. Place in brine tube					0.231	0.170
6. Heat to 152° C			0.149	0.092	0.380	0.319
7. Centrifuge to Sor			0.649	0.402	0.880	0.819
8. Cool to 23° C			0.555	0.402	0.786	0.819
9. Place in oil tube					0.786	0.819
10. Heat to 152° C	0.099	0.099			0.687	0.720
11. Centrifuge to Swr	0.599	0.599			0.187	0.220
12. Cool to 23° C	0.555	0.599			0.231	0.220
*Sw(1) is from the displaced fluid vol. in the centrifuge tube at the respective temp.						
**Sw(2) is inferred from elevated-temperature centrifuge tube measurements only.						