WETTABILITY LABORATORY EVALUATION UNDER RESERVOIR CONDITIONS: A NEW APPARATUS

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Abstract

It is increasingly thought that knowing a reservoir's wettability is essential for establishing the Special Core Analysis procedures in order to estimate S_{wi} and S_{or}. However, the conditions (T, P, kind of oil) under which this parameter is evaluated can have an important influence on the measurement result, and contradictory results on this subject are found in the literature. This affects the viability of the extrapolation to reservoir conditions from results obtained in the laboratory under different conditions.

The most reliable way to evaluate the wettability of reservoir rock is to reproduce reservoir conditions; however, no description of satisfactory apparatus for making this kind of measurement can be found in the literature.

This paper describes the first apparatus which can carry out the Amott-Harvey test under reservoir conditions. The influence of the conditions selected for the wettability measurements (T, P, kind of oil) on the wettability index obtained is studied for four reservoirs (three sandstone and one carbonate). For sandstone, it is demonstrated that an increase in temperature often (but not always) increases the rock's affinity for water, but this increase strongly depends on the reservoir rock considered. For the carbonate, no influence of thermodynamic conditions has been observed.

Finally, it is shown that, currently, it is impossible to predict the change in wettability that occurs when the previously considered conditions vary. Each reservoir is unique, and assessing wettability under reservoir conditions remains the most reliable and strongly recommended option.

Introduction

Fifty years ago, most reservoir engineers believed that all oil reservoirs were water wet. At the time, Special Core Analysis programs could even be occasionally carried out, in good faith, using cleaned samples (typically made water wet), refined oil instead of crude oil, and operating under room temperature and pressure conditions [1]. Since that time, these "certainties" have changed. Some authors [2-5] now think that more than half of all reservoirs are not strongly water wet. This tendency is doubtless even more pronounced in the case of carbonate reservoirs. The importance of the "wettability" parameter for capillarity, electrical properties, and fluid/fluid displacement characteristics, for a particular rock/fluids system, has also been demonstrated [1, 5, 6].

A consequence of this change is that many researchers, such as Verma [7], believe that knowledge of a reservoir rock's wettability (including knowledge of lateral or depth-related variations) is necessary for establishing the SCAL procedures necessary to determine S_{wi} and S_{or}.

No in-situ measuring method is really accepted as yet by the oil industry. This implies that the laboratory should have samples available whose surface conditions are representative of those of the in-situ rock, and that valid measuring methods and conditions should be selected. The solution to the first problem, that is, how to provide representative samples, may be found by preserving or restoring their surface conditions [5]. However, there are still some doubtful cases. It is also useful to sample one or more reservoir zones depending on its heterogeneity. The second problem concerns measuring methods and conditions. Numerous measuring methods have been proposed. The two most frequently used, and which are considered the most satisfactory, are described below.

The Amott test consists of calculating an index based on spontaneous and forced displacements of oil by water and of water by oil. It relies on imbibition and displacement experiments using a centrifuge or imposing a pressure gradient or flow rate. Different indices can be calculated (cf. Figure 1): the water wettability index I_W and the oil wettability index I_O. The difference $I_{AH} = I_W - I_O$, which varies from -1 to +1, is often used to quantitatively evaluate wettability.

The USBM test consists of obtaining, by centrifuging for example [8], the capillary pressure curves for imbibition and drainage. The wettability index proposed is the logarithm of the ratio of the area under the secondary drainage curve to that under the imbibition curve (cf. Figure 1). Different ways of calculating this index are possible, depending on whether or not the spontaneous drainage and spontaneous imbibition parts are included.

The conditions for measuring wettability using these two tests vary from one laboratory to another: native, preserved or restored reservoir rock (preservation and restoration conditions may be particular to each laboratory); reservoir brine, possibly reconstituted, or brine chosen arbitrarily; refined oil or crude oil; various pressures and temperatures.

Experimental Conditions and Wettability: Literature Review

A review of the literature shows that the equilibrium conditions of the rock/fluids system and/or of the wettability measurement can influence the results. As for temperature, numerous studies have shown that there is a tendency to increase the affinity for water (or to decrease that for oil) as temperature increases [9 - 17]. Thus Hjelmeland [9] demonstrated that a strongly oil wet system at room temperature becomes water wet at a higher temperature (60°C). Rivas [17] observed that the effect of temperature depends on the initial wettability condition: thus, a system which is partially or totally oil wet at low temperature becomes more water wet at high temperature. On the other hand, for a system which is water wet at low temperature, the change is slight or non-existent. According to Anderson [10], two temperature effects can be envisaged that explain this behavior. First, the solubility of compounds which are liable to be fixed to the mineral surfaces increases with temperature. Some of these compounds can thus be desorbed from the surface, and increase the affinity of the rock for water. Otherwise, interfacial tension and contact angle (measured in the water) diminish as temperature increases independent of the presence of adsorbable compounds. If considered in terms of contact angle, the affinity for water is thus increased.

However, other results can be found which show the opposite tendency: that water wettability diminishes when temperature increases [18-22]. Thus, in recent studies, Jadhunandan [21-22] has demonstrated that Berea sandstone aged with various crude oils becomes more oleophilic when the aging temperature of the rock/fluids system increases. These researchers realize that this result is difficult to interpret, as the adsorption of polar compounds of oils should decrease with an increase in temperature. Other studies have nonetheless shown that adsorption can increase with temperature [23, 24]. To explain the result, reference is made to the potential for a change in the stability, the solubility or the dispersal of asphaltenes in the oil phase, as well as to the influence of temperature on the stability of water films.

These variations in behavior must be due to the fact that several kinds of phenomena intervene in mineral/water/oil interactions (adsorption across a water film, transition of a water film from existing to non-existing conditions, appearance of asphaltene deposits etc.). An improvement in basic understanding of these phenomena should help reconcile the diverse results found in the literature.

As regards the effects of pressure on wettability, little is found in the literature. The studies show that, in general, pressure has little or no influence on wettability [10, 16, 25]. One recent study [26] shows that operating under reservoir pressure during the process of wettability restoration leads to a decrease in oil wettability (compared to a restoration at a lower pressure).

As for the role of dissolved gas, Hjelmeland [9] has shown that for a carbonate/brine/crude oil system, the use of stock-tank oil instead of live oil (for identical T and P) leaves the value of the contact angle virtually unchanged.

The use of refined oil instead of stock-tank oil in the case of preserved (native) or restored samples had been advised in the past, as it was thought [27] that the time required to modify wettability due to the desorption of products fixed on the rocks would be long compared to the duration of experiments performed in the laboratory. Since then, Wendel (cf. page 1139 in Ref. 10) has verified that this sort of oil exchange could modify wettability (reducing oil wettability). In a study where pressure, temperature and the kind of oil changed simultaneously, Kyte et al. [11] have shown that a rock/fluids system of intermediate wettability under normal T and P conditions becomes water wet under reservoir temperature and pressure conditions and with dissolved gas added to the oil.

Finally, the current state of knowledge regarding the influence that temperature, pressure and the presence of a dissolved gas have on wettability is insufficient to predict the influence of these parameters for a particular reservoir. Consequently, when evaluating the wettability of a reservoir rock/fluids system, it is best to match the particular reservoir's conditions as closely as possible (even when establishing the equilibrium). Until now, little work has been done to design equipment for measuring wettability under reservoir conditions.

Olsen et al. [15] modified a centrifuge to carry out the USBM test at reservoir temperatures (T_{max} = 177°C or 350°F) and at a maximum pressure of 35 bar (500 psi). This is an improvement over room temperature and pressure conditions, but does not always permit operating under reservoir pressures nor, above all, allow the use of recombined oil. Wiley [28] recently proposed an apparatus to carry out imbibition experiments under reservoir conditions. Since 1961, Kyte et al. [11] have also described apparatus for performing, on the one hand, imbibition experiments in water, and on the other, displacements, under reservoir conditions. However, these systems cannot carry out the four stages of the Amott-Harvey test while constantly maintaining reservoir conditions; a release of pressure and a reduction in temperature are needed to move from one apparatus to another.

This paper describes a new apparatus which overcomes these difficulties. Then, it presents experimental results that attempt to evaluate the influence of operating conditions (T, P, kind of oil) on the results of the wettability test, for different reservoirs. These experiments were carried out as part of different studies and in different contexts. However, in spite of some variations in experimental conditions, we thought it useful to present the combined results to the petroleum scientific community.

Experimental

• *Program*

The Amott-Harvey test was used to measure the wettability of the reservoirs considered under three types of conditions (room temperature and pressure, intermediate, and reservoir), as indicated in Tables 1 to 4. The study was carried out using reservoir rock samples which had previously been cleaned by sweeping with a series of solvents (whose efficacy had been previously determined). Synthetic brine from each reservoir was always used. The oil used (stock-tank oil, topped stock-tank oil, recombined oil) for establishing initial saturations, for aging and for the wettability test is specified in Tables 1 to 4.

Four reservoirs were studied: three clayey-sandstone, and one carbonate, all of which exhibited mixed wettability in previous core analysis studies (that is, with the potential for spontaneous displacement of oil by water and water by oil). Imbibition experiments were pursued until a recovery plateau was obtained. The experiments lasted several weeks.

• *Equipment*

For the experiments under room temperature and pressure conditions (20°C and atmospheric pressure), standard glass equipment was used for the imbibitions, and Hassler-type cells for the displacements. For the experiments carried out under reservoir conditions, a new apparatus was designed and built that operates at pressures of up to 450 bar (6520 psi) and a maximum temperature of 170°C (338°F). It is described in Appendix 1 and Figure 2. For the experiments carried out under "intermediate" conditions, a new apparatus was built using the same principle as above. It can operate at pressures of up to 25 bar (360 psi) and a maximum temperature of 80°C (176°F) (see App. 1 and Fig. 3).

• *Procedures* (see Appendix 2)

Results and discussion

* Clayey sandstone No. 1 (Table 1)

An Algerian sandstone with low porosity and permeability was used, whose facies considered contained about 6% clay [29]. The three wettability tests were carried out on three different samples from the same well that were taken at depths several meters apart.

It was observed that in spite of long imbibition periods, only small volumes of oil or water are generally recovered. The wettability test carried out under room temperature and pressure conditions showed a slight preferential oil wettability, while the other two tests showed mixed wettability (spontaneous displacement in both directions), that was preferential on the whole to water under intermediate conditions, and preferential on the whole to oil under reservoir conditions. No continuous trend was observed for recovered volumes during imbibitions in water and in oil, or for the value of the wettability index I_{AH} when going from room temperature and pressure conditions to reservoir conditions.

When comparing tests carried out under room temperature and pressure conditions to those performed under intermediate conditions, in which only the wettability temperature measurement changes, the affinity for water is observed to increase with temperature.
Another notable result is the significant increase in spontaneous displacement of water by oil under reservoir conditions when compared to the other two tests. However, this result cannot be interpreted, as the temperature, pressure and kind of oil changed.
The fact that the sample tested under intermediate conditions has a higher Soi than the other two samples implies some limitations to the previous interpretation of the results.

* Clayey sandstone No. 2 (Table 2)
This sandstone from the North Sea contains a few percent of clay, mostly kaolinite, illite, chlorite and trace amounts of pyrite. The tests were carried out on three samples taken one after the other from the midst of the same core sample (adjacent depths). Significant differences are observed for porosity and permeability for the sample tested under intermediate conditions. The difference observed for Soi for the other two samples (5.3%) is probably linked to the differences in experimental conditions for establishing Soi.

The test under room temperature and pressure conditions was carried out twice in succession. That is, the first test was performed after the rock/fluids system had aged 12 days, and the second test after an additional aging period of 55 days at 20°C which corresponded to the duration of the first test. It was observed that the rock tended to be oil wet in both cases. A slight increase of this character was noted from one test to the other, but it stayed at a low level. When thermodynamic conditions are modified, an increase in the affinity for water is noted (cf. oil volumes displaced during imbibition in water) as is a decrease in the affinity for oil (cf. water volumes displaced during imbibition in oil).

Finally, the wettability index goes from about -0.75 under room temperature and pressure conditions to +0.24 under intermediate conditions and +0.42 under reservoir conditions. Thus, an increase in affinity for water and a decrease in affinity for oil is noted when operating under reservoir conditions. Moreover, wettability becomes mixed as soon as there is a deviation from room temperature and pressure conditions. The behavior of this sandstone differs from that of the preceding one when going from intermediate to reservoir conditions.

* Clayey sandstone No. 3 (Table 3)
This North Sea sandstone contains approximately 10% clay (essentially kaolinite and a little illite). Two neighboring samples (from the same core, and taken one next to the other, that is, at the same depth) were used to evaluate wettability under room temperature and pressure and under intermediate conditions. Observations pointed once again to the existence of mixed wettability with a tendency for lower oil wettability when temperature and pressure are increased. Measurements under reservoir conditions were not made.

* Carbonate (from the Middle East - Table 4)
The tests under room temperature and pressure conditions and intermediate conditions were carried out on the same sample; the test under reservoir conditions was carried out on a neighboring sample [30].
The high value for S_{oi} for the latter sample was probably caused by sweeping with viscous oil before introducing the reservoir oil. Regardless of which conditions are

adopted, there is a slight spontaneous displacement of oil by water and of water by oil that is noted. There is thus a mixed-type wettability with a barely perceptible preference for one fluid over the other. The wettability indices are in all cases close to 0.

Finally, it can be inferred that the wettability of this carbonate is affected neither by the kind of oil used (refined oil, stock-tank oil, recombined oil), nor by thermodynamic conditions (20°, 85° or 121°C, atmospheric pressure or 204 bars). The adsorption phenomena whose equilibrium conditions are sensitive to thermodynamic conditions do not seem to explain the surface properties of this rock after restoration. It is the mineral surface that is at issue. The neutral wettability obtained after cleaning this rock with a complex series of solvents supports this hypothesis. This neutral wettability could come from several sources: hydrophobic materials contained in the remains of organisms which make up the mineral, fixation of molecules (organic acids?) found in the waters before migration [31], heavy products (asphaltenes, for example) of crude oil which are irreversibly fixed (and not completely removed by the cleaning procedure).

The evolution of the wettability index when going from one set of conditions to another for the four reservoir rocks has been drawn on Table 5. For the carbonate the results are independent of measurement conditions. For sandstone, when going from room temperature and pressure conditions to intermediate conditions, it is observed that I_{AH} increases slightly to strongly in all three cases. Temperature is the parameter that changes the most between the two sets of conditions (from 20° to 70° or 75°C, while pressure remains constant in one case, or goes from atmospheric pressure to 7 bars in the other two); it can be surmised that the evolution of I_{AH} is principally caused by an increase in temperature. When passing from intermediate to reservoir conditions, an increase in I_{AH} is noted for one of the sandstones, and a decrease is noted for the other. This behavior difference is not easy to interpret.

Finally, it seems that depending on the reservoir, the wettability index measurement is or is not affected by the measurement conditions, without it being possible to predict the influence. This confirms the opinion, widely held in petroleum laboratories, that each reservoir is unique. Additional work needs to be done to better understand the influence of temperature, pressure, and dissolved gas on the wettability of the reservoir rock/fluids system. Meanwhile, as the result of the wettability test *can* depend heavily on the operating conditions, if a realistic extrapolation of results to the in-situ reservoir rock is desired, reservoir conditions should be matched as closely as possible when assessing wettability.

Conclusions

1. A new apparatus for evaluating wettability by means of the Amott-Harvey test, under reservoir conditions, has been designed and built.
2. For a carbonate reservoir, it has been observed that thermodynamic conditions have no significant influence on wettability.
3. For the three sandstone reservoirs, when temperature was the sole or principal parameter modified, the trend most often observed was that water wettability increases with temperature, but the reverse can also be observed.
4. Our results nonetheless confirm that each reservoir is unique and that is it currently impossible to predict the influence of a change in thermodynamic conditions (T, P, kind of oil) on the wettability of a rock/fluids system.
5. Until other research leads to a better understanding of phenomena in this field, wettability should be evaluated under conditions as close as possible to those of the reservoir.

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APPENDIX 1 : Equipment description

The apparatus which operates under reservoir conditions includes:

- a thermoregulated chamber containing a sample-holding cell and various cells containing brine, refined viscous oil (to achieve the required value of S_{wi}), stock-tank oil or recombined oil.
- a pump,
- fluid cooling systems at the oven outlet,
- a back-pressure valve, a gas/oil/water separation system and a gas meter,
- a continuous recording system for temperature, absolute pressure, differential pressure, flow rate, gas volume, etc.

All elements in contact with the brine are made of Hastelloy. The sample (length: 10 cm, diameter: 5 cm) is covered with a heat-shrinkable PTFE sheath and then installed at the center of the cell using a low melting-point metal alloy (the alloy is chosen for its resistance to brine and for having a melting point 10-20°C above the temperature of the experiment). Before mounting, the sample's faces are dry cut to conform to a pre-established position. The cell's two piping ends are then mounted with metal joints, which prevent any loss of dissolved gas during the experiments. A bypass with valve, with an inside diameter sufficiently large (1.0 cm or 0.39 in.) to prevent oil drops from clinging is mounted as indicated in Figure 2.

The apparatus which operates under intermediate conditions was built according to the same principle. The only differences are in the nature of the steel (stainless), its thickness, the seals (Viton "O" rings), and the absence of a system to separate and measure the gas produced (Figure 3).

APPENDIX 2 : Experimental procedure

For the experiments carried out under room temperature and pressure conditions, the procedure is that already described in the literature [5]. The procedure for measuring wettability in the newly-designed equipment described in Appendix 1 is as follows:

- mount the cleaned sample in the cell, install the cell and create a vacuum,
- saturate with brine and bring up to reservoir pressure (porosity),
- bring up to reservoir temperature,
- close bypass, measure absolute permeability and put in recombined oil (with preliminary sweeping with viscous oil and stock-tank oil if necessary),
- age the system (for 7 to 12 days in the current study),
- open the bypass and rapidly replace the oil contained in the bypass and the dead volumes (known volumes) with brine,
- imbibition experiment in the brine with a slow circulation of brine from the bottom towards the top over the sample's two faces (via the bypass) in order to evacuate the oil produced; given the inclination of the cell, gravity helps evacuate the oil.
- once imbibition is complete, close the bypass and pump water from bottom to top across the sample,
- rapidly replace the water contained in the bypass and the dead volumes with recombined oil,
- imbibition in oil with slow circulation of oil from the top towards the bottom over the sample's two faces via the bypass in order to evacuate the brine produced; again, gravity helps evacuate the fluid.
- once imbibition is complete, close the bypass and pump oil from top to bottom across the sample.

Thus, the four volumes needed to calculate the Amott-Harvey wettability index are available. Using the "reservoir conditions" apparatus, the values of R_s and B_{oi} are checked. Relative permeabilities are generally measured after sweeping. The procedure was similar for the two apparatus (reservoir conditions and intermediate conditions).

Table 1 - Influence of experimental conditions on wettability evaluation for sandstone reservoir No. 1

		Preparation of samples		
Porosity, %		8.6	10.2	9.2
Abs. perm., k_w, md		13.9	25.4	34.0
Flood with oil, S_{oi}, %		72.7 (topped STO)*	84.3 (topped STO)	70.6 (recombined oil)
Aging:	temperature, °C	75	75	109
	pressure, bar	atm. press.	atm. press.	185
	duration, days	12	12	12
		Wettability test		
Conditions		room topped STO 20°C, atm. press.	intermediate topped STO 75°C, atm. press.	reservoir recombined oil 109°C, 185 bar
Imbibition in brine:	oil recovery, % PV	0	8.0	5.0
Displacement by brine:	oil recovery, % PV	54.5	49.6	14.3
	k_{rw} at S_{orw}	-	0.14	0.46
Imbibition in oil:	brine recovery, % PV	5.6	2.0	11.6
Displacement by oil:	brine recovery, % PV	45.1	54.0	10.0
Wettability indices:	I_w	0	0.14	0.26
	I_o	0.11	0.04	0.54
	$I_{AH} = I_w - I_o$	-0.11	+0.10	-0.28

** topped STO = stock tank oil without the fraction boiling before 75 °C*

Table 2 - Influence of experimental conditions on wettability evaluation for sandstone reservoir No. 2

		Preparation of samples			
Porosity, %		25.8		23.3	25.4
Abs. perm., k_w, md		3600		170	3600
Flood with oil, S_{oi}, %		64.7 (STO)*	64.8	63.3 (STO)	70.0 (recombined oil)
k_{ro} at S_{wi}		-	0.5	- -	0.6
Aging:	temperature, °C	70		70	97
	pressure, bar	atm. press.		7	270
	duration, days	12		12	12
		Wettability test			
Conditions		room STO 20°C, atm. press.	room** STO 20°C, atm. press.	intermediate STO 70°C, 7 bar	reservoir recombined oil 97°C, 270 bar
Imbibition in brine:	oil recovery, % PV	0	0	12.2	15.7
Displacement by brine:	oil recovery, % PV	30.8	37.2	19.3	6
	k_{rw} at S_{orw}	0.14	0.22	0.26	0.47
Imbibition in oil:	brine recovery, % PV	21.8	26.0	4.4	3.8
Displacement by oil:	brine recovery, % PV	9.1	7.6	26.5	8.9
	k_{ro} at S_{wr}	0.4	0.7	0.4	0.4
Wettability indices:	I_w	0	0	0.39	0.72
	I_o	0.71	0.77	0.14	0.30
	$I_{AH} = I_w - I_o$	-0.71	-0.77	+0.25	+0.42

* *STO = stock tank oil* ** *This test has been performed after the previous one.*

Table 3 - Influence of experimental conditions on wettability evaluation for sandstone reservoir No. 3

		Preparation of samples	
Porosity, %		19.6	25.3
Abs. perm., k_w, md		104	289
Flood with oil, S_{oi}, %		84.0 (STO)	66.0 (STO)
k_{ro} at S_{wi}		0.78	0.72
Aging:	temperature, °C	70	70
	pressure, bar	atm. press.	7
	duration, days	12	12
		Wettability test	
Conditions		room STO 20°C, atm. press.	intermediate STO 70°C, atm. press.
Imbibition in brine:	oil recovery, % PV	3.1	2.8
Displacement by brine:	oil recovery, % PV	55.7	38.5
	k_{rw} at S_{orw}	0.28	0.17
Imbibition in oil:	brine recovery, % PV	26.7	14.9
Displacement by oil:	brine recovery, % PV	*	25.0
	k_{ro} at S_{wr}		0.6
Wettability indices:	I_w	0.05	0.07
	I_o	(0.45)	0.37
	$I_{AH} = I_w - I_o$	-0.40	-0.30

* *Displacement disturbed by paraffin deposit. I_o was evaluated assuming an expected behavior for the considered displacement*

Table 4 - Influence of experimental conditions on wettability evaluation for a carbonate reservoir

		Preparation of samples		
Porosity, %		33.1	33.1	32.0
Abs. perm., k_w, md		155	155	38
Flood with oil, S_{oi}, %		63.3 (STO)	61.7 (STO)	80.0 (recombined oil)
k_{ro} à S_{wi}		0.78	0.76	0.53
Aging:	temperature, °C	85	85	121
	pressure, bar	10	10	204
	duration, days	7	7	7
		Wettability test		
Conditions		room refined oil 20°C, atm. press.	intermediate topped STO 85°C, atm. press.	reservoir recombined oil 121°C, 204 bar
Imbibition in brine:	oil recovery, % PV	1.3	1.3	3.2
Displacement by brine:	oil recovery, % PV	36.7	35.4	45.3
	k_{rw} at S_{orw}	0.26	0.27	0.18
Imbibition in oil:	brine recovery, % PV	3.1	4.2	2.4
Displacement by oil:	brine recovery, % PV	36.7	34.1	31.0
	k_{ro} at S_{wr}	0.9	0.53	-
Wettability indices:	I_w	0.03	0.04	0.06
	I_o	0.08	0.11	0.07
	$I_{AH} = I_w - I_o$	-0.05	-0.07	-0.01

Table 5 - Evolution of wettability index $I_{AH} = I_w - I_o$ vs. measurements conditions

Conditions	**room**	variation	**intermediate**	variation	**reservoir**
Sandstone No. 1	-0.11	+0.21	+0.10	-0.38	-0.28
Sandstone No. 2	-0.75	+1	+0.25	+0.17	+0.42
Sandstone No. 3	-0.40	+0.1	-0.30		n.a.
Carbonate	-0.05	-0.02	-0.07	+0.06	-0.01

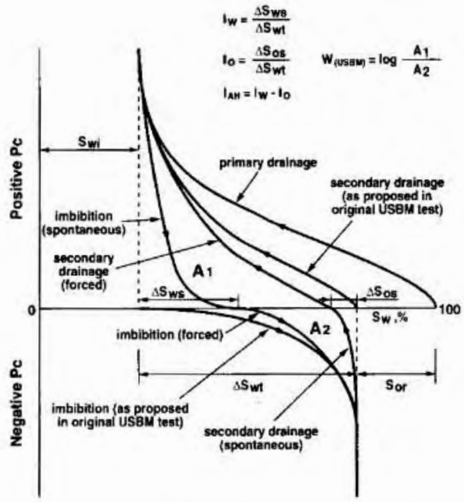

Figure 1. P_c - S_w Relationship and Wettability Measurement.

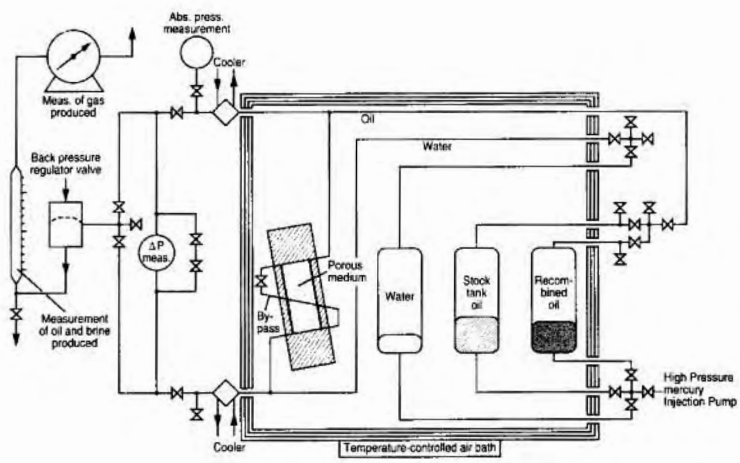

Figure 2. Schematic Diagram of Apparatus for Wettability Evaluation under Reservoir Conditions.

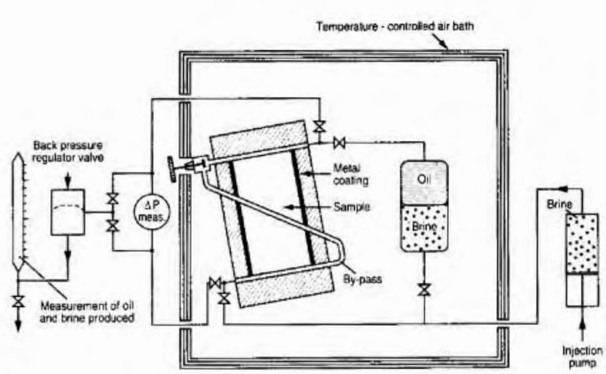

Figure 3. Schematic Diagram of Apparatus for Wettability Evaluation under "Intermediate" Conditions