

DEVELOPMENTS IN I-S_w MEASUREMENTS

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Abstract Resistivity index measurements (I-S_w) are nowadays routinely carried out at Shell Research, Rijswijk with the continuous injection (CI) method. In this technique one fluid phase is continuously displaced by another, while the injection pressure, the fluid saturation and the resistivity across the sample are recorded. The growing demand for measurements at more realistic reservoir conditions stimulated the development of a CI technique that enables isostatic stress conditions and injection pressures up to 60 bar to be applied, the latter being of importance for tight core material. Experiments carried out so far indicate a possible effect of stress on the resistivity index, this depending on the porosity. To study the effects of wettability and to gain a better grasp of the relationship between resistivity logs and laboratory core measurements, the RIMARC (resistivity index measurement at reservoir conditions) equipment was developed, in which CI-like measurements can be carried out at reservoir conditions of pressure, temperature and stress, with 'live' reservoir crude and brine as pore fluids. Scouting experiments have revealed a good similarity in results between this equipment and the 'standard' CI set-up when measurements are carried out under the same conditions.

The first experiments at reservoir conditions are compared with the standard measurement.

INTRODUCTION

The hydrocarbon saturation of reservoirs is often calculated from resistivity logs. For these calculations, the exact relationship between the electrical resistivity and the water saturation of the rock is required. A wide variety of techniques is used within the industry for measuring the electrical properties of core samples as a function of water saturation (Sprunt et al., 1988).

In 1984 at Shell Research, Rijswijk, The Netherlands, equipment was developed and built to measure the resistivity index of core samples according to a continuous injection (CI) technique (de Waal et al., 1989). This method was intended to replace the capillary equilibrium (CE) method which had been in use until then, and which was very time-consuming. Both the CI and CE methods are modifications of the method as described by Waxman

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and Thomas (1972) (see also Longeron et al. 1989). Although both techniques allow only one sample in a core holder to be examined at one time, they are preferred to the porous plate technique, in which several samples mounted on a large semi-permeable plate can be desaturated simultaneously. The reasons for this preference are discussed by de Waal et al. (1989).

In recent years, the realization has grown that measurements on cleaned core material under laboratory conditions using substitute formation fluids may yield results that differ from the electrical behaviour of the rock under reservoir conditions. Several workers have reported on the effect of stress (Longeron et al., 1986), temperature (Søndenå et al., 1990) or wettability (Lewis et al., 1988, Anderson, 1986) on the resistivity index values.

The need for resistivity index data at in-situ conditions has led to the development at our laboratory of the RIMARC (resistivity index measurement at reservoir conditions) equipment. RIMARC enables resistivity index measurements to be made using live crude and formation water at reservoir pressure and temperature, both under drainage and imbibition (waterflood) conditions. Hence, the effect of wettability and core cleaning can also be investigated. The RIMARC technique, which is based on the CI technique, makes it possible to determine directly the in-situ relationship between resistivity and saturation.

In this paper, details of the CI equipment and measuring technique are presented. Some results obtained with this technique are given, including some measurements that were carried out at elevated confining stress. In addition, the first experiments carried out with RIMARC at reservoir conditions are compared with the standard CI measurement.

CONTINUOUS INJECTION TECHNIQUE

History

At Shell Research, Rijswijk, the semi-permeable diaphragm technique as described by Waxman and Thomas (1972) was used for the combined measurement of the resistivity index (I) vs. water saturation (S_w) relationship and the oil/brine capillary pressure curve. In this capillary equilibrium method, measurements are taken at 6 to 10 pressure points. At each of these points, pressure equilibrium has to be reached, and this can take up to 7 days. This method thus requires on average 6 weeks per sample in the drainage mode, with another 6 weeks for the imbibition mode, if this is called for.

The growing demand for I - S_w measurements stimulated the development of a much faster but equally reliable and accurate measuring technique. By modifying and automating the conventional semi-permeable diaphragm technique, this goal was achieved and eighteen units for measuring resistivity indices according to the CI technique became operational in 1984.

Equipment

A schematic of the experimental set-up is shown in Fig. 1. The main components of the equipment are described below.

- (1) A Hassler-type core holder fabricated from AISI-316 with Monel-400 end pieces and pressure tested to 110 bar. Hydraulic oil is used to apply a constant confining pressure of 70 bar to a Viton sleeve surrounding the core sample. A spring mounted in the core holder prevents loss of axial stress of the assembly during transportation to the measuring site (see Fig. 2).
- (2) A cell computer connected to each of the measuring units is used for data acquisition and control of the measurement. Data are transmitted to a main frame at pre-set time intervals for further processing.
- (3) A fluid displacement pump equipped with a piston and a stepping motor. The pump is designed to displace 50 nl per step. The pump cylinder volume is 5.4 ml and the flow rate can be adjusted from 0.0005 ml/h to 100 ml/h. The amount of displaced fluid is measured by a calibrated potentiometer connected to the piston of the pump. The pump is regularly calibrated at experimental conditions.
- (4) Resistance-measuring equipment. Two platinum black electrodes mounted on the top and the bottom of the sample are connected to an automatic Hewlett Packard LCR bridge, type HP4261A. A frequency of 1 kHz is applied to avoid polarization effects. The platinum electrodes are blackened by the electrolytical deposition of platinum from a platinum chloride solution, to enlarge the surface area and thus minimize contact potential errors.
- (5) A semi-permeable membrane, to prevent passage of the injected fluid (kerosene) at the outflow end of the sample. The membrane used for consolidated samples is a Sartorius filter type 14549 and for unconsolidated samples a Metricel alpha-200 filter. The latter membrane will withstand flushing with methanol in the cleaning stage. Both membranes can withstand a differential pressure up to 9 bar.

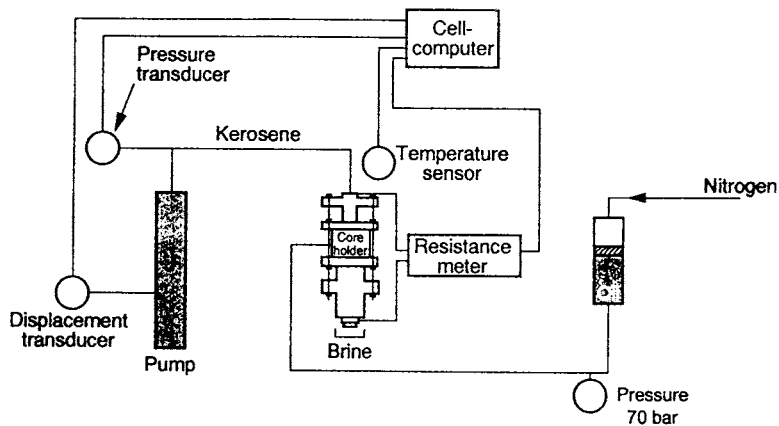


FIGURE 1 Schematic of CI test set-up

The measuring units are installed in a temperature-controlled laboratory room in which the temperature is kept constant at $21.5 \pm 0.5^\circ\text{C}$.

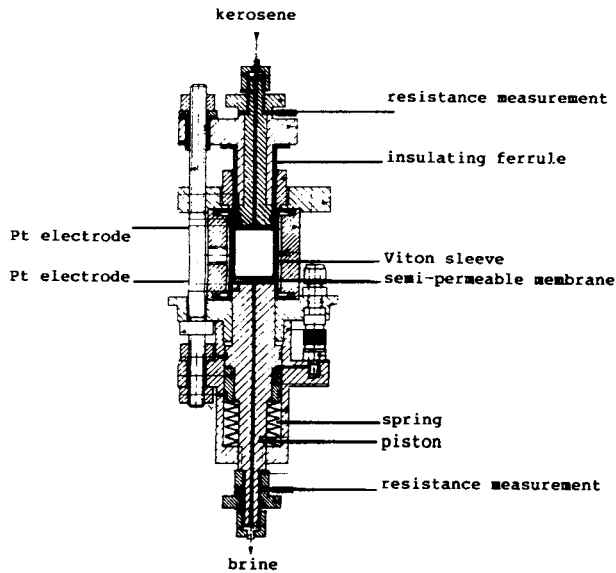


FIGURE 2 Core holder

Sample preparation

Measurements can be carried out either on consolidated or unconsolidated core samples with a length of 3 to 4 cm and a diameter of 2.54 cm (1 inch). Prior to testing, the sample is scanned by X-ray tomography (CT scanning) to check its homogeneity and to aid in the interpretation of the experimental results.

Consolidated samples are cleaned by solvent reflux, while unconsolidated samples are cleaned by flushing in a separate sleeve pressure cell. The solvents used for cleaning depend on the sample lithology. Typical solvents are chloroform methanol(-water) azeotropes and toluene. The samples are oven-dried under vacuum at 95°C. Following the cleaning procedure, the cleaned consolidated samples are subjected to porosity and permeability measurement. Porosity is measured by determining both the bulk volume and the grain volume according to the buoyancy method, i.e., the bulk volume is determined by measuring the buoyancy of the dry sample when it is submerged in mercury; the grain volume by measuring the buoyancy of the toluene-saturated sample when it is submerged in toluene. Prior to the resistivity index measurement, the wettability of the sample is qualitatively tested by observing the imbibition of a water droplet placed on the surface of the toluene-saturated sample. After drying, the consolidated sample is ready for the resistivity index measurement.

Unconsolidated material is dried in the cleaning cell by flowing nitrogen at ambient temperature through it. Next, low-salinity brine is introduced into the sample, after which the sample is frozen in the cell using dry ice. Now the sample can be dismantled from the preparation cell and can be treated as if it is consolidated.

Measuring procedure

The consolidated sample is mounted in the measuring core holder in which the resistivity index curve will be determined. Here, the sample is placed inside a rubber sleeve between two plungers with platinum electrodes. A semi-permeable membrane is placed between the sample and the bottom electrode. A confining stress of 70 bar is applied to the sample.

The frozen unconsolidated sample is mounted in the measuring core holder, pressurised to 70 bar and is allowed to thaw. Methanol is flushed through the sample to remove salt water while the electrical resistance across the sample is measured. When the resistance becomes infinite, this is an indication that the sample is clean. The sample is then dried by flowing nitrogen through it.

In the core holder both the consolidated and unconsolidated samples are evacuated and subsequently saturated with deaerated brine of known resistivity value R_w . The brine is fed into the sample with a Ruska displacement pump and the volume is recorded with an accuracy of 0.001 ml. A pressure of 10 bar is applied to the brine to ensure the adequate filling of all pores. From this procedure, the stressed pore volume of the sample can be calculated.

Then brine is flushed through the sample until the resistance across the sample becomes constant and equal to r_0 . The core holder with the sample is then transferred to an automatic resistivity index measuring unit. During transfer the isostatic stress on the sample is maintained.

To begin the measuring procedure the displacement pump starts to inject kerosene into the core holder. Initially this is done at a high injection rate (about 100 ml/h) first displacing brine from the flowlines. During this stage the pump displacement, the resistance across the sample, the injection pressure and the temperature are monitored continuously. At regular intervals readings are taken and stored in the cell computer. Once the kerosene has reached the sample, its resistance (r_t) will increase. When a pre-set value about 4% higher than the initial resistance (r_0) is reached, the cell computer adjusts the injection rate to the measuring rate (about 8 μ l/h). This rate is chosen such that the brine is expelled from the sample in about 14 days, i.e. the flowrate is governed by the pore volume only. During this period, resistance, pump displacement, injection pressure and temperature are again read at regular intervals and stored in the cell computer. The continuous injection is stopped when a pressure of 9 bar is reached, because at higher pressure kerosene will enter the semi-permeable membrane. The pressure is maintained constant at 9 bar, resulting in a declining rate.

The experiment is stopped when no more brine is expelled from the sample or, as happened occasionally, when the semi-permeable membrane collapses.

The data obtained in the initial stage are used to calculate the resistivity at 100% water saturation (R_0) and the pump displacement value at which kerosene started to enter the sample. Resistance readings are corrected for cell blank and converted to a standard temperature of 21.5°C. The water saturation of the sample at each measuring point is derived from the pump displacement readings.

Although not routinely performed, the experiment can be reversed following the drainage cycle. Water expelled in the drainage cycle and collected in a burette will spontaneously imbibe into the sample. During this cycle the amount of water imbibed can be measured together with the resistance and temperature, resulting in an imbibition resistivity index curve.

RESULTS AND DISCUSSION

The continuous injection technique results in an accurate and precise determination of the resistivity index vs. water saturation relationship of core material (de Waal, 1989). In Fig. 3 some typical experimental results are given to illustrate the variety of $I-S_w$ curves that can be encountered and the density of data points obtained with the equipment. The data presented in this figure are corrected for shaliness by applying the Waxman and Smits equation (Waxman and Smits, 1968); Q_v values were determined by a wet chemistry method according to Mortland and Mellor (1954). The value of n^* for a straight-line Archie relationship on log-log scale can be determined with the continuous injection technique with a precision of $\pm 2\%$.

In some instances a curved I^*-S_w relation is observed. This has been reported in literature earlier (Diederix, 1982; Anderson, 1986), and is ascribed to microstructural properties of the sample (pore size distribution, shale distribution, grain roughness) or the wettability of the sample. This curvature has also been derived from theoretical calculations as published by Spalburg (1988). An advantage of the continuous injection technique is that semi-continuous data are obtained over the full $I-S_w$ range, which results in a well-defined $I-S_w$ curve. This is of particular importance in the case of curved $I-S_w$ relations.

Figure 4 is an illustration of a resistivity index curve as measured on a seemingly homogeneous sandstone sample. CT scanning of the sample indicated the presence of a shale streak parallel to the sample axis. In this case non-linearity is probably related to sample heterogeneity. Figure 4 also demonstrates the repeatability of the experimental technique.

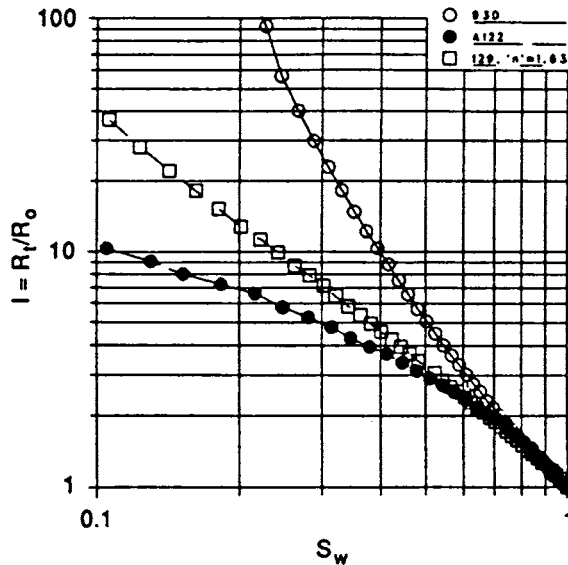


FIGURE 3 Typical $I-S_w$ curves

Figure 5 gives an example of a drainage experiment followed by an imbibition measurement.

Drainage experiments often yield different resistivity index values than the imbibition cycle does, at identical water saturations (see Fig. 5). This difference may be attributed to variations in the oil and water distribution on a microscopic scale (Longeron et al., 1986). Depending on the pore structure, different types of water and/or oil isolation can occur in the imbibition cycle. A programme of experiments is planned to quantify further these differences in the drainage and imbibition $I-S_w$ relationships.

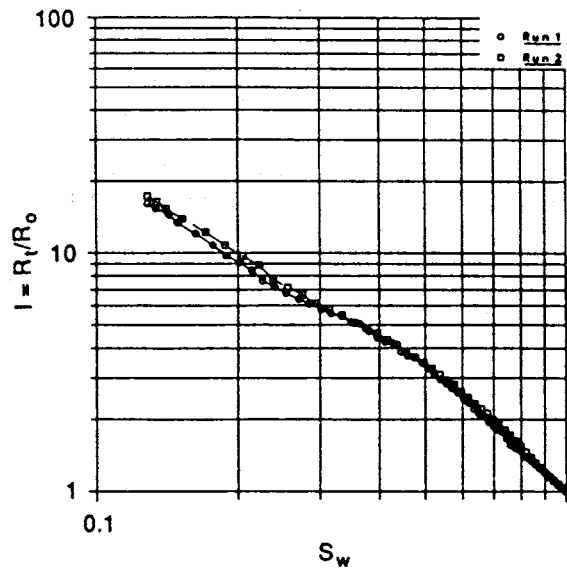


FIGURE 4 $I-S_w$ relationship of heterogeneous sandstone

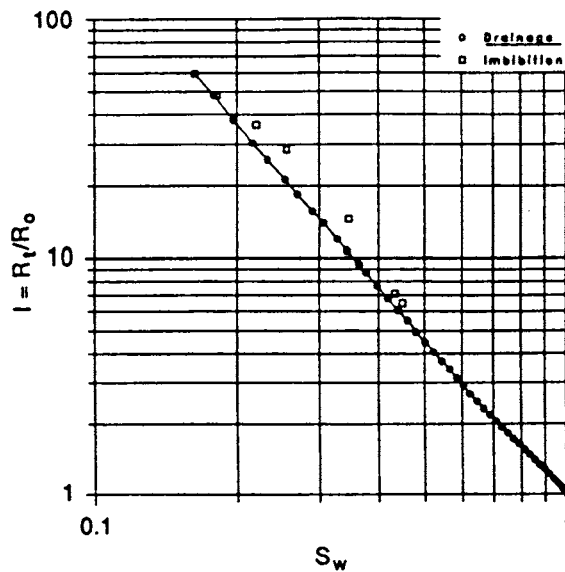


FIGURE 5 Drainage $I-S_w$ curve, followed by Imbibition cycle

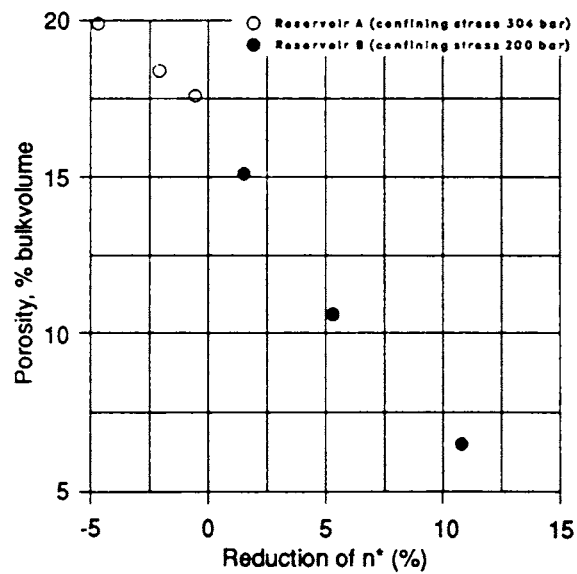
Experiments by de Waal et al. (1989) using CT scanning showed that inhomogeneous fluid distribution can arise when the kerosene is injected too quickly, particularly in highly permeable samples (piston-like displacement). A non-uniform saturation in the sample will result in erroneous resistivity index values. To allow the capillary forces to establish a condition of sufficiently uniform fluid saturation in the sample, a displacement rate of typically 5 to 10 $\mu\text{l/h}$ is applied. This is generally sufficient to displace 80% of the sample pore volume in 14 days. Numerical simulations suggest that at such a flow rate uniform fluid distributions are maintained throughout the experiment, with the exception of the very beginning of the injection phase. Experiments are currently under way to confirm the simulation results and to establish the effects on the measured capillary pressure curve.

Measurements at elevated stress

To study the effect of reservoir confining stress on the resistivity index, part of the equipment was modified to allow an isostatic confining stress of up to 300 bar during the measurement. Resulting clay-corrected n^* values of six sandstone samples from two different reservoirs, measured both at standard conditions and at reservoir stress conditions, are given in Table 1. The change in saturation exponent is plotted versus the sample porosity in Fig. 6. Though it is clear that stress can have an influence on the resistivity index, we have no explanation for the observed effects as yet.

TABLE 1 Stress effect on saturation exponent values

Sample id	Φ % bulk volume atm stressed		K mD	n^* 70 bar	n^* stressed	confining stress (bar)
Reservoir A						
542B	18.4	16.3	140	1.92	1.96	304
748B	17.6	15.7	35	1.82	1.83	304
477B	19.9	18.1	250	1.92	2.01	304
Reservoir B						
133	15.1	12.9	2.3	1.32	1.30	200
138	10.6	9.7	0.2	1.32	1.25	200
130	6.5	5.6	0.1	1.58	1.41	200

**FIGURE 6 Change of saturation exponent vs. atmospheric porosity as a result of confining stress increase**

RESISTIVITY INDEX MEASUREMENT AT RESERVOIR CONDITIONS (RIMARC)

Equipment

The wish to conduct $I-S_w$ measurements at true reservoir conditions stimulated the construction of a robust test system, suited for high pressures, high temperatures and capable of coping with aggressive fluids such as saline hot water and sour crudes. In the pilot RIMARC set-up, the measurements have been carried out in accordance with the capillary equilibrium technique. The system is currently being upgraded to enable measurements with the continuous injection technique.

The heart of the RIMARC system consists of a core holder in which core samples of both 2.54 and 5.08 cm diameter can be placed. The length of the sample must be between 2 cm and 6 cm. An isostatic stress of maximum 600 bar can be applied to the sample through a Viton sleeve, pressurized with oil from an accumulator vessel. A nitrogen-driven mercury vessel supplies injection pressure to the oil that has to replace the water in the sample. This pressure (maximum 500 bar) is kept constant throughout an experiment. The back pressure is applied through a mercury/water vessel, connected to a Ruska displacement pump. The water in the sample is displaced by **withdrawing** water from the sample by means of this pump. This technique was chosen to minimize the impact of leakage in the system, since the bulk of the dead volume of the system is located at the oil injection side. A Vycor glass membrane is fitted at the outflow of the sample, which prevents oil from breaking through, up to differential pressures of some 50 bar. The resistivity is measured with a Wayne Kerr automatic precision bridge (type B905a) at a frequency of 1 kHz, and again using blackened platinum electrodes. The (capillary) tubings and storage vessels in the system that are in contact with mercury are constructed of stainless steel, while the parts of the system that are in contact with saline water are made of Monel-400. The upper temperature limit of the system is 150°C. During the experiment, temperature, isostatic stress, oil pressure and water pressure are recorded continuously, while at each displacement step the saturation can be calculated with the aid of readings of the Ruska pump.

The stressed pore volume of the sample is measured separately in a Hassler-type cell at room temperature. The change in pore volume due to temperature is thus ignored, but it is thought to be very small: if a thermal expansion coefficient for quartz of approx. $10^{-6}/^{\circ}\text{C}$ is taken, the change in pore volume of a sandstone sample caused by a temperature rise of 130°C is estimated at some 0.015%.

A schematic diagram of the RIMARC equipment is shown in Fig. 7.

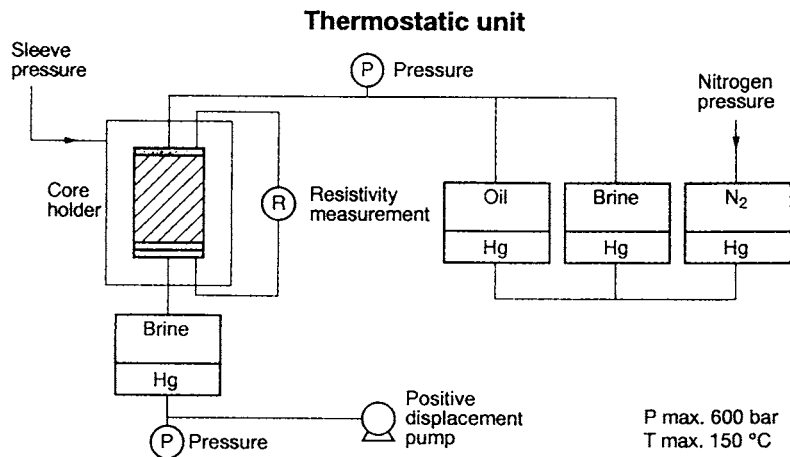


FIGURE 7 RIMARC test set-up

Sample preparation

Oil samples

Preferably, down-hole PVT samples should be used in the experiments. Alternatively, separator oil and gas samples can be used, which then will have to be recombined into the reservoir fluid, using the right gas/oil ratio. The saturation pressure of either the down-hole reservoir fluid or the recombined crude has to be determined at reservoir temperature. Wax and asphaltene content together with the cloud point need to be considered to assess whether the capillary tubing of the RIMARC equipment is likely to become plugged.

Water samples

Again, down-hole water samples from the field are preferred. If these are not available, the formation water can be prepared in accordance with its given chemical composition. Solubility index calculations on both CaSO_4 and CaCO_3 precipitates have to be carried out to establish the likelihood of scaling problems arising because of the elevated temperature of the water. Measures to alleviate this problem can be taken accordingly.

Core material

Core material is prepared in accordance with its nature and state. Preserved or native-state samples can be fixed in the core holder without being cleaned. Then, formation water is gently flooded through the sample, keeping the oil saturation as constant as possible, until a resistivity equilibrium is reached.

Alternatively, core material is first cleaned following standard procedures. To restore reservoir wettability conditions, the material can be aged with reservoir fluids in the RIMARC equipment (Hirasaki et al., 1990).

Procedure

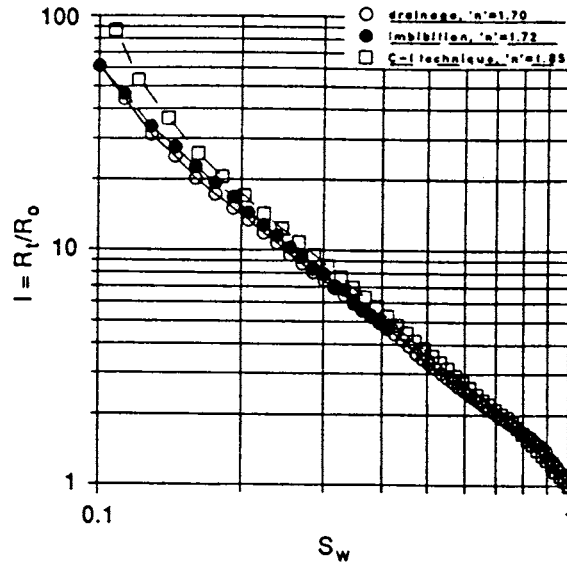
After the sample has been mounted in the core holder, water is flooded through until resistivity equilibrium is established. Subsequently, the injection fluid is changed over from water to oil. By withdrawing water from the sample, the oil phase is forced to enter the sample. As the first distinct change in resistance across the sample is observed, the data acquisition is started. The temperature of the cell, the injection pressure and the net effective stress are recorded continuously, while at any moment the water and oil saturations can be calculated from the readings from the Ruska metering pump.

Experiments

Measurements have so far been performed on cleaned, clay-free Bentheim sandstone samples. These experiments were primarily aimed at checking and calibrating the system.

At first a test sample was subjected to a standard CI measurement at room temperature and a net effective stress of 70 bar, in the equipment described earlier in this paper, using saline water (100 g/l NaCl) and kerosene as pore fluids. In this way a drainage $I-S_w$ curve was obtained. After the sample had been cleaned, this measurement was repeated in the RIMARC equipment.

In the next experiment, the drainage $I-S_w$ curve of a second sandstone sample with a permeability of 3.1 Darcy and a porosity of 24.0% was determined in the CI equipment at standard conditions and, after the sample had been cleaned, in the RIMARC equipment at a temperature of 92°C and an isostatic stress of 125 bar using the same pore fluids as in the first experiment. In the RIMARC equipment the drainage cycle was followed by an imbibition experiment. During this test the kerosene injection pressure was maintained at 55 bar, resulting in a net effective stress of 70 bar, equal to the net stress conditions in the CI equipment. Results of these experiments are shown in Fig. 8.



**FIGURE 8 RIMARC test at elevated pressure and temperature
Results and discussion**

The first measurements showed good agreement between the CI and the RIMARC results at room temperature. The second experiment in the RIMARC equipment at elevated fluid injection pressure and temperature resulted in an 'n' value of 1.70 for the drainage cycle and 1.72 for the (spontaneous) imbibition cycle (Fig. 8). The corresponding CI measurement, conducted under standard conditions, resulted in a much higher saturation exponent of 1.85. Since the net isostatic stress on the sample in this experiment was equal to that in the standard CI measurement, this change in 'n' is fully attributable to the temperature effect. This decrease in 'n' at higher temperatures is in line with the observations made by, for example, Søndena et al. (1990).

Future work

At present the RIMARC equipment is being upgraded to a computer-controlled continuous injection system.

Experiments are in preparation using live reservoir fluids, to study the effect of wettability changes (through ageing) on the resistivity index. A separate series of experiments is planned to investigate further the effect of stress and temperature on the measurement.

The ultimate goal of this work is a more accurate interpretation of resistivity logs and from this a more realistic calculation of oil and water saturations in the reservoir.

Conclusions

- (1) The continuous injection technique for resistivity index measurement gives accurate and precise results.
- (2) Confining stress at levels prevailing in the reservoir has an effect on the resistivity index for the subject sample set. The cause of this effect is as yet not quantitatively understood.
- (3) The first results in the RIMARC equipment show a temperature effect on the resistivity index. Experiments involving live reservoir fluids will be conducted in the near future to study the effects of wettability on the resistivity index.

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