# USING A DENSITOMETER FOR QUANTITATIVE DETERMINATIONS OF FLUID DENSITY AND FLUID VOLUME IN CORE FLOODING EXPERIMENTS AT RESERVOIR CONDITIONS

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#### ABSTRACT

A densitometer is used for quantitative density determinations of fluids being produced from core samples during flooding experiments at reservoir conditions. The densitometer is situated in the flow-line immediately after the core holder, and measures the density of all fluids being produced from the core sample at the actual Pressure/Temperature (P/T) conditions of the flooding experiment. In addition the densitometer provides timing information about dynamic events during the experiment, e.g. water breakthrough or gas breakthrough.

In the case of two-phase experiments, the densitometer may be used for determining the volumes of the two produced phases, if the density of each of the two fluid phases is known. This is the case in many flooding experiments using oil and water. In such cases the densitometer may provide data for the produced volumes of oil and water that agrees reasonably with fluid volumes determined by an acoustic separator. In complex and prolonged flooding experiments the densitometer volume determinations may provide an independent confirmation of the volume determinations of an acoustic separator or possibly other devices.

#### **DENSITOMETER SET-UP**

During core-flooding experiments at reservoir conditions it is important to keep track of the fluids being produced from the core sample. For this purpose a densitometer situated in the flow-line immediately downstream to the core sample has proved useful. The densitometer (Paar DMA HPM) has been used at GEUS for obtaining precise density measurements of the fluids being produced from core samples at temperatures up to 115 degree C and fluid pressures up to 420 bara [1]. However, the rating of the device allows use up to 200 degree C and 1400 bara.

The densitometer is situated in the flow-line immediately downstream to the core holder containing a core sample, and measures the density of whatever fluid mixture is produced from the core sample. After passing the densitometer, the fluid flow continues to an acoustic, two-phase separator (Nisep-200) that measures the produced fluid volumes. A water reservoir, the core sample, the densitometer, and the separator are all situated inside an oven with temperature control better than 1 degree C. Figure 1 gives a sketch of the set-up. Depending on the number of fluid phases in the experiment, the two-phase separator can be replaced by a three-phase separator situated outside the oven. The present paper is only concerned with experiments where the two-phase separator inside the oven is used.



## **USING THE DENSITOMETER**

Data from a core-flooding experiment, Exp-1, on a chalk sample from a North Sea oil reservoir is used to illustrate the use of the densitometer. The core sample was free of fractures, and was initially saturated with live crude oil and simulated formation water, while synthetic sea water was used as injection water for the water-flooding. The experiment was conducted at the P/T conditions of the North Sea reservoir. Figure 2 shows the density log obtained during the water-flooding. Several observations can be made from the log:

(1) Only oil with a constant density of  $0.7821\pm0.0001$  g/ml is produced from the core before water breakthrough.

(2) Water breakthrough occurs when the amount of injected water, *Vinj*, reaches 0.481 pore volumes (PV).

(3) After water breakthrough the measured fluid density fluctuates between a density that represents water and a density that represents a mixture of water and oil. This is best seen in the bottom plot of Figure 2 that shows a blow-up of a typical part of the log. It is seen that the majority of the density measurements reports a value around 1.0015 g/ml, which is the density of the injection water. With regular spacing occur narrow downward deflections of the density trace that usually reach down to a fluid density between 0.92



Figure 2. Density log obtained during water-flooding experiment of a chalk core sample at reservoir conditions. The fluids were recombined live oil, simulated formation water, and simulated injection water. The log contains 16785 density measurements. Water breakthrough occurs at Vinj= 0.481 PV. The top plot shows the complete log, the bottom plot, shows a small part of the log in detail.

and 0.965 g/ml. Each of these downward deflections represents an oil drop that passes through the densitometer. The density of the oil was constant during the oil-only production period before water breakthrough, and it is estimated that it remained nearly constant throughout the water-flooding. Whenever the volume of an oil drop entering the measuring cell is smaller than the sensitive volume of the measuring cell volume, the reported density is the average density of the fluids present in the cell. This effect is termed *the partial volume effect*. Because most of the downwards deflections of the density log only reach between 20% and 35% of the distance from the density of water to the density of oil, it is concluded that the associated oil drops only filled between 20% and 35% of the densitometer cell. The sensitive volume of the cell is approximately 0.35 ml and therefore most of the oil drops were between 0.07 ml and 0.12 ml.

(4) Immediately after water breakthrough the water density is considerably higher than the density of the injection water, i.e. 1.0245 g/ml vs. 1.0015 g/ml. The heavy water represents the connate formation water of the core sample being displaced by the injection water. It is seen that the displacement of the connate water is rather efficient, being completed before Vinj=0.79 PV. Similar behaviour of the connate water being mobilized ahead of the injection water has been observed in other studies, e.g. Brown [2], Nielsen et al. [3], and Korsbech et al. [4].

## **QUANTIFICATION OF PRODUCED FLUID VOLUMES**

In many experiments with flow of water and oil in tubes of more or less transparent materials, e.g. nylon or Teflon, it has been observed that the oil and water moves in sections of fluids separated by curved interfaces, Figure 3. Furthermore, it has been noted that for tubes with inner diameter between 1.0 and 1.6 mm, i.e. commonly used 1/8" tubes, oil and water do not seem to flow past each other, but instead moves as fixed "trains" (Figure 3). The curvature of the oil-water interface indicates that oil is the wetting phase, which agrees with Long et al. [5]. The measuring cell of the densitometer is Hastelloy C-276, and the wetting properties of this alloy against live oil-water are not known. However, because the density log of Exp-1 (Figure 2) and many other experiments show the characteristic movement in "trains", it is reasonable to assume that



**Figure 3.** Photo of a Teflon tube filled with alternating sections of water (colourless) and crude oil (very dark brown) during a flow experiment. The curvature of the oil-water interfaces shows that oil is the wetting phase. Note that the inner wall of the tube do not show any oil staining in the water sections, except adjacent to interfaces. The tube has an outer diameter of 3.2 mm (1/8 inch) and an inner diameter of 1.6 mm. Fluid flow was from right to left. While the photo was taken the flow was stopped. Pressure was 1 atm, temperature was 22 degC.

the movement of oil and water is similar in the densitometer, even though it cannot be verified visually. In this treatment it is assumed that water and oil cannot flow past each other in the tube.

A density log may with certain presumptions be used for calculation of a log of produced fluid volumes. The theory of the calculation of oil and water production from densitometer data is given below.

Consider a core flooding experiment conducted at constant pressure and temperature with two immiscible fluids. For ease of reference the fluids are referred to as water and oil, but the presented technique is considered generally applicable for immiscible fluid pairs. The flow rate of the experiment may vary, but is constant in most experiments.

Let *WF* be the fraction of water in a small volume, *dVol*, at a measuring site in the flow line as a function of the total injected volume, *InjVol*, at that site. The cumulative amount of water that has passed the measuring site, *CumProdWater*, is then

$$CumProdWater = \int WF \, dInjVol \tag{1}$$

For measurements in discrete steps from start at zero to end at N Eq. 1 becomes

$$CumProdWater(0, N) = \sum_{0}^{N} WF(i) * [InjVol(i) - InjVol(i-1)]$$
(2)

The conversion of Eq. 1 to Eq. 2 requires that the density measurements are made so often that every fluid molecule participates in at least one density measurement, i.e. [InjVol(i)-InjVol(i-1)] must be less than the measuring volume dVol. In the present work [InjVol(i)-InjVol(i-1)] is typically between 0.03 and 0.07 ml while dVol is 0.35 ml. Therefore each fluid molecule participates in between 5 and 12 density measurement.

Being a system with two phases separated by interfaces, i.e. a water phase and an oil phase, a measured density of the mixture is always intermediate between the density of the water phase and the density of the oil phase. WF at step *i* is then calculated as

$$WF(i) = \frac{MeasDens(i) - LowerEnvelope(i)}{UpperEnvelope(i) - LowerEnvelope(i)}$$
(3)

where *MeasDens* is the measured density of the mixture, *LowerEnvelope* is the density of the oil, and *UpperEnvelope* is the density of the water phase.

Combining Eqs. 2 and 3 gives

$$CumProdWater(0, N) = \sum_{0}^{N} \frac{MeasDens(i) - LowerEnvelope(i)}{UpperEnvelope(i) - LowerEnvelope(i)} * [InjVol(i) - InjVol(i-1)]$$
(4)

Because the experiment is conducted at constant pressure and temperature

$$CumProdOil(0, N) = CumInjFluid(0, N) - CumProdWater(0, N)$$
(5)

After defining the functions *LowerEnvelope* and *UpperEnvelope*, Eqs. 4 and 5 can be used to calculate the volumes of produced water and oil in the experiment. The functions *LowerEnvelope* and *UpperEnvelope* must be carefully considered. In many situations the fluid densities are readily available from the densitometer measurements, but in particular the *LowerEnvelope* densities may be difficult to obtain. Whenever possible, the pure fluids should be flowed through the densitometer to obtain the density of the pure fluids.

Because the fluid volumes determined by the densitometer and the separator are made at the same temperature and pressure they are directly comparable, without any P/T corrections.

## **EXAMPLE OF FLUID VOLUME QUANTIFICATION**

A log of produced oil volume calculated from the densitometer data of Figure 2 is given in Figure 4. Also given is a log of produced oil volume calculated from two-phase separator data. The two methods do not share any parameters, and are therefore fully independent volume determinations. The two oil volume traces are seen to match within 1.3% relative. Furthermore, the shapes are nearly identical. The only notable deviation is that the separator log lags a little below the densitometer log immediately after water breakthrough, as oil production according to the separator log seems nearly to stop. This is contrary to the densitometer data, where a significant oil production is documented in the time just after water breakthrough, cf. Figure 5. It is considered most likely that the





separator data are slightly biased by the influx of heavy formation water just after water breakthrough and that the densitometer data in this instance may actually be more precise.

The use of equations 1 to 5 requires that the lower density envelope and the upper density envelope are defined. The densitometer oil volume log of Figure 4 is based on a constant lower envelope density of 0.7821 g/ml, which is the very stable density of oil recorded before water breakthrough, cf. Figure 4. The upper density envelope is calculated from the densitometer log, and is shown as a red line in Figures 5 and 6. The calculation of the upper density envelope is automated by using an algorithm that identifies sections with nearly constant high density values, and interpolates linearly across the downward deflections of the density log. This is well seen is Figure 5.

The measurement of density in the densitometer and volume in the separator are done downstream to the outlet of the core sample with a separation between the units of several milliliters. In Figures 2, 4, 5 and 6 the densitometer log and the separator log have been shifted by the volume of the connecting flow lines. In this way the densitometer and separator data are shifted to correspond to the outlet of the core sample, i.e. events recorded by the densitometer and separator are shown in the diagrams at the value of the injection volume, *InjVol*, where the corresponding fluids left the outlet of the core sample. In this way the densitometer log and the separator log become directly comparable.



## DISCUSSION

The assumption that the fluid phases within the densitometer moves in "trains" without bypassing each other is crucial to the use of the densitometer data for calculation of produced fluid volumes. The main argument that this is the case comes from the fact that the measured fluid density returns exactly to the density of pure water after the passage of an oil drop, cf. Figure 2 bottom plot and Figure 5. If bypassing took place, an amount of oil would sometimes be present in the measuring cell in the intervals between passing oil drops. This is never seen in situations where the oil drops are clearly spaced as in Figure 2. In situations where the measured fluid density returns to the density of water after passage of every drop of oil, both two-phase flow and bypassing of immobile oil within the measuring cell is not possible, and the calculation procedure proposed in this paper is considered valid. In situations where the fractional flow of water and oil are approximately equal, the measured fluid density sometimes stays in an intermediate interval between the density of water and the density of oil (Figure 5). In this situation two-phase flow within the densitometer cannot be ruled out, and the calculation procedure may or may not be valid. In water-flooding experiments this situation has only been seen right after water breakthrough.

The measuring cell of the densitometer consists of a metal tube with an inner diameter of 2 mm (Anton Paar Nordic AB, personal communication). This is slightly larger than the Teflon tube shown in Figure 3, and it is possible that significant bypassing may take place in fluid systems with significantly lower interfacial tension than standard North Sea water-oil systems.

The specifications of the densitometer states that the "amount of sample in the measuring cell" is "approximately 2 ml" [6]. The stated cell volume of 2 ml is clearly larger than the sensitive volume of the measuring cell. At GEUS the sensitive cell volume has been determined to be  $0.35 \pm 0.03$  ml by observing the passage of a single water-oil interface through the cell, cf. Figure 7.



**Figure 7.** Density log of the passage of a water-oil interface through a Paar DMA HPM densitometer. The measured density starts changing at approximately Vinj=4.9 ml and ends at approximately Vinj=5.25 ml, indicating the volume of the sensitive part of the measuring cell to be approximately 0.35 ml. The density change is not linear as the interface passes through the sensitive volume, indicating that the sensitivity to fluid density varies through the sensitive volume, being largest at the center of the sensitive volume.

It is not known how the densitometer will perform at flow rates above 25 ml/h, and neither how it will perform with oil that are significantly heavier than typical North Sea oil. If a water-oil emulsion is produced, it is estimated that the quantitative volume calculation procedure will probably fail.

In general the densitometer has proved to be very reliable. However, it is susceptible to fines that settle in the measuring cell. Such incidents typically cause the densitometer trace to display a sudden offset towards higher density values. In such case the densitometer data cannot be used for quantitative determination of fluid volumes as the true density values become unknown. In some cases it is possible to flush the fines out of the measuring cell by briefly increasing the flow rate.

The densitometer at GEUS is mounted in a horizontal position. A vertical orientation with the closure of the densitometer U-tube pointing upwards has been considered as a means to avoid settling of fines within the measuring cell. The point should be that gravity then would prevent the fines from settling in the measuring cell. However, this has not been implemented at GEUS, because it seems to introduce a risk of oil collecting at the top of the U-tube.

## CONCLUSIONS

An in-line densitometer provides a method for obtaining reliable fluid density data continuously during core-flooding experiments at reservoir conditions. Furthermore, it provides precise timing information of breakthrough events. These applications are possible both for two-phase experiments and three-phase experiments.

In the case of two-phase experiments conducted at constant temperature and pressure conditions, data from an in-line densitometer allows calculation of a log of produced fluid volumes that is comparable in precision to data obtained from an acoustic separator. The application requires the logging time of the density parameter and the injected fluid volume parameter to be so short that every fluid volume molecule participates in at least one density measurement. This method is presently used at GEUS as a secondary method for calculating produced fluid volumes, with separator determination being the primary method.

## REFERENCES

- 1. Olsen, D., "CO2 EOR Production Properties of Chalk", SPE 142993 (2011).
- 2. Brown, W.O., "The Mobility of Connate Water During a Water Flood", *Trans. AIME*, (1957), **210**, 190-195.
- 3. Nielsen, C.M., Olsen, D., and Bech, N., "Imbibition Processes in Fractured Chalk Core Plugs with Connate Water Mobilization", SPE 63226 (2000).
- 4. Korsbech, U., Aage, H.K., Hedegaard, K., Lohmann Andersen, B. and Springer, N., "Measuring and modeling the displacement of connate water in chalk core plugs during water injection", *SPE Reservoir Eval. & Eng.* (2006) **9,3**, 259-265.
- 5. Long, Z., Shetty, A.M., Solomon, M.J., and Larson, R.G., "Fundamentals of magnet-actuated droplet manipulation on an open hydrophobic surface", *Lab Chip.* **9(11)**, 1567-1575 (2009).
- 6. Instruction Manual, DMA HPM, Document no. C34IB06A.fm, Anton Paar GmbH, Graz, Austria (2005).