ESTABLISHMENT OF VISCOMETER CAPABILITY FOR GEOPRESSURED FLUIDS

MASTER

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**Evelyn Rockar
Philip Randolph**

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**Prepared by
Institute of Gas Technology
IIT Center, 3424 S. State Street
Chicago, Illinois 60616**

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EXECUTIVE SUMMARY

This report describes work carried out with the IGT capillary viscometer to overhaul, modify, test, and calibrate it for the measurement of the viscosity of methane-saturated brines at 10,000 psi and 200°C. The viscometer had been originally designed to operate with hydrocarbon gases and liquids at up to 10,000 psi and 340°F (171°C).

The viscometer was cleaned, modified, reassembled and pressure tested. In tests carried out separately from the viscometer itself, most of the component materials were found to be compatible with brine, but some corrosion at the mercury surface may give trouble with the electrical timing device. Calibration of the density cells was only achieved with a precision of 6% to 7%, considered adequate for present purposes.

In operation, it was found that the turn-around time between runs was excessively long, the requirements for meticulously cleaning the equipment were very time-consuming, and problems were encountered due to gas bubbles becoming trapped in the mercury lines and the capillary. The best calibration value obtained with pure water was 6% below literature values.

Because of the difficulty encountered in making measurements, data on methane-saturated brine could not be obtained within the funding limitations of this program. Recommendations are made for future work, which include justifying why viscosity data are needed, defining the accuracy and precision required, and enumerating several options for the design and construction of a completely new apparatus for this purpose. We conclude that further work on the existing viscometer would not be cost-effective.

TABLE OF CONTENTS

	<u>Page</u>
I. INTRODUCTION	1
II. DETERMINATION OF VISCOSITY	3
A. Viscometers	3
B. Empirical Predictions of Viscosities	4
III. MEASURING THE VISCOSITY OF METHANE-SATURATED BRINE WITH THE IGT VISCOMETER	5
IV. DISCUSSION	9
V. RECOMMENDATIONS	11
REFERENCES CITED	14
APPENDIX. Operating Characteristics of the Viscometer	17

I. INTRODUCTION

Among the many unknown engineering parameters required for the analysis of reservoirs containing geopressured-geothermal brines is the dynamic viscosity of the brine with natural gas in solution. The dynamic viscosity of gas-free aqueous NaCl solutions at pressures up to about 4500 psi and at 150°C has been measured¹ with specific application to geothermal brines. Geopressured-geothermal brines, on the other hand, are found at pressures to 20,000 psi at 200°C, and they may be saturated with methane. The amount of methane in the brine decreases with the concentration of dissolved solids in the brine but increases with pressure and with temperature above a minimum at about 80°C. Data accumulated at this time² indicate that the amount of methane could exceed 50 SCF/bbl or approximately 0.7 mole percent, depending on pressure, temperature, and salinity. Although the value in mole percent may seem a small number, Dodson and Standing³ have measured the compressibility of oil field brines at 5000 psi, 250°F (171°C), and 35,000 ppm total solids, saturated with natural gas (88% CH₄) at 20 SCF/bbl, and found it to be 18% higher than that of the brine alone. On the other hand, they also found that the formation volumes of the gas-saturated water were only about 1% higher than those of pure water (no gas or solids) at 5000 psi and 250°F.

These data indicate that the viscosity of a gas-saturated brine might differ significantly (20%) from that of one with no dissolved gas.

An examination of the literature has turned up only two references to liquid viscosity measurements of liquid containing a gaseous component. One reference examined the effect of dissolved air on the viscosity of water at 20°C and 14.7 psi after compressing the system to 5250 psi.⁴ No effect was observed. The other reference (abstract only) examined the effect of dissolved gases on the viscosity of n-heptane.⁵ The temperature, pressure, and gases were not specified in the abstract. Nevertheless, the author reports that "a change in the concentration of the dissolved gaseous component has a considerable effect on the dynamic viscosity coefficient."

No empirical expressions have been developed at this time to estimate the viscosity of a liquid with a gaseous component. In fact, the empirical expressions for estimating the viscosity of mixtures of gases or mixtures of liquids at high temperatures and pressures are only claimed to be good to 15%

for systems in which the viscosities of the individual components are known. Thus, if we need to know the value of the viscosity of gas-saturated geopressured-geothermal brines, the only alternatives are to try to measure it or to make some simplifying assumptions about the contribution of the dissolved gas.

Unfortunately, the measurement would not be a simple one to carry out. We hoped to be able to make some preliminary measurements of viscosities in the Institute of Gas Technology (IGT) capillary viscometer at minimal cost to determine whether a more complete study of this measurement is warranted. The IGT viscometer is a modified Rankine capillary viscometer designed for measuring the viscosity of gases to 10,000 psi and 340°F. In the next section, we will discuss the materials and operating problems that we encountered, resulting in disappointing accuracy when calibrating with a known fluid, and resulting in our not being able to obtain data on methane-saturated brines within the funding limitations of the contract. Section IV contains a discussion of making viscosity measurements on this difficult system. Section V consists of recommendations for further experimental work in this area.

II. DETERMINATION OF VISCOSITY

A. Viscometers

There are several types of instruments designed for measuring viscosities. They are the capillary viscometer, the falling ball viscometer, the oscillating disk viscometer, and the rotating disk (or cylinder) viscometer. All four types of designs are absolute measuring devices. That is, if the dimensions of all of the critical components are known precisely and the density of the fluid is known or measured, the dynamic viscosity of the fluid can be computed. The units of dynamic viscosity are poise or Pa·S. If the density is not known, then a ratio of viscosity and density can be determined. This ratio is called the kinematic viscosity; its units are stokes or m^2/s . For petroleum reservoir engineering analyses, the dynamic viscosity is required. It is not sufficient to know the kinematic viscosity.

The fourth type of design, the rotating disk (or cylinder) relates dynamic viscosity to the torque required to rotate the disk through some angle. Usually the instrument is calibrated on some fluid with a viscosity that is approximately the same as the viscosity of the unknown fluid. Most commercial viscometers are of the rotating cylinder type and have widest application for fluids with higher viscosities, such as oils or polymer solutions.

In general, the most precise types of viscometers are the capillary and oscillating disk viscometers. Viscosities with these instruments can be measured to a precision of 0.1% or better. The critical determining factor is very often the precision with which the density of the fluid can be evaluated. For those measurements of viscosity that do not require a high degree of accuracy, the instrument can be calibrated with a fluid of known viscosity — usually water or a silicon oil for liquids and nitrogen for gases. Each of these types has been modified at one time or another to measure viscosities at extremes of temperature or pressure.

An excellent review of viscometer types and their theoretical and practical limitations is given by Bruges and Whitelaw.⁶ One conclusion they draw is that capillary viscometers should not be used near a fluid critical point. The implication for gas-saturated liquids is that a capillary viscometer should not be used near saturation because it is difficult to keep the gas in solution near the saturation pressure.

B. Empirical Predictions of Viscosity

The state of the art in making empirical predictions for viscosities of gas-saturated liquids is primitive to say the least.⁷ We know that, for highly saline aqueous solutions, the viscosity decreases with increasing temperature and is relatively insensitive to pressure changes below the critical point.¹ This behavior is very similar to that of pure water and, in fact, the viscosity of aqueous solutions could probably be scaled to that of water according to the change in fluid density brought about by the additional salt.

The situation for gas in solution, however, cannot be so simply handled. Take, for example, natural-gas-saturated pure water at 3000 psi and 220°F. The mole fraction of gas in solution is only 0.0024 (~17 SCF/bbl), which, according to Dodson and Standing³ would increase the compressibility of the fluid by 15% over pure water at the same temperatures and pressures. The density of gas-saturated water at those temperatures and pressures is 0.963,⁸ whereas that of pure water at the same temperature and pressure is 0.967.⁹ Because the data of McKetta and Katz⁹ is probably good to only 5% in this region, we could infer that there is essentially no change in the density of the water when saturated with gas at this temperature and pressure. The mole fraction of methane in solution could be at most 0.01,¹⁰ so we could also infer that the change in density of the gas-saturated fluid would not exceed 1%. The density of gas-saturated water seems to be more a function of temperature than of pressure, much like the viscosity of pure or saline water.

Based on the above discussion, the most that could be said about the viscosity of a gas-saturated brine is that it is at least 1% lower than that of the brine alone at the same temperature and pressure. The increase in compressibility observed by Dodson and Standing for gas-saturated brines leads one to suspect that the viscosity could be somewhat further reduced.

NOTE: There has been extensive effort in developing models for determining the viscosity of two-phase fluids.¹¹ We are concerned here with single phase solutions in which the solute is a gas.

III. MEASURING THE VISCOSITY OF METHANE-SATURATED BRINE WITH THE IGT VISCOMETER

The IGT viscometer is a capillary viscometer of the modified Rankine type. It was originally designed to measure the kinematic viscosity of hydrocarbon gases and liquids at pressures up to 10,000 psi and temperatures up to 340°F.¹² (For a description of the principles of operation, see Appendix.) When the densities of the fluids could also be measured, computed, or taken from other sources, the dynamic viscosities could be determined as well. For dense fluids (such as propane) the calculated dynamic viscosities were poorly correlated with the experimental values.¹³ Because this system capability seemed to exist already, it was proposed that this viscometer be used (with slight modifications) to measure the viscosity of a methane-saturated brine at 10,000 psi and 340°F. The tasks proposed were -

1. To procure and fabricate new system components
2. To overhaul the viscometer
3. To test and calibrate the viscometer.

Tasks 1 and 2 are completed. In these two tasks, the viscometer was disassembled and cleaned, components were pressure-tested or evaluated in other ways, and new components were purchased to bring the system up to specifications. Some aspects of this work were delayed because items were received late from manufacturers. Two no-cost time extensions were requested and granted to compensate for this delay.

Task 3 consisted of calibration and testing and had four objectives. The first objective was to evaluate the degree of brine reactivity with the system components. This evaluation was carried out in separate teflon-lined autoclave vessels to avoid possible damage to the viscometer components. From the evaluation we concluded that a) as long as oxygen is kept out of the system, the corrosion of the stainless steel is minimal; b) etching is observed on cut glass surfaces, and as the drawn glass surfaces do not seem to be affected, this etching should not be a problem; c) an opaque grey film developed on the mercury surface when the mercury was heated to 140°C (284°F) in the presence of a 60,000 ppm NaCl solution, a piece of 316 SS, and methane gas. The film seemed to be well-confined to the surface of the mercury and should not adversely affect the fluid flow characteristics. The interference of

the film with the behavior of the timing device at operating conditions remains to be evaluated, however.

The second objective of Task 3 was to develop a timing device which will operate in the presence of an electrically conducting fluid, i.e., aqueous NaCl. This device has been built and has been tested in a simulated mode. It essentially makes use of the natural emf that develops between the two electrodes and ground and amplifies that voltage, instead of imposing a voltage on the solution. The timing device also operates when measuring the viscosity of a nonconducting medium. The device has not been tested while the system is at a temperature and pressure for the several hours or days required to form gas-saturated brine. An insulating film may form on the surface of the mercury and preclude a measurement. Usually the contacts of the timing device were activated infrequently when the system was filled with deoxygenated water for several days. The surface of the mercury was found to be covered with patches of "scum" and the inconel electrodes seemed to have a dull "oxide" coating. Neither the "scum" nor the "oxide" was analyzed. These contaminants appeared even when the timing device was "off."

The third objective, of calibrating the density cells, has also been completed. The precision to which they can be calibrated is 6% to 7%. This calibration sets a limit on the precision of the viscosity measurements. It also sets a limit on the degree to which the gas solubility can be determined. After the fluid had been cooled in the density cells, the gas was to be expanded into calibrated volumes and the pressure in these volumes measured. The gas-to-water ratio could then be computed. The degree of saturation of the brine with gas can only be determined after the system has cooled down, because the density cells cannot be removed safely from the hot box while under temperature and pressure. The accommodation of at most six density cells in the system means that at most six different values could be determined during one run. The turn-around time between runs is at least 3 to 4 days; this allows for cooling down, refilling, and equilibrating the system.

The precision of better than 1% that has been claimed for the viscosity measurements previously made on hydrocarbon gases is based on the use of literature values for the gas densities at temperature and pressure.

The final objective of Task 3 was to calibrate the system as a whole and determine the viscosity of test solutions at various temperatures and pressures.

This final aspect of the project proved to be more time-consuming than anticipated. The calibration on a known fluid, in this case pure water, has been difficult. Some of the problems arise from the need for the system to be meticulously clean. Other problems seem to arise from the presence of bubbles in the capillary. Procedures to avoid this difficulty were attempted. The calibration on a known fluid is an extremely important activity; it would determine the degree of accuracy and precision of the viscosity values of test solutions. After attempting this calibration in various ways, we concluded the following:

- Cleaning the system is a tedious operation. Because small particles of whatever nature can remain suspended in the fluid to be measured and therefore clog the capillary, the system must be meticulously clean. This degree of cleanliness requires that most of the high-pressure connections be opened and the various components cleaned separately. It is not enough to merely circulate a solvent through the system and rinse several times.
- Filling the lines with the fluid to be measured and the mercury requires care. The system is first cleaned and flowed with dry nitrogen and then evacuated using a vacuum pump. The mercury lines of the system are filled in such a manner that no air bubbles are in the mercury lines. The same procedure is used to fill the fluid lines. Both filling operations are done slowly to prevent mercury from entering the fluid lines. Small beads of mercury have been observed to float on the water surface. A gas/fluid interface also exists in the saturation vessel. The possibility does exist that very small beads of mercury could be carried through the fluid lines and interfere with the measurement in one of two ways. The first way would be to clog the capillary; the second way would be to short either of the electrodes to the vessel. This second way is more likely to occur in the high-pressure "Conax" electrical feed-through where the leads are in very close proximity to the fitting and to each other.

Although several attempts were made at calibrating the system with water, the best value obtained was about 6% too low. It took a long time to obtain a few values, because of interruption for cleaning and keeping mercury confined to its vessels; this indicated that meaningful values could not be obtained even for CO₂ in water within the budget of the contract. The nature of the operating problems encountered indicated that the cost of a preliminary assessment with this particular instrument would be much greater than originally anticipated. Cost could possibly be as high as for constructing and operating a new facility designed to provide data over the full pressure and temperature range of interest for geopressured aquifers.

Considering the difficulty of making the measurements, it seemed appropriate to think about other possible ways of carrying them out. Some thoughts in this direction are presented in the following section, and recommendations are given in section V.

IV. DISCUSSION

Despite the difficulties to be encountered, viscosity measurements of geopressured-geothermal brines should be carried out. Viscosity measurements under extreme conditions of temperature and pressure are difficult enough to things further. It is as important to determine the density of the fluid and establish the quantity of dissolved gas in solution simultaneously as it is to determine the viscosity. At this time, a high degree of precision is not required for these measurements; 5% to 10% is adequate because other reservoir parameters are not known to a higher accuracy. For example, the results of Blount² on methane-saturated NaCl solution are claimed to be good to $\pm 5\%$. Actual reservoir fluids contain other salts and gases in solution, so for some time assumptions and approximations for each reservoir will have to be made regardless of the precision and accuracy of laboratory data from "simple systems."

Lowering the requirements for precision and accuracy of the measurements, however, will not make it significantly easier to obtain these simultaneous measurements at high temperatures and pressures.

Some of the difficulty in making very high-temperature (200°C) and high-pressure (20,000 psi) measurements with methane in solution can be overcome by examining other systems for which the gas solubility is higher. For example, the solubility of CO₂ in water at 1000 psi and 25°C is greater than that for methane at 10,000 psi and 200°C. Perhaps a commercial viscometer or a modified one could examine the effect of CO₂ saturation on the viscosity of water. However, most commercial viscometers require significant modification to measure viscosities less than 1 cP. Some previous experience¹⁴ at studying hot geothermal brines at atmospheric pressures with a modified commercial viscometer has been less than successful.

On the other hand, a design based on a commercial viscometer for measuring the viscosities of drilling muds at high temperature and pressure has been built by Sandia Labs. It operates at pressures to 12,500 psi and temperatures to 260°C.¹⁵ It measures viscosities to 300 cP in cP divisions and would obviously have to be modified to measure fluids whose maximum viscosities are 1 cP and could drop to less than 0.2 cP¹. The present version

of the Sandia mud viscometer uses Dow Corning Fluid 200-10 CS as the hydraulic fluid. Whether this or a similar hydraulic fluid can be used when the experimental brine is saturated with methane remains to be evaluated. An opaque and more dense fluid such as mercury could not be used in the viscometer with its present design. The hydraulic fluid "floats" on the mud and must be optically transparent. The presence of gas bubbles in the viscometer is known to affect its accuracy, so a brine would have to be less than saturated to be evaluated in this system. A means to evaluate the extent of gas saturation would have to be devised. The system could be calibrated with a low-viscosity fluid at low temperatures and pressures.

Another flow-through (i.e., capillary) viscometer could be assembled with calibrated constant-rate displacement pumps providing the fluid drive at a constant (known) flow rate. A precision differential pressure transducer could measure the pressure drop across the capillary, and the viscosity could be calculated using Poiseuille's equation. This is a much simpler version of the IGT capillary viscometer, which instead uses a mercury head for the drive and volume flow rate.

The density and solubility of the fluid would still have to be determined, and the fluid would have to be kept slightly undersaturated to keep the gas in solution. An alternative would be to flow the gas-saturated fluid through a porous plug of known permeability and calculate the viscosity of the fluid using Darcy's equation. In this last case, the density of the fluid would not need to be known. A drawback here is that it is not clear that the gas would stay in solution in the plug or that Darcy's law can be applied to a gas-saturated liquid.

Any of the above schemes would require sufficient time to make a series of careful measurements at a wide variety of saturations to establish the validity of the method. A capillary viscometer would, in addition, require meticulous cleanliness.

V. RECOMMENDATIONS

Following is a list of recommendations that address the problems raised in this report. They are in order of increasing cost and level of effort; each is accompanied by discussion of the advantages and disadvantages.

- A. Measure the viscosity of CO₂-saturated water and brine in a conventional or slightly modified existing viscometer. The viscometer should have a demonstrated capability of measuring fluid viscosities that are less than that of water at room temperature and pressure. For example, one like Professor Kestin's (oscillating disk) at Brown University might be modified and calibrated to make and maintain CO₂ in solution. The pressure and temperature capability is more than adequate for CO₂ in solution. The solubility of CO₂ in water as a function of temperature and pressure is well-known as is the density of the solution. Therefore, after equilibrium was assured, separate solubility and density values would not have to be determined.

The drawback of this approach is that values directly available for reservoir engineering in geopressured aquifers would not be forthcoming. The effects of gas in solution on viscosity might allow some empirical extrapolations to be made.

The level of effort required is difficult to assess, but any program should be broken into two tasks.

1. Demonstrate the capability of measuring the viscosity of a fluid with gas in solution.
2. Make measurements over a prescribed temperature and pressure range.

Task 2 should not be funded until Task 1 has been satisfactorily completed. Depending on the starting capability, Task 1 would require a minimum of one-half of a man-year. Equipment expenses should be minimal. Task 2 could take up to another one-half of a man-year.

- B. Measure the viscosity of a methane-saturated brine in an apparatus that measures permeability at elevated temperatures and pressures. Use cores whose permeability is known (capillary bundles, Berea sandstones, etc.). In this case, the density of the fluid is not required, but the degree of solubility must be known for the measurement to have any meaning.

The advantage here is that determined values of viscosity could be used immediately in reservoir engineering calculations. The disadvantage is that it is a completely unproven approach. For that reason, to minimize costs, the system capability of measuring absolute permeability and solubility should be previously established. Several existing systems come close to having the above capabilities. They include those developed by Professor Somerton at the University of California at Berkeley; Professor Gray at the University of Texas, Austin; Terra Tek, Inc.; and IGT. The first two systems do not currently have the capability of flowing a liquid with gas in solution at high temperatures and pressures. The first three

cannot use brines at high temperatures because the steels of which they are made are not corrosion-resistant enough. The last system can in principle perform the measurements, but they have not been carried out.

Therefore, the task work should proceed as follows:

1. Establish the capability of maintaining gas in solution at elevated temperature and pressure. Be able to determine the degree of solubility.
2. Measure the viscosity by flowing the fluid through a core of known permeability. Demonstrate that the gas remains in solution and that Darcy's law is applicable in this situation.

Unless the system in question has been used for gas-saturated fluids previously, Task 1 could take considerable time — as much as 6 to 8 man-months. Similarly, Task 2 could take as long as 1 man-year. The savings is made here because the systems in question are 90% to 100% equipped and no major equipment needs to be built or purchased. The time involved could be cut down again by examining CO₂ in solution first. The lower temperature and pressure requirements give a faster turn-around time in the debugging processes.

C. A third approach is to start from scratch to build a high-pressure, high-temperature absolute viscometer for gases in solution. A system like the Sandia mud viscometer could be constructed. Although density of the fluid would not be required, the degree of solubility again is important. The task work would proceed as follows:

1. Design the viscometer.
2. Construct the viscometer.
3. Test the viscometer using liquids with gas in solution.
4. Make measurements of methane-saturated brine solutions.

Task 1 would require at least 6 man-months, including a complete study of the state of the art viscosity measurements, with visits to laboratories currently making viscosity measurements. The cost of building the viscometer would be greater than the cost of Professor Kestin's recently built viscometer or the cost of the Sandia mud viscometer. This is due to the additional hardware required to dissolve gas in the high-pressure, high-temperature brine and measure the concentration. Task 3 again could take at least 6 man-months to establish credibility, perhaps longer. The level of effort in Task 4 depends on the complexity of the measurements and the number to be made. About 1 man-year should be allowed.

The above estimates for all three recommended routes are approximate, but the difficulty of making measurements with gas in solution cannot be understated. For this reason, the lower temperatures and pressures allowed by examining a CO₂-

9/80

61024

water system should be a part of any investigation. The measurements are so difficult to make that approaching the whole problem by supporting recommendations A and B or C would provide important validation of results.

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9/80

61024

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APPENDIX. Operating Characteristics of
the Viscometer

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The IGT viscometer consists of two reservoirs connected by two horizontal capillary tubes. (See Figure A-1.) The bottom capillary tube and about half the reservoir volume are filled with mercury. The high-pressure sample fills the top capillary tube and remaining reservoir volume. The entire assembly is an oven. Viscosity is calculated from the rate of mercury and sample flow between reservoirs when the assembly is tilted.

The viscometer components are mounted on a rigid bedplate, which has a pivot point on the vertical axis of the receiver. The reservoir end of the instrument is raised or lowered to obtain the various operating positions. After the proper amount of mercury has been charged to the system, the test fluid is charged to the instrument with the bypass valve open and the bed essentially level. With the bypass open, the reservoir is successively raised and lowered to pass the sample back and forth, assuring homogeneity.

The reference level is established by raising the reservoir so that the mercury just touches the short receiver electrode, with the reservoir electrode still covered a few millimeters (position B). To make a run, the reservoir is lowered (position C) to let mercury flow to the left and test fluid to the right. Then the bypass valve is closed and the reservoir raised rapidly to give a predetermined mercury driving head (position D).

At this time there is some mercury flow before the test fluid begins to flow. This compresses the fluid in the receiver and expands that in the reservoir. Then the test fluid flows through the capillary under constantly decreasing mercury head and pressure difference. The time for the run is that required for the mercury to rise from the top of the lower receiver electrode to the tip of the upper electrode. The volume of mercury flow is determined from the bore of the receiver.

The reservoir elevation is determined so that the Reynolds number of the flow in the capillary is always safely within the laminar range. With proper precautions, any single-phase fluid can be run — gas, liquid, or dense fluid. Fluids only slightly outside the phase boundary or just barely above the critical point have been tested successfully with reproducible results.

Figure A-1. SCHEMATIC OPERATION OF VISCOMETER

The equation for viscosity used in this viscometer is:

$$\mu_f = \frac{\pi g r_c^4 \theta_E}{8 L_c V_D} \left[\frac{\Delta h_{LM} (\rho_m - \rho_f) g}{g_c} - \frac{\rho_f V_D^2 \beta}{g \pi r_c^4 \theta_E^2} \right] - \frac{L_m r_c^4 \mu_m}{r_m^4 L_c}$$

where

- g = acceleration due to gravity
- L_c = length of capillary
- L_m = length of mercury flow line
- r_c = radius of capillary
- r_m = radius of mercury flow line
- V_D = total volume of fluid displaced
- β = constant for entrance effect correction
- Δh_{LM} = mercury elevation difference between reservoir and receiver
- θ_E = experimental flow time
- μ_f = viscosity of fluid
- μ_m = viscosity of mercury
- ρ_f = density of fluid
- ρ_m = density of mercury

Units are cgs.

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