Improved High Pressure Capillary Tube Viscometer

B. E. EAKIN
JUNIOR MEMBER AIME
R. T. ELLINGTON
MEMBER AIME

INSTITUTE OF GAS TECHNOLOGY
CHICAGO, ILL.

ABSTRACT

An apparatus and a procedure for determining the viscosity behavior of hydrocarbons at pressures up to 10,000 psia and temperatures between 77 and 400° F are described. The equipment is suitable for measuring viscosity of either the liquid or vapor phases or the fluid above the two-phase envelope for systems exhibiting retrograde phenomena, according to the phase state of the system within these ranges of temperature and pressure. Equations are developed for calculation of viscosity from the experimental measurements, and new data for the viscosities of ethane and propane at 77° F are reported.

INTRODUCTION

With the advent of higher pressures and temperatures in industrial processes and deep petroleum and natural gas reservoirs, demand has increased for accurate values of physical properties of hydrocarbons under these conditions. Proportionately, more frequent occurrence of natural gas and condensate-type fluids is encountered as fluid hydrocarbons are discovered at greater depths. This increases the importance, to the reservoir engineer, of being able to predict accurately the physical properties of light hydrocarbon systems in the dense-gas and light-liquid phase states.

Reliable gas viscosity data are limited primarily to measurements made on pure components near ambient temperature and at low pressures. Few investigations have been reported for high pressures, and except for methane, data on light hydrocarbons are subject to question. This is demonstrated by the large discrepancy between sets of data on the same component reported by different investigators. For mixtures in the dense gas and light liquid regions and for fluids exhibiting retrograde behavior there are very few published experimental data.

Viscosity data for methane have been reported by Bicher and Katz,[1] Sage and Lacey,[12] Comings, *et al*,[5] Golubev,[8] and Carr,[2] with good agreement among the last three sets of data. Comings, Golubev and Carr utilized capillary tube instruments for which the theory of fluid flow is well established. The theory permits calculation of the viscosity directly from the experimental data and dimensions of the instrument alone. Sage and Lacey, and Bicher and Katz used rolling-ball viscometers. The theory of the rolling-ball viscometer has not been completely established, and these instruments presently require calibration by use of fluids of known viscosity behavior before viscosities of test fluids can be measured. To obtain accurate data it is necessary that the rolling-ball viscometers be calibrated by use of fluids of density and viscosity similar to the test fluids, a difficult selection for the gas phase.

From the methane data and experimental tests on various natural gases, Carr developed a correlation for predicting the PVT behavior of light natural gases.[2,3,4] This correlation was based on data for a very limited composition range; its application to rich gases and condensate fluids is questionable.

The object of this investigation is to develop an instrument which can be used to obtain viscosity data at reservoir temperatures and pressures, for rich gases, condensate-type systems above the two-phase envelope and light liquid mixtures. These data will be used in an effort to develop correlations to represent the viscosity behavior of these fluids.

APPARATUS

In a previous viscosity study Carr[2] utilized a modified Rankine capillary viscometer configuration,[11] Fig. 1. In this instrument the gas to be tested is forced through the capillary tube in laminar flow by motion of a mercury pellet in the fall tube, the measured displacement time being that required for the mercury slug to move between the brass timer rings. The viscometer is constructed of glass and mounted in a steel pressure vessel. The test gas pressure in the viscometer is balanced by an inert gas (usually nitrogen) in the vessel.

Excellent results have been obtained with instruments of this type, with Carr[2] and Comings[5] reporting reproducibilities of 99.5 to 99.3 per cent and an estimated absolute accuracy of 99 per cent. However, these instruments have limitations which have precluded their use for liquids. The need for maintaining a balance between pressures of the test fluid and inert gas in the viscometer vessel presents operating problems, and requires charging the test fluid to the viscometer very slowly. The principle drawback to the Rankine unit is behavior of the mercury slug which provides the pressure differential across the capillary. When even trace quantities of propane or heavier hydrocarbons are present in the test gas, the mercury tends to subdivide

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[1]References given at end of paper.

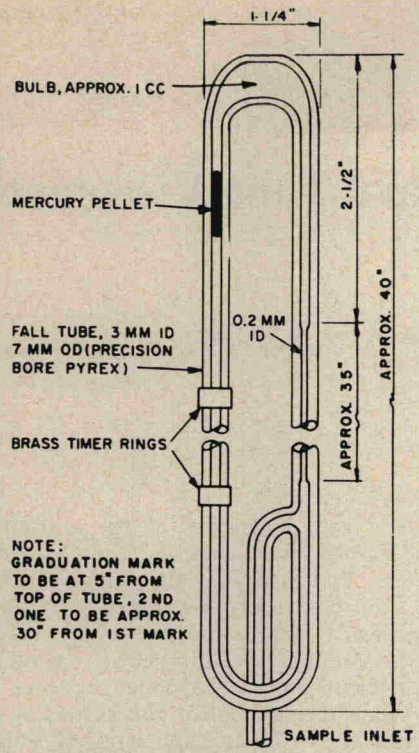

FIG. 1—DETAILS OF RANKINE-TYPE CAPILLARY TUBE VISCOMETER.

into small pellets which cannot be made to recombine in the fall tube; this necessitates disassembling and cleaning the viscometer.

A study was made of the major types of viscometers reported in the literature,[8] and it was decided to base the new design on the principle of the Rankine viscometer, i.e., transpiration of the fluid through a capillary tube, but to devise a new method for providing the pressure differential. Accordingly, glass models of several different configurations were constructed and tested until a relatively simple apparatus was developed.

A schematic diagram of the new viscometer and associated equipment is presented in Fig. 2. The principle components of the instrument are: mercury receiver, K; mercury reservoir, N; high pressure swivel joints, H; glass capillary tube, L; capillary tube jacket, M; by-pass valve, J; and the mercury flow tube. The mercury receiver is fixed in position while the reservoir is free to move in a vertical plane.

The mercury reservoir and receiver were machined from 3.125-in. diameter 316 stainless steel round stock, and with tops in place are 3.688-in. high. Both vessels were bored to give chambers 1.0000 ± 0.0002-in. diameters by 1.625-in. deep, and are equipped with O-ring closures. The receiver body is tapped near the bottom

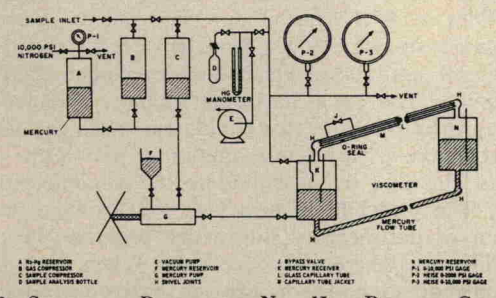

FIG. 2—SCHEMATIC DIAGRAM OF NEW HIGH PRESSURE CAPILLARY TUBE VISCOMETER.

for two 0.25-in. high pressure "Amico"-type fittings, and two fittings are also provided in the caps. The receiver cap is also drilled to receive two electrodes. The reservoir has only one fitting in the body and one in the cap, and the cap is drilled for only one electrode.

The simplified electrode assembly,[15] shown in Fig. 3, was developed to facilitate accurate location of the electrodes, and minimize chance of dimension change during operation of the apparatus. This is very important since the calculated volume of fluid displaced and the change in driving force during the run are both based on the measured distance between receiver electrode tips. The long receiver electrode extends to within about 0.5 in. of the bottom, thus providing a volume below the electrode to permit flow conditions to stabilize before flow timing begins.

When the rising mercury surface contacts the long receiver electrode the timer starts, and when the mercury contacts the short electrode, the timer stops. The spacing between the electrode tips, measured to ± 0.0003 cm by use of a microcomparator, is about 1.45-cm (9/16 in.). The volume above the short electrode was provided to contain the mercury in the flow tube and eliminate the possibility of mercury getting into the upper swivel joint or the capillary tube. The reservoir electrode extends to within about 0.125 in. of the bottom, and is used in adjusting the volume of mercury in the system.

The receiver is rigidly fixed in place with the reservoir free to move in an arc of radius equal to the length of the mercury flow tube. The flow tube and capillary jacket are attached to the receiver and reservoir by 0.25-in. high pressure swivel joints, illustrated schematically in Fig. 4. The reservoir will remain upright as it is raised, since the mercury flow tube and the capillary tube jacket assembly act as parallel linkages. In this manner the pressure differential across the capillary tube (which is equivalent to the difference in levels of the mercury surfaces in the reservoir and the receiver) can be varied from 3 to 40 cm by raising the mercury reservoir to predetermined positions.

The mercury flow tube is a length of 0.25-in. OD, 0.125-in. ID, stainless steel tubing, rated for 15,000-psi working pressure. The capillary tube jacket is 9/16-in. OD, 0.25-in. ID, stainless steel tubing, also rated for 15,000 psi. The annulus between the capillary tube and the tube jacket is utilized as a fluid by-pass to permit rapid transfer of the test fluid between the reservoir and the receiver. This provided a means for rapid return of the mercury to the reservoir after a test run, and also can be used to insure homogeneity of the test fluid. The annulus is sealed at the receiver end by a 7/32-in. ID, 11/32-in. OD, O-ring fitted into a standard 9/16-in. tee utilizing the tube jacket as a follower. The by-pass valve is connected across the O-ring annulus seal, as shown in detail in Fig. 5.

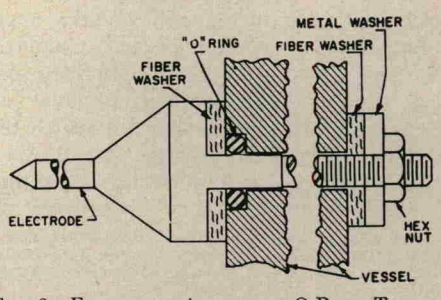

FIG. 3—ELECTRODE ASSEMBLY, O-RING TYPE.