

Measuring PVT Characteristics of Base Oils, Brines, and Drilling Fluids Under Extreme Temperatures and Pressures

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Abstract

Quality correlations for the thermal expansion and compressibility of liquid components used in drilling fluids are necessary to accurately predict static and dynamic downhole pressures. However, data suitable for deepwater, HTHP, and other wells that experience extreme temperatures and pressures are fairly inaccessible, due at least in part to the lack of functional and affordable PVT test equipment. These issues are being addressed by a recently commercialized pycnometer offered as an add-on module to that company's ultra-HTHP rheometer. The primary focus of this paper is to evaluate the accuracy and usability of their equipment over a wide range of temperatures and pressures.

The add-on module is suitable for testing base oils, brines, and fully formulated synthetic, oil, and water-based drilling fluids. The equipment is designed to measure liquid density changes at temperatures from ambient to 600°F, and pressures from atmospheric to 30,000 psi. A large chiller can lower temperatures to as low as 20°F to test fluids for use in deepwater, Arctic regions, and other cold environments.

Results and recommendations from this independent evaluation of the equipment and test procedures are presented in the paper. Test data and correlations from several widely used drilling fluid liquid components also are included for comparison to those published in the literature.

Introduction

The primary barrier for well integrity is the column of drilling fluid that provides the hydrostatic pressure required to overbalance formation pressures. While the impact of temperature and pressure on the downhole density of drilling fluids is now well accepted,¹ prediction of hydrostatic pressure can be challenging, especially for synthetic and oil-based drilling fluids and all fluids used in HTHP and deepwater environments.²

Engineers responsible for these calculations during well planning activities require correlations for specific base fluids, as these are necessary inputs to an appropriate density model to calculate downhole drilling fluid densities. Unfortunately, compressibility and thermal expansion relationships for the wide range of base oils, synthetics, and brines in use today to formulate drilling fluids are not readily accessible. Likewise, PVT equipment is not often available to routinely test field muds on jobs where well integrity could be critical. This is somewhat surprising since volumetric measurements under elevated temperature and pressure are commonplace in other segments of the petroleum industry.

Table 1 lists some of the better-known published resources for drilling-fluids-related data in this subject area. As noted in the table, the data were derived either from the technical literature, calculated using equations of state, or measured in

Table 1: Data from Industry Publications Summarizing PVT Studies

Year	Reference	Equipment	Tmin (°F)	Tmax (°F)	Pmax (psi)	Test Fluids
1982	Hoberock, <i>et al.</i> ⁴	Derived only	75	575	25,000	Water, sea water, saturated salt water, diesel
1982	McMordie, <i>et al.</i> ¹	Autoclave	70	400	14,000	WBM, OBM
1990	Peters, <i>et al.</i> ⁵	Blind PVT cell	78	350	15,000	Diesel, 2 mineral oils
1996	Isambourg, <i>et al.</i> ⁶	PVT cell	68	392	20,300	OBM, CaCl ₂ , mineral oil
2000	Zamora, <i>et al.</i> ⁷	Huxley-Bertram viscometer	70	400	14,500	LVT 200, C ₁₆ C ₁₈ LAO, Saraline 200, EMO-4000
2005	Hemphill and Isambourg ⁸	Reference only	40 - 77	302 - 400	20,000 - 24,000	CaCl ₂ , diesel, mineral oil, IO, paraffin
2006	API RP-13D ³	Reference only	40 - 77	302 - 400	20,000 - 24,000	CaCl ₂ , diesel, mineral oil, IO, paraffin
2007	Demiral, <i>et al.</i> ⁹	Mercury-free PVT cell	80	280	5,000	n-Paraffin-based oil, mud
2007	Demirdal and Cunha ¹⁰	Mercury-free PVT cell	77	347	14,000	C ₁₆ C ₁₈ IO, C ₁₂ C ₁₄ LAO
2010	Hussein and Amin ¹¹	Vapor-Liquid Equilibrium	80.6	300	5,000	Vegetable oil, mineral oil, blend of the two
2012	This work	PVT pycnometer ¹⁵	40	500	30,000	CaCl ₂ , diesel, C ₁₆ C ₁₈ IO, undecane, water

mostly customized calibrated-screw or variable-volume HTHP autoclaves. Some of data were measured on fully formulated drilling fluids, while other data were taken on various liquids used to formulate water, oil and synthetic-based drilling fluids. The last row in the table summarizes the data provided in this paper.

Comparisons among the data sets are not straight forward. In some cases, for example, generic names such as “mineral oil” are used for some of the base fluids. Moreover, even fluids properly described can behave differently depending on sourcing and those supplied by the same vendor can vary by batch. Also, most of the data were taken on customized equipment that could interject unexpected variability.

Some of the references in Table 1 provided regression analyses of their data that permit others to model downhole hydrostatic pressure. Table 2 is a copy of a table in an API recommended practice³ that gives constants and test conditions for five commonly used fluids. The three pressure and three temperature coefficients are matched to the following 2nd order polynomial equation:

$$\rho_{\text{base or brine}} = [(a_1 + b_1 P + c_1 P^2) + (a_2 + b_2 P + c_2 P^2) T] \quad (1)$$

Units for this equation are ρ (lb_m/gal), temperature (°F) and pressure (psi). Units for the constants are consistent with the other variables. Eq. 1 constants were determined by curve fitting data based on the volumetric behavior of base oil (or synthetic) and brine exposed to a range temperatures and pressures.

The API equation and table were reproduced from a paper⁸ that provided limited information on the data sources. Some confusion has arisen over the constants because an earlier paper⁷ used the same polynomial equation arranged differently so that their six constants are out of sync with Eq. 1. This

means that the correlation constants must always be paired with the equation they are based on.

The primary goal of this paper is to provide an evaluation of the test procedures and functionality of a recently commercialized, lower-cost device offered as an add-on module to an ultra-HTHP rheometer offered by the same company. The variable-volume module has been designed to measure volumetric behavior of base oils, brines, and synthetic, oil, and water-based drilling fluids at temperatures from ambient to 600°F, and pressures from atmospheric to 30,000 psi. Reportedly, a chiller can lower temperatures as low as 20°F to test fluids for use in cold environments. Comparisons are made to data available in the literature, although exact matches are virtually impossible in some cases.

PVTP Equipment

The PVT Pycnometer (PVTP) under evaluation was developed by Grace Instruments to measure volumetric changes of liquid and solid samples under conditions of temperature and pressure over a period of time.¹² The add-on module replaces the rotor/bob assembly in their ultra-HPHT viscometer as shown in Fig. 1. This could be a low-cost option for users that already have this particular HTHP viscometer.

Figs. 2a and 2b are schematic drawings of the PVTP module with two different pistons. All the tests in this series used the large piston. Samples can be tested without contamination by pressurization fluid. The unit can be configured with a large or small piston depending on the expected volumetric change of the test sample – the large piston for larger sample changes and the small piston for increased accuracy when testing samples not expected to change volume significantly. The large piston was used for the most part because of the fluid types tested in this evaluation. The internal components are shown in Figs. 3a and 3b.

Table 2: Correlation Constants to Match Eq. 1 for Common Base Fluids^{3,8}

	Calcium Chloride 19.3 wt%	Diesel	Mineral Oil	Internal Olefin	Paraffin
Reference	[6]	[5]	[6]	Chevron Phillips	Total
Pressure Coefficients					
a ₁ (lb _m /gal)	9.9952	7.3183	6.9912	6.8358	6.9692
b ₁ (lb _m /gal/psi)	1.77 E-05	5.27 E-05	2.25 E-05	2.23 E-05	3.35 E-05
c ₁ (lb _m /gal/psi ²)	6 E-11	-8 E-10	-1 E-10	-2 E-10	-5 E-10
Temperature Coefficients					
a ₂ (lb _m /gal/°F)	-2.75 E-03	-3.15 E-03	-3.28 E-03	-3.39 E-03	-3.46 E-03
b ₂ (lb _m /gal/psi/°F)	3.49 E-08	7.46 E-08	1.17 E-07	1.12 E-07	-1.64 E-08
c ₂ (lb _m /gal/psi ² /°F)	-9 E-13	-1 E-12	-3 E-12	-2 E-12	2 E-13
Fitting Statistics for Modeled Data					
Avg. Error %	0.135	0.237	0.166	0.194	0.214
r ² coefficient	0.998	0.997	0.998	0.998	0.999
Range of Validity					
Max. Applied Pressure (psi)	20,300	20,000	20,300	24,000	14,500
Min. Temperature (°F)	77	40	77	56.4	68
Max. Temperature (°F)	392	400	392	392	302

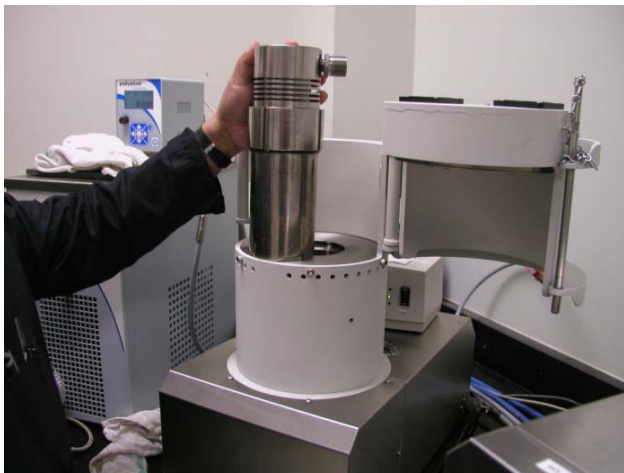
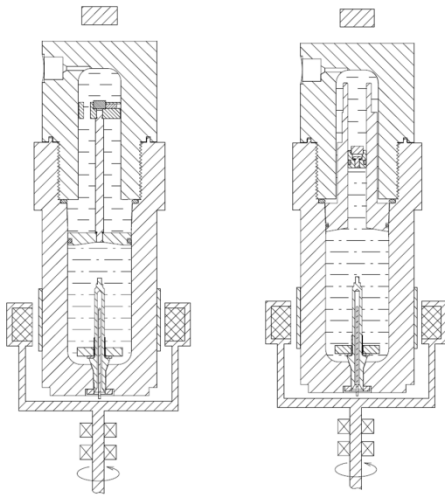


Fig. 1: PVTP ready for installation in the HTHP viscometer pressure tower



Figs. 2a-b: Schematic drawings of the PVTP with large piston shown installed on the left and small piston shown installed on the right.



Figs. 3a-b: Full set of internal PVTP components and head installation with the large piston in place.

Specifications on the PVTP from the manufacturer are presented in Table 3. The temperature and pressure ranges match those of the host HTHP viscometer. Note that the device can run at cold temperatures if a proper chiller is available.

Table 3: PVT Pycnometer (PVTP) Specifications

Resolution: 0.5% of initial density
Density Range: 71 to 142%
Sample Size: Small piston: 185 to 193 mL Large piston: 120 to 170 mL
Temperature Range: Ambient (20°F w/chiller) to 600°F
Pressure Range: Atm to 30,000 psi
Maximum Compressibility: 71% @ 170 mL starting volume (50% optional)
Maximum Expandability: 142% @ 120 mL starting volume (200% optional)
Stirring magnet: 0 to 600 rpm
Computer Requirements: Win PC

The following test procedure was supplied by the manufacturer:

1. Carefully measure test sample density at room temperature. Also record the sample temperature.
2. Ensure pressure vessel bottom cell is clean, dry and close to the temperature that the sample density was measured.
3. Weigh proper amount of sample to pour into pressure vessel. The sample volume should be around 140 mL. Sample volume can be slightly altered if the volume is expected to change significantly during testing.
4. Carefully install piston on top of test fluid and inside the pressure vessel. Make sure to replace all piston o-rings after each test. Also ensure that the magnet on top of the piston is aligned properly according to the user's manual.
5. Pour some extra pressurization oil on top of the sample piston. Screw on the pressure vessel top with o-ring and backup ring installed.
6. Install pressure vessel in pressure tower.
7. Press front keypad "purge" button until oil comes out from the drain without any bubbles.
8. Close the front bleeding valve. Press front keypad to exit "purge" mode. The front LCD should read between 1,000 and 2,000 psi.
9. Start PC software and go into "Simple Test" screen.
10. Set pressure target to 200 psi, temperature to 75°F or room temperature, and speed at 0 rpm. (Note: the purpose of Steps 10 to 12 is to minimize trapped air in the initial test sample.)
11. After pressure reaches 200 ± 50 psi and temperature is within 3°F of room temperature, stop the Simple Test and exit the Simple Test screen.
12. Start the Real-Time Test screen and zero the piston position to the 200 ± 50 psi condition.
13. Load desired test sequence to start test. During the test, ensure that the pressure does not drop below 200 psi during test since this is the initial density point.

Validation Tests

Deionized water and undecane were among the fluids selected to validate results from the PVTP. Fig. 4a shows PVTP-measured isothermal data for water, and Fig. 4b superimposes measured data at three temperatures over those from the literature¹² plotted by Hoberock, *et al.*⁴ The match is acceptable, considering that others reportedly have encountered issues with the literature data. The density variable in these graphs usually is presented in g/mL; however, units of lb_m/gal are used throughout this paper to be consistent with the units adopted in API RP 13D.³

Undecane is a liquid paraffin (CH₃(CH₂)₉CH₃). It is a component of diesel fuel, and readily available in high purity. Its purity can be checked with a simple freezing point determination. Conversely, its freezing point is lower than the freezing point of seawater, which allows testing over the range expected for deepwater risers from tropical to arctic waters. For undecane, Fig. 5 shows that the density measured by the PVTP is in good agreement with the literature¹³ at 86, 300, and 480°F and up to 30 kpsi. There is some small deviation at low and very high pressures, possibly due to dissolved air.

Experimental Results

The temperature/pressure test matrix is summarized in Table 4. The initial plan was to test each fluid only over the range for which it is normally used. For example, there was no need to test at very high temperatures a C₁₆C₁₈ IO commonly used in deepwater drilling. This plan was changed and every attempt was made to test all fluids over the full range.

P (psi)	T (°F)					
	40	77	150	250	400	500
200	X	X	X			
500	X	X	X			
2,500				X	X	X
5,000	X	X	X	X	X	X
10,000	X	X	X	X	X	X
15,000	X	X	X	X	X	X
20,000	X	X	X	X	X	X
25,000		X	X	X	X	X
30,000			X	X	X	X

Fluids selected to report in the paper are listed in Table 5, with the initial intent to compare results with the correlations presented in API RP 13D³ in Table 2. However, only the CaCl₂ brine could be compared directly.

Diesel used in this test series was a red-dyed diesel #2, low-sulfur, fuel oil. The mineral oil, internal olefin blend synthetic, and paraffin blend in Table 2 unfortunately were not sufficiently described. These materials can vary in composition and behavior. Therefore, some differences should be expected. LVT 200 was selected as the mineral oil tested and C₁₆C₁₈ IO was tested for the internal olefin. A paraffin blend was not tested in this series, so correlation constants for water were substituted in Table 5.

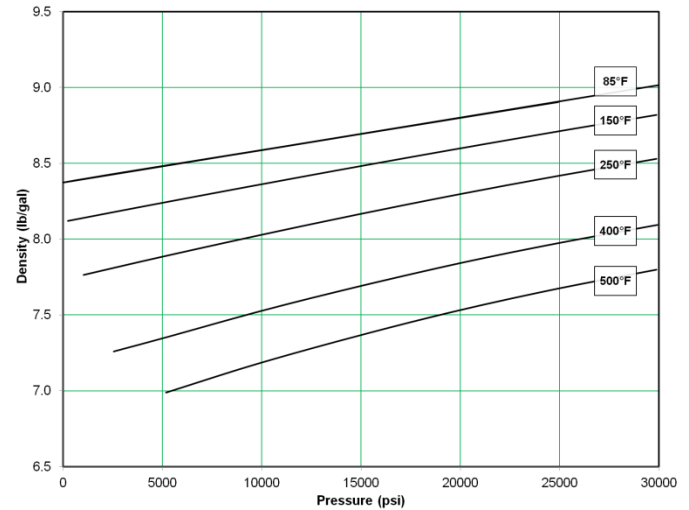


Fig. 4a: PVT data for deionized water measured with the PVTP.

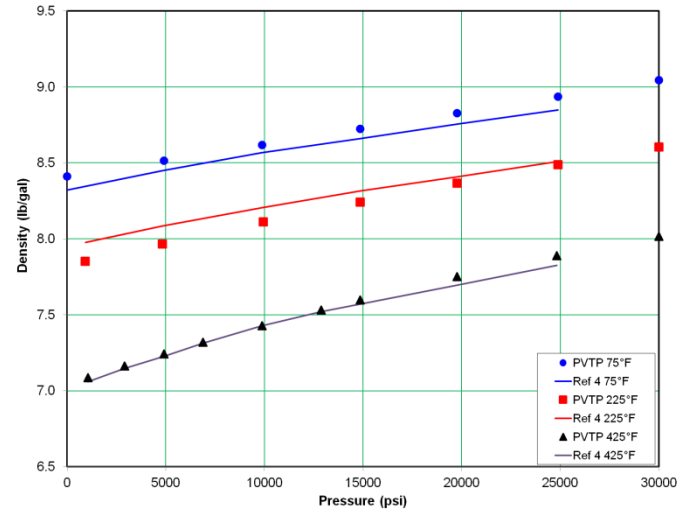


Fig. 4b: Comparison of PVTP-measured deionized water data with Refs 4 and 12 at three different temperatures and pressures to 25 kpsi.

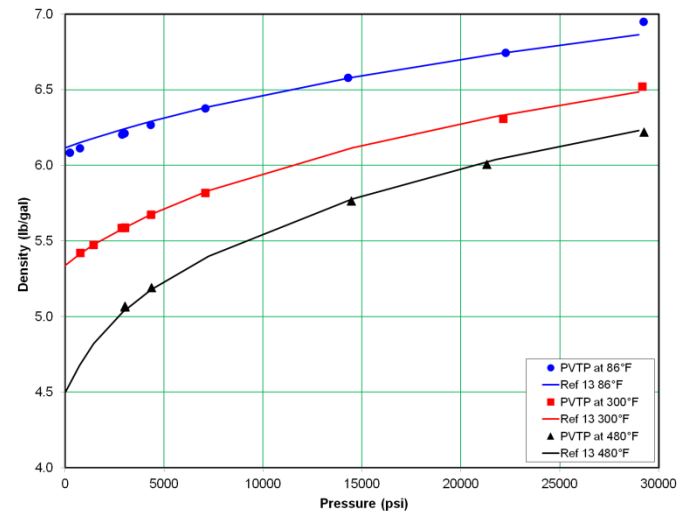


Fig. 5: Comparison of PVTP-measured undecane data and Ref 13 at three different temperatures and pressures to 30 kpsi.

Table 5: Correlation Constants to Match Eq. 1 for Common Base Fluids Tested on PVTP					
	Calcium Chloride 19.3 wt%	Diesel	LVT 200	IO C ₁₆ C ₁₈	Water
Reference	PVTP	PVTP	PVTP	PVTP	PVTP
Pressure Coefficients					
a ₁ (lb _m /gal)	10.0290	7.3459	7.0844	6.6962	8.7471
b ₁ (lb _m /gal/psi)	1.68 E-05	3.00 E-05	3.03E-05	2.83 E-05	1.65 E-05
c ₁ (lb _m /gal/psi ²)	1 E-10	-2 E-10	-2 E-10	-2 E-10	7 E-11
Temperature Coefficients					
a ₂ (lb _m /gal/°F)	-3.09 E-03	-2.99 E-03	-2.80 E-03	-2.72 E-03	-3.91 E-03
b ₂ (lb _m /gal/psi/°F)	3.43 E-08	8.62 E-08	6.85 E-08	6.87 E-08	6.06 E-08
c ₂ (lb _m /gal/psi ² /°F)	-6 E-13	-2 E-12	-1 E-12	-1 E-12	-9 E-13
Fitting Statistics for Modeled Data					
Avg. Error %	0.41	0.81	0.17	0.41	1.22
r ² coefficient	0.996	0.992	0.998	0.996	0.987
Range of Validity					
Max. Applied Pressure (psi)	30,000	20,000	30,000	30,000	30,000
Min. Temperature (°F)	76	37	78	75	84
Max. Temperature (°F)	500	500	500	500	500

PVT charts for the test series and comparisons are presented in Figs. 6a – 9b. Data taken on the PVT were first curve fit to a 2nd order polynomial equation (Eq.1). Charts that provide the PVT data were generated by Eq. 1 for the particular fluid. In the comparison charts, the data shown only as markers represent actual measurements on the PVTP. For these, only three temperatures are plotted to minimize clutter.

Overall, the data look quite reasonable and the comparisons are very good. The C₁₆C₁₈ IO data set plotted in Fig. 9b is interesting as it compares the PVTP-measured data to two references. The minor differences suggest variations in the materials, although the different testing apparatus and procedures also could have contributed.

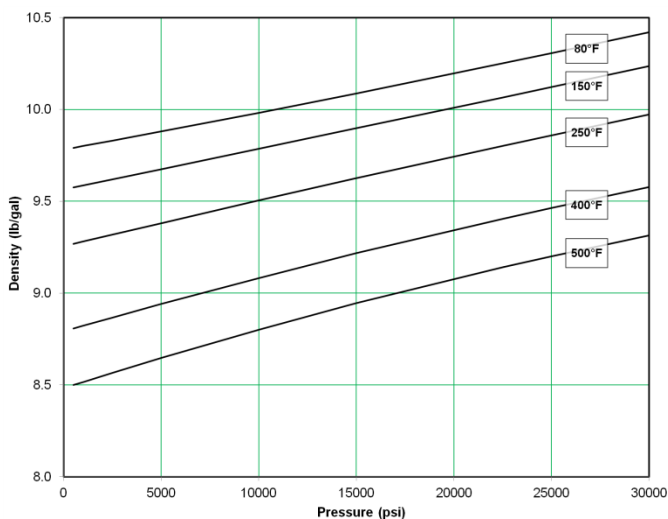


Fig. 6a: PVT data for Calcium Chloride (19.3 wt%) measured with the PVTP.

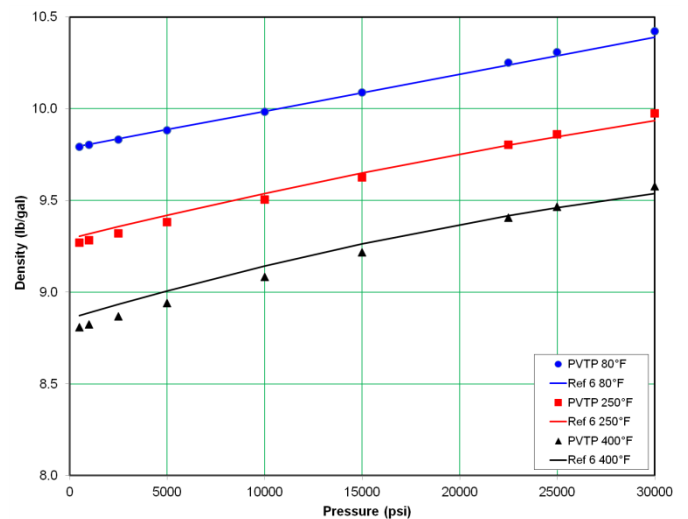


Fig. 6b: Comparison of PVTP-measured Calcium Chloride (19.3 wt%) with Ref 6 at three temperatures and pressures to 30 kpsi.

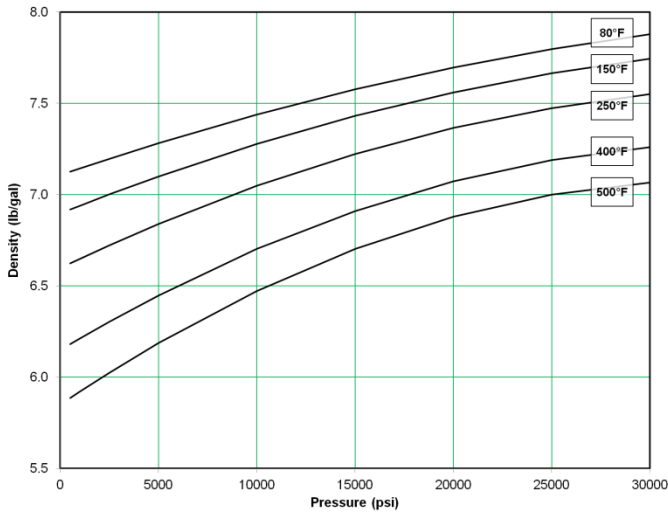


Fig. 7a: PVT data for diesel measured with the PVTP.

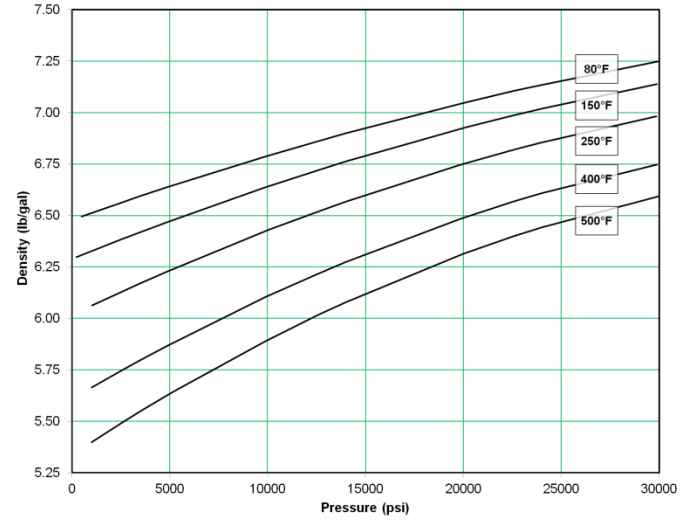


Fig. 9a: PVT data for C₁₆C₁₈ IO synthetic measured with the PVTP.

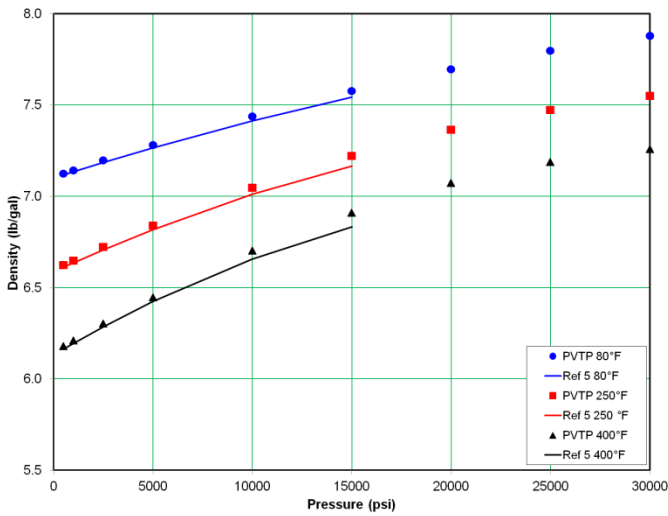


Fig. 7b: Comparison of PVTP-measured diesel data and Ref 5 at three different temperatures and pressures to 30 kpsi.

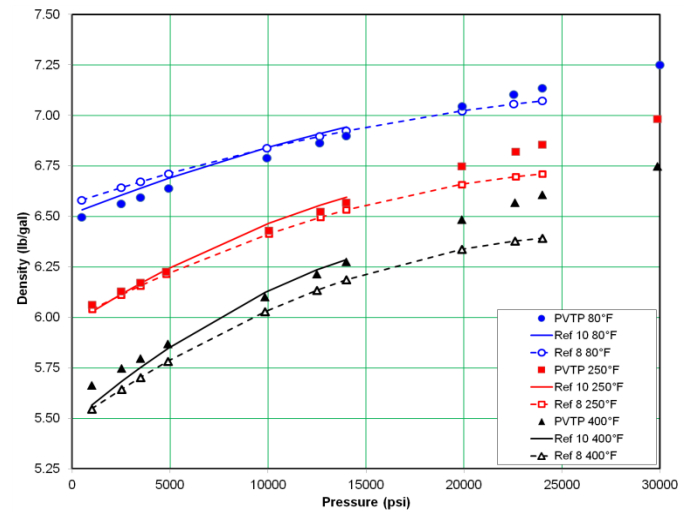


Fig. 9b: Comparison of PVTP-measured C₁₆C₁₈ IO data and Refs 8 and 10 at three different temperatures and pressures to 30 kpsi.

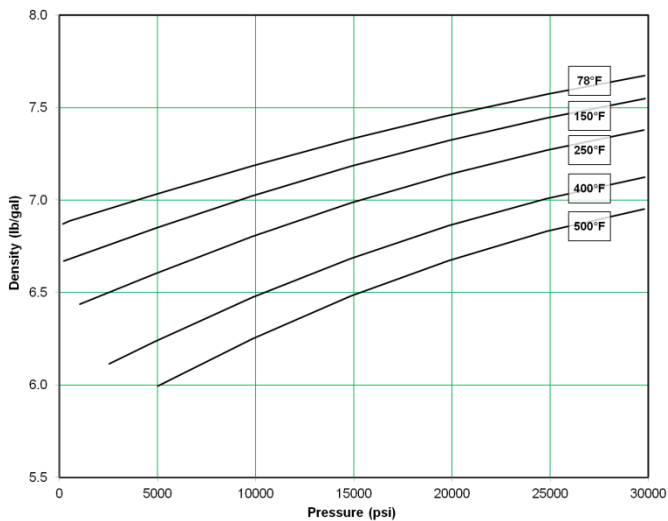


Fig. 8: PVT data for LVT 200 mineral oil measured with the PVTP.

Table 6: Properties for a 12.23-lb _m /gal SBM after Hot Rolling at 150°F	
Mud Weight (lb _m /gal)	12.23
Synthetic	C ₁₆ C ₁₈ IO
S/W Ratio	75 / 25
S/W Volumes (mL)	197 / 65
Brine (wt%)	CaCl ₂ (19 wt%)
Heat Aging	150°F for 16 hr
Rheology Temp (°F)	40 100 150
Plastic Viscosity (cP)	61 21 17
Yield Point (lb/100 ft ²)	16 16 15
LSYP (lb/100 ft ²)	7 5 7
10-sec Gel (lb/100 ft ²)	11 10 12
10-min Gel (lb/100 ft ²)	18 21 20

The final task was to compare measured data on a full formulated drilling fluid to a prediction model that incorporates relationships generated on the liquid components in the mud. This is precisely how the correlations generated from the PVTP data can be used in practice.

Table 6 listed the properties for a 12.23-lb_m/gal SBM after hot rolling at 150°F for 16 hr. The formulation was typical for drilling offshore Gulf of Mexico. The synthetic base fluid was C₁₆C₁₈ IO. The internal phase was 19 wt% CaCl₂ brine – a concentration selected to match the brine concentration in Table 2.

For modeling, the compressibility and thermal expansion of the IO and brine need to be combined with the virtually incompressible solids in the mud. Several density models have been proposed and evaluated¹⁴; however, this compositional, mass-balance model^{4,5} in general use by the fluids industry is suitable for this calculation:

$$\rho(p, T) = \frac{\rho_0 f_{v0} + \rho_w f_{vw} + \rho_s f_{vs} + \rho_c f_{vc}}{1 + f_{v0} \left(\frac{\rho_0}{\rho_{0i}} - 1 \right) + f_{vw} \left(\frac{\rho_w}{\rho_{wi}} - 1 \right)} \quad (2)$$

This model considers the individual contributions of the external phase (synthetic or oil), brine internal phase, and the solids. Many assume that the solids are incompressible, but this may not always be the case.

Fig. 10 compares data measured on the fully formulated SBM and the predictions based on Eqs. 1 and 2 and the correlation constants provided in Table 5 for the IO and the brine. Results are notably close.

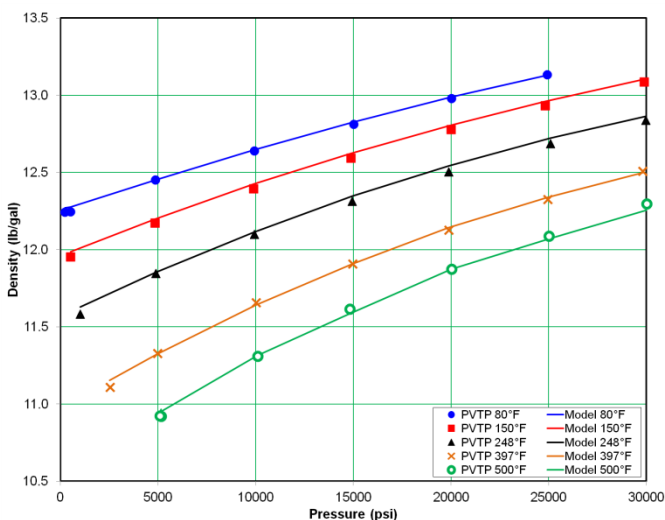


Fig. 10: Comparison of PVT-measured data for 12.23-lb_m/gal SBM to modeled results using Table 6 and Eq. 2.

Evaluations

Since the main purpose of this paper is to evaluate a piece of equipment, it is worthwhile to include some objective and subjective comments from those running the tests. Overall, the PVTP worked well and was easy to use. As expected, however, there were a few minor software issues that needed

to be addressed. There was concern that the two cobalt magnets could be affected by temperature, but this issue had been considered and calibrated out. Some irregularities with the magnets were solved by substituting a different size magnet.

Because the unit is fairly short in order to fit inside the viscometer pressure tower, selecting the correct sample volume was critical. Low-pressure/high-temperature and high-pressure/low-temperature test limits played a role in determining the proper sample volume. For most cases, 140-143 mL sample volumes seemed to work best.

The initial sample density was also a consideration. Use of Class A glassware or a pycnometer that meets ASTM methods is important, as is the temperature at which the density was measured. Slight adjustments to the pressure/temperature schedule in the start-up procedure to match this density/temperature pair helped ensure the base data was consistent.

Finally, there were recurring issues with test conditions lower than room temperature. It is possible that the test procedure did not allow fluids enough time to reach equilibrium. Also, the lower magnet could have failed to properly turn at cold temperatures. Regardless, further investigation will be required to determine the source of the problem and address the issue.

Conclusions

1. Laboratory testing suggests that the commercially available pycnometer discussed in this paper is suitable for measuring the PVT characteristics of base oils, brines, and drilling fluids under extreme temperatures and pressures.
2. Test conditions of temperatures from 40 to 500°F and pressures to 30,000 psi generally exceeded those used in similar testing published in industry literature.
3. Tests run on undecane helped validate the accuracy of the unit.
4. For the most part, new data generated on the PVT pycnometer correlated very well with published results.
5. Correlation constants were very similar to those published in API RP13D for four similar fluids.
6. Data generated on the fluids of interest were adequately fitted to the 2nd order polynomial equation published in API RP13D.
7. Excellent comparison was achieved between SBM measurements and modeled results using a compositional, mass-balance model.
8. The add-on module could be a low-cost option to those who already own the HTHP rheometer host for which it was designed.

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Nomenclature

API	= American Petroleum Institute
ASTM	= American Society of Testing and Materials
c	= Chemical subscript (Eq. 2)
f_v	= Volume fraction, dimensionless (Eq. 2)
HTHP	= High Temperature / High Pressure
i	= At elevated pressure and temperature subscript (Eq. 2)
IO	= Internal Olefin
LAO	= Linear-Alpha Olefin
LSYP	= Low-Shear Yield Point, $lb_f/100ft^2$
o	= Oil subscript (Eq. 2)
OBM	= Oil-Based Mud
PVT	= Pressure, Volume, Temperature
PVTP	= PVT Pycnometer
s	= Solids subscript (Eq. 2)
SBM	= Synthetic-Based Mud
w	= Water subscript (Eq. 2)
WBM	= Water-Based Mud
ρ	= Density, lb_m/gal

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