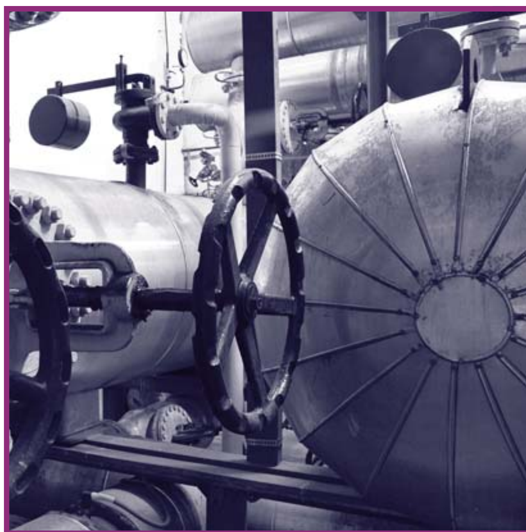


Density Measurement in the Petroleum Industry

Sandra Chirk, PAC
8824 Fallbrook Drive, Houston, TX, 77064, USA
Tel: +1.281.940.1803 • Email: pacsales@paclp.com • Web: www.paclp.com

Density is one of the most prevalent physical property used to classify and characterise fluids, not only in the environmental, cosmetics, pharmaceuticals, and food and beverage industries, but also in hydrocarbon processing industry (HPI). Petroleum products are everywhere in today's life, and have become almost natural parts of the modern world. Numerous products used on a daily basis are derived from petroleum, such as all kinds of fuels and oils, or the wide range of petrochemicals (solvents, plastics, synthetic fibers, etc.)
The raw material, crude oil, and its refinery products are complex mixtures of various carbon hydroxides that are usually characterised through their physical properties, including the density value. Used in conjunction with the specific-industry API Gravity scale*, its accurate and precise measurement is necessary for converting the measured volumes into volumes at reference temperatures (15°C; 20°C and 60°F).

Manual digital analysers, based on the oscillating U-tube method, have helped make density determination more precise and faster, as well as requiring smaller amounts of sample than the traditional pycnometers and hydrometers. Unfortunately, not every petroleum product lends itself to easy and straightforward sample handling and measurement. Highly viscous samples, such as fuel oils or waxes, are in need of high temperatures, whereas gaseous samples require pressure to be liquefied for correct density determination. Due to the wide variety from one product to another, characterisation of petroleum products is often complex to handle, therefore leading to significant analysis errors and inconsistent results. Improper sample filling, presence of air bubbles, weak control of temperature, and contaminated measuring cells are frequently named as the main reasons for an exhausting workflow. To address these challenges, a new generation of digital analysers, powered by smart automation, is introduced to improve analysis performance and reliability in oil and petroleum laboratories. This in turn increases laboratory productivity and profitability.

Addressing the key challenges

Density measurement by oscillating U-tube method, which is specified in the ASTM D4052 standard, is fairly simple: a small volume (approximately 0.7 ml) of liquid specimen is introduced into an oscillating U-shaped tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample.

While the concept is simple, the challenge with petroleum samples, especially very viscous and/opaque specimens (ASTM D5002), involves continual accurate measurement in a repeatable manner, which is required in the demanding environment of an oil and petroleum testing laboratory. To obtain high consistency, the ASTM D4052 and D5002 specifications recommend several critical test parameters to be controlled by the instrument:

- Operators must ensure no bubbles are trapped in the tube, and that it is filled to just beyond the suspension point on the right-hand side. The sample being tested must be homogeneous and free of even the smallest bubbles.
- The ability to accurately control and regulate the sample temperature as it affects the density is also important. As the density is temperature-dependent, the system should be capable of maintaining the temperature of the circulating liquid constant to +/- 0.05°C in the desired range to ensure precise measurement.
- Once the test is completed, the system cleaning consists in a 3 steps-procedure: flush the sample tube with petroleum naphtha, followed with an acetone flush and dry with dry air. The need for a system recalibration is attributable to deposits in the sample tube that are not removed by the routine cleaning process.

Smart Design for Reliability Performance

Automated density analysers powered by intelligent software can provide an alternative platform that better addresses the industry challenges, therefore ensuring laboratory superior performance, but also productivity by eliminating the need to redo testing. For instance, automatic sample injection combined with a bubble detection system can eliminate most of the reading errors caused by presence of air bubbles in the measurement cell. Samples are loaded in a smooth and repeatable way, independently from specimen viscosity and/or operators' subjectivity.

Figures 1 and 2 display the results obtained with automated and manual sample injection. This comparison shows that laboratories using manual sample loading tends to decrease in measurement performance, leading to poor repeatability, with results heavily spread out. Automatic sample injection not only improves the analysis efficiency and repeatability but also optimises operator intervention by reducing the number of multiple redo testing.

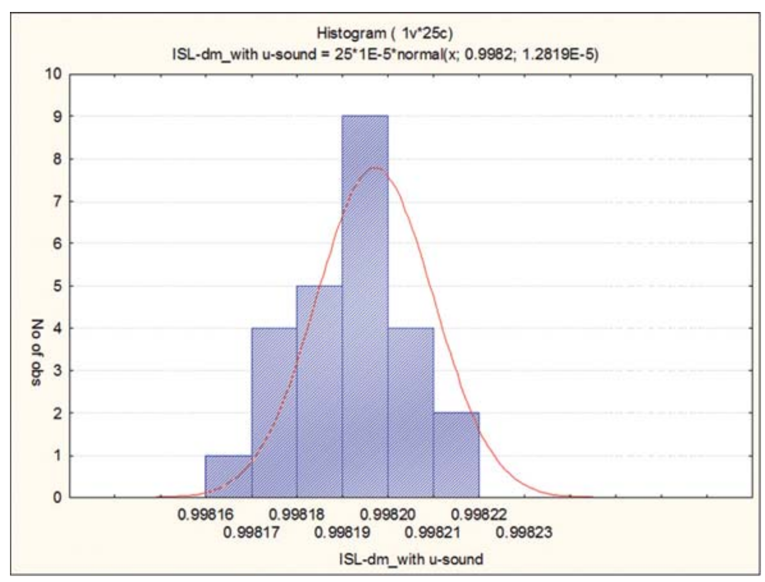


Figure 1: Standard deviation of measurement repeatability (automatic injection method).

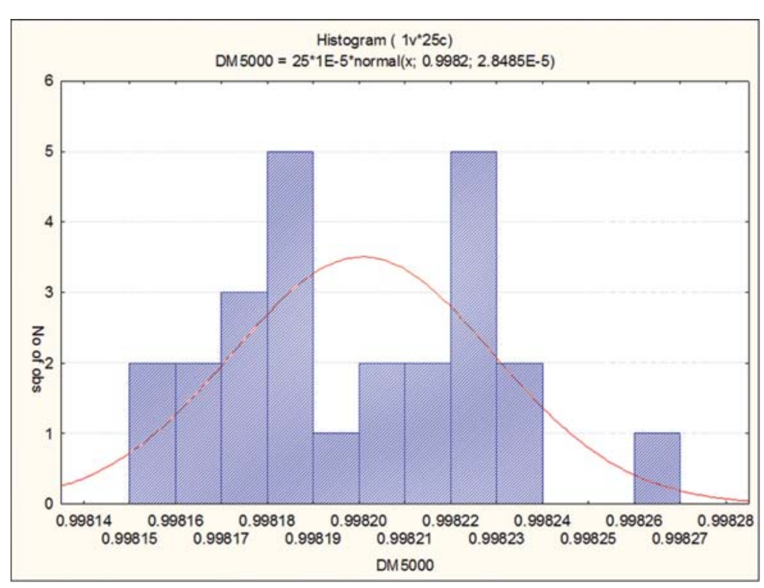


Figure 2: Standard deviation of measurement repeatability (manual injection method)

Automation of the cleaning/drying steps also helps to reduce the operator intervention. Due to the risks of deposits in the sample tube that cannot be removed by routine procedure, the cleaning efficiency can be enhanced by the use of ultrasounds. Apart from drastic reduction of time and cleaning cost per test, the use of ultrasonic cleaning is proven to be one of the most efficient and powerful means available for removing any strongly adhered deposits, yet gentle enough not to damage cell surface (see Figure 3).

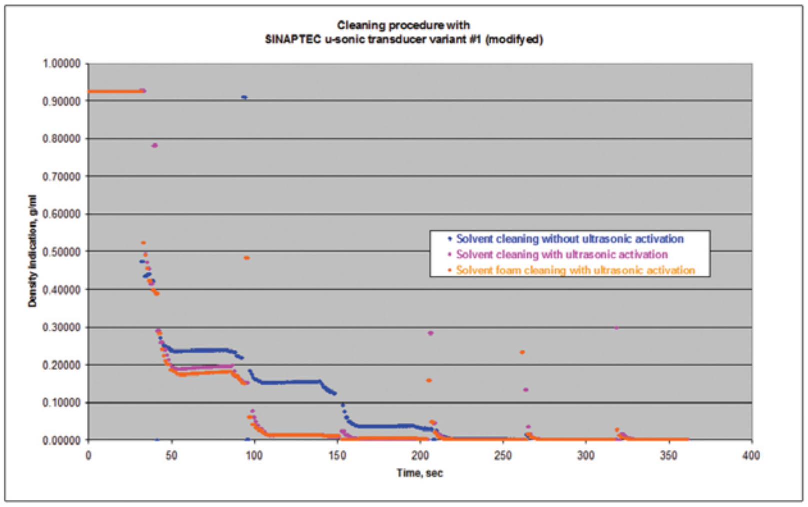


Figure 3: Ultrasonic Effect on cleaning efficiency after mazut sample testing

	Time of density stabilisation, sec	**Volume of the solvent,** ml
Without ultrasound	>270	2.5
With ultrasound	<160	1.5
Improvement	>40%	≈40%

Such practices have also become increasingly popular due to the restriction on the use of aggressive solvents, which are proved harmful for the operators, especially when exposure is on a daily-basis. A final control by checking density of air enables the operator to validate the cleanliness of the measuring cell.

Finally, whereas Peltier thermostats can easily ensure stable temperatures within a wide range (0 up to 100°C), the combination of Pt100 sensors together with a high thermal conductivity measurement cell (for instance: stainless steel, which is proved to conduct at least 10 times more heat than borosilicate glass) has revealed to provide fast response to any change of temperature, ensuring the density is effectively determined at the specific temperature set up by the operators.

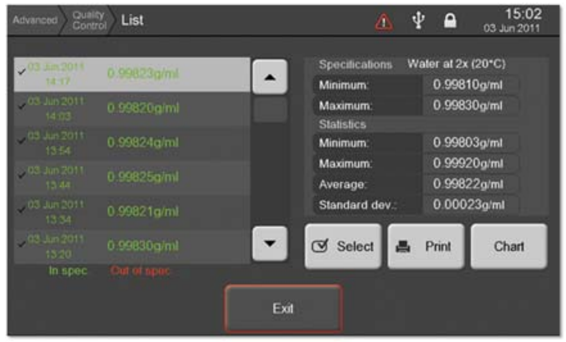
Increased Laboratory Productivity and Instrumentation Usability

With the increased scrutiny on budgets, increasing overall productivity with limited resources is imperative in order to keep reasonable profit margins. Typical test cycle time using automated analysers is about 5-10 minutes and require half the involvement from the operator compared to conventional manual systems. This greatly increases the number of tests laboratories are capable to perform within a day, whereas freeing the personnel to perform others priority tasks.

In addition, with the baby boom generation retiring, laboratories are losing some of their most skilled and experienced operators. Mature industries, such as petroleum, are even more affected than others since they hired a significant amount of their workers years ago. In fact, 49.3% of manufacturing industry employees is considered part of the baby boom generation1. Even though the recent recession has reduced the number of retirees in recent years, the lingering threat of the baby boom generation exiting the workforce in masses still looms. Considering these unavoidable trends, smart automated systems that operate as "plug-and play" solutions, thanks to "push-of-a-button" approach, eliminate the need for operators expert knowledge and/or extensive training to be run at its full performance and capability.

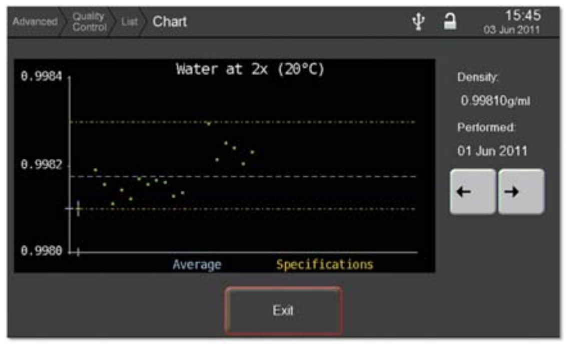
Safety and Quality Compliance (QC)

Since automated systems are designed as stand-alone, self-contained solutions, this is an extra assurance for testing laboratories that potentially harmful vapours are not exuded from the analyser during operation, therefore protecting their personnel from inadvertently being exposed to health risks. Maintenance and regular inspection, which are also essential parts of the laboratory quality assurance, are facilitated by special functions embedded, as standard, in the software.



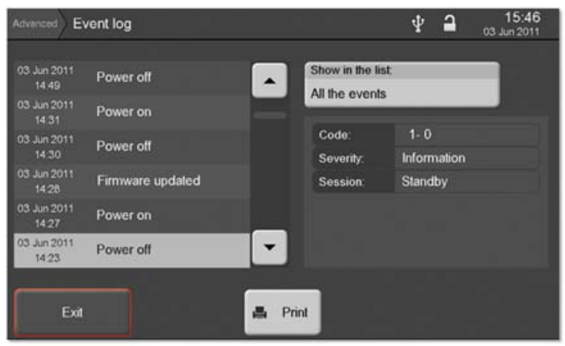
The automatic Pass/Fail Indicator provides a fast validation of QC of products as the results are automatically matched with the specifications limits or target values requirements.

Automatic Pass/Fail Validation of QC Samples



The QC chart enables operators to monitor the correct performance of the analyser with time, with reference material, and stay aware of when system calibration is needed.

Control Chart for Quality Samples or CRM Monitoring



The event log is the primary resource when it comes to diagnosis or troubleshooting issues. All events generated during operating – from information to alarms – are recorded in tamper-proof log files.

Event Log Viewer (filter by severity type)

Conclusion

While manual density meters can be cumbersome to use and susceptible to measurement errors, the new generation of analysers, based on fully automated processing, offers a high level of reliability for a wide variety of petroleum products testing, while meeting increasingly rigorous industry regulations. Laboratories now can benefit from higher accuracy, improved safety and increased productivity, therefore increasing their overall profitability, with a single "push-button" method.

References:

1. Linda Levine, CRS Report for Congress: Retiring Baby-Boomers = A Labor Shortage?, http://aging.senate.gov/crs/pension36.pdf, (2008).