NEW ANALYZER FOR VISCOSITY MEASUREMENT AT REFERENCE TEMPERATURE

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ABSTRACT

Temperature is one of the parameters that has the most influence on the viscosity of a product. If we take the example of glycerin, viscosity drops from about 1400 cP to about 140 cP when the temperature increases from 68°F/20°C to 122°F/50°C. For this reason, it is common to give viscosity measurements at a specific temperature (e.g.: heavy fuel oils at 50°C).

Due to the property of viscosity described above, measurements made under process conditions do not always provide sufficient information to achieve optimal process control or make the right decisions regarding quality specifications. On the other hand, laboratory controls are made under specific conditions so that samples can be compared one with the other but the difficulty of recreating process conditions in the lab complicates the use of the information for process control. This problem led to the process world developing a measurement to be able to compare samples in process condition with standards at a specific temperature: Viscosity measurement at reference temperature. Unfortunately, some solutions are readily available but don't meet the full requirements of all industries.

In this paper we will introduce a new, simple, rugged and economical on-line analyzer capable of measuring viscosity at reference temperature. This analyzer is based on vibration at resonance frequency, a renowned and proven principle in viscosity measurement. We will then go over the results obtained on a reference product sampled in the analyzer.

INTRODUCTION

Viscosity is one of the most commonly measured physical properties. Many methods and technologies exist to measure viscosity. It is almost systematically measured in laboratories using rotational or cone and plate viscometers and rheometers but the recent years have seen an increase in the amount of in-line and on-line viscosity measurements.

The importance of viscosity relates to a couple of factors. It has a lot of influence on processes involving heat transfer and matter transfer as high viscosities lower heat transfer and complicate homogenization of fluids. Viscosity is also a very influential factor in the sizing of pumps as higher viscosities cause higher power drain on pump motors (similarly for agitators). Finally, viscosity is a quality specification for many products. Those 3 elements mean that viscosity is measured for almost every fluid whether in the lab or in process conditions.

Viscosity is itself highly affected by temperature. If we take the example of glycerin, viscosity drops from about 1400 cP to about 140 cP when the temperature increases from 68°F/20°C to 122°F/50°C. Therefore, due to the effect of temperature on viscosity, it is often required to measure viscosity at a constant temperature.

In this paper, we will describe a single point analyzer (i.e.: used to monitor a process around a single operation point) used to measure on-line viscosity at a reference temperature. We will give an overview of the existing technologies to measure viscosity at reference temperature and describe the sensing technology used for this analyzer. We will then look at the series of tests conducted to demonstrate that the analyzer bears very good results even when taking into consideration the simplistic approach taken in the design.

1. EXISTING TECHNOLOGY FOR VISCOSITY AT REFERENCE TEMPERATURE MEASUREMENTS

Viscosity measurement at reference temperature is a measurement where the sample is drawn from the process line at process temperature and brought to an analyzer with an internal temperature control system to heat/cool the product to the desired temperature before measuring the viscosity.

The first principle patented for this measurement was the Hallikainen capillary oil bath system. Developed in the 1950s, it is still widely used and is the process measurement closest to the ASTM D445 standard for viscosity measurement. Its drawbacks include the use of an oil bath, heavy maintenance requirements and its unavailability for viscosities above 2,500 cP.

The technology used by capillary systems measures the pressure difference between the inlet and the outlet of the capillary to make a direct measurement of viscosity. The capillary is inserted in an oil bath to bring the sample to the required temperature and ensure that the

diameter and length of the capillary do not change with temperature. Other systems based on the same capillary technology are available but use an oven instead of an oil bath.

The other commonly employed solution is to use 2 viscosity sensors and measure the viscosity for 2 temperatures surrounding the reference temperature, one slightly above and the other slightly below. Then, according to the formula given by the ASTM D341, it is possible to interpolate the viscosity of the sample. The drawbacks of this solution are that it is not a true viscosity measurement but a calculation as well as the requirement of a series of heat exchanger and controllers to bring the sample near reference temperature which significantly increases the costs.

Based on the issues raised for the previous two solutions, we can see there is a need for an instrument with easy maintenance, measuring (by opposition to calculating) viscosity with a large range of measurement for more viscous products. We will therefore describe in the next section the equipment that was designed to meet those requirements.

2. DESIGN OF AN ANALYZER TO MEASURE VISCOSITY AT REFERENCE TEMPERATURE

2.1 SCIENTIFIC AND TECHNOLOGICAL REQUIREMENTS

Various requirements were taken in consideration during the development of the analyzer:

- Since the analyzer is connected to the process it had to be able to handle process conditions in the sampling loop
    - Maximum Temperature: 200 °C
    - Maximum Pressure: 16 bars
    - Maximum Flow: 100L/h
- As the analyzer is based on cyclical measurements, the maximum acceptable response time had to be under 5 minutes
- The analyzer will be used for process monitoring and/or control so repeatability is the key to this equipment. A repeatability of 1% of reading was needed for this equipment
- The analyzer will be used with refinery products (amongst others) so it had to have the ability to be correlated to the ASTM D445 : Standard Method of Test for viscosity of transparent and opaque liquids (Kinematic and Dynamic viscosities)
- The analyzer will be operating with hazardous products so it had to have the ability to receive NEC and ATEX certification

2.2 PRESENTATION OF THE VIBRATING TECHNOLOGY USED

In 1981, Sofraser created and patented the first vibrating viscometer using the vibrating technology at resonance frequency (Patent N° FR 2 544 496) [1].

Today, vibrating viscometers are recognized worldwide as the optimal solution to measure viscosity in process. The sensor response time is close to zero, and the viscosity information (stability or variation) is available continuously. This allows the control of processes even in the presence of transitory phenomenon or rapid disturbances. The absence of wearable parts guarantees no drift in time and no maintenance.

The vibrating viscometer at resonance frequency is a sensor working at a high shear rate to reduce measurement fluctuations due to fluid speed or flow rate when the product is pseudo-plastic or shear-thinning and is perfectly adapted to process measurements. This process viscometer is able to measure viscosities over one million centipoises (cP).

The principle of the vibrating viscometer at resonance frequency is simple. The active part of the sensor is a vibrating rod held in oscillation at its resonance frequency. The vibration amplitude of this movement varies according to the viscosity of the product in which the rod is immersed. Similarly, as the rod vibrates in the fluid, the frequency of the movement varies with the density of the product.

The motion of the rod is created by a magnet fixed on the rod and placed in front of a coil driven by an alternative current. Another magnet attached to the rod induces a current in a separate coil which is an image of the motion of the rod (Figure 1). The resulting voltage amplitude is an image of the viscosity.

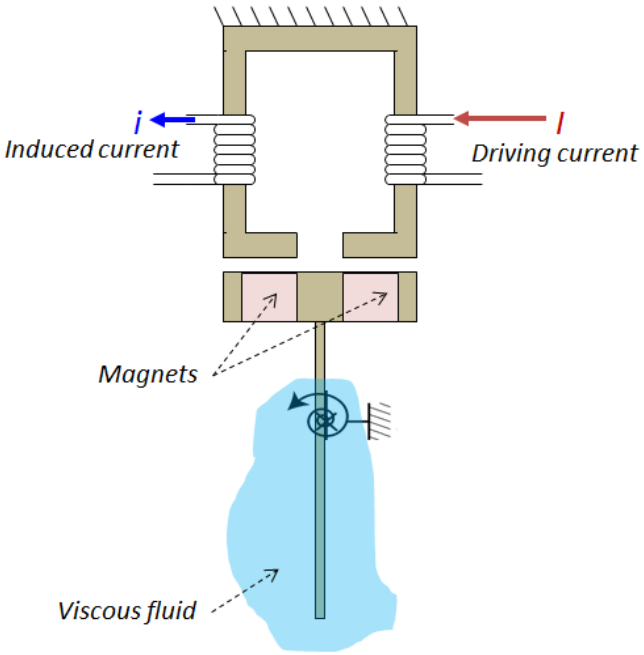

Figure 1. PRINCIPLE OF THE VIBRATING VISCOSITY SENSOR AT RESONANCE FREQUENCY

During calibration, the amplitude of the vibration is correlated to the viscosity of the product by comparing the vibration in the air (maximum vibration) and in the viscous fluid (Figure. 2), thus providing a reliable, repeatable and continuous viscosity measurement. This principle is described in Patents FR 2 911 188 B1 [2] and FR 2 921 726 B1 [3].

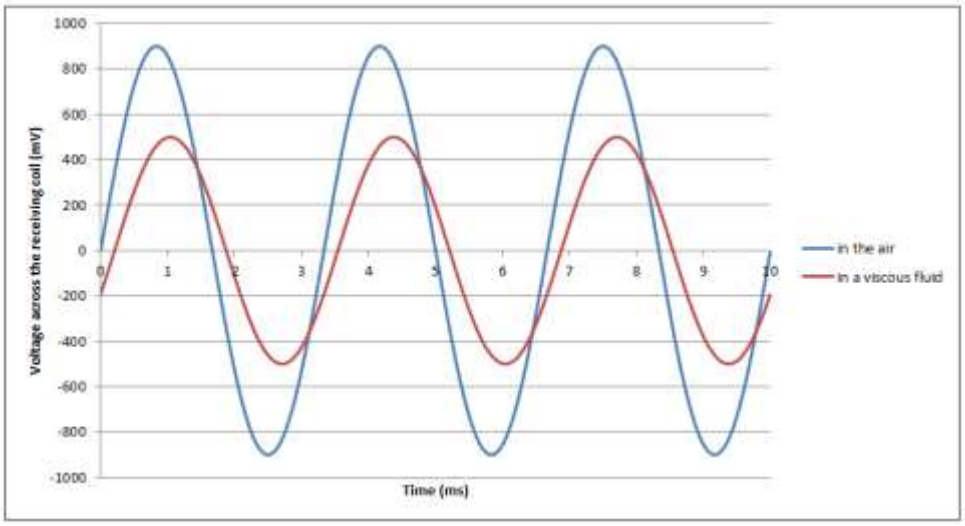

Figure 2. AMPLITUDE DIFFERENCE IN AIR AND IN VISCOUS FLUID

2.3 FIRST ANALYZER AT REFERENCE TEMPERATURE

The first design of the analyzer at reference temperature came out in the late 1990s. The viscometer at resonance frequency described in the previous section is the core around which the analyzer is built. The schematic (Figure 3) of the analyzer can be seen below:

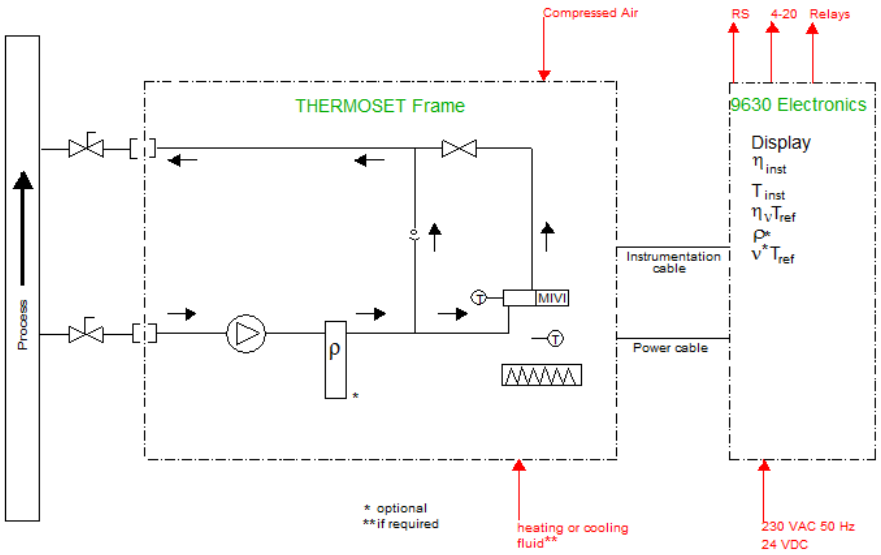

FIGURE 3. SCHEMATIC OF THE FIRST ANALYZER MODEL

The analyzer is based on cyclic operations, it continuously draws sample from the process. At the beginning of a cycle, the flow-through cell containing the viscometer is filled with the fluid. This branch of the flow loop is then closed by a pneumatic valve. The sample is left in the chamber to cool or heat depending on the measurement needed. While the sample cools/heats, the bypass loop is opened and the sample keeps on flowing in the analyzer.

Over the years, this analyzer has proven efficient and reliable and is still highly regarded for its simplicity of use and ruggedness. It is the only analyzer capable of measuring viscosity at reference for viscosities up to 10,000 cP and the simple design including a pump allows it to operate autonomously in nearly all conditions without requiring sampling systems or advanced maintenance.

This analyzer though, still has some weaknesses:

- The analyzer was designed to meet ATEX certification which makes it costly to certify to NEC standards
- Due to the presence of a pump system and control, the analyzer represents a higher price tag
- The installation, even taking into account the low footprint (3’ x 1’ x 2’), does not allow wall mountings

2.4 NEW ANALYZER DESIGN AND ADAPTATION OF THE VIBRATING TECHNOLOGY TO THE ANALYZER

As previously shown, there was no process monitoring solution satisfying the demand for viscosity measurement at a reference temperature especially with regards to costs. The new analyzer is also based on cyclic measurements but works without a pump (We assume in this paper that the process already possesses a pump or that the pressure difference generated by the process between the inlet and outlet of the analyzer is sufficient to move the fluid as the pressure drop in the analyzer is minimal).

It is inserted in an enclosure that can be purged and hence bear a hazardous rating for NEC Class 1 Div 2 or ATEX Zone 2. The absence of a pump system also simplifies the maintenance and reduces the potential for failures.

The other major difference from the first model is the permanent implementation of a water cooling system instead of an air heat exchanger. This modification allows for better temperature control and stability.

The analyzer can be seen on Figure 4 below.

FIGURE 4. SCHEMATIC OF THE NEW ANALYZER AT REFERENCE TEMPERATURE

3. RESULTS

In this section, an overview and analysis of the tests conducted with the analyzer will be presented. Figure 5 shows a picture of the analyzer on which the tests were conducted. On the left side of the picture are, from top to bottom, the viscometer, the water-cooled flow-through cell and a Pt100 temperature probe.

FIGURE 5. PICTURE OF THE ANALYZER IN PROCESS CONDITIONS

3.1 TEST CONDITIONS

In order to be able to compare data, all the tests were conducted at a reference temperature of 50°C (122°F). All the tests were conducted with the Canon S60 Mineral Oil with a certified viscosity of 29.37 cP at 50°C (122°F).

The following tests were made in order to evaluate the response of the analyzer:

- Various input product temperatures: 90°C, 110°C 130°C (Approx. 195°F, 230°F and 265°F), all other conditions constant
- Various cooling water temperatures: 5°C, 10°C, 15°C, 25°C (Approx. 40°F, 50°F, 60°F, 75°F), all other conditions constant
- Continuous operation over 3 days (same conditions all the time)

3.2 RESULTS ON REFERENCE PRODUCT

In this section we will analyze the results from the tests described above. We will also be able to evaluate the performance of the analyzer and hence report accuracy, repeatability and reproducibility.

3.2.1 DISCUSSION

As it has been previously shown, the technology at resonance frequency offers a relative measurement of viscosity. Secondary measurements rely on the calibration of an analyzer with standards in order to guarantee performance. One approach to evaluate the quality of a calibration is to look at the “scale ratio” between the physical quantity measured and the desired physical quantity [4]. The typical parameters used to evaluate an analyzer are repeatability, reproducibility and accuracy [5].

Repeatability is an indication of how close one measurement is from another under the same experimental conditions. In order to calculate the repeatability of an analyzer, the same measurement is made repeatedly under the same conditions. Practically, observers will run the analyzer under the same conditions and compare one measurement with the other, the closer the measurements, the more repeatable the analyzer is. These experiments allow the determination of a “scale ratio”, then, the dispersion of measurements gives the overall repeatability of the analyzer.

Reproducibility is an indication of how an analyzer performs when the experimental conditions change from one measurement to the other. When evaluating the reproducibility of an analyzer, various experiments are run with different operating parameters. Similarly to repeatability, the differences between “scale ratio” variations are used to calculate the reproducibility. For each of the experiments, the difference between the model and actual

value is calculated and the overall differences between all the experiments give the reproducibility of the analyzer.

Finally, accuracy is the indication of how close from the reference value a measurement is. Accuracy is a measurement of how consistent the “scale ratio” is between every measurement. Practically, this means that the observers calculate the difference between the value predicted by the model and the value of the standard.

After correction of any bias by the adjustment of the “scale ratio”, the accuracy of the equipment is then included in the reproducibility limit which is “the lowest or equal value to which the absolute difference between two test results obtained under reproducibility conditions may be expected to have a probability of 95%” [5].

As a matter of fact, these corrections have to be made in all measurements whether absolute or relative as it has been explained in the ASTM standards [6, 7] and demonstrated by Barbosa et al. [8].

In the scope of this article, due to the method used to quantify accuracy, repeatability and reproducibility, the accuracy performance of the analyzer can be extracted from the reproducibility tests. We consider that by knowing the error on the “scale ratio” from the reproducibility data then a proper rescaling of the correlation allows reducing the error and therefore increasing accuracy.

3.2.2 REPEATABILITY

In order to determine the repeatability of the analyzer, several tests were run under constant temperature conditions. These tests were realized as describe in test condition at a reference temperature of 50°C (122°F) for three input fluid temperature (130°C, 110°C and 90°C) and four different cooling temperature (5°C, 10°C, 15°C, 25°C).

Figure 6 shows the viscosity recorded versus time for four of these tests. The first three have the same cooling temperature but different input temperature and the two last have the same input temperature but different cooling temperature. Then, Figure 7 shows the distribution of the measurement for the previous tests.

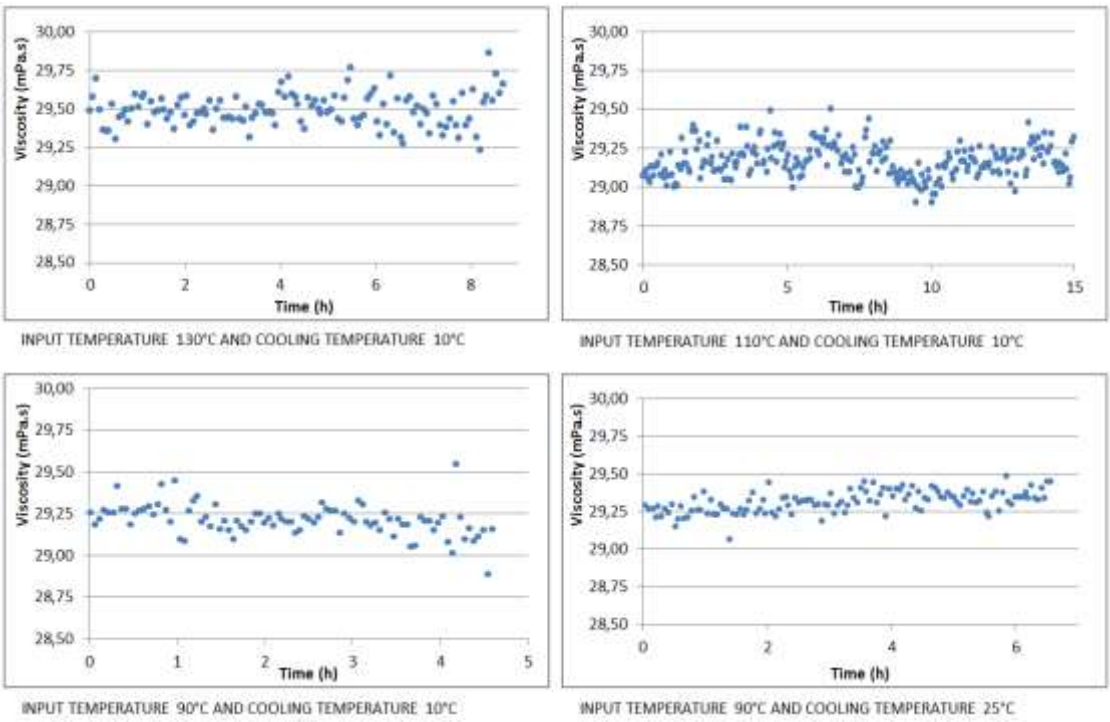

FIGURE 6. VISCOSITY VERSUS TIME

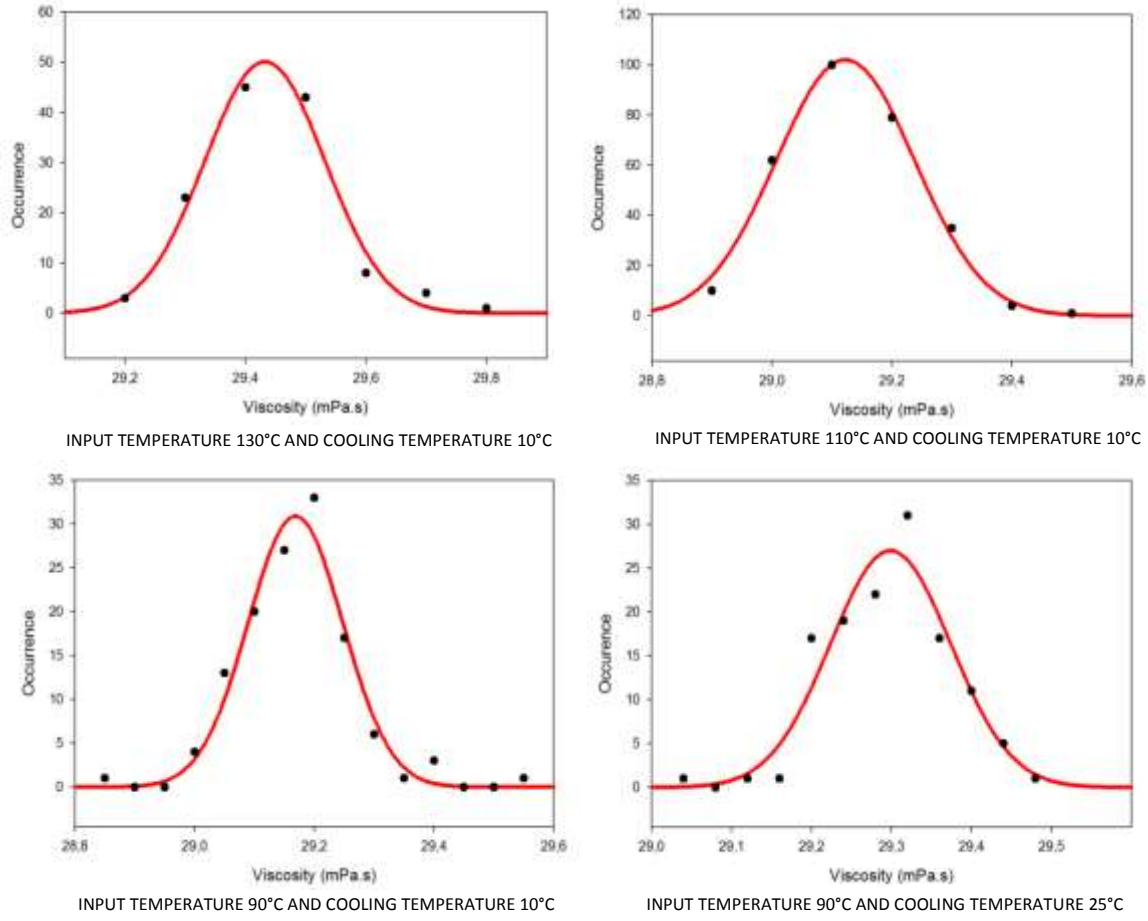

FIGURE 7. DISTRIBUTION OF VISCOSITY VERSUS TIME TESTS

Analysis of the measurements distributions from the previous tests are summarized in table 1 below:

Input product temperature (°C)	130	110	90	90
Cooling water temperature (°C)	10	10	10	25
Average viscosity (mPa.s)	29.491	29.177	29.194	29.318
Standard deviation (mPa.s)	0.107	0.106	0.091	0.071
Standard deviation (%)	0.36	0.36	0.31	0.24

TABLE. 1 ANALYSIS SUMMARY OF REPEATABILITY TESTS CONDUCTED ON THE ANALYZER

These tests show that under repeatability conditions, the standard deviation of the measurements made by the analyzer is lower than 0.5% of reading, (for an expected viscosity of 29.37 mPa.s, the standard deviation is lower than 0.11 mPa.s). This repeatability standard deviation was verified for several temperature conditions. The repeatability limit (i.e. The value less than or equal to which the absolute difference between two test results obtained under repeatability conditions may be expected to be with a probability of 95 %.) is lower than 1% of the reading.

3.2.3 REPRODUCIBILITY AND ACCURACY

Figure 8 below shows the summary of the tests conducted with the analyzer and allows calculating the overall reproducibility of the measurement.

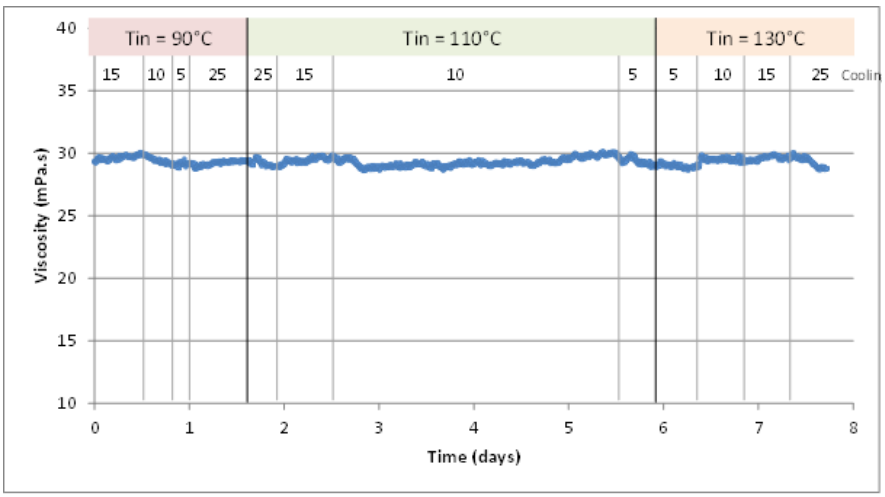

FIGURE 8. OVERALL TESTS AT VARIOUS INPUT TEMPERATURES AND COOLING TEMPERATURES

Figure 9 shows the distribution of the measurements from the previous graph. The average is 29.32 cP (viscosity of standard: 29.37 cP) with an experimental standard deviation of 0.31 cP.

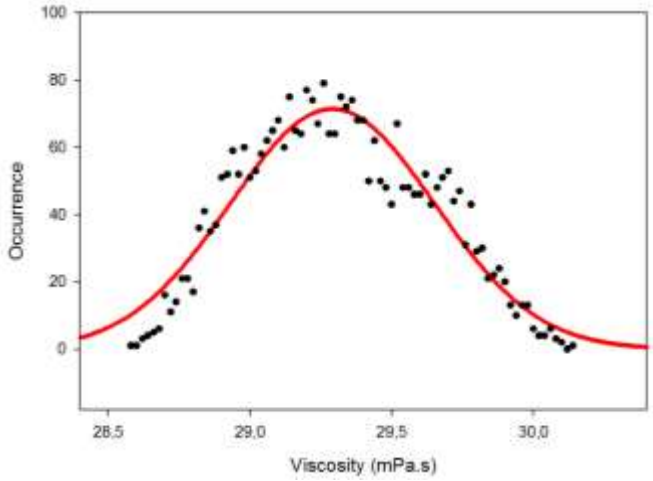

FIGURE 9. DISTRIBUTION OF THE MEASUREMENTS

Based on those results and the ISO definition, the reproducibility standard deviation of the equipment is 1%. Since the measurements distribution is a normal distribution, the 95% probability is included in two times the standard deviation. Following the definition exposed in chapter 3.2.1, the accuracy is therefore expected to be about 2% of reading.

4. COST ANALYSIS

In this section, we will do a cost analysis of ownership for the analyzer presented. We will therefore compare on various grounds the analyzer with existing solutions.

When looking at the price of an analyzer, various costs must be taken into account:

- Cost of the analyzer (Capital expense, CAPEX)
- Cost of installation (CAPEX)
- Cost of operation and maintenance (Operation expense, OPEX)

In this study we will assume that indirect costs such as training, administrative fees, shipping and handling and other indirect fees are the same for both analyzers.

4.1 COST OF THE ANALYZER

The average price of a viscosity analyzer at reference temperature starts at $50k (average standard cost is usually closer to $75k). The price tag for the new analyzer starts at $35 k (if the analysis loop does not already have a pump, an additional cost, lower to $10k needs to be added).

4.2 COST OF INSTALLATION

The main difference in installation costs with existing solutions is usually the need for a conditioning system. Conditioning systems are not systematically required but the internals of existing technologies restrict the type of fluid (particles and bubbles presence) that may enter the analyzer and will therefore generally require the installation of a conditioning system.

A conditioning system would be composed of a pumping system, a 200 mesh (or smaller) filter, a heat exchanger (could also be required for the new analyzer) and various valves. The starting cost is $25k with standard prices closed to $50k.

4.3 COST OF OPERATION AND MAINTENANCE

Over the life of the analyzer, OPEX is where most of the cost of ownership difference between both systems can be seen. Existing solutions, due to their fine tuned design and conditioning system will require more maintenance time and hence a higher maintenance budget. Table 2 compares the time required per year for the new and existing technologies, based upon a comparison conducted by end-users and internal data.

	Existing Solutions	**New Analyzer**
Scheduled maintenance	5 to 7.5 hours per week Average of 325 h/year	45 minutes per week Average of 39 h/year
Preventive maintenance	14h per year	14h per year
Total per year	339 h/year	53 h/year

TABLE. 2 COMPARATIVE MAINTENANCE HOURS PER YEAR

4.4 SUMMARY

Table 3 summarizes the costs associated with both analyzers in the case where no a heat exchanger is required for neither equipments

	Existing solution	**New Analyzer**
CAPEX		
Analyzer Cost	From $50k	From $35k
Conditioning system	From $25k	-
OPEX		
Maintenance Cost ($65/h)	$22k	$3.5k
Total Cost	From $97k 1st year $22k/year afterward	From $38.5k 1st year $3.5k/year afterward
TOTAL COST OVER 10 YEARS OF OPERATION	**From $317k**	**From $73.5k**

TABLE. 3 COMPARATIVE TOTAL COST BETWEEN ANALYZERS

As the table shows, over the life of the viscometer, the new analyzer is more economical than the existing technologies. This allows for the purchase of an analyzer for applications where budget was too limited for existing technologies or where viscosity sensors with viscosity calculations were used, thus providing an easy and economical single point process monitoring with the performance of a real-time analyzer.

CONCLUSIONS

Over this article, we have presented one of the problems commonly encountered with viscosity: temperature dependence. We went over the existing technologies and their weaknesses and presented an alternative based on a pre-existing principle without the weaknesses of the other technologies.

We then demonstrated that the performance of this new analyzer, that can be correlated to the ASTM D445, correspond to most of the industry's needs, that given stable conditions the repeatability and accuracy of the analyzer are excellent and that even in the case of variable conditions, good performances are still maintained. Due to its design, the analyzer is easy to maintain and clean and supports very well difficult external conditions: particulates, bubbles, dirty products, high viscosity. Finally, due to the simple design and simple principle of operation, the analyzer has a low overall cost of ownership compared to other existing technologies and allows monitoring applications, at a single control point, where previous budget restrictions did not permit it.

REFERENCES

[1] FR2 911 188, Sofraser, "Procédé et système mettant en oeuvre un élément oscillant pour déterminer les caractéristiques physiques d'un produit".

[2] FR2 921 826, Sofraser, "Method and system for determining the viscosity of a product".

[3] FR 13 62 507, Sofraser, "Système et procédé de mesure en ligne de la viscosité d'un produit"

[4] T. L. Saaty, « What is relative measurement? The ratio scale phantom", Mathl. Comput. Modelling, Vol 17, No. 4/5, pp. 1-12, 1993.

[5] ISO 5725-1 :1994 « Accuracy (Trueness and precision) of measurement methods and results – Part 1 General principles and definitions", International Organization for Standardization, Geneva, Switzerland, 1994.

[6] ASTM D3764-09, "Standard practice for validation of process stream analyzer systems", ASTM International, West Conshohocken, PA, USA, 2009.

[7] ASTM D6708-08, “Standard practice for statistical assessment and improvement of expected agreement between two test method that purport to measure the same property of a material”, ASTM International, West Conshohocken, PA, USA, 2008.

[8] E. Barbosa, “Case study – Validation and assessment of the correlation according to ASTM D3764”, ISA 57th Analysis Division, Session 08-2, Anaheim, CA, USA, 2012.