White Paper:

An Automatic Test Method with Improved Precision for Wax Appearance Temperature (WAT) of Crude Oils

Problem:
Determining the Wax Appearance Temperature (WAT) or cloud point of crude oil has heretofore been an imprecise, tedious, and subjective process. Various manual methods have been used, but all are difficult and time-consuming, yielding results with unacceptably wide error margins.

Solution:

Phase Technology's WAT-70Xi analyzer is a fully automatic solution, measuring WAT of crude oils in a fraction of the time of manual methods, with no setup or additional cleaning required. Based upon ASTM D5773, Phase Technology's proprietary optical light scattering technique detects phase changes with extreme sensitivity and accuracy, yielding results that are repeatable within 1.0° C.

Background:

Wax Appearance Temperature (WAT), or cloud point, is the temperature at which a crude oil sample first precipitates solid wax as it is being cooled under prescribed test conditions. Similarly, Wax Disappearance Temperature (WDT) is the temperature at which the last wax solids are melted into liquid during a warming cycle.

Wax appearance temperature (WAT) and wax disappearance temperature (WDT) are useful indicators that can predict the occurrence of wax deposition in crude oils, with application in both the upstream and midstream petroleum sectors.

Many different approaches have been used in attempts to determine WAT, with less than optimal results.

Estimations derived from various calculation methods are inexact and yield a wide range of results. Inferred measurement methods are generally less sensitive, producing colder results which may be risky and unreliable.

One such method, Differential Scanning Calorimetry (DSC), detects WAT by measuring the difference in heat absorbed or released between a reference sample and the test sample at a given cooling or warming rate. The WAT is detected by a deviation of experimental data from the reference baseline and typically requires a significant amount of wax formation for sensible detection. Consequently, it may be difficult to obtain a reliable baseline and to pinpoint the deviation from the baseline, especially when the crystallization rate is low and signal noise overshadows thermal effects. Measured WAT tends to be lower than the actual temperature of initial wax formation, and interpretation of WAT by DSC depends on the subjective experience of the operator.

Fourier Transform Infrared Spectroscopy (FT-IR) has also been used to measure WAT. This complicated method requires the operator to identify the linear regions in wave numbers and to calculate the WAT by determining the intersection of two nonparallel lines generated when temperatures are higher and lower than the calculated WAT. This makes it difficult to detect the WAT if wax formation is gradual and the deviation of parallel lines is subtle. Similar to DSC, this method requires large amount of wax for detection, and the interpretation of WAT depends on the operator's experience.

Direct measurement methods yield more reliable results, but are tedious, difficult to perform and lack standardization. Cross polarization microscopy (CPM) is generally accepted to be the most sensitive visual method for measuring WAT, but is time-consuming and requires experienced human interpretation. With CPM, a sample is preheated and transferred to a microscope slide. Wax crystals on the slide are detected by rotating a polarization plane and observing the effects of light on the sample. The sensitivity of CPM depends on the size of wax and film thickness, as well as scale of magnification. However, the restricted field view makes it difficult to detect the first crystal. The CPM method requires an experienced operator to prepare the microscopy slides, set up the microscope and distinguish the first wax crystals from the images. It is therefore not a practical method to use in a daily routine as an analytical tool for WAT measurement.

Visual detection by ASTM method D2500 is limited to fuels that are transparent in a minimum of 40 mm thickness, with cloud points below 49°C. The method's sensitivity depends on the amount and size of wax, and the subjective judgment of the operator. It is a time consuming method. Despite this, D2500 has been used by some to measure cloud point or WAT of crude oils. But since most crude oils are opaque visually before reaching 40mm in thickness and some may have a WAT that is warmer than 49°C, D2500 is not applicable for use with most crude oils.

Solution Details:

Phase Technology's standardized cloud point test method, ASTM D5773, designed for transparent petroleum liquids has been successfully modified to measure the wax appearance temperature (WAT) of opaque crude oils. The method can also determine the temperature needed to completely dissolve the wax after the crude has partially solidified. This is known as the wax disappearance temperature (WDT).

Using this method, relative amount of wax formation and dissolution among different crudes can be compared as a function of temperature. The technique involves a direct detection of light scattered by wax crystals. It is sensitive, precise and reasonably quick in delivering results, making it a useful tool to generate design and operational data to address wax deposition challenges.

The WAT-70Xi crude oil analyzer utilizes an enhanced optical configuration and reduced sample thickness to penetrate through visually opaque samples. As shown in Fig. 1 (below), a thin layer (< 1 mm) of sample is sandwiched between a transparent flat cover and a metal surface which is continuously being heated or cooled by a thermoelectric cooler. An RTD temperature sensor is also installed to measure the temperature of the sample. When there is no wax in the sample, incident light at an oblique angle penetrates the transparent cover and the sample, and is specularly reflected off to the opposite direction. The minimal amount of light detected by an overhead optical detector represents the baseline signal of the device.

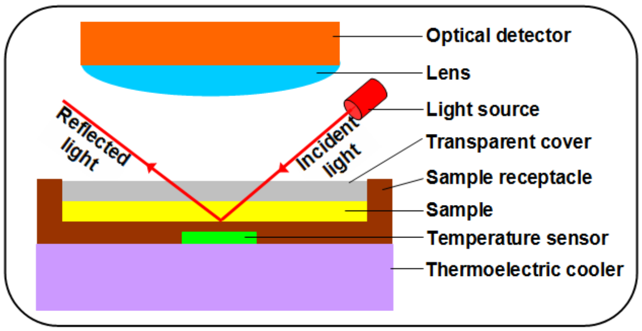

Fig. 1
Absence of wax in the sample

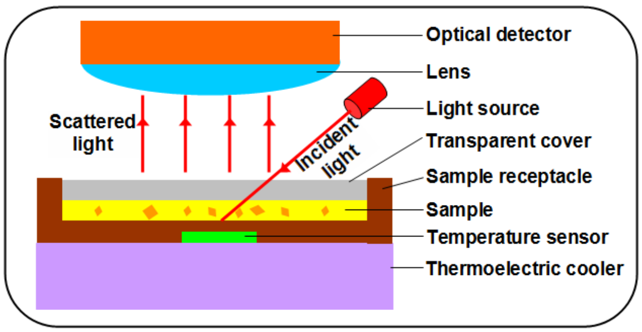

Fig. 2
Presence of wax in the sample

When waxes are present in the sample incident light is scattered, as shown in Fig. 2 (above). The scattered light emerges from the sample and is focused through the lens to the optical detector. WAT is the temperature at which light signal monitored by the optical detector starts to increase from the baseline, and WDT is the temperature at which the light signal returns to the baseline.

Phase Technology's WAT-70Xi determines both WAT and WDT automatically. Test results also provide comparative information on the rates of wax deposition and melting. The analyzer is simple to operate, sensitive to first wax appearance, and repeatable to within 1°C. The method is relatively fast, typically requiring 20-40 minutes to complete.

Fig. 3 (below) illustrates the determination of WAT and WDT using the Phase Technology WAT-70Xi. The plot shows the thermal trajectory of a Saudi Arabian crude starting at 65°C, at which point it is completely liquid with no wax (low scattered optical signal). The sample is cooled and its optical signal is continuously being monitored. The signal begins to rise as wax starts to form. WAT is determined to be 25.1°C.

To measure the WDT, the sample is cooled further to incur more waxes before it is warmed and allowed to melt completely. The WDT is determined to be the temperature at which the optical signal returns to the baseline; in this case, 43.9°C.

Fig. 3 additionally provides information of the rate of optical signal rise during cooling and the rate of decrease during warming, related to the rate of wax deposition and melting, respectively.

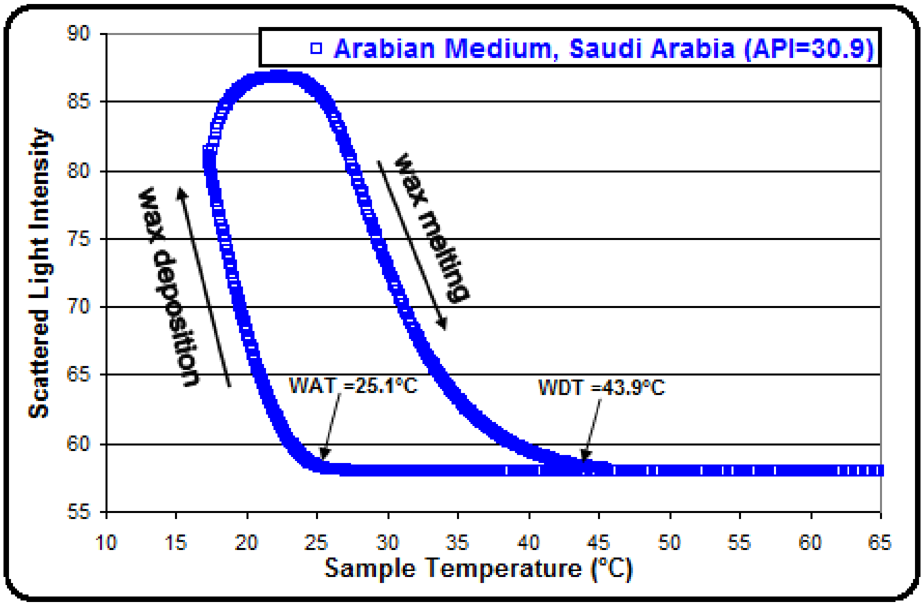

Fig. 3
Phase plot of Arabian Medium crude oil, Saudi Arabia

Solution Benefits:

Phase Technology's WAT-70Xi analyzer provides a valuable tool for crude oil analysis in upstream and midstream operations. As the first and only instrument of its kind, the WAT-70Xi is fully automatic, capable of testing opaque samples with speed and precision that has previously been unachievable. Each test yields two useful results, WAT and WDT. Subjective interpretation is eliminated.

In oilfield applications, users will find that WAT or WDT can assist in determining optimum levels of wax crystal modifiers and/or wax deposition inhibitors. WAT is also an indicator of potential crude incompatibility and a monitor of evolving crude quality. Crude oils from the same region may have quite different characteristics, with disparate rates of wax deposition and dissolution. Changes of location, extraction depth, evolution of time, or even methods of production and blending can all be verified by WAT.

For transport of crude oil via pipeline, railway or tanker, as well as for oil storage, wax crystals may restrict flow or create a total blockage. Companies engaged in midstream operations will find that WAT and WDT can help define acceptable operability limits and prevent the downtime and expense associated with cleaning. The design and development of subseas and land pipeline systems and implementation of wax remediation options benefit from analysis of WAT data.

The WAT-70Xi's intuitive interface and ease of use makes it simple to operate, with minimal training required. Since sample is injected directly into the analyzer, there is no tedious manual set-up needed. A built-in self-cleaning system relieves operators of an otherwise time-consuming task.

Summary:

Wax appearance temperature (WAT) and wax disappearance temperature (WDT) are now easily measured with precise, reliable results using the Phase Technology WAT-70Xi analyzer. For monitoring of ongoing crude quality and blending, as well as the design and management of flowlines, the WAT-70Xi provides a cost-effective solution that will positively impact ROI for companies in the upstream and midstream petroleum sectors.

Contact Phase Technology for further information, including a complimentary evaluation of specific crude oil samples using the WAT-70Xi analyzer

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