

70Xi Phase Temperature Control Software (PTCS)

Phase Technology's Phase Temperature Control Software (PTCS) is an optional, but substantial, feature to the 70Xi Analyzer. It extends the utility and flexibility of the analyzer by allowing customized tests for the analyzer's powerful crystallization and motion detection methods.

Phase Change Characterization

The 70Xi Analyzer's crystallization detection method utilizes an innovative, patented Diffusive Light Scattering (DLS) technique to characterize phase changes with extreme sensitivity and accuracy. DLS detection of crystal formation is shown below:

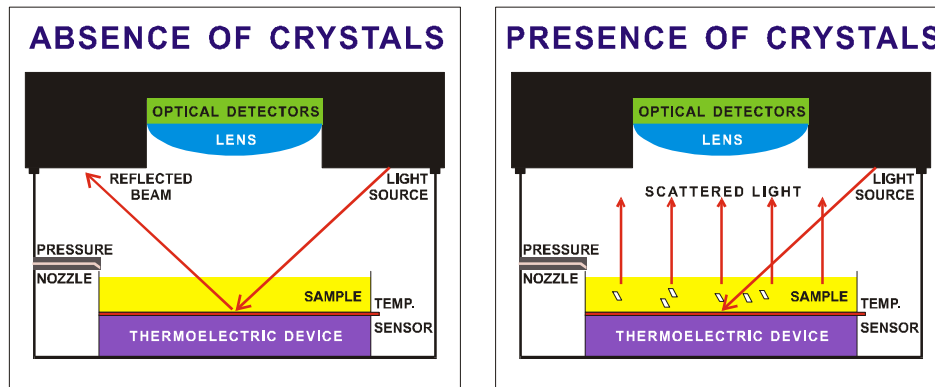


Figure 1: *Crystallization Detection Method*

How Phase Technology's Diffusive Light Scattering (DLS) technology works:

- A light beam focuses onto the test sample, while a matrix of optical detectors continuously monitors the light scattered by the sample as it is subjected to prescribed temperature changes.
- The optical sensors are able to detect minute quantities of crystals wherever they are formed in the sample.
- With this design, crystal initiation and growth (or phase transition in general) during a cooling cycle, as well as crystal melting and final disappearance in a warming cycle, can all be elucidated in a sensitive and repeatable manner.

PTCS provides the tools to experiment with various temperature programs. The multiple steps of target temperature, cooling/warming rate and soak time are user-defined. The 70Xi Analyzer will execute the program and simultaneously output the scattered-light signal in graphic form (Fig.2) as functions of sample temperature. The generated data can then be stored for further processing and analysis, and the format is compatible with most spreadsheet software.

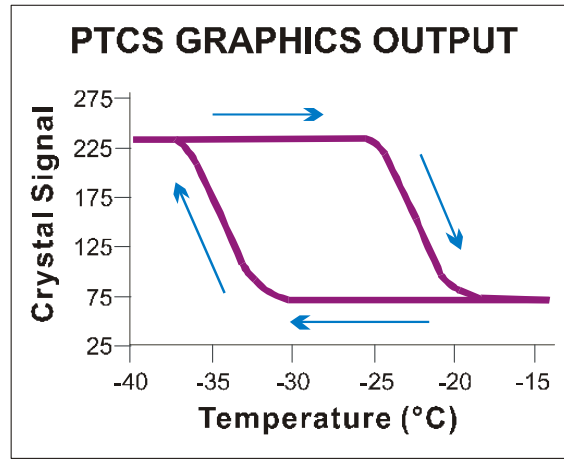


Figure 2: *Temperature vs. Optics Signal Plot*

Flow Resistance Characterization

The 70Xi Analyzer utilizes a patented motion detection method (demonstrated below) to quantify the test sample's resistance to flow under a calibrated perturbation.

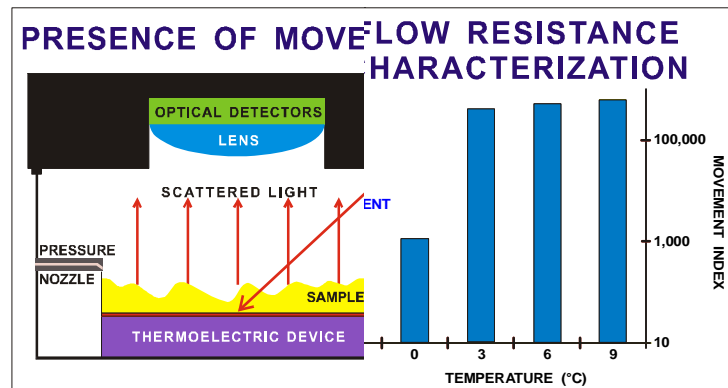


Figure 3: *Motion Detection Method*

How Phase Technology's motion detection method works:

- At a given test temperature, a pulse of dried air through a built-in nozzle is directed onto the sample surface while the analyzer's matrix of optical detectors continuously monitors the sample for movement caused by the perturbation.
- By examining and comparing hundreds of signal profiles before, during and after the pulsing event, the 70Xi Analyzer is able to determine the resultant relative amount of motion.
- In turn, the movement signal can be correlated with the sample's viscous response—for example, flow resistance as a function of temperature

PTCS provides the tools to experiment with various pulsing programs. The user can specify the target temperature, the cooling or warming rate to arrive at the target temperature, the magnitude and force of the pulse, and the temperature interval for applying each pulse. The 70Xi Analyzer will execute the program and simultaneously output a movement index corresponding to each pulse.

Additionally, the user can switch between the movement index plot (Motion Detection Method, above) and the Diffusive Light Scattering (DLS) signal plot (corresponding to crystallization). This allows for monitoring of both Sample Flow Resistance and Crystallization simultaneously

PTCS in Action

Example 1: Study of Crystallization as a Function of Sample Temperature

The following PTCS program shows a typical crystallization study of a crude oil:

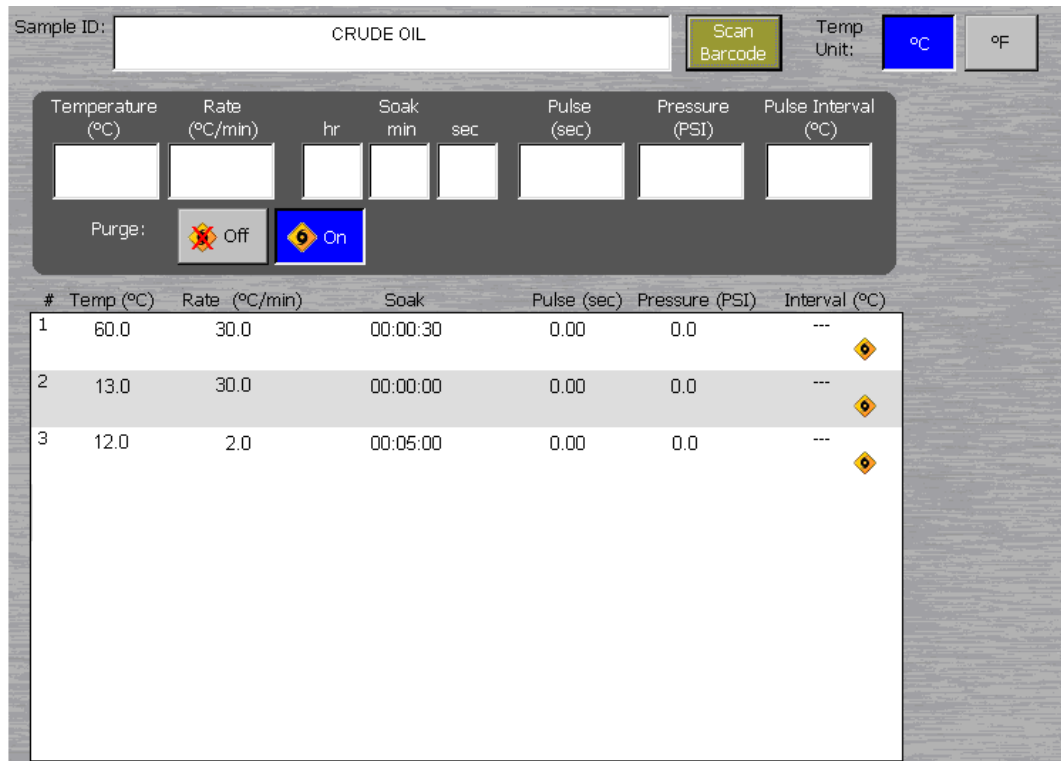


Figure 4: *PTCS Crystallization Study*

1. In step 1, the sample is warmed to 60°C (starting at room temperature) at a rate of 30°C/min. Once it has reached 60°C, it soaks at this temperature for 30 seconds. This step is designed to remove or minimize any moisture effect in the oil sample.
2. The sample cools (from 60°C) to 13°C at 30°C/min.

- The sample cools to 12°C at a slower rate of 2°C/min. Once the sample has reached 12°C, it soaks at this temperature for 5 minutes to ascertain whether or not crystallization takes place. At the conclusion of the 5-minute soaking period, the analyzer will automatically return to room temperature and be ready for another test.
- The Phase Plot corresponding to this PTCS program is shown in the following diagram. This figure plots the scattered-light signal as a function of sample temperature. The sharp rise in signal at 12°C indicates that many crystals were formed during the 5 minute soak period.

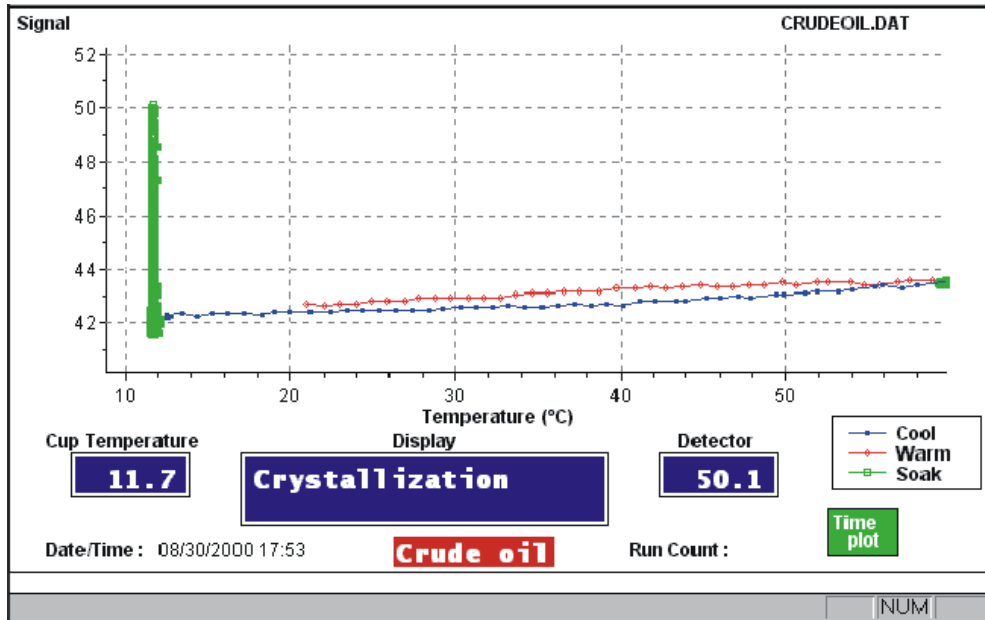


Figure 5: Phase Plot Showing Scattered-Light Signal as a Function of Sample Temperature

Example 2: Study of Flow Resistance of a Base Oil as a Function of Temperature

In this example, the PTCS program consists of only two steps.



Figure 6: **Flow Resistance Test**

1. The base oil cools to 3°C (starting at room temperature) at a rate of 10°C/min.
2. The analyzer first soaks the sample for 30 seconds at the current temperature of 3°C so that the sample has sufficient time to equilibrate. This is followed by a perturbing pressure of 1 psig pulse for duration of 0.65 sec. at the sample at every 3°C interval
3. After the pulse, the 70Xi Analyzer cools the sample at a rate of 1°C/min until it reaches the next pulse temperature (0°C). At this temperature, the analyzer again soaks for 30 sec before pulsing for 0.65 sec using 1 psig of air.
4. The analyzer continues cooling and pulsing in 3°C pulse intervals until the sample temperature has reached -12°C. After the last pulse at -12°C, the PTCS program ends, and the analyzer automatically warms back up to room temperature.
5. The Phase Plot corresponding to this PTCS program is shown below. This Phase Plot demonstrates the relative movement index as a function of sample temperature. The amount of motion experienced by the base oil as a result of each pulse of air is quantified by the green bars. At 3°C, a large movement signal of 40574 indicates a good deal of flow. The amount of sample movement decreases with decreasing temperature until -12°C when there is no flow.

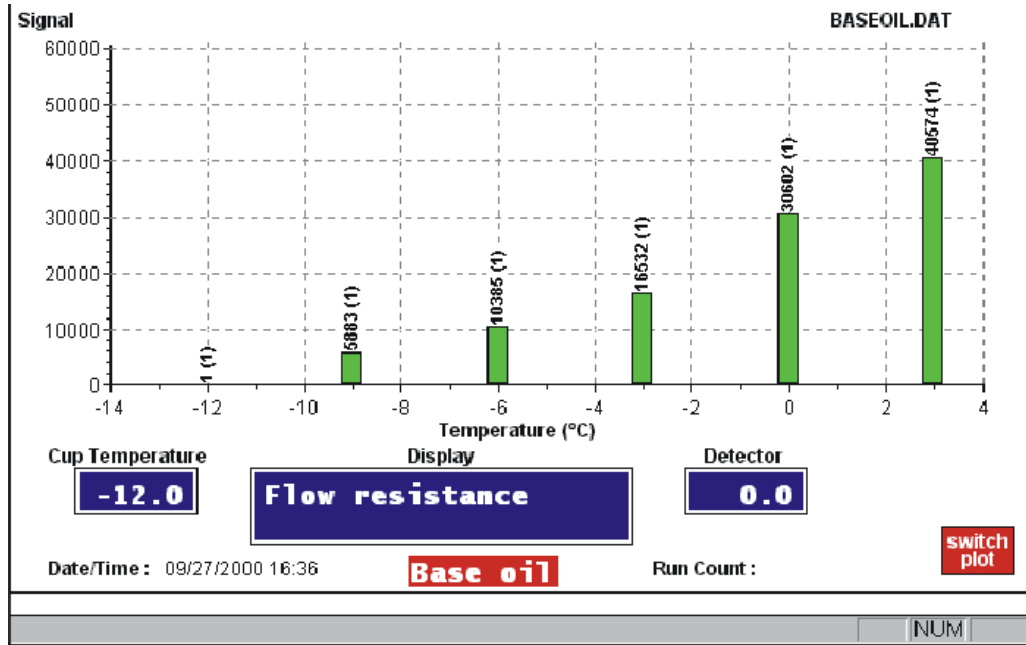


Figure 7: Phase Plot Showing Relative Movement index as a function of sample temperature