Preheating for No-Flow Point and Pour Point of Petroleum Products and Liquid Fuels According to ASTM D7346.

- Proven technology per ASTM D7346 no-flow point
- High accuracy & precision
- Excellent correlation with standard method
- Pour Point ASTM D 7346 at 3°C intervals

Keywords: ASTM D7346, No-Flow Point, ASTM D97, Correlation, Preheating for Pour Point

INTRODUCTION

OptiMPP Automatic Mini Cloud and Pour Point Analyzer provides accurate and precise no-flow point, pour point, cloud point, and, if required, the non-rounded temperature of the cloud point of most hydrocarbons. These include lubricating oils, base stock oils, polyalphaolefins, distillate fuels, liquid petroleum products, and biodiesel.

OptiMPP is the next generation of MPP 5Gs and is designed with the same patented technology in compliance with ASTM D7346-19 and later standard methods for pour and no-flow points determination, and with ASTM D7689-17 and later standard methods for cloud point determination. It also correlates with the ASTM D2500, ASTM D5771, ASTM D5772, ISO 3015, IP 219, IP 444, JIS K2269 standard methods for the cloud point determination and with D6892, ASTM D97, ASTM D5950, ISO 3016, IP 15, JIS K2269 for the pour and no-flow points determination.

MEASURING PRINCIPLE

OptiMPP Automatic Mini Cloud and Pour Point Analyzer automatically controls cooling and heating of the specimen following standard prescribed rates. Optical detectors monitor the appearance and disappearance of hydrocarbon crystals, while the differential pressure detector monitors air pressure variation in the vial for the entire test, down to -95°C with 0.1°C resolution.

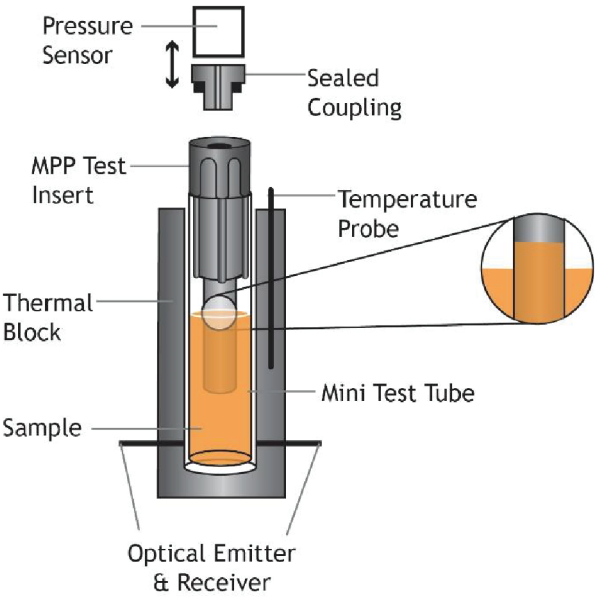
Pour Point

Pour point is the lowest temperature at which the product continues to flow. Pour point is generally close to the cloud point for base oil, although the difference between these two points may be as much as 30°C for mixed oil. The OptiMPP deduces the pour point from the no-flow point and the required interval if programmed (from 0 to 5°C with 0.5°C resolution).

No-Flow Point

By definition, the no-flow point of a product is the precise temperature of a test specimen at which a wax crystal structure and/or viscosity increases and impedes the movement of the surface of the test specimen under the test condition. When found, the sample can be heated up back to the ambient temperature.

No-Flow Point Determination by Mini Method (ASTM D7346)
As they cool, the sample and the air gap on top of the sample and air volume will decrease, causing the sample to flow to the measuring cell. When the viscosity is too high (pour point), the sample will not flow and a differential pressure is measured. The pour point is detected in this manner.

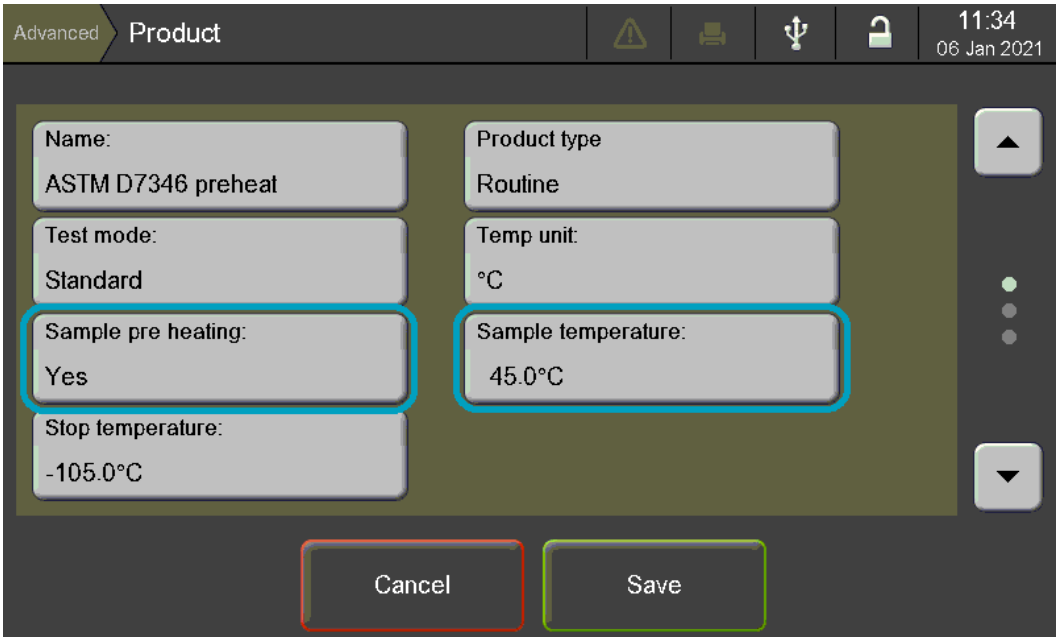


While cooling the sample and the air gap on top of the sample, the sample and air volume will decrease, causing the sample to flow to the measuring cell. When the visocsity is too high (pour point), the sample will not flow and a differential pressure is measured. In this way, the pour point is detected.

TEST PROCESS
OptiMPP measures the no-flow point of the product and calculates pour point. The difference between these is that the no-flow point is detected when the surface of the test specimen is not moving, while pour point is the lowest temperature at which the product continues to flow. The difference between these two values is so small that method D7346 defines the calculation of no-flow point as “round the no flow point temperature to the next warmer 1 °C interval or 3 °C interval, as specified, and record.”

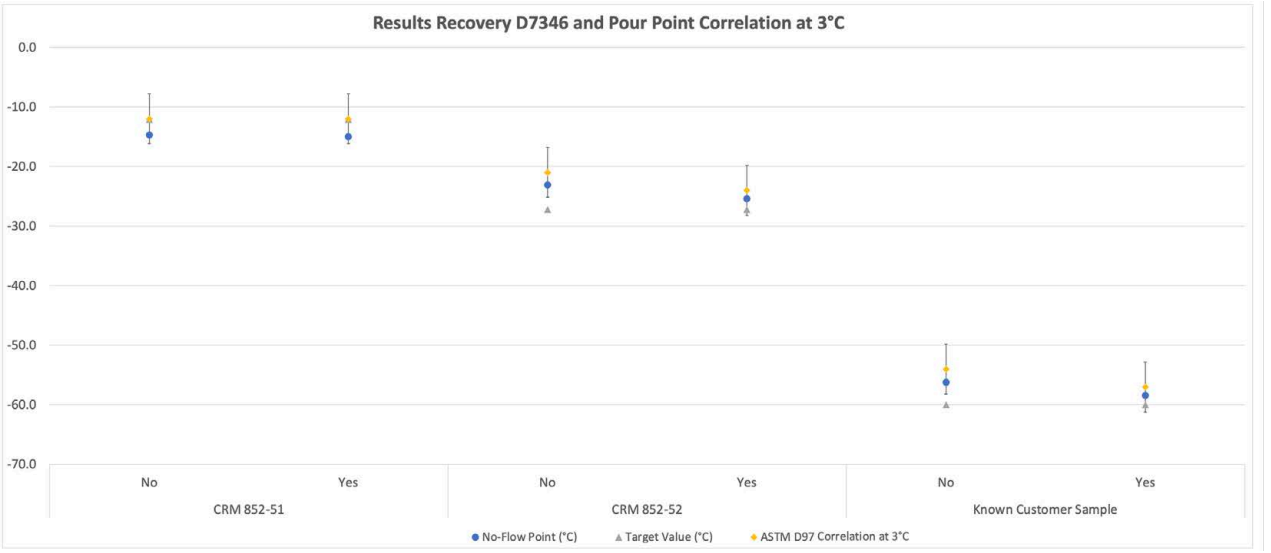
OptiMPP deduces the pour point from the no-flow point at the required interval by default at 3 °C, as described in the D6892, ASTM D97, ASTM D5950, and SO 3016 methods.

TEST PARAMETERS
By default, the preheating is not active and should be activated in a product setting. Select “Yes” to heat the sample up to the configured temperature according to applicable Standard Method. The preheating temperature cannot above 60 °C and usually set up at 45 °C for low pour point.



Results Recovery D7346						
Sample Name	Preheating at 45 °C	Target Value (°C)	Tolerance (°C)	No-Flow Point (°C)	ASTM D97 Correlation at 3 °C	In Specification
CRM 852-51	No	-12.1	±4.2	-14.8	-12.0	Yes
CRM 852-51	Yes	-12.1	±4.2	-15.0	-12.0	Yes
CRM 852-52	No	-27.2	±4.2	-23.1	-21.0	No
CRM 852-52	Yes	-27.2	±4.2	-25.4	-24.0	Yes
Known Customer Sample	No	-60.0	±4.2	-56.2	-54.0	No
Known Customer Sample	Yes	-60.0	±4.2	-58.4	-57.0	Yes

Table 1: Overview of two PAC CRM reference samples and one known customer sample with and without preheating.



ANALYSIS

Preheating has a low effect for CRM 852-51, slightly increasing the no-flow point and keeping the result in the tolerance of D97 method. However, preheating has a higher effect, by increasing the no-flow point and changing the result from -21°C to -24°C in the D97 method.

CONCLUSION

- When heated to a temperature higher than 45 °C during the preceding 24 hours, some samples do not yield the same pour point results as when they are kept at room temperature for 24 hours prior to testing. Examples of materials which are known to show sensitivity to thermal history are residual fuels, black oils, and cylinder stocks.
- It is considered best practice to keep samples of residual fuels, black oils, and cylinder stocks that have been heated to a temperature higher than 45 °C during the preceding 24 hours at room temperature for 24 hours before testing. This should also be done for these sample types when the thermal history is unknown. Samples which are known not to be sensitive to thermal history do not need to be kept at room temperature for 24 hours before testing.
- The preheating has clearly improved the result for the no-flow point and, by consequence, the D97 result.

The OptiMPP complies with ASTM D7346-19 and later standard methods for pour point and no-flow point determination and has an accuracy and precision with perfect correlation with D6892, ASTM D97, ASTM D5950, ISO 3016, IP 15, JIS K2269 for the pour point determination.