TESC-5133 Low Temperature Viscometer

Fully automated ASTM D5133 conditioning and testing on the compact, economical CANNON® TESC-5133

Determination of flowability at low temperatures is a critical aspect of a lubricant's performance. ASTM D5133, Standard Test Method of Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature Scanning Technique[i], provides a procedure to determine the flowability of a lubricant over a temperature range as the temperature is slowly lowered while the apparent viscosity is continuously measured. The test results provide both the viscosity at multiple temperatures and Gelation Index for the sample.

Cannon Instrument Company developed the TESC-5133 to fully automate the D5133 procedure using the Thermoelectric Sample Conditioner (TESC) with a digital rotational viscometer. By consolidating the Brookfield® digital viscometer and a Cannon-developed thermoelectrically controlled sample chamber, the TESC-5133 automates the entire conditioning and testing process prescribed in ASTM D5133. This includes preheating, room temperature stabilization, cooling of the sample to test start temperature of -5 °C, and continuous apparent viscosity measurement of the sample throughout the cooling cycle of the sample from -5 °C to -40 °C. A computer software program collects the data throughout the process and analyzes the Gelation Index, Gelation Index temperature, and apparent viscosity of the sample, upon the end of the test. Automation minimizes the hands-on time needed to prepare and run a D5133 test, reduces variability of the data, and improves test precision.

Procedure

The CANNON TESC-5133 follows a sample preparation and test procedure equivalent to ASTM D5133. To minimize test variability, the TESC-5133 system automates/eliminates many of the steps required to transfer the sample between the conditioning and testing steps. Automation is achieved via a thermoelectrically controlled sample chamber that conditions and measures the sample viscosity without any operator intervention throughout the conditioning and testing processes.

Using a calibrated TESC-5133, an operator runs a D5133 test as follows:

1. Auto-Zero the Brookfield DV2T viscometer.
2. Pour 20 mL of sample into a 25 mm × 150 mm rimless test tube.
3. Carefully place the test tube with the sample in the TESC-5133 sample chamber.
4. Attach TESC-5133 spindle to the DV2T Head Unit and lower the viscometer into the run position.
5. Launch software in controlling computer and establish connection with the TESC instrument.
6. Press Run to start the measurement.

Once the temperature control program starts, the TESC-5133 system heats the sample to the preheat temperature from 20 °C to 90 °C in 15 minutes and holds at that temperature for the required time (1 hour and 45 minutes). The TESC low-temperature Viscometer then cools the sample to room temperature (20 °C) in 15 minutes and then further cools the sample to the desired test start temperature of -5 °C in 10 minutes, and holds at this temperature for 20 minutes in accordance with D5133 requirement. This automated heating and cooling is critical to reducing variability. The TESC-5133 program records the temperature of the sample chamber throughout the thermal conditioning process. Upon completion of the automated thermal conditioning steps of the sample, the TESC-5133 system automatically measures the sample viscosity while cooling down the sample temperature at a rate of 1°C per hour, from -5 °C to -42 °C. Once the viscosity measurements are completed, the TESC-5133 returns the sample temperature to 20 °C. The operator is then able to review the data and enhance the digital record with notes and any additional information. The TESC-5133 instrument is shipped with a set of thermal conditioning programs for D5133, as shown in Table 1.

Table 1: TESC-5133 Thermal Conditioning Steps

Program	TESC-5133	ASTM D5133
Start Preheat	20 °C to 85 °C in 15 minutes	Place in bath >85 °C
Preheat	Hold at 85 °C to 90 °C for 1 hour and 45 minutes (105 minutes)	Hold at 88 °C to 92 °C for 1.5 to 2 hours (90 minutes to 120 minutes)
Cool to room temperature	85 °C to 20 °C in 15 minutes	Cool to room temperature in 10 to 20 minutes
Time at – 5 °C	Cool to -5 °C in 10 minutes, hold for 20 minutes	Equilibrate for 15 to 30 minutes
Measure Viscosity	Cool at 1 °C per hour to -42 °C	Cool at 1 °C per hour to -40 °C

Data

The data presented in Table 2 demonstrate testing of eight ASTM Performance Test Program (PTP) lubricant oil samples on two different TESC-5133 instruments. The data was collected by two different operators in two different laboratories. Note: ASTM D5133 prescribes that Gelation Index values less than 6 be reported as <6 however the actual gelation index values from the TESC-5133 instruments is recorded in the data below.

Table 2: ASTM PTP Program LUxxxx Sample Data Summary

			Unit 1			Average		
ASTM PTP LU1509	**ASTM Data**	**ASTM Std Dev**	**TESC-5133 Run 1**	**TESC-5133 Run 2**	**TESC-5133 Run 3**	**TESC-5133 Unit 1**	**Sample Std Dev**	**Diff to ASTM**
SBV 5,000 cP	-24.2 °C	----	-24.8 °C	-23.3 °C	----	-24.1 °C	1.06	0.15
SBV 10,000 cP	-29.2 °C	----	-29.6 °C	-28.0 °C	----	-28.8 °C	1.13	0.40
SBV 20,000 cP	-34.0 °C	----	-34.5 °C	-33.0 °C	----	-33.8 °C	1.06	0.25
SBV 30,000 cP	-36.6 °C	----	-37.0 °C	-35.4 °C	----	-36.2 °C	1.13	0.40
SBV 40,000 cP	-38.4 °C	----	-38.8 °C	-36.9 °C	----	-37.9 °C	1.34	0.55
Gelation Index	5.6	----	5.6	5.2	----	5.4	----	-0.20
Gelation Index Temperature	----	----	-24.5 °C	-24.1 °C	----	-24.3 °C	0.28	----

			Unit 1			Average		
ASTM PTP LU1805	**ASTM Data**	**ASTM Std Dev**	**TESC-5133 Run 1**	**TESC-5133 Run 2**	**TESC-5133 Run 3**	**TESC-5133 Unit 1**	**Sample Std Dev**	**Diff to ASTM**
SBV 5,000 cP	-25.8 °C	----	-25.6 °C	-25.7 °C	-25.0 °C	-25.4 °C	0.38	0.37
SBV 10,000 cP	-30.8 °C	----	-30.8 °C	-31.0 °C	-30.4 °C	-30.7 °C	0.31	0.07
SBV 20,000 cP	-35.1 °C	----	-35.1 °C	-35.1 °C	-34.7 °C	-35.0 °C	0.23	0.13
SBV 30,000 cP	-37.4 °C	----	-37.4 °C	-37.5 °C	-36.9 °C	-37.3 °C	0.32	0.13
SBV 40,000 cP	-38.7 °C	----	-38.9 °C	-38.8 °C	-38.3 °C	-38.7 °C	0.32	0.03
Gelation Index	< 6	----	5.1	5.2	5.3	5.2	----	----
Gelation Index Temperature	----	----	-40.0 °C	-16.5 °C	-16.5 °C	-24.3 °C	13.57	----

			Unit 1			Average		
ASTM PTP LU1909	**ASTM Data**	**ASTM Std Dev**	**TESC-5133 Run 1**	**TESC-5133 Run 2**	**TESC-5133 Run 3**	**TESC-5133 Unit 1**	**Sample Std Dev**	**Diff to ASTM**
SBV 5,000 cP	-29.2 °C	0.74	-28.5 °C	-28.2 °C	-28.9 °C	-28.5 °C	0.35	0.67
SBV 10,000 cP	-33.3 °C	0.64	-32.7 °C	-32.3 °C	-33.1 °C	-32.7 °C	0.40	0.60
SBV 20,000 cP	-37.5 °C	0.63	-36.7 °C	-36.2 °C	-37.1 °C	-36.7 °C	0.45	0.83
SBV 30,000 cP	-39.4 °C	0.79	-38.9 °C	-38.4 °C	-39.3 °C	-38.9 °C	0.45	0.53
SBV 40,000 cP	----	----	-40.3 °C	-39.8 °C	-40.8 °C	-40.3 °C	0.50	----
Gelation Index	< 6	----	5.2	4.9	5.5	5.2	----	----
Gelation Index Temperature	----	----	-10.4 °C	-32.6 °C	-11.3 °C	-18.1 °C	12.57	----

			Unit 2			Average		
ASTM PTP LU1801	**ASTM Data**	**ASTM Std Dev**	**TESC-5133 Run 1**	**TESC-5133 Run 2**	**TESC-5133 Run 3**	**TESC-5133 Unit 2**	**Sample Std Dev**	**Diff to ASTM**
SBV 5,000 cP	-25.9 °C	0.38	-25.0 °C	-25.5 °C	-25.3 °C	-25.3 °C	0.25	0.66
SBV 10,000 cP	-31.5 °C	0.18	-30.9 °C	-31.4 °C	-31.4 °C	-31.2 °C	0.29	0.37
SBV 20,000 cP	-36.5 °C	0.17	-36.0 °C	-36.5 °C	-36.5 °C	-36.3 °C	0.29	0.27
SBV 30,000 cP	-39.2 °C	0.14	-38.7 °C	-39.1 °C	-39.2 °C	-38.9 °C	0.26	0.30
SBV 40,000 cP	----	----	----	----	----	----	----	----
Gelation Index	< 6	----	4.2	3.7	3.6	3.83	0.32	----
Gelation Index Temperature	----	----	-10.2 °C	-39.3 °C	-37.9 °C	-24.8 °C	16.41	----

			Unit 2			Average		
ASTM PTP LU1809	**ASTM Data**	**ASTM Std Dev**	**TESC-5133 Run 1**	**TESC-5133 Run 2**	**TESC-5133 Run 3**	**TESC-5133 Unit 2**	**Sample Std Dev**	**Diff to ASTM**
SBV 5,000 cP	-27.8 °C	0.65	-27.8 °C	-28.4 °C	-27.6 °C	-28.1 °C	0.42	-0.29
SBV 10,000 cP	-33.2 °C	0.91	-33.3 °C	-33.8 °C	-33.1 °C	-33.6 °C	0.36	-0.40
SBV 20,000 cP	-37.9 °C	0.39	-38.0 °C	-38.7 °C	-38.1 °C	-38.4 °C	0.38	-0.42
SBV 30,000 cP	----	----	-40.5 °C	-41.2 °C	-40.5 °C	-40.9 °C	0.40	----
SBV 40,000 cP	----	----	----	----	----	----	----	----
Gelation Index	< 6	----	5.2	4.5	4.2	4.9	----	----
Gelation Index Temperature	----	----	-10.2 °C	-10.0 °C	-10.9 °C	-10.4 °C	0.47	----

			Unit 2			Average		
ASTM PTP LU1901	**ASTM Data**	**ASTM Std Dev**	**TESC-5133 Run 1**	**TESC-5133 Run 2**	**TESC-5133 Run 3**	**TESC-5133 Unit 2**	**Sample Std Dev**	**Diff to ASTM**
SBV 5,000 cP	-26.0 °C	0.79	-26.5 °C	-25.9 °C	-25.7 °C	-26.2 °C	0.42	-0.22
SBV 10,000 cP	-31.1 °C	0.44	-31.9 °C	-31.5 °C	-31.3 °C	-31.7 °C	0.31	-0.60
SBV 20,000 cP	-35.7 °C	0.33	-36.6 °C	-36.2 °C	-36.1 °C	-36.4 °C	0.26	-0.68
SBV 30,000 cP	-38.2 °C	0.35	-39.0 °C	-38.7 °C	-38.7 °C	-38.9 °C	0.17	-0.64
SBV 40,000 cP	----	----	-40.6 °C	-40.4 °C	-40.2 °C	-40.5 °C	0.20	----
Gelation Index	<6	----	4.8	4.1	4.1	4.3	0.40	----
Gelation Index Temperature	----	----	-11.6 °C	-12.2 °C	-10.9 °C	-11.6 °C	0.65	----

			Unit 2			Average		
ASTM PTP LU1905	**ASTM Data**	**ASTM Std Dev**	**TESC 5133 Run 1**	**TESC 5133 Run 2**	**TESC 5133 Run 3**	**TESC 5133**	**Sample Std Dev**	**Diff to ASTM**
SBV 5,000 cP	-16.0 °C	0.45	-16.1 °C	-15.7 °C	-15.5 °C	-15.8 °C	0.31	0.23
SBV 10,000 cP	-22.0 °C	0.31	-22.1 °C	-21.8 °C	-21.6 °C	-21.8 °C	0.25	0.18
SBV 20,000 cP	-27.3 °C	0.26	-27.4 °C	-27.2 °C	-26.9 °C	-27.2 °C	0.25	0.17
SBV 30,000 cP	-30.2 °C	0.3	-30.3 °C	-30.2 °C	-29.9 °C	-30.1 °C	0.21	0.03
SBV 40,000 cP	-32.1 °C	0.32	-32.2 °C	-32.1 °C	-31.8 °C	-32.0 °C	0.21	0.03
Gelation Index	< 6	----	3.8	3.9	4.0	3.9	----	----
Gelation Index Temperature	----	----	-35 °C	-34.7 °C	-35.9 °C	-35.2 °C	0.62	----

			Unit 2			Average		
ASTM PTP LU2001	**ASTM Data**	**ASTM Std Dev**	**TESC 5133 Run 1**	**TESC 5133 Run 2**	**TESC 5133 Run 3**	**TESC 5133**	**Sample Std Dev**	**Diff to ASTM**
SBV 5,000 cP	-12.8 °C	0.79	-12.2 °C	-12.6 °C	-12.8 °C	-12.5 °C	0.31	0.24
SBV 10,000 cP	-18.7 °C	0.55	-18.2 °C	-18.6 °C	-18.9 °C	-18.6 °C	0.35	0.17
SBV 20,000 cP	-23.8 °C	0.28	-23.6 °C	-23.9 °C	-24.0 °C	-23.8 °C	0.21	-0.08
SBV 30,000 cP	-26.5 °C	0.25	-26.3 °C	-26.7 °C	-26.6 °C	-26.5 °C	0.21	0.00
SBV 40,000 cP	-28.5 °C	0.22	-28.1 °C	-28.6 °C	-28.4 °C	-28.4 °C	0.25	0.08
Gelation Index	< 6	----	3.9	3.8	4	3.9	0.10	----
Gelation Index Temperature	----	----	-26.9 °C	-32.1 °C	-25.8	-28.3 °C	3.37	----

Precision data as per ASTM D5133 is presented in Table 3.

Table 3: ASTM D5133 Precision

ASTM D5133	ASTM D5133	TESC System Estimates
Repeatability at specified viscosity temperature	2 °C	1.2 °C
Reproducibility at specified viscosity temperature	2 °C	1.5 °C
Repeatability Gelation Index value	16%	10%
Reproducibility Gelation Index value	29 %	15%
Repeatability Gelation Index temperature	2 °C	2 °C
Reproducibility Gelation Index temperature	2 °C	2 °C

Discussion and Conclusion

Results from ASTM ILCP lubricant samples clearly demonstrate the TESC-5133 system capability to measure the low temperature behavior of samples according to ASTM D5133 within the tolerances and precision prescribed in the standard. The TESC-5133 measurements from 8 LU samples showed results comparable reported from ASTM ILCP. Since all eight tested samples from ASTM ILCP have Gelation Index less than six, the Gelation Index temperature was not reported in the ILCP results. Table 4 compares six samples using TESC-5133 and independent laboratory testing using ASTM D5133. Both Gelation Index (GI) and Gelation Index Temperature of TESC-5133 for these six samples are well within the reproducibility tolerances prescribed in the ASTM D5133 test method.

Table 4: Comparing TESC-5133 and ASTM D5133

Sample	ASTM D5133		TESC-5133	
	Gelation Index	GI Temperature	Gelation Index	GI Temperature
V-B3	<6	N/A	4	-40.1 °C
LTEP02	12.8	-22.0 °C	14.3	-22.3 °C
E	15.8	-22.0 °C	17.3	-23.5 °C
SP-B3	11.2	-23.0 °C	14.7	-23.1 °C
SP-B4	9.0	-23.0 °C	9.4	-23.7 °C
LTGI-1	16.5	-30.0 °C	22.3	-30.4 °C

As demonstrated in the data comparison between the TESC system and the method's published precision, the TESC System meets measurement requirements in ASTM D5133 for LU samples as well. This is the first ASTM D5133 sample conditioning system that automates not only the low temperature portion of the thermal conditioning process, but also the preheat and room temperature stabilization steps. Because all conditioning steps are carried out with the sample in place within the sample chamber, the TESC System minimizes or eliminates many data quality issues associated with temperature fluctuations and sample disruption during the D5133 sample conditioning and viscosity measurement steps, thereby decreasing variability in the data.

The TESC-5133 low temperature viscometer also incorporates programmed preheat and cooling to test start temperature, and stabilization at the test start temperature, ensuring that all samples are subjected to the same conditions throughout the conditioning process. Furthermore, since the samples are not transferred from an external conditioning set up to the viscometer, temperature fluctuations resulting from such transfer are effectively eliminated. This ensures more consistent thermal conditioning and a repeatable viscosity measurement temperature. The TESC system also incorporates a compact sample chamber, only slightly larger than the sample stator, creating a tightly controlled conditioning environment and minimizing temperature control and variation concerns that can arise with coolant circulation systems. In the run position, the Brookfield® DV2T viscometer completely covers the sample chamber and isolates it from ambient conditions throughout the sample conditioning and testing steps to ensure consistent temperature control. Henderson and Mastropierro[ii] demonstrated that automation of all the procedural steps in the D5133 test reduced variability by approximately 50% (JTE-2019-0944).

A reference standard is used to verify appropriate instrument programming, calibration, and temperature control during instrument set up. Sample chamber temperatures are automatically measured by an integrated RTD temperature sensor throughout the conditioning and testing process. The thermal conditioning history is included in the data file along with measured viscosity and may be obtained by the user after test completion.

In addition to advanced thermal control, elimination of the need for sample transfer from a conditioning instrument to a viscometer allows the TESC System to improve test precision due to reduced sample disruption. Attaching and positioning the spindle during initial set up in the TESC system, rather than immediately prior to the viscosity measurement in a non-consolidated instrument, also helps to lessen the impact of sample disruption on test results and enhances- measurement precision as the sample remains undisturbed until testing is initiated in the TESC.

Along with improvements in test precision and accuracy, the TESC-5133 low temperature viscometer offers a number of advantages over alternative ASTM D5133 sample conditioning options. Automation and consolidation of the entire conditioning and testing process reduces operator hands-on time and minimizes operator errors in the testing procedure. Pre-programmed test profiles for typical lubricant engine oil viscosity ranges reduces set up time and ensures consistency in sample conditioning.

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[i] ASTM D5133-20a, Standard Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique, ASTM International, West Conshohocken, PA, 2020, www.astm.org, http://www.astm.org/cgi-bin/resolver.cgi?D5133

[ii] K. Henderson and J. Mastropierro, "Automatically Assessing Low-Temperature Low-Shear Viscometric Response of Engine Lubricants with Decreasing Temperature," *Journal of Testing and Evaluation 49*. Published ahead of print, 16 June 2020, https://doi.org/10.1520/JTE20190944 .