Investigation of viscosity, density and cloud point of diesel and biodiesel blends

Relevant for: producers (QC), fuel research, fuel wholesale traders, bulk consumers (e.g. vehicle fleet managers for incoming QC), testing labs, R&D

Besides kinematic viscosity and density according to ASTM D7042 and D4052, SVM 3001 Cold Properties also simultaneously determines the cloud point of diesel fuels thanks to its built-in optical cell.

1 Introduction

Legislation and the importance of sustainability necessitate more investigation into fuels for our every-day needs. This results in an increased focus on renewable resources.

Diesel fuels both from petroleum and renewable sources and blends thereof need to fulfill a number of criteria, including

- Kinematic viscosity at 40 °C
- Density at 15 °C,
- Kinematic viscosity at -20 °C for winter diesel used in cold and arctic climate areas
- Cold flow properties: Cloud Point (CP) / Cold Filter Plugging Point (CFPP) / Low Temperature Flow Test (LFTF)

in order to satisfy various specifications as defined by ASTM D975, ASTM D6751, ASTM D7467, EN 590, EN 14214, EN 16734, EN 16709 and others.

SVM 3001 Cold Properties provides kinematic viscosity, density and the cloud point in one go as a scan. Measuring kinematic viscosity at -20 °C does not require external counter cooling equipment.

2 Why determine viscosity?

Specification of a lower and upper limit for kinematic viscosity is important, as too low viscosity leads to increased wear and failure of injectors and injection pump. Too high viscosity impacts the droplet size and causes poor injection, incomplete combustion and higher emissions. High viscosity could also cause excessive fuel injection pressure during engine warm-up. Further, the engine could be starved for fuel at low temperatures, as the fuel moves slowly through the fuel filter and fuel lines.The maximum viscosity is limited by design of engine and injection system. For example, the viscosity of diesel fuel. No. 2 is typically in the range of 2.5 to 3.2 mm²/s at 40 °C to achieve optimum combustion.

3 Why determine cold flow properties?

There are two important cold flow parameters to indicate at which temperatures a diesel fuel or home heating oil can be used: The cloud point (CP) and the cold filter plugging point (CFPP).

The first one is the CP. It is the temperature in °C, at which the clear liquid starts to separate paraffin crystals, which make it turbid or cloudy. The CP is determined under defined testing conditions depending on the standard test method that is employed.

While the CP is not explicitly specified for all diesel types and in all countries, it is nevertheless an important parameter. ASTM D6751 (B6 – B20) and D7467 (B100) require to report the CP, although there is no set limit. EN 590 specifies maximum CPs for arctic and severe winter climates. Typically, the CP is approx. 10 °C above the CFPP.

The second important cold flow property is the cold filter plugging point (CFPP) for diesel fuels and home heating oil extra-light (HHO EL). It indicates the temperature at which the filter in the fuel system clogs up, so that the liquid cannot be filtered any more. The fuel flow will be reduced and eventually interrupted completely. CP and CFPP together are important to

indicate at which temperatures the diesel fuel or home heating oil can be used.

The cold flow properties can be changed by using flow improver additives. Another strategy to prevent clouding or clogging is to heat the filter and parts of the fuel system.

4 Samples

For this report, a variety of samples were tested:

Standards, diesel fuels, home heating oil:

Sample	Type
CRMU-CPGO 8180801	Diesel Fuel Standard (Paragon)
CRM-CPGO 1162610	Diesel Fuel Standard (Paragon)
ASTM DF2 1302	Diesel Fuel No. 2 from ASTM PTP
ASTM BIOD 1104	Biodiesel B100 from ASTM PTP
HHO	Home heating oil "extra light" according to DIN 51603-1

Blend stocks and diesel-biodiesel blends:

Sample	Type
DF2	100 % Diesel fuel No. 2 (DF2) blend stock
B100	100 % Biodiesel (FAME) blend stock
B30, B20, B10, B7, B5, B2	Blends of 30 % v/v BioD with 70 % v/v DF2, 20 % v/v BioD with 80 % DF2 v/v, etc.

5 Measurement

5.1 Instrumentation

SVM 3001 Cold Properties provides viscosity measurement according to ASTM D7042 and density according to ASTM D4052. Additionally, it offers bias-corrected ASTM D445 viscosity results for this application (diesel and biodiesel at 40 °C).
For viscosity, density and cloud point determination of common diesel fuels, blends and home heating oil down to -20 °C, SVM 3001 Cold Properties does not require external counter cooling. For cell temperatures below that (arctic or polar diesel), counter cooling and drying equipment is required. For recommended low temperature equipment, accessories, installation and settings refer to the SVM x001 Reference Guide.

5.2 Instrument preparation

Before starting measurements at low temperatures, remove any humidity from the cells. Open the measuring cell lock and remove the measuring rotor. Purge the thermostat tube for a few minutes with dry air. Insert the measuring rotor immediately afterwards, purge the area where the measuring cell lock sits. Close the measuring cell lock. Now purge the measuring cells with dry air for a few minutes to remove any traces of air humidity from the cells.

Air humidity will cause ice formation in the cold cell which influences the results and the operating behavior of the measuring rotor.

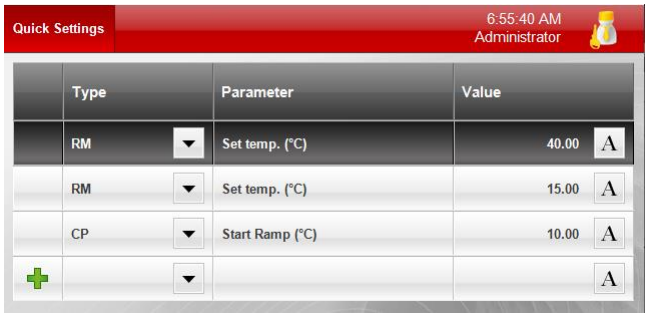
5.3 Settings SVM 3001 Cold Properties

Method settings

SVM 3001 Cold Properties – Method Settings	
Method	Diesel Fuel (CP)
Measurement Mode	CFP Cold Flow Properties
Precision Class	Precise
RDV limit	0.10 %
RDD limit	0.0002 g/cm³
Automatic prewetting	Yes
Filling check	Activated
Filling warning threshold	3

CFP Cold Flow Properties settings

The measurement mode CFP can be adapted in the Quick Settings:



Tips:

To comply with ASTM D7042, the viscosity measurements of the cold flow properties mode are repeated measurements. If this is not required, perform single point measurements at the respective measuring temperature. Refer to the SVM x001 Reference Guide.

If only the cloud point is required, just delete the above lines for viscosity and density measurement.

For low temperature viscosity testing of winter diesel, consult the application report "Viscosity measurement of diesel fuel".

For home heating oil (HHO, fuel oil No. 2, burning fuel), in some areas the kinematic viscosity is specified at different temperatures, e.g. ASTM D396 at 40 °C, ÖNorm C1109 at 20 °C.

CP start ramp temperature

Parameter: Start Ramp (temperature).
The cooling ramp for the CP determination starts from this temperature. The defined cooling rate for the ramp is 1 °C/min (1 K/min).
Recommended: 9 K above the expected CP.

Setting the Start Ramp temperature is a compromise between measuring time and the risk to get a wrong result. A Start Ramp temperature too far off the CP elongates measuring time unnecessarily. A Start Ramp temperature too close to the CP can lead to a faulty measurement.

In any case, if the measured CP is higher than the Start Ramp temperature, the measurement must be repeated with a different Start Ramp Temperature.

5.4 Calibration

The instrument shall be calibrated periodically. According to ASTM D7042, the used reference standards shall be certified by a laboratory which meets the requirements of ISO/IEC 17025 or a corresponding national standard. Viscosity standards should be traceable to master viscometer procedures. The uncertainty for density standards must not exceed 0.0001 g/cm³ for each certified value. The uncertainty should be stated (k = 2; 95 % confidence level).

Use a standard in the viscosity range of your diesel sample(s). If required, apply a calibration correction to improve the reproducibility.

Perform also periodical cloud point calibration measurements. Use either certified cloud point reference standards or at least liquids with reliably known cloud point. The cloud point and the standard type shall be in the range of your samples. If the cloud point is off, check the reference liquid, clean the cell thoroughly and retry. Only if the cloud point is repeatedly off in the same direction, apply a calibration correction.

To perform the calibration and to apply the correction, refer to the SVM x001 Reference Guide.

5.5 Sample preparation

Note: Diesel fuel and HHO contain volatile components. Avoid leaving sample containers open, as that will influence the measuring results.

Homogenize the test specimen to obtain correct and repeatable results. Warm the sample to be tested to a temperature at least 14 °C above the expected cloud point. At this temperature, remove any moisture from the sample by e.g. filtration through dry and lint free filter paper until the liquid is perfectly clear. Water droplets will lead to a wrong CP. If a (bio) diesel fuel drops below the cloud point temperature during transport or storage, ensure that all separated paraffin particles are dissolved before homogenizing and filtering the sample.

For further information on sample preparation refer to ASTM D2500 and to the SVM x001 Reference Guide

5.6 Filling

Use a glass syringe. Diesel fuel chemically attacks plastic syringes, which may influence the results.

A 5 mL glass syringe with Luer-Lock is supplied with the instrument. For repeat measurements, a 10 mL glass syringe (with Luer-Lock) is recommended. Fix an adapter Luer/UNF PEEK to the measuring cell lock to connect the glass syringe directly to the cell inlet.

Fill at least 1.5 mL as first filling. After prewetting refill at least 1 mL or until the sample in the waste hose is free of bubbles.

5.7 Cleaning

5.7.1 Solvents

- Petroleum benzine (boiling range 100/140 °C) alternatively or as second solvent for better drying:
- *n*-heptane

5.7.2 Cleaning procedure

- Open the cleaning screen (tap ) and observe it during the cleaning procedure.
- Remove the sample from the cells using an air-filled syringe.
- Fill ~2 mL of solvent using a syringe and leave it connected.
- Move the plunger of the syringe several times sharply back and forth (motor at filling speed) to improve the cleaning performance in the cells.
- Blow air through the cells for some seconds to remove the sample-solvent-mixture.
- Repeat the procedure once or twice.
- Perform a final flush with a drying solvent.
- Dry the measuring cells until the cleaning value turns green and stays steadily green.

6 Results

The results in this report are mean values from a series of repeat measurements per sample with cleaning after each completed measurement.

The results from the ASTM diesel fuel samples are compared to the data obtained from the ASTM proficiency testing program for these samples.

Where applicable, the results from the blend stocks and from the diesel-biodiesel blends are compared to the specification standards for petroleum derived diesel fuel, biodiesel and blends.

For the heating oil no reference data are available. The general specifications of DIN 51603 are used as reference instead.

6.1 Kinematic viscosity

The kinematic viscosity at 40 °C for all diesel fuels is specified in a certain range given in the respective standard. For diesel fuels used in cold climates, a maximum kinematic viscosity at -20 °C can be stated.

Diesel fuels - standards and specified viscosity:

Diesel fuel type(s) Specification standard	Specified kin. visc. 40 °C [mm²/s]
Petroleum diesel, grades No. 1-D ASTM D975	min. 1.3 max. 2.4
Petroleum diesel, grades No. 2-D, Biodiesel Blends up to B5 ASTM D975	min. 1.9 max. 4.1
Petroleum diesel, Grade No. 4-D ASTM D975	min. 5.5 max. 24
Petroleum diesel MIL-DTL-16884M (F-75/F-76)	min. 1.7 max 4.3
Biodiesel B100 (FAME) * ASTM D6751	min. 1.9 max. 6.0
Biodiesel B100 * EN 14214	min. 3.5 max. 5.0
Biodiesel blends B6 to B20 ASTM D7467	min. 1.9/1.3 max. 4.1
Biodiesel blends up to B7 EN 590	min. 2.0 max. 4.5

* The viscosity limits of the ready blend product of biodiesel with petroleum diesel must not exceed the values stated in ASTM D975.

Results:

Sample	Meas. kin. visc. [mm²/s]	Ref. kin. visc. [mm²/s]	Dev. to ref. [mm²/s]	Repeatab. r, 2 σ [%]
ASTM DF2	2.216	2.2075	0.37	0.59
ASTM BIOD	4.082	4.0142	1.68	0.24
HHO	4.502	2.5 … 6	within range	0.76

Table 1: Kinematic viscosity and precision data of ASTM diesel fuels (40 °C) and HHO (20 °C)

Sample	SVM (D7042) measured kin. visc. [mm²/s]	Repeatability * r, 2 σ [%]	Within specs.
DF No. 2 blend stock	2.499	0.09	yes
B2	2.535	0.10	yes
B5	2.589	0.15	yes
B7	2.661	0.05	yes
B10	2.680	0.08	yes
B20	2.862	0.09	yes
B30	3.069	0.09	n.a.**
B100 blend stock	4.998	0.10	yes

Table 2: Kinematic viscosity and precision data of blend stocks and diesel fuel blends

*** ASTM D7042** (SVM) states for biodiesel and biodiesel blends at 40 °C a repeatability of 0.004647 mm²/s and a reproducibility 0.009603X or (0.96 %) where X is the measured viscosity value.

ASTM D445 (glass capillary viscometers) states for kerosene, diesel fuels, biodiesel fuels, and biodiesel fuel blends at 40 °C a repeatability of 0.56 % and a reproducibility of 2.24 %.

** For the biodiesel blend B30 no specified viscosity range was available.

All tested samples provided results within the specs and within the limit for repeatability specified by both, ASTM D7042 and ASTM D445.

6.2 Cloud Point (CP)

The CP results obtained from the standards are compared to the reference data of the cloud point standards according to their certificates (precision data limits according to ASTM D2500, as this standard is applicable to all samples).

Sample	Test methods employed
CRMU-CPGO	IP 219, ASTM D2500, ISO 3015
CRM-CPGO	ASTM D2500, EN 23015, ISO 3015, ASTM D5771, ASTM D5772, ASTM D5773
ASTM DF2	ASTM D2500
ASTM BIOD	ASTM D2500
HHO-EL	EN 23015

Table 3: Cloud point – applicable test methods

Sample	Determined Cloud Point SVM [°C]	Ref. CP [°C]	Dev. to ref. [°C]	Repeatab.* r, 2 σ [°C]
CRMU-CPGO	-7.23	-7.6	-0.37	0.41
CRM-CPGO	-2.33	-2.4	-0.07	0.16
ASTM DF2	-13.0	-12.5	0.50	0.67
ASTM BIOD	-0.71	-0.5	-0.21	0.08
HHO-EL	-4.07	max. +3	fulfilled	0.06

Table 4: Cloud point and precision data of CP standards, ASTM diesel and HHO

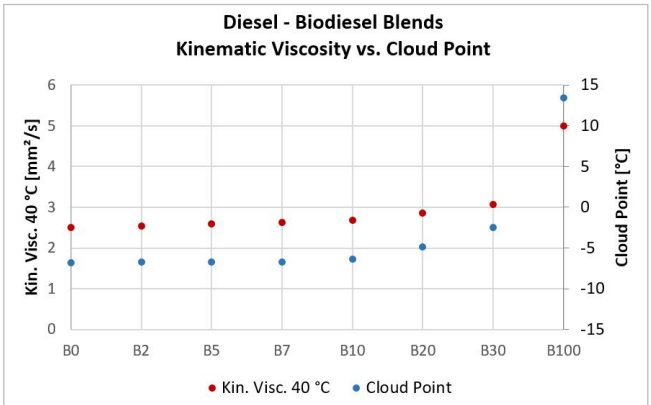
According to ASTM D975, D7467 and D6751, the CP needs to be reported only. EN 590 specifies a maximum CP for diesel fuel and blends up to B7, class 0 to class 4, used in areas of arctic or rough winter climates.

Sample	Determined Cloud Point SVM 3001 Cold Prop. [°C]	Repeatability* r, 2 σ [°C]
DF No. 2 blend stock	-6.80	0.07
B2	-6.73	0.03
B5	-6.72	0.11
B7	-6.68	0.05
B10	-6.38	0.06
B20	-4.88	0.02
B30	-2.47	0.01
B100 blend stock	13.47	0.34

Table 5: Cloud point and precision data of blend stocks and diesel blends

*** ASTM D2500** specifies a repeatability of the CP for biodiesel and biodiesel blends of 2 °C, a reproducibility of 4 °C for petroleum derived diesel and a reproducibility for biodiesel and blends of 3 °C.

The graph shows the change of viscosity and cloud point with rising biodiesel content.



6.3 Density

EN 590 (diesel and blends with biodiesel up to B7) and EN 14214 (B100) specify density ranges at 15 °C. ASTM D6751 (B100) states that biodiesel, which meets the specifications of this standard, is in the range of 0.86 g/cm³ to 0.90 g/cm³, where the typical values are around 0.88 g/cm³ to 0.89 g/cm³.
ASTM D7467 (B6 to B20) does not specify density.
For HHO-EL, ISO 51757 specifies a maximum of 0.86 g/cm³.

Sample	Meas. density [g/cm³]	Ref. density [g/cm³]	Dev. to ref. [g/cm³]	Repeatability r, 2 σ [g/cm³]
ASTM DF2	0.82566	0.8256	0.00006	0.00013
ASTM BIOD	0.88475	0.8839	0.00085	0.00006
HHO-EL	0.83234	max. 0.86	fulfilled	0.00004

Table 6: Density and precision data from ASTM diesel and HHO

Sample	SVM (D4052) measured density. [kg/m³]	Repeatab. *** r, 2 σ [kg/m³]	Within specs.
DF No. 2 *	829.0	0.04	yes
B2 *	830.0	0.08	yes
B5 *	831.6	0.06	yes
B7 *	832.5	0.08	yes
B10	833.9	0.02	n.a.****
B20	838.6	0.02	n.a.****
B30	843.4	0.03	n.a.****
B100 **	877.7	0.04	yes

Table 7: Density and precision data from blend stocks and blends

*** EN 590** specifies 820 to 845 kg/m³ at 15 °C for these products.
** **EN 14214** specifies 860 to 900 kg/m³ at 15 °C for B100.
*** **ASTM D4052** states a repeatability of 0.00011 g/cm³ (average of 2 determinations) for distillates in the range of 0.80 to 0.88 g/cm³
**** No specified density value was available.

7 Conclusion

SVM 3001 Cold Properties is perfectly suited for determining kinematic viscosity, density and the cloud point of diesel fuels, biodiesel and blends hereof as well as of home heating oil provided all requirements described in this report are fulfilled.

Figure 1: SVM 3001 Cold Properties

8 Literature

Related standards – refer to current versions

ASTM D975, ASTM D2500,
EN 590, ASTM D7467, ASTM D6751,
EN 14214, EN 16734, EN 16709, ASTM D7042,
ASTM D445, ASTM D4052, ASTM D2500
ÖNorm C1109, ISO 51757, ASTM D396, DIN 51603

Related documents

Application report "Viscosity measurement of diesel fuel".

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