

### Performance Evaluation of the Ignition Quality Testers Equipped with TALM Precision Package (TALM-IQT<sup>TM</sup>) Participating in the ASTM NEG Cetane Number Fuel Exchange Program

2017-01-0720 Published 03/28/2017

Omar Ramadan, Luc Menard, David Gardiner, Aaron Wilcox, and Gary Webster Advanced Engine Technology Ltd.

**CITATION:** Ramadan, O., Menard, L., Gardiner, D., Wilcox, A. et al., "Performance Evaluation of the Ignition Quality Testers Equipped with TALM Precision Package (TALM-IQT™) Participating in the ASTM NEG Cetane Number Fuel Exchange Program," SAE Technical Paper 2017-01-0720, 2017, doi:10.4271/2017-01-0720.

Copyright © 2017 SAE International

### **Abstract**

This paper is a continuation of work previously discussed in SAE 2014-01-0179 [1] and SAE 2015-01-0805 [2], which was intended to improve the capability and precision of the Ignition Quality Tester (IQT<sup>TM</sup>) and associated ASTM D6890 [3]/CEN EN 15195 [4]/EI IP 498 [5] Test Methods. The results presented in those two papers indicated how the new generation of IQT<sup>TM</sup> with the TALM Precision Package upgrade can markedly improve the precision of the ASTM D6890, CEN EN 15195 and EI IP 498 Derived Cetane Number (DCN) test methods.

This paper will evaluate the performance of the upgraded instruments over the past 21 months of their participation in ASTM's National Fuel Exchange Group (NEG) diesel fuel exchange program. It will also present a comparison of the published precision of the ASTM Cetane Number (CN) and Derived Cetane Number (DCN) standard test methods that participated meaningfully in the ASTM NEG diesel fuel exchange program (ASTM D613 [6]/CEN EN ISO 5165 [7]/EI IP 41 [8] and ASTM D6890 [3]). In addition, it will present and discuss a comparison between the precision of these ASTM standard methods, the mini Inter-Laboratory Study presented in SAE 2015-01-0805 [2] and the recent test results from the ASTM NEG fuel exchange program (real world data).

The real world results extracted from the NEG fuel exchange program's monthly reports strongly support the findings of the two previous SAE papers. This paper shows that the D6890/EN 15195/IP 498 instruments equipped with the TALM Precision Package have clearly demonstrated a marked improvement in precision of the ASTM D6890 and CEN EN 15195 Test Methods.

### Introduction

Ignition quality is one of the more important fuel characteristics that need to be measured for the quality control of diesel (compression ignition) fuels. In diesel engines, the ignition quality of the fuel

determines the time delay between the initial injection of fuel and the beginning of combustion, known as the ignition delay (ID) time. The cetane number (CN) is the diesel fuel quality parameter related to the ignition delay (ID) time and combustion quality of a fuel obtained by comparing it to reference fuels in a standardized engine test. A fuel with a higher CN will have a shorter ignition delay period, thus reducing the amount of fuel that will be injected and pre-mixed with air before combustion begins. This affects emission formation, thermal efficiency, and injection timing requirements.

There are number of standard test methods that have been established over the years at ASTM International (ASTM), and other organizations worldwide, to measure and estimate the CN of fuels. These standards involve engines such as Cooperative Fuels Research (CFR<sup>TM</sup>) engine (cetane engine), and the Constant Volume Combustion Chamber (CVCC) instruments such as the Ignition Quality Tester (IQT<sup>TM</sup>), which are both used specifically for testing purposes to measure the CN or the DCN. In addition to those standard test methods there is an alternative (calculated) method used as supplementary tools for estimating CN, the Cetane Index (CI). The CI can be used for CN estimation when the results by the cetane engine or the CVCC instruments are not available, and if cetane improver is not used and if the bio diesel content is sufficiently low. <u>Table 1</u> lists the ASTM cetane rating standard test methods. The CN applicable ranges stated in the table were extracted from the precision statement of each ASTM method [3, 6, 9, 10, 11 and 12]

Table 1. ASTM cetane rating Standards and Applicable Ranges

ASTM Standard	CN applicable range	Range (max min.)	Instrument
D6890 (DCN) [3]	33-64	31	IQT (CVCC)
	64-100*	36	iQ1 (CVCC)
D613 (CN) [6]	40-56	16	CFR Engine
D7170 (DCN) [9]	39.5-55.2	15.7	FIT (CVCC)
D7668 (DCN) [10]	39.4-66.8	27.4	CID 510 (CVCC)
D976 (CI) [11]	30-60	30	Correlation
D4737 (CCI) [12]	32.5-56.5	24	Correlation

# The Cooperative Fuels Research (CFR<sup>TM</sup>) cetane Engine

The CFR<sup>TM</sup> cetane engine is the oldest (since 1930s) standard test method (ASTM D613 [6]/CEN EN ISO 5165 [7]/EI IP 41 [8]) used for measuring the CN of a diesel fuel. ASTM D613 is essentially the same as the EI IP 41 (Energy Institute (UK) Standard) and EN ISO 5165 (European Standard) test methods and from now on the term ASTM D613 or simply D613 will represent all the above stated CN standards. In D613 test method, the CN is determined by comparing the ID of a fuel in a standard single cylinder, variable compression ratio diesel engine with the IDs of blends of reference fuels of known cetane number. The compression ratio is varied by adjusting a calibrated hand wheel to obtain the same ID for the sample and for each of two bracketing reference fuels, which permits interpolation of cetane number in terms of the hand wheel readings [6]. The cetane number scale in ASTM D613 presently covers the range from 15 CN to 100 CN, but, as stated in ASTM D613, the fuels tested typically cover the range from 30 to 65 CN [6]. However, the precision statement in the ASTM standard D613, which was based on real world data, only covers the CN range from 40 CN to 56 CN [6]. The term "real world data" used in this paper, means the test data extracted from both the ASTM, and the Energy Institute (EI) Fuel Exchange Programs (FEPs). The fuel exchange program participants are less likely to unreasonably tighten the control of critical parameters relative to an Inter-Laboratory Study (ILS), and so the results tend to be more representative of how the participating instruments actually perform in normal usage from a global perspective.

The ASTM D613 test method is currently the CN referee method in the ASTM diesel fuel specification (i.e.: the D613 CN is assumed to be correct in the case of a disputed test result). However, the use of this method has serious drawbacks including high capital cost, high operator skill requirements, and relatively poor reproducibility between labs, which limits the precision with which CN measurements can be made. In ASTM D613-15a the precision limits (e. g. repeatability (r) and reproducibility (R)) are only stated for the range of 40 to 56 CN [6]. Above this range (paraffinic diesel fuels) D613 is less precise [13] than the other combustion based test methods. A comparison between the precision of the D613, D6890 and EN 15195 CN/DCN test methods will be briefly discussed in a later section of this paper.

### The CVCC Instruments

In CVCC instruments, such as the D6890 instrument, the fuel is injected into a heated CVCC. The chamber contains heated air with a controlled pressure and temperature. After injection, the chamber pressure initially decreases. The decrease in pressure is due to evaporative cooling of the injected fuel, which, along with fuel/air mixing, is a part of the physical delay process. Once sustained combustion begins to occur, the pressure in the combustion chamber increases very rapidly. The ID, the time between the start of injection until the start of combustion, is measured and an average of 32 injection/combustion cycles is calculated. The Derived Cetane Number (DCN) is then calculated from the average ID, measured in milliseconds, using a conversion equation [3].

<u>Table 1</u> lists the other ASTM standards that use CVCC instruments such as the Fuel Ignition Tester (FIT) (ASTM D7170 [9]) and the Cetane Ignition Delay CID 510 (ASTM D7668 [10]). Both the D7170

and D7668 methods use similar principles to ASTM D6890, with some differences in the injection system, operating conditions, and the number of injection/combustion cycles used to calculate the average (32 cycles for D6890, versus 25 cycles for D7170 and 15 cycles for D7668). Among the three CVCC ASTM standard methods, D6890 has the widest applicable DCN range, and its precision statement is based on real world data and not solely on ILS data, like the D7170 and D7668 precision statements. For more information regarding the differences between these methods refer to ASTM standards D6890, D71770 and D7668 [3, 9 & 10].

#### Cetane Number Alternative Methods

When the results by the cetane engine or the CVCC instruments are not available, and the test sample does not contain cetane improver, cetane indices can be used to estimate the CN value of a sample [8]. In ASTM, there are two standard test methods used for estimating the cetane index of diesel fuel, ASTM D976 [11] and ASTM D4737 [12]. Both standards are based on correlation of CN with selected bulk physical properties of the tested sample. The ASTM D976 Cetane Index (CI), correlation relies on two properties, and the ASTM D4737 Calculated Cetane Index (CCI) correlation uses four properties. For more information regarding the different correlations and properties used, refer to the cetane index standard methods ASTM D976 [11] and ASTM D4737 [12]. The expected error of prediction for both ASTM D976 and D4737 will be less than  $\pm 2$  CN when used through the method applicable range shown in Table 1 [11, 12].

### Diesel Fuel Real World Data

ASTM International and the EI in Europe both operate monthly Fuel Exchange Programs. In these proficiency test programs, the uniform fuel samples are distributed among participating laboratories, and a monthly statistical report of the results is circulated to participants. These test programs work as statistical quality control tools, which enable the participating organizations to assess their performance in conducting testing according to ASTM or EI standard test methods within their own laboratories. As one of the CN rating test methods, the ASTM D6890 instrument started participating in the ASTM NEG FEP in January 2003, and in the EI FEP in January 2004 under the standard test methods EN 15195 [4] and IP 498 [5]. During this period (2003-2016) a variety of fuel samples, with different chemical compositions, were tested successfully using the D6890/EN 15195 instrument. The fuel samples tested in these FEPs contain all the different diesel fuel grades (with and without cetane improver), biodiesel, jet fuels, gas to liquids fuel (GTL), and other renewable fuels. All the test results used in this paper were extracted from the ASTM NEG FEP monthly reports.

### Diesel Fuel Specification

Diesel fuel refers to any liquid fuel for a compression ignition engine. At ASTM and CEN, diesel fuel quality is specified in the ASTM D975 [14] and CEN EN 590 [15] standards respectively. These specification standards describe a limited number of properties that diesel fuels must meet or exceed for compliance with the standard. The standard only defines some of the property values needed to provide acceptable engine operation, safe storage and transportation of diesel fuel. As the main topic of this paper is related to the use of some of the ASTM NEG fuel sample's test results, the ASTM D975 specification will be highlighted more than that of the EN 590

specification. According to ASTM standard D975, there are different diesel fuel grades such as Grade No. 1-D, Grade No. 2-D and Grade No. 4-D. The grades are numbered in order of increasing density and viscosity, with No. 1-D the lightest and No. 4-D the heaviest [14]. Table 2 contains some of the requirements for diesel fuel oils. These requirements have been extracted from the ASTM D975 and EN590 standards. Table 2 also lists some other properties, such as API Gravity or density, which is not specified in ASTM D975. The API range stated in the table was extracted from other sources such as the Goodheart-Willcox Company [16] or the Engine Manufacturers Association (EMA) [17].

### ASTM D6890 (CEN EN 15195

The automated diesel fuel Ignition Quality Tester (IQTTM), developed by Advanced Engine Technology Ltd. (AET), allows measurements of the ignition delay and hence, the ignition quality of compression ignited fuel samples. Since the introduction of the ASTM D6890 instrument, approximately 190 units have been commissioned worldwide in petroleum refineries, regulatory bodies, test laboratories, combustion research centers, and universities. The importance of the D6890 instrument in the refinery and research community continues to grow. It has been shown that the D6890 instrument can determine the auto-ignition characteristics of high cetane fuel types (such as biomass derived fuels and paraffinic middle distillate, renewable fuels etc.) more accurately than is possible using the ASTM D613 instrument.

According to the ASTM D6890 test method the chamber temperature, pressure oxygen content and the amount of mass injected must be held within relatively narrow limits when fuel samples are tested. The potential to control these variables beyond the limits of the test method is provided to users who employ the D6890 instrument in research applications. D6890 instruments equipped with the Flexible Research variant of the instrument's software have been used extensively for evaluating kinetic models. This software permits wide variations in chamber temperature and pressure. Research-grade fuel injection systems, equipped with a variable displacement fuel injection pump, allow the operator to quickly and easily change the mass of fuel injected into the combustion chamber while the test is running. The controllable variables within the D6890 instrument, in its research configuration, makes the system capable of experimentally validating the combustion kinetic models of many novel fuel formulations [18].

Table 2. Requirements for Diesel Fuel Oils (ASTM D975/EN 590/others)

Property	Specification	No. 1-D (S15,	No. 2-D (S15,	No. 4-D
		S500 & S5000)1	S500 & S5000)	
Flash Point, °C , min	ASTM D975	38	52	55
	EN 590		55	
Distillation Temperature, °C	ASTM D975			
90%, % vol. recovered	min	_	282	_
	max	288	338	-
Kinematic Viscosity, mm2/sec	ASTM D975			
at 40°C	min	1.3	1.9	5.5
	max	2.4	4.1	24
	EN 590		2.0 to 4.5	
Sulfur Content, mg/kg, max	ASTM D975	15	15	_
	EN 590		10	
Cetane Number, min	ASTM D975	40	40	30
	EN 590		51	
Cetane Index, min	ASTM D975	40	40	_
	EN 590		51	
Gravity API at 60 °F	G-W[16]/EMA[17]			
	min [16]	40	33	-
	max [16] ([17])	44 (43)	37 (39)	_
Density at 15 °C, kg/m3	EN 590	1 /	820 to 845	

**Note:** the ASTM D6890 instrument is the same as the IP 498 (Energy Institute (UK) Standard) instrument or EN 15195 (European Standard) instrument.

Relative to the D613 engine, the D6890/EN 15195 instrument has very good CN determination capability (accuracy) in addition to the best precision. This has been demonstrated by over a decade of experience with the ASTM and EI FEPs involving the D6890 instruments and cetane engines, in which over 315 different fuel samples have been tested thus far. In recognition of the escalating need for even higher precision in real-world ignition quality measurements, there have been ongoing efforts to enhance the precision of the D6890 instruments. This has produced an evolution from the initial series of semi-automated instruments, which were used to produce the precision statements in each of the applicable test methods, to a new generation of instruments that employ additional levels of automation (along with other improvements) to attain even greater levels of precision.

### The Upgraded Version of the D6890 (EN 15195) Apparatus

The latest generation of the ASTM D6890 instrument (and upgrade kits for existing instruments) employs additional levels of automation to provide further improvements to its precision. This upgraded generation is known as the Totally Automated Laboratory Model (TALM-IQT<sup>TM</sup>) configuration. Figure 1 illustrates the upgraded version of the ASTM D6890, EN 15195 and IP 498 methods apparatus. The instrument shown, in Figure 1, is equipped with the Totally Automated Laboratory Model Precision Package and the TALM Electronic Pressure Regulator Panel (EPRP). The Totally Automated Laboratory Model Precision Package includes the control system cabinet (TALM-K1), Totally Automated Laboratory Model System Control Software package (TALM-K2), nozzle tip temperature controller (TALM-K3) and the combustion chamber pressure sensor temperature controller (TALM-K4). The Totally Automated Laboratory Model EPRP (TALM-K7) provides automated control of the pressure of the charge air supply, fuel reservoir nitrogen supply, and injection pump actuator air supply. For the full details regarding the benefits of the Totally Automated Laboratory Model Precision Package and how those benefits can be maximized to get the most precise test results, refer to AET previous publications [1, 2]and 19].

**Note:** From now on, the term "D6890 instrument" will replace the conventional Laboratory Model (LM-IQT<sup>TM</sup>) and the term "upgraded D6890 instrument" will replace the Totally Automated Laboratory Model (TALM-IQT<sup>TM</sup>) configuration.

The upgraded D6890 instruments have demonstrated a great improvement in precision in the mini Inter-Laboratory Study (mILS) conducted at AET in 2015 [2]. The mILS was carried out using four different upgraded D6890 instruments, and nine fuel samples from the ASTM NEG and EI FEPs, covering the DCN range from 34 DCN to 81 DCN. The upgraded D6890 instruments achieved a very low average standard deviation of 0.27 DCN for the tested samples, compared with 0.62 DCN for conventional D6890 instruments and 1.33 CN for D613 instruments [2]. Based upon the mILS results, the

upgraded D6890 instruments have demonstrated better reproducibility than all existing ASTM standard test methods used for measuring diesel fuel ignition quality [2].

In addition to the mILS results, the upgraded D6890 instruments have demonstrated improved precision (relative to the conventional D6890 instruments) in the ASTM NEG fuel exchange program, with monthly standard deviation results as low as 0.15 DCN [19]. Starting from January 2015, the majority of the results to date for upgraded D6890 instruments participating in ASTM NEG fuel exchange program are consistent with the reproducibility demonstrated in the mILS [19, 20]. In the following subsection, an overview of the D6890/EN 15195 predictive capability using the NEG/EI real world data is presented. Both upgraded D6890 and conventional D6890/EN 15195 instruments were used in those tests.

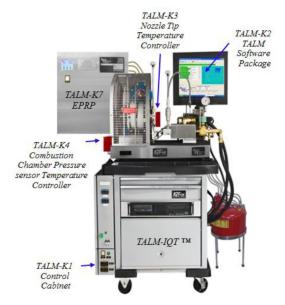


Figure 1. The Upgraded D6890/EN15195/IP498 instrument configuration (TALM-IOT<sup>TM</sup>)

# ASTM Standard D6890/EN15195/IP498 Predictive Capability (CN versus DCN

Over 315 fuel monthly samples have been tested since the D6890/EN 15195 instruments started participating in the ASTM NEG and EI FEPs. Since 2003, 164 fuel samples were tested in the ASTM NEG FEP, and starting from 2004, 151 fuel samples were tested in the EI FEP. To get an insight into the predictive capabilities of the D6890 instrument, the test results of the ASTM NEG FEP for the different participant laboratories performed since 2003 are plotted in Figure 2. Because of the ASTM standard D613/EN ISO 5165 is the referee cetane method, Figure 2 compares the average DCN (D6890) and the average CN (D613) values reported in the NEG Fuel exchange program for 164 samples. The NEG test program results have shown good comparative results between both methods over a wide CN/DCN range (33 to 80).

<u>Figure 2</u> shows that 5 of the 164 samples (3%) tested have some disagreement between D6890 and the referee method (D613). The difference between the CN and DCN values for those 5 samples are as follows:

- 1. Sample D1026: ΔCN=4.9
- 2. Sample D1078: ΔCN=2.4
- 3. Sample D1090:  $\Delta$ CN = 6.8 (the highest since 2003)
- 4. Sample D1095:  $\Delta$ CN = 5
- 5. Sample D1109:  $\Delta$ CN=5

All 5 of those ASTM NEG samples have API gravities or viscosities that are outside of the limits of the diesel fuel specifications presented in Table 2. Physical properties such as API gravity (density) and viscosity affect the fuel injection characteristics. In addition to that, there are substantial differences between the fuel injection systems of the D6890 instrument and the D613 cetane engine. These include much tighter barrel/plunger clearances in the D6890 fuel injection pump, and a higher nozzle opening pressure (2600 psi D6890 versus 1500 psi D613). Thus, the differing impact of a fuel's physical properties on fuel spray characteristics (Spray angle, width, length, velocity and droplet size) would be expected to affect the relationship between the CN of the D613 engine and the DCN (which is based on ignition delay) of the D6890 instrument. Regarding both methods' applicable ranges, 4 samples out of the 5 have average CN values that are outside of the 40 to 56 CN precision range of the referee test method (D613). In contrast, D6890 has a wider applicable range of 33 to 64 DCN; however, D6890's European counterpart, EN 15195, has an applicable DCN range from 35 to 70 DCN. The question one should ask is, which of the available ASTM cetane rating standards should be used as referee method at lower (CN<40) and higher (CN>56) CN scale? This topic will be further discussed in the following two subsections.

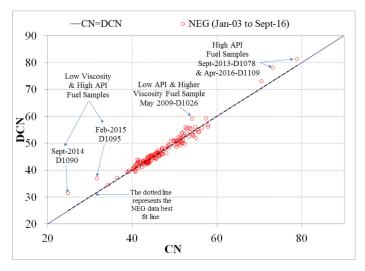


Figure 2. D6890 determination capabilities using ASTM NEG FEP tested fuel samples (January 2003 – September 2016)

# Comparisons between the D6890/EN 15195/IP 498 Precision and CN (D613) Scale Method

Experimental results of any inter-laboratory study contain variability that arises within a given laboratory and variability that arises between laboratories. The terms repeatability (r) and reproducibility (R) are used to differentiate between different measurement variability [21]. Repeatability characterizes the ability of an individual laboratory to repeat measurements (Conditions: single operator, short time between tests, specific test apparatus and a random sample taken from a quantity of homogeneous material). On the other hand reproducibility characterizes the ability of two

independent laboratories to reproduce each other's test results (Conditions: different laboratories, applying same test method and random sample taken from a quantity of homogeneous material) [21].

ASTM E691 is the standard practice for conducting an interlaboratory study to determine the precision of test method [21]. This method provides an estimate of the standard deviation of the repeatability (Sr) and reproducibility (SR). These standard deviations were then used to calculate the 95% repeatability (r) and 95% reproducibility (R) statistics. Table 3 lists the formulas used in ASTM E691 for the estimation of these precision terms. This method will be used in a later section to estimate the repeatability and the reproducibility of the upgraded D6890 instruments that participated in the ASTM NEG FEP. This section presents a comparison and an update to the reproducibility (R) of both the DCN D6890/EN15195 and the CN D613 standards that have participated in the NEG FEP.

Table 3. Formulas used in the estimation of the upgraded D6890 instrument precision terms (r) & (R) [21].

$$r = 95\%$$
 repeatability statistic =  $2.77 * S_r$  (1)

$$R = 95\% reproducibility statistic = 2.77 * S_R$$
 (2)

$$S_r = repeatability \, STDEV = \sqrt{\frac{1}{p} \sum_{i=1}^p S^2} \tag{3}$$

$$S_R = reproducibility \ STDEV = \sqrt{S_{\bar{x}}^2 + S_r^2 \left(\frac{n-1}{n}\right)}$$
 (4)

$$s = cell \ STDEV = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x - \bar{x})^2}$$
 (5)

$$S_{\vec{x}} = \text{STDEV of cell averages} = \sqrt{\frac{1}{p-1} \sum_{i=1}^{p} (x - \bar{X})^2}$$
 (6)

$$\bar{x} = cell \ average = \frac{1}{n} \sum_{i=1}^{n} x$$
 (7)

$$\bar{X} = average \ of \ cell \ averages = \frac{1}{p} \sum_{i=1}^{p} \bar{x}$$
 (8)

Where, n is the number of test results per cell, p is the number of laboratories and x is an individual test results. The precision multiplier shown in equations (1) and (2) is calculated from the  $(t\sqrt{2}=1.96\sqrt{2}=2.77)$ , where (t) is the two tailed Student's t for 95% probability.

Figure 3 presents a comparison of the reproducibility of CN (D613) and DCN (D6890 and EN15195) test methods, and the estimated reproducibility of the upgraded D6890 instruments. The data sets used in Figure 3 were calculated from the precision statements in the most recently published versions of each test method [2, 4 & 6]. The estimated precision of the upgraded D6890 instrument, represented by the dotted line, was based on earlier mini Inter-Laboratory Study (mILS) [2]. The data of ASTM D613-15a, ASTM D6890-15a and CEN EN 15195:2014 used in the published precision calculations is based on real world data from FEPs.

Of the CVCC instruments, the D6890/EN 15195 instrument has the most extensive FEP data set, having participated in these programs since 2003/2004. These FEPs have been used to successively update the precision of D6890 and EN 15195. The published precision for other CVCC instruments (ASTM D7170-14 and ASTM D7668-14) was based only on initial ILS data, since their FEP participation has been limited. The average number of both D7170 and D7668

instruments that participated in the NEG FEP during 2016 (9 months) was two instruments. Therefore, only the ASTM methods that participated meaningfully in the ASTM NEG FEP were used in Figure 3.

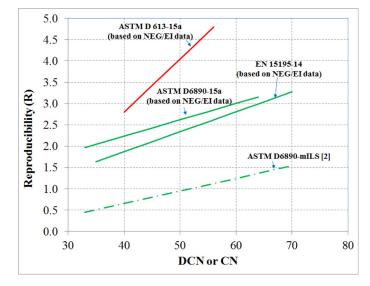


Figure 3. Comparison of the reproducibility of DCN (D6890/EN15195) and CN (D613) test methods, combined with the estimated precision of the upgraded D6890 instruments [Updated from [2]]

For the test methods whose reproducibility is based on FEP data (ASTM D613-15a, ASTM D6890-15a, and CEN EN 15195:2014), Figure 3 shows that CEN EN15195:2014 test method has the best reproducibility, when compared with other established methods over a wide CN/DCN range (34 to 71 DCN).

For the D6890/EN 15195 instrument, the preliminary estimates of combined results from the ASTM and EI study indicated an average improvement of approximately 13% in reproducibility, compared to CEN EN 15195:2014 [2]. The comparison was made based on the results that cover the range of 33 to 64 DCN. However, the applicable range of the EN 15195 test method is from 33 to 70 DCN. Over the larger DCN range, the upgraded D6890 instruments mILS showed, on average, an improvement of approximately 62% in reproducibility relative to the updated standard CEN EN 15195:2014 [2]. The reproducibility of the upgraded D6890 instruments was also better than any of the published reproducibility values for CN or DCN test methods [2].

In the ASTM D613-15a updates shown in Figure 3, the precision limits for reproducibility (R) are for the range of 40 to 56 CN. Above this cetane range D613 is less precise. If the 95% confidence level reproducibility stated in ASTM D613-15a is extrapolated to 75 to 90 CN, reproducibility limits will be  $\pm 7.2$  to  $\pm 9.1$  CN respectively. Due to this limitation the D613 test method, for higher CN fuels such as paraffinic diesel samples (e.g. Gas to Liquid fuels), the CN cannot be precisely determined by a single laboratory [13]. Both the D6890 and EN 15195 test methods provide better precision at higher CN/DCN values than the referee method (D613), which is only applicable over the range of 40 to 56 CN. The improved precision of the CEN EN 15195:2014 test method has led to EN 15195 being allocated as the referee method in the new European standard for high cetane paraffinic diesel fuels (EN 15940 [22]).

### ASTM D6890 DCN and ASTM D613 CN Connection

In ASTM D6890/EN15195/IP498, the DCN conversion equation is based on the correlation between ignition delay measurements and Accepted Reference Values (ARVs) of Cetane Number (CN) for diesel fuel samples. Such ARVs are the mean values that were obtained when each sample was tested by a relatively large number of cetane engines (typically over 30). Averaging so many engine results overcomes the poor reproducibility between individual engines, resulting in 95% confidence intervals on the order of  $\pm 0.5$  CN for the mean values. This reliable average CN data has made possible the development and ongoing verification of an accurate Ignition Delay to DCN equation for the D6890 instrument. In contrast, the CN results from an individual cetane engine can deviate from the mean value or ARV by (for example)  $\pm 4$  at 50 DCN (see Figure 3), to  $\pm 8$  at 75 DCN (see Figure 4). Nevertheless, in cases where D613 has been defined as the referee method, the results from a single cetane engine could be used to over-rule the results of another test method (even a method with markedly superior reproducibility) in the event of a dispute. Thus, granting referee status to a single cetane engine does not seem appropriate.

# Ignition Quality Tester and the CEN Standard EN 15940 for Paraffinic Diesel Fuels

Recently, the European Committee for Standardization (CEN¹) approved EN 15940:2016 [22], a new standard for paraffinic diesel fuels. These materials are liquid fuels that can be synthetically made from feedstocks such as natural gas (Gas-to-Liquid (GTL)), biomass (Biomass-to-Liquid (BTL)), coal (Coal-to-Liquid (CTL) or from hydro-treating vegetable oil (HVO) or animal oils. These fuels are also referred to as Renewable Diesel Fuels (RDFs). RDFs do not fully meet the ASTM D975 or the EN590 specification stated in Table 2. These fuels have slightly lower density, higher energy content and higher cetane number. These high quality RDFs combust cleaner (less oxides of nitrogen and particulate matter emissions) than conventional crude-oil based diesel fuels [13].

According to the CEN EN 15940 standard, paraffinic diesel fuel has two classes; Class A & Class B. Class A has a minimum CN of 70, a density range from 765 to 800 kg/m³ and a viscosity range from 2 to 4.5 cSt. However Class B has a minimum CN of 51, a density range from 780 to 810 kg/m³ and the same viscosity range as Class A. CEN EN 15940 does not specify an upper CN limit for paraffinic diesel fuels. The CN number of most paraffinic diesel fuels is very high and it varies from 70 to 95 [24]. Aatola et al. 2008 [23] stated that the CN range for GTL fuels is from 73 to 81 CN and HVO fuels are in the range from 80 to 99.

As described in EN 15940, the cetane number for these fuels can be measured with two methods; EN ISO 5165 (D613,CFR engine) and by CEN EN 15195 (Ignition Quality Tester). When determining precision data for CN/DCN, it was noted that DCN measured by CEN EN 15195 is the more precise method for paraffinic diesels [22]. The precision (R) statements for the different CN/DCN referee methods stated in CEN EN 15940 are plotted in Figure 4. The plot shows the reproducibility of both test methods at high CN/DCN values.

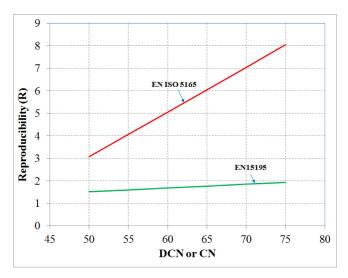


Figure 4. Precision comparison for the two referee methods used in the CEN Standard EN 15940:2016

### **ASTM NEG FEP Test Results**

As an update to the previous work [1, 2, 19 and 20] this paper will analyze the performance of upgraded D6890 instruments in the ASTM NEG FEP for the period of time from January 2015 to September 2016. In this period, 21 different fuel samples were tested. It is only in this period that the ASTM NEG FEP distinguished between upgraded D6890 instruments, and standard ASTM D6890 instruments, in the monthly test reports. In this period of time, 3 to 5 of the new generation of ASTM D6890/CEN EN 15195 instruments participated in the NEG FEP. It is anticipated that there should be more than 7 units next year.

This analysis will demonstrate how the upgraded D6890 instrument can improve the precision and the accuracy, relative to the conventional ASTM D6890 instruments. In this analysis, the ASTM NEG FEP fuel samples (FS) tested will be listed with some of their properties extracted from the ASTM NEG FEP reports. Then the effect of using the upgraded D6890 instrument on precision and accuracy (STDEV, CN versus DCN, repeatability, reproducibility and accuracy) will be covered.

# Fuel Samples Used in the Evaluation of the Upgraded D6890 Instrument and Their Properties

The CN of a fuel is a parameter based on the measured ignition delay period in which the physical and chemical properties of fuel play an important role. In this section, the fuel samples will be defined and some of the main properties extracted from the ASTM NEG FEP report will be presented. These properties will give the reader general information about the real world fuel samples tested in this program. These properties also will be used to examine compliance with the ASTM D975 diesel fuel requirements.

<u>Table 4</u> lists the NEG fuel samples used in this evaluation and their main average measured properties. These properties are the average flash point, average API gravity, average kinematic viscosity, average

<sup>1.</sup> Comité Européen de Normalisation

sulfur content, average CN (ASTM D613), average DCN (ASTM D6890), average CI (ASTM D976), and average CCI (ASTM D4737)). The CI and CCI values listed in <u>Table 4</u> were calculated from the other fuel properties, using the correlation equations from ASTM D976 and ASTM D4737 [11, 12].

Using the diesel fuel requirements presented in <u>Table 2</u> and comparing it with the average properties presented in <u>Table 4</u>, one can conclude that for fuel sample #2 (D1095) and sample #16 (D1109):

- 1. Both fuel samples had API Gravity values that were outside of the limits of the diesel fuel specifications presented in <u>Table 2</u>.
- Both fuel samples had ASTM D613 CN values that were outside of the applicable range of 40 to 56 CN for the test method's precision statement.
- 3. Fuel sample #2 had a viscosity that was outside of the minimum limit for ASTM D975 1-D and 2-D diesel fuels.
- Fuel sample #2 had a sulfur content that was outside of the specifications for the S15 (ULSD) grade of ASTM D975 1-D and 2-D diesel fuels.

The CI and CCI did not predict the CN value for sample #2 (D1095). The difference between the CN and the CCI for this sample was approximately 20, and the difference between CN and DCN was 5. Fuel sample #2 (D1095), #7 (D1100), and #11 (D1104) all had CN, CI, or CCI values that were below the minimum diesel fuel requirements shown in Table 2.

The expected error of prediction for both ASTM D976 and ASTM D4737 is less than  $\pm 2$  CN, when used within the method's applicable precision range. Table 4 shows that 4 out of the 21 test samples had CCI errors of prediction larger than  $\pm 2$  CN:

- 1. Sample #2 (D1095): ΔCN=19
- 2. Sample #15 (D1108):  $\Delta$ CN=3
- 3. Sample #16 (D1109):  $\Delta$ CN=4.2
- 4. Sample #18 (D1111):  $\Delta$ CN=3

For fuel sample #16 (D1109), which had the highest CN/DCN/CCI in this set of samples, some agreement among the DCN and CI methods was observed, while the CN showed the largest discrepancy. The same observation for this sample was recorded between the CN and the other CVCC methods, such as ASTM D7170 (DCN= 79.8) and ASTM D7668 (DCN=81.2).

Of the 21 fuel samples tested, fuel samples #2 (D1095) and #16 (D1109) have extremely different properties from the ASTM D975 diesel fuel specifications in <u>Table 2</u>. The property limits discussed in this section demonstrate that these samples are outside of the scope of the ASTM D6890/CEN EN 15195, which may affect the accuracy and precision of the method.

Table 4. NEG fuel samples used in the evaluation and comparison between the different average properties (January 2015 to September 2016)

	Fuel		Flash	Gravity	Viscosity	Sulfur	Cetane	Derived	Cetane	Calculated
#	Sample	FS	Point	API	at 40°C	Content	Number	CN	Index (CI)	CI (CCI)
	FS	ID#	[°C]	at 60°F	cSt	[mg/kg]	D613	D6890	D976	D4737
1	Jan-15	D1094	65.7	38.9	2.75	5.9	53.4	52.3	55.2	55.0
2	Feb-15	D1095	44.4	51.7	1.21	100.7	31.7	36.8	50.6	53.5
3	Mar-15	D1096	48.0	35.4	2.00	11.5	41.9	43.2	42.1	41.3
4	Apr-15	D1097	63.1	37.9	2.39	8.0	48.5	49.7	49.1	49.0
5	May-15	D1098	60.8	36.2	2.41	110.8	46.0	46.4	47.8	47.0
6	Jun-15	D1099	61.6	36.6	2.59	7.3	46.4	45.1	48.1	48.0
7	Jul-15	D1100	76.8	33.0	2.23	11.9	39.3	39.8	39.5	39.6
8	Aug-15	D1101	70.3	38.5	2.37	6.2	51.5	50.6	50.8	51.2
9	Sep-15	D1102	51.9	40.9	2.16	4.7	51.8	50.0	53.4	52.7
10	Oct-15	D1103	50.4	36.7	2.54	7.1	47.0	45.6	49.2	48.5
11	Nov-15	D1104	80.2	31.8	2.38	6.8	39.2	39.2	38.2	38.5
12	Dec-15	D1105	62.2	33.9	2.82	7.2	43.7	42.7	44.7	44.5
13	Jan-16	D1106	70.9	39.2	2.36	1.5	54.5	52.7	52.2	52.8
14	Feb-16	D1107	57.7	35.3	3.29	8.6	51.0	51.3	50.6	51.1
15	Mar-16	D1108	66.4	38.1	2.78	9.4	54.9	56.1	51.7	51.6
16	Apr-16	D1109	70.2	50.5	2.56	0.4	73.2	78.1	77.4	83.1
17	May-16	D1110	47.1	42.4	1.50	6.4	43.7	42.4	44.7	45.8
18	Jun-16	D1111	57.1	35.1	2.49	7.8	43.8	42.5	46.7	45.2
19	Jul-16	D1112	69.5	38.6	3.19	5.5	56.5	55.5	55.9	57.8
20	Aug-16	D1113	60.0	36.4	2.45	7.5	47.1	45.9	48.4	47.6
21	Sep-16	D1114	64.2	32.4	2.84	8.2	43.2	42.7	44.4	43.2

### Comparisons between the DCN of the D6890 Instrument and the CN of the D613 Instrument in Terms of Standard Deviation (STDEV)

Figure 5 summarizes the precision data from the ASTM NEG FEP for the last 21 months (January 2015 to September 2016). This plot compares the results from the D6890 instrument with those of the D613 cetane engine. The plot includes the standard deviation (STDEV) of the DCN and CN measurements, and the number of laboratories that participated in these tests. The average D6890 DCN STDEV, over the 21 months of data presented in Figure 5 was 0.85 DCN. An average of 12 instruments participated over that time. The average D613 CN STDEV was 1.40 CN, with an average of 34 engines participating.

Over the period of January 2015 to September 2016), the NEG FEPs showed a general improvement in precision for the D6890/EN15195 instruments, compared with D613/EN ISO 5165 instruments, on a monthly basis. In the ASTM NEG FEP data shown in Figure 5, the test results for the fuel sample #6 (D1099) produced a D6890 DCN STDEV of only 0.19 DCN. The D613 CN STDEV for that fuel sample was 1.54 CN. The D6890 DCN STDEV for fuel sample #6 was the lowest STDEV value reported for any NEG or EI FEP fuel sample since reporting of D6890/EN15195 DCN values began in 2003/2004.

In the ASTM NEG FEP results, using the combined upgraded D6890 and conventional D6890 instruments, the D6890 DCN STDEV is lower than the D613 CN STDEV for 84% of the test samples. This has been the case since the reporting of D6890 DCN values began in 2003. The two exceptions were fuel samples #8 (D1101) and #16 (D1109).

As shown in <u>Table 2</u> and <u>Table 4</u>, fuel sample #16 (D1109) had properties that deviated significantly from the ASTM D975 diesel fuel requirements. This puts this fuel outside the upper scope limit of D6890/EN 15195, which may affect the accuracy and precision of the method.

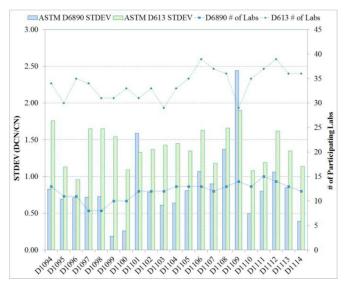


Figure 5. Number of Labs and Standard Deviations for the ASTM NEG Program (Data from January 2015 to September 2016)

Given the limited number of D6890 instruments participating in the ASTM NEG FEP, a problem with a single instrument can have a substantial impact on the statistical analysis of the test results. Fuel sample #8 (D1101)'s exceptionally high DCN STDEV was the result of a single laboratory's test result (referred to as "Laboratory X"), which was 3.85 DCN above the mean D6890 DCN value for the fuel. The ASTM NEG FEP does reject outlying data, but Laboratory X's test result was not considered an outlier by the statistical test used, as discussed in the following sub-section.

#### Outlier Rejection in the ASTM NEG and EI FEPs

The Generalized Extreme Studentized Deviate (GESD) Many-Outlier Procedure (ASTM D7915 [25]) is used by the ASTM NEG FEP as an outlier rejection practice. In this practice, the standard score or Z score<sup>2</sup> of each result is calculated and the minimum or maximum Z score for the data set is compared with a critical value from a table. This table states the critical values, for various data set sizes, at the 99% confidence level. If the minimum or maximum Z score of a certain laboratory equals or is greater than the critical value, the result from that laboratory is rejected as an outlier. The procedure is then repeated with the remaining results, until no more outlying results are identified.

In the EI FEP reports, the Grubbs test is used as an outlier rejection practice. This test practice is good for identifying a single outlier, however the GESD is recommended for identifying multiple outliers in a data set [ $\underline{25}$ ]. Similar to the GESD test, the Grubbs test uses the Z score procedure mentioned above. In the EI FEP report, the highest calculated Z score, and both the 99% and 95% confidence limits, are all reported.

During the period of time from January 2015 to September 2016 Laboratory X reported test results 14 times, of which 3 were rejected as outliers by GESD. According to the Grubbs outlier test, 3 of the remaining 11 test results have Z scores that lie at or between the critical values for the 95% and 99% confidence limits. Table 5 summarizes the results of the Grubbs test performed on fuel sample #8 (D1101), #12 (D1105), and #20 (D1113). The table shows that the critical values for the 95% and 99% confidence limits for the tested samples, and also the effect of rejecting Laboratory X's test results on

the D6890 DCN STDEV. When Laboratory X's test results were rejected for the 3 additional fuel samples, the D6890 DCN STDEV improved significantly. From the data analysis conducted on the performance of Laboratory X, it is obvious that problems with a single instrument can have a substantial impact on the precision of the method used.

Table 5. Grubbs outlier test results for some NEG samples

Sample ID	Z score	99% Critical	95% Critical	NEG	STDEV
	max.	Value	Value	STDEV	(without Lab. X)
Aug-15 D1101	2.443	2.636	2.412	1.58	1.07
Dec-15 D1105	2.497	2.699	2.462	0.81	0.56
Aug-16 D1113	2.680	2.699	2.462	0.85	0.53

The results from the NEG fuel exchange programs, presented in Figure 5, have shown ongoing improvements in the precision (STDEV) of test method D6890. The average DCN STDEV for the combined upgraded D6890 and D6890 instruments is significantly less than that of the average D613 CN STDEV. The following section of the paper compares the performance of the conventional and upgraded D6890 instruments in this study.

# Comparison between the Performance of the Upgraded and Conventional D6890 Instruments Participating in ASTM NEG FEP

The majority of the D6890 instruments participating in the NEG and EI studies are conventional instruments rather than the upgraded instruments that are equipped with the precision package. As mentioned in previous section, an average of 4 upgraded D6890 instruments participated in the NEG FEP for the last 21 months. This section of the paper updates the comparison between the performance of the conventional instruments and the upgraded D6890 instruments that participated in the ASTM NEG FEP over the period of January 2015 to September 2016.

The ASTM NEG FEP test results for the period of time under consideration were analyzed and presented in Table 6. The table compares the separate DCN STDEV values from the upgraded and conventional D6890 instruments with the reported overall NEG DCN STDEV. The superscript of each value shown in Table 6 represents the number of participating laboratories that produced the collective result (e.g. 0.19<sup>10</sup> means that 10 Laboratories were represented in the calculation of a STDEV value of 0.19 for the June 2015 (D1099) sample). The average DCN STDEV, over the 21 months, for the conventional D6890 instruments (see Table 6) is 0.97 DCN, with an average of eight (8) instruments participating. The average DCN STDEV for the upgraded D6890 instruments is 0.48, with an average of four (4) instruments participating. The combined DCN STDEV for both the upgraded and conventional D6890 instruments was 0.85 DCN.

Table 6 shows significant improvements in precision (STDEV) with the upgraded D6890 instruments. The sole exception (D1098) was a case where the sample was evaluated with only two upgraded D6890 instruments. Even for the test sample that was out of the scope of D6890 (D1109), the DCN STDEV for the upgraded D6890 instruments is still lower than the overall DCN STDEV and the D613 CN STDEV (see Figure 5 and Table 6).

 $<sup>\</sup>frac{1}{2} Z Score = \frac{DCN_i - \overline{DCN}}{STDEV_{DCN}}$ 

Table 6. Comparison between the performance of the upgraded and the conventional D6890 instruments participated in ASTM NEG 2015-2016 test results

		NEG	Conventional	Upgraded
FS		D6890 (both Instruments)	D6890 Instrument	D6890 Instrument
#	ID#	STDEV # of IQTs	STDEV # of IQTs	STDEV # of IQTs
1	D1094	0.83 13	0.94 9	0.37 4
2	D1095	0.69 11	0.74 8	0.68 3
3	D1096	0.71 11	0.77	0.62 4
4	D1097	0.72 8	0.86 5	0.45 3
5	D1098	0.73 8	0.71 6	0.92 2
6	D1099	0.19 10	0.23 6	0.15 4
7	D1100	0.26 10	0.26 6	0.26 4
8	D1101	1.59 12	1.83 9	0.36 <sup>3</sup>
9	D1102	0.79 12	0.96 <sup>9</sup>	0.39 <sup>3</sup>
10	D1103	0.61 12	0.70 9	0.33 3
11	D1104	0.64 13	0.75 9	0.22 4
12	D1105	0.81 13	0.94 9	0.52 4
13	D1106	1.07 13	0.94 9	0.52 4
14	D1107	0.91 12	0.99 8	0.79 4
15	D1108	1.36 13	1.55 9	0.68 4
16	D1109	2.44 14	2.77 10	1.23 4
17	D1110	0.5 13	0.44 10	0.32 3
18	D1111	0.8 15	0.90 11	0.41 4
19	D1112	1.05 14	1.24 10	0.39 4
20	D1113	0.85 13	0.98 9	0.52 4
21	D1114	0.39 12	0.46 8	0.21 4

# Repeatability and Reproducibility Estimation for the Upgraded D6890 Instruments Participating in the ASTM NEG FEP

In this paper, the precision of the upgraded D6890 instruments that participated in the ASTM NEG FEP was estimated using ASTM E691. The formulas presented in <u>Table 3</u> were used to calculate the 95% repeatability (r) and 95% reproducibility (R) statistics. The estimated precision terms (r & R) were used for a comparison with the CN referee method (ASTM D613 standard).

The estimated results of both the upgraded D6890 instrument precision terms (r and R) are presented in Figure 6 and Figure 7 respectively as data points. The two figures also show comparisons between the estimated precision of the upgraded D6890 instrument and the CN referee method (ASTM D613). The numbers shown in both figures represent the chronological sequence number of the sample as listed in Table 4 and Table 6. Using this real world data (NEG FEP data), the precision of the upgraded D6890 instruments shows on average the lowest values compared with the other CN (D613) and DCN (D6890/EN15195) test methods.

On average, only 3 or 4 upgraded D6890 instruments have participated in the NEG program over the last 21 months. As indicated above, problems with a single instrument or fuel sample can have a substantial impact on the precision reported by the program. This was the case for some of the highest values shown in Figure 6 and Figure 7 (e.g. sample #16), where deterioration of one instrument component led to overall reproducibility that was comparable to that of the updated CEN EN 15195:2014 method.

As discussed in the NEG fuel sample properties, Sample #16 has a higher API gravity compared with the diesel fuel specifications presented in <u>Table 2</u>. Sample #16 has also a higher CN/DCN than the

precision applicable ranges of all the CN/DCN methods. Please refer to <u>Table 4</u> to compare the performance of the test samples to their required properties.

Most of the data points shown in Figure 7 are consistent with the reproducibility demonstrated in the upgraded D6890 instruments mILS [2]. Thus, the available results from the fuel exchange program support the view that the upgraded D6890 instruments can achieve reproducibility comparable to that previously reported for the upgraded D6890 instrument mILS [2] while operating under demanding, real-world conditions.

### ASTM NEG FEP Test Results; Accuracy Improvement, CN versus DCN (January 2015 to September 2016

Figure 8 presents the ASTM NEG FEP test results for the period from January 2015 to September 2016, as well as the published reproducibility limits for both the D613 and D6890. Figure 8 also shows the ±1 STDEV limits, and the 95% confidence limits, for the average D613 CN values reported in the ASTM NEG FEP. In Figure 8, all of these limits are plotted to show the results of the 21 ASTM NEG FEP fuels samples relative to ASTM D613's statistical limits.

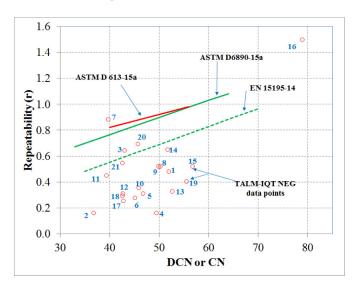


Figure 6. Comparison between the upgraded D6890 instruments estimated repeatability and the repeatability of D613/D6890/EN15195 test methods

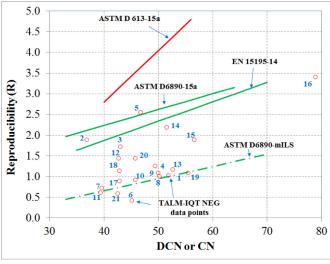


Figure 7. Comparison between the upgraded D6890 instruments estimated reproducibility and the reproducibility of D613/D6890/EN15195 test methods

Figure 8 plots the overall average D6890 DCN values for the 21 ASTM NEG FEP samples (empty circles), along with the average D6890 DCN values for the upgraded D6890 instruments (solid triangles). To make the plot easier to read, Figure 8 does not show fuel samples with DCN values that are outside the scope of D6890 (samples D1095 and D1109). Both the overall average D6890 DCN values and the average upgraded D6890 instruments DCN values show a good correlation with the average D613 CN values for those fuel samples.

Of the 21 fuel samples tested, 19 samples have overall average D6890 DCN values within the  $\pm 1$  STDEV limits of D613, and 6 of the tested samples (~29%) have their overall average D6890 DCN values within the 95% confidence limits of ASTM D613. In case of the upgraded D6890 instruments' results, 9 of the tested samples (~43%) have their average DCN values within the 95% confidence limits of D613. Since the majority of the upgraded D6890 instruments' data points are closer to the CN/DCN parity line, the test results presented in Figure 8 indicate that the upgraded D6890 instruments not only improves the precision it also improves the accuarcy of the ASTM D6890.

The overall average D6890 DCN value for samples D1095 and D1109 were outside of the D613 ±1 STDEV limits. However, only sample D1095's overall average D6890 DCN value was also outside of the 95% confidence limits of D613. Both of these fuel samples had properties outside of the limits of the diesel fuel specifications presented in Table 2. Sample D1095's D613 CN and viscosity were below minimum limits, and both samples had API gravity above the maximum limit.

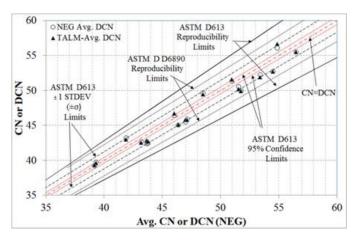


Figure 8. Correlation of ASTM NEG average DCN and the upgraded D6890 instruments average DCN values with ASTM D613 statistics for ASTM NEG samples

### **Summary and Conclusions**

The D6980/EN 15195/IP 498 instrument is a CVCC instrument that offers an alternative to the CFR cetane engine for measuring the ignition quality of diesel fuels. Of the CVCC instruments used to measure the DCN of a fuel sample, the D6890/EN 15195/IP 498 instrument has the most extensive FEP data set (since 2003/2004). This real world data set continues to be used to update the published precision for the instrument. The D6890, EN15195, and IP 498 standards have the widest applicable DCN range of all the ASTM and CEN CN/DCN/CI standards and provides better precision at higher

CN/DCN values compared to the referee method (D613/EN ISO 5165). According to the recently published EN 15940:2016 fuel standard, EN 15195 allocated as the referee method is the more precise method for measuring the high CN/DCN fuel samples such as the paraffinic diesel fuels (or renewable diesel fuels) from synthesis or hydrotreatment.

The ASTM NEG FEP results have shown good comparative results between the D613/EN ISO 5165/IP41 and D6890/EN 15195/IP 498 instruments over a wide CN/DCN range. The results of 21 fuel samples were extracted from the ASTM NEG FEP reports and used to evaluate the performance of both the conventional (LM-IQT<sup>TM</sup>) and the upgraded D6890 instruments (TALM-IQT<sup>TM</sup>).

Some of the average measured fuel sample properties were presented, and used to examine compliance of each sample with the ASTM standard D975 diesel fuel requirements. The property limits discussed in the paper show that a fuel sample that deviates significantly from the requirements of the ASTM D975 diesel fuel specification, such as Sample #16, may affect the accuracy and precision of the test results for that fuel sample, and hence the D6890 test method relative to ASTM D613.

The ASTM NEG FEP test results covering the period from January 2015 to September 2016 show that the overall average DCN STDEV for the conventional D6890 and the upgraded instruments was 0.85 DCN. The average CN STDEV for the D613 instruments over the same period was 1.40 CN. During the month of June 2015, overall average DCN STDEV was only 0.19 DCN for both instrument configurations, while the corresponding D613 CN STDEV was 1.54 CN). The 0.19 DCN STDEV was the lowest DCN or CN STDEV value reported for any result in the ASTM NEG or EI FEPs since the D6890 DCN testing starting in 2003/2004.

The upgraded D6890 instruments have demonstrated improved DCN STDEV relative to the conventional D6890 instruments in the ASTM NEG FEP, with monthly DCN STDEV values as low as 0.15 DCN. Over the period from January 2015 to September 2016, the average DCN STDEV for the upgraded D6890 instruments was 0.48 DCN, with an average of 4 instruments participating.

The precision (r, R) of the upgraded D6890 instruments participating in the ASM NEG FEP were estimated using ASTM E691. The majority of the ASTM NEG FEP test results for the upgraded D6890 instruments were consistent with the reproducibility demonstrated in the TALM-IQTTM mILS. Thus, the mILS reproducibility levels can be achieved by the upgraded D6890 instruments while operating under demanding, real-world conditions.

The upgraded D6890 instruments test results presented in this paper indicate that the TALM Precision Package not only improves the precision it also improves the accuarcy of the ASTM D6890. The results show that for the conventional D6890 instruments and the upgraded D6890 instruments, approximately 29% of the tested samples have their NEG average DCN values within the 95% confidence limits of D613 when all the test results are combined together. For the upgraded D6890 instruments alone, the results show approximately 43% of the tested samples have their average DCN values within the 95% confidence limits of the ASTM D613.

The upgraded D6890 instruments (TALM-IQT<sup>TM</sup>) while operating under demanding, real-world conditions, have demonstrated better repeatability, reproducibility and accuracy than all existing test methods used for measuring diesel fuel ignition quality.

#### References

- Ramadan, O., Webster, G., Menard, L., Wilcox, A., "Diesel Fuel Totally Automated Laboratory Model of the Ignition Quality Tester Technology Updates, Part-1: IQT<sup>TM</sup>- TALM Description and Micro Intra-Laboratory Study (µILS) Results," SAE Technical Paper 2014-01-0179, 2014, doi:10.4271/2014-01-0179.
- Ramadan, O., Webster, G., Menard, L., Wilcox, A., "Ignition Quality Tester (IQT<sup>TM</sup>) Precision Improvements from Using the Totally Automated Laboratory Model (TALM) Technology: Technology Update, Part-2: Mini Inter-Laboratory Study Using the IQT<sup>TM</sup>-TALM," SAE Technical Paper <u>2015-01-0805</u>, 2015, doi:10.4271/2015-01-0805.
- ASTM D6890-15a, ASTM International Standard Test Method, "Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber", 2015.
- EN 15195-14, British Standards Institute "Liquid Petroleum Products – Determination of Ignition Delay and Derived Cetane Number (DCN) of Middle Distillate Fuels by Combustion in a Constant Volume Chamber", BS EN 15195, 2014.
- 5. EI IP 498, Energy Institute IP Test Method, "Determination of ignition delay and Derived Cetane Number (DCN) of middle distillate fuels by combustion in a constant volume chamber", February 2015.
- 6. ASTM D613-15a, ASTM International Standard Test Method, "Cetane Number of Diesel Fuel Oil", 2015.
- EN ISO 5165, International Organization for Standardization (ISO) Test Method, "Determination of the ignition quality of diesel fuels – Cetane engine method", 1998.
- EI IP 41, Energy Institute IP Test Method, "Determination of the ignition quality of diesel fuels – Cetane engine method", January 1999.
- ASTM D7170-14, ASTM International Standard Test Method, "Determination of Derived Cetane Number (DCN) of Diesel Fuel Oils — Fixed Range Injection Period, Constant Volume Combustion Chamber Method", 2014.
- ASTM D7668-14a, ASTM International Standard Test Method, "Determination of Derived Cetane Number (DCN) of Diesel Fuel Oils—Ignition Delay and Combustion Delay Using a Constant Volume Combustion Chamber Method", 2014.
- 11. ASTM D976 06, Standard Test Method for Calculated Cetane Index of Distillate Fuels, 2016.
- 12. ASTM D4737-10, Standard Test Method for Calculated Cetane Index by Four Variable Equation, 2016.
- Yates, A., Viljoen, C., and Metcalf, O., "An Accurate Determination of the Cetane Number Value of GTL Diesel," SAE Technical Paper <u>2007-01-0026</u>, 2007, doi:<u>10.4271/2007-01-0026</u>.
- ASTM D975-15c, ASTM International Standard Specification for Diesel Fuel Oils, 2015.
- 15. EN 590, European Standard, BS EN 590, "Automotive fuels-Diesel-Requirements and test methods, 2013.

- 16. Goodheart-Willcox (G-W) Co., "Diesel Fuels, Chapter 14, Figure 14.2 (General classification chart for diesel fuels (Detroit Diesel)). <a href="https://www.g-w.com">www.g-w.com</a>.
- 17. Engine Manufacturers Association (EMA) report, EMA Recommended Guideline on Diesel Fuel, <u>www.enginemanufacturers.org</u>.
- 18. Bogin G. E., DeFilippo A., Chen J. Y., Chin G., Luecke J., Ratcliff M. A., Zigler B. T., and Dean A. M., "Modeling the Fuel Spray and Combustion Process of the Ignition Quality Tester™ with KIVA-3V", 2009 Fall Meeting of the Western States Section of the Combustion Institute, University of California-Irvine, Irvine, CA, October 26–27. 2009
- 19. Ramadan O., Gardiner D., Webster G., and Bell T. "How Precision Improvements for Derived Cetane Number (DCN) from the Ignition Quality Tester (IQT™) Employing the Totally Automated Laboratory Model (TALM) Technology can Lead to More Optimal Diesel Fuel Blending", Presented at Gulf Coast Conference, Oct. 20–21, 2015, NRG Park, Houston, Texas.
- Ramadan, O., Gardiner, D., Webster, G., and Bell, T. "IQT<sup>TM</sup>s with the TALM Precision Package Demonstrate the Potential for Best Precision of Derived Cetane Number (DCN)", Presented at Gulf Coast Conference, Oct. 11–12, 2016, George R Brown Convention Center, Houston, Texas.
- ASTM E691-15 "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method," Annual Book of ASTM Standards, ASTM International, West Conshohocken, PA.
- EN 15940:2016, "Automotive fuels. Paraffinic diesel fuel from synthesis or hydrotreatment. Requirements and test methods, BS EN 15940:2016, July 2016.
- 23. Aatola, H., Larmi, M., Sarjovaara, T., and Mikkonen, S., "Hydrotreated Vegetable Oil (HVO) as a Renewable Diesel Fuel: Trade-off between NOx, Particulate Emission, and Fuel Consumption of a Heavy Duty Engine," *SAE Int. J. Engines* 1(1):1251–1262, 2009, doi:10.4271/2008-01-2500.
- 24. Neste Corporation, May 2016, "Neste Renewable Diesel Handbook", <a href="www.neste.com/neste\_renewable\_diesel\_handbook.pdf">www.neste.com/neste\_renewable\_diesel\_handbook.pdf</a>
- 25. ASTM D7915-14, ASTM Standard Practice for Application of Generalized Extreme Studentized Deviate (GESD) Technique to Simultaneously Identify Multiple Outliers in a Data Set, 2014.

#### **Contact Information**

Advanced Engine Technology Ltd. 17 Fitzgerald Road, Suite 102 Ottawa, Ontario Canada, K2H 9G1 Contact: Gary Webster

gary@aet.ca

Phone: 613-721-1234

#### **Definitions/Abbreviations**

**ASTM** - ASTM International, formerly the American Society for Testing and Materials

**CEN** - European Committee for Standardization

CI - Calculated Cetane Index using ASTM D976-06

CCI - Calculated Cetane Index using ASTM D4737-10

**D613** - ASTM D613, ASTM standard test method for the CFR<sup>TM</sup> cetane engine

D6890 - ASTM D6890, ASTM standard test method for the  $\mathrm{IQT^{TM}}$  instrument

EI - Energy Institute

**EN** - European Standard (Norme Européenne)

 $\mathbf{EN}$  15195 - CEN EN 15195, European standard test method for the  $\mathbf{IOT^{TM}}$ 

**EN ISO 5165** - CEN EN ISO 5165, European and global standard for the CFR $^{TM}$  cetane engine

EN 15940 - CEN EN 15940:2016, European standard for paraffinic diesel fuels

**IP** - Institute of Petroleum (UK), merged with the Institute of Energy in 2003 to form the Energy Institute

**IP 41** - EI IP 41, UK standard test method for the CFR<sup>TM</sup> cetane engine, equivalent to EN ISO 5165

 ${\bf IP~498}$  - EI IP 498, UK standard for the IQT<sup>TM</sup>, equivalent to EN 15195

**NEG** - ASTM National Exchange Group

**TALM** - Totally Automated Laboratory Model, IQT<sup>TM</sup> instrument with enhanced automation features

The Engineering Meetings Board has approved this paper for publication. It has successfully completed SAE's peer review process under the supervision of the session organizer. The process requires a minimum of three (3) reviews by industry experts.

All rights reserved. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording, or otherwise, without the prior written permission of SAE International.

Positions and opinions advanced in this paper are those of the author(s) and not necessarily those of SAE International. The author is solely responsible for the content of the paper.

word版下载:	http://	/www.ixueshu.co	m

ord版卜载: <u>http://www.ixueshu.com</u>