TOP TIER DIESEL FUEL PERFORMANCE STANDARD

Revision A - February 2017

1. Scope.

1.1 Performance Description. This document describes the performance standard for diesel fuel at a retail level or private fueling site that has detergents to prevent deposit formation on injectors, enhanced stability, improved lubricity, and lower water & particulate contamination.

2. References.

Note: Only the latest versions of standards are applicable or as indicated.

2.1 ASTM International (American Society for Testing and Materials)

ASTM D975, ASTM D6751, ASTM D7467, ASTM D6079, ASTM D6304, ASTM D6079, ASTM D6217, ASTM D7545, ASTM D7501, ASTM D2274, ASTM D524, ASTM D2500, ASTM D23015, ASTM D6371, ASTM D130, ASTM D664, ASTM D93, ASTM D971, ASTM D471, ASTM D6201

2.2 Coordinating European Council (CEC)

CEC F-98-08 (DW10B) and CEC F-110-16 (DW-10C), EN590, EN 15751, EN 14214, DIN EN 12662, DIN EN 116

2.5 International Organization for Standardization (ISO)

ISO 17025, General Requirements for the Competence of Testing and Calibration Laboratories, ISO 4406, ISO 16889, ISO 12205, ISO 10370, ISO 2160, ISO 2719, ISO 1817, ISO 37

3. Definitions

3.1 Independent Laboratory – a mechanical and/or chemical testing organization which has been approved by General Motors for testing the performance of diesel fuel to the TOP TIER Diesel Fuel Performance Standard; provided, however, that the Independent Laboratory is not affiliated with the entity seeking TOP TIER Diesel Fuel status or with the manufacturer of any additive package approved for use in TOP TIER Diesel Fuel.

4. Standards.

4.1 Fuel Additive Requirements. The additive(s) to be used as part of TOP TIER diesel requirements described in the sections below must meet all the requirements of the regulatory body in country of sales. Documentation demonstrating such compliance is required. For example, in the United States, the additive must be registered as per regulations of Title 40 CFR Part 79 of Section 211 as stipulated by Environmental Protection Agency (EPA) to assess impact of the product on emissions.

4.2 Diesel Performance Standards. The performance standards of diesel fuel conforming to section 4 of this document shall be met at the point of delivery to the vehicle at the retail level or private fueling site of diesel sold by a fuel company in all or selected marketing areas of a nation. In addition, conformance to the standards shall mean that the diesel fuel sold in the selected nation will meet the latest existing standards for diesel/biodiesel blends such as ASTM D975 and ASTM D7467 standards in the United States.

4.2.1. Fuel Stability. Biofuels such as fatty acid esters, commonly called “biodiesel,” are increasingly being used in the marketplace. In the United States, the quality of common fatty acid alkyl esters is defined in the ASTM D6751 standard for biodiesel fuel blend stock (B100) intended for use in blends at concentrations of up to 20 volume percent. There are two additional standards that define the quality of the blended fuels, ASTM D975 which applies to blended fuels up to B5 and ASTM D7467 which applies to blended fuels from B6 to B20.

Field warranty and validation tests have shown significant concerns with stability of biodiesel fuels including fuel system deposits, engine oil deterioration, and efficiency loss of the after treatment system. Maintaining high stability of biodiesel blended fuels is critical for its optimal performance as a vehicle fuel.

In the United States, the ASTM D6751 standard for FAME has a stability requirement as measured by an induction period of 3 hours minimum, which is significantly lower than the induction period of a minimum of 8 hours that is required by the European EN 14214 standard. There are no stability requirements in ASTM D975, while the stability

requirements for B6 to B20 blends as per ASTM D7467 is a minimum of 6 hours. In comparison, the EN590 standard which allows biodiesel content up to 7 volume percent has a limit for stability of a minimum 20 hours.

Fuel injection equipment and after-treatment systems could be significantly damaged from the use of oxidized fuel. Diesel OEMs supporting the TOP TIER diesel program would like to ensure that the level of oxidation stability in the diesel as well as biodiesel blended fuel is sufficient to prevent damage due to oxidized fuel under all normal scenarios in which the fuel is stored and used.

Fuel injection equipment and other components could have significant maintenance and/or operating issues from use of oxidized fuel. Diesel OEMs supporting TOP TIER diesel program would like to ensure that the level of oxidation stability in the diesel as well as biodiesel blended fuel is sufficient to prevent any issues due to oxidized fuel in all scenarios of fuel storage and use.

4.2.1.1 Test Method. Fuel stability of biodiesel and biodiesel blended fuels (≥B2) is best described by the parameter 'ageing reserve' determined as Rancimat induction period (IP) according to the EN 15751 test method.

Fuel stability of diesel fuels without any biodiesel (B0) or up to B2 blend must be determined by Rapid Small Scale Oxidation Test (RSSOT) - PetroOXY test as per ASTM D7545 method which measures the induction period under specified conditions and can be used as an indication of the oxidation and storage stability of diesel fuels.

4.2.1.2 Acceptable Performance Limit. The fuel should have following stability properties to meet the TOP TIER diesel performance:

1. The FAME Biodiesel (B100) to be used to make biodiesel blends must have induction period of minimum of 8 hours as measured by EN15751 test method
2. If it is not possible to qualify the stability performance of B100 used to make a biodiesel blend then following stability requirements must be fulfilled at the blend levels:
    a. For biodiesel blends up to 5 volume percent as allowed by ASTM D975 standard, the induction period should be minimum of 24 hours as measured by EN15751 test method.
    b. For biodiesel blends from B6 to B20 as per ASTM D7467 or for blended fuels above B20 biodiesel blends, the induction period should be minimum of 20 hours as measured by EN15751 test method.
3. For petroleum diesel without any biodiesel the expected stability as measured by petro-oxy test (ASTM D7545) should have an induction period of > 60 minutes.

4.2.2 Lubricity. Good lubricity of diesel fuel is important to minimize friction and damage in diesel fuel system components, especially in the fuel pumps. A position statement by the Diesel Fuel Injection Manufacturers of 2012 suggests that the lubricity of the fuel as measured by the HFRR according to ASTM D6079 must have a lubricity value of 460 microns maximum. Fuel with lubricity exceeding 460 microns can adversely affect the lifetime of some of the fuel lubricated injection system components.

4.2.2.1 Test Method. The lubricity of the diesel fuel should be measured by the *High Frequency Reciprocating Rig* (HFRR) method which measures the wear scar on the test specimen ball as per the procedure described in ASTM D6079.

4.2.2.2 Demonstration of Performance. To meet TOP TIER diesel performance the fuels must have an HFRR at 60°C wear scar diameter of less than 460 µm to provide sufficient lubricity.

4.2.3 Particulate Contamination in Fuel. Particulates and water are the most common contaminants in diesel fuel. Modern diesel engines have fine clearances and high operating pressures which makes them highly sensitive to particulate contamination in the fuel. High level of particulate contamination in the diesel fuel can lead to premature clogging of vehicle fuel filters or if the hard particles are carried past the fuel filters, they can cause premature wear of fuel injection systems as well as engine failures. For effective control of particulate contamination in the fuel, it is important to control both the number of particles as well as size of the particles.

4.2.3.1 Test Method. The ISO 4406 test standard provides a measure of particulate contamination based on size and number of particles. The standard defines three code numbers corresponding to the numbers of particles of size greater than 4, 6 and 14 µm per milliliter, respectively.

4.2.3.2 Demonstration of Performance. There are numerous ways particulate contamination can enter the fuel during storage and transportation some of which are beyond the control of the fuel station operators. Also, there is increased risk that improper fuel sampling procedures during audits will artificially increase particulates in the fuel. Thus, for the TOP TIER diesel program, there is no specific requirement for particulate contamination but it is expected that the sediment content be within the specifications defined in latest version of ASTM D975 or ASTM D7467 standards. Although there is no specification for particulate contamination, it is preferable that the fuel cleanliness for particulates meets the typical engine manufacturer requirements identified as per an ISO 4406 Code18/16/13 fuel cleanliness rating at the dispenser nozzle. This rating reflects scale #18 (1300-2500 particles greater than 4 microns), scale #16 (320-640 particles greater than 6 microns), and scale #13 (40-80 particles greater than 14 microns) in any given milliliter sample of fuel.

4.2.4 Water Contamination in Fuel. Water contamination, if not controlled, can led to many problems in the fuel system such as corrosion, fuel degradation, microbial growth, fuel pump wear and ice formation during cold weather. Water is also a good solvent for inorganic salts and can contain dissolved acids or other contaminants that can harm the fuel system and engine either directly or indirectly by deposits formation due to interactions of these contaminants with additives in the fuel. Water may enter the fuel in various ways, such as part of the refining process, as rain, as ship ballast water or as condensation in storage tanks and equipment. Good water management will help minimize the contamination of the fuel during storage and transportation. It is recommended to follow the guidelines and procedures provided in CRC Report 667 – Diesel Fuel Storage and Handling Guide to lower risks for water and particulate contamination in fuel.

Good housekeeping practices and routine maintenance are the best ways to keep water accumulation and particulate contamination in control during storage. In addition, removal of free water from the fuel before the fuel is filled into vehicle fuel tank is also desirable.

4.2.4.1 Test Method. Total water content in diesel fuel shall be measured by procedure described in ASTM D6304 “Standard Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Titration.”

4.2.4.2 Demonstration of Performance. There are numerous ways water can enter the fuel during storage at the fuel station, some of which are beyond the control of the station operators. Thus, for the TOP TIER diesel program there is no specific requirement for absolute water content of the fuel but it is expected that the water content be within the specifications defined in latest version of ASTM D975 or ASTM D7467 standards Additionally, it is preferable that the water content be less than 200 mg/kg at 25°C as measured by ASTM D6304. Audit samples will be tested for water content as per ASTM D6304.

4.2.5 Dispenser Filters. Diesel dispenser filters are last line of defense before particulate or water contamination in the fuel can be delivered into a vehicle’s fuel tank. Thus, better requirements on the fuel dispenser filters can help to significantly reduce concerns associated with fuel contaminations like particulates and water. Generally, the dispenser filters are of the 30 microns to 10 microns in size. A finer size filter is expected to be more efficient in cleaning up the particulate contamination and reduce vehicle concerns related to these contamination including increased filter plugging within defined vehicle filter service intervals.

4.2.5.1 Test Method. The filtration efficiency shall be as per the multipass test procedure defined in ISO 16889 method which describes a procedure for determining the contaminant capacity, particulate removal and differential pressure characteristics and a test using ISO medium test dust contaminant and a test fluid. For water absorption capacity testing, there is no official test due to variety of applications and conditions in the field.

4.2.5.2 Demonstration of Performance (Particulate Filter). Dispenser stations with low speed pumps (fuel flow of less than 15 gallons per minute) shall be equipped with a filter having 10 micron or smaller pore size with a filtration efficiency of greater than 90% at removing 10 micron size particles as demonstrated by ISO 16889 procedure. For dispensers with flow rates greater than 15 gallons per minute, it is recommended to have a 10-micron or smaller nominal pore-sized filter; however, it is a requirement to install a dispenser filter of at least 30-micron or smaller nominal pore size filter with filtration efficiency greater than 50% as demonstrated by ISO 16889 procedure.

4.2.5.3 Demonstration of Performance (Water Absorbing Media). For the water absorption capacity, the filter or a separate water absorption unit shall act as detection devices to alert the station operator of excess water conditions

by restricting or stopping flow when excessive water contamination is present. Alternately, it would be acceptable if the retail station or private fueling site uses fuel monitoring technology with robust process to monitor and control excess water in the fuel tanks. Fueling sites using monitoring or detection technology should review their process and quality procedure with the TOP TIER Diesel Fuel program to obtain written approval.

4.3 Detergent Performance Standard. All performance testing and fuel composition analysis shall be conducted by an Independent Laboratory. Initial deposit control performance shall be demonstrated using the tests shown below.

4.3.1 Diesel Injector Nozzle Deposit Test

4.3.1.1 Test Method. Diesel Injector nozzle keep clean performance shall be demonstrated using CEC F-98-08 (DW10B) *Diesel injector nozzle fouling*. In the above test, the base reference fuel minimum deposit level and additive performance shall be conducted using the same engine block and cylinder head. For the CEC-F-98-08 (DW10B), performance data shall be reported in terms of torque loss for each cycle of the test. The final result shall be reported as loss of engine power as measure of injector fouling performance. Reported results should be within the stated reproducibility of the test procedure.

4.3.1.2 Base Fuel. For the CEC F-98-08 (DW10B) test, the base fuel shall conform to the specifications as per RF-06-03 (European Certification test fuel) reference fuel to break in the fuel injectors. For the key part of the CEC F-98-08 (DW10B) test to induce external nozzle deposits, one part per million (ppm) of zinc (Zn) in form of zinc neodecanoate is added to the reference fuel as defined in CEC F-98-08 test procedure.

4.3.1.3 Demonstration of Performance. The base fuel from 4.3.1.2 treated with additive at the concentration meeting the standard found in 4.3.1.1 shall not result in more than 2% power loss while the power loss for the base fuel with Zn neodecanoate and without detergent additive should exceed 6% power loss.

4.3.2 Internal Diesel Injector Deposits Test

4.3.2.1 Test Method and Demonstration of Performance. Engine test method using a common rail fuel system engine demonstrating dirty-up without use of additive and clean-up performance with use of the detergent additives for internal diesel injector deposits caused by carboxylates salts of calcium and sodium. Additional demonstration of keep clean performance of additive in presence of any other contaminants that can cause internal injector deposits is optional but would be valuable for demonstrating effectiveness of additive formulation. In addition, any test data demonstrating effectiveness of the detergent additive to prevent deposit formations in vehicle tests or fleet tests should be submitted for evaluation. The decision on the approval for additives performance for IDID deposits shall be made after review of data by the OEM sponsors. Reported results should be within the stated reproducibility of the test procedure.

4.3.2.2 DW-10 C Test Method (As of January 2017, this test method is a placeholder until formally approved by CEC). Internal Diesel Injector deposit test keep clean performance shall be demonstrated using CEC F-110-16 (DW10C), *Internal Diesel Injector Deposits.* In the above test, the base reference fuel minimum deposit level and additive performance shall be conducted using the same engine block and cylinder head. For CEC F-110-16 (DW10C) test performance data shall be reported in terms of global rating system derived from complex demerit scale for operational issues. Reported results should be within the stated reproducibility of the test procedure.

4.3.1.2 Base Fuel. For the CEC F-110-16 (DW10C) test, the base fuel shall conform to the specifications as per RF-06-03 (European Certification test fuel) reference fuel to break in the fuel injectors. For the key part of the CEC F-98-08 (DW10B) test to induce external nozzle deposits, one part per million (ppm) of zinc (Zn) in form of zinc neodecanoate is added to the reference fuel as defined in CEC F-98-08 test procedure.

4.3.1.3 Demonstration of Performance. The base fuel from 4.3.1.2 treated with additive at the concentration meeting the standard found in 4.3.1.1 shall not result in more than 2% power loss while the power loss for the base fuel with Zn neodecanoate and without detergent additive should exceed 6% power loss.

4.4 No-Harm Testing Requirements. The requirements for no-harm testing should demonstrate suitable interaction of the candidate detergent deposit control additive used in the fuel marketer final blend package. The additive

package will have to pass 'no-harms' testing with fuel containing biodiesel at highest blend rate along with lubricity, conductivity, stability additives tested with each detergent deposit control additive.

4.4.1 Demonstration of Performance. The acceptable reference fuels shall be the representative retail pump fuel meeting the ASTM D975 or ASTM D7467 specifications to which this package is expected to be added. The additive package will have to pass the 'no- harms' testing listed in Table 1 and section 4.4.2 that shall be the representative of the retail pump fuel meeting the ASTM D975 or ASTM D7467 specifications. The fuel for 'no-harms' testing must contain biodiesel at highest blend rate with additives for lubricity, conductivity, and stability required to meet TOP TIER diesel performance tested with the detergent deposit control additive. If there is more than one type of detergent deposit control additive that will be offered to meet the TOP TIER diesel performance than each of the formulation must be evaluated in market representative fuel. For each test, the entire TOP TIER diesel package shall be mixed at 3x the recommended concentration of the additives in the reference fuel and its performance will be compared to that of the relevant reference fuel. Tests defined in Table 1 shall be used to determine the candidate additive final package compatibility. All the final additive package blend combinations must be tested under these conditions.

Table 1: Tests for Fuel Compatibility

Properties	Standard	Unit	Pass/Fail Criteria
Fuel Contamination by Additive Precipitation	ASTM D6217 or DIN EN 12662	mg/L mg/kg	</= 20 </=24
Cold Soak Filterability	ASTM D7501	Seconds	< 360
Gum Forming Potential	ASTM D2274 or ISO 12205 or ASTM D7462 (for > B6 fuels)	mg/L	< 25
Carbon Residue (on 10% Distillation Residue), mass%	ASTM D524 or ISO 10370	%	0.35
Cloud Point	ASTM D2500 or ASTM D23015	°C	No change relative to reference fuel unless additive used to effect cold properties of fuel **Note 1**
Cold Filter Plugging Point	ASTM D6371 or DIN EN 116	°C	No change relative to reference fuel unless additive used to effect cold properties of fuel **Note 1**
Copper Strip Corrosion (3 h at 50 °C), Maximum	ASTM D130 or ISO 2160	Rating	No. 3
National Association of Corrosion Engineers (NACE International)	NACE TM0172	Rating	Same or Better than reference fuel
Acid Number, mg KOH/g, Maximum	ASTM D664	mg KOH/g	0.3 Maximum
Flash Point	ASTM D93 or ISO 2719	°C	55 Minimum (Summer Grade) 38 Minimum (Winter Grade)
Water Interfacial Tension	ASTM D971	Dynes/cm	To Report

Fuel Additive Resistance Diesel			
Elastomer Testing [Note 2]	A minimum of three (3) specimens to be tested per elastomer type. Required for fuels that have additives		
SAE J2643 Fluorocarbon (FKM-1)	ASTM D471 or ISO 1817, 168 h -2 h, at 23 °C ± 3 °C. Test ratio fuel to test pieces = (40 ± 5) to 1 All tests performed 1 minute after removal from test liquid Elongation at break, Tensile strength, Change in Mass, Properties after additional drying at +150 °C ± 3 °C for 24 h -2 h Elongation at break, Tensile strength, Change in Mass,	 % MPa % % MPa %	 120 Minimum 3 Minimum 0 to +30 150 Minimum 6 Minimum -10 to +5
SAE J2643 Hydrogenated Nitrile Butadiene Rubber (HNBR-1)	ASTM D471 or ISO 1817, 168 h -2 at 23 ± 3 °C Test ratio fuel to test pieces = (40 ± 5) to 1 All tests performed 1 minute after removal from test liquid Elongation at break, ASTM D412 or ISO 37 Tensile strength, ASTM D412 or ISO 37 Change in Mass, ASTM D471 or ISO 1817 Properties after additional drying at +150 °C ± 3 °C for 24 h -2 h Elongation at break, Tensile strength, Change in Mass,	 % MPa % % MPa %	 150 Minimum 5 Minimum 0 to +35 140 Minimum 5 minimum 0 to +25

Note 1: Values within the precision of the described test methods will be acceptable as no change.
Note 2: Purchase elastomer samples from Akron Rubber Development Laboratories at www.ardl.com.

4.4.2 Compatibility with Engine Oil.

4.4.2.1 Test Procedure. Samples must be tested per modified ASTM D2274 or ISO 10370 test procedure. The standard preparation and test procedures in accordance with ASTM D2274 specifications must be followed, except that bubbling oxygen during 16 hours heating period shall not be performed. The following two (2) samples shall be evaluated side by side:

a. Reference fuel without candidate additive but with 0.5% of engine oil which meets service fill specification for dexos2™.
b. Reference fuel with 3x the recommended treat rate of the candidate additive and with 0.5% of engine oil which meets the service fill specification for dexos2™.

The amount of insoluble material generated during the test shall be measured gravimetrically as defined in the test procedure for ASTM D2274. The amount of insoluble generated per 100 mL of the test fuel can provide measure of potential of fuel additive to react adversely with lubricant additive.

4.4.2.2 Demonstration of Performance. The amount of insoluble generated per 100 mL as determined by the modified ASTM D2274 method for sample (**a)** in 3.4.1, (reference fuel with engine oil but no candidate additive) and sample (**b)** in 3.4.1 (reference fuel with engine oil and with 3x candidate additive) shall be ≤ 2.5 mg/100 mL.

Note: Values within the precision of the described test methods will be acceptable as no change. Detection limit of test method ASTM D2274 is 1.0 mg/100 mL. Values lower than the detection limit of the test method shall be reported as < 1.0 mg/100 mL.

For a list of dexos2™ approved brands for diesel engine oil, visit: www.gmdexos.com.

It is recognized that no-harm compatibility testing can take significant resources, and, therefore, it is recommended that compatibility data generation test plan be reviewed by the TOP TIER coordinator in advance of conducting the tests.

The above no-harms tests should be regarded as minimum requirements to qualify the additive. Any additional information in terms of vehicle demonstration or field experiences or engine dyno tests would form a favorable basis to confirm consistent performance of fuel additive package.

5. Process to Attain TOP TIER Diesel Fuel Status.

5.1 Submission of Test Results. A fuel company desiring TOP TIER Diesel status shall forward the test results issued by the Independent Laboratory ("Test Results") to the following address:

Center for Quality Assurance
Attn: TOP TIER™ Licensing Program
4800 James Savage Rd.
Midland, MI 48642, USA
Telephone: +1 989-496-2399
Facsimile: +1 989-496-3438
Email: TopTier@CenterForQA.com

5.2 Notification of receipt. The Test Results shall be reviewed by representatives of General Motors & other OEM sponsors and, if deemed acceptable in its sole discretion, the fuel company will be provided a TOP TIER License Agreement for their execution. Only upon complete execution of the TOP TIER License Agreement by both the Fuel Company and General Motors shall the fuel company be entitled to begin use the TOP TIER name in connection with the distribution, promotion and sale of their gasoline, pursuant to the terms and conditions of the TOP TIER License Agreement.

6. Release, Effective Date, and Revisions.

6.1 Release. This document was first released in January 2017.

6.2 Revisions. Revision A is effective February 2017.

Revision	Date	Description of change
Original	January 2017	• Initial Release
A	February2017	• Remove reference to ASTM D6201 in section 4.3 • Update language in section 4.3 to clarify keep clean performance requirement for detergent for external coking and IDID. • Update section 4.4 to clarify requirements for no harms testing