

TOP TIER™ DIESEL FUEL - PERFORMANCE STANDARD – Plus Level

Revision A, January 2025

1. Scope.

1.1 Performance Description. This document describes the Plus level TOP TIER™ performance standard for diesel fuel at a retail level and/or private fueling sites. The Plus level standard provides the following enhanced protections over conventional ASTM D975 compliant diesel fuel; detergent additives to prevent internal and external fuel injector deposit formation, enhanced fuel dispenser filtering for both water and particulate contamination controls, improved lubricity requirements to help protect fuel system hardware, and increased oxidation stability requirements to prevent build-up of corrosive and gum forming chemistries in the fuel.

2. References.

Note: The latest revision versions of the standards are applicable unless otherwise indicated.

2.1 ASTM International

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 D6371, ASTM D130, ASTM D664, ASTM D93, ASTM D971, ASTM D471, ASTM D6201, ASTM D4176, ASTM D2709

2.2 Coordinating European Council (CEC)

CEC F-98-08 (DW10B) and CEC F-110-16 (DW-10C), RF-79-07, 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 third-party engine testing and/or chemical testing organization, which is ISO17025 certified or has been approved by the TOP TIER™ OEM program Sponsors for testing the performance of diesel fuel to the TOP TIER™ Diesel Fuel Performance Standard.

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 governing fuel additives in the country of sales interest. 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. TOP TIER™ diesel performance standards shall be met at the retail point of sale and/or private fueling site dispenser pump. In addition, conformance to the TOP TIER™ standards means that the diesel fuel sold shall meet the latest standards for diesel and/or biodiesel blends, such as ASTM D975 and ASTM D7467 standards in the United States.

4.2.1 Fuel Detergency Vehicle fuel system and fuel injection equipment deposit control performance shall be demonstrated using the tests detailed below.

4.2.1.1 Diesel Fuel Injector - Nozzle Face Deposit Test

4.2.1.1.1 Test Method.

Diesel injector nozzle keep clean performance shall be demonstrated using CEC F-98-08 (DW10B) Direct Injection, Common Rail Diesel Engine Nozzle Coking Test. This test is the established benchmark for assessing the impact of fuel and additives on injector nozzle coking (fouling) in diesel passenger vehicles equipped with Direct Injection (DI) engines. Utilizing a Peugeot DW10B Euro 4 build common rail engine with prototype piezo injector nozzles prone to deposit formation, the test incorporates the addition of a zinc dopant (1 mg/kg) to the standard reference

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fuel to expedite deposit formation. The DW-10B test protocol involves executing a one-hour test cycle primarily characterized by high speed and load conditions. Preceding the actual test phase with zinc-enhanced reference fuel, there is a recommended break-in period for the injectors using standard reference fuel without zinc dopant.

During testing, the one-hour test cycle is repeated eight times, with engine power measurements recorded at the culmination of each cycle. Subsequently, a 4-hour engine-off soak period is observed. After a 16-hour break-in period the entire test regimen encompasses four sets of 8-hour running cycles interspersed with three 4-hour engine-off soak periods, resulting in 32 hours of total running time and 12 hours of cumulative soak periods.

Two separate tests are required for a detergent additive efficacy assessment. The 'base reference fuel deposit test' and the 'additive performance test' shall be conducted using the same engine block and cylinder head combination. For the CEC-F-98-08 (DW10B), performance data shall be reported in terms of power loss. The power loss is not merely the difference between the SOT and EOT values; it reflects the trend observed over the first five and the last five measurements. Reported results should be within the stated reproducibility of the test procedure.

4.2.1.1.2 Base Fuel. For the CEC F-98-08 (DW10B) test, the base fuel shall conform to the specifications as per RF-79-07 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.

4.2.1.1.3 Demonstration of Performance. The base reference fuel deposit test without additive shall demonstrate $\geq 5\%$ power loss. The additive performance test shall demonstrate $\leq 2\%$ power loss. This performance requirement aims to ensure that injectors remain clean, promoting optimal engine performance.

If the fuel marketer aims to surpass the minimum keep-clean performance requirements and wants to offer fuel capable of cleaning existing deposits, the following performance standard is recommended: For the DW-10B test, the base fuel containing zinc neodecanoate, without any detergent additive, should exhibit a power loss $\geq 5\%$. Subsequently, the same test setup should be used to evaluate the base fuel with zinc neodecanoate, but this time with a detergent additive at an "enhanced" clean-up concentration. The test should demonstrate power restoration by reducing the power loss relative to the dirty-up test, achieving a power loss of no $\leq 2\%$.

4.2.1.2 Diesel Fuel Injector - Internal Deposit Test

4.2.1.2.1 Test Method. Performance for internal diesel injector deposits (IDID) keep clean shall be preferably demonstrated using CEC F-110-16 (DW10C), *Internal Diesel Injector Deposits*. In this test, the base reference fuel minimum deposit level and additive performance shall be conducted using the same engine block and cylinder head combination. The IDID DW10C test was developed to discriminate between a fuel that produces no measurable deposits and one that produces deposits that cause startability issues considered unacceptable by OEMs. The test's objective is to discriminate between fuels that differ in their ability to produce IDID in direct injection common rail diesel engines and subsequently demonstrate the ability of detergent fuel additives to prevent or control these deposits. These deposits differ from injector nozzle coking based on the location of the deposits and on their effects on engine performance.

The DW-10C test protocol involves executing a one-hour test cycle primarily characterized by high speed and load conditions. The one-hour test cycle is repeated 6 times followed by a 4-hour cold soak. This sequence is repeated for total of 5 times with cold starts rated in between the phases. Test results are based on a complex rating system rated from 1 to 10 based on demerits. The demerits are combination of 6 different parameters with 2 parameters associated with cold starts and 4 parameters associated with operability while the engine is running. A demerit rating of 10 indicates excellent IDID protection while lower value indicates issues related to IDID formation.

Reported results should be within the stated reproducibility of the test procedure.

4.2.1.2.2 Base Fuel. For the CEC F-110-16 23(DW10C) test method, the test fuel shall conform to the specifications as per the code RF-79-07 + 0.5 ppm Na (in form of Sodium Naphthenate) + 10 mg/kg DDSA as defined in the test procedure.

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4.2.1.3 Demonstration of Performance. DW10C test results are based on the demerit ratings described in the test procedure. The reference test without detergent additive must have a demerit rating of ≤ 7.5 while the test with detergent additive must show a ≥ 9 merit rating.

4.2.1.4 Alternate Approaches for IDID Qualification.

An alternative approach to obtaining approval for IDID performance involves presenting test data that demonstrates the effectiveness of the detergent additive. This can include results from heavy-duty engine tests or modified DW-10B tests conducted with elevated levels of contaminants known to induce IDID. The key is to show that, in the presence of the detergent additive, deposit formation is either prevented or significantly reduced compared to scenarios without the additive. Additionally, similar data from vehicle or fleet tests illustrating the prevention of deposit formations through the use of detergent additives can be used as an alternate approach for additive qualification.

If an alternative approach is pursued for IDID approval, please note that the final decision will be made after a thorough review of the data by the OEM Sponsors. Fuel additive manufacturers or fuel retailers are encouraged to submit any additional performance data beyond the preferred DW-10C test for IDID qualification.

If the fuel marketer aims to surpass the minimum keep-clean performance requirements and wants to offer fuel capable of cleaning existing deposits, the following performance standard is recommended: DW10C test results are based on the demerit ratings described in the test procedure. The reference test without detergent additive must have a demerit rating of ≤ 7.5 . Subsequently, the same test setup should be used to evaluate the test with “enhanced” clean up detergent additive and must show a ≥ 9 merit rating.

4.2.2 Dispenser Filters. Diesel dispenser filters are the 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 contamination (particulates and water.) Generally, diesel retail dispenser filters feature elements between a 10-to-30-micron rating. A finer size dispenser filter will reduce vehicle concerns related to this contamination, especially reduced vehicle fuel system filter plugging over the filter service interval.

4.2.2.1 Test Method.

The filtration efficiency should adhere to the multi-pass test protocol outlined in ISO 16889, which describes a method for assessing contaminant capacity, particulate removal, and the characteristics of differential pressure. This method involves conducting a test utilizing ISO medium test dust contaminants and a specified test fluid. For water absorption capacity testing, there is no official test due to a variety of applications and conditions in the field.

4.2.2.2 Demonstration of Performance (Particulate Filter).

It is recommended that all dispenser stations—whether low-speed pumps with fuel flow below 15 gallons per minute or high-flow pumps exceeding 15 gallons per minute—are outfitted with filters featuring a pore size of 10 microns or smaller. These filters should exhibit a filtration efficiency surpassing 70% in removing 10-micron size particles, as demonstrated by the ISO 16889 procedure. Given that seasonal variations and the use of biodiesel may affect fuel flow through dispenser filters, it may be permissible to utilize filters with a minimum pore size of 30 microns and 50% efficiency.

Recommendations for options on diesel dispenser filters that meet TOP TIER™ Diesel requirements can be found through the following supplier links.

<https://www.cim-tek.com/products/petroleum/fuel-dispenser-filters>

<https://www.donaldson.com/en-us/engine/filters/products/bulk-fluids/filter-kits-carts/diesel-kits/>

<https://petroclear.com/index.php>

4.2.2.2.1 Particulate Monitoring

Demonstration of Performance. As required by the workmanship requirements of ASTM D975 or ASTM D7467, diesel fuel shall be visually free of undissolved water, sediment, and suspended matter. Typically, this evaluation is done using Test Method D4176, Procedure 2 To comply with the workmanship requirement fuel is expected to have less water than the ASTM D975 or ASTM D7467 standard requirement for “Water and Sediment” of less than 0.05 % by volume determined using Test Method D2709.

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There are no additional requirements for conformance to TOP TIER™ Diesel Particulate contamination requirement. For Audit purposes only, ISO 4406 test standard may be used for particulate monitoring and comparison against the cleanliness requirements of ASTM D975 or ASTM D7467. ISO 4406 standard defines three code numbers corresponding to the numbers of particles of size greater than 4, 6 and 14 microns (μm) per milliliter, respectively. The result will show the number of particles in 100mL of fluid that is specified against each number in the range.

4.2.2.3 Water Monitoring.

Fuel distribution and dispenser stations shall maintain some level of water contamination control to meet the cleanliness standards set by ASTM D975 or ASTM D7467. Filters should have a water absorption capacity or a separate water absorption unit, which detects excess water and alerts the operator by restricting flow. Alternatively, fuel dispensing sites may use water monitoring technology with a robust process to manage and control excess water in the fuel tanks. Sites using such technology should review their processes and quality procedures with the TOP TIER™ Diesel Fuel program to obtain written approval.

Demonstration of Performance. The expectation is that the diesel/biodiesel blend fuel should appear visually clear of any undissolved water, sediment, or suspended particles as expected in the ASTM D975/D7467 standards. Total water content in diesel fuel shall be measured by the ASTM D6304 test procedure. For the TOP TIER™ diesel program, it is desired that the water content be less than 200 mg/kg at 25°C as measured by ASTM D6304.

4.2.3 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 no greater than 460 microns. Fuel with lubricity exceeding 460 microns can potentially adversely affect the lifetime of some of the fuel lubricated injection system components.

4.2.3.1 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.

4.2.3.2 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. If the TOP TIER™ fuel contains more than 2% biodiesel, there is no need to add a lubricity additive. Otherwise, a lubricity additive is necessary to meet the requirement of less than 460 microns.

4.2.4 Fuel Stability: Biofuels such as fatty acid esters, commonly called “biodiesel,” are widely 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) and later intended for use in diesel blends at concentrations of up to 20 volume percent (B20). There are two additional standards that define the quality of the biodiesel blended fuels; ASTM D975, which applies to diesel blends up to B5, and ASTM D7467, which applies to diesel blends from B6 to B20.

When compared to hydrocarbon diesel, biodiesel tends to have a lower oxidative stability period. This can pose challenges when fuel is stored for an extended duration. An oxidative stability additive will enhance the stability of biodiesel and blended fuels and prevent degradation.

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

In the United States, the ASTM D6751 standard for biodiesel (B100) has a stability requirement as measured by a Rancimat induction period of 3 hours minimum. There are no stability requirements in ASTM D975, which allows up to B5. And the stability requirement for B6 to B20 blends per ASTM D7467 is a minimum of 6 hours.

4.2.4.2 Demonstration of Performance: The fuel should have following stability properties to meet the TOP TIER™ diesel performance:

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1. If documentation can confirm that Biodiesel (B100) for making the blends exhibits an induction period of at least 8 hours, as determined by the EN15751 Rancimat test method, then the blends are eligible to fulfill the stability criteria for TOP TIER™ Diesel fuel.

In the US and Canada, the Clean Fuels Alliance of America, the trade association for the biodiesel industry, administers a quality certification program known as BQ-9000®. Certification is granted after a successful formal review and audit of an applicant's capacity and commitment to produce or market biodiesel fuel that meets the ASTM D6751 specification for Biodiesel Fuel (B100) Blend Stock. The quality system includes documentation requirements for stability testing using the Rancimat IP method. It is estimated that over 90% of the biodiesel volume produced in the US is BQ-9000® certified. This certification and its associated analysis certificate can be used to demonstrate stability requirements for the TOP TIER™ Diesel program. The analysis should show that the B100 fuel has an induction period of at least 8 hours, as determined by the EN15751 Rancimat test method.

2. An alternative approach is to add an oxidative stability additive directly to biodiesel blends (B6-B20) as per the qualification process described below. It is to be noted that nearly all the marketplace biodiesel blends have stability additive and TOP TIER™ additive package will provide additional boost to ensure adequate protection.

The qualification process involves obtaining TOP TIER™ – B20 test fuel (1 gallon) along with the TOP TIER™ DIESEL reference stability additive. The TOP TIER™ – B20 test fuel is a blend containing 20% soy-based biodiesel without any stability additives and 80% of EPA Tier 2 certification diesel test fuel while , the reference stability additive represents a well-established antioxidant stability chemistry representative of mainstream stability additives.

To assess stability performance, two test fuels must be prepared. One consists of TOP TIER™ – B20 test fuel blended with the TOP TIER™ DIESEL reference stability additive at the treat rate of 10 ppmv. The other comprises TOP TIER™ – B20 test fuel combined with the candidate stability additive at the desired treat rate. Both test fuels should undergo Rancimat IP testing within the same week or sooner, with the Rancimat IP value reported in hours.

For satisfactory performance, the Rancimat IP value of the B20 test fuel with the candidate stability additive at the desired treat rate must either match or exceed that of the B20 test fuel with the reference stability additive. If the value falls short, adjustments to the treat rate of the candidate additive are necessary to align its performance with that of the reference stability.

TOP TIER™ – B20 test fuel and the TOP TIER™ DIESEL reference stability additive should be obtained from: Gage Products Company, 821 Wanda St, Ferndale, MI 48220. Please reach out to 1fuelsales@gageproducts.com to purchase.

3. For standard ultra-low sulfur diesel fuel devoid of biodiesel and compliant with the minimum ASTM D975 standard, TOP TIER™ Diesel does not require additional stability specifications. This is because there have been no significant stability issues detected in the market over the past decade with use of this fuel.
4. For the biodiesel blends up to B5 in the conventional ultra-low sulfur diesel fuel and meeting minimum of ASTM D975 standard, it is expected that biodiesel used to make the blend as per the latest ASTM D6751 specifications. ASTM D6751 requires neat biodiesel to have a minimum stability value of 3 hours on Rancimat IP test and this value is deemed to be adequate for stability protection for lower levels of biodiesel blends up to B5.

4.3 Renewable Diesel (RD): Paraffinic renewable diesel is stable and is distributed through a dedicated network, unlike conventional diesel, which helps avoid contaminants associated with internal deposit formation. Consequently, it has minimal concerns regarding deposit formation. Therefore, there are no separate injector deposit control requirements for neat or very high blends of paraffinic renewable diesel with conventional diesel (– >

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R95 through RD99) if it meets the ASTM D975 standard and does not contain any biodiesel. Any blends of renewable diesel less than 95 volume percent or any blends that contains biodiesel > 0.2 volume percent should contain deposit control additives to meet the requirements of the program.

4.4 No-Harm Testing Requirements. The requirements for no-harm testing should demonstrate suitable interaction of the candidate detergent, deposit control additive package with widely used marketplace diesel fuel blends, i.e., conventional D02, biodiesel (FAME) blends, and renewable diesel blends (\leq RD80). If the TOP TIER™ additive package is expected to be used in either conventional diesel, renewable diesel blends, or in biodiesel blends then testing of additive is required in biodiesel blended fuel with the highest anticipated biodiesel content for the sales market of interest, which is typically at B20 level.

No-harm compatibility testing can take significant resources, so it is recommended that the test plan be reviewed by the TOP TIER™ coordinator before conducting the tests. The tests outlined below for assessing no-harm effects should be considered as the baseline criteria for additive qualification. Additional data for fuel treated with the additive, including assessments of engine oil compatibility, obtained through vehicle demonstrations, field trials, or engine dynamometer tests, would offer valuable evidence confirming the consistent performance of the fuel additive package.

4.4.1 Demonstration of Performance. The additive package will have to pass the 'no-harms' testing listed in Table 1 when blended with retail pump fuel meeting the ASTM D975 for conventional diesel or ASTM D7467 for biodiesel blend specifications. Each of the deposit control additive formulation must be evaluated in market representative fuel mixed at three times the recommended concentration of the additives in the reference fuel. Compatibility testing is necessary at higher treatment rates beyond the recommended levels to guarantee that no problems arise in case of accidental additive overuse in the fuel.

Note: Testing for cold flow and corrosion impacts as outlined in Table 1 necessitates assessments both in the base reference fuel and with additized fuel to discern the relative effects of incorporating additives into the fuel blend.

Table 1: Tests for Fuel Compatibility

**Testing in biodiesel blends should be conducted with the highest anticipated FAME content for the sales market of interest.*

Properties	Standard	Unit	Pass/Fail Criteria
Fuel Contamination by Additive Precipitation	ASTM D6217	mg/L	\leq 20
	DIN EN 12662	mg/kg	\leq 24
Cold Soak Filterability	ASTM D7501	Seconds	< 360
Gum Forming Potential	ASTM D2274, ISO 12205, ASTM D7462 (for > B6 fuels)	mg/L	< 25
Carbon Residue (on 10% Distillation Residue)	ASTM D524, ISO 10370	wt.%	< 0.35
Cloud Point	ASTM D2500 or equivalent	°C	No change relative to reference fuel unless additive used to effect cold properties of fuel ^{Note 1}
Cold Filter Plugging Point	ASTM D6371, DIN EN 116	°C	No change relative to reference fuel unless additive used to effect cold properties of fuel ^{Note 1}

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Copper Strip Corrosion (3 h at 50 °C)	ASTM D130, 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
Flash Point	ASTM D93, ISO 2719	°C	> 55 (Summer Grade) > 38 (Winter Grade)
Water Interfacial Tension	ASTM D971	Dynes/cm	Report

Note 1: Values within the precision of the described test methods will be acceptable as no change.

5. Process to Attain TOP TIER™ Approved 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 with 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 2025.

6.2 Revision Control.

<i>Revision</i>	<i>Date</i>	<i>Description</i>	<i>Approver</i>
A	January 2025	Initial Release	V. Reilly