

TOP TIER™ Approved GASOLINE DEPOSIT CONTROL PERFORMANCE STANDARD Revision G, January 2025

1. Scope

1.1 Performance Description. This document describes the TOP TIER™ Deposit Control Performance Standard of an unleaded gasoline that minimizes carbon deposits on fuel injectors, intake valves, and combustion chambers of all spark-ignited internal combustion engine technologies. This newest update to the Performance Standard (Rev. G) adds engine test requirements designed to specifically protect gasoline direct injected (GDI) vehicle engines from fuel injector deposits.

2. References

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

2.1 ASTM International (American Society for Testing and Materials)

D86 D381 D1319 D2622 D4806 D4814 D4815 D5453 D5580 D5845 D6201 D6550 D6729.

2.2 California Air Resources Board (CARB)

Advisory letter (April 19, 2001).

Test Method for Evaluating Intake Valve and Combustion Chamber Deposits in Vehicle Engines (March 12, 1999).

2.3 Coordinating European Council (CEC)

CEC F-16- 96, Assessment of the Inlet Valve Sticking Tendency of Gasoline Fuels

2.4 Code of Federal Regulations (CFR)

CFR 1090; 40 Part 79 Registration of Fuels and Fuel Additives and 40 CFR Part 80 Regulation of Fuels and Fuel Additives.

The requirements for the registration of fuel and fuel additives under 42 U.S.C. 7545(a), (b), and (e) are specified in 40 CFR part 79. A party that must meet the requirements of this part (CFR 1090) may also need to comply with the requirements for the registration of fuel and fuel additives under 40 CFR part 79.

2.5 International Organization for Standardization (ISO)

ISO 17025, General Requirements for the Competence of Testing and Calibration Laboratories

2.6 Southwest Research Institute (SwRI)

Intake Valve Sticking Test in GM 5.0L V-8

2.7 General References

a. Worldwide Fuel Charter, Sixth Edition, 2019 (https://www.autosinnovate.org/energy-environment/fuel-publications/Fuel%20Publications-WWFC-19_GASOLINE-15.10.2019-graphist-1.pdf)

b. Technical Committee of Petroleum Additive Manufacturers in Europe (<https://www.atc-europe.org/public/Doc113%202013-11-20.pdf>)

c. AAA encouraged drivers to prioritize TOP TIER™ Approved Gasoline when filling up their tank. (<https://www.aaa.com/autorepair/articles/not-all-gasoline-is-created-equal>)

3. Definitions

3.1 “Independent Laboratory” - a mechanical and/or chemical testing organization which is accredited by a national or international accreditation agency such as the American Association for Laboratory Accreditation (A2LA) for testing competence in mechanical and chemical testing and / or ISO 17025 “General Requirements for the Competence of Testing and Calibration Laboratories”. Additionally, the Independent Laboratory cannot be affiliated with a TOP TIER Licensee, a fuel marketer or the manufacturer of any additive package approved for use in TOP TIER™ Approved Gasoline. Other accreditations may be accepted upon review with GM engineering.

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3.2 “Minimum Treat Rate” – the lowest dose or concentration (PTB and ppmv) of the complete additive package as tested with defined test fuels which meet the requirements of this TOP TIER™ program performance specification.

3.3 “Approved Additive Treat Rate Range” shall be defined as the additive concentration range between the “Minimum Treat Rate” and 3 x the “Minimum Treat Rate.”

4. Standards

4.1 Retail Gasoline Performance Standards. The deposit control performance of an additive package meeting the requirements of section 4 of this document shall be included in all octane grades of gasoline sold by a fuel marketer and in all marketing areas of the country of sales interest. In addition, conformance to the standards shall mean gasoline sold in the country shall not contain metallic based additives, including methylcyclopentadienyl manganese tricarbonyl (MMT), ferrocene, and/or any other organo-metallic compounds.

4.2 Deposit Control Additive Requirements. The deposit control additive used to meet the performance Standards described in 4.3 shall meet the substantially similar definition under Section 211(f) of the U.S. Federal Clean Air Act. Also, the additive shall be certified to have met the minimum deposit control requirements established by the U.S. Environmental Protection Agency (EPA) in 40 CFR Part 80. Lastly, the additive shall be registered with the EPA in accordance with 40 CFR Part 79.

4.3 Deposit Control Performance Standard. All performance testing and fuel composition analysis intended to be submitted as part of a TOP TIER™ Approved Fuels application shall be conducted by an Independent Laboratory. The deposit control performance of a gasoline additive package shall be demonstrated using the tests shown below:

4.3.1 Intake Valve Keep Clean Initial Performance Standard

4.3.1.1 Test Method. Intake valve deposit (IVD) keep clean performance shall be demonstrated using ASTM D6201, *Standard Test Method for Dynamometer Evaluation of Unleaded Spark-Ignition Engine Fuel for Intake Valve Deposit Formation*. Tests demonstrating base fuel minimum deposit level (4.3.1.2) and additive performance (4.3.1.3) shall be conducted using the same engine block and cylinder head. All results shall be derived from operationally valid tests in accordance with the test validation criteria of ASTM D6201. IVD results shall be reported for individual valves and as an average of all valves.

4.3.1.2 Base Fuel. The base fuel shall conform to ASTM D4814 and shall contain commercial fuel grade ethanol conforming to ASTM D4806. All gasoline blend stocks used to formulate the base fuel shall be representative of normal territory refinery operations and shall be derived from conversion units downstream of distillation. Butanes and pentanes are allowed for vapor pressure adjustment. The use of chemical streams to accelerate deposit build-up are prohibited unless approved by GM engineering in advance of engine testing. The base fuel shall have the following specific properties after the addition of ethanol:

1. Contain enough denatured ethanol such that the ethanol content is no less than 8.0 and no more than 10.0 volume percent as measured by ASTM D 4815 or D 5845. In markets with lower fuel ethanol content, fuel matching the market conditions of fuel ethanol content can be used up on approval.
2. Contain no less than 8 volume percent olefins as measured by ASTM D 1319 or D 6729.
3. Contain no less than 15 volume percent aromatics as measured by ASTM D 1319 or D 6729.
4. Contain no more than 80 mg/kg sulfur as measured by ASTM D 2622 or D 5453.
5. Produce a 90% evaporated distillation temperature no less than 290°F as measured by ASTM D86.
6. Produce IVD no less than 500 mg averaged over all intake valves.
7. A Certificate of Analysis showing both the detailed test fuel composition results and source should accompany the additive results package. This certificate should also contain the unwashed and washed gum level of the base fuel according to ASTM D381.

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4.3.1.3 Demonstration of Performance. The base fuel from 4.3.1.2 shall contain enough deposit control additive such that IVD is no more than 50 mg averaged over all intake valves. This amount of deposit control additive shall be the Minimum Treat Rate. Results for individual valves and an average shall be reported. The unwashed gum level of the fuel containing deposit control additive shall be determined according to ASTM D 381 and reported.

4.3.2 Combustion Chamber Deposit Initial Performance Standard

4.3.2.1 Test Method. Combustion chamber deposits (CCD) shall be collected and weighed along with IVD using ASTM D 6201, *Standard Test Method for Dynamometer Evaluation of Unleaded Spark-Ignition Engine Fuel for Intake Valve Deposit Formation*. ASTM D 6201 does not contain a procedure for collecting and measuring CCD. Adapting a scrape and weigh procedure developed by CARB is recommended (see referenced test method dated March 12, 1999). Results for individual cylinders and an average shall be reported.

4.3.2.2 Base Fuel. Combustion chamber deposits shall be measured for the base fuel from 4.3.1.2.

4.3.2.3 Demonstration of Performance. The base fuel from 4.3.1.2 treated with additive at the concentration meeting the standard found in 4.3.1.3 shall not result in more than 140% of the average CCD weight for the base fuel without additive.

4.3.3 Intake Valve Sticking Initial Performance Standard

4.3.3.1 Test Method. Intake valve sticking tendency shall be determined using either the 1.9 L Volkswagen engine (Wasserboxer according to CEC F-16-T-96) or the 5.0 L 1990-95 General Motors V-8 engine (SWRI IVS test). Two options are available for demonstrating intake valve sticking tendency.

4.3.3.2 Option 1. The valve-sticking tendency of the test fuel by itself will not have to be demonstrated prior to testing the candidate additive. The following shall be required of all tests:

1. Test fuel shall be either the same as in 4.3.1.2 or CEC valve sticking reference fuel.
2. Concentration of deposit control additive in the test fuel shall be at three times the amount determined in 4.3.1.3.
3. Test temperature shall be -20°C.
4. Three 16-hr cold soak cycles, each followed by a compression pressure check, shall constitute a complete test.

4.3.3.2.1 Demonstration of Performance. A pass shall result in no stuck valves during any of the three cold starts. A stuck valve is defined as one in which the cylinder pressure is less than 80% of the normal average cylinder compression pressure.

4.3.3.3 Option 2. The valve-sticking tendency of the test fuel together with an additive known to cause valve sticking shall be demonstrated prior to testing the candidate additive. The following shall be required of all tests:

1. Test fuel shall be either the same as in 4.3.1.2 or CEC valve sticking test reference fuel.
2. An additive known to cause valve sticking shall be selected, and, when blended into test fuel, shall demonstrate valve sticking tendency as follows: (a) for the Volkswagen engine, at least two valves shall be stuck; (b) for the GM engine, at least three valves shall be stuck.
3. Tests demonstrating performance of the candidate additive shall be conducted at a concentration that is at least three times the amount determined in 4.3.1.3
4. Test temperature shall be -20°C.
5. One 16-hr cold soak cycle followed by a compression pressure check shall constitute a complete test.

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4.3.4 Gasoline Direct Injection (GDI) Engine, Fuel Injector Cleanliness Performance Standard

4.3.4.1 Test Method. GDI engine **Fuel Injector Cleanliness Performance** shall be demonstrated using the TOP TIER™ GDI Fuel Injector Deposit Test Method. There are two primary metrics for this test. The first is a **target** of 5 – 10% of the engine control module's (ECM) Long Term Fuel Trim (LTFT) shift. The second is a **requirement** of the ECMs "injector pulse-width (IPW)" closed-loop, fuel control parameter greater than 5%. Engine tests demonstrating the base fuel's dirty-up performance of the fuel injector and subsequent additized clean-up performance shall be conducted using the same engine block and cylinder head run in a continuous 120-hour test. The test method allows for periodic engine shutdowns and details documenting the root cause(s). The resultant delta (percent change) IPW fuel injector plugging results are reported from the two separate phases of the test; the delta IPW from zero engine hours (new fuel injectors) to 60 hours of engine dyno operation (dirty fuel injectors), and then again from 61 to 120 hours of operation (additive cleaned fuel injectors). All results shall be derived from operationally valid tests in accordance with the test validation criteria of the TOP TIER™ GDI Fuel Injector Deposit Test Method.

4.3.4.2 Base Fuel. Testing must be conducted using Haltermann Test Fuel Solutions Product No. HF2208 Gasoline for LHU Injector Deposit Test. This test fuel has a specific formulation proven to cause deposits on the inside of the injector and fuel spray holes and therefore should only be sourced from Haltermann Test Fuel Solutions.

4.3.4.3 Demonstration of Performance. Acceptable demonstration of performance occurs when the base fuel from 4.3.4.2 causes a target of 5 – 10% LTFT shift and a result in IPW shift greater than 5% over the course of 60 hours and then the base fuel plus candidate additive removes injector deposits resulting in at least 50% reduction in the delta LTFT shift and delta IPW relative to the first 60 hour dirty-up phase.

Submit the following test data for review; a copy of the engine test lab report including LTFT and IPW results during each phase of the test as well as the end of test (EOT) injector face pictures and CRC ratings, a copy of the base fuel Certificate of Analysis (CofA) showing purchase date, batch ID, and composition results. The test report pictures of each fuel injector at the end of test (EOT) should be similar in form, style, and quality as the one shown in Figure 1.

Figure 1: Fuel Injector Face Image at EOT



4.4 No-Harm Testing Requirements

4.4.1 Stochastic Pre-ignition (SPI) No-Harm Demonstration

4.4.1.1 Test Method GDI engine **SPI No-Harm Performance** shall be demonstrated using the TOP TIER™ GDI Stochastic Preignition (SPI) Test Method. The primary metric for this test is the SPI frequency or count of abnormal combustion cycles measured using in-cylinder pressure transducers, the defined signal conditioning and counting

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software, and derived in engineering units of “problems (SPI events) per million” combustion cycles, or PPM. This test will measure SPI events during three repeat tests between the base fuel response and the additive plus base fuel response. All results shall be derived from operationally valid tests in accordance with the test validation criteria of the TOP TIER™ GDI Stochastic Preignition (SPI) Test Method.

4.4.1.2 Test Fuel – Testing must be conducted using Gage Products Test Fuel TTG-1-002 TOP TIER™ SPI Test Fuel. This Test Fuel has a highly controlled fuel composition from batch to batch yielding tight control of its particulate matter index (PMI) and therefore should only be sourced from Gage Products in Ferndale, Michigan.

4.4.1.3 Test Motor Oil - Testing must be conducted using the reference oil specified in the TOP TIER™ GDI Stochastic Preignition (SPI) Test Method.

4.4.1.4 Demonstration of Performance Acceptable demonstration of performance occurs when the SPI PPM frequency from the combined 3 base fuel plus additive phases of the test are measured to be statistically equivalent to or better than (lower) the 3 base fuel phases of the test, at a statistical p-value level of 0.05.

Submit the following test data for review; a copy of the engine test lab report including SPI results during each phase of the test as well as the fuel dilution in the motor oil results during each phase, copies of both the base fuel Certificate of Analysis (CofA) and motor oil CofA showing purchase dates, batch IDs, and composition results.

4.4.2 No-Harm Particulate Emissions Requirements

4.4.2.1 Test Method GDI engine Particulate Emissions No-Harm Performance shall be demonstrated at the same time as running the TOP TIER™ GDI Fuel Injector Deposit Test Method. For the particulate emissions no-harm demonstration, the primary metric is the exhaust soot (elementary carbon) concentration (mg/m^3) measured over the full 120 operating hours of the TOP TIER™ GDI Fuel Injector Deposit Test Method. All results shall be derived from operationally valid tests in accordance with the test validation criteria of the TOP TIER™ GDI Fuel Injector Deposit Test Method.

The TOP TIER™ GDI Fuel Injector Deposit Test Method specifically references and utilizes the AVL Micro Soot Sensor, AVL sample conditioning equipment, and the AVL 483 sampling probe to measure exhaust soot. Other instrument manufacturer’s soot measurement equipment can be used to generate the soot measurements during the Fuel Injector Deposit test, however sufficient correlation data to the AVL Micro Soot data will need to be submitted to demonstrate compliant performance. It is recommended this be done in advance of any non-AVL Micro Soot generated particulate emissions additive demonstration testing.

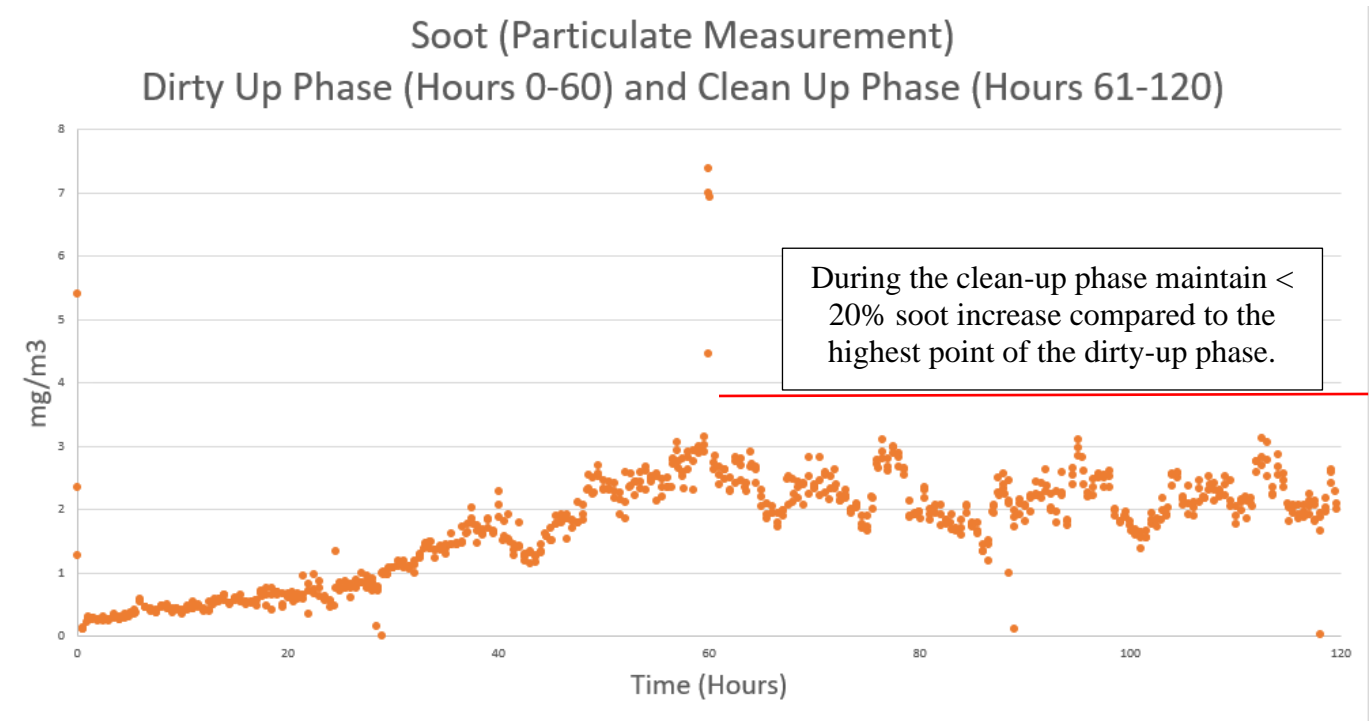
4.4.2.2 Base Fuel Both phases of Soot Testing (Base Fuel and Base Fuel plus candidate Additive Package) must be conducted using Haltermann Test Fuel Solutions Product No. HF2208 Gasoline.

4.4.2.3 Demonstration of Performance Soot emissions performance interpretation will be done using a 1-minute averaged emissions data chart as shown in Figure 2 (below). Typically, acceptable demonstration of performance occurs by maintaining the soot emissions concentration for the full 60-hour Clean-up portion of the test lower than the highest point of the 60 hour Dirty-up phase of the test. However, because of soot emissions measurement variation, it is necessary to offer some compliance flexibility while interpreting the acceptable emissions performance. Note the sporadic, very high soot points such as those on the far-left side Figure 2 and at the 60-hour mark are caused by operating condition transitions and would not be included in the performance interpretation. Also, as long as the soot emissions of the Clean-up phase are lower at the end of the 60 hours than the Dirty-up phase, a 20% margin of soot increase is acceptable in the earlier part of the Clean-up phase. The intent of this no-harm test is to assure that no increased soot emissions are being created by the detergent additives themselves as they work to clean other parts of the fuel injector.

Submit the following test data for review; a copy of the engine test lab report including emissions soot results averaged at a 1-minute frequency during each phase of the TOP TIER™ GDI Fuel Injector Deposit Test Method.

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Figure 2: Example of 1 minute avg. Soot Emissions Chart with Acceptance Limit (red line).



4.4.3 No-Harm Gasoline Chemistry Performance Gasoline detergent additive packages are added to a base gasoline at concentrations generally equal to or less than a few hundred parts per million by mass (ppm). And although these additive packages represent a minor concentration of the gasoline blend, they can have large benefits to the vehicle’s engine performance over time because of their unique chemical characteristics relative to gasoline. It’s therefore important to ensure that they are fully miscible with marketplace base gasolines.

4.4.3.1 Test Method(s) – as outlined below in Table 1.

4.4.3.2 Base Fuel – as outlined below in Table 1.

4.4.3.3 Demonstration of Performance – as outlined below in Table 1.

Table 1: No-Harm Gasoline Chemistry Testing Requirements

**There is no pass/fail criteria for D381 Unwashed Gums. Report your 1X value or TOP TIER value. (The value relating to the amount of additive needed to pass the performance standard limits.)*

| Properties | Test Method | Engineering Units | Reference Fuel | Pass/Fail Criteria |
|---|-------------|-------------------|--|--|
| Existent Gum Content – Unwashed Gum (Basefuel) | ASTM D381 | mg/100 ml | SPI 4.4.1.2 Base Fuel | Report |
| Existent Gum Content – Washed Gum (Basefuel) | ASTM D381 | mg/100 ml | SPI 4.4.1.2 Base Fuel | Report |
| Existent Gum Content – Unwashed Gum (Additized) | ASTM D381 | mg/100 ml | SPI 4.4.1.2 Base Fuel + Additive Package | Report |
| Existent Gum Content – Washed Gum (Additized) | ASTM D381 | mg/100 ml | SPI 4.4.1.2 Base Fuel + Additive Package | Report |
| Additive Package Miscibility – Fuel temperature | ASTM D4176 | | SPI 4.4.1.2 Base Fuel + Additive Package | Clear & Bright Tested: 70 – 75 F Tested: < 25 F |

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|---|------------|--|---|---|
| Additive Package Miscibility – Ethanol Content | ASTM D4176 | | SPI 4.4.1.2 Base Fuel + 5.0 V% Ethanol + Additive Package | Clear & Bright Tested: 70 – 75 F Tested: < 25 F |
|---|------------|--|---|---|

5. Process to Register Additive Packages for use in Licensed TOP TIER™ Approved Gasoline

5.1 Submission of Test Results. An additive or fuel company desiring to register an additive package for use in licensed TOP TIER™ Approved Gasoline products 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
Email: TopTier@CenterForQA.com

5.2 Notification of receipt. The Test Results shall be reviewed by the TOP TIER™ Approved Fuels program. If approved, the additive company will be notified that the additive package will be registered for use in licensed TOP TIER™ Approved Gasoline products. Such notification does not allow the use of the TOP TIER™ trademarks or logos.

6. Release, Effective Date, and Revisions

6.1 Release. This document was first released in April 2004.

6.2 Revision Control.

| <i>Revision</i> | <i>Date</i> | <i>Description</i> | <i>Approver</i> |
|-----------------|--------------|---|-----------------|
| G | January 2025 | Extensive revisions were made to include GDI fuel injector cleanliness requirements, as well as Soot and Stochastic Pre-ignition (SPI) no harms testing. | V. Reilly |
| F | October 2019 | 4.1 An approved additive package at the correct treat rate must be used at all grades of gasoline at a station vs. all gasoline must meet the standard. 4.3.1.2 Minimum aromatics changed to 15% from 28% as it is difficult to obtain fuel with over 28% aromatics. 4.3.3.2 Valve sticking test concentration changed from 2 times to 3 times to align with allowable treat rate range. 5 Updated languages to reflect the process to obtain TOP TIER approval. Added additive company as a submitter and also removed reference to licensing- which is handled separately. | |

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|---|---------------|---|--|
| E | March 2017 | <p>3.1 Modified from A2LA to Independent Laboratory with further definition to accommodate international laboratory accreditation.</p> <p>4.3.1.2 Base Fuel: ethanol changed to 8-10% to align with CARB and to market ethanol outside US.</p> | |
| D | May 2016 | <p>Changed contact to CQA or submission of results.</p> <p>Changed reference in option 1 valve-sticking to 4.3.1.3 (TOP TIER Concentration) from Injector Fouling concentration reference.</p> | |
| C | February 2015 | <p>Removed Fuel Injector Fouling Initial Performance Standard requirement.</p> <p>Changed ethanol limit from 8.0-10.0% up to 10% ± 1%.</p> <p>Changed sulfur to a maximum limit of 80 mg/kg.</p> <p>Removed base fuel requirement for proof of 75% olefin and 60% sulfur content from FCC streams.</p> <p>Added requirement to provide full base fuel composition analytical results and gum values</p> | |
| B | May 2008 | Changed minimum sulfur limit to 24 mg/kg. | |
| A | March 2008 | Modified document to reflect new license agreement; removed attestation forms. | |