EPAct/V2/E-89: Assessing the Effect of Five Gasoline Properties on Exhaust Emissions from Light-Duty Vehicles Certified to Tier 2 Standards

Final Report on Program Design and Data Collection

United States Environmental Protection Agency

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Assessment and Standards Division
Office of Transportation and Air Quality
U.S. Environmental Protection Agency
and
National Renewable Energy Laboratory
U.S. Department of Energy
and
Coordinating Research Council

NOTICE

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United States Environmental Protection Agency

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APPENDICES

Abbreviations Used in this Report

API	American Petroleum Institute
CARB	California Air Resources Board (also ARB)
CFR	Code of federal regulations
CH_4	Methane
CO	Carbon monoxide
CO_2	Carbon dioxide
CRC	Coordinating Research Council
EPA	US Environmental Protection Agency
DOE	US Department of Energy
DNPH	Dinitrophenylhydrazine
DVPE	Dry vapor pressure equivalent (equivalent to RVP)
E0	Gasoline containing no ethanol
E10	Gasoline blend containing 10 vol% ethanol
FBP	Final boiling point
FID	Flame ionization detector
FTP	Federal test procedure
HPLC	High performance liquid chromatograph
MON	Motor octane number
MSAT	Mobile Source Air Toxics
NMHC	Non-methane hydrocarbons
NMOG	Non-methane organic gases
NO_x	Nitrogen oxides
NREL	National Renewable Energy Laboratory
OBD	Onboard diagnostics
(R+M)/2	Average of RON and MON
RON	Research octane number
RVP	Reid vapor pressure
SwRI	Southwest Research Institute
T50	Temperature at which 50 vol% of a fuel has been evaporated (°F)
T90	Temperature at which 90 vol% of a fuel has been evaporated (°F)
THC	Total hydrocarbons

I. EXECUTIVE SUMMARY

This report describes program design and data collection in the EPAct/V2/E-89 light duty gasoline vehicle fuel effects study, which examined the exhaust emission impacts of changes in five fuel properties (ethanol, T50, T90, aromatics, and RVP (specified as DVPE)) over a range covering current market fuels and potential mid-level ethanol blends. Testing was performed by Southwest Research Institute, and program sponsors were the U.S. Environmental Protection Agency, U.S. Department of Energy via the National Renewable Energy Laboratory, the Coordinating Research Council, and the Lubrizol Corporation.

The fuel matrix consisted of 27 fuels arranged according to a partial factorial design optimized for selected interactions of interest, plus an E85 fuel tested on a subset of vehicles. The test vehicle fleet consisted of 15 new light duty cars and trucks of 2008 model year selected from among high sales makes and models to provide a representative sample of the fleet of properly-operating vehicles meeting the U.S. Federal Tier 2 emission standards.[1] Given the relatively low level of emissions from these vehicles, a number of design and procedural steps were undertaken to minimize the impacts of measurement variability and other artifacts on data quality.

Data collected included typical regulated pollutants by bag and second-by-second plus speciated emissions for a subset of tests and bags. Data for 16 relevant onboard diagnostic parameters were also captured for each test on a second-by-second basis. Complete data were generated for 926 tests, with 30 additional tests containing valid measurements for regulated emissions.[2] Sponsors agreed that analysis of the resulting dataset would be performed and published independently.

[1] An additional (16^{th}) vehicle performed testing on the E85 fuel.
[2] Program data is available via the EPA OTAQ website at http://www.epa.gov/otaq/fuelsmodel.htm

II. INTRODUCTION

Prior fuel effects models, such as the EPA Predictive Model and Complex Model,[3] were based on data taken on 1990s-technology vehicles meeting the Tier 0 and Tier 1 emission standards, levels an order of magnitude higher than current (Tier 2-compliant) vehicles. With the fleet turning over to much lower-emitting vehicles, the Agency and stakeholders were interested in generating a coherent body of updated fuel effects data on which policy could be based going forward. Recognizing this issue, Congress, in Section 1506 of the Energy Policy Act of 2005, directed EPA to produce an updated fuel effects model representing the gasoline vehicle fleet at the time of the study. Carrying out other statutory requirements, such as an anti-backsliding assessment of the effects of increased renewable fuels use on air quality as outlined in Section 209 of the Energy Independence and Security Act of 2007, were also dependent upon updated fuel effects data.

In September 2007, Southwest Research Institute (SwRI) began conducting work in San Antonio, Texas, on a series of tasks and assignments for EPA related to the statutory requirement. By January 2009, SwRI had completed Phases 1 and 2 the EPAct program. These pilot phases, described in a separate document, involved testing of 19 light duty cars and trucks (subsequently referred to as the "EPAct fleet") on three fuels, at two temperatures.[4]

In March 2009, SwRI began work on Phase 3 (the main program), which was jointly supported by EPA, the U.S. Department of Energy (DOE) through the National Renewable Energy Laboratory (NREL), and the Coordinating Research Council (CRC). In addition, Lubrizol Corporation provided material and analytical support. This report covers work conducted for Phase 3, also known as EPAct/V2/E-89, which involved the testing of fifteen of the high-sales Tier 2 compliant vehicles from the EPAct Phase 1 fleet using twenty-seven test fuels covering typical ranges of five fuel parameters. Phase 3 also included testing of the four

[3] See "Technical Support Document: Analysis of California's Request for Waiver of the Reformulated Gasoline Oxygen Content Requirement for California Covered Areas", document number EPA420-R-01-016. See also 40 CFR 80.45.
[4] EPAct program Phases 1-2 are described in a memorandum to the Tier 3 docket EPA-HQ-OAR-2011-0135.

flexible-fuel vehicles (FFVs) from the EPAct fleet on an E85 fuel (three of these vehicles were among the 15-vehicle test fleet). Phase 3 data collection was completed in June 2010.

III. TEST FUEL SPECIFICATION AND BLENDING

A. Design of Fuel Matrix

1. Selection of Fuel Parameters for Investigation

Gasoline is a mixture of hundreds of hydrocarbon species, with boiling points spanning the range of 100-400°F, and molecular size ranging from four to ten carbons. Depending on the source of the parent crude oil and configuration of the refinery producing the gasoline, the proportions of particular chemical species can vary widely. This is acceptable so long as a number of key bulk properties of the mixture remain within a range that ensures acceptable operation of vehicles and other equipment designed to use it. Over the past few decades, EPA and other agencies have also found it useful to regulate some of these properties to control harmful emissions.

While an exhaustive study of effects of gasoline properties on emissions could include dozens of parameters, producing results with an acceptable level of statistical power would require such a large and complex design that it would be impractically lengthy and expensive. To narrow the scope a survey was made of available data for recent technology vehicles, with special attention paid to the expected magnitude of emission changes caused by fuel properties, as well as how important characterization of an effect might be in a regulatory context. Databases used for estimating the magnitude of emission impacts included those associated with the EPA Complex and Predictive models, the California Air Resources Board Predictive Model, the CRC E-67 and E-74b test programs, as well as the 2005-6 MSAT test program conducted by EPA and several automakers. Table III-1 lists the parameters initially considered as candidates for study and summarizes selection criteria related to each.

Table III-1. Fuel parameters considered for inclusion in the study.

Fuel Parameter	Data Availability for Latest Technology Vehicles	Expected Magnitude of Emission Impact	Fuel Policy Relevance
Aromatics	Little	High	High
Ethanol	Some	Uncertain	High
RVP	Some	Uncertain	High
T50	Little	Uncertain	Low
T90	Little	Uncertain	Low
Sulfur	Some	High	High
Olefins	Little	Uncertain	Uncertain
Octane number	Little	Low	Low
Drivability index	Little	Low	Low
Total oxygen	Little	Uncertain	Low
Polyaromatics	Little	Uncertain	Uncertain

Initial assessment of costs and program duration suggested it would not be feasible to include more than five fuel parameters and their interactions. Based on a combination of the criteria shown, the five parameters listed at the top of this table were chosen as the study targets. Two other parameters, sulfur and olefin content, were also of interest; examination of sulfur would require a different program design due to involvement of the exhaust catalyst, and olefins were deferred for study later.[5,6]

2. Selection of Fuel Parameter Ranges and Levels

Fuel parameters may have linear or nonlinear impacts on emissions. To capture a nonlinear impact, three or more treatment levels of a given parameter must be included in the study design. Statistical models of data from prior studies suggested that T50, T90, and ethanol content may have nonlinear impacts on emissions. Based on this information, EPA originally planned to test three levels of ethanol spanning the range of E0-E10 (the limit for legal market fuels at that time). During the design process, DOE (via NREL) offered additional funding to add E15 and E20 fuels to broaden the database to include fuel blends that might be expected

[5] A subsequent study on the effects of olefins using the same 15-vehicle test fleet as this study was published as Maryam Hajbabaei, Georgios Karavalakis, J. Wayne Miller, Mark Villela, Karen Huaying Xu, Thomas D. Durbin, Impact of olefin content on criteria and toxic emissions from modern gasoline vehicles, Fuel, Volume 107, May 2013, Pages 671-679, ISSN 0016-2361, 10.1016/j.fuel.2012.12.031.

[6] A subsequent study on the effects of sulfur content using the same makes and models as this study was published by EPA as document EPA-420-D-13-003, "The Effects of Gasoline Sulfur Level on Emissions from Tier 2 Vehicles in the In-Use Fleet".

appear in the market in the future. After this adjustment, four levels of ethanol were selected at 0%, 10%, 15%, and 20% by volume. Five levels of T50 were chosen to allow detailed characterization of any interaction with ethanol. Finally, to examine potential nonlinear impacts of T90, two fuels were added with an intermediate level of this parameter..[7] The remaining two parameters, aromatic content and RVP (reported in this program as DVPE), had two levels.

The parameter ranges to be covered for T50, T90, aromatic content, and RVP were selected to represent the range of in-use fuels based on a review of the Alliance of Automobile Manufacturers' 2006 North American Fuel Survey. As the emissions tests were to be performed at the nominal temperature of 75°F, summer survey data was used. Since the effect of fuel changes on emissions was expected to be small in comparison to other sources of test-to-test variability, the span of fuel parameter ranges was maximized in order to increase the likelihood of discerning statistically significant results. Test fuel parameter ranges were originally drafted to span roughly the 5^{th} to 95^{th} percentiles of survey results for in U.S. gasoline, though some test fuel parameters were adjusted after the actual blending process began (discussed further in section III.B). An intermediate level of T50 in E0 fuels was selected to coincide with the high level of T50 in E10 fuels. Similarly, an intermediate level of T50 in E10 fuels was selected to coincide with the low level of T50 in E0 fuels.

For E15 and E20 fuels, the T90, aromatic content, and DVPE ranges selected for E0 and E10 fuels were applied. A single level of T50 was selected for E20 blends based on the information obtained from CRC report No. 648 (2006 CRC Hot-Fuel-Handling Program) as well as petroleum industry sources which indicated that it was largely independent of the hydrocarbon fraction of the fuel and would not deviate more than several degrees from 160°F due to the presence of a large fraction of ethanol.[8] At the time this fuel matrix was being designed, no information was available on distillation properties of E15 fuels. Two levels of T50 were

[7] The intermediate level of T90 occurs along one edge of the fuel domain in Phase 3. Statistical analysis of any nonlinear T90 effect was intended to rely on a fuel used in Phase 1 of the program as an additional source of data for the intermediate T90 level.

[8] As ethanol blend level moves beyond 10 vol%, T50 becomes increasingly correlated (inversely) with ethanol content. At E15, the two can be manipulated independently with some effort within a relatively limited range. By E20, the behavior of the center of the distillation curve (where T50 lies) is dominated by ethanol's boiling point, and thus T50 cannot be moved outside a narrow range around 165°F. Thus, T50 and ethanol should only be understood to be independently blended parameters at E10 and below.

selected for the E15 fuels, the low level equal to the lowest T50 assumed for E10 fuels and the high level being a linear interpolation between the highest T50 of E10 fuels and the sole T50 level of E20 fuels. Table III-2 shows the levels and range of parameters chosen during the initial design of the Phase 3 fuel set. Some of these parameters were adjusted during subsequent blending steps (discussed further in section III.B).

Table III-2. Summary of initial Phase 3 test fuel set design.

Fuel Parameter	Number of Levels	Values to Be Tested
Ethanol (vol%)	4	0, 10, 15, 20
T50 (°F)	5	150, 160 (E20 only), 190, 220, 240
T90 (°F)	3	300, 325, 340
Aromatics (vol%)	2	15, 40
RVP (psi)	2	7, 10

3. Statistical Design of the Fuel Matrix

Studies involving multiple levels of multiple variables are ideally conducted in such a way that each parameter is varied independently through all its levels while the others are held constant, thus generating data from all possible combinations of treatment levels (referred to as a factorial design). Given the levels of treatments outlined above, the Phase 3 fuel set would require some 240 fuels to be blended and tested to examine each point of a factorial matrix. This clearly being impractical, use of a partial factorial design was utilized. This arrangement uses a subset of all the possible fuel blends to allow characterization with statistical confidence of the main effects (i.e., the fuel parameters themselves) plus a pre-selected subset of interactions between the main effects.[9] Interactions of interest were chosen based on models from prior studies as well as engineering judgment, and are as follows: ethanol interactions with each of the other fuel parameters, plus T50-squared and ethanol-squared. With the five main effects plus these six interactions, the design was to be optimized for 11 potential model terms.

[9] What is sacrificed in a partial factorial design is the ability to properly characterize all interactions between the study parameters. Interactive effects beyond those for which the design is optimized are confounded with others to varying degrees, and thus conclusions about them can't generally be made with statistical confidence.

Selection of the best subset of fuels for a partial factorial design is a computationally intensive process, for which specialized software has been developed. This work was done by SwRI statisticians Robert Mason and Janet Buckingham. A matrix of 27 fuels as shown in Table III-3 was eventually constructed to maximize statistical performance over the desired range of parameters. The statisticians' report describing the matrix development process is available as Appendix A.[10]

[10] The statisticians' report covers matrix design through several stages of evolution of program objectives and funding levels. The fuel matrix shown in Table 9 of the report was eventually used for Phase 3, with the exception of fuels 17-19, which were tested in earlier phases of the program. The Phase 3 matrix of 27 fuels was intended to stand on its own for modeling purposes, with optional inclusion of fuels 17-19 in the database as "centered" fuels. The development of fuels 17-19 is described in the SwRI EPAct/V2/E-89 Fuel Blending Final Report attached as Appendix B.

Table III-3. Phase 3 fuel matrix resulting from partial factorial design.

Test Fuel Number[a]	T50, °F	T90, °F	Ethanol, vol.%	DVPE, psi	Aromatics, vol.%
1	150	300	10	10	15
2	240	340	0	10	15
3	220	300	10	7	15
4	220	340	10	10	15
5	240	300	0	7	40
6	190	340	10	7	15
7	190	300	0	7	15
8	220	300	0	10	15
9	190	340	0	10	40
10	220	340	10	7	40
11	190	300	10	10	40
12	150	340	10	10	40
13	220	340	0	7	40
14	190	340	0	7	15
15	190	300	0	10	40
16	220	300	10	7	40
20	160	300	20	7	15
21	160	300	20	7	40
22	160	300	20	10	15
23	160	340	20	7	15
24	160	340	20	10	15
25	160	340	20	10	40
26	150	340	15	10	40
27	190	340	15	7	15
28	190	300	15	7	40
30	150	325	10	10	40
31	160	325	20	7	40

[a] Fuels 17-19 were tested in an earlier phase of the program. Fuel 29 is an E85 fuel not included in the statistical matrix design.

4. Test Fuel Specification

In addition to the five fuel properties that were the focus of this program, the test fuel specifications included a number of parameters which were known or suspected to affect emissions. In an attempt to produce as controlled an experiment as possible, the levels of those parameters were specified within certain bounds across all test fuels. Examples of these properties include olefin and sulfur content and octane number. An olefin specification of 7.0 ± 1.5 vol% was used, based on the U.S. average computed from the Alliance of Automobile

Manufacturers' 2006 Summer North American Fuel Survey. Because of its important impact on benzene emission performance, the fuel benzene content specification was set at 0.62 ± 0.15 vol%, the level of the refinery average gasoline benzene standard effective on January 1, 2011. The sulfur content specification of 25 ± 5 mg/kg (ppmw) was selected to ensure that the level remained within a reasonably narrow range capped by the current refinery/importer annual average standard of 30 mg/kg. The minimum (R+M)/2 octane specification of 87 was based on minimum requirements of test vehicles selected for this program. Blending tolerances for all properties are shown in Table III-4. Note that there was no upper limit on octane number, so its value tends to be correlated with ethanol content since ethanol has a high octane number and its level varied widely across the blends.

Special attention was paid to the distribution of aromatic molecule sizes because of potential impacts on particulate matter emissions. An attempt was made to maintain consistent proportions of aromatic-containing molecules with seven or more carbon atoms. The ratios of 2:2:2:1 were chosen for C_7:C_8:C_9:C_{10} aromatics based on detailed hydrocarbon analysis data for commercial gasolines that was available to EPA at the time of fuel blending. As a practical matter of meeting the distillation targets, the proportions had to be adjusted to include more C_7 and C_8 aromatics for fuels with a combination of low T90 and high aromatics.

The fuel specification also included T10, FBP, oxidation stability, copper strip corrosion and solvent-washed gum content requirements taken from the ASTM D4814 Standard Specification for Automotive Spark-Ignition Engine Fuel. Furthermore, a limit on total content of oxygenates other than ethanol was adopted to safeguard the test fuels against such contamination. Finally, a number of uncontrolled fuel properties used in emissions test calculations were appended to the fuel specification table to make sure they were measured prior to the launch of the emissions test program and in the EPAct/V2/E-89 Fuels Round Robin to be conducted. They included carbon, hydrogen and oxygen content, density, and heat of combustion.

A single E85 fuel (fuel number 29) was also incorporated in this program for testing in flexible fuel vehicles. This fuel was not blended along with other fuels used in Phase 3 of the

EPAct/V2/E-89 Program, but was obtained from CRC. Its properties are provided in Section III.D.

B. Test Fuel Development and Blending

1. Development and Verification of Test Fuel Formulations

All fuels tested in Phase 3 of the EPAct/V2/E-89 Program were formulated by EPA in conjunction with Haltermann Solutions, Houston, Texas (with the exception of fuel 29, the E85 blend). To facilitate the formulation process, Haltermann made a detailed set of property data for their gasoline blending components available to EPA for use in designing fuels for this program. (These data were deemed confidential business information and are not provided in this report.) The majority of these components were blendstock streams taken from various points in refinery operations (e.g., reformate, alkylate, isomerate, light naphtha, etc.), ensuring that the resulting test fuels contained the typical range of components found in actual market fuels.

In the development of each test fuel formulation, EPA used a computational blending model to define and adjust the blend recipe from the available components while Haltermann prepared and characterized lab-scale hand blends. Distillation by ASTM D86 method, aromatics and olefins by D1319, DVPE by D5191 and ethanol content by D5599 were always measured at this stage. Sulfur by D5453 was measured if its predicted level was $\geq$ 27 mg/kg, benzene by D3603 when its predicted content was $\geq$0.70 vol% and octane numbers by D2699 and D2700 if predicted knock index (RON+MON)/2 was $\leq$88.0. The use of distillation stills equipped with OptiDist or equivalent technology was required for all E10, E15, and E20 fuels.[11]

The distillation parameters, DVPE, as well as the aromatic and olefinic content of hand blends were measured by Haltermann and verified by a third-party laboratory of Haltermann's

[11] Accurately measuring T50 becomes increasingly difficult as more ethanol is blended into the fuel. Older still models were found to generate results with unacceptable levels of variability. OptiDist is a trade name denoting enhanced automated control of distillation parameters that produces better results than traditional methods.

choice (typically Core Laboratories, Dixie Services, BSI-Inspectorate, or Saybolt). Single measurements were allowed for benzene, sulfur, and ethanol content, as well as MON and RON, though confirmation could be performed at one of the third-party laboratories at Haltermann's discretion.

As the development of fuel formulations progressed, it became clear that some property values in the initial fuel matrix design (Table III-3) could not be met within acceptable tolerances. They included:

- T50 target of 150±4°F for the E15 fuel 26
- T50 target of 160±4°F for all E20 fuels
- DVPE target of 6.65±0.15 psi for the vast majority of "low" DVPE fuels
- RON target of 93±2 for many fuels
- MON target of 85±2 for many fuels
- (RON + MON)/2 target of 87±2 for many fuels

The initial blending experiments also revealed that the upper T50 limit for E15 fuels was as high as 220°F, considerably higher than the 190°F target assumed for fuels 27 and 28 in the absence of relevant information in the technical literature.

Finally, a decision was made to replace the aromatic content level of 40 vol% with a target of 35 vol% to facilitate blending of "high" aromatic content fuels. It was also thought that the lower target would be more representative of future market fuels.

As a consequence of the observations and decisions mentioned above, the following modifications were incorporated in the fuel specification:

- T50 target of 160±4°F for the E15 fuel 26
- T50 target of 220±4°F for the E15 fuels 27 and 28
- T50 target of 165±4°F for all E20 fuels
- DVPE range of 7±0.25 psi for the "low" DVPE fuels

- Aromatic content target of 35±1.5 vol% for all “high” aromatic content fuels
- Separate RON and MON targets were eliminated, replaced by (RON + MON)/2 target of ≥87 for all fuels

These specification changes are reflected in Table III-4, and the resulting domain of ethanol content vs. T50 of the test fuel matrix is shown in Figure III-1.

Table III-4. Final test fuel specification for blending.

PROPERTY	UNIT	METHOD	BLENDING TOLERANCE	TEST FUELS													
				1	2	3	4	5	6	7	8	9	10	11	12	13	14
Density, 60°F	-	D4052	NA	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
API Gravity, 60°F	°API	D4052	NA	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
Ethanol Content	**vol. %**	**D5599**	**E0: < 0.1; E10: ± 0.5; E15: ± 0.5; E20: ±0.5; E85: ±2**	**10**	**0**	**10**	**10**	**0**	**10**	**0**	**0**	**0**	**10**	**10**	**10**	**0**	**0**
Total Content of Oxygenates Other Than Ethanol	vol. %	D5599	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
T10	°F	D86	-	<158	<158	<158	<158	<158	<158	<158	<158	<158	<158	<158	<158	<158	<158
T50	**°F**	**D86**	**± 4**	**150**	**240**	**220**	**220**	**240**	**190**	**190**	**220**	**190**	**220**	**190**	**150**	**220**	**190**
T90	**°F**	**D86**	**± 5**	**300**	**340**	**300**	**340**	**300**	**340**	**300**	**300**	**340**	**340**	**300**	**340**	**340**	**340**
FBP	°F	D86	-	<437	<437	<437	<437	<437	<437	<437	<437	<437	<437	<437	<437	<437	<437
DVPE	**psi**	**D5191**	**± 0.25**	**10**	**10**	**7**	**10**	**7**	**7**	**7**	**10**	**10**	**7**	**10**	**10**	**7**	**7**
Aromatics	**vol. %**	**D1319**	**± 1.5**	**15**	**15**	**15**	**15**	**35**	**15**	**15**	**15**	**35**	**35**	**35**	**35**	**35**	**15**
Olefins	vol. %	D1319	± 1.5	7	7	7	7	7	7	7	7	7	7	7	7	7	7
Benzene	vol. %	D3606	± 0.15	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62
S	mg/kg	D5453	± 5	25	25	25	25	25	25	25	25	25	25	25	25	25	25
(RON + MON)/2	-	Calc.	-	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0
C	mass %	D5291 mod*	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
H	mass %	D5291 mod*	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
O	mass %	D5599	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
Water Content	mg/kg	E1064	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
Net Heat of Combustion	MJ/kg	D4809	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
Oxidation Stability	minute	D525	-	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240
Copper Strip Corrosion, 3h at 122°F	-	D130	-	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1
Solvent-Washed Gum Content	mg/100 ml	D381	-	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5

* Method adapted to gasoline by the testing laboratory

Table III-4 Cont. Final test fuel specification for blending.

PROPERTY	UNIT	METHOD	BLENDING TOLERANCE	TEST FUELS												
				15	16	20	21	22	23	24	25	26	27	28	30	31
Density, 60°F	-	D4052	NA	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
API Gravity, 60°F	°API	D4052	NA	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
Ethanol Content	**vol. %**	**D5599**	**E0: < 0.1; E10: ± 0.5; E15: ± 0.5; E20: ±0.5; E85: ±2**	**0**	**10**	**20**	**20**	**20**	**20**	**20**	**20**	**15**	**15**	**15**	**10**	**20**
Total Content of Oxygenates Other Than Ethanol	vol. %	D5599	-	<0.1	<0.1	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.15	<0.15	<0.15	<0.1	<0.2
T10	°F	D86	-	<158	<158	<158	<158	<158	<158	<158	<158	<158	<158	<158	<158	<158
T50	**°F**	**D86**	**± 4**	**190**	**220**	**165**	**165**	**165**	**165**	**165**	**165**	**160**	**220**	**220**	**150**	**165**
T90	**°F**	**D86**	**± 5**	**300**	**300**	**300**	**300**	**300**	**340**	**340**	**340**	**340**	**340**	**300**	**325**	**325**
FBP	°F	D86	-	<437	<437	<437	<437	<437	<437	<437	<437	<437	<437	<437	<437	<437
DVPE	**psi**	**D5191**	**± 0.25**	**10**	**7**	**7**	**7**	**10**	**7**	**10**	**10**	**10**	**7**	**7**	**10**	**7**
Aromatics	**vol. %**	**D1319**	**± 1.5**	**35**	**35**	**15**	**35**	**15**	**15**	**15**	**35**	**35**	**15**	**35**	**35**	**35**
Olefins	vol. %	D1319	± 1.5	7	7	7	7	7	7	7	7	7	7	7	7	7
Benzene	vol. %	D3606	± 0.15	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62
S	mg/kg	D5453	± 5	25	25	25	25	25	25	25	25	25	25	25	25	25
(RON + MON)/2	-	Calc.	-	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0	> 87.0
C	mass %	D5291 mod*	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
H	mass %	D5291 mod*	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
O	mass %	D5599	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
Water Content	mg/kg	E1064	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
Net Heat of Combustion	MJ/kg	D4809	-	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report	Report
Oxidation Stability	minute	D525	-	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240
Copper Strip Corrosion, 3h at 122°F	-	D130	-	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1	<No. 1
Solvent-Washed Gum Content	mg/100 ml	D381	-	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5

* Method adapted to gasoline by the testing laboratory

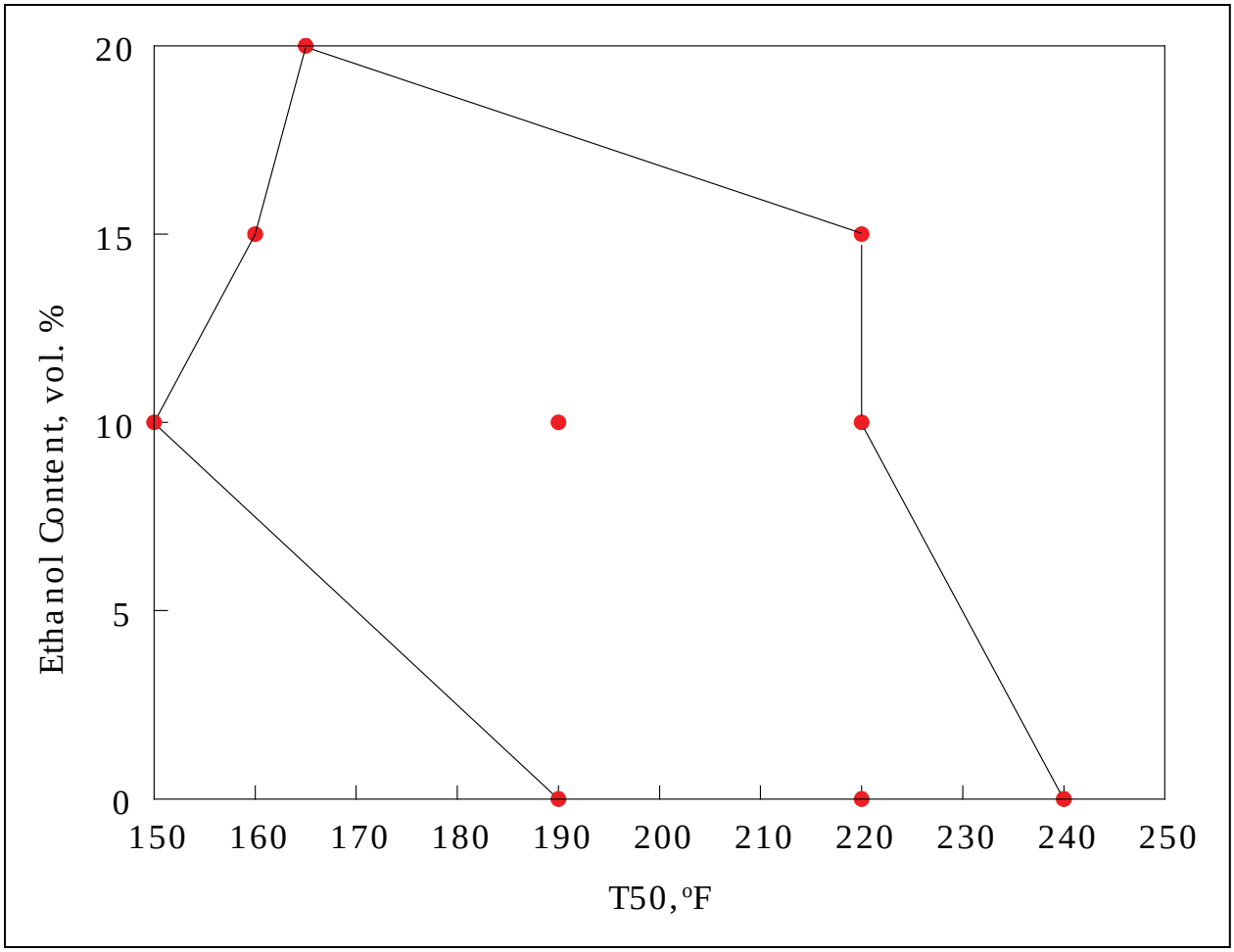

Figure III-1. Nominal ethanol content vs. T50 domain of E0-E20 fuels (Final version)

Overall, 21 different blending components were used in this program, between 9 and 16 per fuel. The process of developing formulations for each test fuel required as many as seven lab-scale iterations in some cases with particularly challenging combinations of fuel parameters.

Once the T10, T50, T90, FBP, DVPE, knock index as well as the aromatic, olefinic, benzene, sulfur and ethanol content requirements of the specification shown in Table III-4 were met, the remaining parameters listed in that specification were measured. If they also met the requirements listed in Table III-4, the final specification for the bulk blend of the fuel was issued by EPA and NREL using the template shown in Table III-4. In each such bulk blend specification, the approved hand blend results were used as targets for the distillation parameters, DVPE as well as the aromatic and olefinic content. The specification shown in Table III-4

included T30 and T70 to make sure that the distillation curves of the hand blends were closely reproduced in the bulk blends. This specification and the corresponding fuel formulation (blend recipe) were subsequently provided to Haltermann for use in the preparation of the bulk blends.

The use of hand blending results as targets for the bulk blends was done to accommodate setting of realistic targets and tolerances for the fuel blending subcontractor to follow. As shown in Section III.D, this approach still kept the parameters in question within tight blending tolerance limits.

Table III-5. Bulk blend specification template.

PROPERTY	UNIT	METHOD	BLENDING TOLERANCE	SPECIFICATION
Density, 60°F	-	D4052	-	Report
API Gravity, 60°F	°API	D4052	-	Report
Ethanol Content	vol. %	D5599	E0: <0.1 E10: ±0.5 E15: ±0.5 E20: ±0.5	Per Table III-4
Total Content of Oxygenates Other Than Ethanol	vol. %	D5599	-	<0.1
Distillation T10	°F	D86	±5	Value based on hand blend data
T30	°F	D86	±5	Value based on hand blend data
T50	°F	D86	±4	Value based on hand blend data
T70	°F	D86	±5	Value based on hand blend data
T90	°F	D86	±5	Value based on hand blend data
FBP	°F	D86	-	<437
DVPE	psi	D5191	±0.15	Value based on hand blend data
Aromatics	vol. %	D1319	±1.5	Value based on hand blend data
Olefins	vol. %	D1319	±1.5	Value based on hand blend data
Benzene	vol. %	D3606	±0.15	0.62
S	mg/kg	D5453	±5	25
(RON+MON)/2	-	Calc.	-	≥87.0
C	mass %	D5291 mod*	-	Report
H	mass %	D5291 mod*	-	Report
O	mass %	D5599	-	Report
Water Content	mg/kg	E1064	-	Report
Net Heat of Combustion	MJ/kg	D4809	-	Report
Oxidation Stability	minute	D525	-	>240
Copper Strip Corrosion, 3h at 122°F	-	D130	-	<No. 1
Solvent-Washed Gum Content	mg/100 ml	D381	-	<5

*Method adapted by a specific laboratory to testing of gasolines

2. Bulk Blending and Analysis of Test Fuels

Haltermann prepared the bulk blends and adjusted their properties until they met the bulk blend specifications. Distillation parameters, DVPE, MON, RON as well as the aromatic, olefinic, benzene and S content were measured during bulk blending by Haltermann and verified by another laboratory, usually Core Laboratories, Dixie Services, BSI-Inspectorate, or Saybolt. Single measurements were required for the remaining parameters listed in Table III-5 and could also be performed at one of the mentioned laboratories at Haltermann's discretion.

Once the bulk blend specification was met, a sample of the fuel was shipped to SwRI for confirmatory testing. If SwRI analytical results fell inside the test method reproducibility limits for all parameters listed in Table III-5, EPA and NREL approved the bulk blend for shipment to SwRI. If they fell outside the method reproducibility limits, further tests and/or blend adjustments followed until the requirements of the specification were met.

C. Shipping, Storage and Handling of Test Fuels

For contingency purposes the procured quantities of test fuels (500 gallons each) exceeded the anticipated needs to complete the program by 20%. The fuels were shipped by Haltermann to SwRI in epoxy-lined, 55-gallon drums and stored on site in a temperature-controlled facility (Figure III-2). The storage temperature for unopened drums was 70°F ± 5°F. Any drums that were to be opened (for vehicle fueling or sampling) were cooled down to a temperature of <50°F at a dedicated cold storage facility located behind the emissions laboratory next to the vehicle refueling bay. The temperature of both fuel storage facilities was continuously recorded, and was verified at least once a day.

Upon arrival at SwRI, all fuels received independent identifiers which included the EPAct/V2/E-89 fuel number, a SwRI fuel code, and a project-specific supplementary three-letter code (Table III-6). All fuel drums and corresponding work requests included all three designators in an effort to assure the correct fuel was being used at any point in the test program.

Additionally, each individual drum received a sequential number. These unique alphanumeric designations assigned to individual drums were recorded and verified by two individuals each time a test vehicle was fueled.

Each time a full drum was opened, the properties of its contents were verified using a portable PetroSpec gasoline analyzer. Based on these results, no mislabeling of fuel drums occurred during this program.

With the exception of drum content verification using the PetroSpec analyzer, the 200 gallons of fuel 29 (E85) provided by the CRC were handled in the same manner as the E0-E20 fuels.

Table III-6. Test fuel identifiers used by SwRI.

EPAct/V2/E-89 FUEL NUMBER	SwRI FUEL CODE	SwRI FUEL NAME
1	EM-6995-F	SAT
2	EM-6953-F	ELP
3	EM-7053-F	FLG
4	EM-6996-F	HOU
5	EM-7061-F	MCI
6	EM-7092-F	IND
7	EM-6954-F	JNU
8	EM-6936-F	BWI
9	EM-6955-F	KAW
10	EM-7093-F	LNK
11	EM-7055-F	MIA
12	EM-6997-F	MLS
13	EM-6965-F	CLF
14	EM-6956-F	BNA
15	EM-6957-F	OAK
16	EM-7056-F	OSH
20	EM-7057-F	PHX
21	EM-7058-F	RNO
22	EM-7001-F	SLC
23	EM-7059-F	SFO
24	EM-6998-F	TEX
25	EM-7073-F	TUL
26	EM-7094-F	YAK
27	EM-7095-F	BOS
28	EM-7096-F	NBA
29	EM-6975-F	E80
30	EM-7060-F	BUF
31	EM-7074-F	GPZ

Figure III-2. Constant-temperature storage of unopened fuel drums

D. Fuels Round Robin

The EPAct/V2/E-89 Fuels Round Robin was launched in October 2009 with the support of the Coordinating Research Council (CRC). Its objective was to supplement the fuel inspection data previously generated for the drum blends by Haltermann, Core Laboratories, Dixie Services, BSI-Inspectorate, Saybolt, and SwRI with additional results, especially for the parameters most critical to this program, i.e. T50, T90, DVPE, and ethanol and aromatic content. The complete lists of parameters which were measured in the 27 E0 through E20 fuels and the E85 fuel 29 are provided in Tables III-6 and III-7, respectively.

Round robin participants included BP, Chevron, ConocoPhillips, EPA, ExxonMobil, Marathon, PAC (distillation equipment manufacturer) and Shell. The total number of laboratories which measured the properties of fuels used in this program thus increased to 14. Tables III-6 and III-7 show the total number of test results which were eventually generated for

each fuel using the specific tests. It is worth noting that at least six T50, T90, DVPE, ethanol content and aromatic content results were available for each E0-E20 fuel.

Table III-7. Parameters measured in E0-E20 fuels in EPAct/V2/E-89 fuels round robin.

PROPERTY	TEST METHOD	TOTAL NUMBER OF TEST RESULTS PER FUEL
Density, 60°F	D4052	4
API Gravity, 60°F	D4052	4
Ethanol	D5599	6
Total Content of Oxygenates Other Than Ethanol	D5599	2-4
Distillation	D86	6-12
DVPE (EPA equation)	D5191	9-10
Aromatics	D1319	7
Olefins	D1319	7
Benzene	D3606	4-5
S	D5453	6
RON	D2699	4
MON	D2700	4
C	D5291 mod.*	4
H	D5291 mod.*	4
O	D5599	3-6
Net Heat of Combustion	D4809	3
Water	E-1064	1-2
Lead	D3237	0-1
Copper Strip Corrosion	D130	1
Solvent Washed Gum Content	D381	1
Oxidation Stability	D525	1

* Method adapted by individual laboratories to testing of gasolines

Table III-8. Parameters measured in E85 fuel during EPAct/V2/E-89 fuels round robin.

PROPERTY	TEST METHOD	TOTAL NUMBER OF TEST RESULTS PER FUEL
Density, 60°F	D4052	8
API Gravity, 60°F	D4052	8
Ethanol	D5501 mod.*	5
Methanol	D5501 mod.*	7
Distillation	D86	11
DVPE (EPA equation)	D5191	10
Benzene	D5580	1
S	D5453	3
C	D5291 mod.**	4
H	D5291 mod.**	4
O	D5501 mod.*	5
Water	E203	6
Net Heat of Combustion	D4809	1
Solvent Washed Gum	D381	2
Unwashed Gum	D381	2
Acidity (as acetic acid)	D1613	1
pHe	D6423	2
Inorganic Chloride	D7319	1
Copper	D1688 modified as outlined in D4806	1

* Method adapted by individual laboratories to testing of E85 fuels

** Method adapted by individual laboratories to testing of gasolines

The instructions issued to the participants at the outset of the round robin included special requirements regarding test methods D86, D5501 and D5291. More specifically, they were requested to:

- Use distillation stills equipped with enhanced distillation rate control technology to perform D86 distillations on ethanol containing fuels. It turned out that the round robin participants who met this requirement all used PAC stills featuring OptiDist technology. They were also instructed to perform charge volume scans for improved measurement precision. Seven round robin participants complied with this request.

- Generate C and H content data using the D5291 test method modified for use with gasoline. The standard version of this method is "not recommended for analysis of volatile materials such as gasoline". However, some contract and petroleum industry laboratories are known to have adapted this method to testing of volatile petroleum products and that support on this issue was available from test equipment manufacturers. Overall, four participants used such a modified D5291 method in this program.

- Generate ethanol content data for the E85 fuel 29 by means of the D5501 test method modified for use with E85 fuels. The standard version of this method is applicable to denatured fuel ethanol containing 93-97 mass % of ethanol. In response to this request, two of the participating laboratories adapted the ASTM D5501 method to ethanol levels typical of E85 fuels. To this end, they utilized draft #4 of the ASTM 5501-09 Proposed Revision of Standard Test Method for Determination of Ethanol Content of Mid and High Level Ethanol Fuels by Gas Chromatography (valid over the 20-100 mass % range) which at the time was being balloted by ASTM Subcommittee D02.04. It was also determined that the three laboratories which had previously generated D5501 data on the E85 fuel 29 for the CRC also used their own, in-house versions of the D5501 method adapted to testing of E85 fuels.

To ensure consistency with the best industry practice, a Procedure for Sampling and Handling of Gasoline Samples was developed with oil company assistance for use in the round robin. It was provided to SwRI and is included in this report as Appendix C.

Finally, to enable blind testing, a new set of designations was assigned to the test fuels. The only fuel which was recognizable to the participants prior to its analysis was the E85 fuel 29 which was subjected to a different battery of tests than the other 27 fuels.

The fuel samples reached round robin participants by mid-October 2009. By mid-March 2010, all promised data were received by the EPA. Shortly thereafter, a blinded set of round robin results was made available to all participants for review, so that each could determine if their data were correctly entered into the EPAct/V2/E-89 database. At the same time, the EPA

and NREL performed a preliminary review of the whole data set, identified results which were obviously in error and requested retesting by the respective laboratories.

Following the approval of round robin database entries by all participants and the incorporation of any results of retesting along with fuel property data generated earlier in the program, the EPA statistician identified outliers in the data using the methodology described in ASTM E178-08 Standard Practice for Dealing with Outlying Observations. The details of this methodology as applied to the Phase 3 EPAct/V2/E-89 fuel data set have been summarized by the EPA statistician and are included in this report as Appendix D.

The final, blinded EPAct/V2/E-89 fuel property data set is provided in Appendix E. Red cell fill color in Appendix E tables indicates outliers. Orange fill color denotes data which were not used in the calculation of parameter averages. The latter occurred when a T50 and/or a T90 result was an outlier or when sample contamination was suspected.

The final set of Phase 3 EPAct/V2/E-89 test fuel properties to be used in emissions modeling is provided in Tables III-8 and III-9. Their values were computed using the round robin test data provided in Appendix E following the elimination of outliers. Table III-9 lists the properties of E0-E20 fuels, while Table III-10 those of the E85 fuel 29.

Table III-9. Final properties of E0-E20 Phase 3 EPAct/V2/E-89 fuels based on round robin results.

PROPERTY	UNIT	TEST METHOD	FUEL													
			1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
Density, 60°F	g/cm^3	D4052	0.7211	0.7220	0.7350	0.7346	0.7573	0.7342	0.7208	0.7191	0.7454	0.7644	0.7596	0.7517	0.7540	0.7223
API Gravity, 60°F	°API	D4052	64.6	64.3	60.8	60.9	55.2	61.1	64.6	65.1	58.2	53.4	54.6	56.5	56.0	64.2
Ethanol	**vol. %**	**D5599**	**10.03**	**<0.10**	**10.36**	**9.94**	**<0.10**	**10.56**	**<0.10**	**<0.10**	**<0.10**	**9.82**	**10.30**	**9.83**	**<0.10**	**<0.10**
Total Content of Oxygenates Other Than Ethanol	vol. %	D5599	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
Distillation IBP	°F	D86 (**OptiDist** or equivalent for E10, E15 and E20 fuels)	92.9	83.5	106.4	89.9	94.1	106.7	100.1	83.7	85.3	104.7	92.0	91.3	96.6	100.4
5% evap	°F		112.5	105.4	136.0	115.9	128.6	130.4	127.6	108.1	105.1	130.0	115.4	110.7	127.0	126.5
10% evap	°F		117.3	121.7	141.7	126.3	145.4	135.9	137.0	123.4	115.1	136.3	124.4	116.9	139.8	135.5
20% evap	°F		123.9	154.4	148.9	140.9	172.6	142.6	149.0	151.6	130.3	144.3	137.6	125.0	158.7	147.3
30% evap	°F		131.2	190.6	155.0	151.7	199.4	148.3	161.7	185.1	147.2	151.0	148.1	133.8	178.2	160.0
40% evap	°F		139.9	218.5	175.1	161.2	222.1	153.4	176.6	204.4	167.7	161.6	156.5	142.8	199.9	175.1
50% evap	**°F**		**148.9**	**236.7**	**217.5**	**221.9**	**237.0**	**188.5**	**193.1**	**221.1**	**192.8**	**217.1**	**189.3**	**152.2**	**222.5**	**192.8**
60% evap	°F		172.3	252.7	230.2	245.9	247.2	228.2	210.2	233.5	224.7	261.5	231.1	198.5	245.2	212.0
70% evap	°F		224.1	271.7	243.6	270.0	258.5	267.7	228.6	246.4	260.3	290.4	251.4	275.1	269.8	237.3
80% evap	°F		254.6	305.9	257.1	303.5	273.1	310.1	251.5	264.0	292.2	317.5	270.0	307.9	303.5	280.1
90% evap	**°F**		**300.2**	**340.1**	**295.9**	**337.5**	**300.0**	**340.4**	**298.4**	**303.1**	**341.8**	**340.2**	**298.6**	**339.8**	**337.9**	**338.5**
95% evap	°F		334.5	353.0	334.4	352.0	323.5	352.7	329.3	330.5	363.5	354.3	325.0	357.7	354.4	354.5
FBP	°F		368.0	375.3	368.9	369.8	357.8	369.2	361.8	360.9	384.7	372.4	360.8	375.9	377.5	377.5
DVPE (EPA equation)	**psi**	**D5191**	**10.07**	**10.20**	**6.93**	**10.01**	**6.95**	**7.24**	**7.15**	**10.20**	**10.30**	**7.11**	**9.93**	**10.13**	**6.92**	**7.14**
Aromatics	**vol. %**	**D1319**	**15.4**	**14.1**	**15.0**	**15.5**	**34.7**	**15.0**	**17.0**	**15.7**	**35.8**	**34.0**	**35.0**	**34.8**	**34.1**	**16.9**
Olefins	vol. %	D1319	7.6	6.8	7.6	6.8	6.9	8.8	7.5	6.4	6.2	6.1	6.9	6.9	6.3	8.5
Saturates	vol. %	100 - D1319Aromatics - D1319Olefins - D5599Ethanol	67.0	79.1	67.0	67.8	58.4	65.6	75.5	78.0	58.0	50.1	47.8	48.5	59.6	74.6
Benzene	vol. %	D3606	0.62	0.51	0.61	0.54	0.51	0.68	0.55	0.50	0.54	0.52	0.54	0.57	0.51	0.52
S	mg/kg	D5453	30	23	22	21	24	23	23	23	23	25	24	19	23	24
RON	-	D2699	94.8	96.0	98.0	97.1	96.7	96.3	91.2	95.5	94.5	98.5	97.8	100.4	95.8	91.5
MON	-	D2700	86.3	88.6	87.6	87.6	86.3	86.6	84.2	87.8	84.8	87.2	85.6	88.0	85.8	84.6
(RON+MON)/2	-	-	90.6	92.3	92.8	92.4	91.5	91.5	87.7	91.7	89.7	92.9	91.7	94.2	90.8	88.1
C	mass %	D5291 mod.*	81.70	85.12	81.61	82.21	86.58	81.52	85.16	85.12	87.03	83.47	83.68	83.32	86.76	85.28
H	mass %	D5291 mod.*	14.02	14.43	14.17	14.12	12.92	14.21	14.25	14.32	12.82	12.83	12.61	12.68	13.15	14.29
O	mass %	D5599	3.9	<0.1	3.9	3.7	<0.1	4.0	<0.1	<0.1	<0.1	3.6	3.7	3.6	<0.1	<0.1
Net Heat of Combustion	MJ/kg	D4809	41.950	43.960	41.536	41.952	42.948	41.785	43.735	44.037	43.209	41.210	41.175	41.373	43.171	43.519
Water	mass %	E-1064	0.071	0.010	0.059	0.077	0.014	0.073	0.019	0.020	0.009	0.067	0.066	0.066	0.014	0.015
Lead	g/l	D3237	-	<0.001	-	-	<0.003	-	<0.001	0.001	<0.001	<0.003	-	<0.003	<0.001	<0.001
Copper Strip Corrosion	-	D130	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A
Solvent Washed Gum Content	mg/100ml	D381	<0.5	<0.5	<0.5	1.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	0.5	<0.5	1.5	<0.5
Oxidation Stability	min.	D525	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240

* Method adapted by individual laboratories to testing of gasolines

Table III-9 Cont. Final properties of E0-E20 Phase 3 EPAct/V2/E-89 fuels based on round robin results.

PROPERTY	UNIT	TEST METHOD	FUEL												
			15	**16**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**30**	**31**
Density, 60°F	g/cm^3	D4052	0.7428	0.7636	0.7425	0.7754	0.7371	0.7476	0.7422	0.7702	0.7593	0.7434	0.7699	0.7508	0.7742
API Gravity, 60°F	°API	D4052	58.8	53.6	58.9	50.8	60.3	57.6	58.9	52.0	54.6	58.6	52.1	56.8	51.1
Ethanol	**vol. %**	**D5599**	**<0.10**	**10.76**	**20.31**	**20.14**	**20.51**	**20.32**	**20.51**	**20.03**	**15.24**	**14.91**	**14.98**	**9.81**	**20.11**
Total Content of Oxygenates Other Than Ethanol	vol. %	D5599	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
Distillation IBP	ºF	D86 (**OptiDist** or equivalent for E10, E15 and E20 fuels)	84.7	104.5	107.9	106.3	89.8	109.0	89.7	89.0	88.7	104.8	103.9	90.9	105.8
5% evap	ºF		105.5	133.0	137.3	134.7	118.8	133.3	115.9	113.7	109.6	135.3	136.3	110.3	132.5
10% evap	ºF		115.6	139.2	142.6	141.3	129.6	138.9	126.9	125.5	117.1	142.3	144.2	116.7	139.1
20% evap	ºF		130.5	147.8	149.7	150.3	144.3	146.2	142.8	142.1	127.8	152.0	154.0	125.4	147.7
30% evap	ºF		146.6	155.1	155.3	157.1	153.7	152.3	153.2	153.3	138.6	158.0	160.2	133.9	155.1
40% evap	ºF		166.3	172.1	159.6	162.6	159.5	157.8	160.4	160.9	149.8	163.8	165.8	143.1	161.3
50% evap	**ºF**		**189.7**	**218.8**	**162.7**	**167.6**	**163.2**	**162.5**	**165.1**	**166.9**	**160.3**	**221.5**	**216.6**	**152.9**	**167.3**
60% evap	ºF		216.2	237.5	179.9	217.3	167.2	171.6	172.9	191.3	174.7	265.1	240.2	197.2	214.0
70% evap	ºF		243.0	251.9	234.8	255.2	233.9	270.9	266.1	281.6	277.0	274.9	251.6	267.3	271.6
80% evap	ºF		265.9	268.6	253.1	275.3	253.6	311.4	305.5	310.3	306.5	311.3	268.4	294.6	297.0
90% evap	**ºF**		**299.4**	**300.6**	**298.7**	**305.0**	**297.3**	**338.2**	**338.1**	**337.9**	**338.7**	**340.3**	**298.8**	**323.8**	**325.2**
95% evap	ºF		329.3	330.8	336.6	331.3	334.5	350.0	350.3	352.7	356.7	351.9	327.3	341.8	342.1
FBP	ºF		363.7	365.6	371.9	360.5	369.9	364.6	368.2	371.8	377.3	372.2	363.2	366.1	365.6
DVPE (EPA equation)	**psi**	**D5191**	**10.23**	**7.12**	**6.70**	**7.06**	**10.21**	**6.84**	**10.12**	**10.16**	**10.21**	**6.97**	**6.87**	**10.23**	**6.98**
Aromatics	**vol. %**	**D1319**	**35.3**	**35.6**	**15.2**	**35.5**	**15.0**	**15.9**	**15.3**	**35.2**	**35.6**	**14.9**	**34.5**	**35.5**	**35.5**
Olefins	vol. %	D1319	7.2	6.8	7.4	7.1	6.9	7.5	7.3	6.6	6.5	7.4	7.0	6.5	6.8
Saturates	vol. %	100 - D1319Aromatics - D1319Olefins - D5599Ethanol	57.4	46.9	57.1	37.3	57.6	56.4	56.9	38.1	42.7	62.9	43.5	48.2	37.6
Benzene	vol. %	D3606	0.54	0.62	0.61	0.61	0.59	0.63	0.62	0.65	0.62	0.56	0.59	0.58	0.60
S	mg/kg	D5453	24	23	22	22	21	21	21	26	23	26	24	23	25
RON	-	D2699	95.0	101.0	101.9	101.4	101.8	97.4	100.8	102.2	101.7	100.8	102.7	100.5	101.7
MON	-	D2700	84.9	88.3	89.3	87.5	89.3	86.8	88.6	88.3	88.5	89.2	89.4	88.1	88.2
(RON+MON)/2	-	-	90.0	94.7	95.6	94.5	95.6	92.1	94.7	95.3	95.1	95.0	96.1	94.3	95.0
C	mass %	D5291 mod.*	86.88	83.40	78.06	79.90	78.24	78.34	78.47	80.62	81.48	80.27	81.78	83.17	79.90
H	mass %	D5291 mod.*	12.79	12.66	14.01	12.43	13.85	13.86	13.86	12.38	12.45	14.01	12.62	13.00	12.49
O	mass %	D5599	<0.1	3.9	7.6	7.1	7.7	7.5	7.6	7.2	5.6	5.5	5.4	3.6	7.2
Net Heat of Combustion	MJ/kg	D4809	43.108	41.013	40.057	39.285	40.031	39.915	40.114	38.855	40.384	41.062	40.383	41.304	39.391
Water	mass %	E-1064	0.012	0.066	0.138	0.128	0.113	0.112	0.108	0.117	0.088	0.090	0.091	0.086	0.143
Lead	g/l	D3237	<0.001	-	<0.003	0.009	0.004	<0.003	0.005	0.001	<0.003	<0.003	<0.003	-	<0.003
Copper Strip Corrosion	-	D130	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A
Solvent Washed Gum Content	mg/100ml	D381	0.5	1	<0.5	0.5	<0.5	0.5	0.5	<0.5	<0.5	0.5	<0.5	<0.5	0.5
Oxidation Stability	min.	D525	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240	>240

* Method adapted by individual laboratories to testing of gasolines

Table III-10. Final properties of the E85 Phase 3 EPAct/V2/E-89 fuel.

PROPERTY	UNIT	TEST METHOD	Average
Density, 60°F	g/cm^3	D4052	0.7797
API Gravity, 60°F	°API	D4052	49.8
Uncorrected Ethanol	mass %	D5501 mod.*	79.59
Uncorrected Methanol	mass %	D5501 mod.*	0.01
Ethanol	vol. %	D5501 mod.*	77.15
Methanol	vol. %	D5501 mod.*	<0.01
Estimated Hydrocarbon Content	vol. %	100 - D5501Ethanol - E203Water	22.14
Distillation IBP	ºF	D86	99.0
5% evap	ºF		132.9
10% evap	ºF		154.3
20% evap	ºF		167.6
30% evap	ºF		170.3
40% evap	ºF		171.2
50% evap	ºF		171.8
60% evap	ºF		172.1
70% evap	ºF		172.5
80% evap	ºF		172.9
90% evap	ºF		173.9
95% evap	ºF		176.2
FBP	ºF		265.8
DVPE (EPA equation)	psi	D5191	8.92
Benzene	vol. %	D5580	0.12
S	mg/kg	D5453	16
C	mass %	D5291 mod.**	57.74
H	mass %	D5291 mod.**	12.80
O	mass %	D5501 mod.*	27.19
Water	mass %	E203	0.93
	vol. %	E203	0.72
Net Heat of Combustion	MJ/kg	D4809	30.058
Solvent Washed Gum	mg/100 ml	D381	1.9
Unwashed Gum	mg/100 ml	D381	1.8
Acidity (as acetic acid)	mass %	D1613	0.0021
pHe	-	D6423	8.08
Inorganic Chloride	mg/kg	D7319	Not detected
Copper	mg/l	D1688 modified as outlined in D4806	0.02

* Method adapted by individual laboratories to testing of E85 fuels

** Method adapted by individual laboratories to testing of gasolines

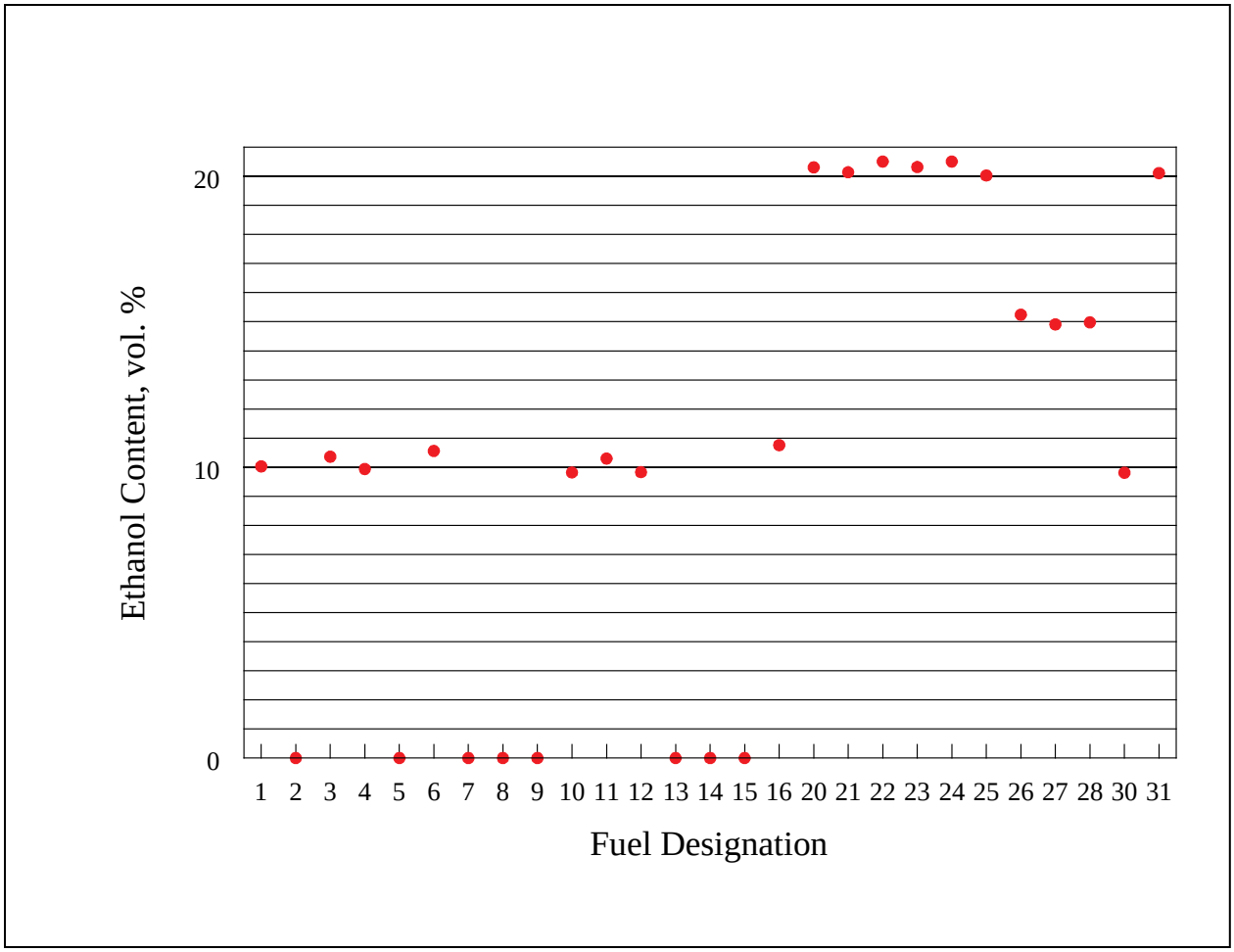

Figure III-3. Ethanol content of Phase 3 EPAct/V2/E-89 fuels.

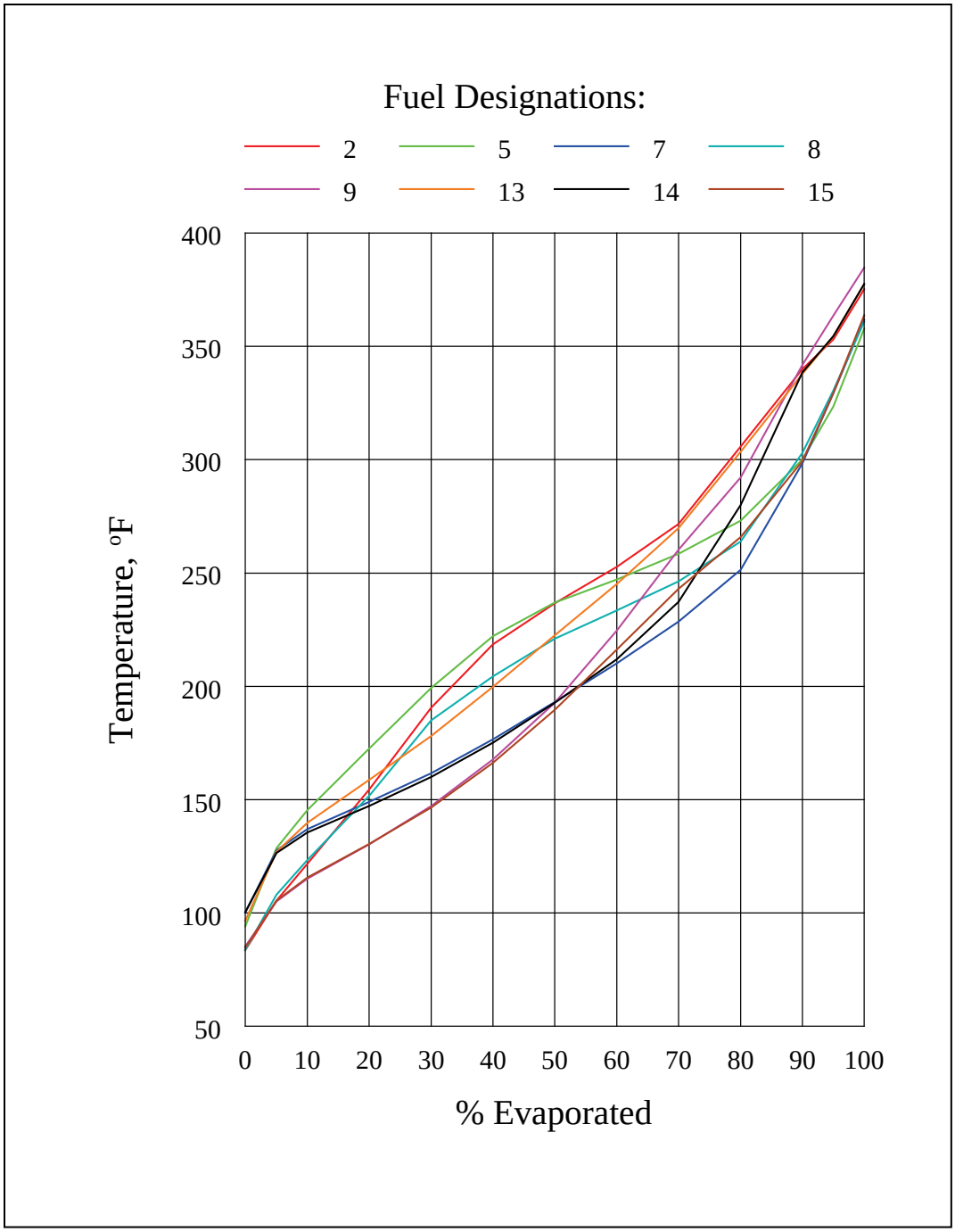

Figure III-4. Distillation curves of E0 Phase 3 EPAct/V2/E-89 fuels.

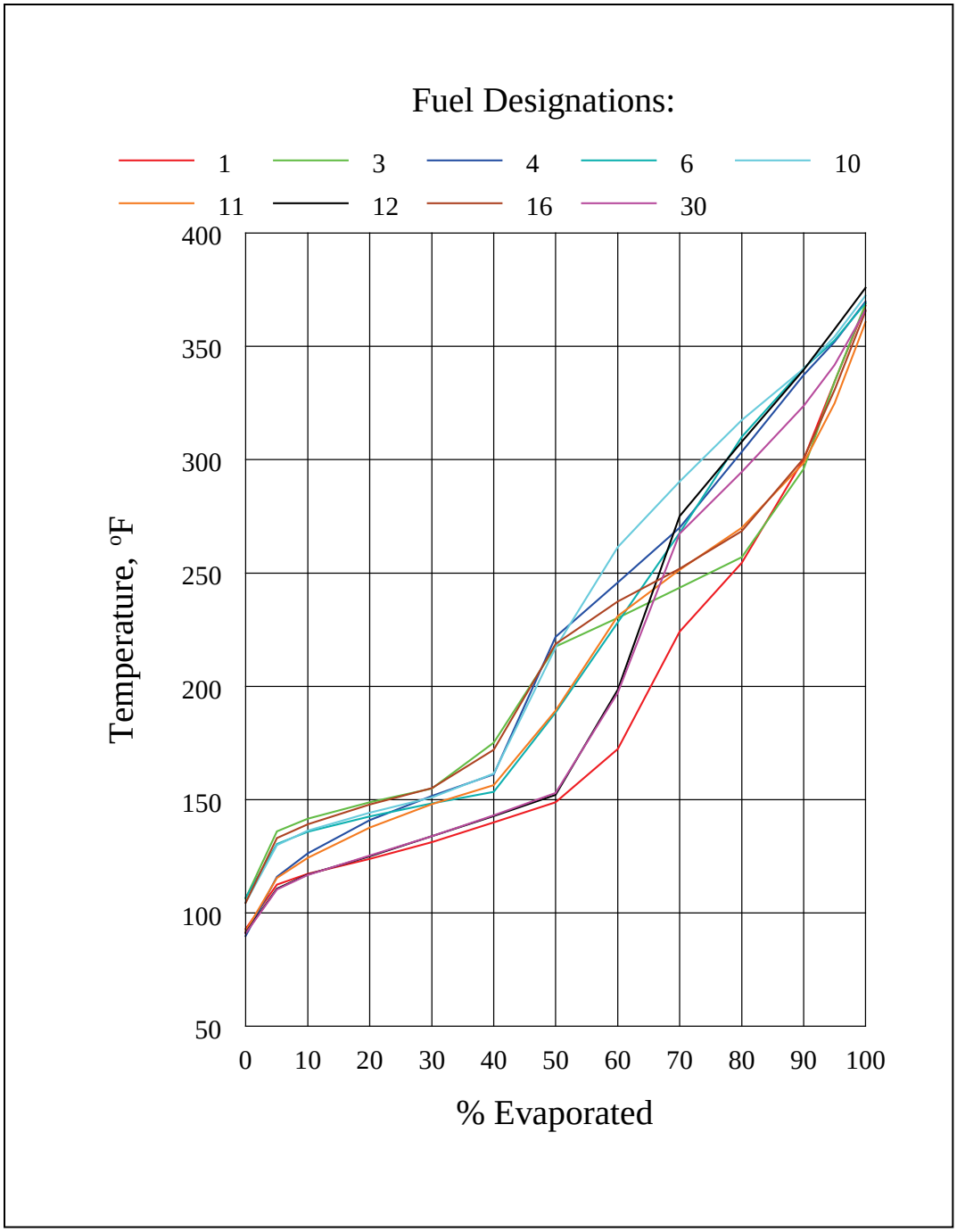

Figure III-5. Distillation curves of E10 Phase 3 EPAct/V2/E-89 fuels.

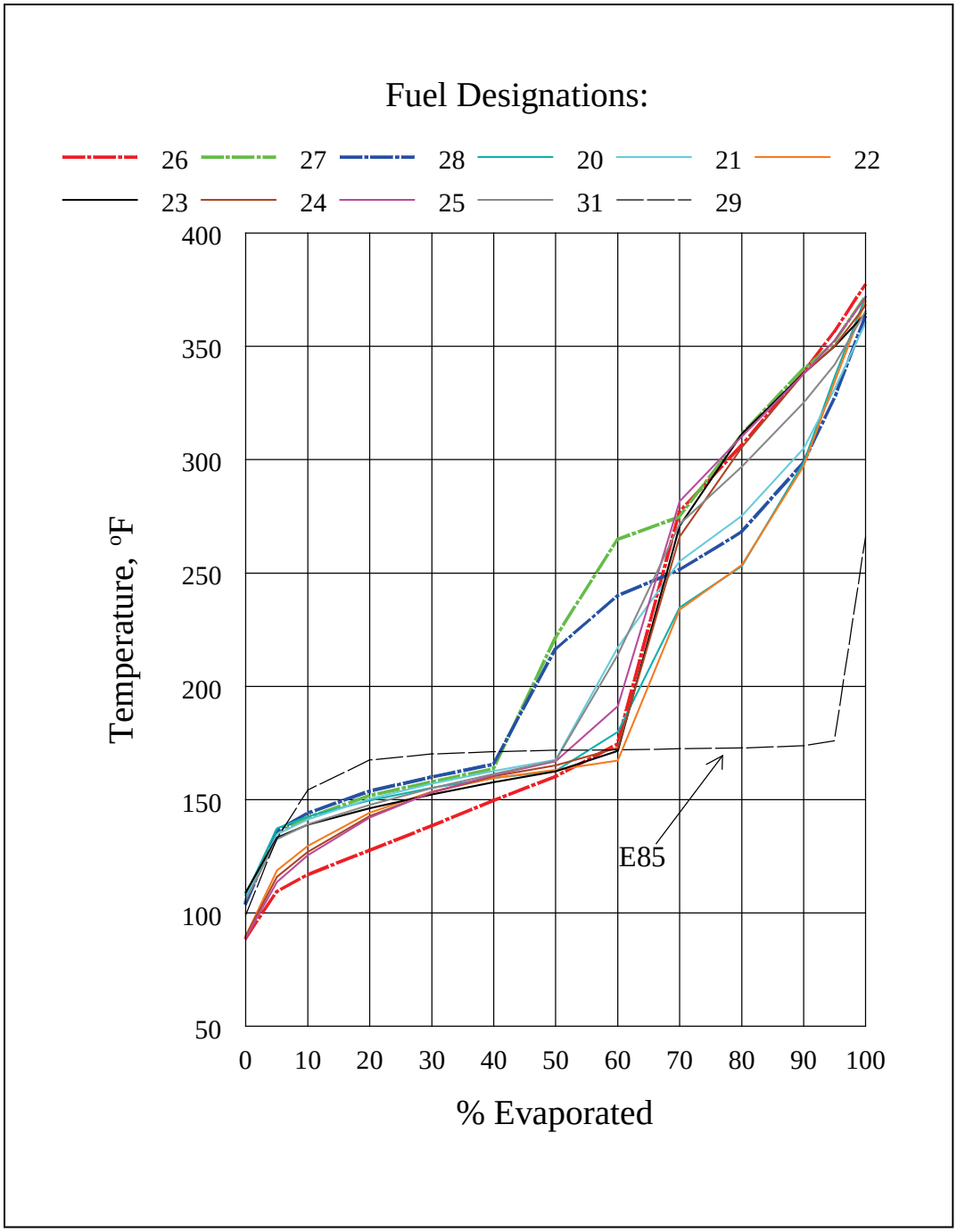

Figure III-6. Distillation curves of E15, E20 and E85 Phase 3 EPAct/V2/E-89 fuels.

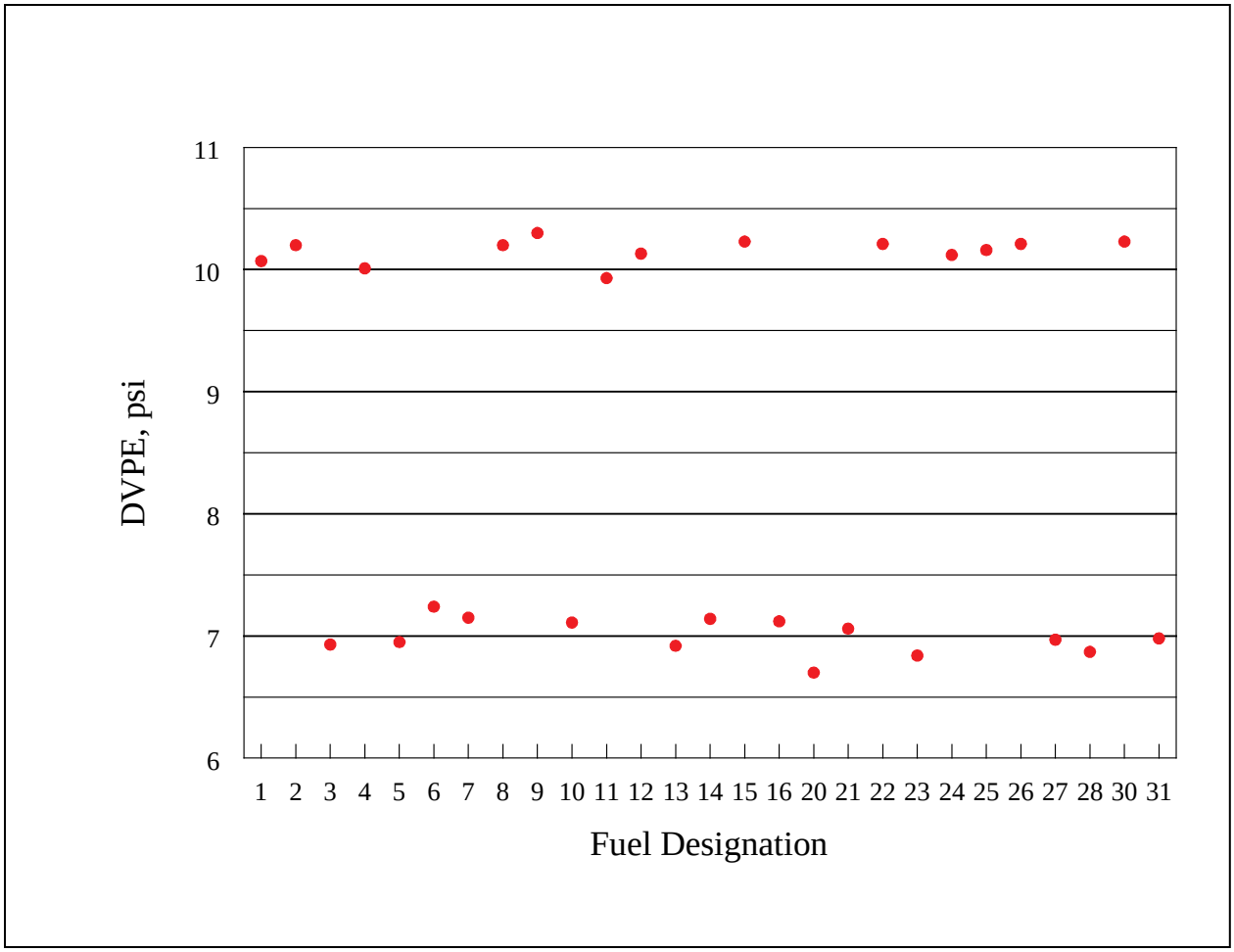

Figure III-7. DVPE of Phase 3 EPAct/V2/E-89 fuels.

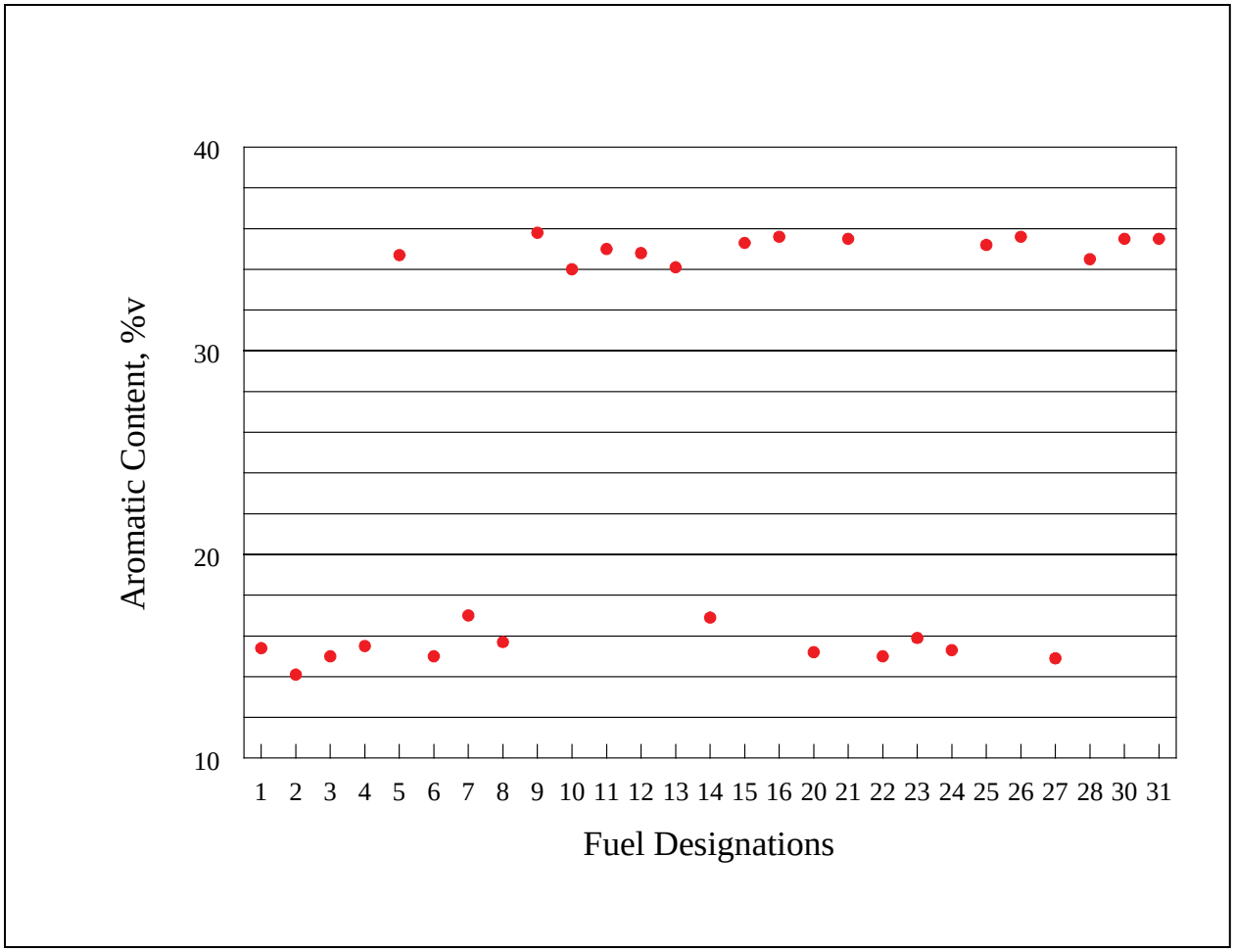

Figure III-8. Aromatic content of Phase 3 EPAct/V2/E-89 fuels.

IV. TEST VEHICLE FLEET SIZING AND SELECTION

A. Fleet Selection

Emission behavior of vehicles originally meeting Tier 0 and Tier 1 emission standards (generally, those sold in the 1990s) is well-characterized in databases supporting the Complex Model for gasoline production compliance and the Agency's California oxygenate waiver decision.[12] Together, these databases contain thousands of measurements taken on hundreds of vehicles and fuel blends. At the time this program was being designed, only a limited amount of fuel effect data existed for vehicles meeting the Tier 2 standards, data that would be required by Congress as highlighted in Section II. Thus, the focus of this program was on filling that data gap.

1. Statistical Determination of Number of Vehicles

Proper design of an experimental study should ensure that the effect being investigated has a good chance of being detected if it exists. This likelihood of detection is referred to as the statistical power of a study. Estimation of power requires specification of a size of effect deemed to be meaningful, as well as information about the variability or noise that will occur in the measurements. These inputs may be informed by previous experiments or, if no such data is available, more arbitrarily chosen based on some assumptions about the behavior of the test subjects.

A power analysis was performed during the initial design of this program to estimate the number of vehicles required to detect a fuel effect of various sizes for both hydrocarbon and NOx emissions. The statistical methods were based on those presented by Snedecor and Cochran, and are consistent with work done for the Auto/Oil Air Quality Improvement Research Program (AQIRP) in the early 1990s.[13,14] Table IV-1 shows estimates of vehicle-by-fuel

[12] See footnote 1.
[13] Snedecor G.W. & Cochran W.G., "Statistical Methods", Iowa State Univ Press, 8th Ed.,1989.
[14] See Painter, J.L. & Rutherford, J.A., "Statistical Design and Analysis Methods for the Auto/Oil Air Quality Improvement Research Program", SAE paper number 920319.

variability and repeat measurement error derived from data collected in recent emission programs.

Table IV-1. Data used in vehicle selection power analysis.

	Study	NOx	NMHC
Vehicle-by-fuel variability (% as COV)			
	CRC E-67	2	18
	2005-6 MSAT	14	13
	Value used	**14**	**18**
Test-to-test repeatability (% as COV)			
	CRC E-67	20	19
	2006 CARB	22	17
	2005-6 MSAT	15	17
	CRC-E74b	22	20
	Value used	**22**	**20**

The prior test programs shown here were conducted in a variety of configurations and were included to give an idea of the range of values expected. CRC E-67 and E-74b represent testing on different vehicles and fuels performed at the same laboratory. The 2005-6 MSAT program tested different vehicles and fuels at different laboratories. The 2006 CARB data represent testing of the same vehicle on the same fuel at different laboratories. Examination of both NOx and hydrocarbon emissions allowed the design to accommodate the more restrictive of the two.

These two measures of variability were combined to compute the vehicle-by-fuel standard error was computed as follows, where n is the number of replicates assumed.

$$SE_{vehicle\ by\ fuel} = \sqrt{variability^2 + \frac{repeatability^2}{n}}$$

The initial vehicle selection done for Phase 1 of the program assumed a power of 0.90 and a significance level of 0.05 for the statistical tests. Thus, the following equation was used to estimate the number of samples (test vehicles) required, denoted here as *r*: [15]

$$r = 1 + 10.5\left(2\left[\frac{SE_{vehicle\ by fuel}}{relative\ difference}\right]^2\right)$$

Table IV-2 shows an example of the results run for different numbers of replicates and relative differences detectable between two treatments (e.g., 0.25 = 25% fuel effect).

Table IV-2. Results of power analysis for number of vehicles to test.

Replicates	$SE_{vehicle\ by\ fuel}$	Relative difference	Vehicles at 0.90 power
1	0.270	0.05	614
		0.10	155
		0.15	70
		0.20	40
		0.25	26
		0.40	11
2	0.230	0.05	446
		0.10	113
		0.15	51
		0.20	29
		0.25	19
		0.40	8
3	0.215	0.05	389
		0.10	98
		0.15	45
		0.20	26
		0.25	17
		0.40	8

A relative fuel effect of 25% was selected during the design of AQIRP as a reasonable size of effect to investigate. Using this level of effect, a design of 19 vehicles with two replicates for each fuel-vehicle combination was originally chosen to meet a 90% study power. This number seemed large, considering that anti-backsliding policy discussions at the time of this study design were focusing on NOx emission changes on the order of 5-10% that occurred when

[15] For more details on this calculation, see Snedecor & Cochran, 1989, p. 102-105.

ethanol was added to gasoline in previous studies on older vehicles. However, there was an expectation that through careful design and execution, this program could achieve better measurement variability and repeatability than suggested by previous programs, which would allow adequate statistical power with smaller sample size (something that could not be known until after data collection was underway). Additionally, the program budget would not have accommodated the hundreds of test vehicles suggested by the calculation to detect relative differences below 10%.

2. Criteria Based on Sales, Engine Size, Technology, etc.

The Phase 1 fleet of 19 test vehicles was chosen with the intent of being representative of latest-technology light duty vehicles being sold at the time the program was being launched. In terms of regulatory standards, the test fleet was to conform on average to Tier 2 Bin 5 exhaust levels and employ a variety of emission control technologies, to be achieved by including a range of vehicle sizes and manufacturers.

Engine family sales data obtained from EPA certification and Wards databases was analyzed to generate a list of high-sales vehicles as candidates for inclusion.[16] Grouping sales data by engine family allowed additional transparency and flexibility in choosing test vehicles that represent a wider group than one specific make and model. The resulting test fleet, shown in Table IV-3, was used in Phases 1-2 of the program and the engine families represented were expected to cover more than half of new vehicle sales for MY 2008. No criteria were used by the sponsors to select the individual test vehicles for lease, and thus this selection was effectively random.

[16] Engine family is a term used in manufacturing and certification to describe a combination of a base engine and after-treatment system that may be used in several vehicle makes and models offered by a manufacturer.

Table IV-3. Test vehicle fleet used in Phases 1 and 2 (19 vehicles).

Model Year	Make	Brand	Model	Program ID	Engine Size	Engine Family	Tier 2 Cert Bin
2008	GM	Chevrolet	Cobalt	CCOB	2.2L I4	8GMXV02.4025	5
2008	GM	Chevrolet	Impala FFV	CIMP	3.5L V6	8GMXV03.9052	5
2008	GM	Saturn	Outlook	SOUT	3.6L V6	8GMXT03.6151	5
2008	GM	Chevrolet	Silverado FFV	CSIL	5.3L V8	8GMXT05.3373	5
2008	Toyota	Toyota	Corolla	TCOR	1.8L I4	8TYXV01.8BEA	5
2008	Toyota	Toyota	Camry	TCAM	2.4L I4	8TYXV02.4BEA	5
2008	Toyota	Toyota	Sienna	TSIE	3.5L V6	8TYXT03.5BEM	5
2008	Toyota	Toyota	Tundra	TTUN	4.0L V6	8TYXT04.0AES	5
2008	Ford	Ford	Focus	FFOC	2.0L I4	8FMXV02.0VD4	4
2008	Ford	Ford	Taurus	FTAU	3.5L V6	8FMXV03.5VEP	5
2008	Ford	Ford	Explorer	FEXP	4.0L V6	8FMXT04.03DB	4
2008	Ford	Ford	F150 FFV	F150	5.4L V8	8FMXT05.44HF	8
2008	Chrysler	Dodge	Caliber	DCAL	2.4L I4	8CRXB02.4MEO	5
2008	Chrysler	Dodge	Caravan FFV	DCAR	3.3L V6	8CRXT03.3NEP	8
2008	Chrysler	Jeep	Liberty	JLIB	3.7L V6	8CRXT03.7NE0	5
2008	Honda	Honda	Civic	HCIV	1.8L I4	8HNXV01.8LKR	5
2008	Honda	Honda	Accord	HACC	2.4L I4	8HNXV02.4TKR	5
2008	Honda	Honda	Odyssey	HODY	3.5L V6	8HNXT03.54KR	5
2008	Nissan	Nissan	Altima	NALT	2.5L I4	8NSXV02.5G5A	5

3. Reduction of Fleet for Phase 3

Due to budget constraints that arose after beginning Phase 3, the fleet was reduced to 15 vehicles from the 19 used in Phases 1-2. (One additional vehicle, the Dodge Caravan, was used only for E85 testing to provide a total of four FFVs tested in the program.) A study power analysis was repeated using data from 15 vehicles in Phase 1, and results suggested a power in the range of 0.7-0.8 for detecting a 25% relative difference at a significance level of 0.05. (Relaxing the significance level to 0.10 will increase the study power. Statistical analysis of Phase 1 data found significant fuel effects smaller than 25%, so this shouldn't be understood as a lower limit of detectable effects, but rather as a screen for the largest effect that is unlikely to be missed.)

Reduction of the fleet meant choosing four vehicles to eliminate. Primary considerations in this process included retaining high-sales engine families, a balance of vehicle and engine sizes, and maintaining representation of all manufacturers originally included in order to represent a range of technologies and emission control strategies. There was also consideration

of the fact that changes in the test fleet could shift the average program results. As a screen for this issue, all nineteen vehicles were ranked according to their NOx and NMHC sensitivity to fuel ethanol level based on the Phase 1 data, with the intent being to avoid removing several vehicles with similar emissions behavior (though the two pollutants could provide conflicting direction). Table IV-4 shows the resulting set of vehicles used to generate the Phase 3 data. Note that the Dodge Caravan was used only for E85 testing, so that 15 vehicles were used to produce the matrix of 27 fuels.

Table IV-4. Reduced vehicle fleet used for Phase 3.

MAKE	MODEL YEAR	BRAND	MODEL	VEHICLE NAME	ENGINE	ENGINE FAMILY	EPA T2 BIN	CA CERT	PHASE 3 STARTING ODOMETER
GM	2008	Chevrolet	Cobalt	CCOB	2.4L I4	8GMXV02.4025	5	NA	4,841
GM	2008	Chevrolet	Impala FFV	CIMP	3.5L V6	8GMXV03.9052	5	L2	5,048[a]
GM	2008	Saturn	Outlook	SOUT	3.6L V6	8GMXT03.6151	5	L2	5,212[a]
GM	2008	Chevrolet	Silverado FFV	CSIL	5.3L V8	8GMXT05.3373	5	NA	5,347[b]
Toyota	2008	Toyota	Corolla	TCOR	1.8L I4	8TYXV01.8BEA	5	U2	5,019[a]
Toyota	2008	Toyota	Camry	TCAM	2.4L I4	8TYXV02.4BEA	5	U2	4,974[b]
Toyota	2008	Toyota	Sienna	TSIE	3.5L V6	8TYXT03.5BEM	5	U2	4,997
Ford	2008	Ford	Focus	FFOC	2.0L I4	8FMXV02.0VD4	4	U2	5,150[a,b]
Ford	2008	Ford	Explorer	FEXP	4.0L V6	8FMXT04.03DB	4	NA	6,799[c]
Ford	2008	Ford	F-150 FFV	F150	5.4L V8	8FMXT05.44HF	8	NA	5,523[a]
Chrysler	2008	Dodge	Caliber	DCAL	2.4L I4	8CRXB02.4MEO	5	NA	4,959
Chrysler	2008	Dodge	Caravan FFV[d]	DCAR	3.3L V6	8CRXT03.3NEP	8	NA	5,282
Chrysler	2008	Jeep	Liberty	JLIB	3.7L V6	8CRXT03.7NE0	5	NA	4,785
Honda	2008	Honda	Civic	HCIV	1.8L I4	8HNXV01.8LKR	5	U2	4,765
Honda	2008	Honda	Odyssey	HODY	3.5L V6	8HNXT03.54KR	5	U2	4,850
Nissan	2008	Nissan	Altima	NALT	2.5L I4	8NSXV02.5G5A	5	L2	5,211[b]

[a] – These vehicles were added to the Phase 3 test matrix at a later date. Prior to their inclusion in the matrix, they received on-road miles every other week.

[b] – These vehicles were included in an FTP interim test program (EPA WA 1-09) conducted between Phases 1 and 2.

[c] – During Phase 1, the initial 4,000 miles of vehicle break-in was conducted with the wrong crankcase lubricant viscosity grade. An additional 2,000-mile break-in was conducted with the correct lubricant viscosity grade.

[d] – Dodge Caravan FFV was only tested with E85

4. Vehicle Procurement and Delivery

Vehicles for this program were obtained brand new and delivered to the SwRI facility in San Antonio, Texas, where testing took place. All vehicles were leased by SwRI for two years at the initiation of Phase 1 of the V2/EPAct/E-89 program. Due to changes and additions to the overall program, the term of the two-year leases expired prior to the completion of all Phase 3 testing. The Coordinating Research Council then purchased the test vehicles and made them available to the test program for the remainder of its duration.

B. Initial Preparation and Storage

1. Mileage Accumulation and Conditioning of Engine Oil

Prior to commencement of emission testing for Phase 1 of the program, each vehicle was brought up to 4,000 odometer miles, the intent being to avoid variability in emissions behavior occurring during engine break-in. This was accomplished by operating the vehicles on mileage accumulation dynamometers over the Standard Road Cycle using a non-oxygenated, commercially-acquired, 87 octane gasoline (see Table IV-5).

Engine crankcase oil meeting GF-4 specifications was provided for the program by the Lubrizol Corporation. Engine oil was drained and replaced with the appropriate manufacturer-recommended viscosity grade at the start of mileage accumulation, and again after 2,000 miles. The 2,000-mile fill remained in the test vehicles throughout Phases 1, 2, and 3 of the program. Each vehicle's oil sump was overfilled by 12 ounces at the 2,000-mile oil change to accommodate oil samples to be taken during the course of the program (see Section V.C.2 for more details on oil sampling). The vehicle odometer readings at the start of Phase 3 are included in Table IV-4.

Table IV-5. Properties of mileage accumulation fuel.

PROPERTY	UNIT	METHOD	Valero RUL
Density, 60°F	g/cm^3	D4052	0.7329
API Gravity, 60°F	°API	D4052	61.5
Ethanol Content	vol. %	D5599	<0.1
IBP	ºF	D86	82
T10	ºF	D86	109
T50	ºF	D86	194
T90	ºF	D86	342
FBP	ºF	D86	416
DVPE	psi	D5191	11.1
Aromatics	vol. %	D1319	26.2
Olefins	vol. %	D1319	7.7
Benzene	vol. %	D3606	0.95
S	mg/kg	D5453	15.9
(R + M)/2	-	Calc.	87.5
Net Heat of Combustion	Btu/lb	D4809	18,734

a. Effect of Oil Age on Emissions

A study completed by EPA and Lubrizol shortly before the start of this program examined the effect of oil age on emissions.[17] The study involved accumulating 2000 miles on two low-mileage, Tier-2-compliant vehicles using a non-ethanol fuel, with emission measurements taken immediately after oil change and at 500, 1000, and 2000 miles. The only significant effect of oil age observed was for PM, which dropped by nearly half after 500 miles in one of the vehicles. Both vehicles showed a general trend of PM reduction out to 2000 miles, though differences between each test interval were not large or statistically significant.

The study also looked at the effect of mileage accumulation using ethanol-blended fuel on emissions and oil quality. After the initial 2000-mile accumulation period on E0, the fuel

[17] Christianson, M., Bardasz, E., and Nahumck, B. *Impact of Lubricating Oil Condition on Exhaust Particulate Matter Emissions from Light Duty Vehicles*. Society of Automotive Engineers International Journal of Fuels and Lubricants, December 2010 issue 3: pp. 476-488.

supply was changed to E10, and another 3000 miles were accumulated. No significant changes were seen in emissions between the beginning and end of the period, and an analysis of oil quality (including metals, acid and base numbers) done by Lubrizol didn't show oil degradation beyond the normal range for the accumulated mileage.

Though this study was relatively small, it was intended to serve as a screening, confirming that 2000 miles of oil age should be sufficient to stabilize emissions, and that use of ethanol fuels didn't appear to cause unacceptable levels of oil degradation.

2. Vehicle Storage Conditions During the Program

Vehicles being actively tested were stored indoors in a temperature-controlled soak area adjacent to the test cell. All vehicles had batteries trickle-charged for at least 12 hours prior to testing. Due to the nature of the randomized test matrix, as well as the incremental addition of test vehicles to the program, certain vehicles were not involved in active testing for several weeks at a time. Those not scheduled to test for approximately two weeks or more were generally stored outdoors in a parking area near the test facility. In an attempt to minimize vehicle maintenance issues due to extended inactivity, those vehicles were operated by an experienced driver once every two weeks over an on-road course around the perimeter of the SwRI campus. Prior to each drive, each vehicle received a brief visual inspection to ensure proper tire inflation and fluid levels. One "lap" was completed, which was approximately 8 miles in length and about 20 minutes in duration. Speed limits ranged from 35 to 45 mph, and the drive included six traffic signals and two stop signs. This task was conducted using a non-ethanol fuel.

V. TEST PROCEDURES, DATA COLLECTION, ISSUES ENCOUNTERED

There are many things that can affect vehicle emissions performance, with the fuel effect potentially being a relatively small factor. Thus, an important consideration in fuel effects studies is the need to minimize measurement artifacts, which include variability, error, or bias in the results that are unintended in the design of the study. Any such artifacts become confounded with the fuel effect itself, which can lead to loss of statistical power to resolve an effect or, perhaps worse, detection of an effect that doesn't exist. As new vehicles have lower and lower emission levels, more and more sources of measurement artifacts have the potential to become influential in the results of the study. A great deal of effort was taken in the design and execution of this program to minimize artifacts related to procedures, including following a particular sequence of steps in vehicle handling before, during, and between emission tests, as well as use of the same driver and test cell for all emission tests. Such procedures are described in more detail in this section. (Another potential source of error in fuel effects models is fuel property measurement error; the fuels round robin process described in Section III was done to minimize this.)

A. Test Cycle, Conditions, and Schedule

All vehicle/fuel combinations were tested using the California Unified Cycle, also known as the LA92. This cycle was chosen because it contains higher speeds and acceleration rates representative of a wider range of typical driving behavior than what is covered in the FTP cycle. For this program, the LA92 was conducted as a three-phase, cold-start test in a manner similar to the FTP.[18] Test procedure quality controls included criteria for maximum deviation in speed and time from the test cycle driving trace to minimize effect of driver behavior on test-to-test variability.

Testing was conducted during the day shift while vehicle preparation, fuel changes, and conditioning were conducted during a second shift. All vehicle soaks and tests were conducted

[18] In order to supplement data being collected during Phase 4 of this test program, the four FFVs were also tested over the FTP cycle when operating on E85.

at a nominal temperature of 72°F. The representative bulk oil temperature of a vehicle's sump was stabilized to 72°F ± 3°F prior to conducting any emission test to minimize any effect of temperature on variability.

SwRI made a number of modifications and optimizations to the test cell air handling system during the initial phases of the program in an effort to maintain vehicle intake air humidity at 75 ± 5 grains H_2O/lb dry air while tests were being conducted, consistent with certification procedures in 40 CFR Part 86, to minimize the impacts on NOx emissions.[19] During Phase 3 and later portions of the program, this target was met 95% of the time, with some exceptions when outdoor weather conditions were rapidly changing. SwRI provided a humidity quality check metric within each individual test file and flagged entries in the test log where humidity was outside the desired range for more than five percent of the time. EPA and NREL provided guidance to SwRI regarding whether or not any individual test should be repeated.

Under Phase 1 of the program, SwRI determined and verified PM sample flow rates that provided proportionality. Those same flow rates were used for Phase 3. The CVS blower was turned on approximately 20 minutes before each emission test in an effort to ensure tunnel stability.

1. Test Matrix

The test matrix was designed to test each vehicle/fuel combination in a randomized order to minimize any effects of biases or artifacts that may not have been addressed through other provisions in design or procedures (e.g., possible effects of season, weather, changes in test fuel properties over time, vehicle or instrument drift, etc.). During the first nine weeks of testing, EPA specified partially randomized vehicle/fuel assignments that alternated between E0 and higher-ethanol blends in an effort to determine the amount of conditioning necessary to allow a vehicle's fuel control system to adapt to a new ethanol concentration. Development of the vehicle conditioning procedure is discussed in further detail in Section V.B. Once this issue was

[19] The calculation of NOx emission rates includes a humidity correction factor, but providing consistent test conditions are preferable to relying on this factor alone.

resolved, vehicle/fuel assignments were made using a spreadsheet tool that tracked which combinations had been tested and chose new assignments randomly from the remaining options.

Testing started with five E0 fuels as shown in Table V-1. Additional fuels were added to the test matrix based on both the requirements of the vehicle conditioning assessment and on fuel availability (fuel blending and delivery took several months). All fuels became available to the randomization algorithm by the 12th week of testing. Due to initial funding limitations, only ten vehicles were included in the original Phase 3 test plan. Two additional vehicles were added to the matrix in the 25th week of testing, and three additional vehicles were added in the 37th week of testing. These additional vehicles were added to the test randomization scheme with randomized fuel/vehicle pairings from the outset.

Table V-1. Schedule of addition of fuels and vehicles to the test matrix.

Phase 3 Week	Fuels Added	Vehicles[a] Added	Vehicle/Fuel Assignments
Week 1	2, 7, 8, 9 and 15	CCOB, TCAM, FEXP, DCAL, HODY	EPA
Week 2	None	CSIL, TSIE, DLIB, HCIV, NALT	
Week 3	None	None	
Week 4	1, 12, 13	None	
Week 5	None	None	
Week 6	22, 24	None	
Week 7	None	None	
Week 8	3, 4, 5, 11, 14, 16, 20, 21, 23, 30	None	
Week 9	None	None	
Week 10	None	None	Randomized for rest of program, except for E85
Week 12	6, 10, 25, 26, 27, 28, 31	None	
Week 25	None	FFOC, SOUT	
Week 37	None	CIMP, F150, TCOR	
Week 55	29 (E85)	DCAR	Last fuel tested
Week 60	End of Phase 3 testing		

[a] - Vehicle designations are explained in Section IV.

Due to imperfect test-to-test repeatability and the possibility of individual tests to significantly influence results, each vehicle/fuel combination was tested at least twice, back-to-back, with the second replicate usually performed on the following day. When replicates were split over weekends, an additional prep cycle was conducted as to maintain a 12-36 hr soak period. After two tests were completed and the acquired data passed all quality control verifications, the need for a third test was determined according to the variability criteria shown

in Table V-2. If the ratio of any of the specified pollutants (THC, NO_X, or CO_2) on a pair of tests for a given vehicle/fuel combination exceeded the levels shown in Table V-2, a third test was conducted and a note was made in the test log.

Table V-2. Repeatability criteria for third replicate.

Dilute Gaseous Emission	**Criterion For Requiring a Third Test (Composite Cycle Emissions)**
CO_2	Ratio of higher / lower > 1.03
NO_X	Ratio of higher / lower > 2.7
THC	Ratio of higher / lower > 2.0

These repeatability criteria were generated based on variance levels found in Phase 1 data, with a target of performing a third replicate for approximately 5% of fuel/vehicle pairs in Phase 3. This figure was chosen with the goal of covering a small number of measurements that might be statistical outliers.

Since emissions performance on current technology vehicles is dominated by what occurs shortly after start-up, engine cranking times between replicates were screened for inconsistency. If a test differed in cranking time from a previous replicate by more than one second, its procedure log and emissions data were reviewed by EPA and NREL to determine if an additional replicate should be performed.

In the end, additional replicates were performed for approximately 3% of vehicle/fuel combinations due to both repeatability and cranking criteria. (Some additional replicates beyond these were done as necessary to cover void tests or other procedural issues.) In the end, 926 fully valid tests were performed with all measurements as specified in the scope of work. Thirty additional tests had valid data for regulated pollutants but were missing other subsets of data (e.g., speciation) due to measurement or data quality issues; inclusion of these (denoted as "makeup tests" and containing an "x" in the test number) brings the total number included in the database to 956.

B. Fuel Change and Vehicle Conditioning Procedures

1. Learned Fuel Trim

The vehicles tested in this program all employed learned fuel adjustments (or trim) to continuously adjust the amount of fuel delivered for proper combustion. Most vehicle manufacturers began using such controls during the 1990s, and today nearly all new vehicles use microprocessor algorithms of varying sophistication to optimize vehicle performance and meet emission standards.

When the combustion process requires a change in air/fuel ratio, such as occurs when ethanol blend level changes, the engine controller must adjust the fuel trim to re-optimize engine and emission performance. This "re-learning" process requires operation of the vehicle for a certain period of time in several speed and load modes. Since this test program used multiple fuels with widely varying properties, sufficient prep procedures were needed to ensure emission behavior was stabilized after a fuel change.

During all testing and prep procedures, vehicles were connected to an on-board diagnostics (OBD) scanner to capture data for parameters of interest, including those related to fuel trim. For more discussion of OBD data collection, refer to section V.D.3.

A vehicle fuel change and conditioning procedure was initially developed during Phases 1 and 2. After those emissions and OBD data were analyzed, it was evident that the conditioning sequence was not sufficient for all vehicles to fully adapt after a change from E20 to E0 fuel. Therefore, the beginning of Phase 3 included a study (done concurrently with emission testing) to reassess and optimize the vehicle conditioning procedures. Long-term fuel trim (LTFT) and short-term fuel trim (STFT) OBD parameters were monitored during successive two-phase (bag 1-2) LA92 test cycles and were analyzed for stabilization. Based on the results of this study, the CCOB, NALT, HCIV, HODY, and TCAM were all conditioned with five successive two-phase

LA92s starting in the third week of testing. For all the remaining vehicles, three LA92s was sufficient. Prior to the third week, all vehicles were conditioned with three LA92 preps.

The final vehicle fuel change and conditioning procedure is given in Table V-3. OBD data, including LTFT and STFT, were collected during all conditioning runs and made available to program sponsors as part of the program metadata.

2. Sulfur Purge

It is well known that fuel (and lubricant) sulfur content affects the performance of the exhaust aftertreatment catalyst. Vehicles emitting at lower and lower levels of regulated pollutants tend to rely on increasingly active catalysts, and thus there was concern that even a modest amount of sulfur contamination over time could produce a drift in emissions performance of the test vehicles that would be confounded with the fuel properties being studied. Thus, a set of high-speed and load cycles were performed after each fuel change in an effort to reset the vehicle catalysts back to some relatively "clean" baseline. The specific procedure used was taken from Appendix C of the CRC E-60 program report and consists of a series of alternating high and low speed cruises with hard accelerations in between.[20]

[20] Durbin T.D., et al. (2003). *The Effect of Fuel Sulfur on NH_3 and Other Emissions from 2000-2001 Model Year Vehicles*, Appendix C. Report number E-60. Coordinating Research Council, Alpharetta, GA. Available at www.crcao.org.

Table V-3. Fuel change, conditioning, and test execution sequence.

STEP	DESCRIPTION
1	Drain vehicle fuel completely via fuel rail whenever possible. When switching to E85 only, drive vehicle to fully warm up engine.
2	Turn vehicle ignition to RUN position for 30 seconds (60 seconds when switching to E85) to allow controls to allow fuel level reading to stabilize. Confirm the return of fuel gauge reading to zero.
3	Turn ignition off. Fill fuel tank to 40% with next test fuel in sequence. Fill-up fuel temperature must be less than 50°F.
4	Start vehicle and execute catalyst sulfur removal procedure described in Appendix C of CRC E-60 Program report. Apply side fan cooling to the fuel tank to alleviate the heating effect of the exhaust system. Engine oil temperature in the sump will be measured and recorded during the sulfur removal cycle.
5[a]	Perform four vehicle coast downs from 70 to 30 mph, with the last two measured. If the individual run fails to meet the repeatability criteria established in Phases 1 and 2 of the program, the vehicle will be checked for any obvious and gross source of change in the vehicle's mechanical friction.
6	Drain fuel and refill to 40% with test fuel. Fill-up fuel must be less than 50°F.
7[b]	Drain fuel again and refill to 40% with test fuel. Fill-up fuel must be less than 50°F.
8	Soak vehicle for at least 12 hours to allow fuel temperature to stabilize to the test temperature.
9[c]	Move vehicle to test area without starting engine. Start vehicle and perform three 2-phase (bags 1 and 2) LA92 cycles. During these prep cycles, apply side fan cooling to the fuel tank to alleviate the heating effect of the exhaust system. Following the first two prep cycles, allow vehicle to idle in park for two minutes, then shut-down the engine for 2-5 minutes. Following the last prep cycle, allow the vehicle to idle for two minutes, then shut down the engine in preparation for the soak.
10	Move vehicle to soak area without starting the engine.
11	Park vehicle in soak area at proper temperature (75 °F) for 12-36 hours. During the soak period, maintain the nominal charge of the vehicle's battery using an appropriate charging device.
12	Move vehicle to test area without starting engine.
13	Perform LA92 cycle emissions test.
14	Move vehicle to soak area without starting the engine.
15	Park vehicle in soak area of proper temperature for 12-36 hours. During the soak period, maintain the nominal charge of the vehicle's battery using an appropriate charging device.
16	Move vehicle to test area without starting the engine.
17	Perform LA92 emissions test.
18	Determine whether third replicate is necessary, based on data variability criteria (see Table V-2).
19	If a third replicate is required, repeat steps 14, 15, 16 and 17.
20	If third replicate is not required, return to step 1 and proceed with next vehicle in test sequence.

a – Vehicle coastdown repeatability criteria referred to in Step 5 were provided by EPA as follows:

- maximum difference of 0.5 seconds between back-to-back coastdown runs from 70 to 30 mph
- maximum ±7 percent difference in average 70 to 30 mph coastdown time from the running average for a given vehicle

b – Some vehicles received only two fuel drains and fills, i.e. Step 7 was skipped. See section V.F.4 for details.

c – Conduct five 2-phase LA92 test cycles for the following vehicles: CCOB, NALT, HCIV, HODY, and TCAM.

3. Fuel Carryover in Vehicle Fuel Tanks

On May 27, 2009, (the 11th week of testing) SwRI noticed low levels of ethanol in exhaust speciation samples collected from the Nissan Altima on Fuel 13, which contained no ethanol. Samples were pulled from the drum used to fuel the vehicle, and from the vehicle's fuel tank, and analyzed with a PetroSpec analyzer. The drum sample showed no ethanol while the vehicle's fuel tank showed 1.5 vol% ethanol, suggesting ethanol-containing fuel had carried over from a previous test in the Altima. The fuel tank sample was sent to EPA for analysis by ASTM D5599 method and was found to contain 1.44 vol% of ethanol. This was equivalent to a fuel carryover rate of 7.2% following two drains and 40% fills, meaning that approximately 3 gallons of the previous fuel (an E20) remained in the Altima's tank after it has been drained via the fuel rail. From this point forward, except for mid- and end-point drift check tests, the Altima received three fuel flushes during the fuel change sequence.

Investigating further, SwRI examined the speciation results for other vehicles and found measurable levels of ethanol when testing with an E0 fuel that was immediately preceded by an E20 fuel. To better understand this situation, fuel samples were collected during tests where fuel changed from an E15/E20 to an E0 fuel. D5599 analyses of the samples by SwRI and EPA suggested that the following percentages of the previous fuel were retained in the tanks of the test vehicles following fuel changes consisting of two drains and 40% fills:

- Honda Odyssey: 8.8 vol%
- Toyota Sienna: 5.0 vol%
- Honda Civic: 4.2 vol%
- Nissan Altima: 6.1 vol%
- Toyota Camry: 5.3 vol%
- All remaining vehicles: 2.1 to 3.2 vol%

Based on these results, EPA and NREL directed SwRI to prepare several 95%/5% and 5%/95% blends of the test fuels with the most extreme combinations of distillation properties and ethanol content to determine the effect of 5% fuel carryover on T50, T90 and RVP. The fuel sampling procedure used during these experiments is given in Appendix G, while the test matrix

and findings are given in Appendix H. The results of these experiments generally suggested that significant (nonlinear) deviations of T50 and T90 would not be expected as a result of carryover, but that shifts in RVP as high as 0.5 psi could be expected for E0 fuels experiencing ethanol contamination.

Since the initial and subsequent carryover samples from the Altima were relatively inconsistent (3.7% vs. 7.2% carryover), SwRI performed additional refueling experiments with the Altima, Odyssey, Camry, Civic, and Sienna to determine the variability of the measurements. These experiments showed that a third fuel flush was effective in reducing fuel carryover to less than one percent. The procedure and results are given in Appendix I. Based on these results, starting on August 1, 2010, SwRI incorporated a third fuel drain and fill into the vehicle change procedure for the Altima, Odyssey, Sienna, Civic, and Camry to address larger carryover observed in their fuel systems.

Fuel carryover was characterized for the Focus, Outlook, Impala, F-150, and Corolla before they were added to the test matrix. Based on results, these five vehicles received triple drains and fills during fuel changes.

During investigation of fuel carryover, questions also arose about the impact of refueling location. Two sites were used during the drain and fill procedures, one with a noticeable slope toward the front of the vehicle, and the other toward one side. (Sloped pavement is typical in refueling areas, related to spill and storm water control.) Starting in August 2009, each vehicle was assigned a particular refueling location to attempt to limit any variability in carryover this may have caused. Prior to this, refueling may have occurred in either location. To assess the magnitude of effect of refueling location, additional experiments were conducted with the Silverado, Camry, Sienna, Caliber, Civic, Odyssey, and Altima. Results did not show any clear evidence of such effect. Additional details are given in Appendix J.

C. Crankcase Oil Level Monitoring

Crankcase oil levels were checked monthly to ensure vehicles were operating as expected and that sufficient volume was available for collection of samples for analysis. No abnormalities in oil levels were noticed, with the exception of the Ford Explorer as described in section F.1.

Oil samples of four ounces in volume were collected from each vehicle at five points during Phase 3: at the start and end of the 2,000-mile lubricant break-in (i.e., around odometer readings of 2,000 and 4,000 miles), and following emissions testing of the 3rd, 15th, and 27th fuels in the Phase 3 test sequence. The oil samples were shipped in batches to Lubrizol for analysis. To accommodate the oil samples taken over the course of the program, each vehicle's sump was overfilled by 12 ounces during the oil change at the mid-point of the 4,000-mile vehicle break-in. A summary timeline of oil changes and samples by approximate mileage is shown in Figure V-1, and details of dates and mileages at collection from each vehicle are given in Appendix F.

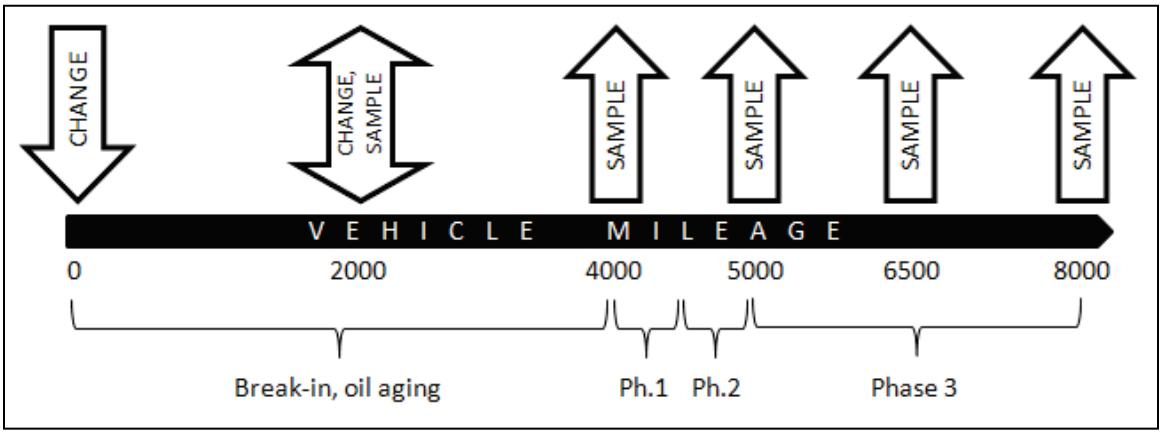

Figure V-1. Summary of oil changes and sampling by odometer mileage.

D. Emissions Measurements and Other Data Collection

Both bag-level and continuous emission measurements were collected during vehicle testing. On-board diagnostics (OBD) parameters relevant to engine operation and emissions were also recorded during testing.

All emission tests were conducted using a Horiba 48-inch single-roll electric chassis dynamometer, which simulates the loads of acceleration, deceleration, and drag experienced by a vehicle when driving a test cycle. The computerized dynamometer control system uses a pre-defined mathematical formula with coefficients specific to each vehicle to simulate its behavior on the road. Vehicle manufacturers generate target road load coefficients for a vehicle (called target coefficients), which can then be used to compute set coefficients appropriate create an accurate simulation in other laboratories. The set coefficients for this program were derived from targets reported by manufacturers in EPA's on-line certification database.[21] These values were approved by EPA prior to the initiation of testing, and are shown in Table V-4.

Differences in electrical and mechanical characteristics and calibration of dynamometer equipment between test sites could be a source of variability in engine load that a vehicle experiences during a test, and thus the emissions it produces. Similarly, the vehicle driver has considerable influence on behavior of the engine during transient portions of the test cycle, also affecting emissions. Therefore, in an effort to minimize variability within the dataset, a single test site and a single driver were used for all data collected in the program, something that is unusual for a program of this size. Different drivers were used for sulfur purges and vehicle conditioning. Tension in the tie-downs used to secure the vehicle against the dynamometer roll was also maintained within a specified range for each test to minimize variability in tire drag and rolling friction.

[21] More details on certification data are available at http://www.epa.gov/otaq/verify/index.htm

Table V-4. Vehicle chassis dynamometer settings.

MODEL YEAR	MAKE	BRAND	MODEL	NAME	ETW, lbs	TARGET COEFFICIENTS			SET COEFFICIENTS			ROAD LOAD HP @ 50 mph
						A, lbs	B, lbs/mph	C lbs/mph^2	A, lbs	B, lbs/mph	C lbs/mph^2	
2008	GM	Chevrolet	Cobalt	CCOB	3,125	21.51	0.5409	0.01521	4.22	0.20100	0.017055	11.5
2008	GM	Chevrolet	Impala FFV	CIMP	3,875	19.87	0.4397	0.01752	8.320	0.11210	0.018601	11.4
2008	GM	Saturn	Outlook	SOUT	5,000	38.61	0.3921	0.02818	19.860	0.07430	0.030294	17.2
2008	GM	Chevrolet	C1500 Silverado FFV	CSIL	5,500	28.80	0.8005	0.03219	18.130	0.31630	0.035662	19.9
2008	Toyota	Toyota	Corolla	TCOR	2,875	22.10	0.1500	0.01886	8.080	-0.02580	0.020902	10.2
2008	Toyota	Toyota	Camry	TCAM	3,625	29.16	0.1659	0.01844	10.110	-0.15630	0.019592	11.1
2008	Toyota	Toyota	Sienna	TSIE	4,500	38.41	0.0249	0.02946	16.270	-0.12110	0.029718	15.1
2008	Ford	Ford	Focus	FFOC	3,000	27.66	0.2892	0.01697	15.240	0.07660	0.018743	11.3
2008	Ford	Ford	Explorer	FEXP	4,750	32.35	0.6076	0.02716	14.350	0.43360	0.028153	17.4
2008	Ford	Ford	F150 FFV	F150	5,250	27.26	0.9495	0.02932	4.300	0.83540	0.029383	19.7
2008	Chrysler	Dodge	Caliber	DCAL	3,500	52.75	-0.3153	0.02826	15.990	-0.20400	0.025692	14.4
2008	Chrysler	Dodge	Caravan FFV	DCAR	4,750	35.94	0.6505	0.02155	18.470	0.30710	0.023981	16.3
2008	Chrysler	Jeep	Liberty	JLIB	4,250	29.53	0.4040	0.02955	9.410	0.13330	0.031781	16.5
2008	Honda	Honda	Civic	HCIV	3,000	23.18	0.1904	0.01699	8.120	0.05150	0.017724	10.0
2008	Honda	Honda	Odyssey	HODY	4,750	28.70	0.6915	0.02167	11.170	0.24850	0.024710	15.7
2008	Nissan	Nissan	Altima	NALT	3,500	47.47	-0.4531	0.02414	19.710	-0.30660	0.021358	11.4

An additional step taken to ensure data quality was the performance of multiple coast-down checks for each vehicle during each fuel change prep procedure (described in Table V-3, step 5). These checks involve bringing the vehicle up to 70 mph on the dynamometer and recording the time it takes to coast down to 30 mph while in neutral. This provides a screen for friction or miscalibration issues that can affect program results. If a difference was found of more than 0.5 s between two back-to-back checks, or more than 7 s from a running average, the results were flagged for follow-up investigation. These values were chosen based on criteria used for quality control in certification testing. While there were occasional deviations noted, no significant, reproducible issues were found with coastdown times.

1. Emission Measurements

The LA92 test cycle used in this program was broken into three portions, or bags, as shown in Figure V-2. The bag-level emissions measured and reported in all bags and replicates were THC, NMHC (by FID), NO_X, NO_2, CO, CO_2, and particulate mass. In addition to the dilute, bagged exhaust samples, continuous raw tailpipe exhaust mass emissions rates were measured on a second-by-second basis for all replicates for THC, CH_4, CO, NO_X, CO_2 and O_2. A subset of tests also had measurement of speciated alcohols, carbonyls, and hydrocarbons, as well as NMOG and NMHC values calculated to adjust for partial response of the test cell FID to oxygenated species. All tests and replicates also have reported composite values computed using

FTP weighting factors (43% bag 1+2 plus 57% bag 2+3), primarily as a matter of convention as no parallel factors exist for weighting portions of the LA92.[22]

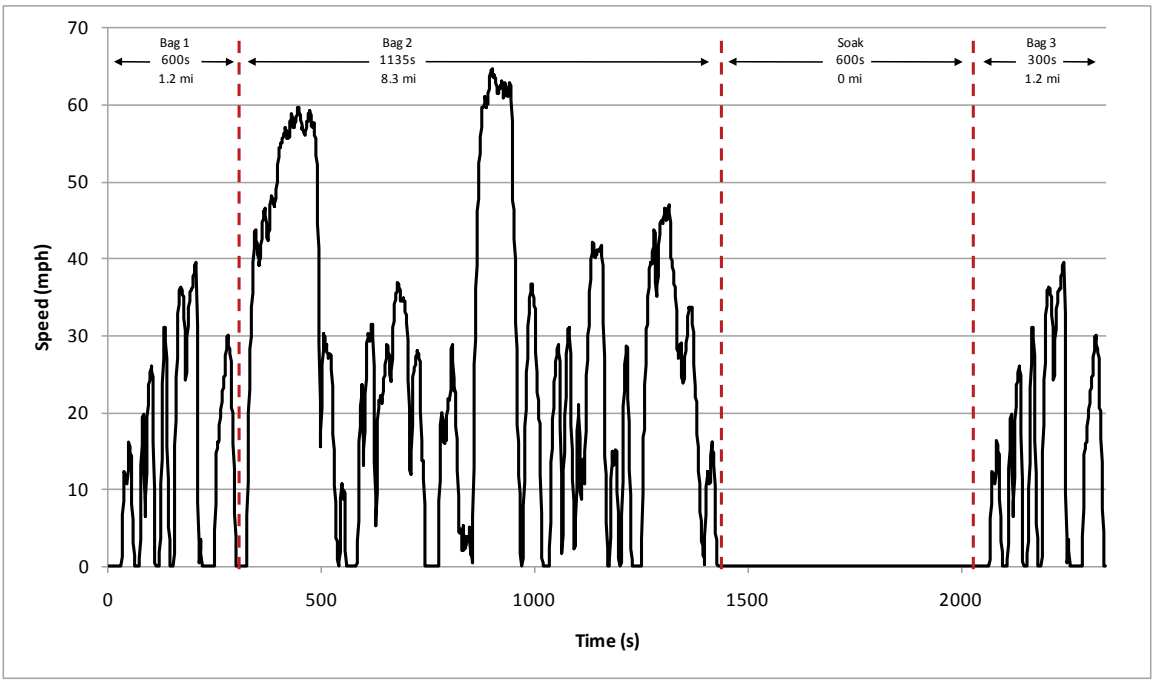

Figure V-2. Speed vs. time schedule of California Unified Cycle (LA92).

a. Methods

Gaseous emissions were determined in a manner consistent with EPA protocols for light-duty emission testing as given in the CFR, Title 40, Part 86. A constant volume sampler was used to collect proportional dilute exhaust in Kynar bags for analysis of carbon monoxide (CO), carbon dioxide (CO_2), total hydrocarbons (THC), methane (CH_4), and oxides of nitrogen (NO_X). Dilution air flow entering the sampling system was measured with a smooth approach orifice, and a critical flow venturi measured bulkstream dilute exhaust flow. Measured dilution air flow

[22] EPA performs inventory modeling using modeling techniques that break emission rates down by individual operating modes. Therefore the application of fixed weighting factors to emission bags to create a single composite result representing average driving is primarily done for historical reasons.

was subtracted from the bulkstream flow to calculate raw exhaust flow to determine continuous raw mass emission rates.[23]

During Phases 1 and 2 of the test program, continuous raw NH_3 measurements were made. Results showed NH_3 spikes of several hundred ppm during testing of many of the vehicles. These concentrations were sufficient to cause poisoning of the NO_2-to-NO converter in the continuous raw NO_X analyzer. In an attempt to minimize this problem, prior to the start of Phase 3 SwRI installed two NH_3 adsorbers in series upstream of the continuous raw emission measurement sample train. These adsorbers were changed daily. Additionally, the NO_2-to-NO converter was purged with 5,000-ppm (nominal) NO_X for five minutes following every test in an effort to reverse any NH_3 poisoning of the converter that may have occurred during testing. The NO_X analyzer was then purged for another three minutes with zero nitrogen prior to initiating the normal pre-test zero-span sequence.

For the measurement of PM (particulate matter) mass emissions, a proportional sample of dilute exhaust was drawn through Whatman Teflon membrane filters after secondary dilution with HEPA-filtered air. A single filter was used for each bag, with both clean and dirty filters being weighed three times after a period of stabilization in a temperature and humidity-controlled clean room. Replicate weights were required to be within ±2 μg or the result was void. The PM sampling method was compliant to CFR, Title 40, Part 1065. Several parameters, such as filter face velocity and temperature and dilution ratio, were monitored and reported for each test.

Sampling and analysis of alcohols was done in a manner similar to CARB method 1001, "Determination of Alcohols in Automotive Source Samples by Gas Chromatography". Dilute exhaust was bubbled through glass impingers containing deionized water. Immediately following the test, samples were sealed and stored below 40°F until analysis. Most samples were analyzed on the day they were collected, but no later than within six calendar days.

[23] An electronic flow measurement device (referred to as an EFM) was used to measure raw exhaust flow during Phase 1 of the program, and results were found to be sufficiently similar to the SAO subtraction method. Given this fact, as well as cost and maintenance issues related to the EFM, it was not used in Phase 3.

Sampling and analysis of carbonyl compounds was conducted in a manner similar to CARB method 1004, "Determination of Aldehyde and Ketone compounds in Automotive Source Samples by High Performance Liquid Chromatography". Dilute exhaust was drawn through cartridges containing DNPH, a compound that selectively binds to carbonyl groups to form stable complexes with known properties. The DNPH media were extracted using acetonitrile within 15 minutes of collection, and the extracts sealed and stored immediately at a temperature below 40°F. Most of these extracts were analyzed on the day they were collected, but no later than within three calendar days. An effort was made to detect the presence of a tautomer of acrolein, acrolein-x, which can be a measurement artifact. No acrolein-x was found in any exhaust sample. Additionally, storage of alcohol and carbonyl samples was segregated to prevent any cross-contamination of samples.

During carbonyl speciation work in Phases 1-2 of the program, SwRI noticed that background levels of some species (e.g., acetone and acetaldehyde) were relatively high and/or shifted in time in ways that were suspicious. SwRI began tracking daily media blanks and found that new, unused cartridges contained background levels of some species that were similar to the levels found in dilute exhaust. Thus, a protocol was developed for determining limits of quantitation (LOQs) of results based on media blank levels. These LOQs were determined for each compound on each test day using recent blank measurements. A detailed description of this process is given in Appendix L.

Sampling and analysis of C_2-C_{12} hydrocarbons was conducted in a manner similar to CARB method 1002/1003, "Procedure for the Determination of C_2-C_{12} Hydrocarbons in Automotive Exhaust Samples by Gas Chromatography". During the analysis of C_2 - C_4 hydrocarbons, special consideration was given to 1,3-butadiene. Because of the instability of 1,3-butadiene, the analysis of C_2 - C_4 hydrocarbon samples collected during Bag 1 of a test cycle was initiated within one hour of collection. The speciation of C_5 - C_{12} hydrocarbon samples collected in Bag 1 of the test cycle was completed within 4 hours of collection.

The following daily sequence was used for the analysis of VOC samples:

- VOC samples collected during Bag 1 of the test cycle were analyzed first, in the sequence of vehicle tests.
- If a vehicle requiring VOC sampling during all three bags of the test cycle was tested, the Bag 1 was analyzed first, followed immediately by the Bag 3 sample and finally by the Bag 2 sample.
- Background samples were analyzed last, in the sequence of vehicle tests.

A summary of measurement methods is shown in Table V-5. Additional details on measurement equipment and related QA procedures are given in Appendix K.

Table V-5. Exhaust emission measurement methods.

Constituent	**Analysis Method**
Total hydrocarbon	Heated flame ionization detector (bag, modal)
Methane	Gas chromatography (bag, modal)
Carbon monoxide	Non-dispersive infrared analysis (bag, modal)
Carbon dioxide	Non-dispersive infrared analysis (bag, modal)
Oxides of nitrogen	Chemiluminescence analysis (bag, modal)
Nitric oxide	Chemiluminescence analysis (bag only)
Oxygen	Magnetopneumatic detector (modal only)
Particulate matter	Part 1065 gravimetric measurement (bag only)
Non-methane hydrocarbons	Calculated from THC and CH_4 (bag, modal)
Non-methane organic gases	Calculated as specified in section VI.B (bag only)
Nitrogen dioxide	Calculated from difference of NO_x and NO (bag only)
$C_1 - C_{12}$ HC speciation	Gas chromatography (bag only)
Alcohols	Gas chromatography (bag only)
Carbonyls	Liquid chromatography (bag only)

b. Schedule of Speciated Emission Measurements

Speciated volatile organic compounds (VOCs) measured in this program included C_1 - C_{12} hydrocarbons, light alcohols, and carbonyls (aldehydes and ketones). Due to the additional time and expense required, the scope of speciation covered a subset of vehicles and fuels. Alcohols and carbonyls were speciated during bag 1 for all tests (vehicles, fuels, replicates). C_1 - C_{12} hydrocarbons were speciated for the first replicate on all vehicles for a subset of twelve fuels (3, 4, 6, 7, 10, 13, 14, 21, 23, 27, 28, and 31) chosen to provide, as nearly as possible, useful comparisons between different levels of ethanol, aromatics, T50, and T90. In addition, all types of speciation were carried out for bags 2-3 for the subset of 12 fuels on a subset of five vehicles (Civic, Corolla, F150, Impala, Silverado) intended to represent the range of sizes and technologies present in the larger test fleet. This information is summarized in Tables V-6 and V-7. All types of speciation were also carried out for all bags of all E85 tests (fuel 29).

Table V-6. Speciation schedule by fuel, vehicle, and bag (excluding E85 tests).

Vehicle	Speciation Type	Replicate 1		Replicate 2+	
		Bag 1	Bags 2-3	Bag 1	Bags 2-3
CIMP, CSIL, F150, HCIV, TCOR	Alcohols, Carbonyls	All fuels	Subset shown in Table V-7	All fuels	-
	Hydrocarbons	Subset shown in Table V-7	Subset shown in Table V-7	-	-
All others	Alcohols, Carbonyls	All fuels	-	All fuels	-
	Hydrocarbons	Subset shown in Table V-7	-	-	-

Table V-7. Summary of fuel properties for speciation subset (excluding E85 tests).

Fuel	Ethanol vol%	T50 °F	T90 °F	DVPE psi	Aromatics vol%
3	10.4	218	296	6.9	15.0
4	9.9	222	338	10.0	15.5
6	10.6	189	340	7.2	15.0
7	<0.10	193	298	7.2	17.0
10	9.8	217	340	7.1	34.0
13	<0.10	223	338	6.9	34.1
14	<0.10	193	339	7.1	16.9
21	20.1	168	305	7.1	35.5
23	20.3	163	338	6.8	15.9
27	14.9	222	340	7.0	14.9
28	15.0	217	299	6.9	34.5
31	20.1	167	325	7.0	35.5

2. OBD Data Collection

Onboard diagnostic (OBD) system data were acquired at 1 Hz from each vehicle during all emissions tests using a DBK70 data acquisition system. On rare occasions, OBD data collection failed during a test; this was not grounds for repeating a test. This dataset includes the following parameters:

- Engine RPM
- Vehicle speed
- Engine load
- Short term fuel trim-bank 1
- Long term fuel trim-bank 1
- MIL status
- Absolute throttle position
- Engine coolant temperature
- Short term fuel trim-bank 2
- Long term fuel trim-bank 2
- Fuel/air commanded equivalence ratio

- Alcohol fuel percent (if available)
- Manifold absolute pressure
- Spark advance
- Engine control module voltage
- Air flow rate from mass air flow sensor

3. Data Quality Control

a. SwRI Procedures

A number of automated data quality checks were used by SwRI during collection and processing of data. The individual test files contain a QA tab showing a number of quantitative checks including: zero/span results for emission analyzers; PM filter weight gains, face velocities, and temperatures; bag-modal emission mass agreement; status of OBD channels on the vehicle CAN bus; test cell humidity. The test log file shows the status of additional checks such as repeatability ratio for requirement of a third replicate, long crank time flag, and pass/fail for chemistry and PM analyses.

b. EPA Procedures

Typically within about one week of completion of a test, SwRI would place the data file on a secure FTP site for review by the program sponsors. On approximately a weekly basis, EPA staff loaded the new data files into a database and ran various queries to look for other potential data quality anomalies. Any questions or issues were then raised during the weekly conference call with SwRI and other involved parties.

E. Drift Check Tests

Measurements may be affected by any of a variety of sources of drift, generally understood to be a systematic or progressive shift in results due to vehicle wear, instrumentation calibration changes, fuel weathering over time, etc. A rigorous scheme for detecting drift in a

test program such as this one would involve a large amount of redundant testing, for instance adding replicates for each vehicle/fuel combination at several points throughout the program. Needless to say, this would have increased cost and effort significantly, and did not seem justified given the quality control procedures already in place to minimize variability, as well as the fact that vehicle/fuel combinations were being tested in a randomized sequence. Nonetheless, as a screen for any gross drift that might affect the program results, each vehicle was re-run on a fuel from early in its sequence again at the mid-point and end.

Due to concerns with vehicles properly adapting to different ethanol contents in the test fuels, the following drift check procedure was conducted. To ensure that the test vehicles were similarly adapted during start-, mid-, and end-point testing, the test matrix was manipulated so the immediate history prior to mid- and end-point vehicle testing was substantially similar to that at the beginning of Phase 3. Specifically, all vehicles were operated on two successive E0 fuels for the first three weeks of testing, which had been immediately preceded by operation on an E20 fuel at the end of Phase 2 of the program. The second E0 fuel for each vehicle was designated as the drift check fuel. With the assistance of EPA, SwRI scheduled mid- and end-point testing to be immediately preceded by an E20 or E15 fuel and then an E0 fuel with properties substantially similar to the first Phase 3 fuel on which each vehicle was tested.

Due to the scheduling of end-point drift checks, five of the original ten vehicles completed testing during Week 34, while the remainder of the original ten vehicles completed testing during Week 37. (The Ford Explorer did not complete end-point testing until much later in the program due to a MIL issue described in Section V.F.2.)

Various statistical analyses were conducted, including graphical assessment of emission trends, modeling measurements as a function of odometer reading, and modeling measurements as a function of the time of test (beginning, middle, end).

No consistent drift in time-of-test measurements was found. For example, in 120 pairwise comparisons of beginning, middle and end measurements (10 measurements × 4 phases × 3 pairwise tests) using Tukey multiple-comparison-adjusted p-values, only one comparison

was found to be statistically significant: bag 1 CH4 means between beginning and end test runs. This sole significant result could easily have occurred by chance. Therefore, drift in emission behavior did not appear to occur in the test program.

F. Issues encountered with Specific Vehicles

1. Ford Explorer Oil Issues

An incorrect oil viscosity was used in the Ford Explorer during break-in. Ford specifies 5W-30 grade for the 4.0L V-6 engine and 5W-20 for the 4.6L V-8. The test vehicle was equipped with the 4.0L V-6, and was incorrectly filled with the 5W-20 oil at both the start of mileage accumulation and at the 2,000-mile oil change. The vehicle had accumulated 4,000 miles when this error was discovered. After discussing this situation with all involved parties, the vehicle received a single flush with 5W-30 oil (2 drains and 2 fills with oil filter changes) and an additional 2,000 miles were accumulated on the Ford Explorer to break-in the correct oil.

There also appeared to be an oil level issue with the Explorer. When the oil sample was collected following testing of the 15th fuel, the technician noticed that the oil level was below the minimum oil level on the dip stick. Following extensive discussions with all sponsors, an additional 20 ounces of fresh crankcase lubricant were added to the Ford Explorer before resuming testing. Details of this incident are given in Appendix M.

As a result of this situation, starting in the 22nd week of Phase 3, the oil level on all vehicles was checked monthly. These checks were taken on a level floor inside the emissions lab following a minimum 12-hour soak at room temperature (72°F ± 2°F). Initial results showed the Toyota Camry oil level was between 1/4 and 1/8 of the distance from the fill level to the full level on the dipstick. The oil level of this vehicle was monitored weekly, but did not change during the rest of the program. No other vehicles had oil level issues.

2. Fuel System Evaporative Leak Check Failures (Explorer)

During the 27th week of testing, the Ford Explorer illuminated a malfunction indicator light (MIL – a.k.a. “check engine light”) for diagnostic trouble code (DTC) P0455-Evaporative Emission System Leak Detected (Gross Leak/No flow). This started a series of troubleshooting events that are summarized in Table V-7. On-road testing seemed to indicate that code was not due to the fuel change procedure or operation of the vehicle on the chassis dynamometer. Following extensive discussions that included input from Ford technical staff, the team decided that the MIL would not have an adverse affect on emissions testing, and the vehicle was placed back into the test matrix.

Table V-7. Troubleshooting of the Ford Explorer evaporative system MIL

DATE	ACTION
9/16/2009	Fuel change to E20 Fuel 31; key off
9/17/2009	MIL light during vehicle conditioning, E20 Fuel 31; PO455-Evaporative Emission System Leak Detected (Gross Leak/ No Flow)
9/23/2009	Vehicle sent to dealer; performed a smoke test; Canister vent solenoid replaced
9/26/2009	Fuel change to E20 Fuel 21; key off
9/27/2009	MIL light during vehicle conditioning, E20 Fuel 21; PO455-Evaporative Emission System Leak Detected (Gross Leak/ No Flow)
10/5/2009	Vehicle sent to dealer; performed a smoke test; capless fuel filler door was cleaned as it had dirt and grime
10/12/2009	Fuel change to E20 Fuel 21; key off
10/13/2009	MIL light during vehicle conditioning, E10 Fuel 12; PO455-Evaporative Emission System Leak Detected (Gross Leak/ No Flow)
10/16/2009	SwRI performed an IDS test and a smoke test and a leak test by pressurizing the evap system and found no leaks.
10/26/2009	FEXP was taken to the test track where we ran 9 WOT up to 70 mph. The MIL did not light. The next day we ran through the three LA 92 (2-bag) prep sequence on the dyno. The MIL did light approximately 500 seconds (~24 miles) into the third LA 92. This means the fuel change procedure is probably not the cause for the MIL.
10/28/2009	Vehicle was driven on road approximately 50 miles. Pending code P0422, but no MIL light
11/20/2009	Vehicle sent to the dealer; The EVAP system was smoke tested and the capless fuel assembly was replaced. I was told that the technician drove the vehicle for more than 10 miles to confirm that the code did not reappear.
11/21/2009	Fuel change to E15 Fuel 28; key off
11/22/2009	MIL light during vehicle conditioning, E15 Fuel 28; PO455-Evaporative Emission System Leak Detected (Gross Leak/ No Flow)
12/1/2009	Dealership performed IDS Diagnosis, PO455 code. EVAP test found capless retainer broken. Replaced retainer and retested ok.
12/8/2009	MIL light during vehicle conditioning, E15 Fuel 28; PO455-Evaporative Emission System Leak Detected (Gross Leak/ No Flow)

Following Phase 3 testing, SwRI performed additional evaporative system leak checks with an IDS scan tool per instructions given by Ford technical staff, and the data files were forwarded to Ford for review. Subsequent coordination among SwRI, Ford technical staff, and the local Ford dealership allowed us to determine that the fuel tank pressure sensor had an internal fault causing the signal to become erratic during vehicle operation, after which the vehicle was repaired.

3. Transmission Module Malfunction (Outlook)

A MIL illuminated during the second test of Fuel 16 with the Saturn Outlook. The codes found were Tran Control Sys Malfunction and U0073 - Control Module Comm. Bus Off. The MIL and same codes had occurred previously while operating the vehicle on the mileage accumulation dynamometer during the initial vehicle break-in. At that time, the vehicle was taken to the dealership where the codes were cleared, and the MIL did not recur until testing Fuel 16.

Review of the emissions results from the two tests on Fuel 16 did not show a significant difference. With EPA's and NREL's approval, the codes were cleared and the vehicle was placed back into the test program. Four days later, the MIL came on again during testing, with the same DTCs. This time the driver noticed that the vehicle's engine was revving higher than usual at cruising speeds and shifting hard during the first two bags. Bag 3 did not have the same issues. With EPA's and NREL's approval, the vehicle was taken to the dealer for diagnosis, but they were not able to find any problems. There was concern that the DBK system used to collect OBD data may have somehow been interfering with proper vehicle operation. The next set of tests was conducted without OBD data acquisition, and the MIL did not illuminate. All subsequent tests were conducted without OBD data acquisition, and the MIL did not illuminate.

VI. DATA REPORTING

A. Products and Formats

This program generated a large amount of primary data covering fuels, emissions, and test conditions, available as follows:

- Individual test files (956 Microsoft Excel files, 3.6 MB each)
- Phase 3 testing log file (1 Microsoft Excel file, 845 KB)
- Phase 3 E85 testing log file (1 Microsoft Excel file, 37 KB)
- Test vehicle fleet data file (1 Microsoft Excel file, 46 KB)
- Fuel property data file with round robin results (1 Microsoft Excel file, 58 KB)
- Fuel speciation results (28 PDF files, 270 KB each)

Summary database files are also available covering gaseous, PM and speciation results.

1. Overview of Individual Test File Contents

The individual test files were produced in Microsoft Excel 2003 format, and use a standardized layout of six tabs with consistent row/column locations of particular data fields across all files. A summary of the contents of the six spreadsheet tabs is as follows:

Output-BagSummary

- Pretest data: date, time, test number, vehicle and fuel identification
- Test conditions: phase times, distances, test cell temp, humidity, crank times
- Emission results: concentration and g/mi results by bag and weighted for all pollutants

Output-QA

- Data and process quality checks, including: bag-to-modal comparison, PM collection parameters, analyzer zero/span check results, OBD CAN data channel status by parameter, test cell humidity time in-spec, average power and work by bag

Output-UnregSummary

- Summary of min/max/average by phase of modal data for various species and pollutants
- Note: This tab was assembled during Phase 1 when additional modal speciation was being done; for Phase 3 only "TP Raw" results are valid here (others may contain zeros or other values that are not necessarily accurate).

Output-ModalData

- Second-by-second measurements of OBD and CVS parameters, tailpipe raw concentrations, computed mass emission rates and cumulative totals

Chemistry Summary

- Mass and concentration data for alcohol, carbonyl, and gaseous hydrocarbon data, as available for a given test, after filtration by LOQs
- Limits of quantification (LOQs) applied to speciation data
- Concentration results for the subset of compounds being used to calculate NMOG results, after filtration according to LOQs

DataProcessingNotes

- File review status
- FID response factors used in NMOG calculations
- Miscellaneous notes on procedure and calculations

2. Treatment of Zeros and Missing Values

Speciation results in the *Chemistry Summary* sheet that were reported as zero after falling below the LOQ are denoted as "-99". In other sheets, non-detects are simply reported as a numerical zero value. Missing data omitted for some reason other than a non-detect, such as due a voided sample, are indicated with the period character (".").

B. Calculation of NMOG and NMHC

NMOG (non-methane organic gases) is defined as the sum of all organic emission species minus methane. It can be compared to NMHC (typically defined as FID THC minus methane), which contains an incomplete accounting of oxygenated emission species like formaldehyde, acetaldehyde, and ethanol due to the way the hydrocarbon analyzer operates.[24] NMOG results can be generated either by fully speciating the exhaust and summing the masses of all compounds, or by starting with a FID-based NMHC result and then correcting it using speciation results for alcohols and carbonyls. A non-oxygenated NMHC total can be generated in the same way. Where alcohol and carbonyl speciation results were present (bag 1 in nearly all tests, and bags 2-3 in a subset per Table V-6), SwRI generated NMOG and non-oxygenated NMHC results based on corrected FID measurements as described in detail in Appendix N. For tests where there was no speciation, the calculation was performed assuming oxygenated species masses were zero, effectively setting the NMOG and NMHC values equal to the traditional NMHC definition of FID THC minus methane.

[24] See Appendix P for details on FID response to oxygenated compounds.

VII. SUMMARY AND CLOSING REMARKS

This program collected regulated and unregulated emission data, by bag and second-by-second, for 15 new, properly-operating light duty vehicles of 2008 model year using a statistically-designed partial factorial matrix of 27 gasoline test fuels covering typical market ranges of ethanol, T50, T90, aromatics, and RVP. Complete data were generated for 926 tests, with 30 additional tests containing valid measurements for most emissions.

During the execution of this program a number of questions about best practices for emission studies were raised, and experiments were carried out to answer some of them. Observations include:

- New vehicles contain sophisticated engine control systems that can have significant effects on emission behavior. Specific fuel change and prep procedures must be followed to isolate to the greatest extent possible the impacts of fuel changes from transient behaviors resulting from learning processes occurring in engine controls.
- Vehicle fuel tanks vary widely in how much fuel remains after being drained by the fuel pump, which can result in fuel carryover and increased variability of actual in-tank fuel properties during testing. Careful fuel change procedures can accommodate this.
- As vehicle emission levels get lower and lower, more care must be taken to reduce sources of measurement error and variability. Speciation of carbonyls was one area found to be especially subject to this issue since DNPH cartridges contain background levels for some species of interest similar in magnitude to dilute emission levels. In addition, dilute gaseous emission measurements such as NOx may have sample concentrations similar in magnitude to background for some portions of the test (e.g., bags 2-3).