# Dispensing Equipment Testing With Mid-Level Ethanol/Gasoline Test Fluid

**Summary Report** 

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# **Executive Summary**

The National Renewable Energy Laboratory's (NREL) Nonpetroleum-Based Fuel Task is responsible for addressing the hurdles to commercialization of fuels and fuel blends such as ethanol that are derived from biomass. One such hurdle is the unknown compatibility of new fuels with current infrastructure, such as the equipment used at service stations to dispense fuel into automobiles. The U.S. Department of Energy's (DOE) Vehicle Technology Program and the Biomass Program have engaged in a joint project to evaluate the potential for blending ethanol into gasoline at levels higher than the present allowance of nominal 10 volume percent (E10).

This project was established to help DOE and NREL better understand any potentially adverse impacts caused by a lack of knowledge about the compatibility of the dispensing equipment with ethanol blends higher than what the equipment was designed to dispense. This report provides data about the impact of introducing a gasoline with a higher volumetric ethanol content into service station dispensing equipment from a safety and a performance perspective.

The project consisted of testing new and used equipment harvested from the field (all equipment UL listed for up to E10). Testing was performed according to requirements in Underwriters Laboratories Inc. (UL) Outline of Investigation for Power-Operated Dispensing Devices for Gasoline and Gasoline/Ethanol Blends With Nominal Ethanol Concentrations up to 85 Percent (E0-E85), Subject 87A, except using a CE17a test fluid based on the scope of this program. The primary focus was to identify leakage and assess other safety-related equipment performance as addressed by applicable UL requirements.

The overall results of the program were not conclusive insofar as no clear trends in the overall performance of all equipment could be established. New and used equipment such as shear valves, flow limiters, submersible turbine pumps, and hoses generally performed well. Some new and used equipment demonstrated a reduced level of safety or performance, or both, during either long-term exposure or performance tests. Dispenser meter/manifold/valve assemblies in particular demonstrated largely noncompliant results. Nozzles, breakaways, and swivels, both new and used, experienced noncompliant results during performance testing. Responses of nonmetals, primarily gaskets and seals, were involved with these noncompliances.

# **Acronyms and Abbreviations**

ASTM ASTM International

CE17a Test fluid composed of predetermined amounts of aggressive ethanol and

ASTM Reference Fuel C

EPA U.S. Environmental Protection Agency

DOE U.S. Department of Energy

NREL National Renewable Energy Laboratory

SAE Society of Automotive Engineers

UL Underwriters Laboratories Inc.

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## Introduction

## Background

The National Renewable Energy Laboratory's (NREL) Office of Deployment and Industry Partnerships and the Center for Transportation Technologies and Systems' Fuels Performance Group are responsible for addressing the hurdles to commercialization of fuels and fuel blends such as ethanol that are derived from biomass. One such hurdle is the unknown compatibility of new fuels with current infrastructure, such as the equipment used at service stations to dispense fuel into automobiles.

According to the U.S. Energy Information Administration, as of 2008 there were almost 162,000 retail gasoline outlets in the United States.<sup>1</sup> The equipment now in use consists of products from various manufacturers (some of which are no longer in business), of varying ages, maintained to varying degrees using different processes. The potential responses of the legacy base of installed fuel dispensing equipment to different fuel compositions such as E15 are unknown.

#### **Purpose**

This project used a systematic method to evaluate the performance of fuel dispensing equipment when exposed to a defined test fluid. The tests provide a methodology for assessing the equipment response to the predetermined test conditions, with a focus on loss of containment (leakage) and other safety-related performance issues.

In the equipment design process, materials are selected based on particular design considerations and performance requirements for the system. A key aspect of the selection is the compatibility of the materials (metals, plastics, and elastomers) with the fuel to which it will be exposed. Thus, an effective selection process is based on a comprehensive understanding of the material's mechanical, physical, and chemical properties. These materials are selected and used to produce component parts of equipment. The intended use of the equipment is a critical parameter for defining the required performance with regard to specific attributes.

In the case of fuel-dispensing equipment, materials that were selected—based on a characteristic compatibility with gasoline and gasoline/ethanol blends up to E10—may not exhibit the same compatibility with different fuel compositions. This program systematically evaluated the response of fuel dispensing equipment to exposure to ethanol/gasoline fuels with higher ethanol content by performing testing in the form of accelerated long-term exposure and subsequent assessment or safety performance.

Tests were conducted on new (previously unused) samples of equipment listed for gasoline and E10 use, and on used equipment that dispensed gasoline or E10 in the field. For harvested equipment, this testing was conducted to reflect a "second life" in dispensing a new fuel.

### **Test Items and Methods**

#### **Test Items**

NREL identified and procured the equipment to be tested. Samples were subsequently delivered and prepared for test at the Underwriters Laboratories (UL) facility. A labeled photo of fueling equipment is available in Appendix B.

#### Selection

NREL identified test items based on discussions with a variety of stakeholders with knowledge of the practical use of fuel dispensing equipment. Stakeholders provided information about the prevalence of particular equipment in the marketplace, and about installation and maintenance conditions and experience. After their input was gathered and evaluated, specific pieces of equipment were targeted as preferred test items for the testing program.

Equipment samples of identified test items were obtained for testing from various sources. Used equipment was obtained from the marketplace based on availability. The used dispensers were employed in different geographic locations for varying durations and may have been subjected to variable levels of maintenance.

The selected test items were listed for use with gasoline and E10. The legacy standards used to evaluate these products specify the use of ASTM Reference Fuel H test fluid (85% ASTM Reference Fuel C and 15% nonaggressive ethanol).

## Preparation

All samples were provided with closures to effectively seal all openings. Dispenser samples were modified to reduce their height to fit in the test chamber and to maximize test chamber space to generate data. Size reduction methods were selected to preserve as much as possible the integrity of the manufacturers' assembled connections, joints, seals, and structure

Dispenser samples were configured for the Long-Term Exposure test with hanging hardware to simulate practical use and promote test efficiency. The hanging hardware consists of the breakaway coupling, flexible hose, swivel, and hose nozzle valve. After the Long-Term Exposure test, these samples were disassembled to perform applicable performance testing on the required equipment.

#### **Test Methods**

Test methods were based on established, recognized protocols that were modified to address the specific focus of this program.

#### Test Fluid

The tests were conducted using CE17a test fluid, as defined by NREL. The test fluid was based on the same standard used to evaluate material compatibility for flexible-fuel vehicles. A 17% ethanol volumetric concentration was selected to address E15 use. This was not a commercial fuel, but rather a test fluid selected for research purposes.

CE17a test fluid consists of a mixture of 83% ASTM Reference Fuel C and 17% aggressive ethanol. Reference Fuel C is a 50/50 v/v blend of isooctane and toluene. Aggressive ethanol as defined in SAE Publication J1681, Gasoline, Alcohol, and Diesel Fuel Surrogates for Materials Testing,<sup>2</sup> is a mixture of synthetic ethanol and the following aggressive elements in defined amounts: deionized water, sodium chloride, sulfuric acid, and glacial acetic acid. The added elements are representative of contaminants found in ethanol. The test fluids were prepared the same day they were used.

## Test Methodology

Tests were conducted in accordance with the applicable methods specified in the Outline of Investigation for Power-Operated Dispensing Devices for Gasoline and Gasoline/Ethanol Blends With Nominal Ethanol Concentrations up to 85 Percent (E0-E85), Subject 87A,<sup>3</sup> except for the use of the CE17a test fluid. The testing methodology was developed with significant industry participation. These test criteria are defined to address reasonable safety of the equipment, focusing on loss of fuel containment and other safety-critical performance such as loss of ability to stop fuel flow or failure of breakaway couplings to separate at appropriate forces.<sup>4</sup> A brief summary of the test protocols follows; unless otherwise noted, references are to UL Subject 87A:

- Long-Term Exposure Section 29. Samples were filled with test fluid and placed in a 60°C ± 2°C chamber for 2,520 hours. A 50 psi leakage test was conducted weekly and the test fluid was replaced with fresh test fluid. Extracted test fluids were retained for subsequent analytical testing from one new and one used dispenser of similar design. Following Long-Term Exposure testing, samples were subjected to applicable performance tests depending on equipment type.
- High-Pressure Leakage Test Section 30. Samples were subjected to a hydrostatic or aerostatic pressure of 150% of the rated value, but not lower than 75 psi.
- Meter Endurance Section 31. Meter samples were operated at rated pressure for 300 hours, and then subjected to a leakage test at 150% of rated pressure, but not lower than 75 psi.
- Endurance Test Pumps: Section 32. Pump samples were operated at the maximum discharge pressure developed by the pump for 300 hours.
- Hydrostatic Strength Test Section 34. Samples were exposed to an internal hydrostatic pressure of 250 psi for 1 minute.
- Leakage and Electrical Continuity Test Section 35. Hose samples were pressurized and the electrical resistance was measured.
- Hose Bending Test (Filled) Section 36. Hose samples were filled with test fluid and subjected to a defined bending process for 3,150 cycles per day for 6 days.
- Low-Temperature Test Section 37. Hose samples were filled with test fluid for conditioning for a specific duration, then drained and capped. Following the conditioning, the samples were placed in a chamber at –40°C to ± 2°C for 16 hours, and subsequently bent around a mandrel with defined properties.

- Seat Leakage Test Breakaway Couplings: Section 38. Breakaway coupling samples were uncoupled and subjected to a hydrostatic or aerostatic pressure of 150% of the rated value for 1 minute. The test was then repeated with a pressure of 0.25 psi.
- Operation Test Electrically Operated Valves: Section 39. Electrically operated valve samples were connected to a test fluid system under rated pressure with the valve in the open position and fluid flowing, then the valve was closed to determine if there was continued fluid flow.
- Electrical Continuity Test Section 42. The electrical resistance across the element was measured.
- Pull Test Breakaway Couplings: Section 43. Breakaway coupling samples were subjected to a pull force to verify that they would separate at a force value not more than the rated value and not less than 100 pounds.
- Endurance Test Breakaway Couplings: Section 44. Reconnectable breakaway coupling samples were subjected to 100 cycles of separation and reconnection.
- Operation Test Swivel Connectors: Section 45. Swivel connector samples were subjected to 100,000 cycles of operation under defined conditions.
- Endurance Test Hose Nozzle Valve: Section 46. Hose nozzle valve samples were subjected to 100,000 cycles of operation.
- Pull Test Hose Assemblies: Section 49. Hose assembly samples with end couplings were subjected to a 400-pound pull force.
- Shear Section Section 61. Shear valve samples were subjected to a bending moment of not more than 650 pound-feet to verify the valve would close.
- Ozone Test Section 62. Specimens from hose samples were exposed to ozone for 70 hours and examined for cracking.
- Dielectric Strength UL 79, Section 61. Pump samples were subjected to a 60 Hz potential of 1,460 V applied between live electrical parts and dead metal for a period of 1 minute.

Equipment testing is typically terminated when a noncompliance is noted. However, in the interest of gathering the most data possible, testing after a noncompliance was continued to the degree possible in this program. In some cases, test results are interdependent and the root cause of noncompliances in one test may lead to noncompliances in others.

# **Results**

Table 1 contains a summary of the test results observed on the new dispenser samples and dispensing equipment subassemblies. Dispenser samples were configured with hanging hardware for the Long-Term Exposure Test.

**Table 1. Tests on New Samples** 

Sample	Tests Conducted	Results
Dispenser #1	Long-Term Exposure High-pressure Leakage	Compliant Compliant
Meter/manifold/electric valve assembly #1	Long-Term Exposure High-Pressure Leakage Meter Endurance	Compliant Compliant Noncompliant. Leakage noted during endurance test from meter and valve seals. As a result, no further testing could be conducted.
Dispenser #2	Long-Term Exposure High-Pressure Leakage	Compliant Compliant
Meter/manifold/electric valve assembly #2	Long-Term Exposure High-Pressure Leakage Meter endurance	Compliant Compliant Noncompliant. Leakage noted during endurance test from valve seals. As a result, no further testing could be conducted.
Breakaway #1 (reconnectable)	Long-Term Exposure High-Pressure Leakage Seat Leakage Pull Endurance  Hydrostatic Strength Electrical Continuity	Compliant Compliant Compliant Compliant Noncompliant. Poppet disengaged and leakage noted. Compliant Compliant
Breakaway #2 (reconnectable)	Long-Term Exposure High-Pressure Leakage Pull Test Seat Leakage Endurance High-Pressure Leakage (repeated) Seat Leakage Pull (repeated) Hydrostatic Strength  Electrical Continuity	Compliant Compliant Compliant Compliant Compliant Compliant Compliant Noncompliant. Leakage noted. Compliant Inconclusive. Sample separated at 180 psi and could not reach 250 psi test pressure Compliant

Sample	Tests Conducted	Results
Breakaway #3	Long-Term Exposure	Compliant
(reconnectable)	High-Pressure Leakage	Compliant
	Seat Leakage	Compliant
	Pull	Compliant
	Endurance	Noncompliant. Poppet o-ring displaced and leakage noted.
	High-Pressure Leakage (repeated)	Compliant
	Seat Leakage (repeated)	Noncompliant. Leakage noted.
	Hydrostatic Strength	Inconclusive. Sample separated at 178 psig
		and could not reach test pressure.
	Electrical Continuity	Compliant
Breakaway #4	Long-Term Exposure	Compliant
(non-reconnectable)	High-Pressure Leakage	Compliant
	Pull	Compliant
	Seat Leakage	Compliant
	Electrical continuity	Compliant
Breakaway #5	Long-Term Exposure	Compliant
(non-reconnectable)	High-Pressure Leakage	Compliant
,	Pull	Compliant
	Seat Leakage	Compliant
	Electrical Continuity	Compliant
Flow Limiter #1	Long-Term Exposure	Compliant
	High-Pressure Leakage	Compliant
	Hydrostatic Strength	Compliant
	Electrical Continuity	Compliant
Hose Assembly #1	Long-Term Exposure	Compliant
	Leakage and Electrical Continuity	Compliant
	Hydrostatic Strength	Compliant
	Ozone	Compliant
Hose Assembly #2	Long-Term Exposure	Compliant
	Leakage and Electrical Continuity	Compliant
	Pull	Compliant
	Hydrostatic Strength	Compliant
Hose Assembly #3, with	Long-Term Exposure	Compliant
integral swivel	High-Pressure Leakage	Compliant
	Swivel Operation	Compliant
	High-Pressure Leakage (repeated)	Compliant
	Leakage and Electrical Continuity	Compliant
	Hydrostatic Strength	Compliant
	Ozone	Compliant
Hose Assembly #4	Long-Term Exposure	Compliant
	Leakage and Electrical Continuity	Compliant
	Pull	Compliant
Hose Assembly #5	Long-Term Exposure	Compliant
	Leakage and Electrical Continuity	Compliant
	Pull	Compliant

Sample	Tests Conducted	Results
Hose Assembly #6	Long-Term Exposure	Compliant
	Leakage and Electrical Continuity	Compliant
	Hydrostatic Strength	Compliant
	Ozone	Compliant
Hose assembly #7	Long-Term Exposure	Compliant
	Leakage and Electrical Continuity	Compliant
	Hydrostatic Strength	Compliant
	Ozone	Compliant
Hose assembly #8	Long-Term Exposure  Leakage and Electrical Continuity Hydrostatic Strength	Noncompliant. Ferrule started leaking during pressure testing in week 8 of long-term exposure.  Compliant Compliant
	Ozone	Compliant
Hose #9	Hose Bending Test (Filled)	Compliant
11030 #3	Leakage and Electrical Continuity	Compliant
	Low Temperature	Compliant
Nozzle #1	Long-Term Exposure High-Pressure Leakage	Compliant Compliant
	Endurance	Inconclusive; nozzle shut off flow after approx. 14,000 cycles of endurance and would not allow further flow. As observed the test terminated in a safe condition.
	High-Pressure Leakage (repeated) Hydrostatic Strength Electrical Continuity	Compliant Compliant Compliant
Nozzle #2	Long-Term Exposure	Compliant
	High-Pressure Leakage	Compliant
	Endurance	Compliant
	High-Pressure Leakage (repeated)	Compliant
	Hydrostatic Strength	Compliant
	Electrical Continuity	Compliant
Nozzle #3	Long-Term Exposure High-Pressure Leakage Endurance	Compliant Compliant Inconclusive; nozzle shut off flow after approx. 83,000 cycles of endurance and would not allow further flow. As observed the test terminated in a safe condition.
	High-Pressure Leakage (repeated) Hydrostatic Strength Electrical Continuity	Noncompliant. Leakage noted. Compliant Compliant
Nozzle #4	Long-Term Exposure	Compliant
	High-Pressure Leakage	Compliant
	Endurance	Compliant
	High-Pressure Leakage (repeated)	Noncompliant. Leakage noted.
	Hydrostatic Strength	Compliant
	Electrical Continuity	Compliant

Sample	Tests Conducted	Results
Nozzle #5	Long-Term Exposure	Compliant
	High-Pressure Leakage	Compliant
	Endurance	Compliant
	High-Pressure Leakage (repeated)	Compliant
	Hydrostatic Strength	Compliant
	Electrical Continuity	Compliant
Nozzle #6	Long-Term Exposure	Compliant
	High-Pressure Leakage	Noncompliant. Leakage noted.
	Endurance	Compliant
	High-Pressure Leakage (repeated)	Noncompliant. Leakage noted.
	Hydrostatic Strength	Compliant
	Electrical Continuity	Compliant
Shear Valve #1	Long-Term Exposure	Compliant
	High-Pressure Leakage	Compliant
	Hydrostatic Strength	Compliant
	Shear Section	Compliant
Shear Valve #2	Long-Term Exposure	Compliant
	High-Pressure Leakage	Compliant
	Hydrostatic Strength	Compliant
	Shear Section	Compliant
Shear Valve #3	Long-Term Exposure	Compliant
	High-Pressure Leakage	Compliant
	Hydrostatic Strength	Compliant
	Shear Section	Compliant
Submersible turbine	Long Term Exposure	Compliant
pump #1	Hydrostatic Strength	Inconclusive. Required test pressure could not
	Dielectric Strength	be applied based on sample configuration.  Compliant
Swivel #1	Long-Term Exposure	Compliant
OWIVEI # 1	High-Pressure Leakage	Compliant
	Operation	Compliant
	High-Pressure Leakage (repeated)	Compliant
	Hydrostatic Strength	Compliant
	Electrical Continuity	Compliant
Swivel #2	Long-Term Exposure	Compliant
OWIVEI II Z	High-Pressure Leakage	Compliant
	Electrical Continuity	Compliant
	Operation	Compliant
	High-Pressure Leakage (repeated)	Compliant
	Hydrostatic Strength	Compliant
	Electrical Continuity	Compliant
Swivel #3	Long-Term Exposure	Compliant
	High-Pressure Leakage	Compliant
	Operation	Noncompliant. Leakage noted after
	High Proseure Leakage (reported)	approximately 26,000 cycles on swivel nut.  Noncompliant – leakage noted at swivel nut.
	High-Pressure Leakage (repeated) Hydrostatic Strength	Compliant – leakage noted at swiver nut.
	Electrical Continuity	Compliant

Table 2 contains a summary of the test results observed on used dispensers and dispensing equipment subassemblies.

**Table 2: Tests on Used Samples** 

Sample	Tests Conducted	Results	
Dispenser #3	Long-Term Exposure	Compliant	
	High-Pressure Leakage	Compliant	
Meter/manifold/electric Long-Term Exposure		Compliant	
valve assembly #3	High-Pressure Leakage	Compliant	
	Meter Endurance	Compliant	
	High-Pressure Leakage repeated	Compliant	
	Hydrostatic Strength	Compliant	
	Operation Test – Electrically Operated Valves	Noncompliant. Valve did not shut off flow.	
Nozzle #7	Long-Term Exposure	Noncompliant. Leakage noted during pressure testing starting in week 10 of long-term	
	LE L Book of Lord	exposure.	
	High-Pressure Leakage Endurance	Noncompliant. Leakage noted.  Noncompliant; 100,000 cycles completed but	
	Lituatance	leakage noted.	
	High-Pressure Leakage (repeated)	Noncompliant. Leakage noted.	
	Hydrostatic Strength	Compliant	
	Electrical Continuity	Compliant	
Breakaway #6	Long-Term Exposure	Compliant	
(reconnectable)	High-Pressure Leakage	Compliant	
	Seat leakage	Compliant	
	Pull Test	Compliant	
	Endurance	Noncompliant. Seat leakage noted at 71 cycles.	
Seat Leakage		Noncompliant. Leakage noted.	
Electrical Continuity		Compliant	
Hose assembly #10	Long-Term Exposure	Compliant	
	Leakage and Electrical Continuity	Compliant	
	Pull	Compliant	
Hose assembly #11, with	Long-Term Exposure	Compliant	
integral swivel	Swivel Operation	Compliant	
	Leakage and Electrical Continuity	Compliant	
	Hydrostatic Strength	Compliant	
	Ozone	Compliant	
Dispenser #4	Long-Term Exposure	Compliant	
	High-Pressure Leakage	Compliant	
Meter/manifold/electric	Long-Term Exposure	Compliant.	
valve assembly #4	High-Pressure Leakage	Compliant	
Meter Endurance		Noncompliant. Leakage noted during endurance test from meter and valve seals. As a result, no further testing could be conducted.	

Sample	Tests Conducted	Results
Nozzle #8	Long-Term Exposure	Noncompliant. Seat leakage noted during pressure testing in week 9 of long-term exposure.
	High-Pressure Leakage Endurance	Noncompliant. Leakage noted.  Noncompliant; 100,000 cycles completed but seat leakage noted
	High-Pressure Leakage (repeated) Hydrostatic Strength Electrical Continuity	Noncompliant. Leakage noted Compliant Compliant
Breakaway #7	Long-Term Exposure	Compliant
(reconnectable)	High-Pressure Leakage	Compliant
,	Seat Leakage	Compliant
	Pull	Noncompliant. Separated above rated value.
	Endurance	Compliant
	High-Pressure Leakage (repeated)	Compliant
	Seat Leakage	Compliant
	Pull (repeated)	Compliant
	Electrical Continuity	Compliant
	Hydrostatic Strength	Inconclusive. Sample separated at 208 psig and could not reach test pressure
Hose assembly #12	Long-Term Exposure	Compliant
	Leakage and Electrical Continuity	Compliant
	Pull	Compliant
Hose assembly #13, with	Long-Term Exposure	Compliant
integral swivel	Swivel Operation	Compliant
	Leakage and Electrical Continuity	Compliant
	Hydrostatic Strength	Compliant
	Ozone	Noncompliant; cracking noted
Dispenser #5	Long-Term Exposure High-Pressure Leakage	Compliant
		Compliant
Meter/manifold/electric	Long-Term Exposure	Compliant
valve assembly #5	High-Pressure Leakage	Compliant
	Meter Endurance	Noncompliant. Leakage noted at valve seal. As a result, no further testing could be conducted.
Nozzle #9	Long-Term Exposure	Compliant
	High-Pressure Leakage	Compliant
	Endurance	Compliant
	High-Pressure Leakage (repeated)	Compliant
	Hydrostatic Strength	Compliant
	Electrical Continuity	Compliant
Breakaway #8	Long-Term Exposure	Compliant
(reconnectable)	High-Pressure Leakage	Compliant
	Seat Leakage Pull Test	Compliant Noncompliant. Separated above rated value.
	1000	After separation, sample could not be
		reassembled to complete other tests.
	Electrical Continuity	Compliant

Sample	Tests Conducted	Results
Swivel #4	Long-Term Exposure High-Pressure Leakage Operation Test	Compliant Compliant Noncompliant. Body joint leaked after approximately 62,000 cycles. Swivel nut leaked after approximately 12,200 cycles.
	High-Pressure Leakage (repeated) Hydrostatic Strength Electrical Continuity	Compliant Compliant Compliant
Hose assembly #14, with integral swivel	Long-Term Exposure	Noncompliant. Ferrule started leaking during pressure testing in week 7 of long-term exposure.
	High-Pressure Leakage Swivel Operation High-Pressure Leakage (repeated) Hydrostatic Strength Leakage and Electrical Continuity Ozone	Compliant Compliant Compliant Compliant Compliant Compliant Noncompliant – cracking noted
Dispenser #6	Long-Term Exposure High-Pressure Leakage	Compliant Compliant
Meter/manifold/electric valve assembly #6	Long-Term Exposure High-Pressure Leakage Meter Endurance	Compliant Compliant Noncompliant. Leakage noted during endurance test from meter and valve seals. As a result, no further testing could be conducted.
Nozzle #10	Long-Term Exposure High-Pressure Leakage Endurance Test  Hydrostatic Strength	Compliant Compliant Noncompliant. Seat leakage noted and automatic shutoff not operating after approx. 61,000 cycles of Endurance Test. Compliant Compliant
Breakaway #9	Electrical Continuity  Long-Term Exposure	Compliant
(non-reconnectable)	High-Pressure Leakage Seat Leakage Electrical Continuity	Compliant Compliant Compliant
Swivel #5	Long-Term Exposure High-Pressure Leakage Operation Test	Compliant Compliant Noncompliant; swivel nut leaked after approximately 3000 cycles. Testing on body joint was compliant.
	High-Pressure Leakage (repeated) Hydrostatic Strength Electrical Continuity	Compliant Compliant Compliant
Hose Assembly #15	Long-Term Exposure Leakage and Electrical Continuity Pull Hydrostatic Strength	Compliant Compliant Compliant Compliant

# **Analysis**

An exhaustive literature search was conducted on gasoline and gasoline-ethanol blended fuel compatibility with fuels infrastructure materials and equipment. From this investigation, numerous published reports have demonstrated that exposure to fuels such as ethanol/gasoline blends may affect materials that come into contact with the fuel. This may affect the performance of a formed part (such as a gasket) manufactured from such materials. The formed part may be affected to the degree that it modifies equipment performance with respect to a critical property. In this case, a change in equipment performance or safety may be noted. For this program, a change in equipment performance was gauged by response to the defined test conditions.

Table 3 summarizes the performance of different types of equipment in the testing program.

Table 3: Summary of Test Results on Different Types of Equipment

Equipment	Compliant Test Results on New Samples <sup>a</sup>	Compliant Test Results on Used Samples <sup>a</sup>	Overall Compliant Test Results <sup>a</sup>
Breakaways	2 of 5	1 of 4	3 of 9
Flow Limiters	1 of 1	_	1 of 1
Hoses/Hose Assemblies	8 of 9	4 of 6	12 of 15
Meter/Manifold/Valve Assemblies	0 of 2	0 of 4	0 of 6
Nozzles	3 of 6	1 of 4	4 of 10
Shear Valves	3 of 3	_	3 of 3
Submersible Turbine Pumps	1 of 1	_	1 of 1
Swivels <sup>b</sup>	3 of 4	3 of 5	6 of 9

<sup>&</sup>lt;sup>a</sup>In the context of Table 3, "compliant" results is used to include fully compliant test results and inconclusive test results that did not directly manifest a hazard such as leakage during the testing that was able to be performed as a part of this research program.

For equipment with noncompliant test results, few leakages occured during the Long-Term Exposure test. The majority of leakages occurred during performance testing. These results may indicate that exposing some equipment to fuel blends with higher ethanol content may not produce an immediate or short-term response that would result in a leakage. However, this equipment may still demonstrate reduced effective life and in time lead to a reduced level of safety as assessed in the subsequent performance testing.

Some equipment, both new and used, demonstrated performance during and after the Long-Term Exposure test that indicated a reduced level of safety or efficacy, or both. These data indicate that some pieces of equipment in the legacy base of installed gasoline dispensing equipment may be adversely affected by exposure to fuel with higher ethanol content. During this testing program, a number of leakages and other noncompliant results were noted on new and used equipment harvested from the field. Leakages are largely attributed to effects of exposure on the gasket and seal materials. The only exceptions were cases in which a polymeric component of a breakaway coupling was degraded and the damage resulted in a consequential leakage.

<sup>&</sup>lt;sup>b</sup> Includes swivels integral to hose assemblies.

#### **Gaskets**

Exposure to gasoline/ethanol blends may cause gasket and seal materials to swell<sup>4</sup> or otherwise be affected. Although mild swelling may produce the short-term effect of a tighter seal, it is indicative of a material response to exposure that may have long-term consequences for seal performance. Previous studies<sup>6</sup> identified volume swelling as one of the most critical measurements when considering tolerances for elastomeric seal housing design; swelling of elastomers greater than 20% have reportedly caused several problems, including overfill of the seal housing groove, seal extrusion damage, extremely high stresses in the seal and in the housing, occasional fracture of metal components, and progressive degradation of elastomers. Studies<sup>7</sup> have also established that elastomers demonstrate increased permeability of gasoline/ethanol blends with increasing ethanol content. Permeation may in turn lead to extraction of organic compounds from exposed nonmetals. In the case of fillers and other compounds that are introduced into the gasket or seal for a specific performance attribute, such extraction may fundamentally alter the material and the corresponding performance of the formed part.

Depending on the configuration, fuel dispensers may contain 20 to 60 (or more) gaskets and seals. Many equipment manufacturers use a variety of gasket materials in their ongoing production of specific pieces of equipment, with potential variations in sourcing over time and different manufacturing locations. The field population of a specific piece of equipment designed for use with gasoline and E10 may incorporate a variety of gasket materials. In the past, these materials were generally selected based on their compatibility with gasoline and E10. The materials may demonstrate varying compatibility with higher ethanol fuel blends.

#### **Metallic Parts**

In this study, there was no noted effect on metallic parts of equipment. The lack of galvanic interaction or other significant corrosion is consistent with the relatively lower ethanol content of E15 fuel serving as the subject of this study and corresponding lower electrical conductivity, compared to higher ethanol fuel blends such as E85.

#### **Used Equipment**

Used equipment has already been subjected to a useful life, which reflects its unique conditions of use and maintenance. Use conditions may vary widely with respect to temperature, fuels the equipment dispensed, duration of use, conditions of practical use, and similar environmental conditions. Maintenance conditions such as adherence to applicable schedules and field modification of the equipment also may vary widely. Based on these practical issues, the response of used equipment to the prescribed test conditions may be inherently variable. Some used equipment demonstrated noncompliant results in this test program. However, various pieces of used subassemblies completed the testing with fully compliant results. In all cases, if legacy dispensers were to be exposed to fuel blends with higher ethanol content, effective supervision, maintenance, and inspection regimes will be important to effectively monitor the equipment's response to the different conditions of use and proactively minimize the occurrence of hazards.

#### **Breakaways**

The breakaway coupling samples demonstrated varying performance in the test program. Three of the nine samples tested, and two of the five new samples, yielded compliant

results. All three non-reconnectable samples yielded compliant results. Two cases of noncompliant results were for reconnectable breakaways, in which the poppet was dislodged during endurance and caused containment loss; a more appropriate poppet material would be expected to produce better practical results. Only one of the four used samples produced compliant results. Two noncompliances were noted for the pull test force on used samples. Two instances of seat leakage were noted on one new and one used sample; more appropriate sealing methods for the seat would be expected to produce better practical results in these cases.

#### Flow Limiter

The flow limiter sample yielded fully compliant results.

#### Hoses

Hoses and hose assemblies, both new and used, fared well overall. Twelve of the 15 samples, and eight of the nine new samples, complied with all tests that were performed. Thirteen of the 14 samples yielded results on the hoses that were compliant. Of the three samples that produced noncompliant results, two leaked at the fitting ferrule, and one used sample yielded noncompliant results in the ozone test. In the cases involving leaks at the ferrule, a more appropriate sealing method would be expected to produce better practical results.

#### Meter/Manifold/Valve Assemblies

The meter/manifold/valve assemblies demonstrated noncompliant results in the six dispensers tested. In five cases, the meter cover seal leaked; in the sixth, the electric valve lost its ability to shut off the flow of fuel. These data indicate that gasket and seal materials used in these applications may be particularly affected by exposure to fuel blends with greater ethanol content. The seal materials used in this part of the hydraulic tree may require careful consideration if fuel blends with higher ethanol content are used.

#### **Nozzles**

The nozzle samples demonstrated varying performance in the test program. Four of the 10 samples tested, and three of the six new samples, yielded compliant results or results that did not involve containment loss. Five of the six noncompliant results noted involved leakage, including seat leakage; more appropriate sealing methods would be expected to produce better practical results. Only one of the four used samples produced compliant results

#### **Shear Valves**

The three new shear valve samples demonstrated compliant results in all cases.

#### **Swivels**

The swivel samples demonstrated varying performance. Six of the nine samples tested yielded compliant results. Three of the four new samples were compliant; this may indicate that more recent designs are better suited to anticipate use with E15 fuel. Three of the five used samples produced compliant results. All three noncompliant results noted involved leakage that started during the operation test. More appropriate seal materials would be expected to produce better practical results.

## **Submersible Turbine Pumps**

The submersible turbine pump sample tested demonstrated compliant results for the long-term exposure and dielectric strength test. The hydrostatic strength test yielded inconclusive results because the required test pressure could not be applied based on the test sample configuration; however, no noncompliant results were noted. These data do not demonstrate an incompatibility of the test item with E15, and the Long-Term Exposure test was successfully completed.

## Conclusion

The overall results of the program were not conclusive insofar as no clear trends in the overall performance of all equipment could be established.

Various pieces of new and used dispensing equipment demonstrated compliant results. Shear valve and flow limiter test items produced compliant results, the submersible turbine pump performed well, and hoses generally yielded compliant results.

Some equipment with noncompliant results did not leak during the Long-Term Exposure test. These results may indicate that exposing some equipment to fuel blends with higher ethanol content may or may not produce an immediate or short-term response that would cause leakage. However, this equipment may still demonstrate reduced effective life and in time lead to a reduced level of safety as assessed in the subsequent performance testing.

Some equipment, both new and used, demonstrated performance during and after the Long-Term Exposure test that indicated a reduced level of safety or performance, or both. These pieces of equipment demonstrated limited ability to safely accommodate exposure to fuels such as E15 with higher ethanol content. Responses of nonmetals to exposure—notably gaskets and seals, but also polymeric parts—were involved with these noncompliances. Dispenser meter/manifold/valve assemblies in particular demonstrated largely noncompliant results; the seal materials used in this portion of the hydraulic tree may require careful consideration if fuel blends with higher ethanol content are used.

Analysis of the extracted test fluids may provide additional insight into the chemical interactions of the test fluids, materials, and the corresponding degradation mechanisms; analysis results are available in Appendix A. Because of the specific nature and goals defined for this program, a finite number of test items were employed. Testing of other items to establish a larger sample size may provide additional insights. Further detailed analysis of the equipment that produced compliant results may establish best practices; conversely, further detailed analysis of the equipment that produced noncompliant results may further identification of root causes of equipment design that may lead to leakages or other potential risks. This work is ongoing and will be reported separately.

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# Appendix A

## Fluid Analysis Summary for Dispensers 1 and 5

Oakridge National Laboratory Mike Kass, Tim Theiss, Sam Lewis and John Storey

During the 15-week conditioning phase of UL Subject 87A, spent fluid samples were extracted from dispensers #1 and #5 for analysis by Oak Ridge National Laboratory (ORNL). Dispenser 1 was a new dispenser while Dispenser 5 has a similar design and was used for five years. The fuel dispensing history of Dispenser 5 is unknown. During the evaluation, the fluids within the dispensers were replaced once per week for 15 weeks. A control fuel sample and tested samples from weeks 1, 2, 3, 4, 8, 10, 12 and 15 were sent to ORNL for analysis. Photographs showing the fluid coloration with sample times are shown in Figures 1 and 2 for Dispensers 1 and 5, respectively. Both sets of fluids exhibited an amber coloration during the first week of experimentation, in contrast to the control fluid, which is clear. In general, the color becomes less pronounced and more clear as the test period progresses. The fluid in Dispenser 1 retains the amber color into week 12, while the fluid extracted from Dispenser 5 loses the amber coloration around week 8. The fuel sample for week 15 for Dispenser 1 is noteworthy in that it did not follow the observed trend and exhibited a clear coloration for week 15. Analysis revealed that this sample was chemically identical to control specimen (uncontaminated CE17a). The results may potentially be attributed to a sample handling error.

The fluids were analyzed using a gas chromatography-mass spectrometer (GC-MS). GC-MS is an established analytical technique for analysis of hydrocarbon compounds in fluid-based samples. Representative GC-MS spectra for fluids extracted from Dispenser 1 and 5 are shown in Figures 3 and 4, respectively. The spectra reveal key differences between the two samples. As shown in Figure 3, fluid extracted from Dispenser 1 (a new unit) showed clear identifiable peaks associated with phthalate and polymer compounds. In contrast, the spectra shown in Fig. 4 for the fluid pulled from the used Dispenser 5 was heavily contaminated with kerosene. The presence of high kerosene levels is a strong indicator that this dispenser unit had been used to dispense kerosene at some point in its operational lifetime. Unfortunately, because the kerosene concentration was so high, any phthalate or polymer compounds that may have been present in the fluid samples would be masked out by the kerosene. Therefore, we cannot state with any certainty whether dissolved phthalates or polymers were present in the fluid samples for Dispenser 5.

The phthalates observed in the Dispenser 1 fluid samples are commonly added to dispenser hoses, and to a lesser extent in the o-rings and gaskets to increase flexibility and durability. Because phthalates are not covalently bonded to the polymer structure, they are highly susceptible to leaching and removal by fluids that are capable of penetrating into the polymer structure. The phthalate concentration as a function of week of exposure to CE17a test fluid is shown in Fig. 3 for Dispenser 1. Except for week 12, the phthalate level decreased with exposure indicating that the phthalate concentration in the diffusion region of the elastomer was decreasing with time. The results may potentially be attributed to a sample handling error.

On the other hand, the decrease in phthalate concentration with sampling time can be attributed to two compounding reasons. First, the level of available phthalates in the elastomer decreases with exposure time as the phthalates are leached away and, secondly, the diffusion distance for the fluid to permeate into the elastomer to reach and dissolve the phthalate compounds also increases, thereby reducing phthalate removal. Because the phthalates are added to polymers to impart flexibility and durability, their removal will result in a stiffer component that is susceptible to cracking when flexed. We cannot state without further investigation whether the phthalate removal was caused by a single component or interaction of the CE17a ingredients. However, results from the ORNL stir-tank materials study have shown that the volume swell (a measure of permeation) for polymers increased with the addition of the aggressive ethanol in most cases.

The sample fluid from Dispenser 1 also contained high concentrations of polymer fragments indicative of fractured molecules of elastomers and rubber seals (see Fig. 4). The longer hydrocarbon chain lengths of the elastomer molecules are too large to be detected using GC-MS; however, fractured elements of the elastomer, such as hexanoic acid (shown in Fig. 4), were detected. The ester and ether molecular groups can be cleaved from the extended hydrocarbon structure through a hydrolysis reaction involving an acid acting as catalyst. Because the hydrolysis reaction requires an acid catalyst to cleave the polymer into the resulting hexanoic acid fragments, the acetic and sulfuric acid components of the test fluid are likely responsible for polymer fragmentation and subsequent detection. The resulting fragments are themselves acids and serve to propagate the hydrolysis reaction. Polymer fractionation and dissolution would eventually lead to structural damage and a weakening of gaskets or o-rings. Prolonged exposure would result in gap formation between the gasket and sealed sections leading to fluid leakage.

ORNL concludes that polymer degradation was caused primarily by the acid constituents of the aggressive ethanol. There was some discussion as to whether the 60°C operating temperature was responsible for the noted polymer degradation, but the observed polymer hydrolysis fractionation cannot be attributed to temperature alone. Thermal-based reactions would result in increased crosslinking and not cleavage of the hydrocarbons chains. Additionally, thermal oxidation of the hydrocarbons would result in the formation of CO, CO<sub>2</sub>, H<sub>2</sub>O, and partially oxidized hydrocarbons (soot). However, the temperatures needed to promote thermal oxidation of the elastomers would be expected to exceed 60°C and no partially oxidized hydrocarbons of either the fuel or the polymers were detected.

Because the kerosene contamination in the Dispenser 5 fluid samples was so high, we were unable to identify any peaks associated with phthalate compounds or polymer fractions. Therefore, we had to rely on the Dispenser 1 fluid samples to assess potential interactions between the test fuel and dispenser materials (especially elastomers). The fluid samples contained large levels of phthalates and fractionated polymers (hexanoic acid, etc.). The presence of phthalates indicates that the fluids were able to penetrate into the elastomer structure and remove the phthalate compounds which were added to improve flexibility. As a result the elastomers can be expected to have reduced durability.

The presence of hexanoic acid is a strong indication that the weak acids present in the test fuels were able to hydrolyze and break down the molecular structure of the gasket and seal materials. Either of these two effects will degrade the physical properties of the elastomers used in the gaskets, o-rings, seals, etc. and would eventually lead to leakage.

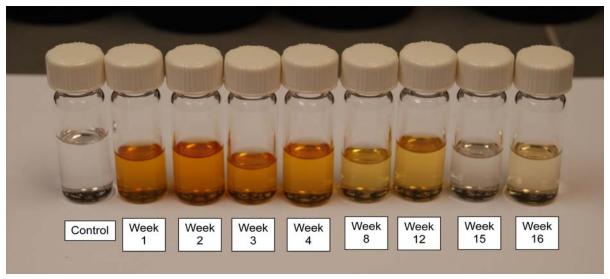


Figure. 1. Photograph showing the weekly change in appearance of fluid extracted from Unit 1.

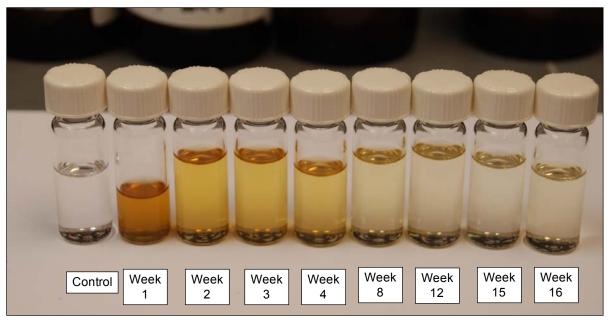


Figure 2. Photograph showing the weekly change in appearance of fluid extracted from Unit 5.

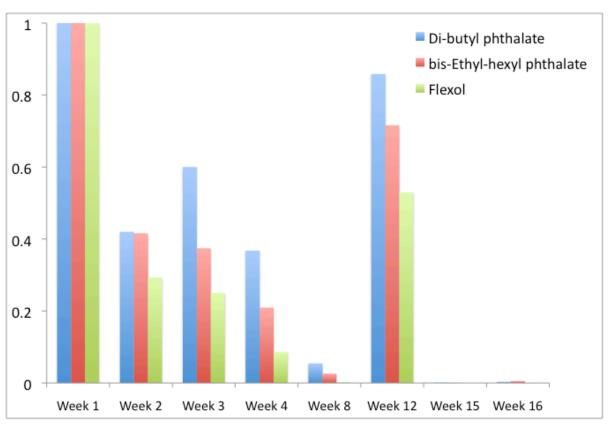


Figure. 3. Phthalate concentration as a function of sample time for fluid samples extracted from Dispenser 1.

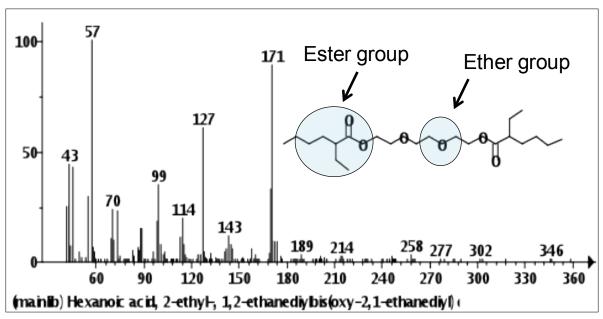
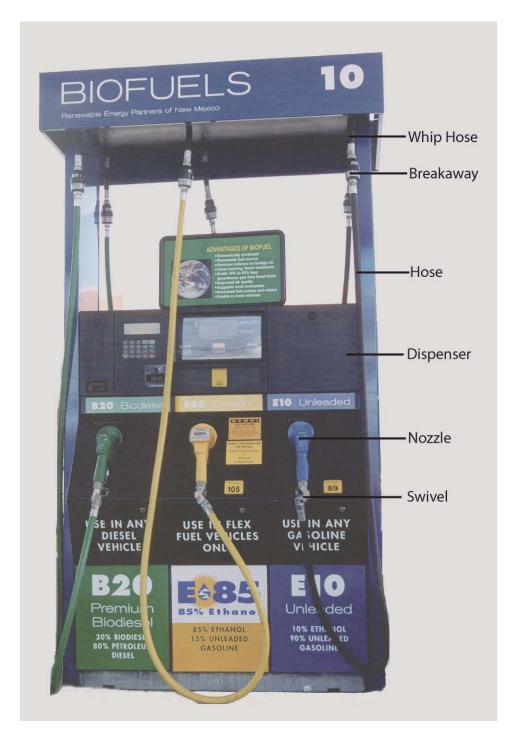


Figure 4. GC-MS graph showing an acid fragment formed by the cleavage of a long chain hydrocarbon elastomer. The ester and ether groups of the hexanoic acid are shown as sites where hydrolysis occurs.

# Appendix B



**NREL/PIX 13531** 

## REPORT DOCUMENTATION PAGE

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	NREL better understand any potentially adverse impacts caused by a lack of knowledge about the compatibility of the dispensing equipment with ethanol blends higher than what the equipment was designed to dispense. This report provides data about the					
	impact of introducing a gasoline with a higher volumetric ethanol content into service station dispensing equipment from a safety					
and a performance perspective.						
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# Subcontract Report NREL/SR-7A20-49187

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