

Regulatory Impact Analysis

Control of Hazardous Air Pollutants from Mobile Sources

Chapter 7 Portable Fuel Container Feasibility and Test Procedures

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Chapter 7: Portable Fuel Container Feasibility and Test Procedures

Section 183 (e) of the Clean Air Act provides statutory criteria that EPA must evaluate in determining standards for consumer products. The standards must reflect “best available controls” as defined by section 183 (e)(3)(A). Determination of the “best available controls” requires EPA to determine the degree of reduction achievable through use of the most effective control measures (which extend to chemical reformulation, and product substitution) after considering technological and economic feasibility, as well as health, energy, and environmental impacts. Chapters 1 through 3 discuss the environmental and health impacts of portable fuel container (PFC) emissions. Chapter 10 discusses the economic feasibility of PFC controls and the fuel savings associated with controlling PFC emissions. This chapter presents the technological feasibility of controlling emissions from PFCs. All of these analyses and information form the basis of EPA’s belief that the evaporative emission standards reflect the “best available controls” accounting for all the above factors.

This chapter presents available data on baseline emissions and on emission reductions achieved through the application of emission control technology. In addition, this chapter provides a description of the test procedures for determining evaporative emissions.

Evaporative emissions from PFCs containing gasoline can be very high.^A This is largely because PFCs are often left open and vent to the atmosphere and because materials used in the construction of the plastic PFCs generally have high permeation rates. Evaporative emissions can be grouped into three main categories:

DIURNAL: Gasoline evaporation increases as the temperature rises during the day, heating the PFC and venting gasoline vapors.

PERMEATION: Gasoline molecules can saturate plastic PFCs, resulting in a relatively constant rate of emissions as the fuel continues to permeate through the walls of the PFC.

REFUELING: Gasoline vapors are always present in typical containers. These vapors are forced out when the container is filled with liquid fuel.

The use of PFCs also results in losses through spillage, both during transportation and usage of the cans to refill vehicles and equipment.

7.1 Permeation Emissions

The California Air Resources Board (ARB) investigated permeation rates from PFCs with no emissions controls.^{1,2} The ARB data is compiled in several data reports on their web site and is included in our docket. Table 7.1-1 presents a summary of this data which was

^A Diesel and kerosene fuels have very low volatility levels and therefore much lower evaporative emissions compared to gasoline.

collected using the ARB Test Method 513.³ Although the temperature in the ARB testing is cycled from 65 – 105° F with 7 pound per square inch (psi) Reid Vapor Pressure (RVP) fuel, the results would be similar if the data were collected at the temperature range and fuel used by EPA of 72-96° F with 9 psi RVP fuel. This is because the lower temperature and higher RVP effectively offset one another. The average permeation emissions from uncontrolled containers were 1.57 g/gallon/day.

Table 7.1-1. Permeation Rates for HDPE PFCs Tested by ARB

PFC Capacity [gallons]	Permeation Loss [g/gal/day]
1.0	1.63
1.0	1.63
1.0	1.51
1.0	0.80
1.0	0.75
1.0	0.75
1.3	0.50
1.3	0.49
1.3	0.51
1.3	0.52
1.3	0.51
1.3	0.51
1.3	1.51
1.3	1.52
2.1	1.88
2.1	1.95
2.1	1.91
2.1	1.78
2.5	1.46
2.5	1.09
5.0	0.89
5.0	0.62
5.0	0.99
5.0	1.39
5.0	1.46
5.0	1.41
5.0	1.47
6.6	1.09

7.2 Permeation Emissions Controls

7.2.1 Sulfonation

The California Air Resources Board (ARB) collected test data on permeation rates from sulfonated PFCs using California certification fuel.⁴ The results show that sulfonation can be used to achieve significant reductions in permeation from plastic fuel containers. This data was

collected using a diurnal cycle from 65 – 105° F. The average emission rate for the 32 sulfonated PFCs was 0.35 g/gal/day; however, there was a wide range in effectiveness of the sulfonation process for these PFCs. Some of the data outliers were actually higher than baseline emissions. This was likely due to leaks in the PFCs which would result in large emission increases due to pressure built up with temperature variation over the diurnal cycle. Removing these five outliers, the average permeation rate is 0.17 g/gal/day with a minimum of 0.01 g/gal/day and a maximum of 0.64 g/gal/day. This data suggests that more than a 90% reduction in permeation is possible through sulfonation. This data is presented in Table 7.2-1.

Table 7.2-1. Permeation Rates for Sulfonated Plastic PFCs Tested by ARB

PFC Capacity [gallons]	Permeation Loss [g/gal/day]
1	0.05
1	0.05
1	0.05
1	0.06
1	0.06
1	0.06
1	0.08
1	0.12
1	0.14
1	1.23
1	1.47
1	1.87
2	0.02
2	0.02
2	0.48
2	0.54
2	1.21
2.5	0.03
2.5	0.08
2.5	0.32
2.5	0.38
2.5	0.42
2.5	0.52
2.5	0.64
2.5	0.80
5	0.01
5	0.04
5	0.05
5	0.06
5	0.11
5	0.13
5	0.15

Variation can occur in the effectiveness of this surface treatment if the sulfonation process is not properly matched to the plastic and additives used in the container material. For instance, if the sulfonater does not know what ultraviolet (UV) inhibitors or plasticizers are used, they cannot maximize the effectiveness of their process. Earlier data collected by ARB showed consistently high emissions from sulfonated fuel containers; however, ARB and the treatment manufacturers agree that this was due to inexperience with treating fuel containers and that these issues have since been largely resolved.⁵

ARB also investigated the effect of fuel slosh on the durability of sulfonated surfaces. Three half-gallon fuel tanks used on small SI equipment fuel tanks were sulfonated and tested for permeation before and after being rocked with fuel in them 1.2 million times.^{6,7} These fuel tanks were blow-molded high density polyethylene (HDPE) tanks used in a number of small SI applications including pressure washers, generators, snowblowers, and tillers. The results of the testing show that an 85% reduction in permeation was achieved on average even after the slosh testing was performed. Table 7.2-2 presents these results which were recorded in units of g/m²/day. The baseline level for Set #1 is an approximation based on testing of similar fuel tanks, while the baseline level for Set #2 is based on testing of those tanks.

The sulfonater was not aware of the materials used in the fuel tanks sulfonated for the slosh testing. After the tests were performed, the sulfonater was able to get some information on the chemical make up of the fuel tanks and how it might affect the sulfonation process. For example, the UV inhibitor used in some of the fuel tanks is known as HALS. HALS also reduces the effectiveness of the sulfonation process. Two other UV inhibitors, known as carbon black and adsorber UV, are also used in similar fuel tank applications. These UV inhibitors cost about the same as HALS, but have the benefit of not interfering with the sulfonation process. The sulfonater claimed that if HALS were not used in the fuel tanks, a 97% reduction in permeation would have been seen.⁸ To confirm this, one manufacturer tested a sulfonated tank similar to those in Set #2 except that carbon black, rather than HALS, was used as the UV inhibitor. This fuel tank showed a permeation rate of 0.88 g/m²/day at 40°C⁹ which was less than half of what the CARB testing showed on their constant temperature test at 40°C.¹⁰ A list of resins and additives that are compatible with the sulfonation process is included in the docket.^{11,12}

Table 7.2-2. Permeation Rates for Sulfonated Fuel Tanks with Slosh Testing by ARB Over a 18-41°C Diurnal

Technology Configuration	Units	Tank 1	Tank 2	Tank 3	Average
Set #1 Approximate Baseline	g/m ² /day	10.4	10.4	10.4	10.4
Set #1 Sulfonated	g/m ² /day	0.73	0.82	1.78	1.11
	% reduction	93%	92%	83%	89%
Set #1 Sulfonated & Sloshed	g/m ² /day	1.04	1.17	2.49	1.57
	% reduction	90%	89%	76%	85%
Set #2 Average Baseline	g/m ² /day	12.1	12.1	12.1	12.1
Set #2 Sulfonated	g/m ² /day	1.57	1.67	1.29	1.51
	% reduction	87%	86%	89%	88%
Set #2 Sulfonated & Sloshed	g/m ² /day	2.09	2.16	1.70	1.98
	% reduction	83%	82%	86%	84%

About a year and a half after the California ARB tested the Set #2 fuel tanks, we performed permeation tests on these fuel tanks. During the intervening period, the fuel tanks remained sealed with California certification fuel in them. We drained the fuel tanks and filled them with fresh California certification fuel. We then measured the permeation rate at 29°C. Because this is roughly the average temperature of the California variable temperature test, similar permeation rates would be expected. The untreated fuel tanks showed slightly lower permeation over the constant temperature test as compared to the ARB test. This difference was likely due to the difference in the temperature used for the testing. However, the sulfonated fuel tanks showed an increase in permeation as compared to the ARB test. This increase in permeation appears to be the result of the 1.5 year additional fuel soak. After this long soak, the average permeation reduction changed from 84% to 78%. Table 7.2-3 presents this comparison.

Table 7.2-3. Permeation Rates [g/m²/day] for Sulfonated Fuel Tanks Tested by ARB and EPA on CA Certification Gasoline with a 1½ Year Fuel Soak Differential

Technology Configuration	Temperature	Tank 1	Tank 2	Tank 3	Average
Baseline, CARB testing	18-41°C	12.1	12.1	12.1	12.1
Baseline, EPA testing after 1.5 year additional fuel soak	29°C	11.5	11.4	11.2	11.4
	% change	-5%	-6%	-7%	-6%
Sulfonated, CARB testing	18-41°C	2.09	2.16	1.70	1.98
Sulfonated, EPA testing after 1.5 year additional fuel soak	29°C	2.48	2.73	2.24	2.5
	% reduction from EPA baseline	78%	76%	80%	78%

After the above testing, we drained the fuel tanks and filled them with certification gasoline splash-blended with 10% ethanol (E10). We then soaked the fuel tanks for 20 weeks to precondition them on this fuel. Following the preconditioning, we tested these fuel tanks for permeation at 29°C (85°F). Table 7.2-4 presents these emission results compared to the emission results for three baseline tanks (untreated) that were subject to the same preconditioning. Percent reductions are presented based on the difference between the sulfonated fuel tanks and the average results of the three untreated fuel tanks.

Table 7.2-4. Permeation Rates for Sulfonated Fuel Tanks on E10 Fuel at 29°C

Technology Configuration	Units	Tank 1	Tank 2	Tank 3	Average
Baseline (untreated)	g/m ² /day	13.9	13.7	14.4	14.0
Sulfonated	g/m ² /day	3.91	4.22	2.92	3.69
	% reduction	72%	70%	79%	74%

One study looked at the effect of alcohol in the fuel on permeation rates from sulfonated fuel tanks.¹³ In this study, the fuel tanks were tested with both gasoline and various methanol blends. No significant increase in permeation due to methanol in the fuel was observed.

7.2.2 Fluorination

Another barrier treatment process is known as fluorination. The fluorination process causes a chemical reaction where exposed hydrogen atoms are replaced by larger fluorine atoms which form a barrier on surface of the container. In this process, PFCs are generally processed post production by stacking them in a steel container. The container is then voided of air and flooded with fluorine gas. By pulling a vacuum in the container, the fluorine gas is forced into every crevice in the fuel containers. As a result of this process, both the inside and outside surfaces of the PFCs are treated. As an alternative, containers can be fluorinated on-line by exposing the inside surface of the PFC to fluorine during the blow molding process. However, this method may not prove as effective as off-line fluorination which treats the inside and outside surfaces.

We tested one fluorinated HDPE fuel tank which we bought off the shelf and sent to a fluorinator for barrier treatment. The fuel tank type used was a 6-gallon portable marine fuel tank. The fuel tank was soaked for 20 weeks with certification gasoline prior to testing. We measured a permeation rate of 0.05 g/gal/day (0.56 g/m²/day), which represents more than a 95 percent reduction from baseline. We then began soaking this fuel tank on E10, subjected it to the required pressure and slosh testing, and retested the fuel tank. The post-durability testing showed a permeation rate of 0.6 g/gal/day (6.8 g/m²/day). As discussed below, we believe that the impact of the durability testing on the effectiveness of fluorination can be minimized if the fluorination process and material properties are matched properly. In addition, this fuel tank was treated to a significantly lower level of fluorination than is now available. However, this data supports the need for the durability testing requirements included in the program.

The California Air Resources Board (ARB) collected test data on permeation rates from fluorinated fuel containers using California certification fuel.^{14, 15} The results show that fluorination can be used to achieve significant reductions in permeation from plastic fuel containers. This data was collected using a diurnal cycle from 65 - 105°F. For the highest level of fluorination, the average permeation rate was 0.04 g/gal/day, which represents a 95 percent reduction from baseline. Earlier data collected by ARB showed consistently high emissions from fluorinated PFCs; however, ARB and the treatment manufacturers agree that this was due to inexperience with treating fuel containers and that these issues have since been largely resolved.¹⁶ The ARB data is presented in Table 7.2-5.

Table 7.2-5. Permeation Rates for Fluorinated Plastic PFCs Tested by ARB

Barrier Treatment*	PFC Capacity [gallons]	Permeation Loss [g/gal/day]
Level 4 (average =0.09 g/gal/day)	1	0.05
	1	0.05
	1	0.06
	5	0.11
	5	0.11
	5	0.15
Level 5 (average =0.07 g/gal/day)	1	0.03
	1	0.04
	1	0.05
	1	0.05
	1	0.07
	1	0.08
	1	0.11
	1	0.11
	1	0.12
	2.5	0.04
	2.5	0.04
	2.5	0.05
	2.5	0.07
	2.5	0.07
	5	0.05
5	0.10	
5	0.11	
SPAL (average =0.04 g/gal/day)	5	0.04
	5	0.04
	5	0.04

*designations used in ARB report; shown in order of increasing treatment

All of the data on fluorinated PFCs presented above were based on PFCs fluorinated by the same company. Available data from another company that fluorinates fuel containers shows a 98 percent reduction in gasoline permeation through a HDPE fuel tank due to fluorination.¹⁷

ARB investigated the effect of fuel slosh on the durability of fluorinated surfaces. Two sets of three fluorinated fuel tanks were tested for permeation before and after being sloshed with fuel in them 1.2 million times.^{18,19} These fuel tanks were 0.5 gallon, blow-molded HDPE tanks used in a number of small SI applications including pressure washers, generators, snowblowers, and tillers. The results of this testing show that an 80% reduction in permeation was achieved on average even after the slosh testing was performed for Set #1. However, this data also showed a 99 percent reduction for Set #2. This shows the value of matching the barrier treatment process to the fuel tank material. Table 7.2-6 presents these results, which were recorded in units of g/m²/day. The baseline level for Set #1 is an approximation based on testing of similar fuel tanks, while the baseline for Set #2 is based on testing of those tanks.

Table 7.2-6. Permeation Rates for Fluorinated Fuel Tanks with Slosh Testing by ARB Over a 65-105° F Diurnal

Technology Configuration	Units	Tank 1	Tank 2	Tank 3	Average
Set #1 Approximate Baseline	g/m ² /day	10.4	10.4	10.4	10.4
Set #1 Fluorinated	g/m ² /day % reduction	1.17 89%	1.58 85%	0.47 96%	1.07 90%
Set #1 Fluorinated & Sloshed	g/m ² /day % reduction	2.38 77%	2.86 73%	1.13 89%	2.12 80%
Set #2 Approximate Baseline	g/m ² /day	12.1	12.1	12.1	12.1
Set #2 Fluorinated	g/m ² /day % reduction	0.03 >99%	0.00 >99%	0.00 >99%	0.01 >99%
Set #2 Fluorinated & Sloshed	g/m ² /day % reduction	0.07 99%	0.11 99%	0.05 >99%	0.08 99%

About a year and a half after the California ARB tests on the Set #2 fuel tanks, we performed permeation tests on these fuel tanks. During the intervening period, the fuel tanks remained sealed with California certification fuel in them. We drained the fuel tanks and filled them with fresh California certification fuel. We then measured the permeation rate at 29°C. Because this is roughly the average temperature of the California variable temperature test, similar permeation rates would be expected. The untreated fuel tanks showed slightly lower permeation over the constant temperature test. This difference was likely due to the difference in the temperature used for the testing. However, the fluorinated fuel tanks showed an increase in permeation. This increase in permeation appears to be the result of the 1.5 year additional fuel soak. Even after this long fuel soak, the fluorination achieves more than a 95% reduction in permeation. Table 7.2-7 presents this comparison.

Table 7.2-7. Permeation Rates [g/m²/day] for Fluorinated Fuel Tanks Tested by ARB and EPA on CA Certification Gasoline with a 1½ Year Fuel Soak Differential

Technology Configuration	Temperature	Tank 1	Tank 2	Tank 3	Average
Baseline, CARB testing	18-41°C	12.1	12.1	12.1	12.1
Baseline, EPA testing after 1.5 year additional fuel soak	29°C % change	11.5 -5%	11.4 -6%	11.2 -7%	11.4 -6%
Fluorinated, CARB testing	18-41°C	0.07	0.11	0.05	0.08
Fluorinated, EPA testing after 1.5 year additional fuel soak	29°C % reduction from EPA baseline	0.56 95%	0.62 95%	0.22 98%	0.47 96%

After the above testing, we drained the fuel tanks and filled them with certification gasoline splash-blended with 10% ethanol (E10). We then soaked the fuel tanks for 20 weeks to precondition them on this fuel. Following the preconditioning, we tested these fuel tanks for permeation at 29°C (85°F). Table 7.2-8 presents these emission results compared to the emission results for three baseline tanks (untreated) that were subject to the same preconditioning. Percent reductions are presented based on the difference between the fluorinated fuel tanks and the average results of the three untreated fuel tanks. The slight increase in permeation on the E10 fuel was similar for the baseline and fluorinated fuel tanks and still resulted in reductions above 95 percent.

Table 7.2-8. Permeation Rates for Fluorinated Fuel Tanks on E10 Fuel at 29°C

Technology Configuration	Units	Tank 1	Tank 2	Tank 3	Average
Baseline (untreated)	g/m ² /day	13.9	13.7	14.4	14.0
Fluorinated	g/m ² /day % reduction	0.43 97%	0.62 96%	0.62 96%	0.56 96%

Another study also looked at the effect of alcohol in the fuel on permeation rates from fluorinated fuel tanks.²⁰ In this study, the fuel tanks were tested with both gasoline and various methanol blends. No significant increase in permeation due to methanol in the fuel was observed.

One automobile manufacturer used fluorination to reduce permeation on HDPE fuel tanks to meet the LEV I vehicle standards. This manufacturer used similar or more stringent requirements for fuel soak, durability, and testing than finalized today. At 40°C, this manufacturer stated that they measured 0.15-0.2 g/day for fluorinated tanks compared to over 10 g/day for untreated HDPE fuel tanks.²¹

7.2.3 Barrier Platelets

Another approach for reducing permeation emissions is to blend a low permeable resin in with the HDPE and extrude it with a single screw. The low permeability resin, typically ethylene vinyl alcohol (EVOH) or nylon, creates non-continuous platelets in the HDPE fuel tank which reduce permeation by creating long, tortuous pathways that the hydrocarbon molecules must navigate to pass through the container walls. The trade name typically used for this permeation control strategy is Selar® for nylon and Selar RB® for EVOH. Although the barrier is not continuous, this strategy can still achieve greater than a 90 percent reduction in permeation of gasoline. EVOH has much higher permeation resistance to alcohol than nylon; therefore, it would be the preferred material to use for meeting our new standard, which is based on testing with a 10 percent ethanol fuel.

We tested several portable PFCs and marine fuel tanks molded with low permeation non-continuous barrier platelets. Six of the containers tested were constructed using nylon as the barrier material. The remainder of the containers were constructed using EVOH as the barrier material. The sixth container was tested on E10 (10% ethanol) to evaluate the effectiveness of this material with alcohol blended fuel. The containers with the EVOH barrier were all tested on E10.

Testing was performed after the containers had been filled with fuel and stored at room temperature. We soaked the containers with gasoline for 22 weeks and the tanks with E10 for 37 weeks. The purpose of the soak period was to ensure that the fuel permeation rate had stabilized. The containers were drained and then filled with fresh fuel prior to the permeation tests. We did not run slosh and pressure tests on these containers. However, because the barrier platelets are integrated in the can wall material, it is not likely that pressure or slosh testing would significantly affect the performance of this technology.

Table 7.2-9 presents the results of the permeation testing on the containers with barrier platelets. These test results show more than an 80 percent reduction for the nylon barrier tested on gasoline. However, the nylon barrier does not perform as well when a fuel with a 10% ethanol blend is used. Testing on a pair of 2 gallon containers with nylon barrier showed 80% percent higher emissions when tested on E10 than on gasoline. We also tested PFCs that used EVOH barrier platelets. EVOH has significantly better resistance to permeation on E10 fuel than nylon. For the containers blended with 6% EVOH, we observed a permeation rate of about 0.08-0.09 g/gal/day on E10 fuel.

Table 7.2-9. Permeation Rates for Plastic Fuel Containers with Barrier Platelets Tested by EPA at 29°C

Percent Selar®*	Capacity [gallons]	Test Fuel	Fuel Soak [weeks]	g/gal/day	g/m ² /day
Nylon barrier platelets					
unknown**	2	gasoline	40	0.54	–
unknown**	2	E10	40	0.99	–
4%	5	gasoline	22	0.35	4.1
4%	5.3	gasoline	22	0.11	1.2
4%	6.6	gasoline	22	0.15	1.6
4%	6.6	gasoline	22	0.14	1.5
EVOH barrier platelets					
2%	6.6	E10	37	0.23	3.0
4%	6.6	E10	37	0.14	1.9
4%	6.6	E10	37	0.15	2.0
6%	6.6	E10	37	0.08	1.4
6%	6.6	E10	37	0.09	1.4

*trade name for barrier platelet technology used in test program

** designed to meet California permeation requirement

Manufacturers raised a concern about whether or not a container using barrier platelets would have a stabilized permeation rate after 20 weeks. In other words, manufacturers were concerned that this technology may pass the test, but have a much higher permeation rate in-use. We tested one of the 4% and 6% EVOH containers on E10 again after soaking for a total of 104 weeks (2 years). The measured permeation rates were 2.0 and 1.4 g/m²/day for the 4% and 6% EVOH containers, respectively, which represents no significant changes in permeation from the 37 week tests. In contrast, we measured the 4% nylon tanks again after 61 weeks and measured permeation rates of 2.8 and 2.7 g/m²/day, which represented about an 80-90% increase in permeation compared to the 22 week tests.

The California ARB collected test data on permeation rates from PFCs molded with Selar® low permeation non-continuous barrier platelets using California certification fuel. This data was collected using a diurnal cycle from 65-105°F. The results show that this technology can be used to achieve significant reductions in permeation from plastic fuel containers. This test data showed that more than a 90 percent reduction in permeation is achievable through the use of barrier platelets. However, all of this testing was performed on California certification fuel, which does not include ethanol.

Table 7.2-10. Permeation Rates for PFCs with Barrier Platelets Tested by ARB on California Fuel

Percent Selar®*	Container Capacity [gallons]	Permeation Loss [g/gal/day]
4% (average =0.12 g/gal/day)	5	0.08
	5	0.09
	5	0.13
	5	0.16
	5	0.17
	6	0.08
	6	0.10
6% (average =0.09 g/gal/day)	5	0.07
	5	0.07
	5	0.07
	5	0.08
	5	0.12
	5	0.17
	6	0.06
8% (average =0.07 g/gal/day)	5	0.08
	5	0.10
	6	0.05
	6	0.06

*trade name for barrier platelet technology used in test program

Table 7.2-11 presents permeation rates for HDPE and three Selar RB® blends when tested at 60°C on xylene.²² Xylene is a component of gasoline and gives a rough indication of the permeation rates on gasoline. This report also shows a reduction of 99% on naphtha and 98% on toluene for 8% Selar RB®.

Table 7.2-11. Xylene Permeation Results for Selar RB® at 60°C

Composition	Permeation, g mm/m ² /day	% Reduction
100% HDPE	285	—
10% RB 215/HDPE	0.4	99.9%
10% RB 300/HDPE	3.5	98.8%
15% RB 421/HDPE	0.8	99.7%

7.2.4 Multi-Layer Construction

PFCs may also be constructed out of multiple layers of materials, and some PFC manufacturers have started using this technology. In this way, the low cost and structural advantages of traditional materials can be utilized in conjunction with higher grade materials which can provide effective permeation resistance.

Coextruded barrier technology has been long established for blow-molded automotive fuel tanks. Data from one automobile manufacturer showed permeation rates of 0.01-0.03 g/day for coextruded fuel tanks at 40°C on EPA certification fuel. They are using this technology to meet LEV II vehicle standards. For comparison, they reported permeation rates of more than 10 g/day for standard HDPE fuel tanks.²³

Another study looks at the permeation rates, using ARB test procedures, through multi-layer vehicle fuel tanks.²⁴ The fuel tanks in this study were 6 layer coextruded plastic tanks with EVOH as the barrier layer (3% of wall thickness). The outer layers were HDPE and two adhesive layers were needed to bond the EVOH to the polyethylene. The sixth layer was made of recycled polyethylene. The two test fuels were a 10 percent ethanol blend (CE10) and a 15 percent methanol blend (CM15). See Table 7.2-12.

Table 7.2-12. Permeation Results for a Coextruded Fuel Tank Over a 65-105°F Diurnal

Composition	Permeation, g/day	% Reduction
100% HDPE (approximate)	6 - 8	–
3% EVOH, 10% ethanol (CE10)	0.2	97%
3% EVOH, 15% methanol (CM15)	0.3	96%

7.3 Diurnal Emissions

The above sections discuss permeation emissions and permeation emissions control. These emissions are part of the overall evaporative emissions, or diurnal emissions, from PFCs. PFCs as a system also emit evaporative emissions from seals and spouts. PFCs have high evaporative emissions when they are left open. In order to meet emissions standards, manufacturers would use cans with spouts that automatically close and seal well around the opening to the can where the spout attaches. Automatic closing spouts have been designed for the California program. These spouts are typically manufactured with springs that close the cans automatically when the cans are not being used to refill equipment. In addition, these cans vent through the spouts, and the vents typically found on the back of the cans are removed. This is important because open vents can be a significant source of evaporative emissions.

CARB conducted a feasibility study for their PFC standards and concluded that a 0.3 g/gal/day standard was feasible in the 2009 time-frame.²⁵ CARB conducted testing of three different PFCs designed to meet emissions standards. They were tested in two ways: with the spout attached and with the spouts removed and the PFCs sealed. The results for the sealed cans

represent the amount of permeation emissions observed. This data was collected using a diurnal cycle from 65-105°F with 7 RVP fuel. As noted above, the results would be similar if the data were collected at the temperature range and fuel used by EPA of 72-96°F with 9 psi RVP fuel, because the lower temperature and higher RVP offset one another. The PFCs with spout were soaked for 160 days and the sealed cans were soaked for 174 days prior to testing. The results of the testing are provided below in Table 7.3-1. The results show the average of three identical cans per manufacturer. CARB did not identify the manufacturers or the permeation barriers used.

Table 7.3-1. Results of CARB Diurnal Testing (g/gal/day)

	Sealed PFC	PFC w/ Spout
Manufacturer A	0.1	0.2
Manufacturer B	0.0	0.7
Manufacturer C	0.2	0.2

CARB indicated that the results from Manufacturer B increased because of one faulty spout which significantly increased the average emissions. The results indicate that the 0.3 g/gal./day standard is feasible. The results also indicate that a faulty spout or seal around the opening of the PFC would likely lead to emissions significantly above the standard. Manufacturers would need to focus on controlling variability in their manufacturing process to ensure spouts are durable and well matched to the PFCs and do not allow evaporative emissions to escape.

7.4 Testing Procedures

The test procedure for diurnal emissions is to place the PFC with the spout attached in a SHED^B, vary the temperature over a prescribed profile, and measure the hydrocarbons escaping from the fuel container. The final result would be reported in grams per gallon where the grams are the mass of hydrocarbons escaping from the fuel tank over 24 hours and the gallons are the nominal PFC capacity. The test procedure is based on the automotive evaporative emission test described in 40 CFR Part 86, Subpart B, with modifications specific to PFC applications. The hydrocarbon loss must be measured either by weighing the cans before and after the diurnal cycle or by measuring emissions directly from the SHED. Three identical containers must be tested for three diurnal cycles. The daily emissions for each container are to be averaged together for comparison with the standard, rounded to the nearest one-tenth of a gram. Each container must meet the standard to demonstrate compliance with the standard.

Manufacturers must test cans in their most likely storage configuration. The key to reducing evaporative losses from PFCs is to ensure that there are no openings on the cans that could be left open by the consumer. Traditional cans have vent caps and spout caps that are easily lost or left off cans, which leads to very high evaporative emissions. We expect manufacturers to meet the evaporative standards by using automatic closing spouts and by

^B Sealed Housing for Evaporative Determination

removing other openings that consumers could leave open. However, if manufacturers choose to design cans with an opening that does not close automatically, we are requiring that containers be tested in their open condition. If the PFCs have any openings that consumers could leave open (for example, vents with caps), these openings thus must be left open during testing. This applies to any opening other than where the spout attaches to the can. We believe it is important to take this approach because these openings could be a significant source of in-use emissions.

Spouts must be in place during testing because this would be the most likely storage configuration for the emissions compliant cans. Spouts will likely still be removable so that consumers will be able to refill the cans, but we would expect the containers to be resealed by consumers after being refilled in order to prevent spillage during transport. We do not believe that consumers will routinely leave spouts off cans, because spouts are integral to the cans' use and it is obvious that they need to be sealed. Testing with spouts in place will also ensure that the cans seal properly at the point where the nozzle attaches to the can. If cans do not seal properly, emissions will be well above the standards.

7.4.1 Temperature Profile, Length of Test, Fill Level

PFCs will be tested over the same 72-96°F (22.2-35.6°C) temperature profile used for automotive applications. This temperature profile represents a hot summer day when ground level ozone emissions (formed from hydrocarbons and oxides of nitrogen) would be highest. This temperature profile would be for the air temperature in the SHED.

The automotive diurnal test procedure includes a three-day temperature cycle. The purpose of this test length is to ensure that the carbon canister can hold at least three days of diurnal emissions without vapor breaking through the canister. For PFCs, we do not believe that a three-day test is necessary. Prior to the first day of testing, the fuel will be stabilized at the initial test temperature. Following this stabilization, a single 24-hour diurnal temperature cycle will be run. Because this technology does not depend on purging or storage capacity of a canister, multiple diurnal cycles per test should not be necessary.

Diurnal emissions are not only a function of temperature and fuel volatility, but of the size of the vapor space in the PFC as well. The fill level at the start of the test will be 50% of the nominal capacity of the PFC. Nominal capacity, defined as the volume of fuel to which the PFC can be filled when sitting in its intended position, is to be specified by the manufacturer. The vapor space that normally occurs in a PFC, even when "full," is not considered to be part of the nominal capacity of the PFC.

7.4.2 Test Fuel

Consistent with the automotive test procedures, we are requiring that the test take place using 9 RVP certification gasoline. About 20-30% of fuel sold in the U.S. contains ethanol and this percentage is expected to increase due to the Energy Policy Act. We are requiring the use of E10, which is a blend of 90% certification gasoline blended with 10% ethanol for diurnal testing

of PFCs. As noted in Section 7.2, ethanol in the fuel can increase permeation emissions for some permeation barriers such as nylons if not properly accounted for in the design of the PFCs. Other available permeation barriers do not allow significantly higher emissions when ethanol is present in the fuel. Testing with E10 helps ensure that manufacturers would select materials with emissions performance that does not degrade significantly when ethanol is present in the fuel.

7.4.3 Preconditioning and Durability Testing

We are applying essentially the same preconditioning and durability testing requirements for PFCs that we have established for permeation control requirements for recreational vehicles. We are also requiring a durability demonstration for spouts. As with the diurnal testing, the preconditioning and durability testing are to be performed on the complete PFC with the spout attached (except for pressure cycling as noted below).

7.4.3.1 Preconditioning

It takes time for fuel to permeate through the walls of containers. Permeation emissions will increase over time as fuel slowly permeates through the container wall, until the permeation finally stabilizes when the saturation point is reached. We want to evaluate emissions performance once permeation emissions have stabilized, to ensure that the emissions standard is met in-use. Therefore, we are requiring that prior to testing the PFCs, the cans need to be preconditioned by allowing the can to sit with fuel in them until the hydrocarbon permeation rate has stabilized. Under this step, the PFC must be filled with E10, sealed, and soaked for 20 weeks at a temperature of $28 \pm 5^\circ\text{C}$. As an alternative, we are allowing that the fuel soak could be performed for 10 weeks at $43 \pm 5^\circ\text{C}$ to shorten the test time. During this fuel soak, the PFCs must be sealed with the spout attached. We have established these soak temperatures and durations based on protocols EPA has established to measure permeation from fuel tanks made of HDPE.²⁶ These soak times should be sufficient to achieve stabilized permeation emission rates. However, if a longer time period is necessary to achieve a stabilized rate for a given PFC, we are requiring that the manufacturer to use a longer soak period (and/or higher temperature) consistent with good engineering judgment.

7.4.3.2 Durability Testing

To account for permeation emission deterioration, we are specifying three durability aging cycles: slosh, pressure-vacuum cycling, and ultraviolet (UV) exposure. They represent conditions that are likely to occur in-use for PFCs, especially for those cans used for commercial purposes and carried on truck beds or trailers. The purpose of these deterioration cycles is to help ensure that the technology chosen by manufacturers is durable in-use, represents best available control, and the measured emissions are representative of in-use permeation rates. Fuel slosh, pressure cycling, and UV exposure each impact the durability of certain permeation

barriers, and we believe these cycles are needed to ensure long-term emissions control. Without these durability cycles, manufacturers could choose to use materials that meet the certification standard but have degraded performance in-use, leading to higher emissions. We do not expect these procedures to adversely impact the feasibility of the standards, because there are permeation barriers available at a reasonable cost that do not deteriorate significantly under these conditions. As described above, we believe including these cycles as part of the certification test is preferable to a design-based requirement.

For slosh and pressure cycling, we are requiring the use of durability tests that are based on draft recommended Society of Automotive Engineers (SAE) practice for evaluating permeation barriers.²⁷ For slosh testing, the PFC must be filled to 40 percent capacity with E10 fuel and rocked for 1 million cycles. The pressure-vacuum testing contains 10,000 cycles from -0.5 to 2.0 psi. The pressure cycling may be performed by applying pressure/vacuum through the opening where the spout attaches, rather than by drilling a hole in the container. The third durability test is intended to assess potential impacts of UV sunlight (0.2 μm - 0.4 μm) on the durability of a surface treatment. In this test, the PFCs must be exposed to a UV light of at least 0.40 Watt-hour/meter² /minute on the PFC surface for 15 hours per day for 30 days. Alternatively, PFCs may be exposed to direct natural sunlight for an equivalent period of time. We have also established these same durability requirements as part of our program to control permeation emissions from recreational vehicle fuel tanks.²⁸ While there are obvious differences in the use of PFCs compared to the use of recreational vehicle fuel tanks, we believe the test procedures offer assurance that permeation controls used by manufacturers will be robust and will continue to perform as intended when in use.

We are also allowing manufacturers to do an engineering evaluation, based on data from testing on their permeation barrier, to demonstrate that one or more of these factors (slosh, UV exposure, and pressure cycle) do not impact the permeation rates of their PFCs and therefore that the durability cycles are not needed. Manufacturers would use data collected previously on PFCs or other similar containers made with the same materials and processes to demonstrate that the emissions performance of the materials does not degrade when exposed to slosh, UV, and/or pressure cycling. The test data must be collected under equivalent or more severe conditions as those noted above.

In its recently revised program for PFCs, California included a durability demonstration for spouts. We are requiring a durability demonstration consistent with California's procedures. Automatically closing spouts are a key part of the emissions controls expected to be used to meet the new standards. If these spouts stick or deteriorate, in-use emissions could remain very high (essentially uncontrolled). We are interested in ways to ensure during the certification procedures that the spouts also remain effective in use. California requires manufacturers to actuate the spouts 200 times prior to the soak period and 200 times near the conclusion of the soak period to simulate spout use. The spouts' internal components are required to be exposed to fuel by tipping the can between each cycle. Spouts that stick open or leak during these cycles are considered failures. The total of 400 spout actuations represents about 1.5 actuations per week on average over the average container life of 5 years. In the absence of data, we believe this

number of actuations appears to reasonably replicate the number that can occur in-use and will help ensure quality spout designs that do not fail in-use. We also believe that adopting requirements consistent with California will help manufacturers to avoid duplicate testing.

The order of the durability tests would be optional. However, as discussed above, we require that the PFC be soaked to ensure that the permeation rate is stabilized just prior to the final permeation test. If the slosh test is run last, the length of the slosh test may be considered as part of this soak period. Where possible, the deterioration tests may be run concurrently. For example, the PFC could be exposed to UV light during the slosh test. In addition, if a durability test can clearly be shown to not be necessary for a given product, manufacturers may petition to have the test waived. For example, manufacturers may have data showing that their permeation barrier does not deteriorate when exposed to the conditions represented by the test procedure.

After the durability testing, once the permeation rate has stabilized, the PFC is drained and refilled with fresh fuel, the spout is placed back on the container, and the PFC is tested for diurnal emissions.

7.4.4 Reference Container

We are requiring the use of a reference container during testing. In cases where the permeation of a PFC is low, and the PFC is properly sealed, the effect of air buoyancy can have a significant effect the measured weight loss. Air buoyancy refers to the effect of air density on the perceived weight of an object. As air density increases, it will provide an upward thrust on the PFC and create the appearance of a lighter container. Air density can be determined by measuring relative humidity, air temperature, and air pressure.²⁹

One testing laboratory presented data to EPA on their experience with variability in weight loss measurements when performing permeation testing on PFCs.³⁰ They found that the variation was due to air buoyancy effects. By applying correction factors for air buoyancy, they were able to greatly remove the variation in the test data. A technical brief on the calculations they used is available in the docket.³¹

A more direct approach to accounting for the effects of air buoyancy is to use a reference container. In this approach, an identical PFC to that being tested would be tested without fuel in it and used as a reference PFC. Dry sand would be added to this PFC to make up the difference in mass associated with the test cans being half full of fuel. The reference PFC would then be sealed so that the buoyancy effect on the reference PFC would be the same as the test PFCs. The measured weight loss of the test PFC could then be corrected by any measured changes in weight in the reference can. The California Air Resources Board has required this approach for measuring PFC emissions, and they refer to the reference PFC as a “trip blank.”³²

References for Chapter 7

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