Welcome to the

ETHANOL FUELS WORKSHOP

Meeting the Demands of Fuel Standards with Ethanol

Tennessee Department of Agriculture
Regulatory Services Division

Sponsored by
Tennessee Fuel and
Convenience Store Association
Tennessee Petroleum Council

In Partnership With
Clean Cities of Tennessee
Tennessee Department of Environment
and Conservation
Tennessee Department of
Transportation

E10 Fuels – Issues and Solutions

- ✓ Review Fundamental Requirements for Ethanol Blends
- ✓ Summer Volatility Control Period
- ✓ Ethanol and Octane Boost – An Advantage for Ethanol
- ✓ Ethanol and Vapor Pressure Boost – A Disadvantage for Ethanol
- ✓ Phase Separation – The Most Critical Control for E10 Blends
- ✓ Violation Trends
- ✓ Pump Labels and UST Marking Requirements

E10 Fuels Requirements

- ✓ All Fuel Standards in TN are based on ASTM International Standards and Tennessee Rules
  - ASTM D4814 is the governing document for Automotive Spark-Ignition Engine Fuel, i.e., gasoline and gasoline-ethanol blends.
  - Covers important parameters such as volatility and workmanship (water, sediment, and other adulterants), gum formation, and corrosion, and 10th percentile temperatures for phase separation.
  - References all Standard Test Methods that are to be used to verify the characteristics of the fuel – from distillation to octane.
- ✓ Legislative Authority T.C.A. 47-18-1301 et seq.

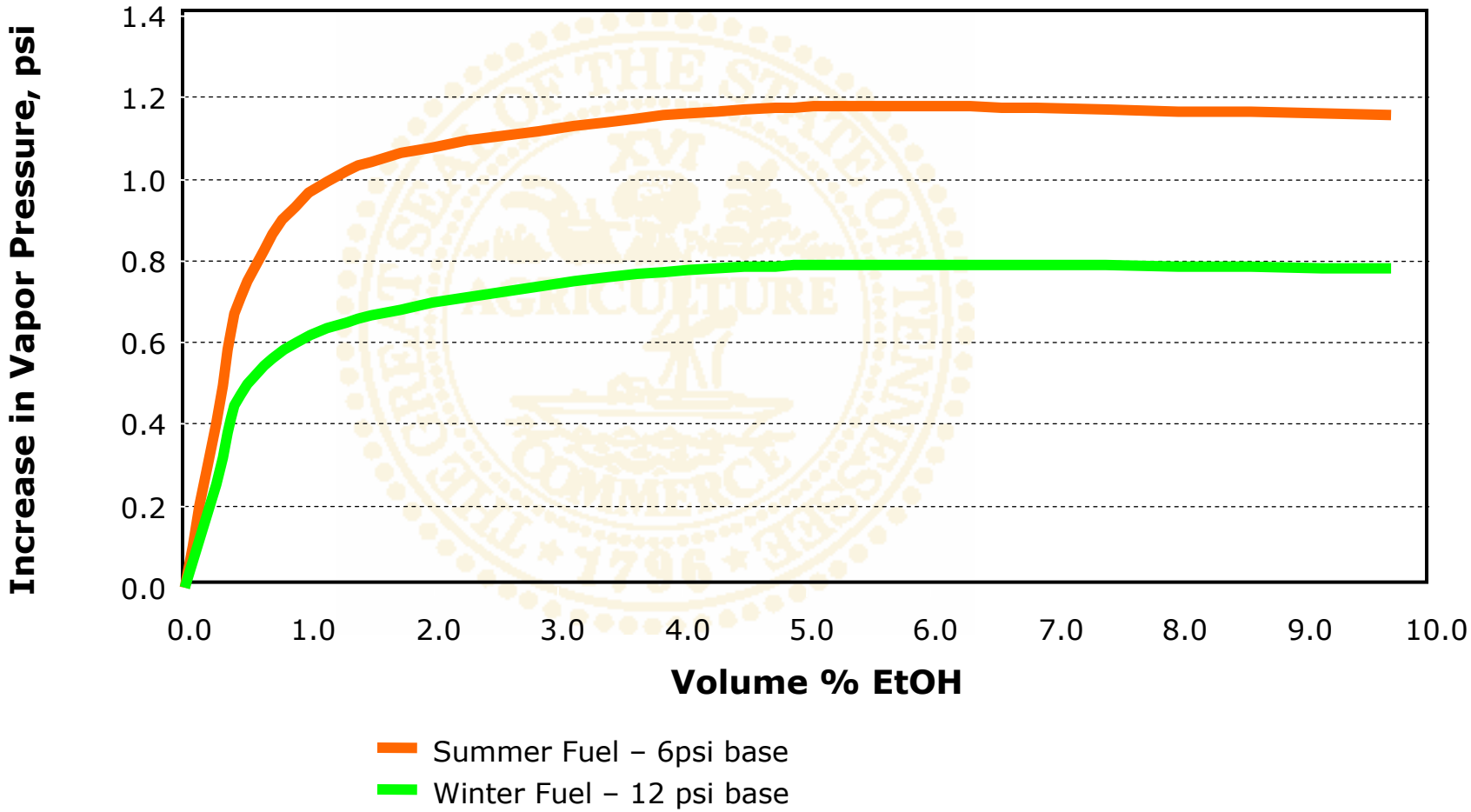
Summer Volatility Control Period Coming Up Soon!

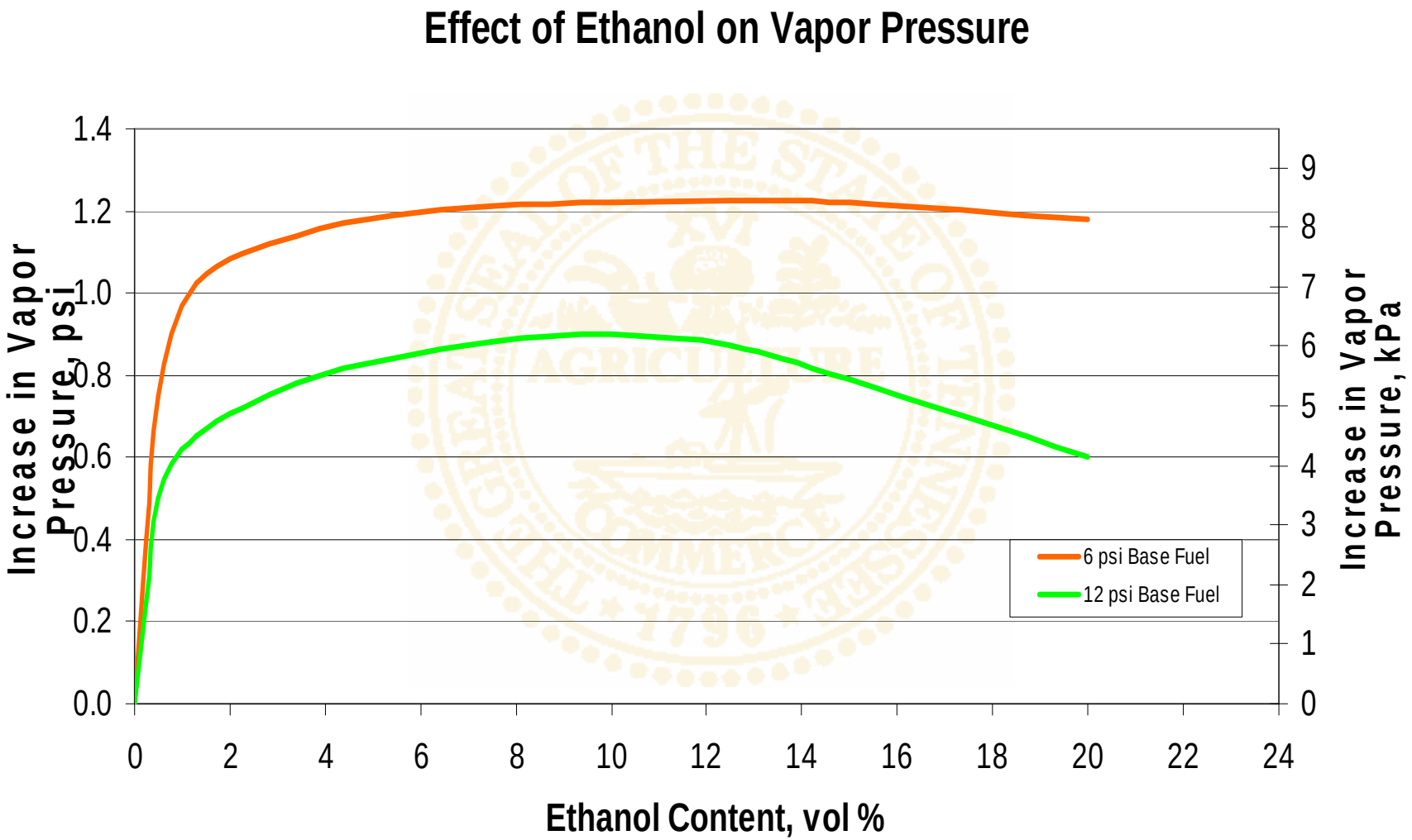
- ✓ Summer EPA Control Period: **May 1 – September 15**
- ✓ Tennessee Maximum Vapor Pressure is **9.0 psi**, except for the following counties:
  - **Davidson, Rutherford, Sumner, Williamson, Wilson and Shelby**

These counties have a **Maximum VP of 7.8 psi**

Ethanol blends have a 1 psi exemption; HOWEVER, to qualify **the ethanol content must be between 9 and 10 volume %!**

Effects of Ethanol on Vapor Pressure

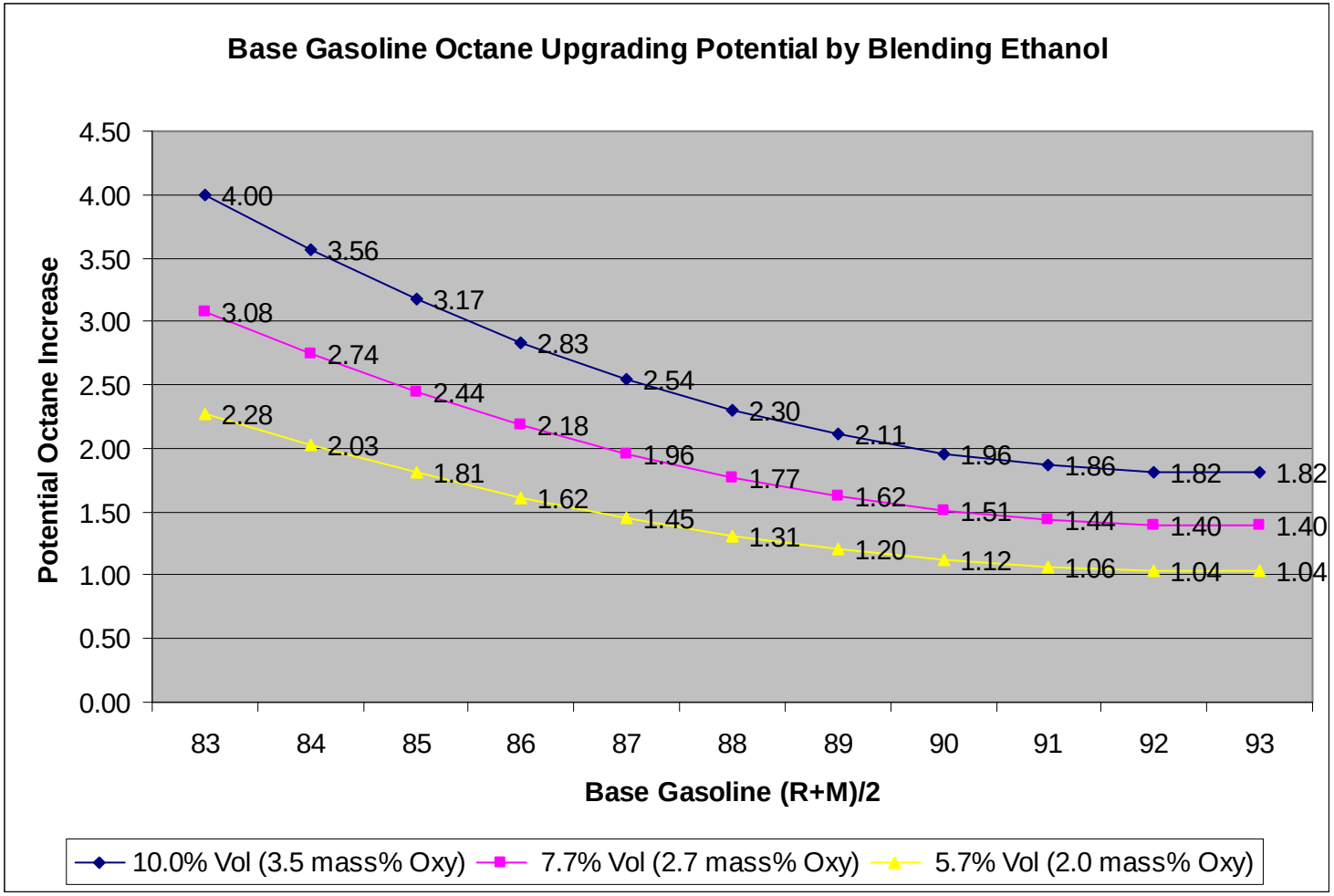



Effect of Ethanol on Vapor Pressure
Increase in Vapor Pressure, psi
Increase in Vapor Pressure, kPa
Ethanol Content, vol %
6 psi Base Fuel
12 psi Base Fuel
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
0
1
2
3
4
5
6
7
8
9
0
2
4
6
8
10
12
14
16
18
20
22
24

Preparing for the Summer Volatility Control Period

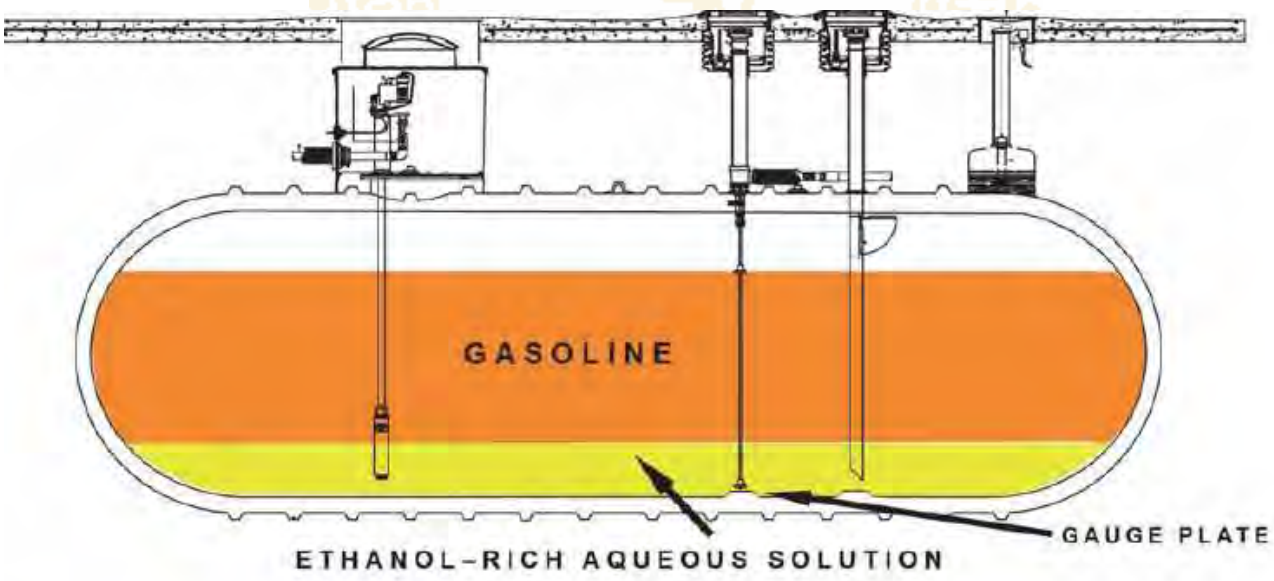
- ✓ Check with your suppliers on anticipated delivery of 7.8 and 9.0 psi fuel.
- ✓ If blending EtOH, make sure that you maintain a 9-10% blend level.
- ✓ Run your retail tanks Low as you approach June 1 and begin to dilute with summer volatility fuel.
- ✓ Know Your Customers and Serve Their Needs:
  - Begin to advise retail owners on the need to take minimal delivery of pre-summer grade fuel prior to the availability 7.8/9.0 fuel.
  - Especially critical of Low Volume and Seasonal locations
    - Boat docks, small retailers, along with their suppliers, face penalties each year that could have been avoided.

Blending Ethanol & Octane Boost



Phase Separation

- ✓ E10 can remain stable at up to its saturation point with water; after the saturation point is reached the water and ethanol will begin to separate from solution. Stability decreases with cold weather and lower aromatic fuels.
- ✓ The Phase separation solution is more dense than gasoline, is polar, and will separate and sink to the bottom of the tank.



Phase Separation – What TDA Sees in the Field

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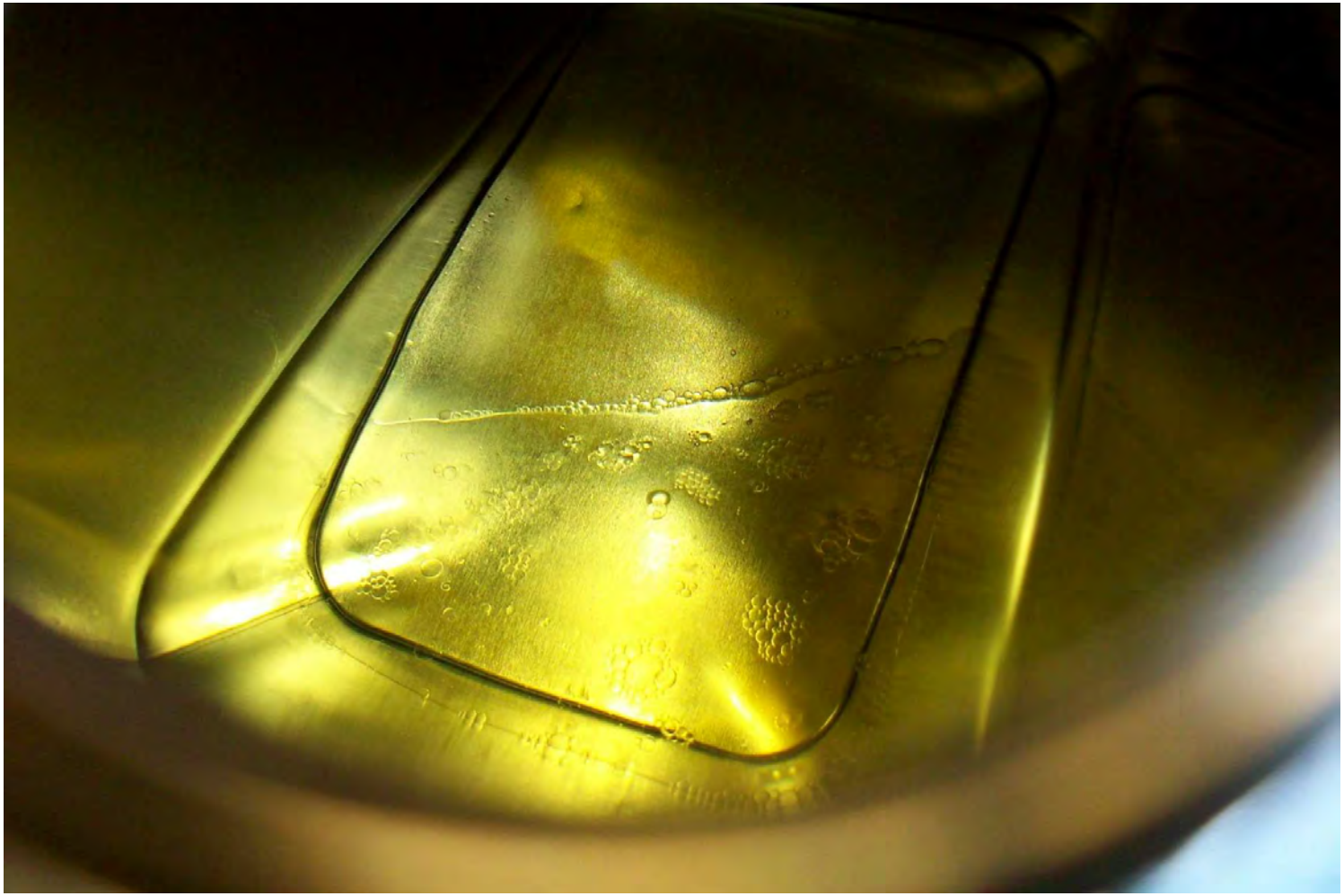
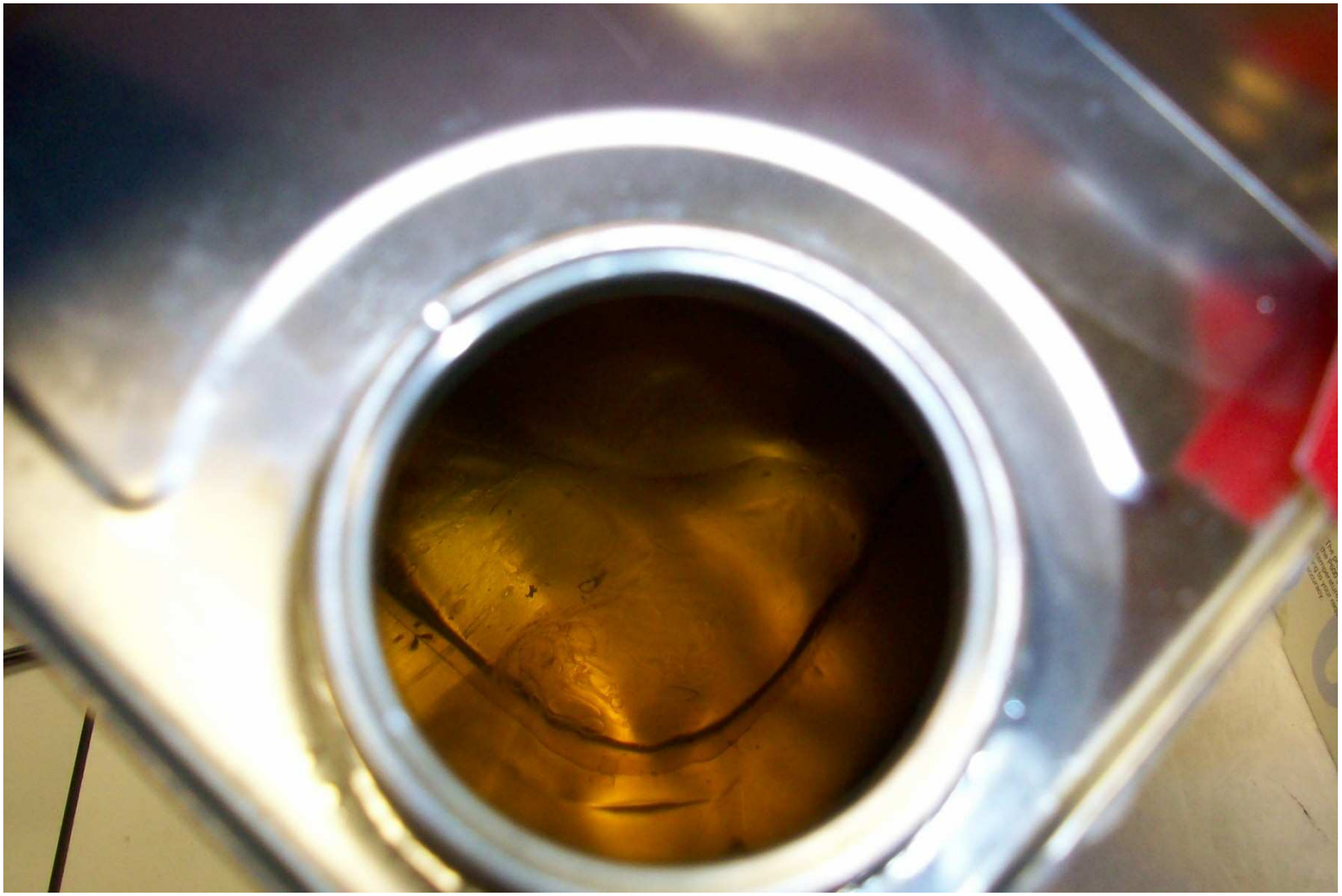


Phase Separation – What TDA Sees in the Field



Tennessee Phase Separation Seasonal Temperatures
10th Percentile 6-h minimum

(The highest temperature of the six coldest consecutive hourly temperature readings in a 24 h period)

Month	Temp, Deg. C (F)
January	-11 (12.2)
February	-8 (17.6)
March	-4 (24.8)
April	2 (35.6)
May	8 (46.4)
June-September	10 (50.0)
October	5 (41.0)
November	-3 (26.6)
December	-7 (19.4)

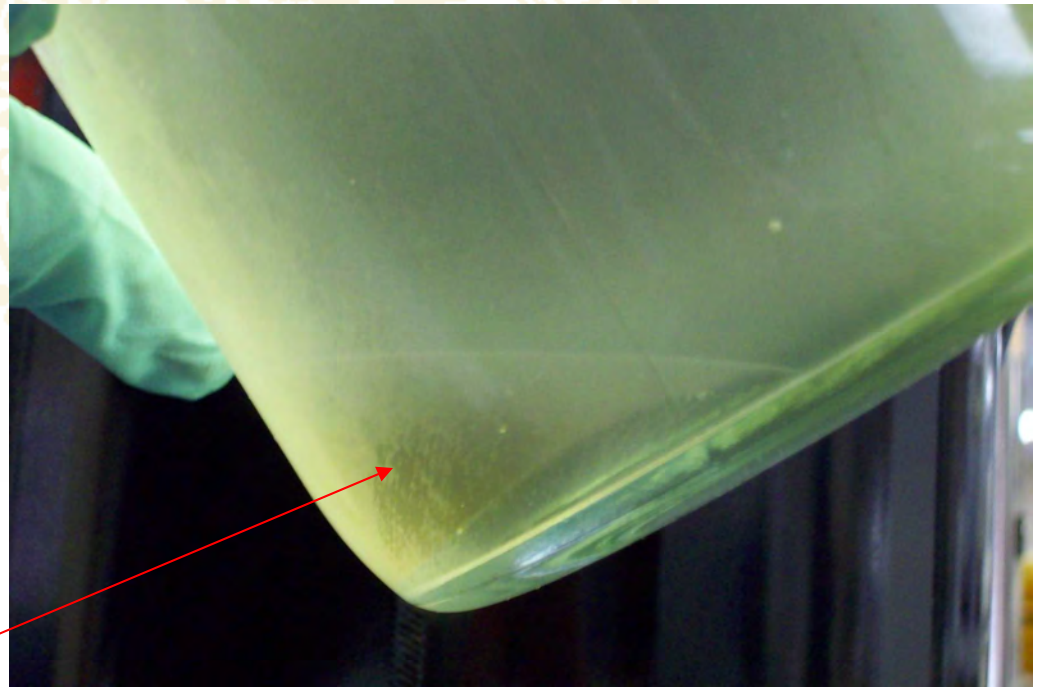
Phase Separation Project
Initiated to help in remediation advice

- ✓ Created a batch of E10 gasoline - blended from gasoline and EtOH samples taken from terminal.
- ✓ Two 1 Gal cans labeled as batch 1 & 2 observed with no signs of phase sep.
- ✓ EtOH used to make blends was pretested and contained 0.629% water.
- ✓ After hand blending to 10% DFE, batch 1 and 2 verified for EtOH – 9.43, 9.42%, respectively.

Phase Separation Project – Protocol 1

Objective – Determine a reasonable remediation step for minor phase separation

- ✓ Three samples selected to represent typical phase sep findings at retail.
- ✓ Sample chilled and shaken to mix then transferred to 1 qt. bottle.



Sample after chilling and shaking to bring fallout into solution. Hazy and phase observed at bottom.

Phase Separation Project Results – Protocol 1

- ✓ 400 mL of phased sample measured into graduated cylinder – transferred to another 1 qt. bottle and left to stand – phase was observed.
- ✓ 400 mL of E10 batch 1 was added and mixed for 20 s.
- ✓ After mixing under these conditions, the 1:1 mix was observed to be clear & bright.

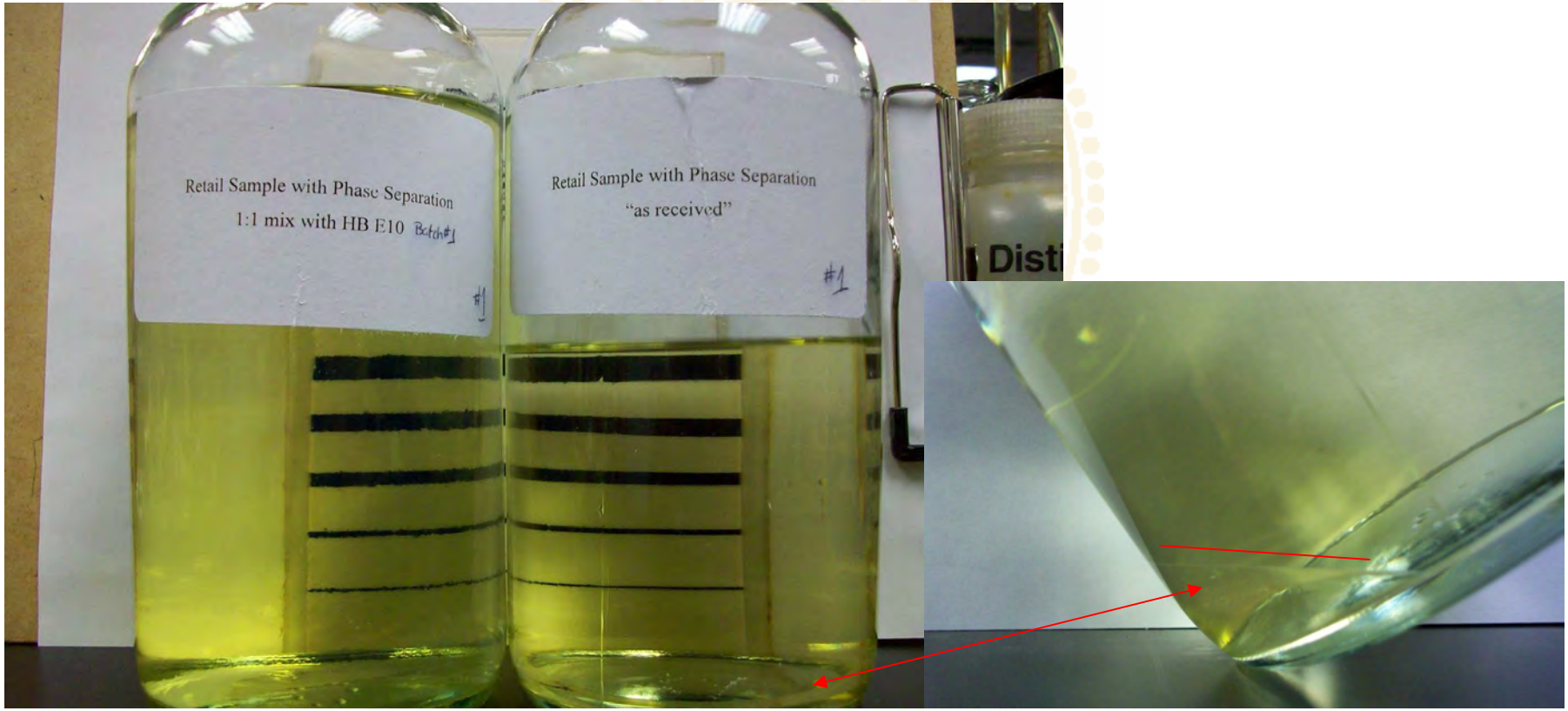
Sample after 1:1 at ~ 5 deg. C.

Results indicate that the 'typical' phase problems that we routinely encounter can be corrected by dilution of 1:1 with fresh E10.

NOTE: This is not a catastrophic phase separation solution!

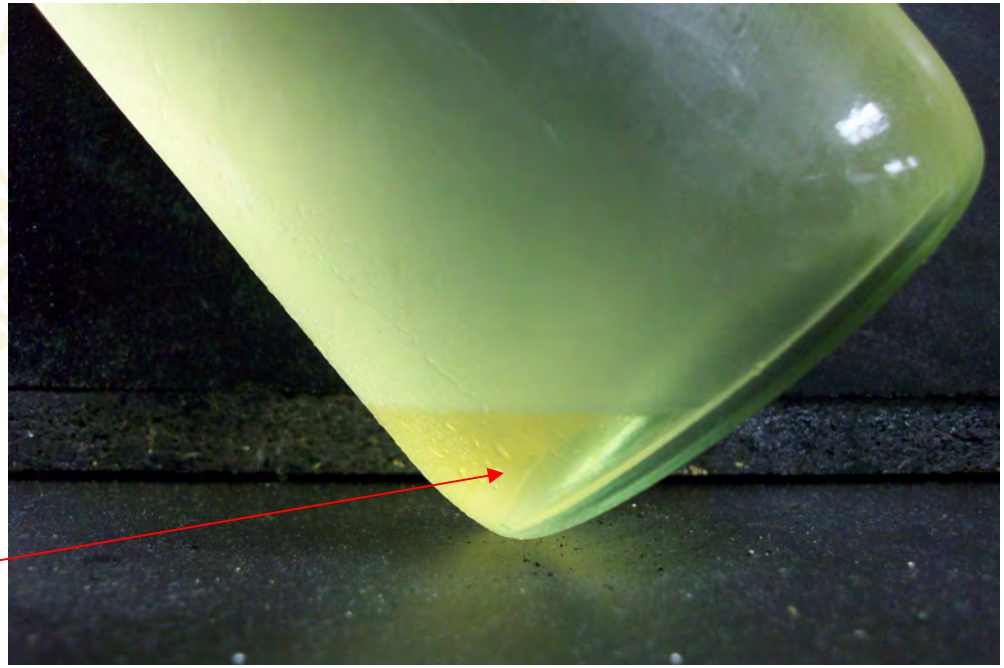
Phase Separation Project Results – Protocol 1

1:1 dilution vs. “as received” – Note phase sep remains in “as received”



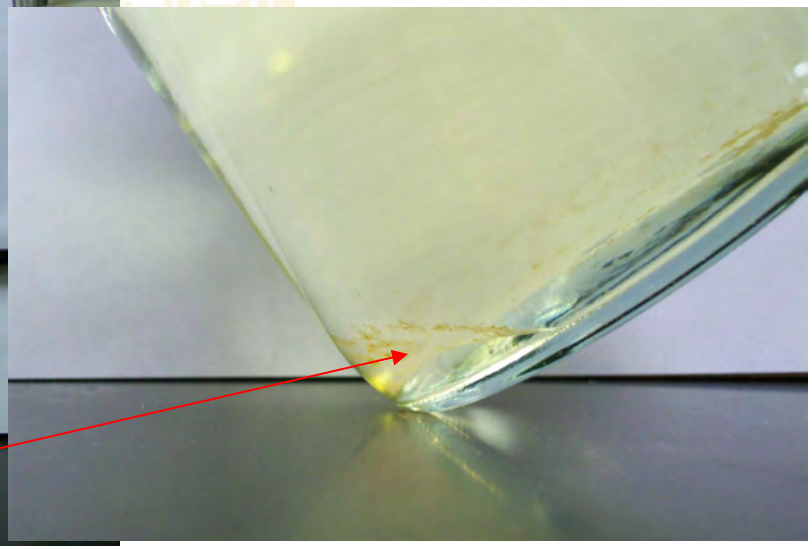
Phase Separation Project Results – Protocol 1

- ✓ A 2nd 'as received' sample was chilled, shaken, and transferred to glass bottle.
- ✓ Photos illustrate hazy and water condensation in corner of tilted bottle.



Phase Separation Project Results – Protocol 1

- ✓ Again, the 1:1 dilution corrected the problem with this fuel sample.
- ✓ A 3rd sample was tested and corrective actions were successful again.



Key Takeaway: 1:1 dilution with fresh, dry E10 corrects minor phase sep problems.

Phase Separation Project – Protocol 2
Objective – Determine if there are practical solutions to catastrophic phase separation issues.

- ✓ 4 L of E10 gasoline made from ethanol and terminal gasoline.
- ✓ Batch labeled as #3 - chilled – no phase separation observed.
- ✓ 10% water contamination introduced into a sample.
- ✓ Evaluation of Upper Phase for remediation attempted
- ✓ Fresh fuel was added up to 25:1 in an attempt to dilute the phase separation.

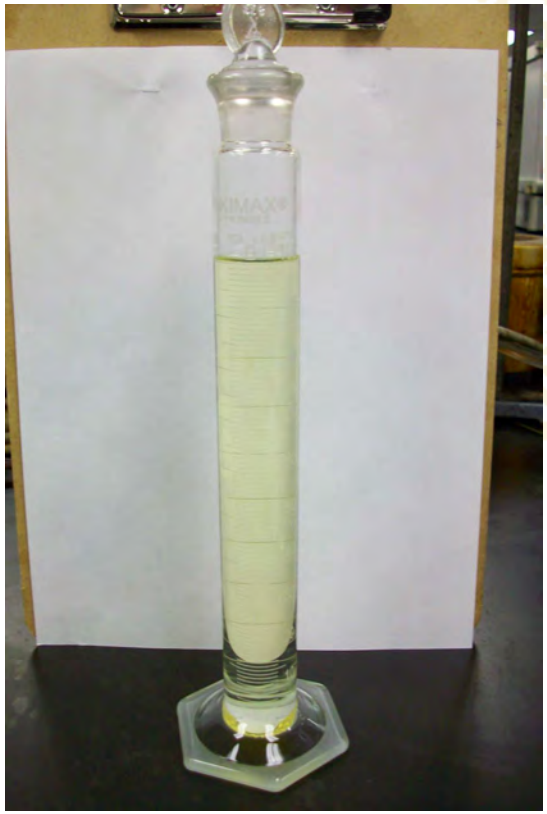
Phase Separation Project – Protocol 2

- ✓ Density determined on lower phase:
  - Lower phase 0.9417 @ 15C (water is 0.9991 – EtOH used was 0.7948).

- ✓ Upper phase tested for water and EtOH:
  - Water content was 382 ppm, mass
  - EtOH content was 1.34% mass

Phase Separation Project – Protocol 2

- ✓ 1:1 dilution of fresh batch #3 E10 was added to the UPPER phase of the 10% contamination batch.
- ✓ Sample was chilled and remained clear and bright.

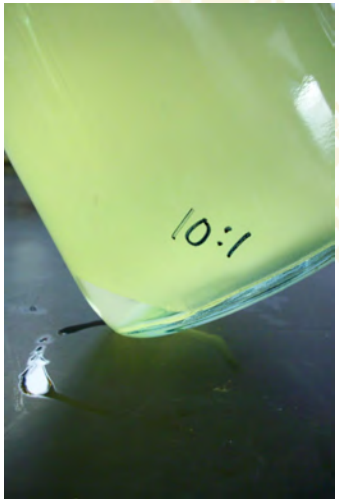


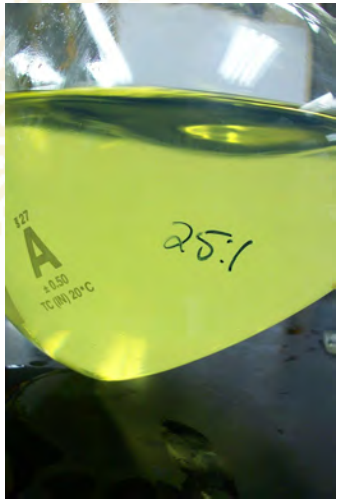
Key Takeaway: When catastrophic phase separation occurs, upper and lower phases can be separated, and upper phase salvaged.

NOTE: The loss of EtOH means loss of AKI. Laboratory analysis will have to be done to determine the exact blending to hit your target product grade!

Phase Separation Project – Protocol 2

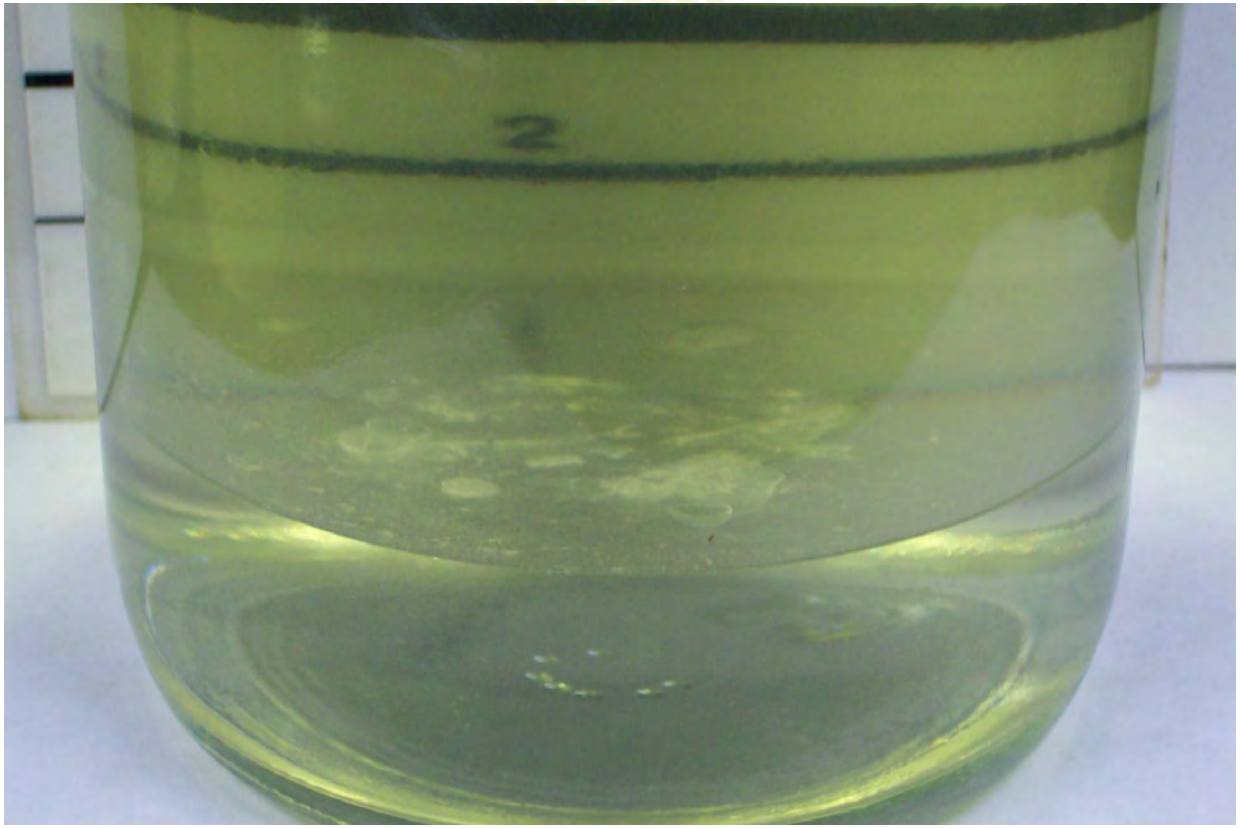
- ✓ Another 10% contamination sample of 40 mL was created.
- ✓ 40 mL increments of fresh batch #3 E10 was added until the dilution ratio was up to 25:1.
- ✓ At 25:1 phase separation was not observed at room temperature, **but after chilling the sample for 30 minutes, phase separation was observed!**
  - **We could not stabilize the contaminated fuel without first removing the lower phase.**





Phase Separation Project Results – Protocol 3
Objective – Evaluate 0.3 – 10% water contamination

E10 hand blends checked for phase sep – both clear & bright after chilling.



A close up of 10% contamination after resting at room temp. Is there a 3rd phase?

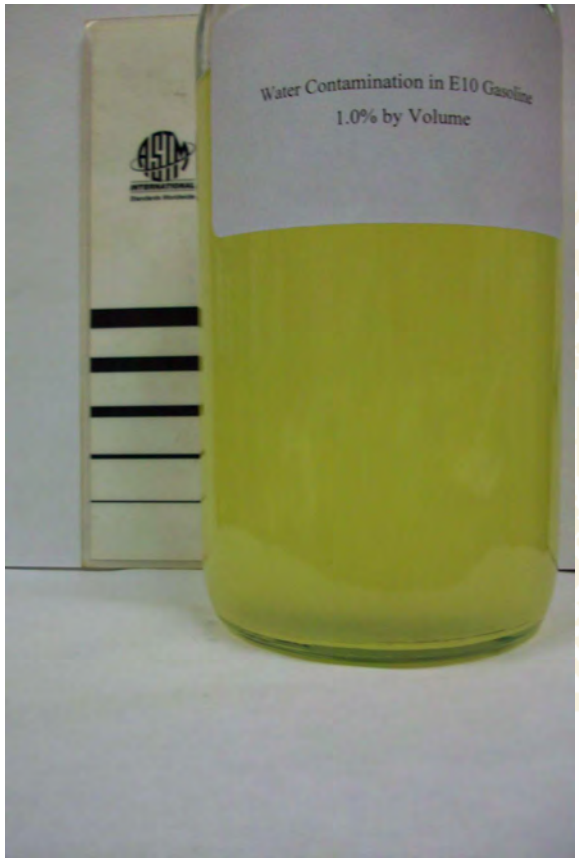
Phase Separation Project Results – Protocol 3

A portion was contaminated with **1%** DI water.

Immediately after mixing.

After shaking.

Phase Separation Project Results – Protocol 3



1% sample after warming to room temp and agitated.

1% sample after warming to room temp and resting. Sample remains hazy and bottom phase forms.

Phase Separation Project Results – Protocol 3

A portion was contaminated with **0.5%** DI water.

Immediately after mixing.

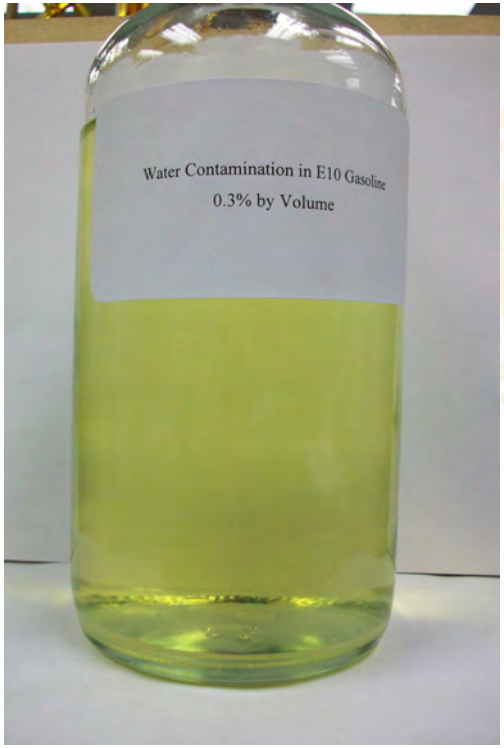
After shaking.

After resting.

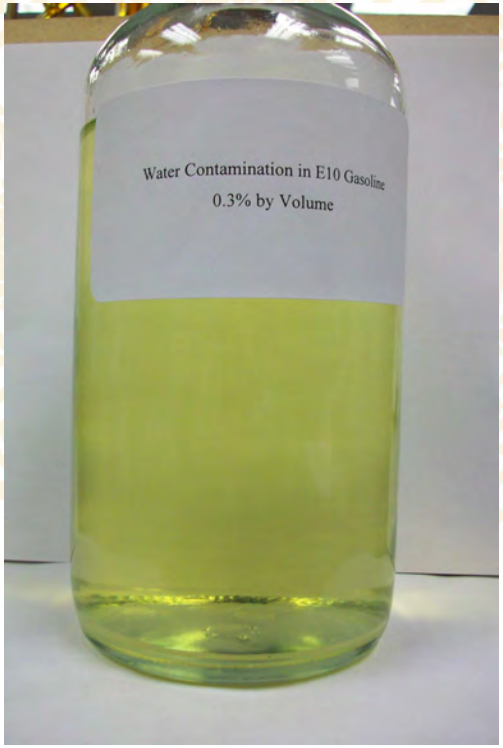
Sample remains hazy and visible phase layers are observed.

Phase Separation Project Results – Protocol 3

A portion was contaminated with **0.3%** DI water.



Immediately after mixing.

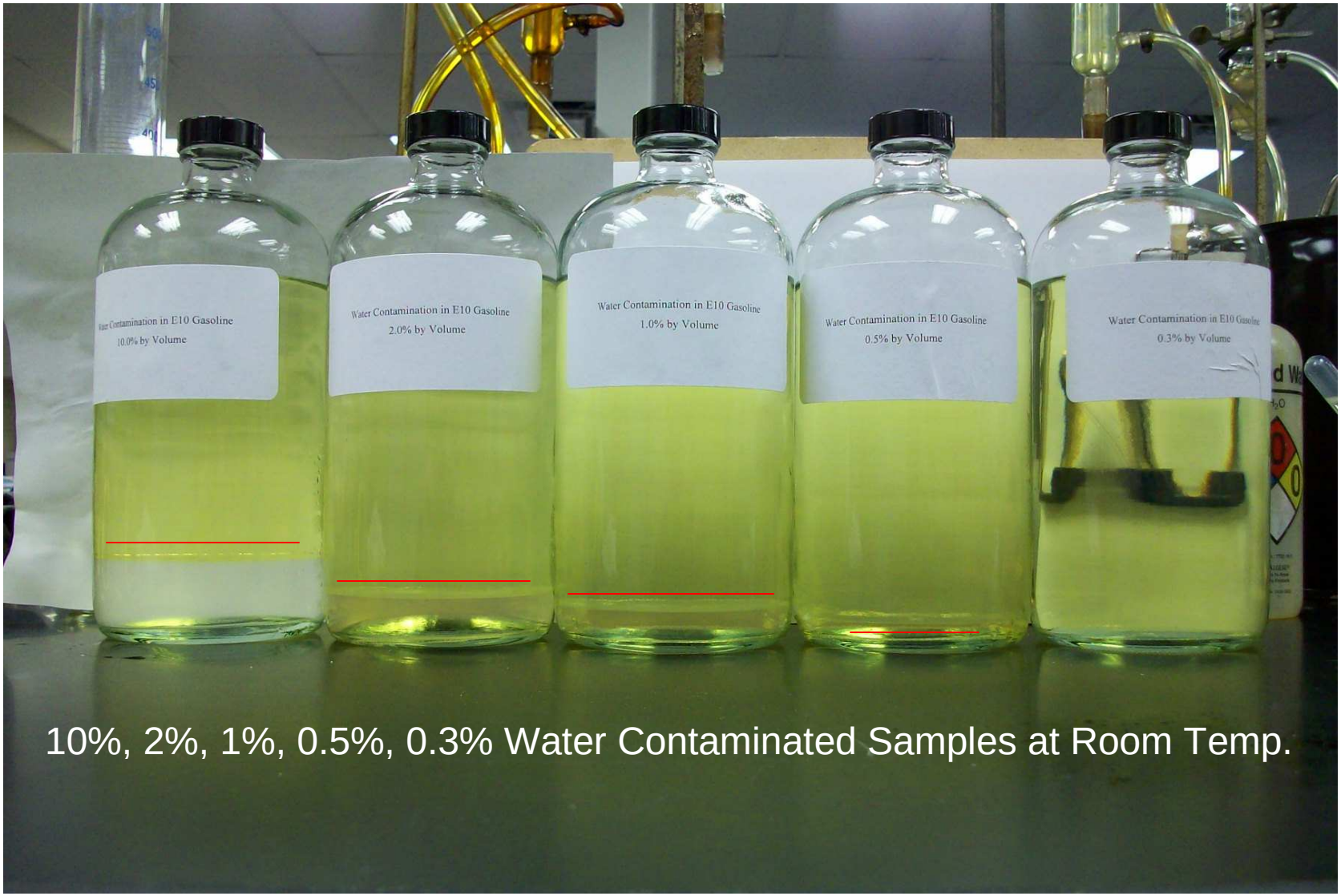


After shaking.

After resting.

Sample remains clear with no phase separation at 3000 ppm water.

Phase Separation Project Results – Protocol 3



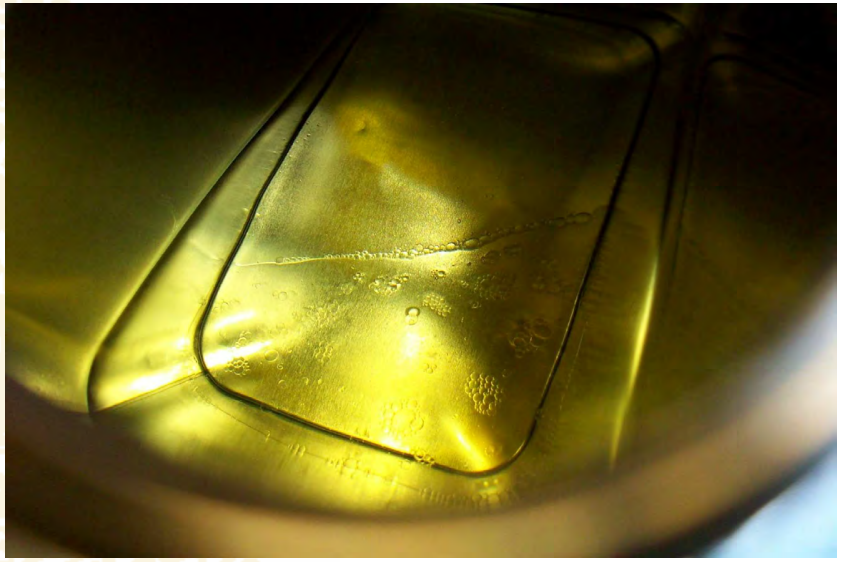
10%, 2%, 1%, 0.5%, 0.3% Water Contaminated Samples at Room Temp.

Remediation of Phase Separated Fuel

For a problem like this:

Check both ends of tank for moisture.

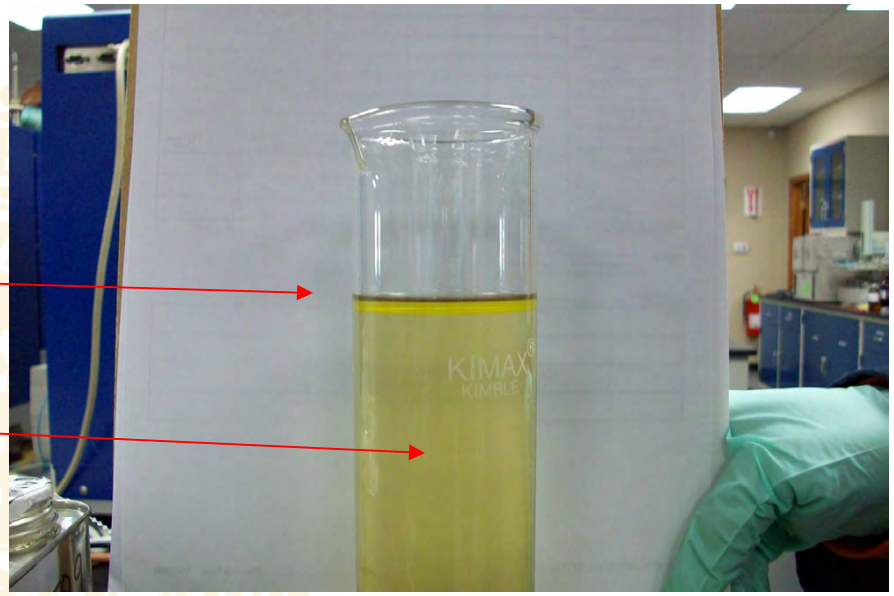
1. Completely remove any signs of water/phase separated material.
2. Water/EtOH Phase to be removed and disposed of.
3. Blend 1:1 Minimum with Fresh E10 Fuel – Retest for compliance before delivering for sale.
4. Inspect filters – replace as needed.



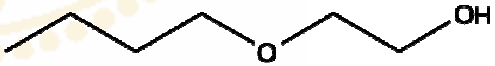
Remediation of Phase Separated Fuel

For a problem this severe:

1. Empty Tank and have all moisture removed.
2. Collect top portion of product.
3. Water/EtOH Phase must be removed and disposed of.
4. Determine the critical parameters of the gasoline phase – AKI, EtOH %.
5. Blend 1:1 Minimum with Fresh E10 Fuel – Retest for compliance before delivering for sale.
6. Replace all filters and purge lines.



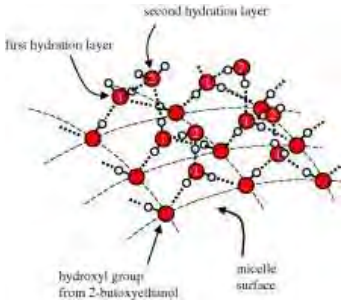
Additives To Correct: Be Cautious!



Some compounds introduced into gasoline-ethanol blends in an attempt to un-do the phase separation can be risky. High treat rates can cause the total mass % oxygen to exceed federal limits!

2-butoxyethanol is 27.08% oxygen (EtOH 34.74%)

Used as a solvent commercially



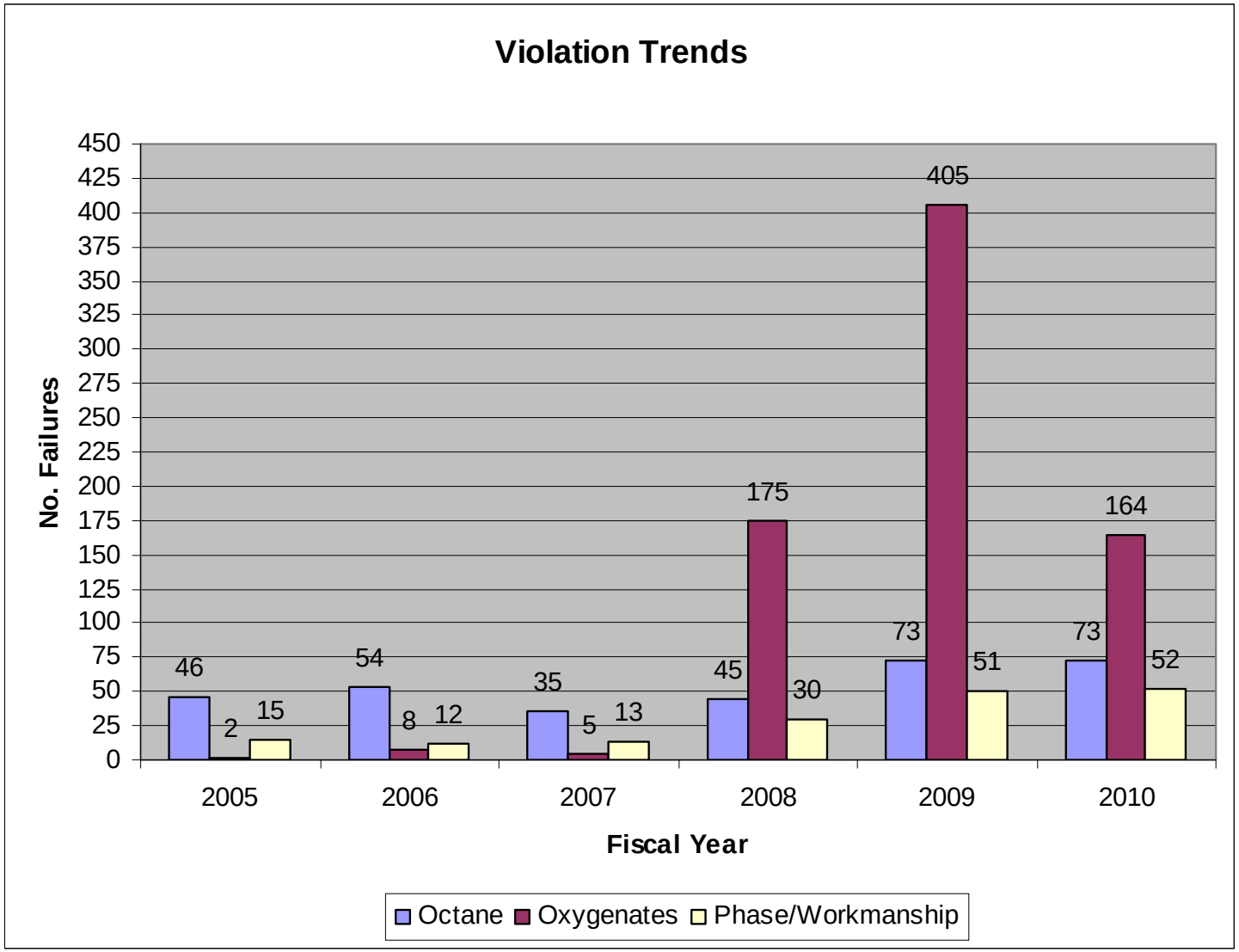
Phase Separation Maintenance Steps

- ✓ Select maximum 10 micron filters. We suggest using Alcohol Monitor filtration that will slow down the dispenser when catastrophic phase separation is occurring.
- ✓ Periodically have tanks swept to remove any accumulated moisture. TN Rules allow up to 0.25" in an ethanol tank; however, we strongly recommend drying tanks when any moisture is present. The presence of any water may be contributing to our problems!
- ✓ Be sure that you are using water finding paste that is designed for ethanol blends.
- ✓ Inspect fill and vapor caps for damage and missing gaskets, replacing if necessary. They must be water tight. Seals and latches must be in excellent condition.

Phase Separation Maintenance Steps

- ✓ Inspect tanks daily – always before and after fuel drop. If any water is present, have your employee contact QC officer and advise.
- ✓ Check vent pipes. They must have rain caps that allow ventilation yet prohibit rain water from entering the system. Do not overlook this item!
- ✓ Inspect product and spill containment buckets – if water is present, do not drain it into the tank but instead, remove and properly dispose of the water.
- ✓ Collect you own samples for visual inspection. Chill samples and look for any presence of water separation.

TENNESSEE DEPARTMENT OF AGRICULTURE
Regulatory Services Division

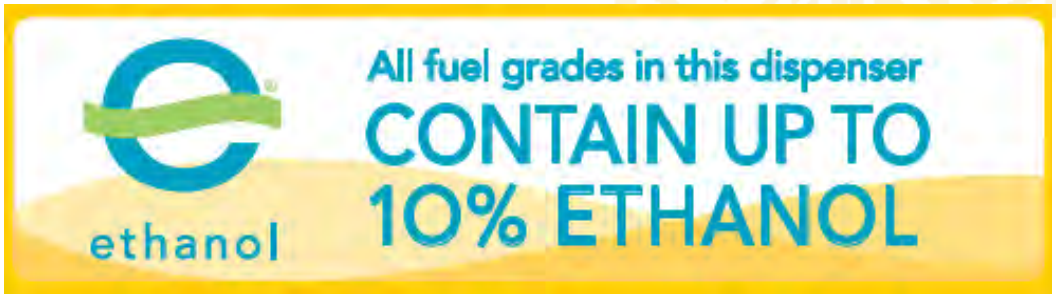


E10 and Lower Blend Requirements

Classification and Method of Sale Requirements

- ✓ Declare ethanol on PTD if greater than 1.5 Mass % Oxygen (~ 4 Vol.% EtOH)

- ✓ Label Dispensers if greater than 1.5 Mass % Oxygen (~ 4 Vol.% EtOH)
  - Label as "with" or "containing" (or similar wording) Ethanol. The label must be posted on the upper 50 percent of the dispenser front panel in a position clear and conspicuous from the driver's position in a type at least 12.7 mm (½ in) in height, 1.5 mm (1/16 in) stroke (width of type).



Product Storage Identification

"Fill Connection Labeling," the fill connection for any petroleum product storage tank or vessel supplying engine-fuel devices shall be permanently, plainly, and visibly marked as to the product contained by means of:

(a) A permanently attached tag or label and;
(b) American Petroleum Institute color codes as specified and published in "API Recommended Practice 1637".

NOTE: For locations storing both blended and unblended gasoline, according to the practice, use the appropriate gasoline symbol surrounded by a white circle for PU and MG, and a black circle for RU to indicate gasoline containing ethanol.

Questions?

Rules & More Information Can Be Found At:

http://state.tn.us/agriculture/regulatory/petroleum.html