



# **Tennessee Department of Agriculture**

## **Biodiesel Quality Program**

**Education, Communication, Cooperation, & Regulation**

**Presented by  
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Tennessee Department of Agriculture  
Regulatory Services  
February 6, 2008  
Orlando, FL**

## Session Outline

### Regulation:

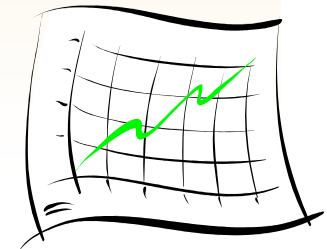
- History of Fuel Quality Regulations in Tennessee
- Biodiesel Requirements in Tennessee
  - Blend Stock
  - Biodiesel Blends
- Results of Inspections and Testing

### Education:

- Outreach – Governors Interagency Biofuels Working Group, 2007 Biofuels Governor's Conference, Biodiesel Workshops

### Communication & Cooperation

- Resolution Field Issue

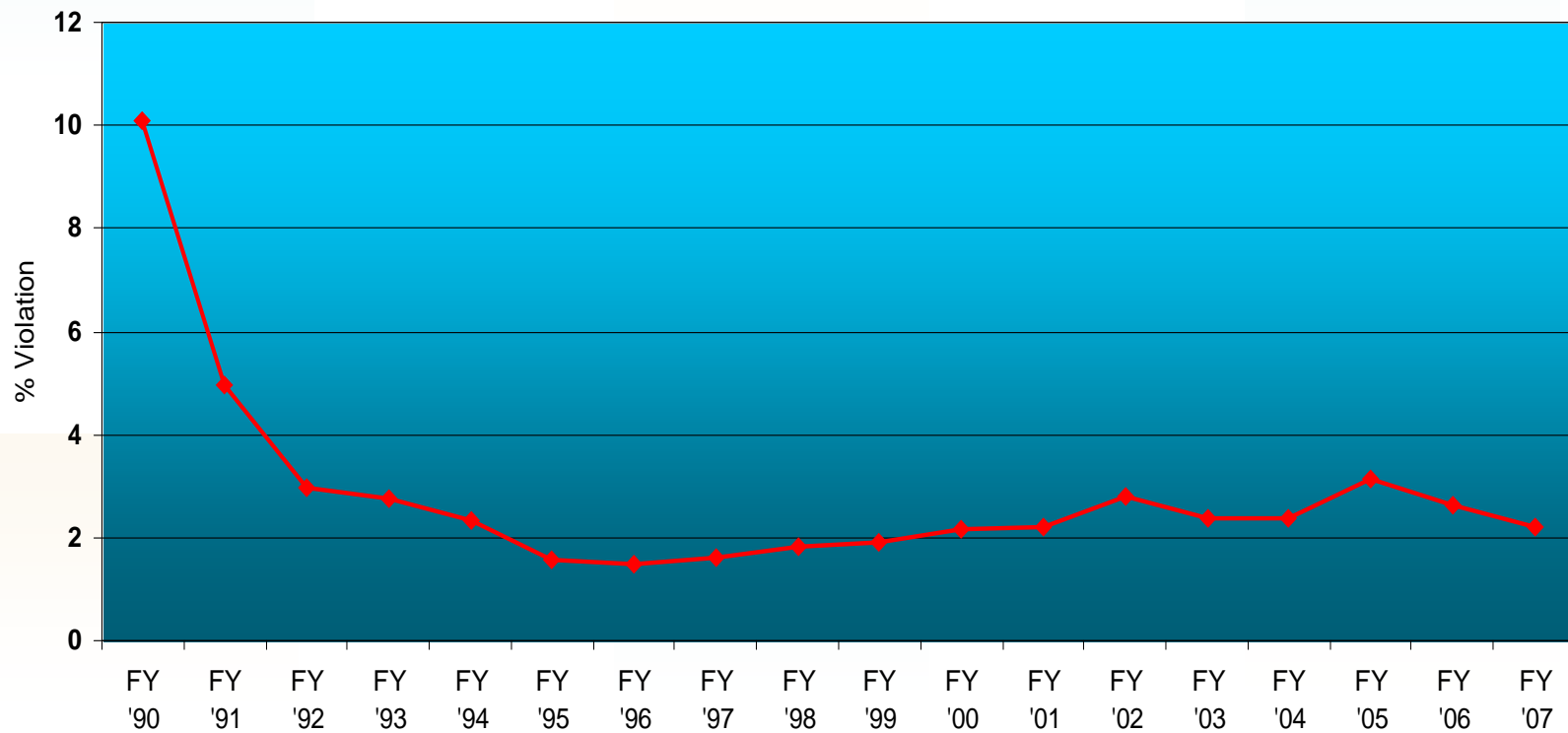


## History of Fuel Quality Regulations in Tennessee

- **Kerosene and Motor Fuels Quality Inspection Act of 1989  
T.C.A. 47-18-1301 et. seq.**
- **Charged the TN Dept. of Agriculture with the responsibility of  
regulating heating oils and engine fuels conveyed in Tennessee**
- **Regulations under TN Rules 0080-5-12**
- **Law sets ASTM standards as the basis for enforcement**
- **TCA 47-18-1304 (c) provides the Commissioner with authority to  
set alternative standards when in the best interest of the public**

## Improvement in Fuel Quality After Implementation of the KMFQI Act

Violation Rates  
All Regulated Products



## What Are The Requirements for Marketing Biodiesel and Biodiesel Blends in Tennessee?



## **Biodiesel Blend Stock Product Requirements in Tennessee**

- **Biodiesel Blend Stock must meet all specifications in the ASTM Standard Specification D 6751**
  - TN tests blend stock for complete specification conformance
- **Biodiesel Blend Stock must be at least 99% biodiesel (no more than 1% diesel fuel). Blend stock less than 99% biodiesel can only be used in commercial fuels if permission is granted by the Commissioner**



## **Biodiesel Blends Product Requirements in Tennessee**

- **Base diesel fuel must meet ASTM D975**
  - Sulfur, Aromatic, Lubricity exception provided that final blend meets specification
- **Biodiesel Blend Stock must meet ASTM D6751**
- **Biodiesel Blends up to 5% by volume must meet ASTM D975**
- **Biodiesel Blends more than 5% and up to 20%:**
  - ASTM D975 except that the maximum temperature of the 90% distillation point is set at 5 degrees C above the D975 specification. At such time that ASTM develops a standard for blends up to 20%, the ASTM specification will prevail as rule

## Biodiesel Blends Product Requirements in Tennessee

- **Low Temperature Operability**
  - All biodiesel blends must meet the 10<sup>th</sup> percentile minimum ambient temperature values for low temperature operability published in ASTM D 975 Appendix X.4, qualified by either D2500 (CP) or D4539 (LTFT)
- **Biodiesel Conveyed at Public Retail Sale Points**
  - Biodiesel conveyed at retail sale points that are available to the general consuming public shall not exceed 20% by volume

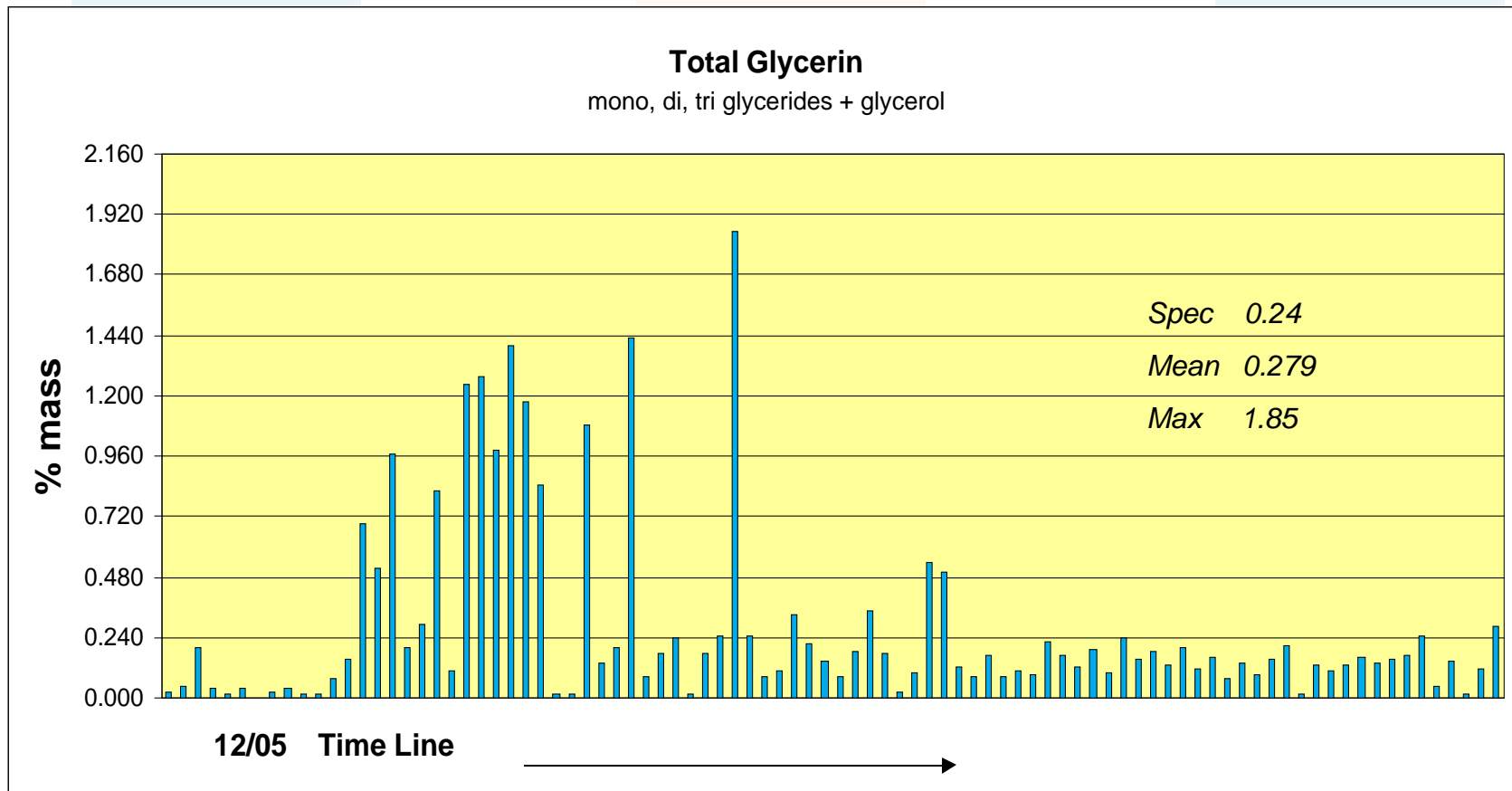


## What Have Investigations Revealed?

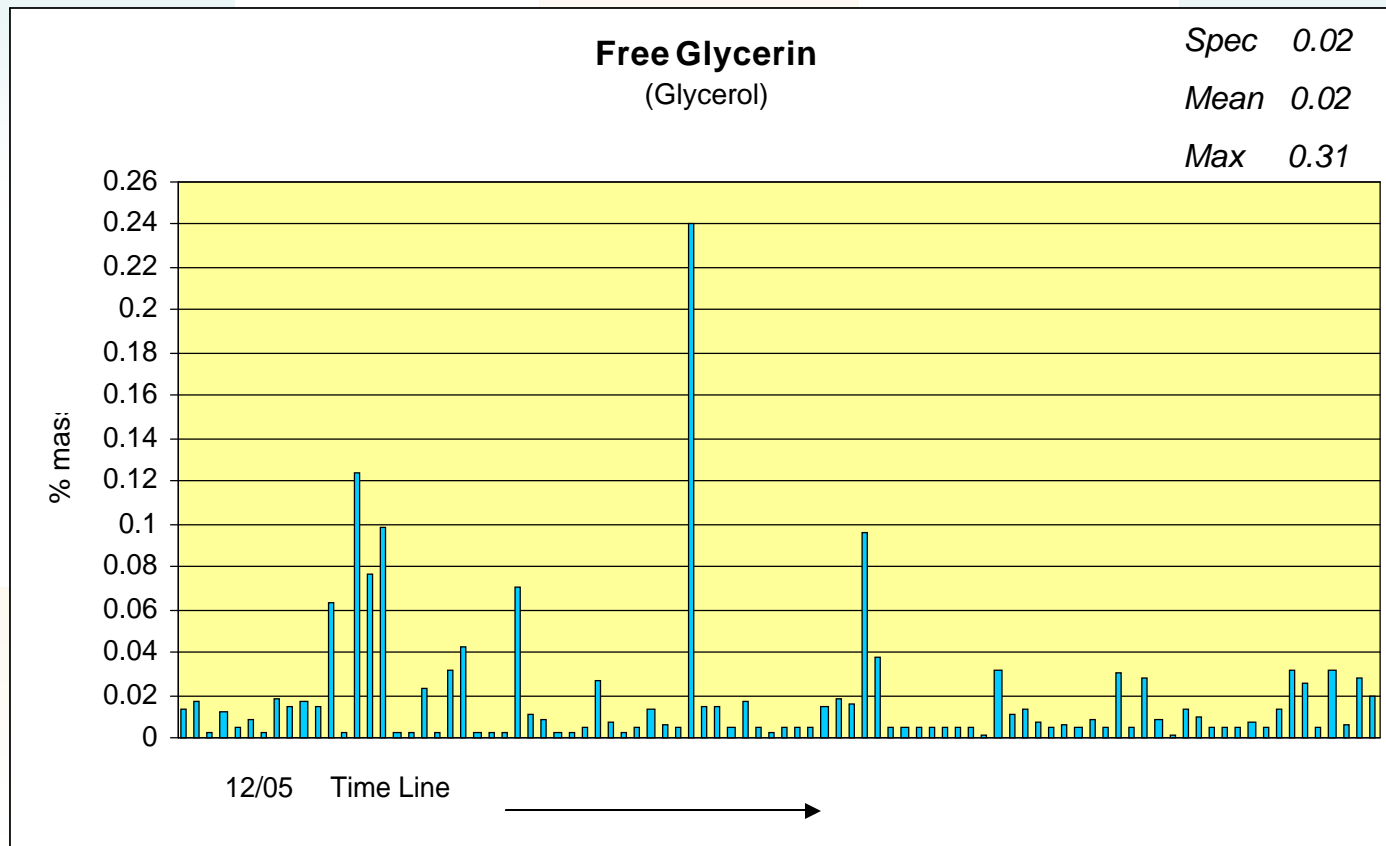
- Many samples found to be on spec – testimony of successful vehicle operability
- Continue to Find Borderline Fuels
- Failure of Biodiesel Blend Stock for multiple parameters of 6751
- Vehicle Failures / Fleet Issues
  - Improper blending may have aggravated some of the situations
  - Sterol Glucosides / Sitosterol Compounds



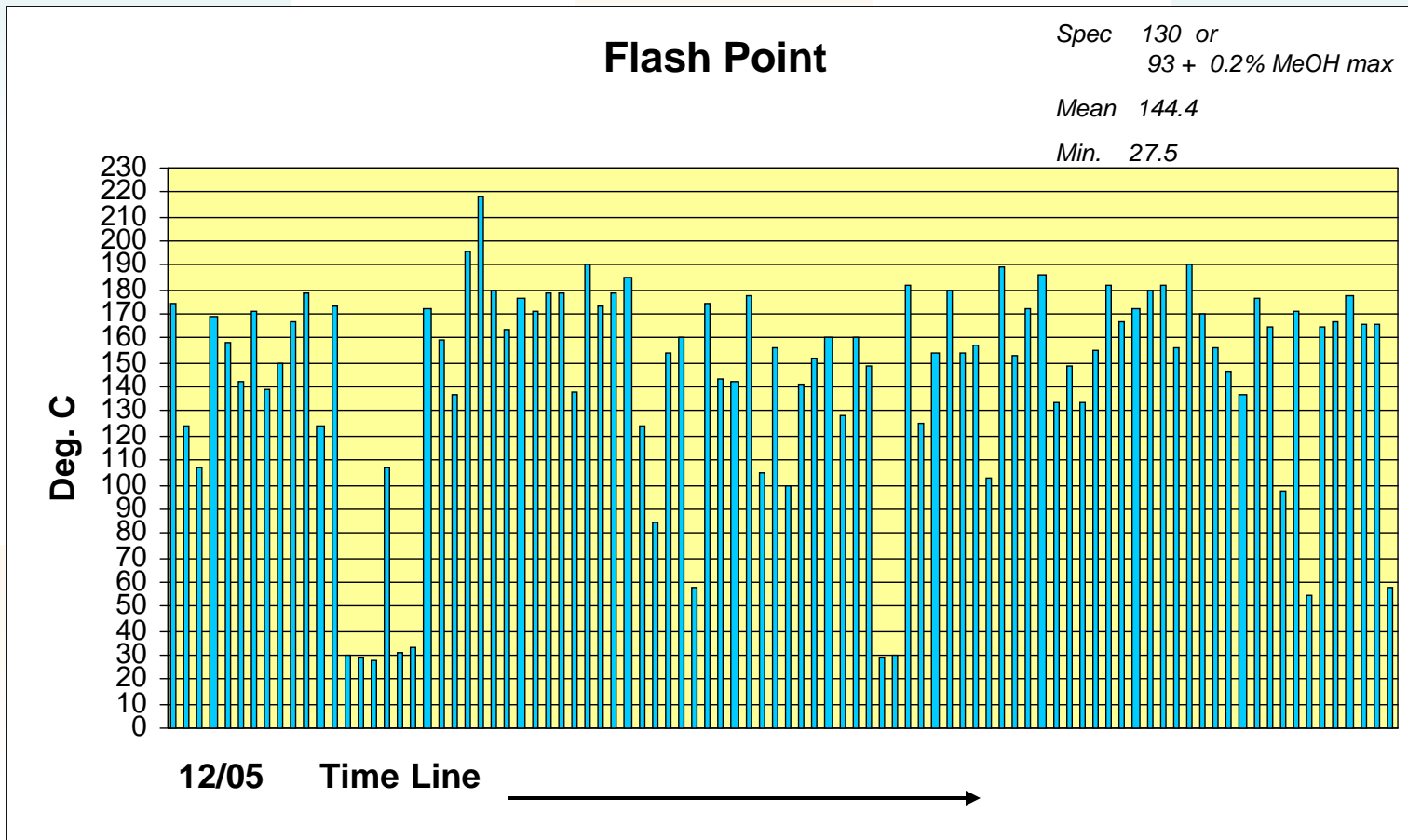
## Results of Biodiesel Blend Stock Testing



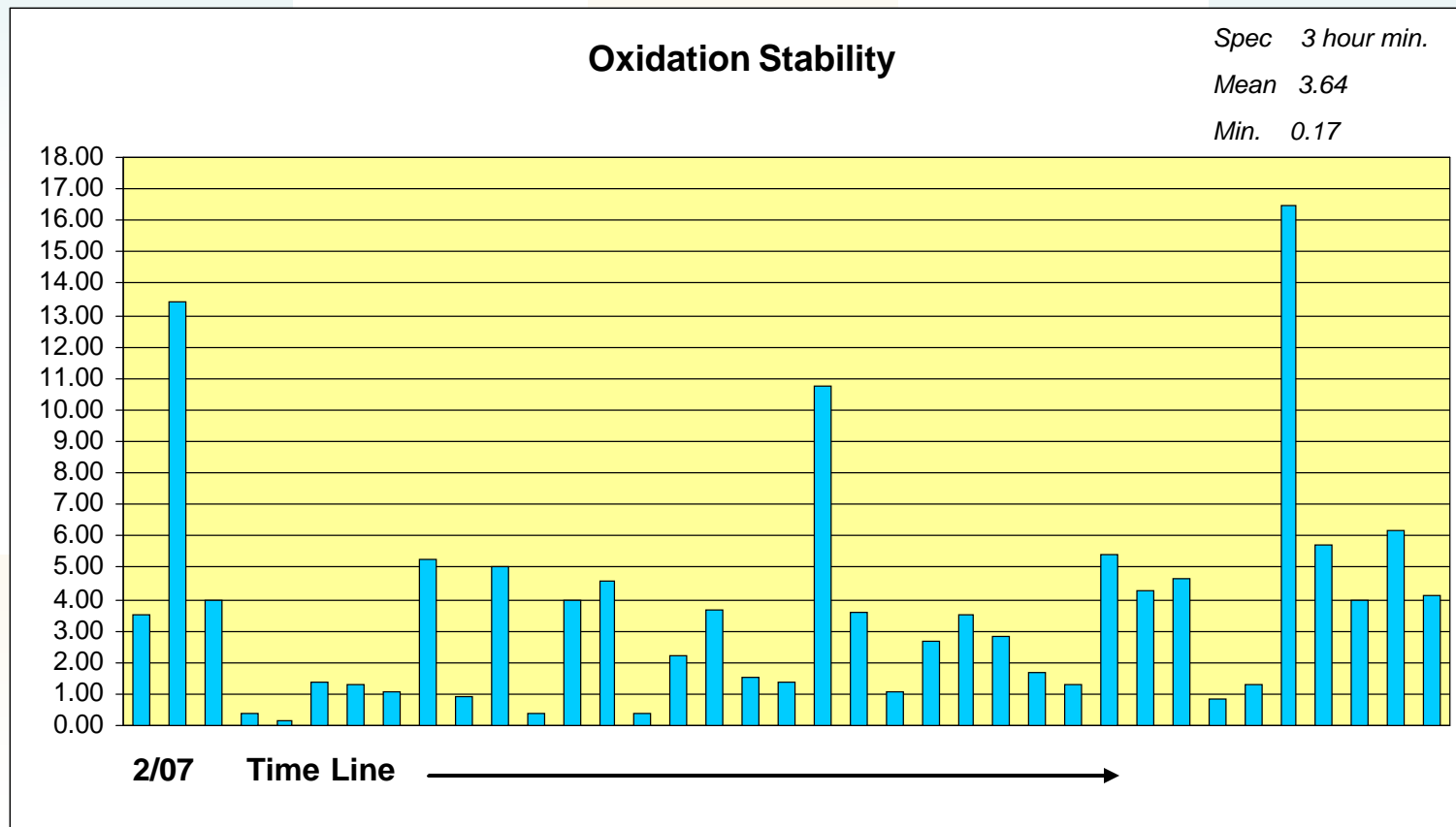
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## Education & Outreach

- **Governors Interagency Alternative Fuels Working Group**
  - BioTenn.org
- **Governor's Conference on Biofuels 2007**
- **Biodiesel Workshop 2007**
- **Collaboration with Clean Cities Organizations**

## **Governor's Interagency Alternative Fuels Working Group**

- **Governor Phil Bredesen signed Executive Order No. 33 on February 14, 2006**
- **Six Departments Charged to Develop State Alternative Energy Strategy**
  - Agriculture, Economic & Community Development, Environment & Conservation, General Services, Health, and Transportation
- **Began to Meet in March of 2006 to Address Directives**
- **Strategic Planning Meeting in July of 2007**
  - Report has been presented to Governors office – leans heavily on incentives, education, and quality

## Governor's 2007 Conference on Biofuels

- **May 30 – June 1 2007**
- **Offered Educational and Networking Opportunities to Stakeholders**
  - Farmers, Agriculture Executives, Entrepreneurs, Biofuel Producers, and Government Officials
- **Panel Discussions on Biofuel Initiatives, Next Generation Feedstocks, Production Trends, & Quality Assurance**



## Statewide Biodiesel Workshop

- **August 15, 2007 at Ellington Agriculture Center Nashville**
  - Sponsored by the Tennessee Oil Marketers Association, National Biodiesel Board, and the Tennessee Soybean Promotion Council
- **Presentation Topics Covered All Aspects of Biodiesel Quality**
  - Biodiesel Overview, Regulations, Ensuring Fuel Quality, Economics and Marketing Biofuels, Additives and Biodiesel, Transportation & Blending, UST Requirements
  - Speakers: Richard Nelson (NBB), Parks Wells (TN Soybean Promotion Board), John Chandler (Infineum USA), Dave Blatnik (Marathon Petroleum)

## Communication & Cooperation

- **Working with Tennessee Clean Cities Organizations**  
Area Workshops, Newsletters, ...
- **Working with Biodiesel Blenders and Marketers**  
Consultation on Laws and Rules, Explain Quality Requirements, ...
- **Working with Biodiesel Producers**  
Quarterly Sampling and Testing, Problem Resolution, ...
- **Working with Biodiesel Fleets**  
Cold Flow Testing & Consultation, Fuel Analysis, ...

## Example of Field Problem Resolution

- **Late 2007 Contacted by a Biodiesel Marketer Regarding Filter Plugging in Customer Fleet**
- **Contacted Fleet Operator For Description of Problem**
  - Verified Fuel Systems in Problem Vehicles
  - Verified when Problem Began
  - Collected Plugged Filters and Samples of the Biodiesel Blends
- **Contacted Biodiesel Marketer**
  - Collected Sample of B100 Currently In Stock

## Example of Field Problem Resolution

- **The Fleet Operator Confirmed That:**
  - All Vehicles Affected Had 2002 and 2003 High Pressure Fuel Systems  
Older Models Without High Pressure Systems Had No Issues
  - Fleet had been operating on ~B10 since May 2007 w/o Issue
  - Within 2-3 Week period, 15 Vehicle Failures
  - No Change in Filter Brands, No Additional Additives Being Used



## Example of Field Problem Resolution

- **TDA Used Network of Experts For Guidance**
  - Several Labs Offered Their Assistance In Analyzing Both the Fuels and the Filters
  - Several Experts Offered Conjectures on Possible Causes
- **Samples of the Fuels and Filters Were Shipped For Analysis**
  - Certain Labs Analyzed Fuel, Others Filter Residue



<b>Lab 1</b>	<b>Result</b>	<b>Spec , Max</b>	<b>R of Method</b>	<b>r of Method</b>
<b>Total Glycerin, Mass %</b>	0.167	0.24	0.095	0.037
<b>Free Glycerin, Mass %</b>	0.031	0.02	0.020	0.004
<b>Lab 2.</b>	<b>Result</b>	<b>Spec , Max</b>	<b>R of Method</b>	<b>r of Method</b>
<b>Total Glycerin, Mass %</b>	0.135	0.24	0.079	0.035
<b>Free Glycerin, Mass %</b>	0.025	0.02	0.018	0.004
<b>Acid No., mg KOH/g</b>	0.33	0.5	0.188	0.059
<b>Cold Soak Filtration, s</b>	>1200	N/A	N/A	N/A
<b>Water Content, ppm</b>	70	500	216	19.42
<b>Phosphorous, ppm</b>	3	10	N/A	N/A
<b>Ca + Mg, ppm</b>	7	5	2.229	0.4334
<b>Na + K, ppm</b>	1	5	1.13	.213
<b>Lab 3</b>	<b>Result</b>	<b>Spec , Max</b>	<b>R of Method</b>	<b>r of Method</b>
<b>Total Glycerin, Mass %</b>	0.140	0.24	0.081	0.035
<b>Free Glycerin, Mass %</b>	0.027	0.02	0.019	0.004
<b>Cold Soak Filtration, s</b>	>900	N/A	N/A	N/A
<b>Phosphorous, ppm</b>	<10	10	N/A	N/A
<b>Ca + Mg, ppm</b>	6	5	2.08	0.4102
<b>Na + K, ppm</b>	<2	5	N/A	N/A

## Filter Analysis Revealed:

### Laboratory 2 Reported That:

- A sample was scrapped off the fuel filter material. It was black in color and more fluid than [other filter samples being analyzed in the lab].
- The sample had a **large amount of free glycerin present**. Also present was **sterol glucosides**, and a small amount of C18:2 monoglyceride. The unsaturated monoglycerides are not usually associated with filter plugging problems.



## Filter Analysis Revealed:

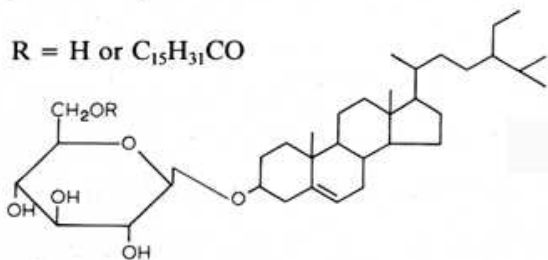
### Laboratory 4 Reported That:

From the identification of the components in the sludge, the following compounds are believed to be involved in the formation of the black sludge observed:

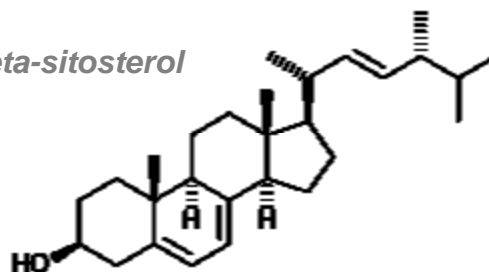
Water, Hydrotreated diesel, Biodiesel, Biodiesel by-products of glycerin, sitosterols, and/or sitosterol glucosides, Metals, Cellulosic filter media from vehicle or dispenser

*β-sitosterol glucosides*

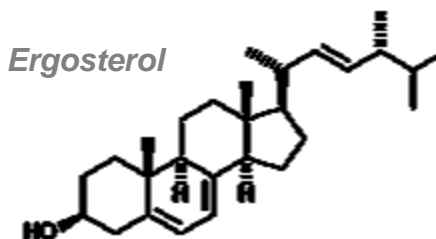
R = H or C<sub>15</sub>H<sub>31</sub>CO



*Beta-sitosterol*



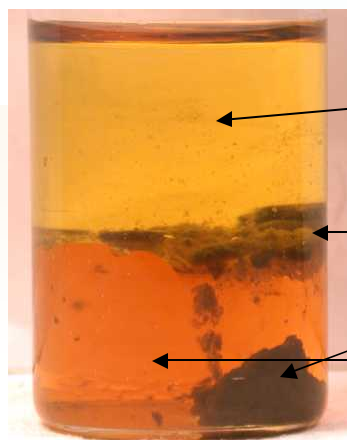
*Ergosterol*



## Filter Analysis Revealed:

### Laboratory 4 Reported That:

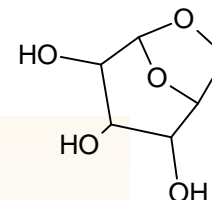
Black sludge reformed in a mixture of the filtered hydrocarbon and methanol. The solids from this mixture were also analyzed by Thermal Extraction and Pyrolysis GC-MS techniques. Thermal Extraction shows a trace of water, a small amount of glycerin and diesel range hydrocarbon, some C18-acid methyl esters, and **mostly sitosterol compounds**. Pyrolysis GC-MS shows additional **sitosterols, levoglucosan, and decomposition of sitosterol compounds**.



Top Phase: Diesel/Biodiesel Solution

Reformed Solids – Mainly Sitosterol Compounds

Bottom Phase: Methanol/Water/Glycerin



*Levoglucosan*

Levoglucosan is a cellulosic material that can be produced from carbohydrates, which were also identified in the filter residue

## Conclusions

- **Problems Have Been Resolved With B100**
- **Additional Quality Controls Are in Place**
- **All Parties Involved Agree that the Cooperation & Communication Approach was Effective in Solving the Problem**

# Questions?

