




**Diethyl Sulfosuccinate  
Chemical Behavior and Liquid  
Chromatography Tandem Mass  
Spectrometry Analysis**

**Oil Spill Response Research &  
Development Forum  
January 10, 2011**

A satellite image of the Gulf of Mexico coastline. The land is green and brown, with a large area of white and light brown material (oil spill) extending from the shore into the dark blue water. The text "The greatest environmental disaster of its kind in our history," Obama '10 is overlaid on the image.

**“The greatest environmental disaster of its kind in our history,” Obama '10**

# Monitoring the Impact

- Alkyl and Polyaromatic Hydrocarbons (PAHs)
- Oil and Grease Analysis
- Metals
  - Vanadium and Nickel
- Air Monitoring
  - Particulate matter,
  - SO<sub>2</sub>
  - H<sub>2</sub>S
  - Xylenes
  - Volatile and Semi-Volatile Hydrocarbons
  - Gasoline and Diesel Range Organics



Gerald Herbert, AP

# Dispersants

- Applied to mitigate the impact of floating oil on sensitive shoreline habitats
- 6.977 million liters applied NOAA Oil budget
  - Surface and subsurface
- Corexit 9500A and 9527A



# Dioctyl Sulfosuccinate

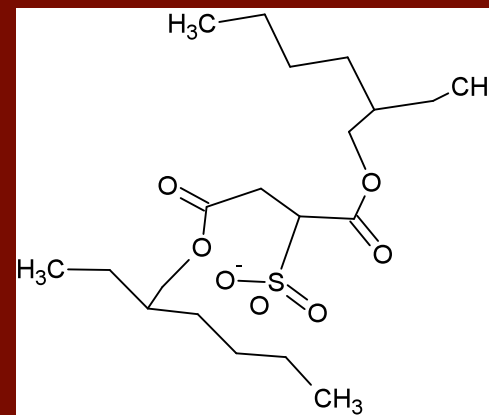
IUPAC: 1,4-bis(2-ethylhexoxy)-1,4-dioxobutane-2-sulfonic acid

CAS # 577-11-7

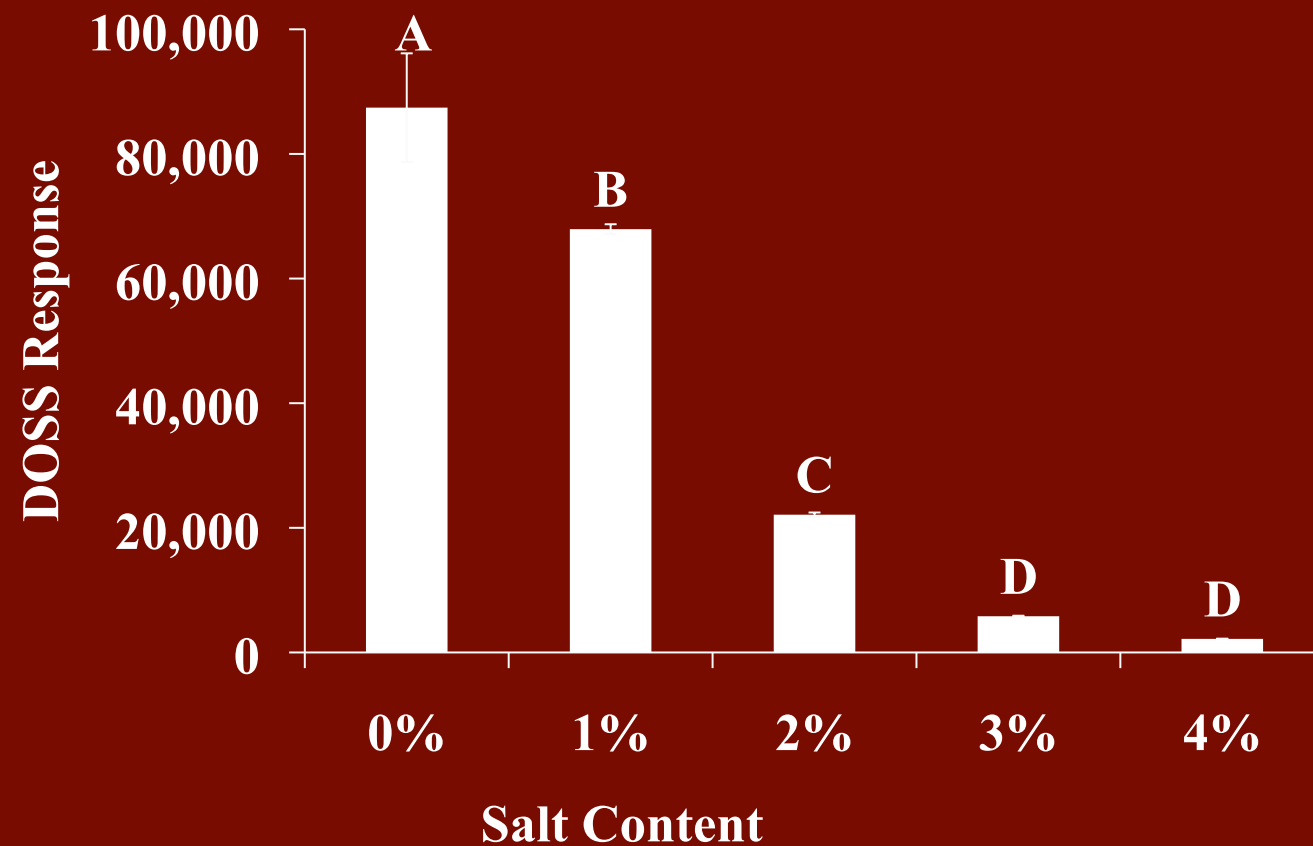
Many uses:

Laxatives, food additive, ....

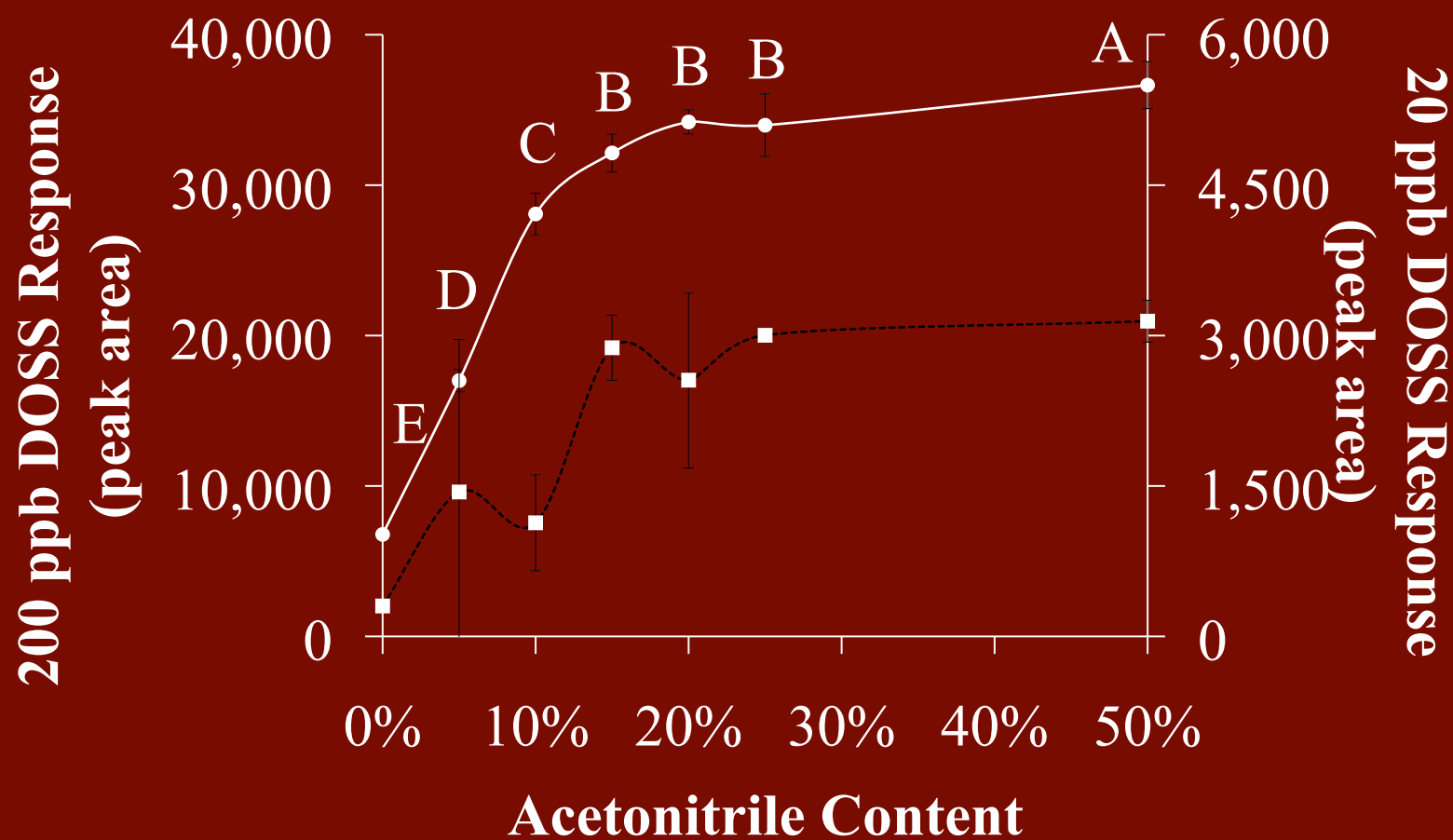
No method for trace analysis in sea water



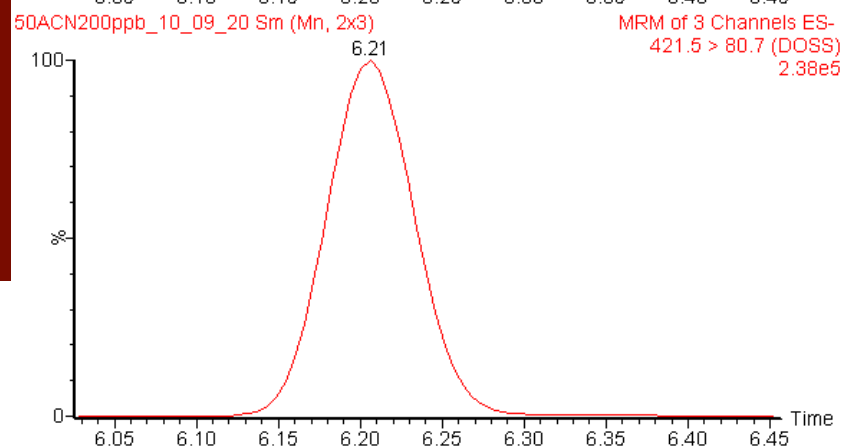
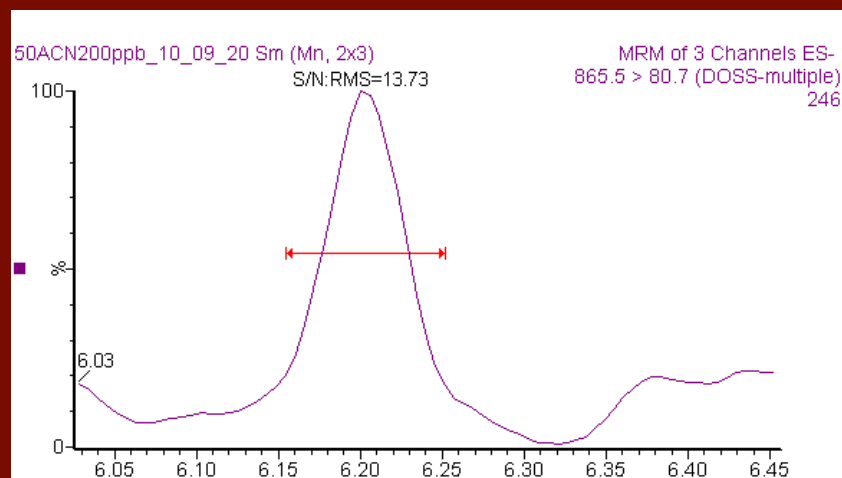
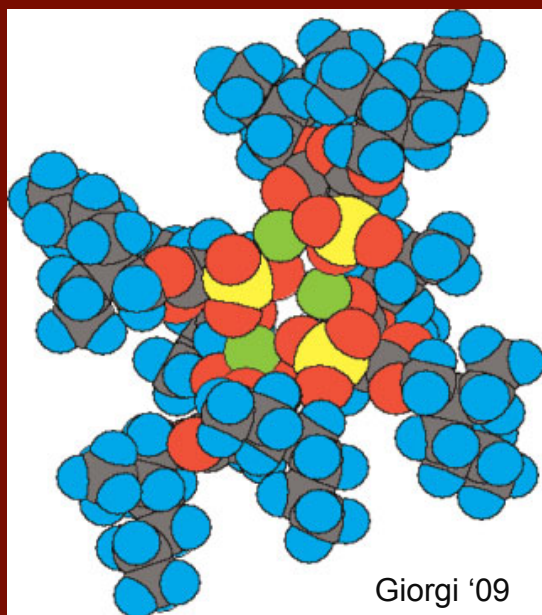
# DOSS / sodium chloride



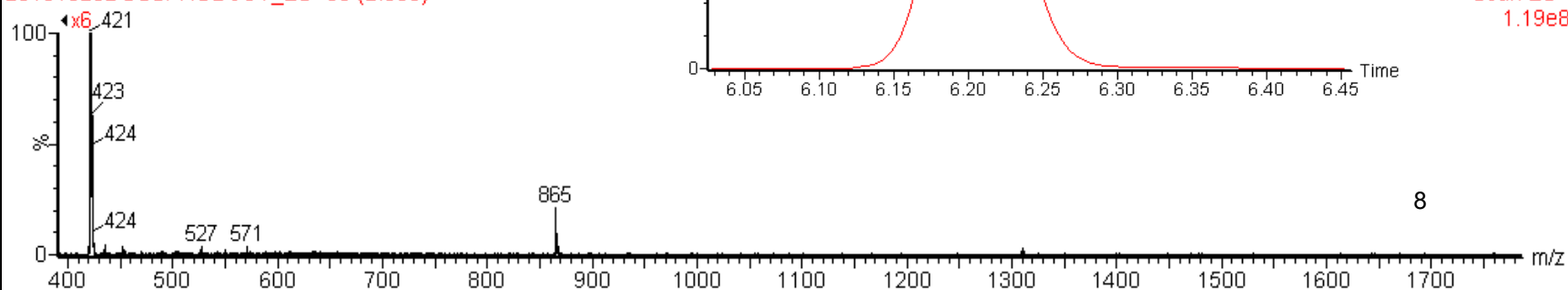
# ACN Addition



# Supramolecular Structure

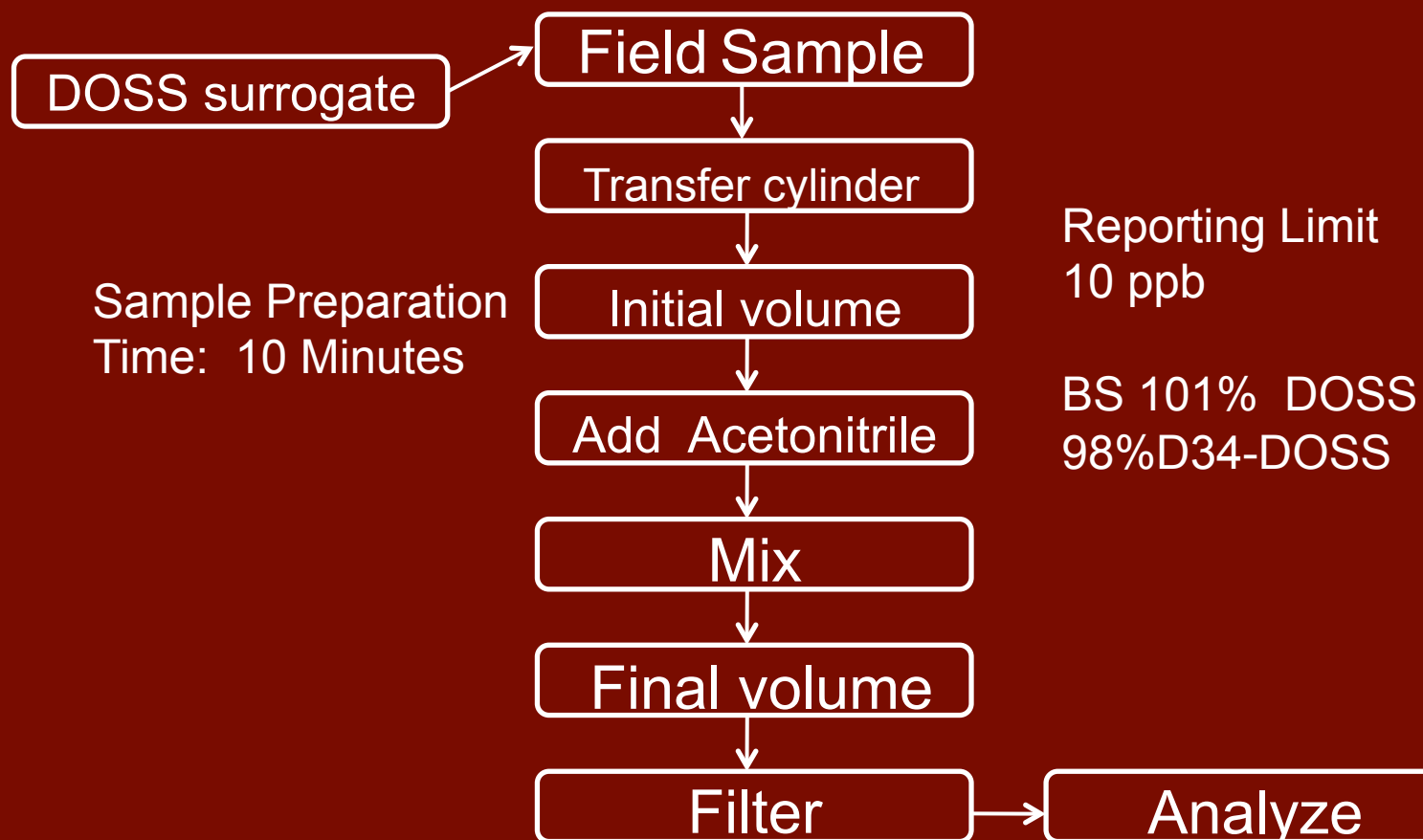


20101020DOSSPRODUCT\_ES- 30 (2.005)



# Regional Analytical Method (RAM)

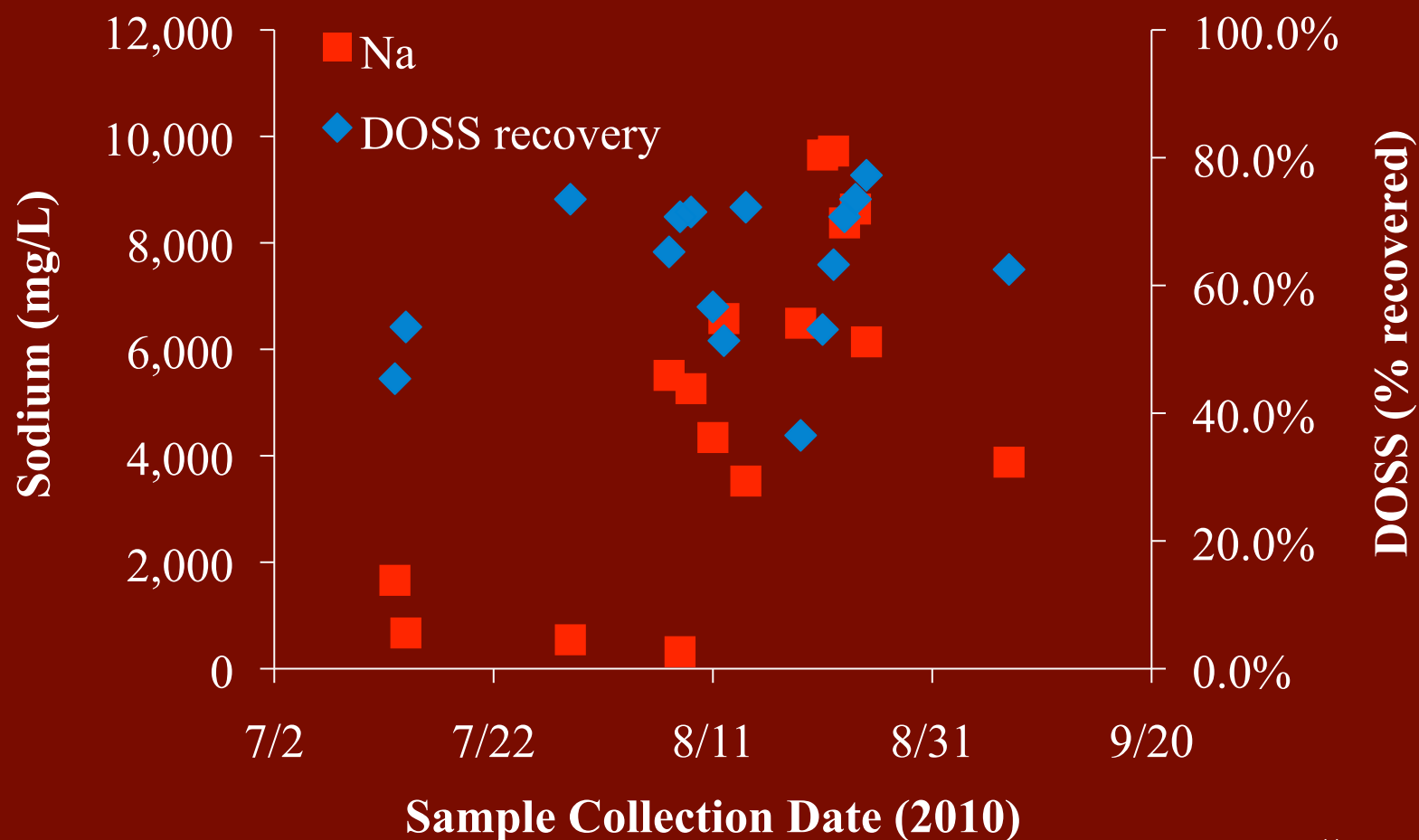
## RAM-DOSS



# Seawater DOSS Spike

- Spiked Seawater with reference oil
  - Aqueous only – 10 % of full sample spike
  - Full sample - 97% recovered
- Spiked Seawater with source oil -
  - 4.5 ppm DOSS
  - D34-DOSS - 97% recovered
- Tubing
  - Polypropylene - 26% DOSS spike collected
  - DOSS bound to surface collected with ACN rinse

# Matrix Spike Recovery



# Deuterated Surrogate

- US EPA near-shore samples
  - Analyzed prior to  $^{13}\text{C}$  labeled surrogate was commercially available
- Deuterated surrogate –DOSS- $\text{D}_{34}$ 
  - Matrix spike/surrogate recoveries varied with LC modifier
    - ammonium formate  $R^2=0.95$
    - formic acid  $R^2=0.27$ 
      - Low for reagent and matrix spikes
      - Retention time difference increased

# Conclusions

- Addition of 50% acetonitrile reduced DOSS surface binding
- DOSS recovery strongly correlated with D34-DOSS
- Analyzed ~ 600 Gulf of Mexico samples
  - None  $\geq 20$  ppb

# ***Questions***

***David Schroeder , Ph.D.  
US EPA, Region 5  
davidschroeder@gmail.com***