

# *Environmental Monitoring and Sampling*



*U.S. Environmental Protection Agency. August, 2022*

# Acknowledgements:



Multiple slides and information also provided by:

U.S. Department of the Interior  
U.S. Fish and Wildlife Service (US FWS)  
National Conservation Training Center



U.S. Department of Commerce  
National Oceanic and Atmospheric  
Administration (NOAA)





# Topics Covered:

- Inland Oil Spill Environmental Sampling Overview
- Sampling and Analyses Plans
- Sample setting, media and constituents
- Ephemeral data collection
- Civil and Criminal sampling for enforcement.

# Sampling and Emergency Response



## First Step, Identify Immediate Monitoring Needs

- A sampling and Analyses plan (SAP) needs to be developed.
  - What are the Data Quality Objectives ?
  - Why am I sampling?
  - When will I stop?
  - What are we going to do with samples?
  - What Parameters should we analyze for ?
  - What media (water, sediments, soils) are to be sampled ?
- Once a Monitoring Plan is created and approved by Unified Command, who is implementing what parts of the plan?

# Immediate Sampling and Monitoring Needs



- Public Health and Safety Questions
  - Rapid turn-around results needed from the lab
- Responder Decision Making
  - Real-time field-data needs
- Goal: Documenting ephemeral data.
  - For the response phase.
  - For long term restoration and enforcement.





# Ephemeral Data

- Ephemeral data are types of information that change rapidly over time and may be lost if not collected immediately (e.g., within hours, days or weeks).





# Urgent Sampling Efforts from a Spill

Goal: Be able to develop a sampling plan that addresses the needs of ephemeral data collection during the Response Phase and for the Restoration\*Phase.

## Immediate Sampling Needs

- A common approach to sampling is to immediately gather upstream, spill area, and downstream water quality samples.
- Act on what is reasonable and necessary to meet the objectives.

*\* In the USA, Natural Resource Damage Assessment and Restoration (NRDAR) must establish environmental harm. Sampling is an important part of this process.*

# Environmental Sampling and Monitoring for Intermediate and Long-Term Data Needs



## Long Term Monitoring:

- Crude Oil contains persistent pollutants. A long-term plan needs to meet the expectation of Federal, State and Local regulatory authorities,
- Long term vegetative and habitat monitoring may also be necessary to evaluate harm and mitigation strategies, often overseen by natural resource trustees.



# Sampling and Analyses Plans (SAP)



A basic SAP should have many of these components:

- A description and map of the area to be studied.
- Sampling Design and Rationale
- Data Quality Objectives.
- Analytical Requirements, contaminants of concern
- Scope of Work, Sampling Team(s)
- Proposed Tasks and Schedule
- Geological/Limnological/hydrological Information



# Sampling and Analyses Plans (SAP) cont.

- Field Methods, Sample collection, Handling procedures  
*(specific for each media – soil-sediment-water)*
- Equipment Calibration, Maintenance, Testing, Inspection
- Quality Assurance/Quality Control Processes
- Field Health and Safety Information
- Data Review and Validation Process
- Decontamination and disposal of sampling derived wastes

# Data Quality Objectives (DQOs) cont.



DQOs Concisely describe the problem to be investigated.

- Identify what questions the investigation will attempt to resolve, what actions (decisions) may result, and who the primary decision maker is.
- Identify the information that needs to be obtained and the measurements that need to be taken to resolve the decision statement(s).
- Define study boundaries and when and where data should be collected.



# Typical Methods of Sampling that go into a SAP.

- Sample Design:
  - Judgemental (biased/targeted) Sampling,
  - Simple Random Sampling,
  - Systematic and stratified & cluster sampling
  - Manual sampling, Automatic Sampling, sorbent sampling
- Sample techniques: Grab, homogenization, filtration

# Before you Sample



- Do your best to review relevant standards, permit conditions, or applicable rules to document violations.



# Environmental Media



Have targeted sampling plans for various media/settings.

- Source oil (fingerprinting)
- Stranded oil on shorelines
- Oil sheens
- Water (surface and water column)
- Intertidal and seasonally inundated sediments
- Subtidal sediments
- Terrestrial soils and ground water



# U.S. NOAA had developed Sampling Guidelines to aid in SAP development.

- Sampling guidelines are general descriptions that can be used to facilitate ephemeral data collection.
- A sampling plan should provide additional details on “what, when, and how” samples and data will be collected.
- *Note: a field sampling plan is different from guidelines in terms of the level of detail and specificity. (e.g., use pre-cleaned stainless steel spoons and glass jars to collect samples).*

# NOAA Guidelines for Habitats and Associated Communities



- Sand beach infauna
- Gravel beach communities
- Rocky intertidal communities
- Ice
- Vegetated habitat (marsh, low lands, tundra)
- Eelgrass
- Exposed organisms/Shellfish tissues
- Fish (ichthyoplankton, juveniles and adults)

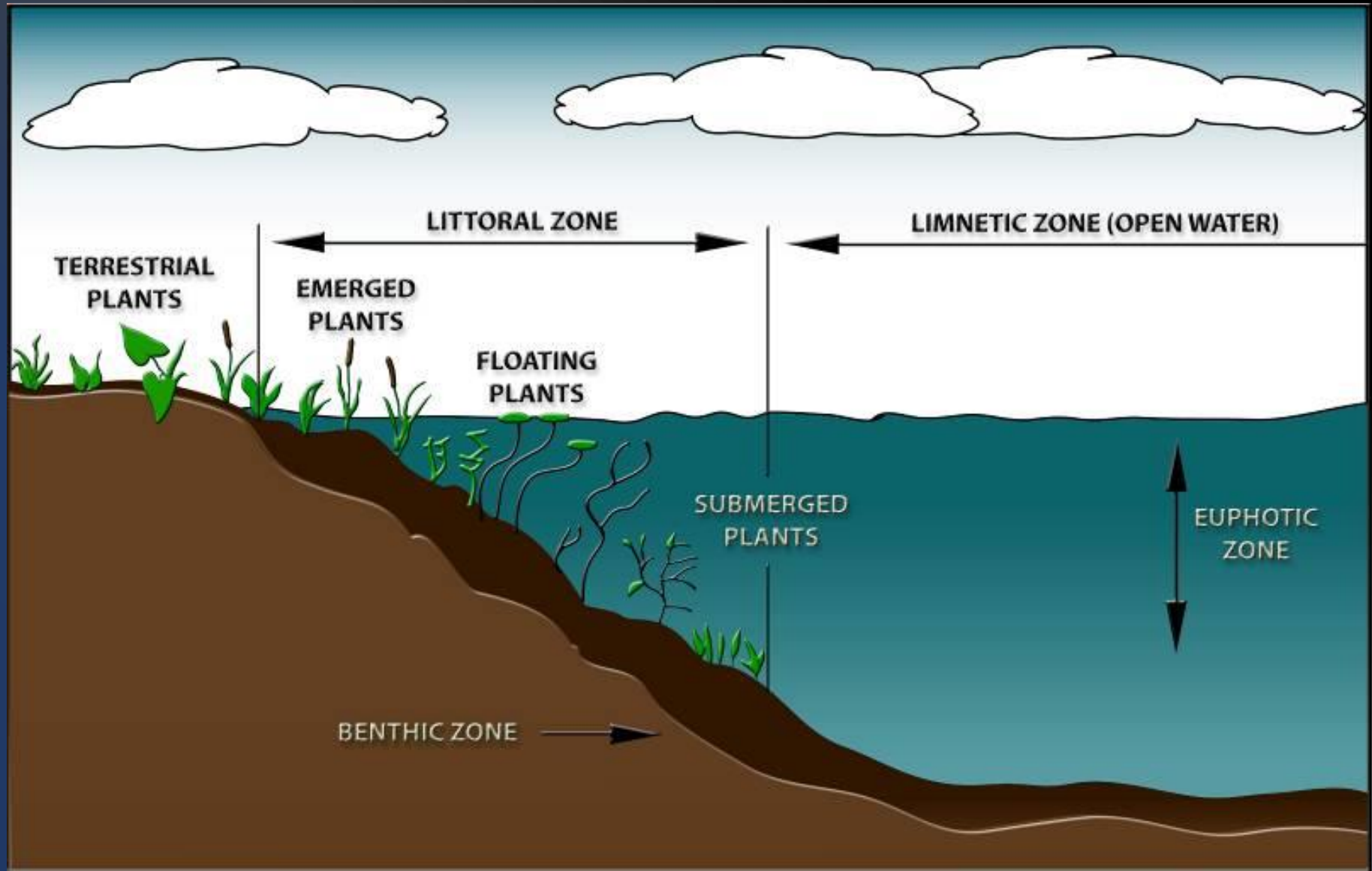


# Contaminants of Concern for Crude Oil

**Crude oil contains many components which behave differently in the environment.**

- The volatile compounds will evaporate soon after the spill.
- Many components will not readily degrade,
- Salts and dissolvable constituents will further cause movement of contaminants into the subsurface.
- Resins & waxes, heavy paraffins, asphaltenes, and lubricating oil components will also make shallow soils water repellant.

# The Importance of Habitat Zones



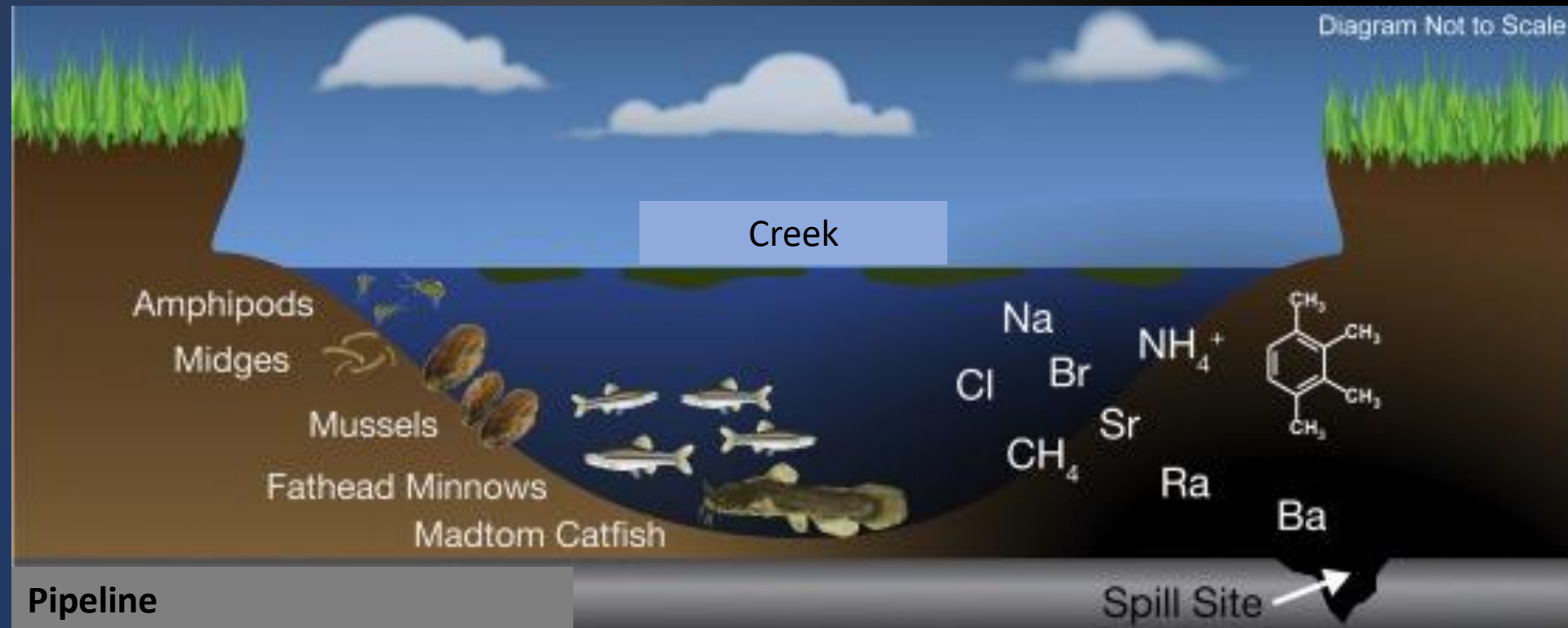


# Still Freshwater Ecosystems: Lakes, Ponds



- Lakes and ponds: **much of the species' diversity is concentrated in the nearshore littoral zone.**
  - Algae and plants thrive in light needed for photosynthesis.
  - Animals include snails, amphibians, crustaceans, insects, and fish.
- Beyond the littoral zone is the **limnetic zone**: zone of open water where light is still able to penetrate (i.e., photic zone).
  - Consumers in this zone include zooplankton, which feed on the algae, some insects, and fish.
- **Benthic zone** is the bottom sediment .
  - This zone dominated by invertebrate species and bottom dwelling fish that feed on them.

If you live there, oil pipelines have more than just Oil.



# Crude Oil Toxicity



**Many Components are:**

- **Poisonous (have established LC50/LD50),**  
Respiratory monitoring and protection needed for all responders.

**Benzene** is a driving risk criteria. Benzene is carcinogenic, persistent, and often field screenings don't correlate with analytical results.



# Crude Oil Toxicity

- **Confirmed Human and Animal Carcinogens,**  
(for example: Seven PAHs are known to cause cancer in animals.

For impacted aquatic sediments, the cleanup criteria is specifically intended to be protective of benthic invertebrate communities.

**Safety Data Sheets for Crude oil often list known Aquatic Toxicity (LC50-EC50).**

Fish kills may be evident in surface waters.



# Unknown or Known Source:



- Provide proof of toxicity
- Run Non-traditional parameters (ex.  $LC_{50}$ )
- If unknown; Run full scans (ex: vocs, svocs)

## $LC_{50}$

- Concentration in the water lethal to 50% of test population

## $EC_{50}$

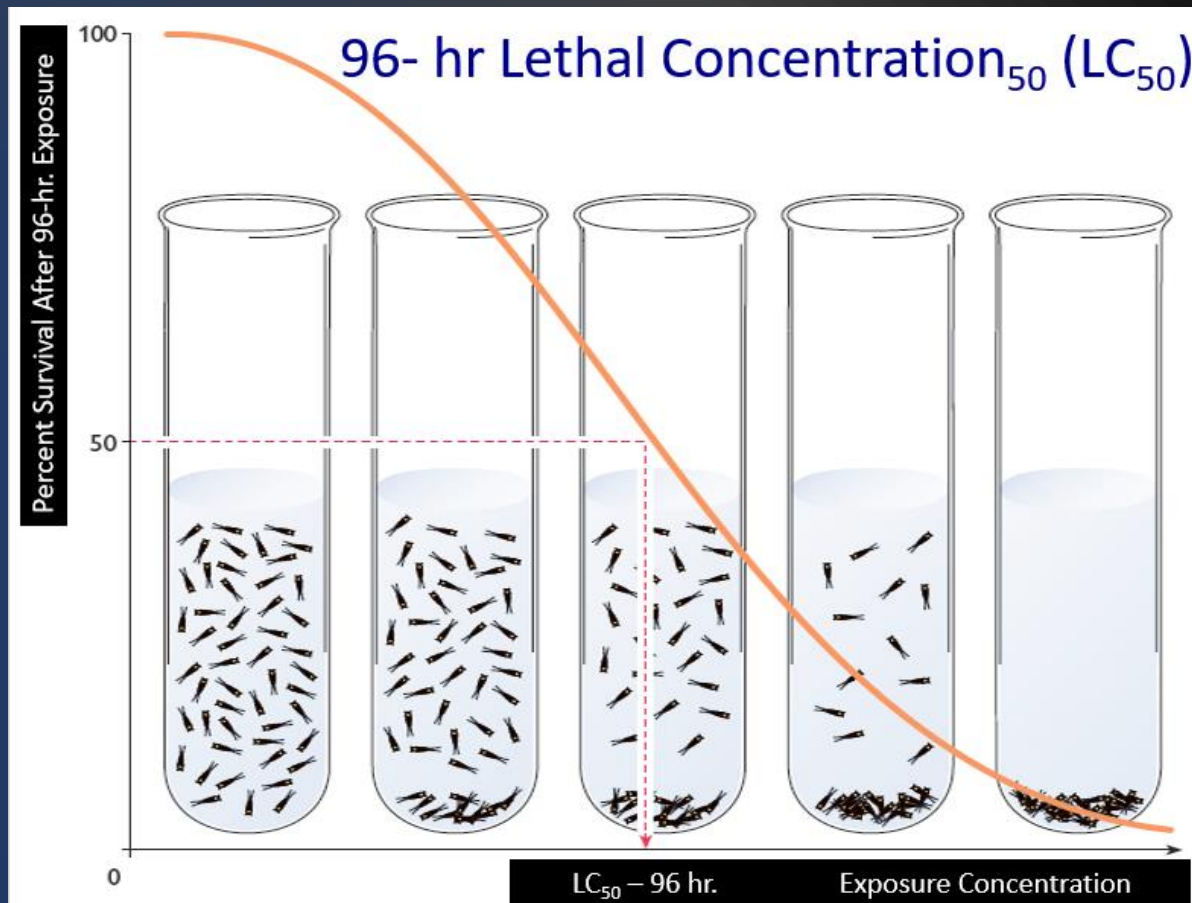
- Concentration in the water causing a **sub-lethal effect** to 50% of test population (e.g., growth rate)

Must state **Exposure Time!**



## Oil Toxicity

You really need a toxicologist to help figure it out, but at least know enough to ask the right questions.



Crude Oil Components.  
Consider sampling strategies for  
families of constituents.



Component

- C10 to C49+ isoparaffins
- C10 to C49+ cyclic paraffins
- C12+ mono-aromatics
- Poly aromatic hydrocarbons
- C10 to C49+ n paraffins
- C16+ di-aromatics
- C7 cyclic paraffins
- C8 cyclic paraffins
- Trimethyl benzenes
- Dimethylnaphthalene
- n-Heptane
- Methylcyclopentane
- Nonane
- Dimethyl benzenes
- Isobutane
- Octane
- Trimethyl naphthalene
- Hexane
- 3-Methylpentane
- 2-Methylheptane
- 2-Methylhexane
- Pentane
- Toluene
- Decane
- Tetramethyl benzenes
- Pentamethyl benzenes
- Isopentane
- Low level and unidentified hydrocarbons



# Interesting fact

Crude Oil with high sulfur and metals content can be acidic.

- Shippers and Refineries test for corrosivity using a Total Acid Number (TAN).
- naphthenic acids and sulfuric acids are a contributor to the overall low pH.

# Get out there and sample !



- Ephemeral Data is Critical to decisions and enforcement.
  - Water and sediment samples
  - Acute/chronic toxicity
  - Wildlife at risk of exposure (species/counts)
  - Specimens to document oil exposure
  - Document lost recreational/cultural use
  - Source oil sample (for “fingerprint”)



*Spill to Stream*



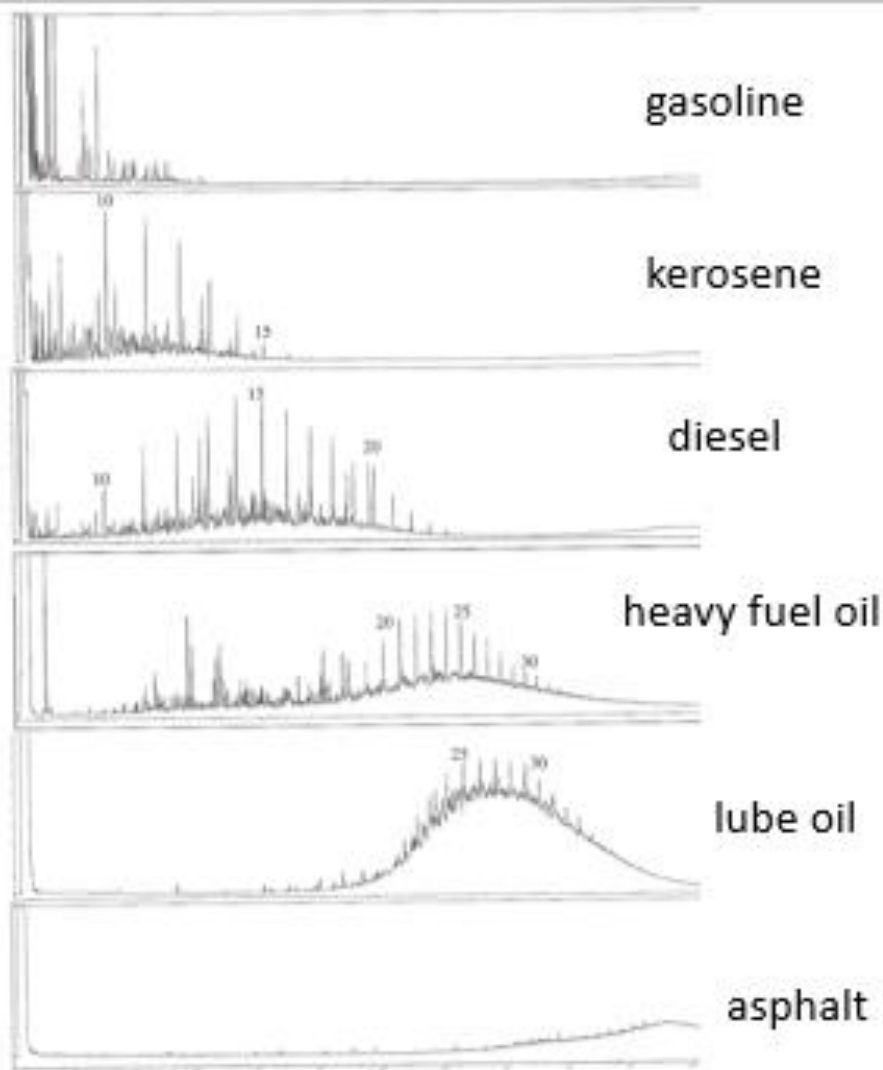
*Downstream Fish kill*

# Unknown Source Fingerprinting



Fuel Products  
from Crude Oil:

Each has their  
own GC  
pattern, which  
is one kind of  
fingerprint



Retain assistance from USCG !

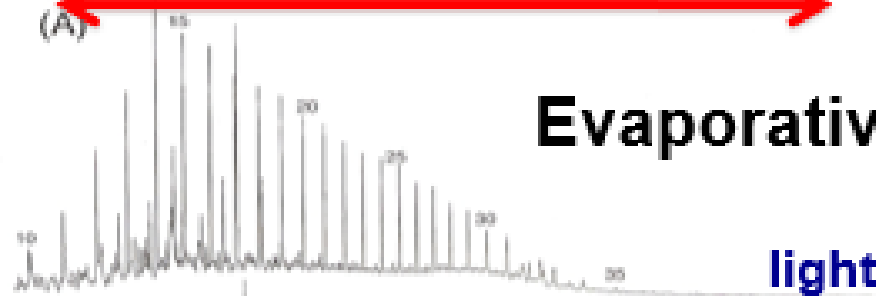


small low MW

large high MW

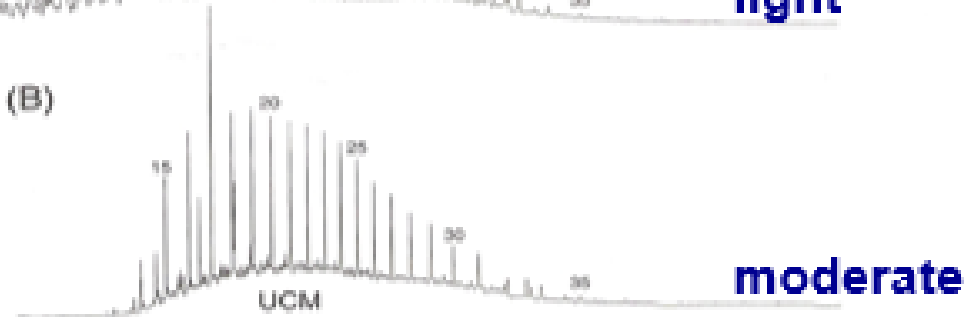


(A)

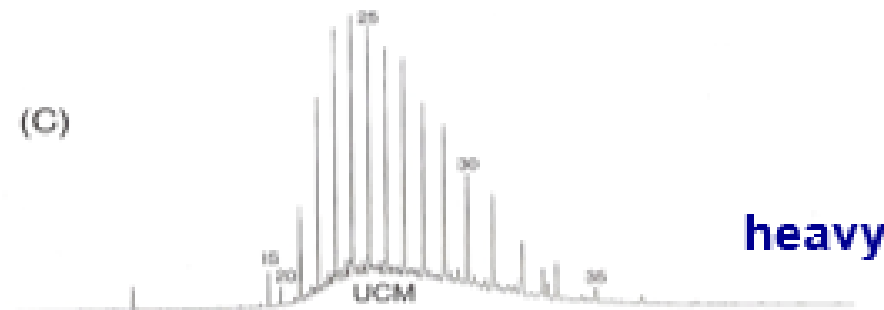


## Evaporative Losses

(B)



(C)



So, the GC fingerprint becomes less definitive as the oil weathers

# Solubility Matters !



When testing for constituents in the water, there will be an unequal picture of what was released into the environment. There will be fractions that dissolve and fractions that don't dissolve.

The monoaromatic compounds have orders of magnitude more solubility, compared to similar molecular weight alkanes.

As the number of carbon atoms increases, the solubility goes down.

Alkanes – hydrocarbons with single bonds, like methane, ethane, propane.

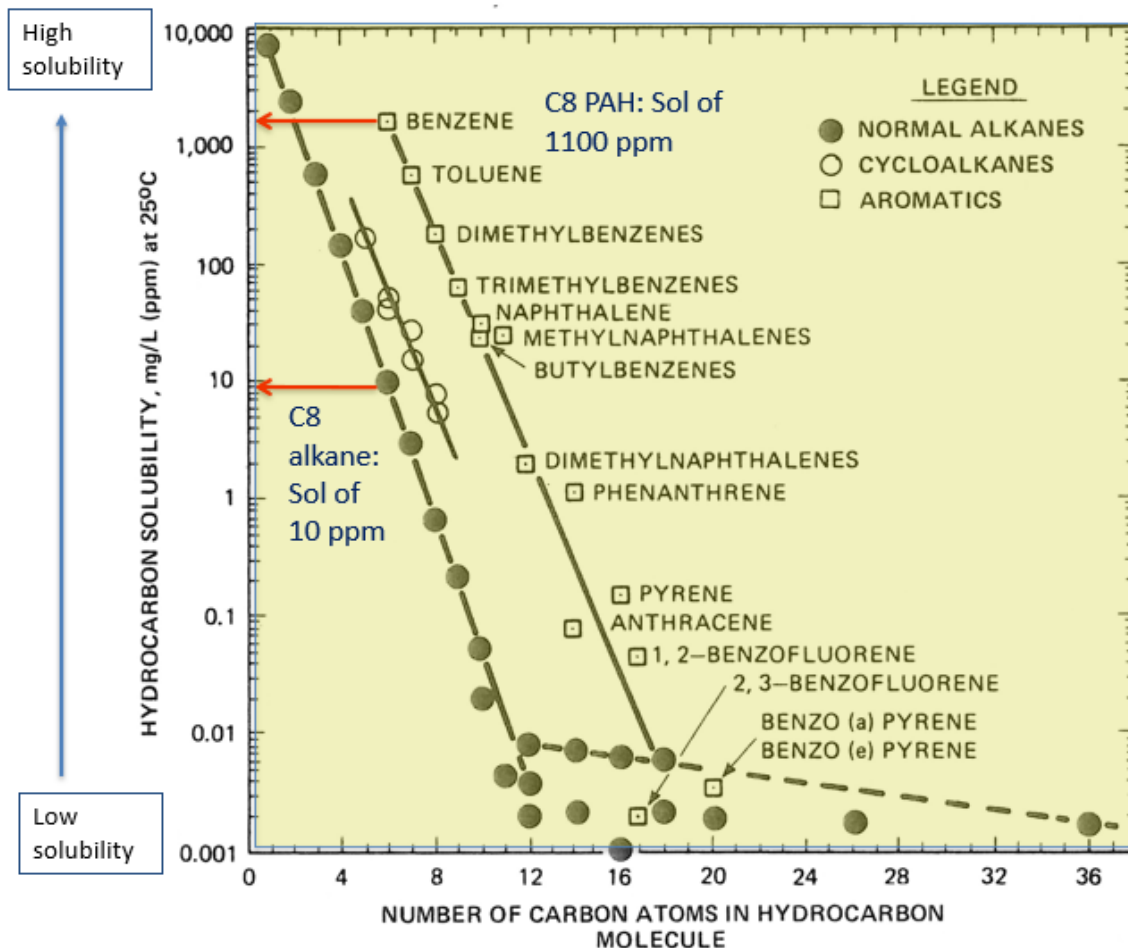
– often toxic but don't leave much of a footprint.

Aromatics – Benzene

- evaporative half-life in terms of hours in surface waters, however..
- to find benzene a month later in wetlands is not uncommon
- Benzene has been found in ground water decades after a crude oil release.

# Log plot of hydrocarbon solubility at 25°C.

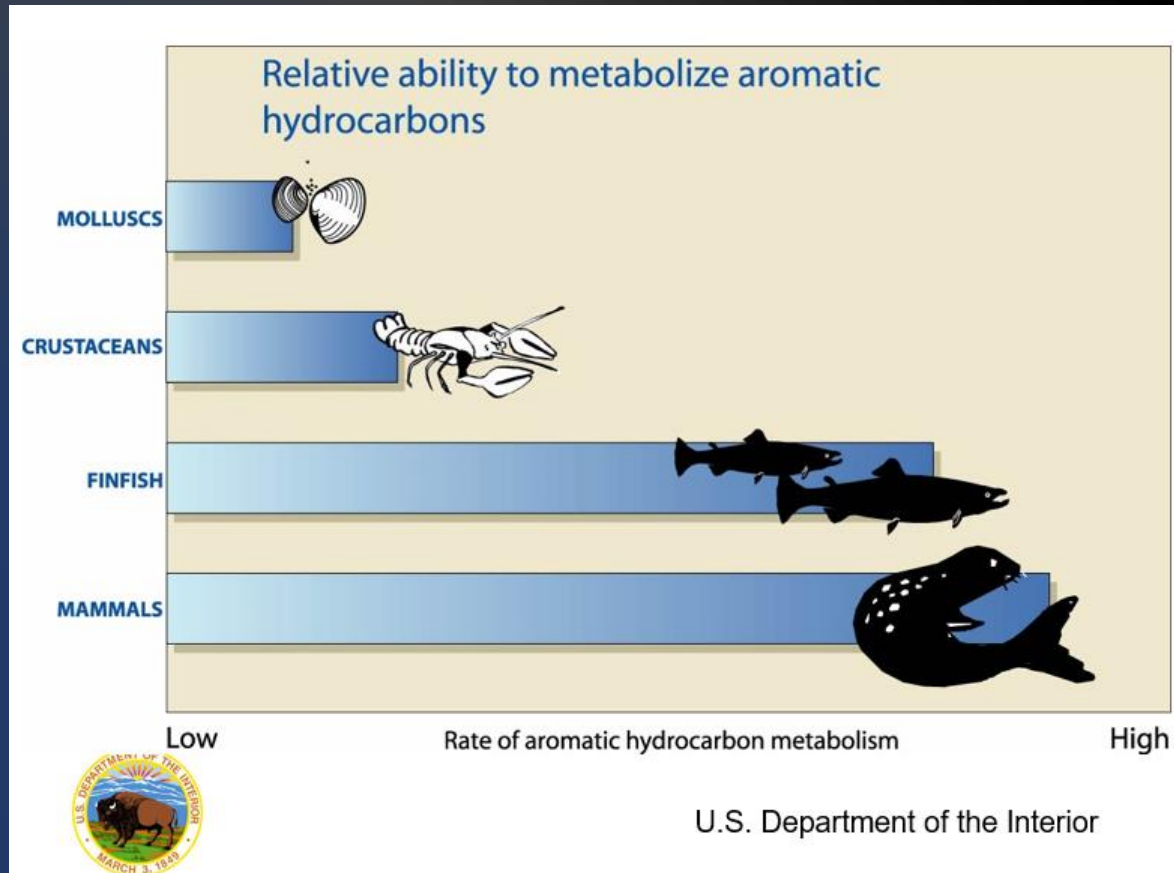
- Benzene has solubility of 1,100 ppm.
- Alkanes are much less water soluble and don't dissolve.  
C8 normal alkane has same number of carbon atoms.





If you are looking for a biological sample to document degree of oil exposure in the water column, shellfish are best!

- Molluscs have very slow metabolism rates.
- Other animals use enzymes to break down and excrete the HCs.





# Field Data Methods for Petroleum

- Sheen Test – grab sample
- (NAPL) Jar Tests – fractionation time
- Headspace Vapor Analysis – PID/FID
- Colormetric Test Kits / Immunoassays
- Fiber Optic Chemical Sensors (measures TPH)
- Field or Mobile Laboratories



# DISCLAIMER

- Information Discussed in this presentation is for information sharing between Environmental Regulatory Agencies and is not Legal Advice.





# Civil and Criminal Sampling for Enforcement

- Beyond the need for characterizing spilled materials for response, adequate documentation must be obtained for potential enforcement.
- Documentation must establish the fact that a violation has occurred.
- *Keep in mind that the lead investigator must establish a link to real persons and will strive to bring the highest responsible individual to justice.*



# Civil Enforcement

- EPA enforces applicable statutes (laws) and rules (written by the Agency and having an effect of law) through agency-delegated means of authority.

# Criminal Enforcement

- EPA has a Criminal Investigation Division (CID) with Special Agents to assist in investigations.
- They are specifically looking for deliberate conduct as evidenced by lying, falsifying records, fraud
- Enforces all Env. Laws (CWA/CAA/SDWA/FIFRA/+..)
- Big cases, CID can supply Haz Mat trained staff to take lab samples.



# Civil and Criminal Distinctions

- Civil:
  - Can manage the time,
  - Obtain consent to enter and sample,
  - Inspections and Formal letters requesting more information.
- Criminal:
  - Time constraints imposed,
  - Search warrants and evidence seizure,
  - Interviews, grand jury proceedings.



# Civil and Criminal Distinctions

- Civil:
  - Discovery by interrogatories and depositions,
  - Burden of proof; Preponderance of the evidence (>50%).
- Criminal:
  - Discovery by witness statements, reports of examinations and tests (laboratory analyses).
  - Burden of Proof; "beyond a reasonable doubt".



# Civil and Criminal Distinctions

- Civil:
  - Trier of Fact: Administrative Law Judge, federal or state judge, jury.
  - Often settled out of court through enforcement tools and negotiated settlements.
  - Penalties: Civil penalties, injunctive relief, site remediation.
- Criminal:
  - Trier of Fact: Federal or state judge, jury. (usually a jury),
  - Penalties: Fines, prison sentences for individuals, site remediation.



# Criminal Cases

- If criminal, what are the expected charges ?
- The way that prosecutors put together a case is often to work backwards from the sentencing guidelines that a Judge would use to impose a sentence.
- Seriousness and History, the offender's role in the incident, and victim(s) concerns, etc.
- The ongoing or repeat nature of the release, actual harm or potential of threat/risk





# Evidence

- Evidence is anything to indicate the guilt or innocence of a person or to aid in determining the truth about any fact in question.
  - Classes of evidence:
    - DIRECT; shows the existence of facts from personal witness knowledge
    - REAL or PHYSICAL; objects which speak for themselves and require no explanation, merely identification.
    - CIRCUMSTANTIAL; evidence that does not directly prove the fact but establishes a fact or series of facts which tend to prove certain elements of a case.
- Proof is the effect of evidence that leads reasonable persons (judge and jury) to draw reasonable conclusions.



# Rules for Evidence to be Admissible

- **RELEVANT**

- Does it tend to establish or disprove a fact ?

- **MATERIAL**

- Does it affect a fact or issue in the case significantly ?

- **COMPETENT**

- Is it adequate or capable evidence ?
- Is the witness qualified to testify about the facts in question ?

A witness commonly fulfills competency requirements if he/she is intelligent enough to understand the nature of an oath.

# Enforcement Sampling



- **Sample to demonstrate that a discharge or release occurred.**
- Sample the Contaminants of Concern listed in the SAP.
- Sample for any constituents or daughter products that may cause harm!

Cultural moment:  
Sampling for oil that  
travels under the ice of  
a frozen river.



# Reminder !

Bare Minimum Discharge Sample Collection.

- Upstream (Background)
- Discharge area (worst case, incident site)
- Downstream areas (fate and affect)





# Evidence of more than Background

**Whether Air, Water, Sediment or Soils it is imperative to determine “Background” in sampling.**

Difficulties:

- Historical data contains outliers and ambiguities
- Inland areas have lots of legacy contaminants
- Chemical fingerprinting may be required





# Standard Operating Procedures (SOPs)

- Review appropriate Agency SOPs and receiving laboratory methodologies for sampling. You may get asked in court if you know and followed data collection standards (validating data and establishing data credibility).







# Laboratory methodologies

- Correct sample bottle compatibility !
- Correct preservatives !
- Correct storage and holding times !
- Correct shipping temperature !





# *EPA has set national Standard Methods* for collection of samples

- Describes types of samples
  - Grab, composite, integrated.
- Spells out chain of custody procedures
  - Sample labels, seals, field log book, COC record, sample analyses request sheet, sample delivery to the laboratory, sample seals, receipt and logging of samples
- Describes sampling methods
- Describes sampling containers selection criteria,
- And quantifies the number and volumes of samples for statistical significance based upon standard deviations, desired level of confidence, etc.



# Sample Analysis Request Sheet

- **This sheet accompanies the samples to the laboratory.**
- The collector completes most of the pertinent information recorded in the log book.
- When the samples are transported via a commercial carrier, place the request sheet in a water-tight zip-lock type plastic bag and place the bag inside the cooler along with the samples.
- Some Analyses requests sheets are customized on tablet forms, can be filled in the field, and sent digitally to the receiving lab (or posted on a web-accessible platform/cloud sync).



## Sample Analysis Request Sheet cont.

- The laboratory portion of the form is completed by laboratory personnel and includes:
- Name of person receiving sample(s)
- Laboratory sample number
- Date and time of sample receipt
- Condition of sample (cold, warm, container full or half-full, color, cloudiness, particulates, more than one phase, e.g., water and sediment)
- Analyses to be conducted on sample(s)

# Chain of Custody Procedures For Environmental Samples:



Taken to **Establish** Laboratory Analysis As **Evidence** for  
A Possible Civil Or Criminal Enforcement Action



**Tetra Tech, Inc. | Biological Research Facility**

Project Manager or Client Contact: **Leanne Stahl**  
USEPA/OST (4305)  
1200 Pennsylvania Ave NW  
Washington, DC 20460  
(202) 260-7055

Address/Phone: \_\_\_\_\_

Contact Name/Phone: **Bob Smith** 717/555-1212

Project Number: **USEPA National Fish Tissue Study**

Sample Location: **Lake Sabula**

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Sample Identification/Station

Date	Time	Sample Identification/Station	Type of Analyses Requested	Preservative (Y/N)	Number of Containers
11/02/99	0815	PA 99 0039 P S Specimen #01	Y	I	✓
11/02/99	0900	PA 99 0039 P S #02	Y	I	✓
11/02/99	1415	PA 99 0039 P S #03	Y	I	✓
11/02/99	1510	PA 99 0039 P S #04	Y	I	✓
11/02/99	1520	PA 99 0039 P S #05	Y	I	✓

Sample check-in:  
DO \_\_\_\_\_  
T \_\_\_\_\_  
pH \_\_\_\_\_  
Cond/Salinity \_\_\_\_\_  
Chlorine \_\_\_\_\_  
Appearance \_\_\_\_\_

Collection Method \_\_\_\_\_ Log Number \_\_\_\_\_

Received by: **Bob Smith** Date/Time: **11/02/99 1310**

Received by: \_\_\_\_\_ Date/Time: \_\_\_\_\_

FORM DISTRIBUTION:  
WHITE - Sample Prep Lab  
GREEN - Tetra Tech  
YELLOW & PINK - Analytical Labs  
GOLD - Sampler





# Chain of Custody - Civil



- Custody is demonstrated by documenting that a sample or item is always in custody.
- Helps fulfill requirements for admissibility (relevance and authenticity).
- Field Custody. The samples or items must be in your actual possession or items are locked to prevent tampering or is placed in a secure area.
- A sample tag is required when complete chain of custody is required.



# Chain of Custody - Criminal



- If the likelihood of criminal investigations may occur, start the Chain of Custody at the time the sample bottles are picked up from the lab. In situations where the sample bottles are shipped, request that the lab ship them under seal for COC.

*A sample is considered to be under a person's custody if:*

- If it is the individual's physical possession,*
- In the individual's sight,*
- Secured and made tamper-proof by the individual, or*
- Secured in an area restricted to authorized personnel*



## Chain-Of-Custody Record (*EPA SOP*)

- Fill out a chain-of-custody record to accompany each sample or group of samples. This record should contain:
- Sample number
- Sample collector's signature
- Date, time, and place of collection
- Sample type
- Sample preservation, if any
- Signatures of all personnel involved in the chain of possession and the dates and times of possession

# Sample Labels



- Use labels to prevent sample misidentification.
- Gummed paper labels or tags are generally adequate.

The label should include the following information:

- A unique sample number
- Sample type
- Sample collector's name
- Date and time of sample collection
- Place of sample collection
- Sample preservative used, if any. *If used, include date and time of sample preservation*

# Sample Seals



Sample seals are used to detect unauthorized sample tampering up to the time of analysis.

- Affix seal so that it is necessary to break it to open the sample container or the sample shipping container, e.g., a cooler
- Affix the seal to the container before the sample leaves the custody of the sampler



# Chain of Custody - Digital

- Software Companies have patented 3-part authentication on digital COCs.
- The cooler seal also acts as the COC and comes with a scannable QR or Bar Code to simplify sample tracking and data entry.





# Field Log Books

- Because sampling situations vary widely, it is essential to record relevant and sufficient information so that one could reconstruct the sampling event without reliance on the sample collector's memory.
- Digital or manual.







# Field Log Book (*EPA SOPs*)

Include the following in the log book:

- Type of sample composition
- Purpose of the sampling
- Sampling Method and preservation (if any)
- Expected concentrations
- number of samples taken
- volume of samples taken
- date and time of collection
- Location and description of sampling point
- collector's sample identification numbers
- Collector's name and contact information
- sample distribution and method of transportation
- If the sample is wastewater, identify the process producing the waste stream
- Potential Responsible Party address (if known) being sampled, if different from location
- Land owner(s) information/permissions (if provided)



## Sample Delivery to the Laboratory

- Deliver samples to the laboratory as soon as practicable and **always within the holding time** of the analysis to be conducted.
- If more than one analysis is to be conducted, the shortest analysis holding time must be observed.
- When short holding times must be observed, make special arrangements to ensure timely delivery to the laboratory.
- Notify the laboratory in advance as to when the samples will arrive.
- Notify the lab if there is a rush on the samples.
- If expected sample concentrations are high, warn the receiving lab so they don't fry their equipment.



## Receipt and Logging of Samples

In the laboratory the sample custodian inspects the condition and seal of the sample and reconciles the sample label information with the chain-of-custody information before accepting the samples for analysis.



## Quality Control (QC) - know when to take sampler blanks and trip blanks and splits.

- Recommended one trip blank/cooler.
- Field blanks to ensure no cross contamination on reusable sampling equipment, one FB/day per sample matrix.
- One field duplicate recommended per ten samples for each matrix. Provides check on laboratory or between laboratories.
- Background samples required to demonstrate concentrations between contaminated and uncontaminated areas.



# Quality Control Methods

- Trip blank (historically also known as a field blank).
- Field (equipment ) blanks.
- Negative and Positive Plates (for bacteria). Field Duplicates.
- Lab Replicates.
- Spike Samples.
- Calibration Blank.
- Calibration Standards.

*Note: QC is a subset of Quality Assurance (QA) which includes a larger program that includes: Sampling plan/study design, SOPs, Staff Training, Data Management.*



# Civil cases often must document violation of a water quality standard

## Examples:

- Unionized Ammonia (>40)
- Turbidity (>25 NTU)
- pH (< 6.5 or > 9.0)
- Dissolved Oxygen (4 mg/l as min., 5 as daily avg. min.)
- Chlorides (salts): 230 mg/l chronic, 860 acute MS (1/2 FAV)
- PCBs, BTEX compounds, GRO/DRO/naphthalene, CBOD, etc.
- TOC or other pollutant loadings
- Other: pollutants orders of magnitude above background.



# Field Data/Monitoring



- **Field readings are enforceable under civil proceedings** unless standards methodologies are not followed.
- Collecting data on field parameters must also follow SOPs. Equipment must be calibrated, etc.
- Examples: Dissolved Oxygen Drops (supporting the COD/CBOD analyses); Temperature shocks; pH changes; salinity and conductivity, etc.

## In the Field:

- Set your own pace (Nervous inspectors will miss a lot).
- Assist the principal investigator in determining; Who, What, When, Where, Why, and How.

# Can't get properly preserved (refrigerated) samples to the lab on time?



- Laboratories will flag analytical results that exceed holding times or temperatures. Opponents to the data will say the data is not valid.
- Collect and Run (analyze) the samples anyway.
- Samples exceeding holding times are not invalid, they just have a larger error margin.
  - Even if a lab says the analytical results are potentially off by 20 to 50 percent, the sample still shows the contamination is present.
  - Often samples show that the contamination is also orders of magnitude above a water quality standard (far outside the analyses variability).
  - Judges will understand logistical problems if explained.

# Photographic Evidence



- Take a representative pictures, document conditions.
- Step Back and take a scene-scale photo, too many site photos show small detail, hard to explain to a judge, paint a story.
- *“Take Photographs like you want them to be discovered”.*



# Proving that there is a persistently generated sheen



## Visual inspections preferred vs. chemical analyses:

- Sampling is difficult – take a photo or video.
- High variability, a random grab sample may show little.
- Turnaround time for results problematic
- A photograph is evidence and proves the point.
- No guidelines on what oil levels are “safe”.





# Terrestrial Ecological Evaluations

## *Monitoring and Sampling*



A site-specific terrestrial ecological evaluation should be conducted by an experienced habitat biologist or ecological risk assessor.

- conducting a site-specific evaluation can be time consuming, expensive, and take more than one season.





# Summary Points on Sampling

- Request that the Responsible Party submit a SAP promptly.
- Document Harm,
- Proceed with the proper acquisition of Data.
  - Entry and private property, get permission if not collecting data from off-site or from “Plain View”





## Summary Points on Sampling

# Document/Document/Document

- You have no personal interest in the case, be absolutely sure of the facts.
  - You may be called as an expert witness or a factual witness (An expert witness can have an opinion in court).
- Be prepared to defend your data in a Civil Appeal.
- Be prepared to defend your data and your credentials in a contested or criminal case.
  - (Picture yourself being deposed or Cross-Examined ).



# Put together an oil spill “go-box” !

- Field Logbook, pen, pencil
- Sample tags
- Sample labels
- Appropriate supply of gloves
- Blank analytical request forms
- COC record form
- Shipping cooler custody seals
- Camera/phone
- Also: be prepared to grab and go with calibrated equipment and fresh sample bottles and preservatives.



# QUESTIONS ?

*David Morrison*  
*Federal On Scene Coordinator*  
*U.S. EPA / Region 5*  
*[morrison.david@epa.gov](mailto:morrison.david@epa.gov)*