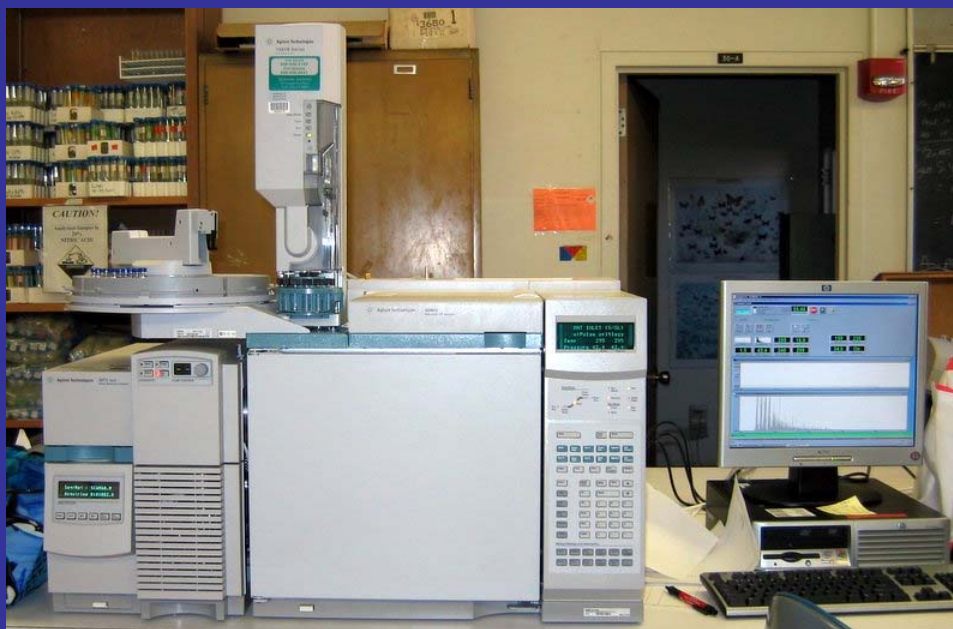


Gas Chromatography Mass Spectrometry (GCMS)



Organics

- Chlorinated pesticides
 - DDT
 - Chlordanes
- Polychlorinated biphenyls (PCBs)
- Polyaromatic hydrocarbons (PAHs)

DDT (Dichlorodiphenyl-trichloroethane)

- Used as an insecticide
 - Control malaria and typhus during WW II



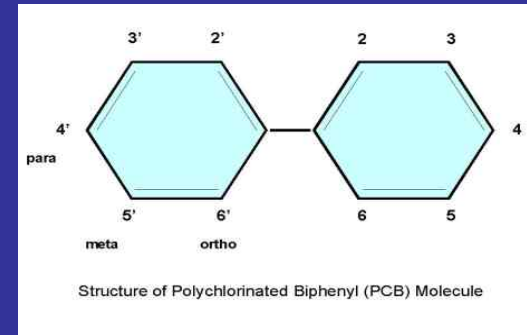
DDT (Dichlorodiphenyl-trichloroethane)

- Used as an insecticide
 - Control malaria and typhus during WW II
 - $\leq 80\%$ 4,4'-DDT, $\leq 20\%$ 2,4-DDT, and $\leq 4\%$ 4,4'- DDD
 - Breaks down into 4,4'-DDE, 2,4'-DDE and 2,4-DDD
- Used as an agricultural pesticide
- Banned in 1972 in the US
 - Causes egg shell thinning
- Southern CA- world's largest DDT manufacturer Montrose Chemical Corporation



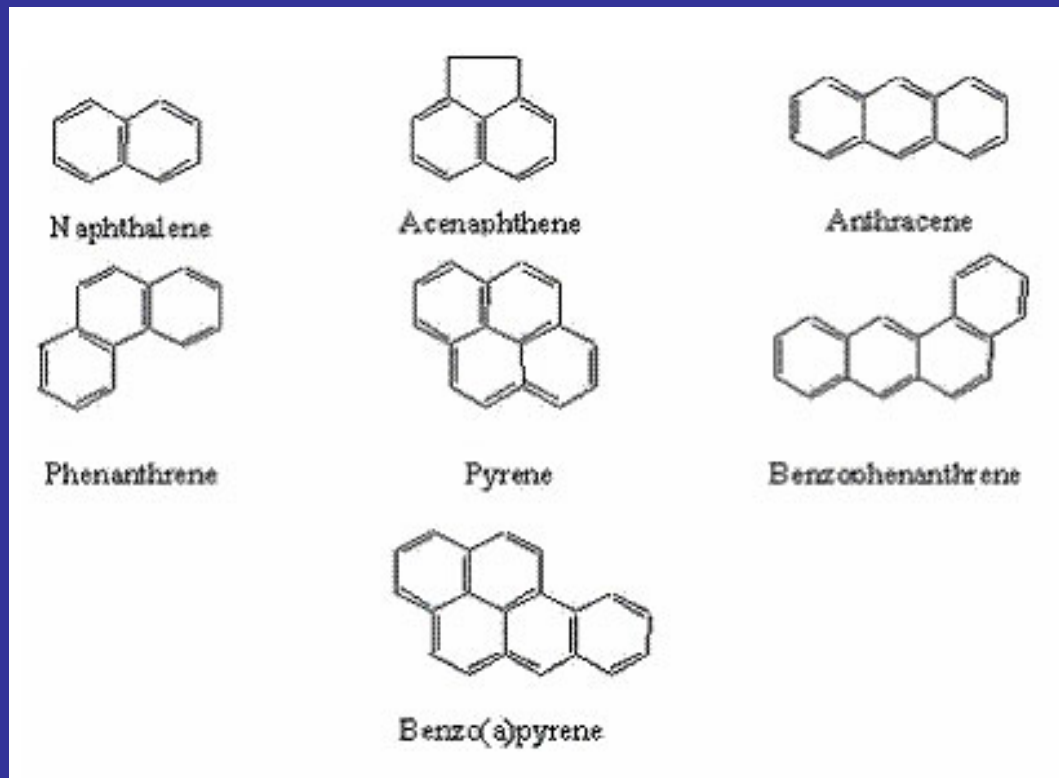
PCBs

- 209 congeners
- Often referred as Aroclor
 - i.e.- Aroclor 1260
- Used for hydraulic fluids, insulators, carbonless papers
- Banned in 1976 in US



Polyaromatic hydrocarbons (PAHs)

- Occur in oil, coal and tar
- Benzo [a] pyrene- 1st chemical carcinogen discovered



Analytical Procedure

Sample Preparation

Extraction

Cleanup

Quantification

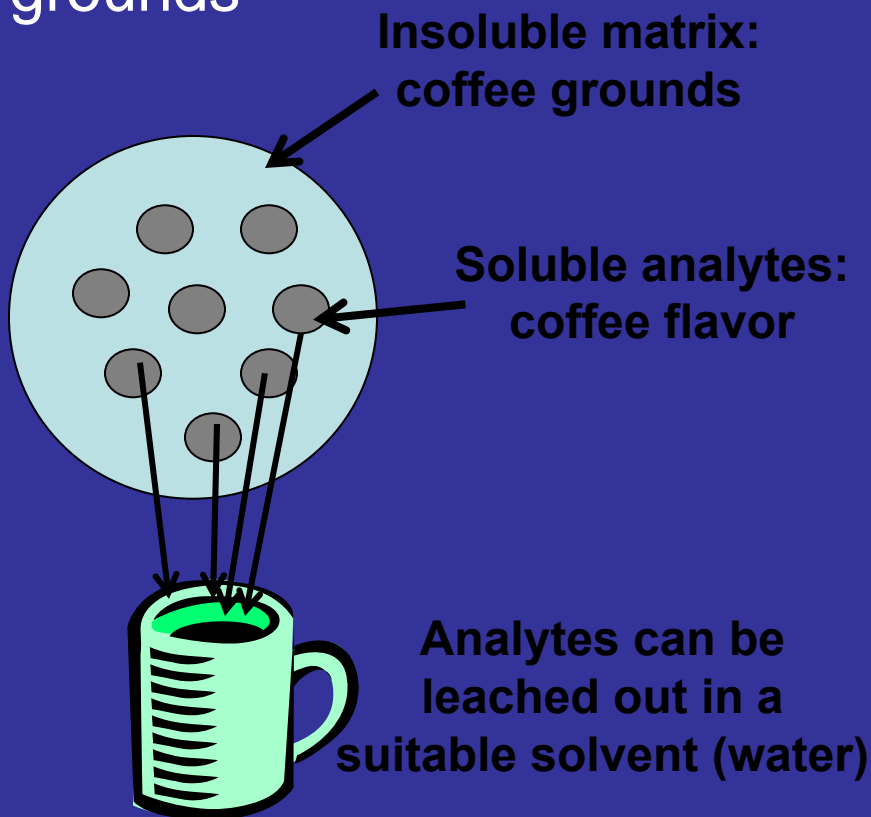
Quality Assurance/Quality Control

Sample Preparation

- Involves modifying the physical form of the sample so that the analytes can be extracted efficiently
 - Homogenize sample

Extraction

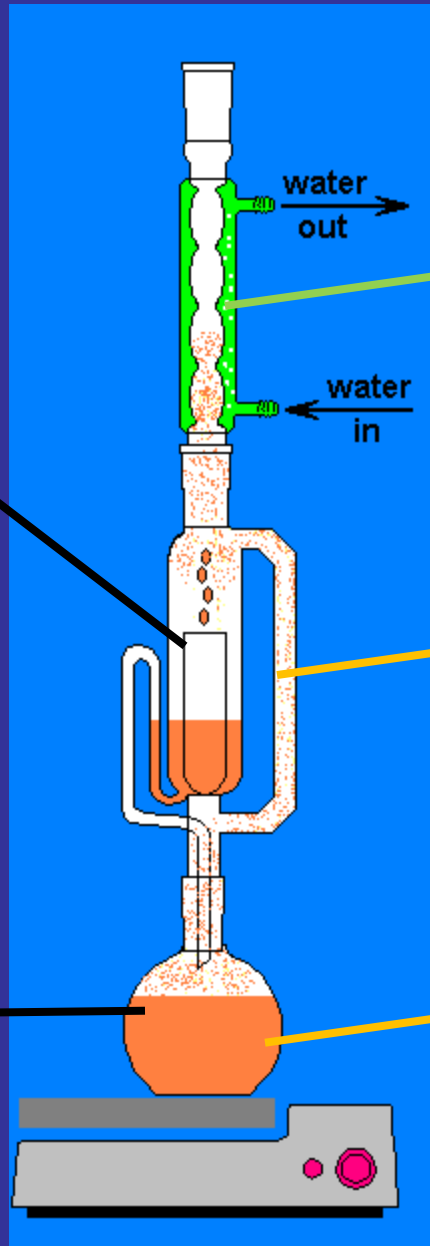
- What is the purpose?
 - Used when we want to separate substances
- How do we separate substances?
 - Use a solvent in which the desired substance dissolves in and the undesired substance does not dissolve in
 - Example: coffee grounds



Soxhlet Extractor

Cellulose thimble:
10-15 g of sample inside

200 ml 3:1
DCM:acetone

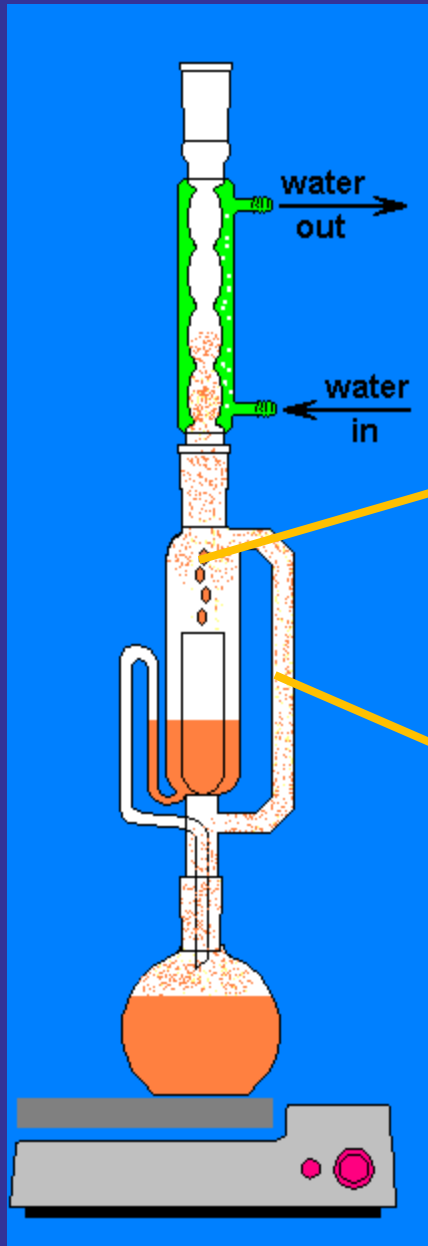


Condenser

Extractor

Boiling flask

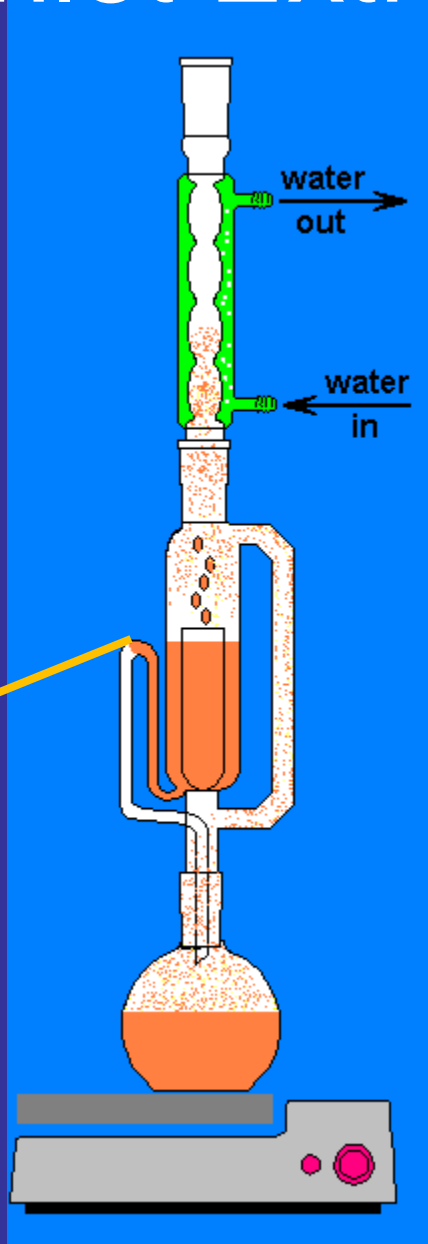
Soxhlet Extractor



2) Condensed drops of solvent fall into thimble, dissolving analytes from the matrix

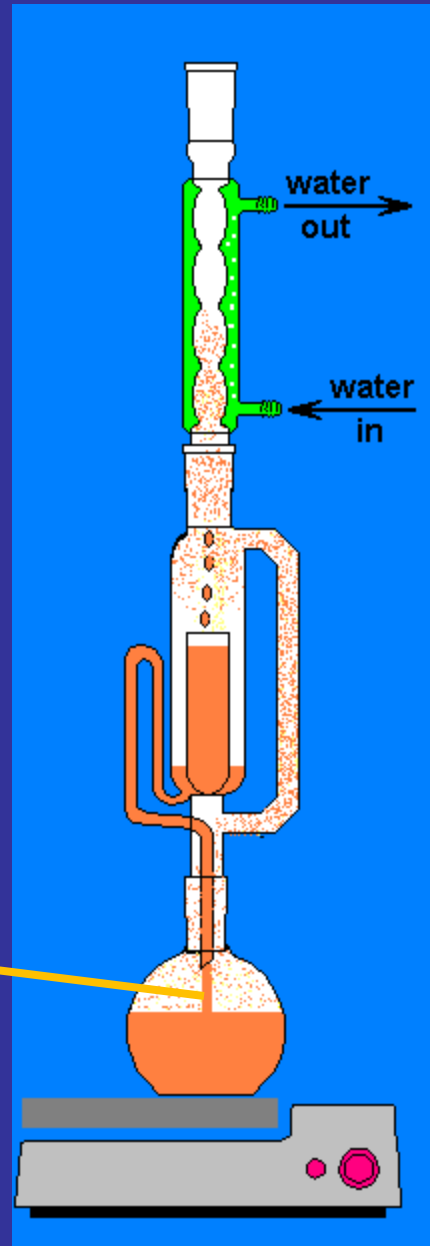
1) Boiling solvent vapors rise up

Soxhlet Extractor



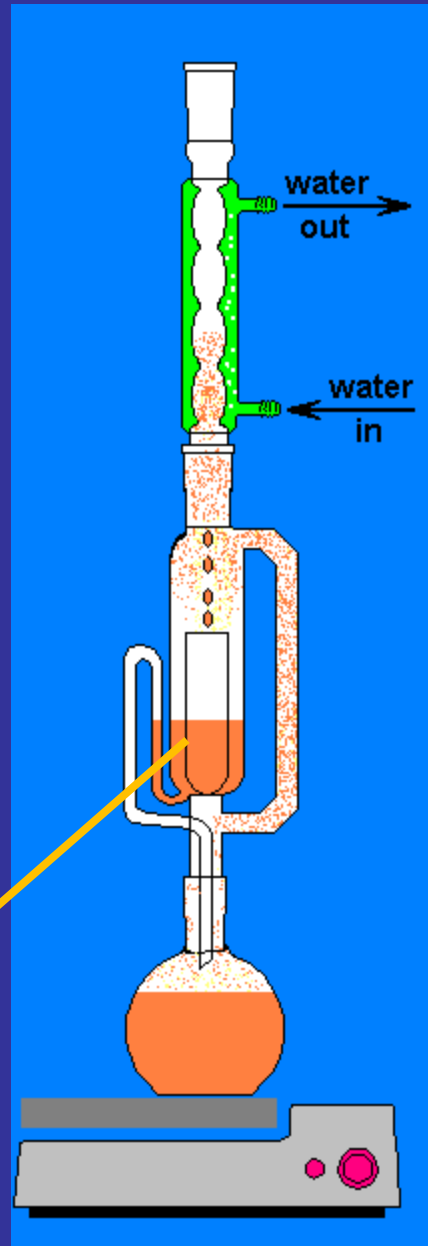
3) When smaller side-arm fills to overflowing, siphoning action is initiated

Soxhlet Extractor



4) Solvent (containing the dissolved analytes) is siphoned into the flask

Soxhlet Extractor



5) Residual solvent drains out of cup, as fresh solvent continues to fall

Concentrate sample

- Called rotovaping
- From ~200 mL to ~ 1 mL
- Lipid determination (infaunal samples only)
 - Split the sample 50:50
- Solvent exchange: DCM to hexane
 - Add ~ 2 mL hexane and concentrate again to ~ 1mL

Extract cleanup

- Column chromatography: silica gel and alumina cleanup
- Aim: to remove compounds that may interfere with analyte analysis
- Principle:
 - Concentrated sample is deposited onto the column
 - Column is washed with the appropriate solvents
 - 30 mL hexane
 - 15 mL 30% DCM/Hexane
 - 15 mL DCM
 - Analytes are eluted with the solvents whereas the interfering compounds remain on the column
 - Concentrate sample
 - From ~60 mL to ~ 1 mL

Quality Assurance/Quality Control (QA/QC)

Quality Assurance vs. Quality Control

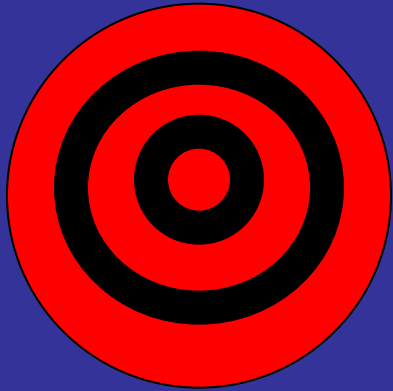
- An overall management plan to guarantee the integrity of the data
- Series of analytical measurements used to assess the quality of the analytical data

Precision

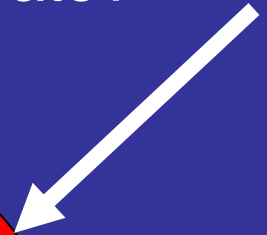
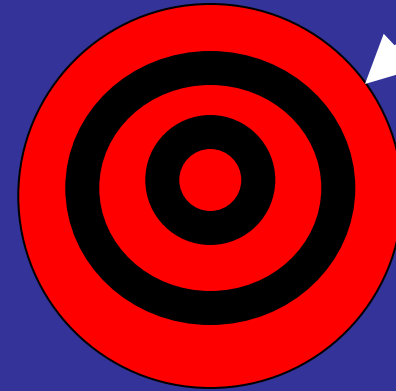
vs.

Accuracy

- Are results reproducible?



- Are results accurate?



Precision

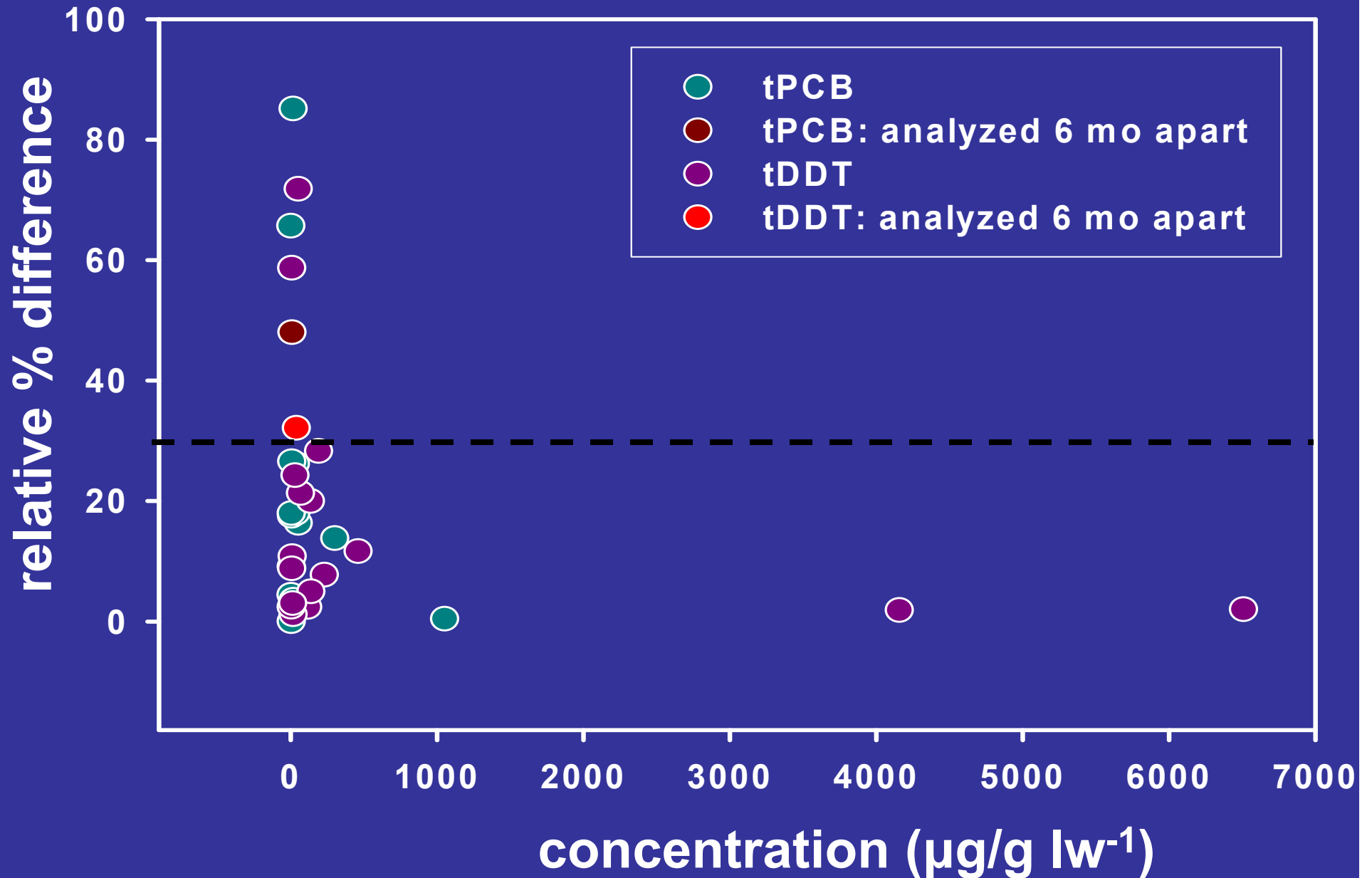
vs.

Accuracy

- Are results reproducible?
 - Field duplicates

- Are results accurate?

Duplicate error, n = 20



Precision

vs.

Accuracy

- Are results reproducible?
 - Field duplicate

- Are results accurate?
 - Surrogates
 - Blanks
 - Matrix spike (MS)
 - Do in duplicate: Matrix spike duplicate (MSD)
 - Standard reference material (SRM)

Surrogates

- Monitors extraction efficiency
 - Most common in organic methods
- Similar to analytes you are analyzing
- Added to ALL field samples and QAQC samples
 - Added prior to extraction
- Surrogate recovery criteria?

Blanks, Blanks, Blanks

- Method Blank
 - Clean sample (solvent only) subjected to ALL sample prep
- Instrument Blanks
 - Clean sample (e.g.- hexane) used to determine instrument contamination
- Field Reagent Blanks
 - Prepared in the lab, taken to the field
 - Opened at the sampling site, exposed to sampling equipment, returned to lab
- Trip Blanks
 - Prepared in the lab, taken to the field
 - Not opened, returned to the lab

Matrix spikes (MS)

- Sample spiked with a known amount of ALL analytes
- Subjected to ALL sample prep and analytical procedures
- Determines the effect of the matrix on analyte recovery
- Done in duplicate
- Laboratory Control Spikes (Blank spike)
 - All analytes spiked into solvent
 - Subjected to ALL sample prep and analytical procedures
 - Done in duplicate

Standard Reference Material (SRM)

- Homogeneous material where analyte values have been well established to validate lab and analytical methods
- Use similar matrix to samples
 - Lake Michigan Fish Tissue (SRM 1947)
 - Sediments (1941b)