STRATEGIES FOR INCREASING SUCCESS RATES OF TOXICITY IDENTIFICATION EVALUATION STUDIES

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Outline

- What is a TIE?
- Common Applications
- Critical Factors for Successful Execution
- Case Study

What is a TIE?

- **Purpose** – to characterize, identify, and confirm the cause of toxicity in a given sample in order to solve a problem
- Range of chemical and physical manipulations

- Why is it toxic?
- What can I do about it?

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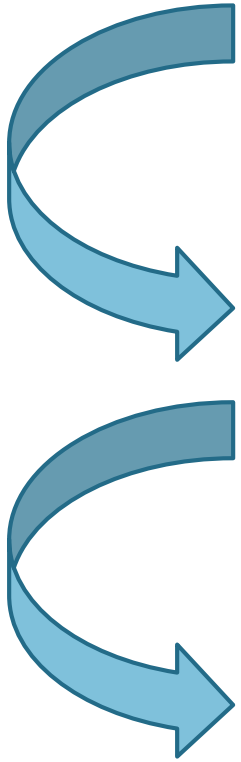
TIE Phases



Phase I: Toxicant Characterization

- Determines general physical and chemical properties and class(es) of constituent(s) of concern
- May also eliminate groups as possible toxicants

Phase II: Toxicant Identification

- Tentative ID of specific toxicants
- Done through additional treatments designed to further isolate and characterize
- Testing approach and cost depends on results obtained during Phase I

Phase III: Toxicant Confirmation

- Toxicant of concern is confirmed
- Techniques may include analytical quantification, spiking studies, correlation analyses, and overall weight of evidence
- Goal is to ensure clear, scientifically sound conclusions

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Common Phase I TIE Procedures

Treatment	Purpose/Target Constituent
Baseline	Unmanipulated sample. Used for evaluation of treatment effectiveness
Solid-Phase Extraction	Non-polar organics
Filtration	Suspended particulate material
Aeration	Volatile/Sublatable Compounds
EDTA Addition	Divalent Cationic Metals
Sodium Thiosulfate (STS) Addition	Chlorine and other Oxidants
Piperonyl butoxide (PBO)	Organophosphate and Pyrethroid Pesticides
Carboxylesterase enzyme (CEE)	Pyrethroid Pesticides

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Common Applications

- NPDES compliance
- Storm water/receiving water studies
- TMDL development
- Ecological risk assessment
- Litigation apportionment

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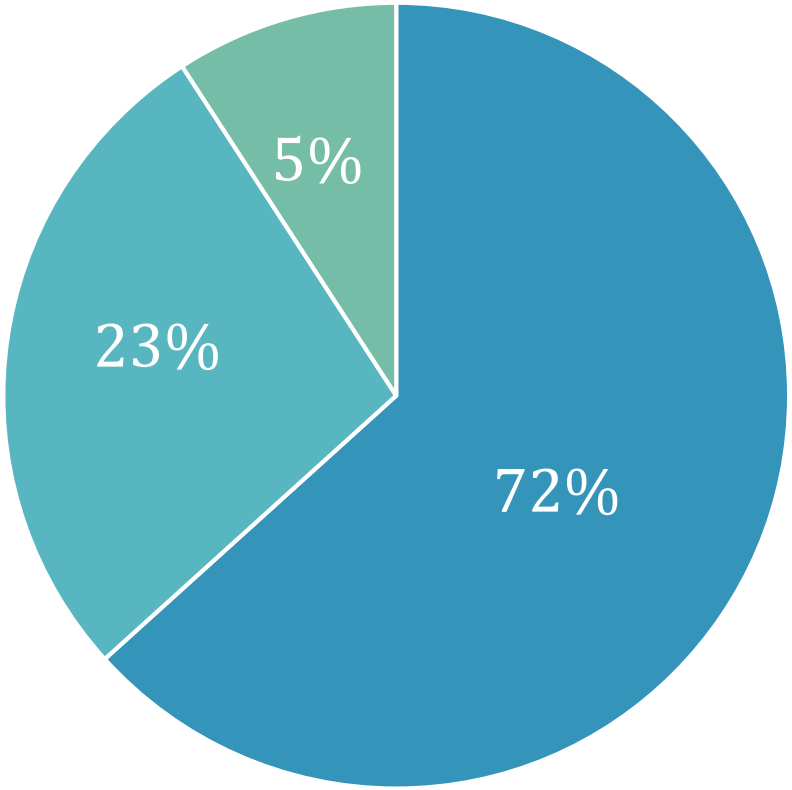
Success Rates



- TIEs **>90%** successful

when designed and applied skillfully

- 22 initial Phase I TIEs over 15 months
- 94% success when baseline toxicity present

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Critical Steps

- **Study Design & Planning**
  - The right questions
  - Sample selection
  - Species-specific considerations
  - Method adaptations
- **Execution**
  - More for another day
- **Interpretation & Communication**
  - Putting it all together

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The Right Questions

- **Defining the right questions before beginning is crucial to success!**
  - Site-specific issues
  - Historical data review; site manager and staff
  - Regulatory context
  - TRE
  - Magnitude of response, replication
  - Interactions and interferences
  - Iterative approach
  - Reduction in treatments

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TIE Sample Selection

- **Requires baseline toxicity…but how much?**
  - Minimum significant differences
  - Endpoint variability impacts
- **Timeline**
  - Is toxicity persistent or transient?
  - Can another sample be collected?
  - Concurrent TIE treatments with routine monitoring
- **Regulatory triggers**
  - Required actions

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Species-specific Considerations

- Sensitivity
- Exposure route
- Study specific goals
- Sufficient background information
- Replication

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Method Adaptations

- Targeting the obvious (but don't assume)
- Iterative approach
- Interactions/interferences
- Adapting outdated methods to the present
- Resulting cost implications

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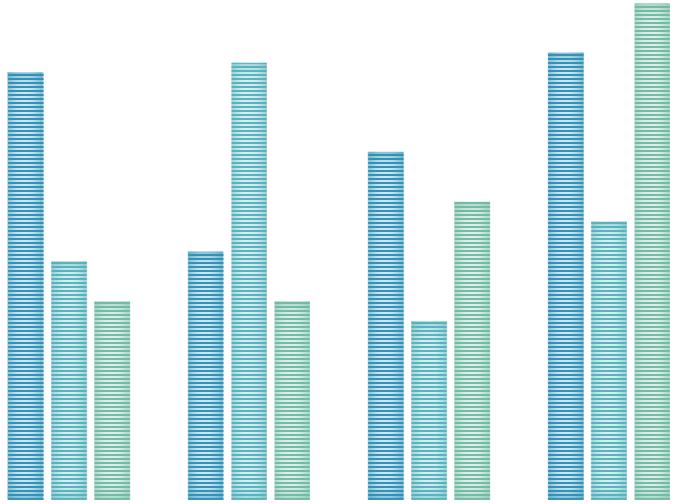
Data Interpretation

- **Most critical factor!**
  - Analyst skill, experience, and training
  - Integration of biology and chemistry
  - Proper method controls
- **Multiple lines of evidence**
  - Defensibility
  - Pair with chemistry data whenever possible
  - Use study designs to exclude/isolate constituents of concern



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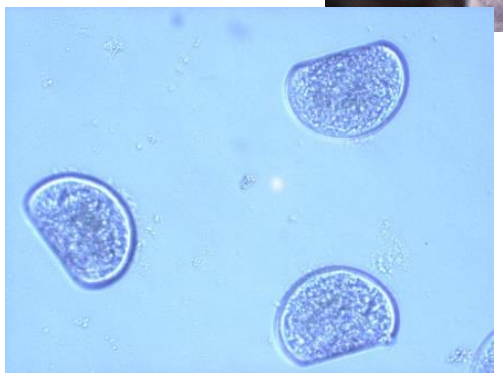
TIE Case Study

- **The Problem:**
  - Statistically significant effects to mussel embryo development, in excess of NPDES permit limit
  - Accelerated monitoring confirmed not an isolated event
  - TIE triggered



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TIE Case Study

- **The Site:**
  - Effluent discharge into marine environment
  - Consists of groundwater and stormwater
  - Limited treatment prior to discharge
  - Known challenges with metals and ammonia based on available chemistry and past performance

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TIE Case Study

- **The Initial Approach:**
  - Initial treatments focused on metals and ammonia
  - pH 10 sample adjustment removed all toxicity; EDTA did not, indicates there is something beyond metals

Sample ID	Treatment/ Concentration	Mean Normal Development (%)	Standard Deviation (%)
Method Controls	Lab Control	97	2.1
	Salt Control	97	1.4
	EDTA 25 mg/L Control	95	1.1
	pH 10.7 + 0.45 µm Filtered Control	96	1.9
	C8 SPE Control	98	3.4
Sample	25% Baseline	95	1.6
	50% Baseline	94	1.3
	100% Baseline	47	9.0
	100% EDTA 25 mg/L	40	4.6
	100% pH 10.7 + 0.45 µm Filtered	94	1.3
	100% C8 SPE	43	5.3
	100% Baseline (no pH control)	21	4.5

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TIE Case Study

- **The Iterative Part:**
  - Sample had high alkalinity
  - Anion scan showed high bicarbonate
  - Treatment method developed to target bicarbonate only (leaving ammonia and metals)

Analyte (mg/L)	Baseline	pH 10.7 + 0.45µm Filtration	pH 5.6 + Aeration
Copper	0.0084	0.0052	0.0087
Chloride	8700	8600	9100
Sulfate	1000	1100	1200
Bicarbonate (HCO_3)	720	220	130
Total Alkalinity ($CaCO_3$)	720	220	130

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Sample ID	Treatment/Concentration	Mean Normal Development (%)	Standard Deviation (%)
Method Controls	Lab Control	93	2.0
	Salt Control	94	1.9
	pH 5.6 + Aeration Control	94	2.3
	pH 10.7 + 0.45 µm Filtered Control	95	1.6
	EDTA 25 mg/L Control	92	2.4
100% Sample	Baseline	0.0	0.0
	pH 5.6 + Aeration	41	7.0
	pH 10.7 + 0.45 µm Filtered	92	2.7
	EDTA 25 mg/L	0.0	0.0

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Sample ID	Treatment/Concentration	Mean Normal Development (%)	Standard Deviation (%)
Method Controls	Lab Control	98	1.2
	Salt Control	98	1.5
	pH 5.6 + Aeration Control	97	1.0
	pH 10.7 + 0.45 µm Filtered Control	95	1.0
	EDTA 25 mg/L Control	96	2.0
100% Sample	100% Baseline	38	2.3
	100% pH 5.6 + Aeration	98	1.2
	100% pH 10.7 + 0.45 µm Filtered	96	2.0
	100% EDTA 25 mg/L	31	3.0

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TIE Case Study

- **The Interpretation & Integration:**
  - Additional TIEs showed metals were a minor/moderate and inconsistent contributor
  - Ammonia occasionally played a role, data could predict when it would be an issue
  - Mechanism of effect for mussels was likely excess bicarbonate that inhibited shell building

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TIE Case Study

- **The Happy Ending:**
  - Concurrent targeted TIEs run with routine monitoring
  - Relief from costly accelerated testing and additional TIEs
  - Confirm patterns as problem was addressed
  - Next steps - bicarbonate spiking

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Questions?

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