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Lab Solutions

Highly Sensitive Detection of Geosmin and MIB by Purge and Trap with GC/MS

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Xylem Lab Solutions, North America

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Agenda

- Part I: Harmful Algal Blooms and Geosmin/MIB
- Part II: OI Analytical Geosmin/MIB Method via EPA Method 524.3

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Harmful Algal Blooms and Geosmin/MIB

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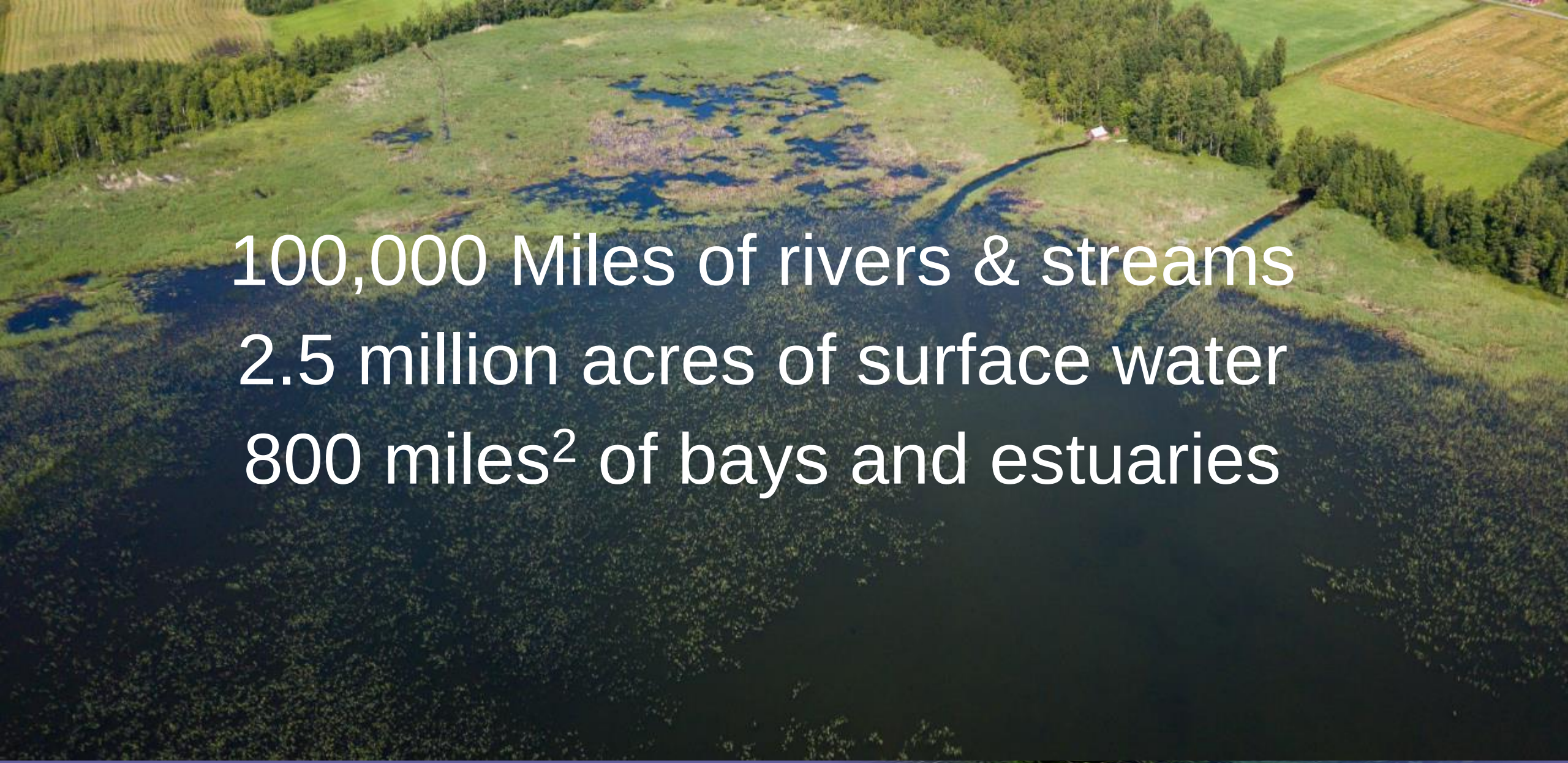
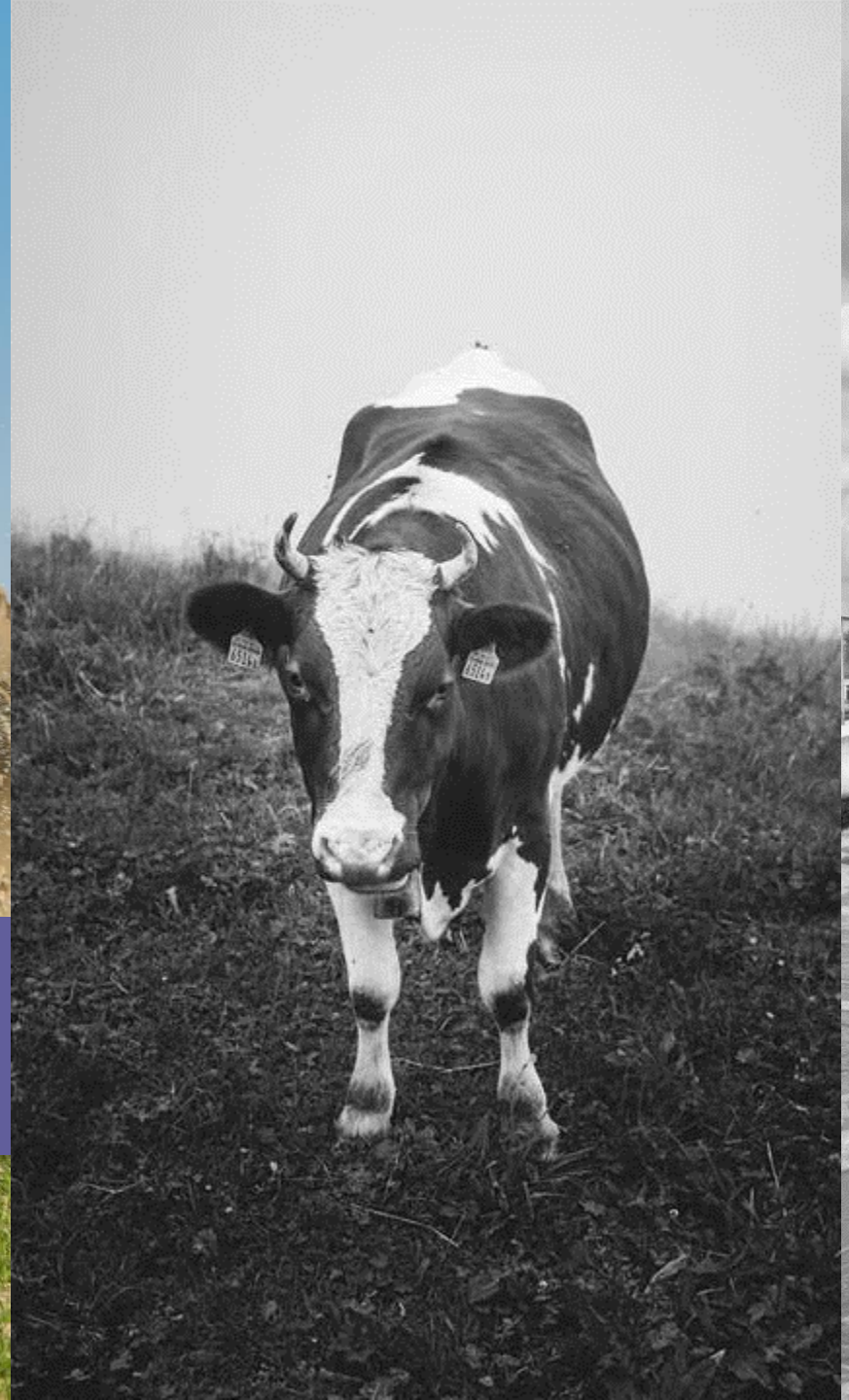
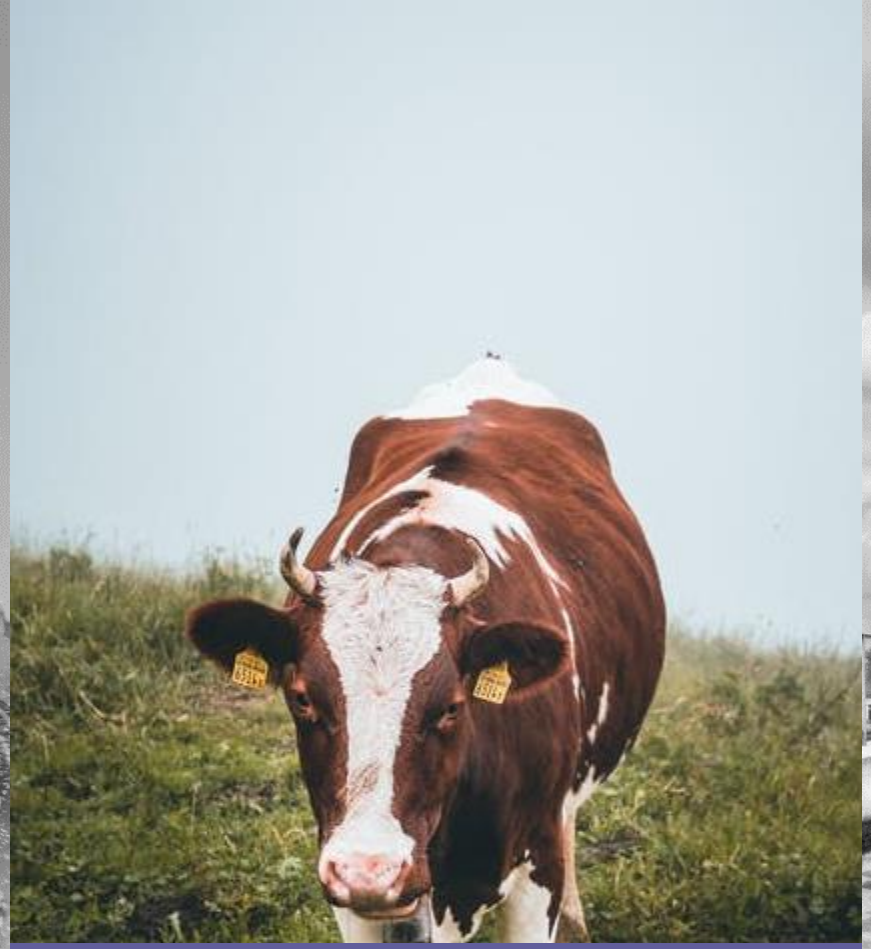


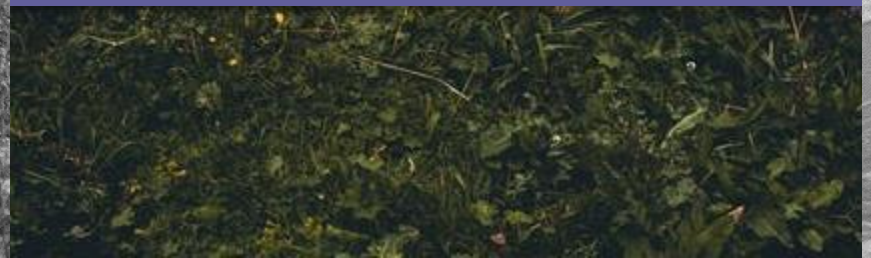
https://www.epa.gov/nutrientpollution/where-nutrient-pollution-occurs, accessed June 2022

Fertilizers





Animal Manure



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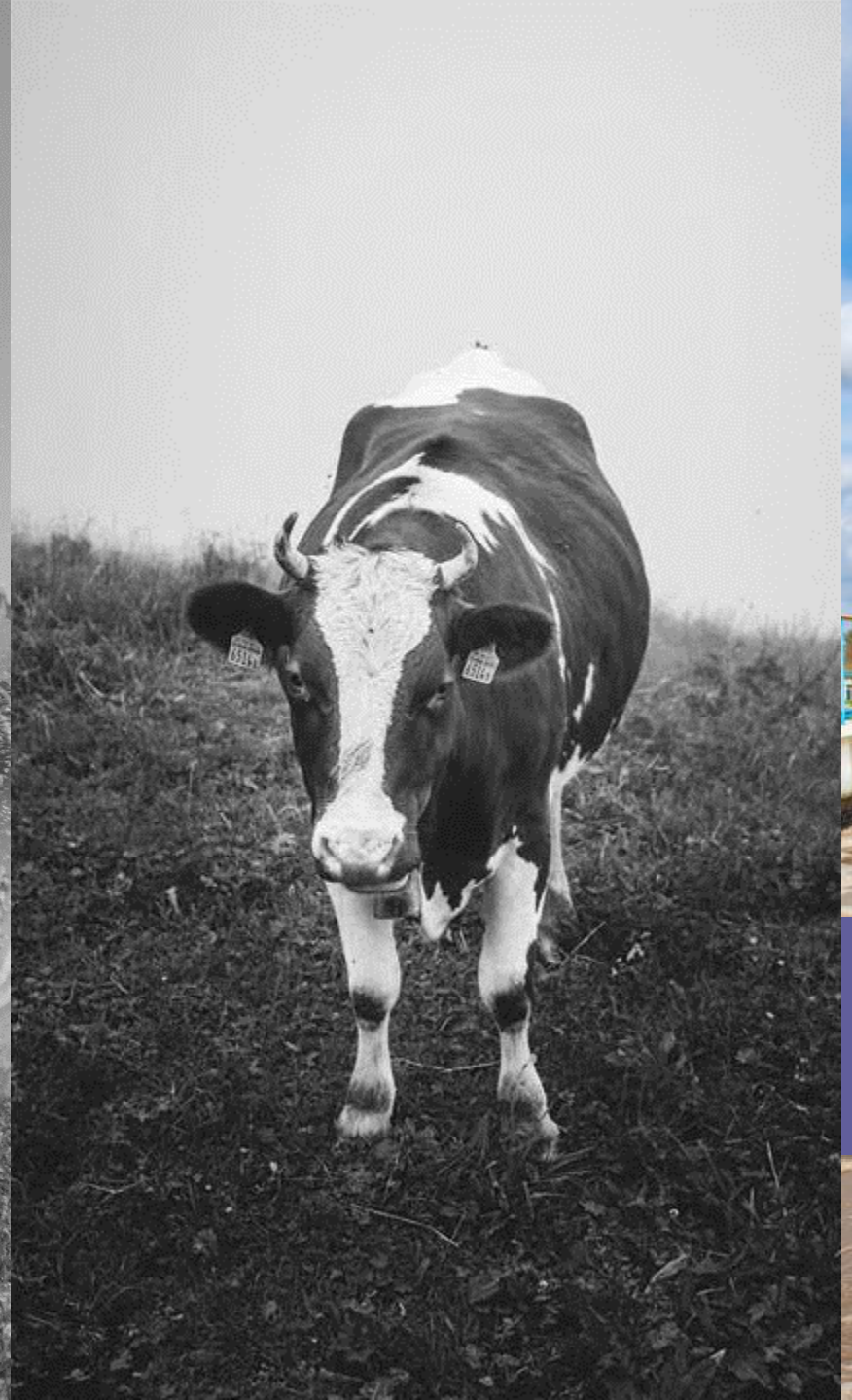

Wastewater Effluents

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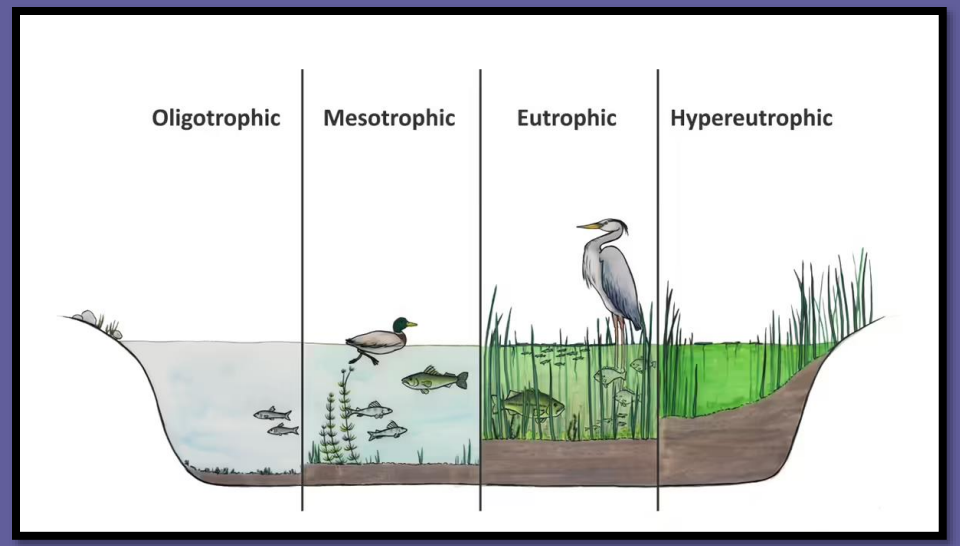
Nutrients and HABs

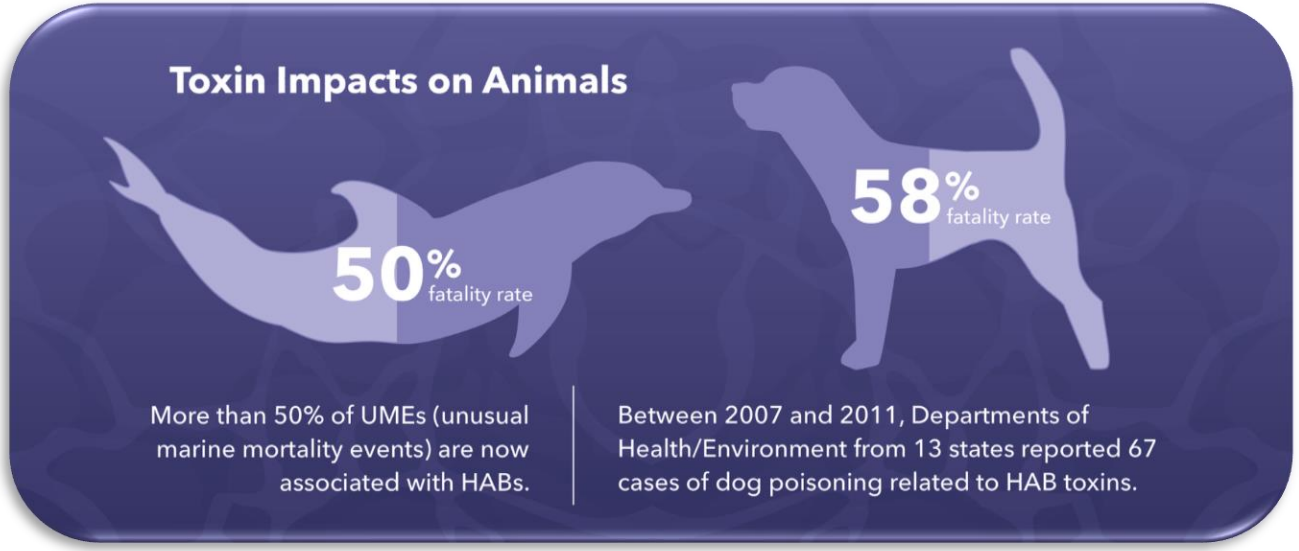
- P is the limiting nutrient for algae in most freshwaters.
- N further stimulates growth, toxin production.
- Eutrophic and hypereutrophic conditions lead to Harmful Algal Blooms (HABs).



https://datastream.org/en/guide/phosphorus, June 2022

HAB Impacts

- Environmental

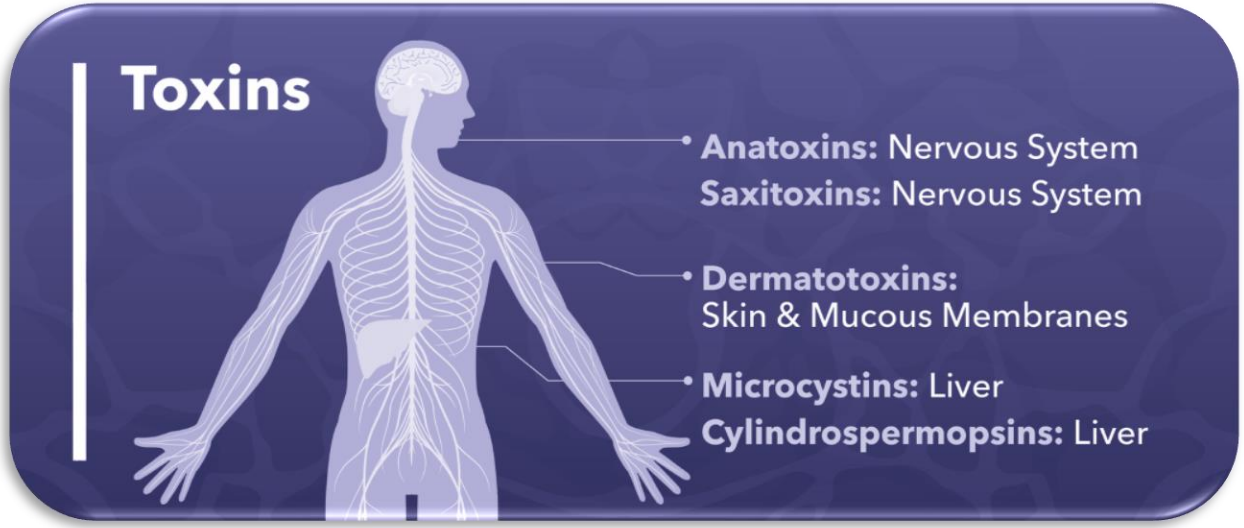


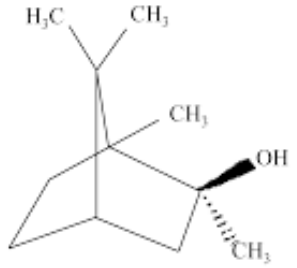
HAB Impacts

- Environmental
- Economic

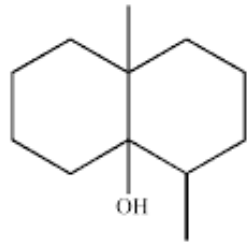
HAB Impacts

- Environmental
- Economic
- Drinking Water





2 Methylisobornel (MIB)

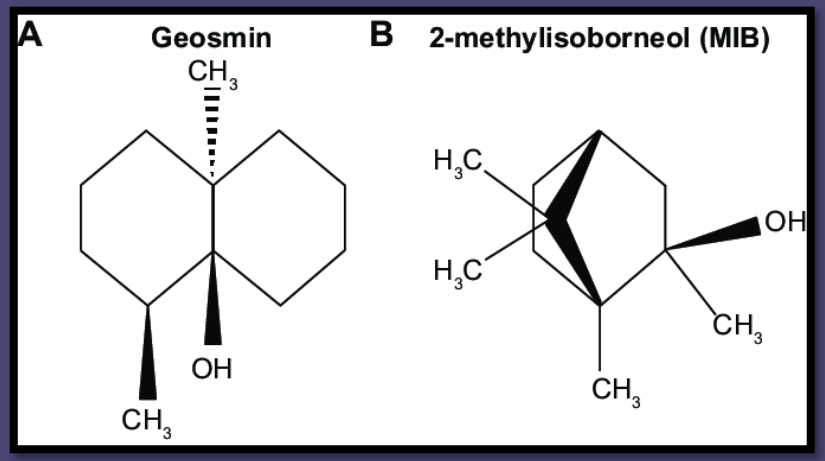


Geosmin

Geosmin & MIB in Drinking Water

What are Geosmin & MIB?

- Volatile organic compounds (VOCs)
- Non-toxic
- Made by microbes
  - Actinomycetes/Streptomycetes
  - Blue-green algae (cyanobacteria)



Geosmin & MIB in Drinking Water

What are Geosmin & MIB?

HABs and Geosmin/MIB

- Humans can detect as low as 5 ppt
- “Early warning sign” of an HAB
- Co-occurred with toxins ≥90% of the time in one study*

*Graham JL, et al. Environ Sci Technol. 2010;44(19):7361–7368. doi: 10.1021/es1008938.

Geosmin & MIB in Drinking Water

What are Geosmin & MIB?

HABs and Geosmin/MIB

Drinking Water Treatment

- Carbon
- Advanced Oxidation
- Breakthroughs common

AOP Article in WWD

Geosmin & MIB in Drinking Water

What are Geosmin & MIB?

HABs and Geosmin/MIB

Drinking Water Treatment

HAB Strategy for Geosmin/MIB

- Source Water Monitoring for HABs
- Mitigate Nutrients in Source Water
- Lab-based Monitoring for Geosmin/MIB

Part 1 Summary

- Eutrophication leads to HABs, and HABs lead to taste and odor in drinking water
- Geosmin/MIB are sensed by humans in the low ppt range
- Breakthroughs to finished water are common
- An HAB strategy should include ppt-range detection of Geosmin/MIB in finished water

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OI Analytical Geosmin/MIB Method via EPA Method 524.3

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OI Analytical Method

Detection Methods*

- Pre-conditioning → Extraction → Separation → Detection
- Sample Concentration (SPME, P&T…)
- GC/MS with selective ion monitoring

*Bristow, R.L., et al. TrAC. 2019;110:233-248 https://doi.org/10.1016/j.trac.2018.10.032.

OI Analytical Method

Detection Methods

EPA Method 524.3

- Purgeable Organics in finished drinking water by GC/MS
- Over 70 VOCs listed in method, *but neither geosmin or MIB*
- Every calibration curve point must meet a %recovery target

OI Analytical Method

Detection Methods

EPA Method 524.3

Our Goals

- ≤ 1 ppt in finished water is the target
- No salting/pre-treatment
- Extraction, concentration, sample intro all in one step

OI Analytical Method

Detection Methods

EPA Method 524.3

Our Goals

Our Approach

- 4100 Autosampler
- 4760 Eclipse P&T
- Agilent 7890A/5975C GC/MS

Geosmin/MIB Method

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OI Analytical Method: Calibration Acceptance & Validation

Compound	1 ppt		100 ppt	
	Std	%Rec	Std	%Rec
1,2-DCB-d4 (SS)	9.81	98.1	10.16	102
2-MIB	1.03	103	97.2	97.2
Geosmin	1.13	113	98.7	98.7

- 1,2-Dichlorobenzene-d4 is the surrogate standard, at 10 ppt
- Concentrations: 1, 2, 5, 10, 25, 50, 75 and 100 ppt
- Low level %Rec must be +/- 50%, all other levels must be +/- 30%

OI Analytical Method: Minimum Reporting Level (MRL)

Compound	Mean	Std. Dev.	HR_{PIR}	Upper PIR Limit (%)	Lower PIR Limit (%)
1,2-DCB-d4 (SS)	9.69	0.137	0.54	102	91.5
2-MIB	1.13	0.060	0.24	137	89.8
Geosmin	1.05	0.061	0.24	129	80.9

- HR – Half Range Prediction of Interval Results
- Upper PIR limit must be ≤150% recovery (true value is 1 ppt)
- Lower PIR Limit must be ≥50% recovery (true value is 1 ppt)

OI Analytical Method: Initial Demonstration of Capability

Compound	Precision (%RSD)	Accuracy (%)
1,2-DCB-d4 (SS)	1.92	98.4
2-MIB	3.73	109
Geosmin	3.46	106

- The 1,2-DCB-d4 surrogate standard was at 10 ppt
- Target compounds at 25 ppt
- %RSD must be ≤ 20% for precision for all
- Accuracy (%) must be ± 20%
  - (80-120% recovery)

OI Analytical Method: Considerations

- Method 524.3 is for *finished* drinking water
  - Haven't tested this method with source water

- Clean sample pathway, clean MS source
- Recommend minimum of three vials for sample testing
- Use same care you would use for all VOC sample collection

Part 2 Summary

- Met Our Primary Goals
  - ≤ 1 ppt MRL
  - No pre-treatment of samples
  - Extraction, concentration, sample intro in one, hands-free step

- Additional Benefits:
  - Common setup found in many environmental labs
  - Didn’t need salt
  - Used lower sample volume than some

Questions?

Contact Us:
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www.YSI.com

Learn More:
YSI.com/HABs

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