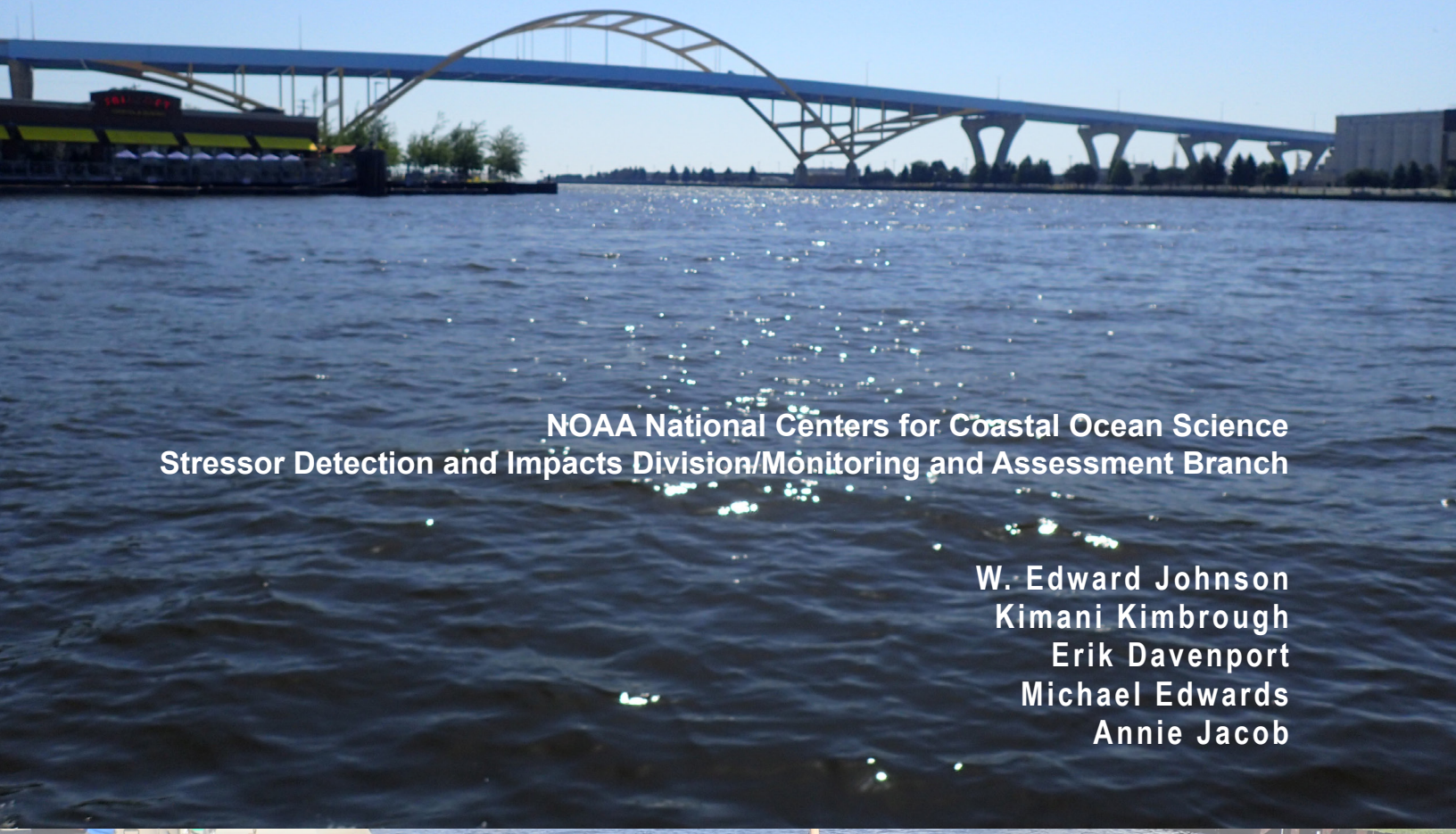
Great Lakes Restoration Initiative Interagency Agreement Final Report: NOAA Mussel Watch FY 17



NOAA National Centers for Coastal Ocean Science
Stressor Detection and Impacts Division/Monitoring and Assessment Branch

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Cover photos.
Top: Milwaukee Bridge, credit NOAA; bottom: NOAA boat, credit NOAA

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Project Information

A. Title: (2017-666)Integrated Emerging Contaminant Monitoring in the Great Lakes by NCCOS Mussel Watch Program.

B. Principal Investigator: Dr. Ed Johnson, NOAA, Great Lakes Chief Scientist,

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C. Project Period: 2017-2019

D. Funded Amount: $450,000

Project Approach for FY17 effort

- Participation in the Integrated Case Assessment Study at Milwaukee Bay Estuary along with several other federal partners.
- Multi-indicator approach to characterize contaminant exposure and effects.
- Use of caged mussels to asssess body burden and bioeffects.
- Use of mussel bioeffects techniques such as untargeted metabolomics, targeted metabolomics and DNA damage assays.
- Deployment of passive samplers to complement body burden data.

The National Oceanic and Atmospheric Administration's (NOAA) Mussel Watch Program (MWP) administered by the National Centers for Coastal Ocean Science (NCCOS) has used dreissenid mussels to monitor contaminants in the nearshore zones of the Great Lakes since 1992. MWP has 25 long-term monitoring sites in the Great Lakes from Duluth, MN on Lake Superior at the mouth of the St. Louis River to Cape Vincent, NY where Lake Ontario flows into the St. Lawrence River. Beginning in 2010, MWP expanded its monitoring activities in the Great Lakes under the Great Lakes Restoration Initiative (GLRI), Action Plan I (2010-2014), Focus Area "Toxic Substances and Areas of Concern". MWP added sites in all the U.S. Areas of Concern (AOC) and data from the basin-wide assessment conducted in 2009-2010 is summarized in Kimbrough et al., 2014.

In FY12 and beyond, MWP initiated a more focused effort in select AOCs and incorporated newer techniques and approaches such as the use of caged mussels, bivalve health metrics, etc. in order to better address contamination and remediation issues of specific AOCs. While the main focus of Phase 1 efforts was on providing data on legacy contaminants, MWP did opportunistic monitoring of contaminants of emerging concern (CEC) in mussels, which paved the way for participation in the GLRI Action Plan II efforts.

The GLRI Action Plan II (2015-2019) Focus Area "Toxic substances and Areas of Concern" calls for federal partners to "identify emerging contaminants and assess impacts on Great Lakes fish and wildlife." Accordingly a multi-agency team of federal scientists formulated a Strategic Plan, which sets forth two major goals:

(1) To characterize and evaluate the extent to which CECs threaten fish and wildlife populations relative to other chemical stressors present in the Great Lakes, and,
(2) To pilot and develop a short and long term state-of-the-art bioeffects surveillance program for the Great Lakes basin.

The major goals are to be achieved through three separate, but integrated study components including Surveillance Program (SP), Integrated Assessment Case Studies (IACS) and Priority Contaminant Mixtures (PCM). While the SP component is conducted at the Great Lakes basin-wide scale, the latter two are conducted at the individual watershed scale.

In FY17, MWP participated in the IACS study initiated by the federal team, the main objective of which is to identify associations between watershed attributes, such as land use and point sources, and CEC prevalence in watersheds. In 2017, the focus was on Polycyclic Aromatic Hydrocarbons (PAH) in the urban watershed of Milwaukee Estuary AOC to help identify CEC stress associated with urban land use.

For MWP, the specific objectives for the 2017 field season were:

Objective 1: Support the GLRI CEC Team's "Year of the PAH" study of the Milwaukee River.
Objective 2: Measure metabolomics and DNA damage response in mussels on a temporal scale.
Objective 3: Characterize chemical body burden in distinct zebra and quagga mussel samples.
Objective 4: Study the cage effect by deploying mussels at the harvest site.

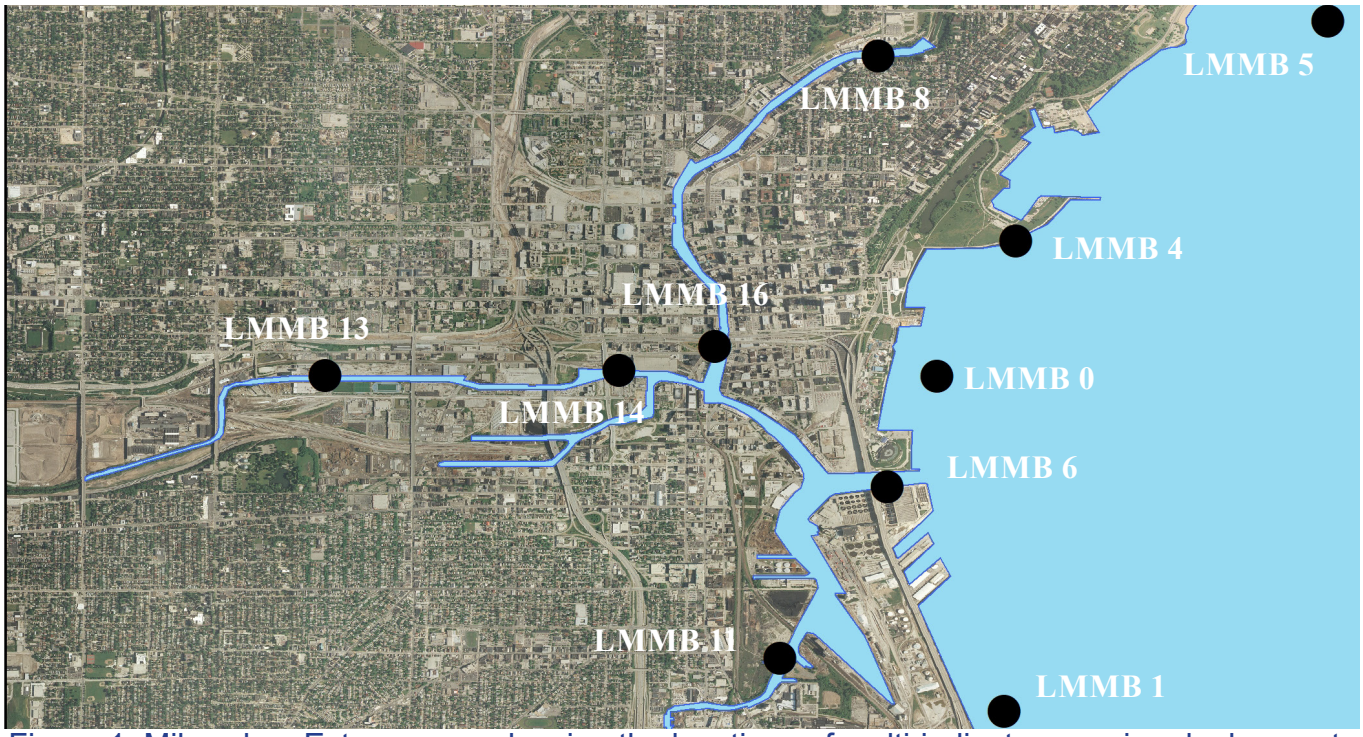

Figure 1. Milwaukee Estuary map showing the locations of multi-indicator mooring deployments.

Sites and sampling methods

The Milwaukee Estuary Area of Concern (AOC) was designed as one of the 31 US AOCs in 1987 by the International Joint Commission. The AOC includes the lower 3.1 miles of the Milwaukee River, the lower 3 miles of the Menomonee River down, the lower 2.5 miles of the Kinnickinnic River and the inner and outer Harbor and nearshore waters of Lake Michigan. In 2008, the boundary was expanded to include the following: Cedar Creek downstream from Bridge Road to confluence with Milwaukee River Milwaukee River and Lincoln Creek from confluence with Cedar Creek to North Avenue Dam;
Little Menomonee River from Brown Deer Road to confluence with Menomonee River, and Menomonee River downstream from confluence with Little Menomonee River to 35th Street.

Past industrial activities coupled with urbanization in subsequent years have caused pollution and impairments to the beneficial use of the waters within Milwaukee estuary AOC boundaries. Out of a total of 14 possible beneficial use impairments (BUIs), eleven were designated within the original AOC and four within the expanded boundary. Legacy contamination from polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and heavy metals are considered to be the predominant cause of the impairments. Further, water quality has been affected by pollution sources associated with land use from the entire drainage basins of the Milwaukee, Menomonee, and Kinnickinnic Rivers. Mussels were collected from the harbor site (LMMB 4) in Milwaukee Estuary, and from a site (LMMB 5) in Lake Michigan, Wisconsin. LMMB 4 is located in the outer harbor north of the harbor entrance slip channel adjacent to Juneau Park, whose land use has remained largely unchanged since the early 1900s. LMMB 5 is approximately 0.4 km offshore of a public swimming beach and about 4 km north of the entrance to Milwaukee harbor.

LMMB 0 is located in the outer harbor north of the harbor entrance ship channel. This site is adjacent to Lakeshore State Park, a 7-ha manmade island constructed mainly of dolomite limestone from a deep tunnel project of the Milwaukee Metropolitan Sewerage District in 1991.

The LMMB 1 site is located adjacent to the Milwaukee Harbor Confined Disposal Facility (dredge spoil) in the outer harbor south of the harbor entrance ship channel and approximately 4 km south of LMMB 4.

Field Component

- Collections of *in situ* mussels from 2 outer harbor sites and relocation in cages at 10 sites.
- Deployment of mooring with caged mussels, data loggers (temperature and dissolved oxygen) and passive samplers (POCIS or PEDs or both).
- Temporal sampling of mussels at 2, 5 and 53-55 days.Retrieval of moorings after 4 weeks.
- Processing of mussels for bivalve health metrics.

Laboratory analyses

- Mussel tissue chemistry (legacy organics and contaminants of emerging concern)
- POCIS chemistry
- Targeted and untargeted metabolomics
- DNA damage analyses

LMMB 6- Jones Island N end. About 35m E of the USGS water quality station building
LMMB 8- Upper Milwaukee River. E side of MKE river (N) about 20m S of Humboldt Ave bridge.
LMMB 11- Upper Menomonee River. N side of MM river near the intersection of Mt Vernon Ave and 21st Street. Mooring tied off near the second mooring bollard E of the first bollard.
LMMB 13- Upper Kinnickinnic River. W side of KK about halfway between the Becher St and Linlcon Ave bridges
LMMB 14- Lower Kinnickinnic River. W side of KK about 25m downstream of the turning basin w/in 3m of the concrete ship wall.
LMMB 15- Lower Menomonee River. N side of MM river about 40m E of the 6th Street bridge.
LMMB 16- Lower Milwaukee River. W side of MKE river about halfway between the Clybourn and St Paul St bridges.

On 6 June, 2017 divers harvested mussels from LMMB 4 and LMMB 5 using a metal paint scraper from rocks and the sheet pile wall on the north side of the island. The mussels were placed into a separate nylon mesh dive bag; the bags were gently shaken underwater to remove debris. Upon surfacing, the harvested mussels in individual mesh bags were immediately placed in a 26 L cooler with aerated site water until deployment. A subsample comprising 200-400 harvested in situ mussels were taken to analyze for baseline tissue chemistry and metabolomics before caging. The cages were deployed for 4 weeks and retrieved between July 31- Aug1, 2017. Approximately 300-500 mussels were placed inside each cage, which consisted of torpedo shaped metal minnow traps that were tightly secured with cable ties. The cages were deployed approximately 0.5 m above the river bottom and secured to shore with #2 zinc plated double loop steel chain. One composite sample comprising 200-400 harvested mussels was taken to analyze for tissue chemistry and metabolomics. Temporal samples for metabolomics and DNA damage were taken at the appropriate times.

Sample Processing

Tissue Chemistry

One composite sample comprising 200-400 mussels was taken from each cage to analyze for tissue chemistry. Mussel samples were rinsed with site water to remove debris, placed in freezer bags, packed on water ice and shipped to laboratories within two days.

DNA damage

Two FEP bags each containing 15-20 mussels were prepared for DNA damage analyses. Each FEP-bag received a unique number. The two FEP bags from each site are placed into one labeled cloth bag, and flash-frozen by placing in a cryogenic dry vapor shipper (-196 degrees C).

Figure 2. Multi-indicator mooring apparatus with mussels, polar organic chemical integrative sampler (POCIS) and data loggers.

Metabolomics
Each metabolomics sample consists of 24 individually FEP-bagged mussel plus two additional composite bags containing 15-20 extra mussels for archive. Prior to bagging the 24 individual mussels, digital calipers are used to measure their longest length and recorded on the data sheet. The composited bags of bivalves are not measured. Each FEP-bag received a unique number. The entire group of 26 FEP bags from each site are placed into one labeled cloth bag, and flash-frozen by placing in a cryogenic dry vapor shipper (-196 degrees C).

Analysis Methods

Tissue Chemistry
Tissue samples were analyzed by TDI-Brooks International, Inc., Texas for PAH and legacy organics, and by SGS AXYS Analytical Services Ltd. in British Columbia, Canada for CECs.

Protocols for analytical methods for organic contaminants including PAHs in mussel tissue are detailed Kimbrough et al. (2006).

The SGX AXYS methods are proprietary and confidential. Hence, in this document, we will refer to the name of the method and revision number provided by SGS AXYS. Note that MLA-075 and MLA-035 are based on EPA methods 1694 and 1699 respectively (U.S. EPA 2007a, b).

DNA damage
DNA damage analyses were conducted by scientists at National Institute for Standards and Technology and methods are described in detail in Jaruga et al., 2017. Briefly, mussels were thawed on ice, then washed with ice-cold deionized water. Mussel tissues (≈100 mg) separated from shells with a scalpel were processed according to the product manual of E.Z.N.A. Mollusc DNA Kit, Omega Bio-tek (Norcross, Georgia) with modification involving homogenization with Bullet Blender Storm

24 high-throughput bead-mill homogenizer (Next Advance, Averill Park, New York). Tissues were placed in the 1.5 mL Rhino Screw cap tubes (Next Advance) kept on ice, containing 350 mL of ML1 Buffer from the kit and three 2 mm zirconium oxide beads. Tubes were transferred into the Bullet Blender kept in the refrigerator at 4 0C and processed 2 x 30 s at speed 12 with 30 s break between runs. Subsequently 25 mL of Proteinase K from the kit was added and samples were incubated for 2 h at 60 0C. Then, subsequent steps of the Mollusc DNA Kit protocol were applied. For the final DNA elution, two portions of 100 mL of sterile high-performance liquid chromatography grade water (Sigma- Aldrich, St. Louis, Missouri) warmed to 70ºC were used. The UV absorbance spectrum of each DNA sample was recorded by absorption spectrophotometry between the wavelengths of 200 nm and 350 nm to ascertain the quality of DNA and to measure the DNA concentration at 260 nm (absorbance of 1 = 50 mg of DNA per mL).

Aliquots (50 mg) of DNA samples were dried in 1.5 mL deoxyribonuclease-free Eppendorf tubes in a SpeedVac under vacuum and then kept at –80 0C for further analysis. Gas chromatography-tandem mass spectrometry with isotope-dilution was used to identify and quantify modified DNA bases and 8,5' -cyclopurine-2'-deoxynucleosides. Six modified DNA bases (5 hydroxy-5-methylhydantoin (5-OH-5-MeHyd), thymine glycol (ThyGly), 5,6-dihydroyuracil (5,6-diOH-Ura), 4,6-diamino-5-formamidopyrimidine (FapyAde), 2,6-diamino-4-hydroxy-5-formamidopyrimidine (FapyGua) and 8-hydroxyguanine (8-OH-Gua)) and three 8,5′-cyclopurine-2′-deoxynucleosides ((5'S)-8,5′-cyclo-2′-deoxyadenosine (S-cdA), (5'R)-8,5′cyclo-2′-deoxyguanosine (R-cdG) and (5'S)-cyclo-2′-deoxyguanosine (S-cdG)) were identified and quantified in the mussels' tissue samples.

Targeted Metabolomics

AXYS Analytical Services Ltd. provided the analytical services for targeted metabolomics on dreissenid mussels .The methods are proprietary and confidential. Mussels were extracted from their shells, exterior water removed and the whole body homogenized. Extraction with methanol and chloroform used a bead blender and a portion of the extract was subject to further workup and analysis for the six lists of metabolites:

· MABA: Amino acids and Biogenic amines (43 metabolites)
· MFAAHEX: Fatty acids (18 metabolites) and Hexose (1 metabolite)
· MLIP: Phospholipids and Acylcarnitines (144 metabolites),
· MNRG: Metabolites associated with energy pathways (17 metabolites).

Untargeted Metabolomics

Mussels samples were anlaysed by scientists at National Institute of Standards and Technology using nuclear magnetic resonance spectroscopy. Methods are described in detail in Watanabe et al., 2015.

SAMPLES COLLECTED	DATE OF COLLECTION	LOCATIONS	DATA	LABS
In situ Mussels	6/6-6/8 T0 days	LMMB 0 LMMB 1 LMMB 4 LMMB 5	Chemistry DNA damage Metabolomics	TDI/AXYS NIST AXYS/NIST
Moorings (mussels, POCIS, SPMD, data logger)	6/8-6/10 - T 2 days 6/12 - T 5 days 7/31- 8/1 - T 53-55 days	LMMB 4 LMMB 5 LMMB 6 LMMB 8 LMMB 11 LMMB 13	Chemistry DNA damage Metabolomics	TDI/AXYS NIST AXYS/NIST
Caged Mussels Only	7/31-8/1 - T53-55 days	LMMB 14 LMMB 15 LMMB 16	Chemistry	TDI/AXYS

Table 1: List of samples collected, dates of collection, locations, data types and laboratories that analyzed the samples.

Table 2. Chemical list A) PAHs

Acenaphthene	Acenaphthylene	Anthracene	Benz[a]anthracene
Benzo[a]pyrene	Benzo[e]pyrene	Benzo[b]fluoranthene	Benzo[k]fluoranthene
Benzo[g,h,i]perylene	Benzothiophene	C1-Benzothiophene	C1-Chrysenes
C1-Dibenzothiophenes	C1-Fluorenes	C1-Fluoranthenes_Pyrene	C1-Naphthobenzo-thiophene
C1-Naphthalenes	C1-Phenanthrenes_ Anthracenes	C2-Dibenzothiophenes	C2-Fluorenes
C2-Fluoranthenes_Pyrenes	C2-Naphthobenzo-thiophe	C2-Naphthobenzothiophene	C2-Naphthalenes
C2-Phenanthrenes_Anthracenes	C3-Benzothiophene	C3-Chrysenes	C3-Dibenzothio-phenes
C3-Fluorenes	C3-Fluoranthenes_ Pyrenes	C3-Naphthobenzothiophene	C3-Naphthalenes
C4-Chrysenes	C4-Naphthalenes	C4-Phenanthrenes_Anthra-cenes	Chrysene
Dibenzo[a,h]anthracene	Dibenzofuran	Dibenzothiophene	Fluoranthene
Fluorene	Indeno[1,2,3-c,d] pyrene	Naphthobenzothiophene	Naphthalene
Phenanthrene	Pyrene		

B) Pesticides by GC/HRMS (MLA-035.R07.02).

2,4'-DDD	Chlorothalonil	Fenitrothion	Nonachlor, cis-
2,4'-DDE	Chlorpyriphos	Flufenacet	Nonachlor, trans-
2,4'-DDT	Chlorpyriphos-Oxon	Flutriafol	Octachlorostyrene
4,4'-DDD	Chlorpyriphos-Oxon	Fonofos	Parathion-Ethyl
4,4'-DDE	Cyanazine	HCH, alpha	Parathion-Methyl
4,4'-DDT	Dacthal	HCH, beta	Pendimethalin
Alachlor	Desethylatrazine	HCH, delta	Perthane
Aldrin	Diazinon	HCH, gamma	Phorate
alpha-Endosulfan	Diazinon-Oxon	Heptachlor	Phosmet
Ametryn	Dieldrin	Heptachlor Epoxide	Pirimiphos-Methyl
Atrazine	Dimethenamid	Hexachlorobenzene	Quintozene
Azinphos-Methyl	Dimethoate	Hexazinone	Simazine
beta-Endosulfan	Disulfoton	Linuron	Tebuconazol
Butralin	Disulfoton Sulfone	Malathion	Tecnazene
Butylate	Endosulfan Sulfate	Methoprene	Terbufos
Captan	Endrin	Methoxychlor	Triallate
Chlordane, alpha (cis)	Endrin Ketone	Metolachlor	Trifluralin
Chlordane, gamma (trans)	Ethalfluralin	Metribuzin	
Chlordane, oxy-	Ethion	Mirex	

C) Octylphenol, Nonylphenol & Nonylphenol Ethoxylates (MLA-080.R02.04).

4-NP	4n-OP
NP1EO	NP2EO

D) Pharmaceuticals and personal care products (MLA-075.R06.01).

1,7-Dimethylxanthine	Citalopram	Glipizide	Progesterone
10-Hydroxy-amitriptyline	Clarithromycin	Glyburide	Promethazine
17 alpha-Dihydroequilin	Clinafloxacin	Hydrochlorothiazide	Propoxyphene
17 alpha-Estradiol	Clonidine	Hydrocodone	Propranolol
17 alpha-Ethinyl-Estradiol	Clotrimazole	Hydrocortisone	Ranitidine
17 beta-Estradiol	Cloxacillin	Ibuprofen	Rosuvastatin
2-Hydroxy-ibuprofen	Cocaine	Iopamidol	Roxithromycin
4-Epianhydrochlortetracycline [EACTC]	Codeine	Isochlortetracycline [ICTC]	Sarafloxacin
4-Epianhydrotetracycline [EATC]	Colchicine	Lincomycin	Sertraline
4-Epichlortetracycline [ECTC]	Cotinine	Lomefloxacin	Simvastatin
4-Epioxytetracycline [EOTC]	Cyclophosphamide	Medroxyprogesterone Acetate	Sulfachloropyridazine
4-Epitetracycline [ETC]	Daunorubicin	Melphalan	Sulfadiazine
Acetaminophen	DEET	Meprobamate	Sulfadimethoxine
Albuterol	Dehydronifedipine	Mestranol	Sulfamerazine
Allyl Trenbolone	Demeclocycline	Metformin	Sulfamethazine
Alprazolam	Desmethyldiltiazem	Methylprednisolone	Sulfamethizole
Amitriptyline	Desogestrel	Metoprolol	Sulfamethoxazole
Amlodipine	Diatrizoic acid	Metronidazole	Sulfanilamide
Amphetamine	Diazepam	Miconazole	Sulfathiazole
Amsacrine	Digoxigenin	Minocycline	Tamoxifen
Androstenedione	Digoxin	Moxifloxacin	Teniposide
Androsterone	Diltiazem	Naproxen	Testosterone
Anhydrochlortetracycline [ACTC]	Diphenhydramine	Norethindrone	Tetracycline [TC]
Anhydrotetracycline [ATC]	Doxorubicin	Norfloxacin	Theophylline
Atenolol	Doxycycline	Norfluoxetine	Thiabendazole
Atorvastatin	Drospirenone	Norgestimate	Trenbolone
Azathioprine	Enalapril	Norgestrel	Trenbolone acetate
Azithromycin	Enrofloxacin	Norverapamil	Triamterene
Benzoylecgonine	Equilenin	Ofloxacin	Triclocarban
Benztropine	Equilin	Ormetoprim	Triclosan
Betamethasone	Erythromycin-H2O	Oxacillin	Trimethoprim
Bisphenol A	Estriol	Oxazepam	Tylosin
Busulfan	Estrone	Oxolinic Acid	Valsartan
Caffeine	Etoposide	Oxycodone	Venlafaxine
Carbadox	Flumequine	Oxytetracycline [OTC]	Verapamil
Carbamazepine	Fluocinonide	Paroxetine	Virginiamycin M1
Cefotaxime	Fluoxetine	Penicillin G	Warfarin
Chlortetracycline [CTC]	Fluticasone propio-nate	Penicillin V	Zidovudine
Cimetidine	Furosemide	Prednisolone	
Ciprofloxacin	Gemfibrozil	Prednisone	

FY17 study was undertaken with the support and collaboration with NOAA office listed below:

- NOAA-GLERL- boat and diving support for SCUBA diving operations, and mooring deployment and recovery; GLERL ecologist provided scientific and logistical support.

Two separate field mission were carried out between June - August, 2017. Both missions included 6 crew members for a total of 75 person-days.

Event	Date
Annex 3 activities update EPA-NOAA brief	Q1
Regarding the mussel tissue data for evaluation using toxCast in the toxEval software &CompTox (corsi emails)	Q1
Participated/supported discussion with Ohio Sea Grant setup by EPA GLNPO	Q2
NOAA GLRI workshop	Q2
IAGLR session on “Discoveries, trends, and implications of chemicals in the Great Lakes” at Detroit	Q3
Field planning and logistics meeting for Milwaukee IACS	Q3
GLRI CEC monthly call	Q1
GLRI CEC session at SETAC in Minneapolis	Q2
GLRI F2F Meeting in Athens NOAA DIVER web tool demo	Q2
GLRI-CEC Phase 1 report	Q2
IACS coordination call	Q2

Table 3: List of significant events during the project period.

What data is there

1. Mussel chemistry
2. Targeted metabolomics for mussels
3. Non-targeted metabolomics for mussels
4. DNA damage assay
5. POCIS chemistry
6. Dissolved oxygen and temperature data

How and when it was collected

1. Mussels for cage depolyment were collected by scuba divers.
2. Mussel collections, mooring deployment and recovery were conducted between June- August 2017.

Purpose of data

1. Contaminant characterization of Milwaukee Estuary using bivalves and passive sampler chemistry.

2. Assessment of bivalve health indicators (metabolomics and DNA damage assays) to inform on bioeffects of contaminants.

3. Temporal study of targeted metabolomics of mussels and its application as a biomonitoing tool.

Where it resides

1. Chemistry data resides on a NOAA web page as downloadable csv files.

2. Chemistry data will permanently reside as part of NOAA's Query Manager database and be accessible through ERMA and DIVER.

3. Bivalve health indicator data reside on local drives and will be made available to public eventually after the publication of journal articles.

Table 4. Compilation of data collected are listed and their associated filename found on the website http://www.coastalscience.noaa.gov/projects/detail?key=179

Description	Filename
Great Lakes Sampling Sites	GL_Sampling sites_MWP_2017
Tissue Chemistry- Contaminants of Emerging Concern	GL_CEC_AXYS_2017.xls
Tissue Chemistry- PAH	GL_PAH_TDI_2017.xls
Tissue Chemistry- Legacy Organics	GL_Organics_TDI_2017.xls
Passive Sampler (POCIS) Chemistry	GL_POCIS_AXYS_2017.xls
Water Quality Data	GL_Water quality_2017.xls

Analysis of the data

Mussel Data Analysis

Mussel and POCIS chemistry data were manipulated using R-code for both data formatting and analysis. In this report, we present the data in heat maps and bar charts. In-depth analysis of the data will be conducted in associated technical memorandums and manuscripts.

List of Project Outputs

Following reports are in development:

- Polycyclic aromatic hydrocarbon characterization and prediction in coastal sediments using regression modeling and machine learning
- Characterization of Polycyclic Aromatic Hydrocarbon Characterization in the Great Lakes Basin using Driessenid Mussels
- Milwaukee Estuary Polycyclic Aromatic Hydrocarbon Summary
- Great Lakes Mussel Watch (2013 - 2018) Pharmaceuticals and Personal Care Products Report.

Conclusions and Recommendations

Specific conclusions/recommendations will be made once the entire data is analyzed.

Contamination monitoring and assessment efforts at Milwaukee Estuary have shown that relocating mussels in cages to targeted sites within a river-harbor is an efficient and practical way to assess environmental condition.

Jaruga, P., E. Coskun, Kimbrough, K.L, A. Jacob,W.E. Johnson and M. Dizdaroglu. 2017. Biomarkers of oxidatively induced DNA damage in dreissenid mussels: A genotoxicity assessment tool for the Laurentian Great Lakes. Environmental Toxicology32:2144–2153.

Kimbrough, K. L., and G. G. Lauenstein (eds.). 2006. Major and trace element analytical methods of the National Status and Trends Program 2000-2006. NOAA Technical Memorandum NOS NCCOS 29 Silver Spring, MD 19pp.

Kimbrough, K. L., G. G. Lauenstein, and W. E. Johnson (eds.). 2006. Organic contaminant analytical methods of the National Status and Trends Program: Update 2000-2006. NOAA Technical Memorandum NOS NCCOS 30 Silver Spring, MD 65pp.

Kimbrough, K.L., W. E. Johnson, A. Jacob, M. Edwards, E. Davenport, G. Lauenstein, T. Nalepa,M. Fulton and A. Pait. 2014. Mussel Watch Great Lakes Contaminant Monitoring and Assessment:Phase 1. Silver Spring, MD. NOAA Technical Memorandum NOS NCCOS 180, 113 pp.

U.S. Environmental Protection Agency, 2007, Method 1694: Pharmaceuticals and personal care products in water, soil,sediment, and biosolids by HPLC/MS/MS: USEPA, Washington, DC, EPA-821-R-08-008, 77 p.

U.S. Environmental Protection Agency, 2007b, Method 1699:Pesticides in water, soil, sediment, biosolids, and tissue by HRGC/HRMS: USEPA, Washington, DC, EPA-821-R-08-001,96 p.

Watanabe, M., K. A. Meyer, T. M. Jackson, T. B. Schock, W. E. Johnson, and D. W. Bearden. 2015. Application of NMR-Based Metabolomics for Environmental Assessment in the Great Lakes Using Zebra Mussel (*Dreissena Polymorpha*). Metabolomics. http://dx.doi.org/10.1007/s11306-015-0789-4. https://repository.library.noaa.gov/view/noaa/13686

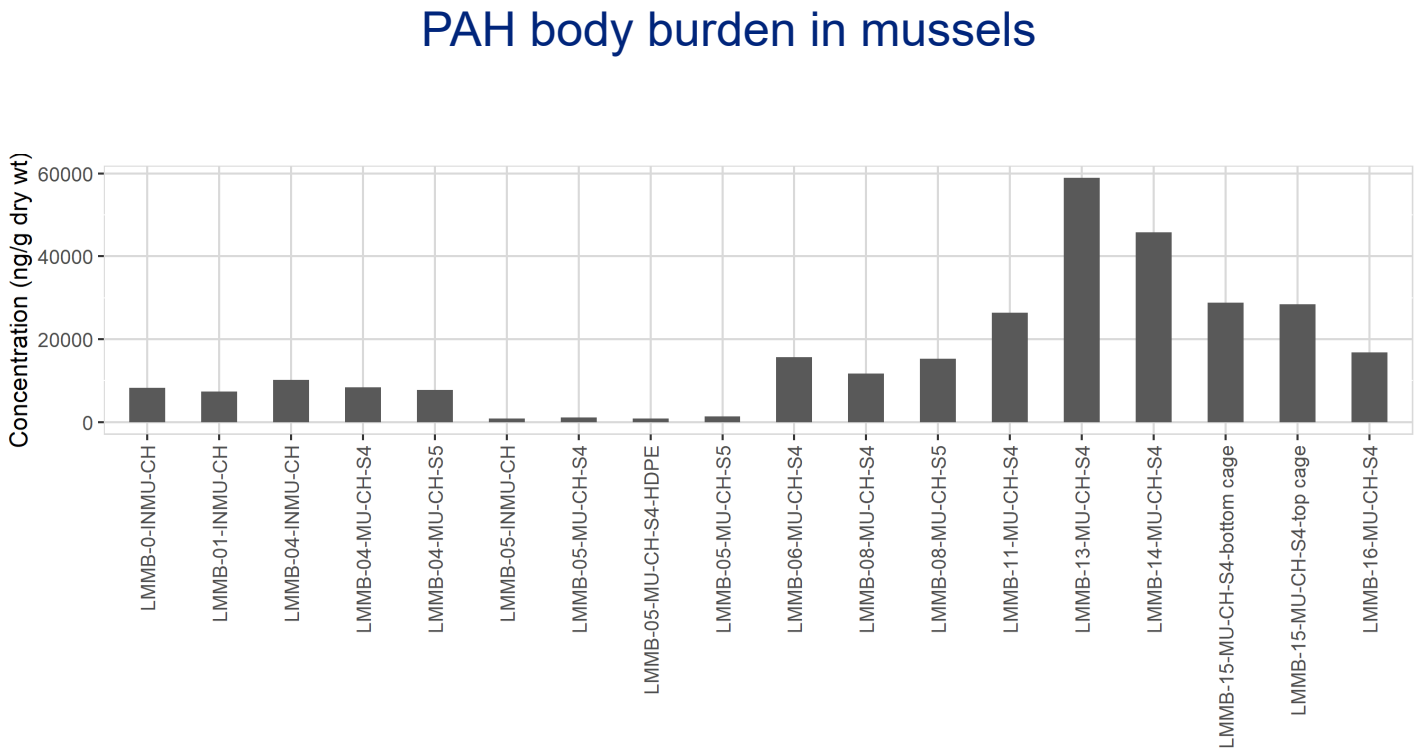

Figure 3: Total Concentration of PAH in mussel tissue samples collected from Milwaukee Estuary sites.

Percent detection of CECs and legacy pesticides in mussel tissue

COMPOUND	PERCENT DETECTION
Amphetamine	5.3
Chlorpyriphos	5.3
Quintozene	5.3
Cocaine	10.5
Fluoxetine	10.5
Hexachlorobenzene	10.5
Sulfadiazine	10.5
Chlordane, oxy-	21.1
Citalopram	21.1
Iopamidol	21.1
Trifluralin	23.5
alpha-Endosulphan	26.3
4,4'-DDT	26.3
Propranolol	31.6
Azithromycin	36.8
Verapamil	36.8
2,4'-DDE	36.8
Diphenhydramine	42.1
Heptachlor Epoxide	42.1
Triclocarban	47.4
Aldrin	52.6
Sertraline	73.7
2,4'-DDD	73.7
Amitriptyline	78.9
Chlordane, gamma (trans)	78.9
Etoposide	78.9
4-NP	78.9
Chlordane, alpha (cis)	84.2
Nonachlor, cis-	84.2
4,4'-DDD	89.5
Nonachlor, trans-	94.7
Dieldrin	100.0
4,4'-DDE	100.0
NP1EO	100.0
NP2EO	100.0

able 5: Percent detection of compounds detected above three times the detection limit in mussel tissue samples from Milwaukee Estuary in 2017.

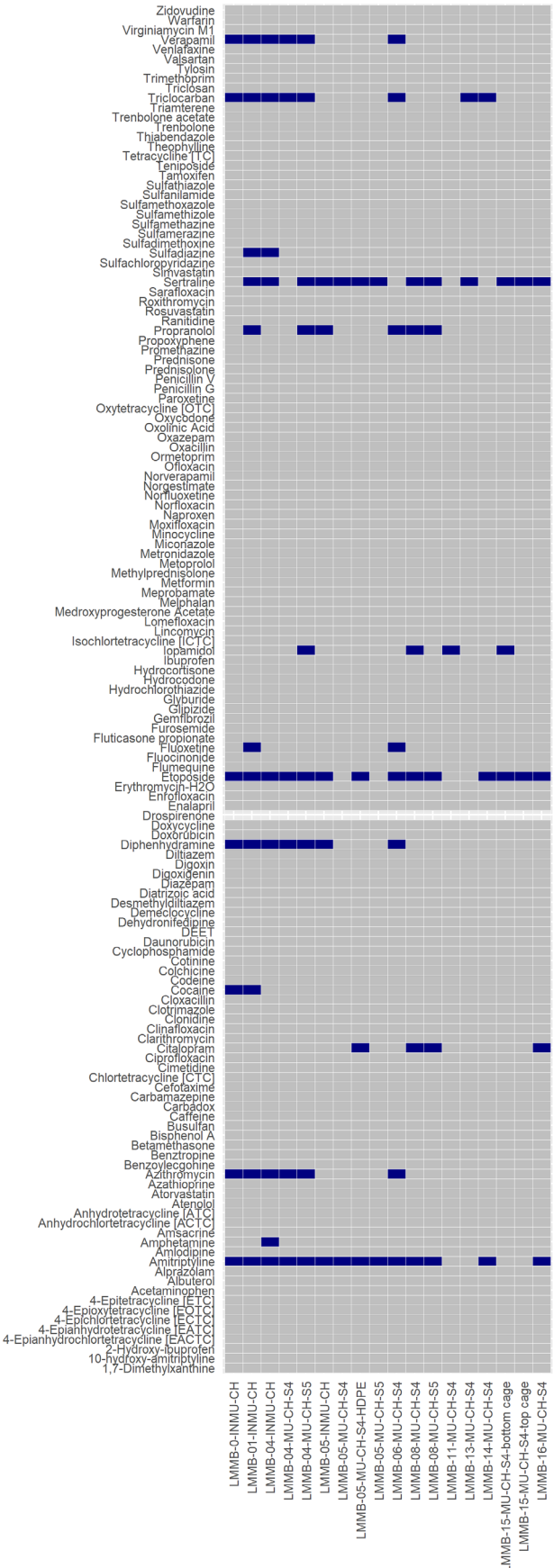

Figure 4: Heatmap of pharmaceuticals and personal care products detected above three times the detection limit in mussel tissue samples from Milwaukee Estuary in 2017.

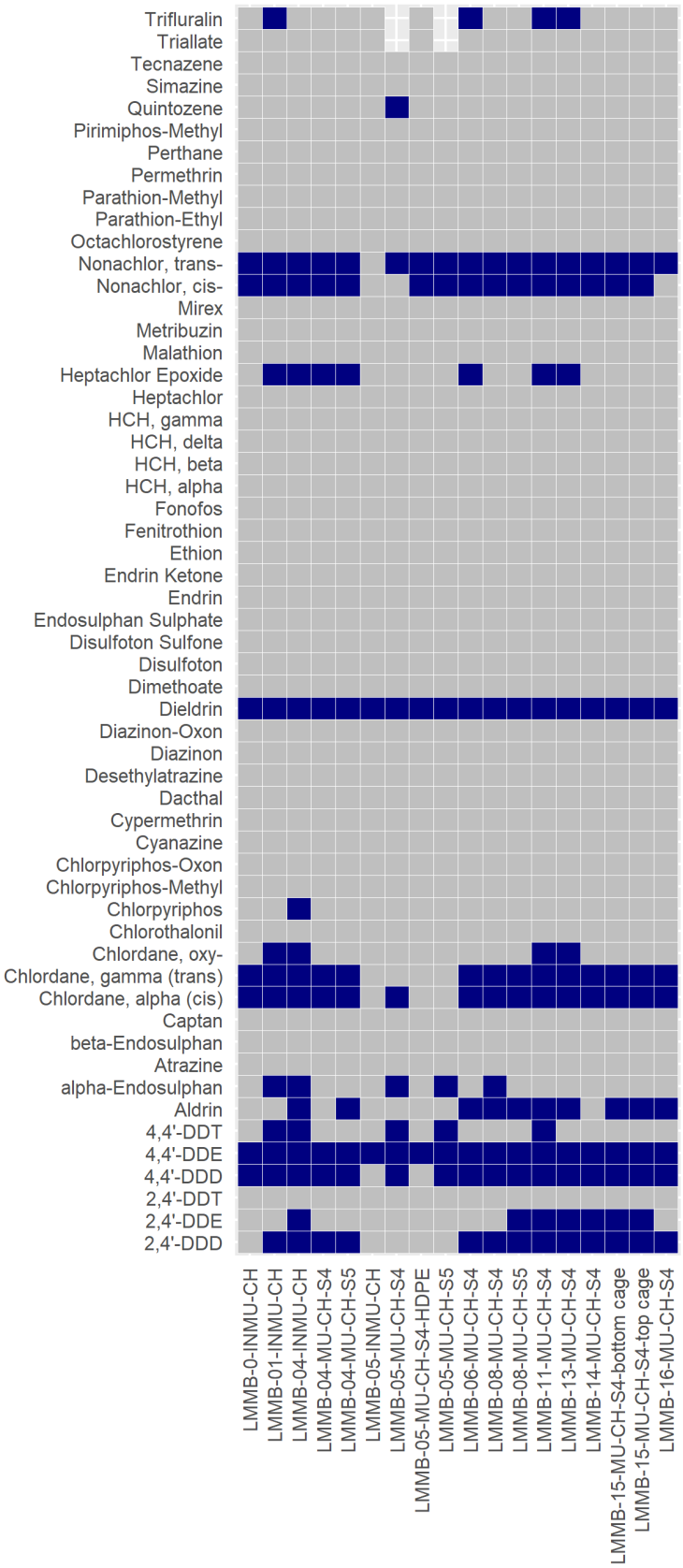

Figure 5: Heatmap of current use and legacy pesticides detected above three times the detection limit in mussel tissue samples from Milwaukee Estuary in 2017.


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