Screening for Organic Pollutants by Gas Chromatography-Mass Spectrometry (GC-MS) in Surface Water and Sediments at Bukana Malaki River, Naic, Cavite

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ABSTRACT

Screening, as an analytical method, is a fast and effective approach in obtaining a holistic picture of organic pollution in a site-specific environment. In this study, surface water and sediment samples from the selected locations along the Bukana Malaki River, in Naic, Cavite which is the tail end of the Labac-Alemang River Watershed were screened to (1) gather qualitative data and gain insights into source identification, and (2) provide quantitative baseline data of organic pollutants for future studies. Using Gas Chromatography - Mass Spectrometry (GC-MS) to screen for organic pollutants in surface water samples, 11 different organic compounds were identified at the upstream, 29 at the midstream and 28 at the downstream. Eight (8) of these compounds have concentrations greater than 10 percent by mass. The downstream sampling station contains the pollutant with the highest concentration: n-hexadecanoic acid methyl ester which is 56.48 percent by mass. Again, using GC-MS for sediments, 41 different organic compounds were identified at the upstream, 13 at the midstream, and 8 at the downstream. Ten (10) of these compounds have concentrations greater than 10 percent by mass. The upstream sampling station contains the pollutant with the highest concentration: acetonyldimethylcarbinol with 94.11 percent by mass. The organic compounds found in the water and sediment samples are considered hazardous with a few exceptions. The hazardous effects include eye, skin, digestive tract and respiratory tract irritations. Some may even cause organ toxicity. Even others are found to pose danger to the environment. As to the sources, these organic compounds are produced from the use of household materials and industrial products released to the environment. The results demonstrated the pollution characteristics and occurrence levels of organic pollutants in the surface waters and sediments at Bukana Malaki River in Naic, Cavite.

Keywords: *Acetonyldimethylcarbinol, Bukana Malaki River, GC-MS, n-Hexadecanoic acid methyl ester, organic pollutants, screening, surface water, sediments*

INTRODUCTION

Essentially, environmental chemistry of organic pollutants is a branch of chemistry that has grown in response to the practical requirement of governments for a clear understanding of the behaviour and effects of chemicals in the environment. In many situations, control measures may have adverse economic and social effects on human communities and, at the same time, may not have beneficial environmental outcomes. Fully effective measures for the management of chemicals require detailed understanding of such factors as: the distribution of chemicals in the environment, their persistence and degradation, and toxicity to humans and other biota. In addition, control and management requires a sound knowledge of methods for the treatment and reduction of the discharge of organic pollutants to the environment (Connell, Wu, Richardson & Lam 2013).

Organic pollution occurs when large quantities of organic compounds, which act as substrates for microorganisms, are released into water courses. During the decomposition process, the dissolved oxygen in the receiving water may be used up at a greater rate than it can be replenished, causing oxygen depletion and having severe consequences for the stream biota. Organic effluents also frequently contain large quantities of suspended solids which reduce the light available to photosynthetic organisms and, on settling out, alter the characteristics of the riverbed, rendering it an unsuitable habitat for many invertebrates. Toxic ammonia is often present.

Organic pollutants consist of proteins, carbohydrates, fats and nucleic acids in a multiplicity of combinations. Raw sewage is 99.9 percent water, and of the 0.1 percent solids, 70 percent is organic (65 percent proteins, 25 percent carbohydrates, 10 percent fats). Organic wastes from people and their animals may also be rich in disease-causing (pathogenic) organism (Lenntech,1998).

According to Connell, et al. (2013), a broad range of organic substances is produced by human society and appears in discharges such as sewage, storm water, and industrial discharges. These discharges can represent major sources of the pollutant substance. For example, the concentration of organic pollutants in sewage is low but the volume is large making it a major source of many pollutants. A similar situation applies with storm water where these substances often originate from discharges from motor vehicles on to road surfaces and are subsequently swept into waterways by storm run-off. Motor vehicles are major sources of petroleum hydrocarbons, polycyclic aromatic hydrocarbons and dioxins which are often discharged to the atmosphere in particulate form. These particulates are deposited close to busy roadways leading to contamination of urban soils and potential human exposure. Urban and industrial wastes are often, either currently or in the past, disposed directly into pits dug into the ground which leads to contamination of the soil and in some cases the adjacent ground water. Outside urban areas, agricultural activities are the major sources of pollutants. The growing of crops often involves the release of pesticides into the environment, which can result in the contamination of waterways and soils, as well as the urban environment. Perhaps, the most spectacular example of contamination of the environment is due to the accidental spillage of petroleum. Over the years, many disasters of this kind have occurred releasing tens of thousands of tonnes of petroleum into the aquatic environment.

Results of the previous study of Callao, et al., (2018) revealed that during the dry and wet seasons, the concentrations of PCBs were both 0. 0003 mg/L for water and 0.05 mg/kg for sediments. The physical condition and smell of the river aroused the interest of the researchers to conduct this study. Water and sediments sampling was conducted on March 23, 2018. The researchers were able to observe that the physical condition of the sampling locations showed evidences of water pollution since there were numerous floating materials on the surface of the river as well as suspended solids. In addition, the surface of the water was found thick and oily and had a foul odor. The sediments also contained solid wastes (mostly plastics), black in color and had a very pungent smell. Thus, this study tried to identify the organic pollutants that are present in water and sediments. Specifically, it aimed to (1) gather qualitative data and gain insights into source identification and (2) provide quantitative baseline data of organic pollutants for future studies.

METHODOLOGY

Study Area

The municipality of Naic, Cavite is located on the western part of the province along the shorelines of Manila Bay. Trece Martires City and Tanza bound it to the east. Situated beyond the southern portion of Naic is Indang and the

western boundary is shared with Ternate and Maragondon. The coordinates of Naic are 14°32 latitudes and 120°768 longitudes.

Labac River Watershed is one of the six major rivers in the province of Cavite. The river is divided into: the western Labac River and the eastern Labac River. It traversed the six municipalities/cities, namely: Tagaytay, Mendez, Indang, Trece, Tanza and Naic. The elevation of the watershed is divided into three: Tagaytay and Mendez for Upland, Indang for Midland, and Trece Martires, Tanza and Naic for Lowland. Elevation of 0 to 162 meters was represented as lowland, 162.1 to 383 meters as midland and 383.1 to 692 meters above sea level. The lowest elevation is 2 meters above sea level and located at Naic (Obstaculo, 2015).

Barangay Bukana Malaki in Naic, Cavite, the tail end of the river, before it drains into the Manila Bay, was chosen as the sampling station as it has the lowest elevation. It is expected that the Labac River will drain towards Naic.

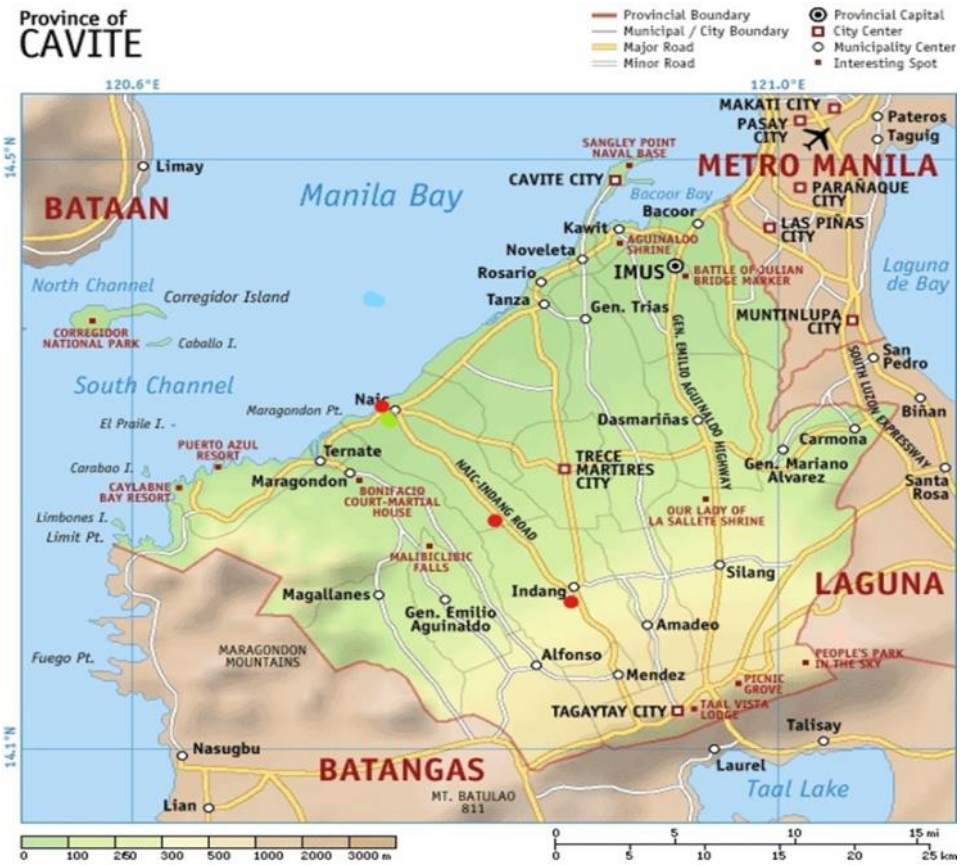

Figure 1. Map of Cavite[1]

Legend: ● -sampling sites

[1] https://upload.wikimedia.org/wikipedia/commons/thumb/1/18/Ph_map_cavite.png/330px-Ph_map_cavite.png

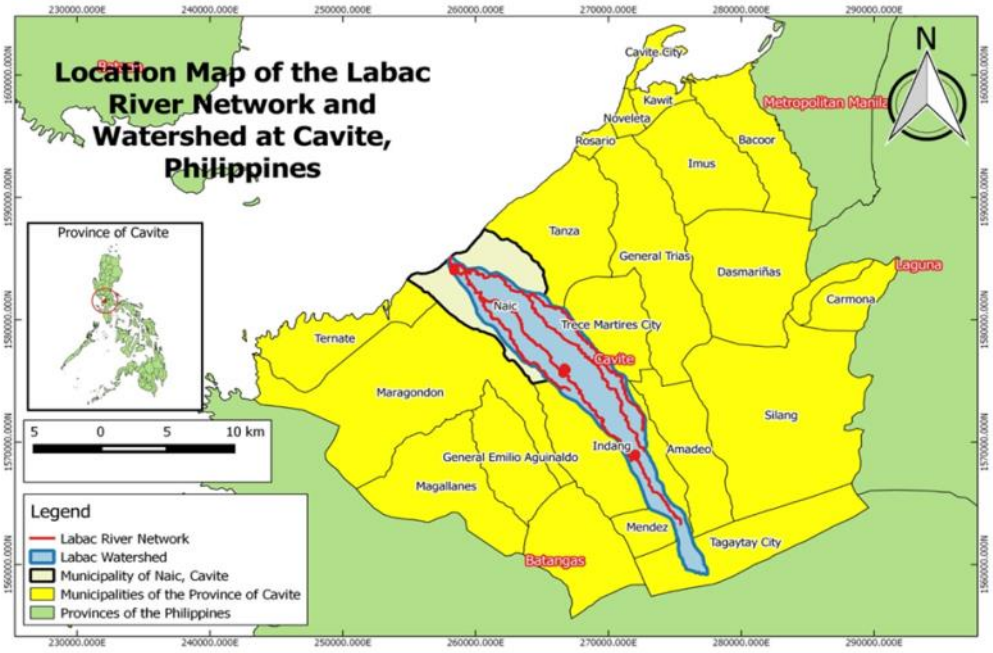

Figure 2. Labac-Alemang Watershed[2]

Sampling

Water Sampling

Surface water samples from different locations, i.e., upstream, midstream and downstream of Bukana Malaki River in Naic, Cavite which is the tail-end of the Labac-Alemang River Watershed, were collected in March 2018. Amber glass bottles (1L) were used for sampling, which were washed successively with detergent, water, distilled water and solvents before sampling. Three-liter (3L) surface water samples per location were collected using stainless bucket. The samples were transferred to the amber glass bottles and the bottles were filled to the brim with the sample water to eliminate air bubbles. After proper labelling, the water samples were transported with ice to the laboratory, stored at 4°C followed by an extraction within seven days.

Sediment Sampling

The collection of the sediment samples was also done simultaneously with the collection of water samples in March 2018. Five hundred-gram composite sediment samples from each sampling location were collected from the surface (10 - 15 cm deep) with a stainless-steel auger and stored in three 1-L amber bottles. The location and description of each sampling site was recorded in detail. After sampling, the sediment samples were brought to the laboratory. A diagram of composite sampling is shown in Figure 3. Samples are taken at regularly spaced intervals in all directions.

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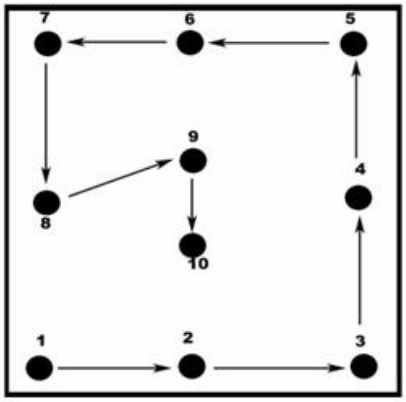

Figure 3. Diagram of composite sampling

Sample Analysis

Screening Analysis in Water

One liter of sample was transferred to a two-Liter separatory funnel and extracted with dichloromethane three times for three (3) minutes for each extraction. The organic layers were combined and allowed to pass through sodium sulfate previously rinsed with 30 mL dichloromethane. The organic layer was concentrated by Kuderna-Danish apparatus and solvent exchanged to n-hexane. The n-hexane extract was transferred to a 2 mL vial and screened by Gas Chromatography with Mass Spectrometer Detector (GC-MSD).

Screening Analysis in Sediments

Five grams of sample was subjected to Soxhlet extraction with a mixture of acetone: n-hexane. The extract was concentrated by Kuderna-Danish apparatus and solvent exchanged to n-hexane. The n-hexane extract was transferred to a 2mL vial and screened by Gas Chromatography with Mass Spectrometer Detector (GC-MSD).

Procedure for GC-MS Analysis

The n-hexane extracts obtained from the water and sediments samples, respectively, were analyzed separately by GC-MS using a Rxi-5MS, 0.25mm x 30m x 0.25micrometer column in a Shimadzu GCMSQP2010. The carrier gas was helium with a constant flow rate of 5 mL/min. The oven temperature was initially kept at 90 ^{o}C for 4 min then ramped at 10 ^{o}C/min to 300 ^{o}C. An amount of 1.0 µL of the sample (100 ppm in dichloromethane) solutions was injected in the split mode. Mass spectra were obtained over the scan range 50-650 m/z. The procedure was done in three replicates for the water and sediment samples, respectively. The readings were recorded and the average of three readings for each sample from the upstream, midstream and downstream were computed. The compounds were identified by comparison of their mass spectra with those of the NIST 17 mass spectral library.

Chromatographic Conditions

The instrument used was GCMSQP2010 with the following specifications and parts: AOC-20i auto injector, Rxi-5MS, 0.25mm x 30m x 0.25micrometer column; oven temperature with a rate of 5.0 @ 300.0 ^{o}C; injector temperature: 250.0 ^{o}C; ion source temperature: 230.0 ^{o}C; interface temperature: 250.0 ^{o}C; injection mode: split; column flow: 0.97mL/min; carrier gas: helium, UHP; purge flow: 5.0 mL/min; split ratio: 35; solvent cut time: 2.50min; acquisition mod: SCAN; and scan range: 50-650 m/z.

RESULTS AND DISCUSSION

The screening analysis by Gas Chromatography-Mass Spectrometry (GC-MS) showed that the river water and sediments contained numerous organic pollutants. The results are presented in Table 1 for water samples and Table 2 for sediment samples. It can be noted that in the

water samples, eleven (11) different organic compounds were identified at the upstream, twenty-nine (29) at the midstream and twenty-eight (28) at the downstream. From the upstream, eight (8) organic compounds were found to have more than 5.0 percent composition, six (6) from the midstream, and two (2) from the downstream. Still from among the water samples, the downstream sampling station contained the pollutant with the highest concentration that is n-hexadecanoic acid methyl ester which is 56.48 percent by mass.

Among the sediment samples, forty-one (41) different organic compounds were identified at the upstream, thirteen (13) at the midstream, and eight (8) at the downstream. From the upstream, only one (1) organic compound had percentage composition greater than 5.0 percent, ten (10) from the midstream, and four (4) from the downstream. The upstream sampling station contained the pollutant with the highest concentration, acetonyldimethylcarbinol which was found to be 94.11 percent by mass.

Only one organic compound, N-(Trifluoroacetyl)-N,O,O',O"-tetrakis (trimethylsilyl) norepinephrine, was found in both the upstream and downstream of the water samples and none for the sediment samples. Two organic compounds, glycerol tricaprylate and lauric acid methyl ester were both found in the upstream of the water samples and downstream of the sediment samples. All others were different compounds from the upstream and downstream of the water and sediment samples.

Several of the organic compounds found in both water and sediment samples are hazardous. The hazardous effects include eye, skin, digestive tract and respiratory tract irritations. Some may even cause organ toxicity. Others pose danger to health and the environment.

The following are some of the organic compounds found in the samples with their specific hazardous effects:

Acetonyldimethylcarbinol is a flammable liquid and vapor. It causes skin and serious eye irritations and may cause damage to organs through prolonged or repeated exposure. It enters the body through the respiratory and digestive tracts, affects the nervous system, and damages the liver and stomach. Inhalation of high concentrations of vapor will form pulmonary edema, even coma. Long-term exposure can lead to dermatitis.

Lauric acid methyl ester and octanoic acid are very toxic to aquatic life. Glycerol tricaprylate is not considered hazardous according to the 2012 OSHA Hazard Communication Standard (29 CFR 1910.1200). It is not a dangerous substance or mixture according to the Globally Harmonized System (GHS) however, specific hazards arising from the chemical are its combustion products: carbon monoxide and carbon dioxide. The same is true with 1, 3-dimethylurea. It is not considered hazardous by the 2012 OSHA Hazard Communication Standard (29 CFR 1910.1200) but it can produce hazardous combustion products such as nitrogen oxides (NOx), carbon monoxide (CO) and carbon dioxide (CO_2). N-hexadecanoic acid methyl ester causes skin irritation and may cause an allergic skin reaction. This also contains hazardous decomposition products such as carbon monoxide (CO) and carbon dioxide (CO_2). Bis (2-ethylhexyl) isophthalate is a colorless, oily organic carcinogen with a slight odor.

A few others do not have complete available information to be considered hazardous. Still some are classified as non-hazardous such as n-Heptacosane, n-Hexacosane, n-hexatriacontane, and n-octacosane.

Others do not have available hazard information such as N-(trifluoroacetyl)-N,O,O',O"-tetrakis (trimethylsilyl) norepine-phrine, 2,3-hexanediol, tert-butyl cyanomethylcarbamate and, Trimethylsilyl-2,5 bis(trimethylsilyl)oxy} benzoate. These information are all based on the material safety data sheets of the said organic compounds.

As to their sources, these organic compounds were produced from the use of household materials and industrial products such as butter, milk, coconut oil, food flavorings, biodiesel, and plastics released to the environment.

SUMMARY AND CONCLUSIONS

The study was conducted in cooperation with the officials of Brgy. Bukana Malaki, Naic, Cavite where the sampling site for screening is located to (1) gather qualitative data and gain insights into source identification, and (2) provide quantitative baseline data of organic pollutants for future studies. It was done in March 2018. The water and sediment samples were screened for the presence of organic pollutants using Gas Chromatography-Mass Spectrometry. In the water samples, 11 different organic compounds were identified at the upstream, 29 at the midstream, and 28 at the downstream. In the sediment samples, 18 different organic compounds were identified at the upstream, 41 at the midstream, and 8 at the downstream. Among the water samples, the downstream sample contained the organic pollutant with the highest concentration (58.46% by mass). And, among the sediment samples, the upstream sample contained the organic pollutant with the highest concentration (94.11% by mass). Only one organic compound, N-(Trifluoroacetyl)-N,O,O',O"-tetrakis (trimethylsilyl) norepinephrine, was found in both the upstream and downstream of the water samples and none for the sediment samples. Two organic compounds, glycerol tricaprylate and lauric acid methyl ester were both found in the upstream of the water samples and downstream of the sediment samples. Several of the organic compounds found in both water and sediment samples are hazardous. The hazardous effects include eye, skin, digestive tract and respiratory tract irritations. Some may cause organ toxicity. Others pose danger to health and the environment.

Their sources of origin were mostly from materials used at home or from sewage and industrial products of which these compounds are components and these were released to the environment.

Thus, this study has demonstrated the utility of screening method using Gas Chromatography-Mass Spectrometry in assessing the presence of organic pollutants in surface water and sediments in the river. These organic compounds were characterized as to their sources and to the hazardous effects they pose to health and environment through their material safety data sheets. This needed further study.

IMPLICATIONS AND RECOMMENDATIONS

Organic pollution affects the organisms living in a stream by lowering the available oxygen in the water. This causes reduced fitness, or, when severe, asphyxiation. The increased turbidity of the water reduces the light available to photosynthetic organisms. Organic wastes also settle out at the bottom of the stream, altering the characteristics of the substratum.

The results of the screening analysis by Gas Chromatography - Mass Spectrometry (GC-MS) for both the surface water and sediments at Bukana Malaki River in Naic, Cavite which is the tail end of the Labac-Alemang River Watershed showed the presence of many organic compounds. Several of these organic compounds were found to be toxic, dermal and/or respiratory irritants and may even be lethal in high doses as described in their material safety data sheets. Others have information not fully established. These organic compounds were produced from the use of household/food materials and industrial products released to the environment and these must be considered in terms of solid waste management and water pollution control.

To complete the components of a holistic assessment of the water and sediment quality, the following are recommended: (1) The toxicity and bio-availability via tissue analysis and sediment toxicity testing may be measured to assess the resident biota, via community bio-assessment/survey and procedures; and (2) The study may be replicated in other river systems of Cavite.

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Table 1. Organic compounds screened in water samples

SAMPLE	SURFACE WATER SAMPLES ORGANIC COMPOUNDS	% COMPOSITION
Water Upstream	Diisooctyl phthalate	19.53
	Bis(2-ethylhexyl)isophthalate	18.77
	Glycerol tricaprylate	13.02
	Methyl-3-methylenecyclopentanecarboxylate	12.34
	Lauric acid, methyl ester	9.45
	Octanoic acid, allyl ester	8.55
	Phenylcyclohexane	5.47
	2-iodo-3-methylbutane	5.09
	1,2,4-trichloro-1,1,2,3,3,44-heptachlorobutane	4.78
	N-(Trifluoroacetyl)-N,O,O',O"-tetrakis(trimethylsilyl) norepeniphrine	1.78
	3-methoxy-2,2,4,4-tetramethylpentane	1.24
Water Midstream	2-(2',4',4',6',6',8',8'Heptamethyltetresiloxan -2'yloxy') - 2,4,4,6,6,8,8,10,10-nanomethylcyclopentasiloxane	16.14
	2-Hexanol	12.22
	2-Chlorocyclohexanol	11.66
	3-Hexanol	8.79
	1,3,3,3-Tetramethyldisiloxanyl tris(trimethylsilyl) orthosilicate	5.54
	Neohexane	5.27
	Bicyclo[3.1.0]hexan-3-one	3.53
	Hexadecamethyl-cyclooctasiloxane	3.40
	N-(Trifluoroacetyl)-N,O,O',O"-tetrakis(trimethylsilyl)norepine-phrine	2.78
	(Z)-4,4-Dimethyl-2-pentene	2.54
	1-Azabicyclo[3,1,0]hexane	2.53
	1-(2-Methoxyethoxy)-2-methyl-2-propanol, methyl ester	2.53
	Monoallylamine	2.29

Continuation of Table 1. Organic compounds screened in water samples

SAMPLE	SURFACE WATER SAMPLES ORGANIC COMPOUNDS	% COMPOSITION
Water Downstream	N-(Trifluoroacetyl)-N,O,O',O"-tetrakis(trimethylsilyl)norepinephrine	2.12
	Dodecamethylcyclohexasiloxane	1.84
	Bicyclo[3,1,0]Hexan-3-one	1.58
	Bromocyloheptane	1.02
	Oxalic acid, allylbutyl ester	0.9
	1-Nitorisobutan	0.88
	Methyl isocyanate	0.76
	1,1,2,2,-Tetramethylcyclopropane	0.71
	P-Dioxane	0.63
	Metyl-3-methylene-2-oxetanone	0.63
	Methyl vinyl carbinol	0.61
	2,2,5-Trimethylhexan	0.52
	Monoallylamine	0.44
	Monoallylamine	0.38
	Tert-Butyl N-hydroxycarbamate	0.32
	Bromocycloheptane	0.28
	Phthalic acid, cyclobutyltridecyl ester	0.28
	Dimethylnitrosoamine	0.17
	3-Fluro-2-propynenitrile	0.13

Table 2. Organic compounds screened in sediment samples

SAMPLE	SEDIMENT SAMPLES ORGANIC COMPOUNDS	% COMPOSITION
Sediments Upstream	Acetonyldimethylcarbinol	94.11
	4-Hydroxy-2-pentanone	0.95
	2-Chlorocyclohexanol	0.87
	3,3-Diethoxy-1-propyne	0.37
	2,2-Dimethyl-3-pentanol	0.35
	2-Cyclohexanone	0.35
	Isobutenyl methyl ketone	0.3
	2-Hexyl hydroperoxide	0.28
	3-Hexanol	0.26
	2-Methylene-cyclopentanol	0.17
	Isopropylcyclohexane	0.12
	Isoprene oxide	0.12
	1-Butene	0.11
	Citrulline	0.11
	Heptane	0.1
	1,2-Diacetylethylene	0.1
	4,6-Diamino-1,3,5-triazine-2-carboxylic acid	0.1
	4-Ethyl-3-methylhexane	0.1
	Diisobutylene	0.08
	Valerphenone	0.08
	2,2,5,5-Tetramethylhexane	0.08
	Sec-Butyl formate	0.06
	2-Hexanol	0.06

Continuation of Table 1. Organic compounds screened in sediment samples

SAMPLE	SEDIMENT SAMPLES ORGANIC COMPOUNDS	% COMPOSITION
Sediments Upstream	Trimethylaminomrthane	0.06
	Oxalic acid, allyl butyl ester	0.06
	3-(Hydroxymethyl)-4,4-dimethylpentanal	0.06
	Cis-4.4-Dimethyl-2-Pentene	0.05
	3-Methylcyclopentanone	0.05
	1,4-Dimethoxybutane	0.05
	4-Trifluoroacetoxyoctane	0.05
	2-Bromo-6-methylheptane	0.05
	Diisobutylene	0.05
	Pivaloactone	0.04
	Dihydroresorcinol	0.04
	2,2,5-Trimethylhexane	0.04
	N,N'-Dimethylurea	0.03
	2-Methyl-3-buten-1-ol	0.03
	1,2,3,4-Tetramethylcyclobutane	0.03
	2-Propenal	0.03
	.mu.-Mercaptodiborane	0.03
	Tert-Butyl Carbazate	0.03
Sediments Midstream	N-Octacosane	15.45
	N-Hentriacontane	11.29
	N-Hexacosane	11.24
	N-Heptacosane	10.85
	Cyclic octaatomic sulfur	10.14

Continuation of Table 1. Organic compounds screened in sediment samples

SAMPLE	SEDIMENT SAMPLES ORGANIC COMPOUNDS	% COMPOSITION
Sediments Midstream	N-Nonacosane	9.94
	N-Hexatriacontane	7.75
	N-Heinecosane	7.08
	2-Methyloctacosane	5.85
	N-Eicosane	5.46
	2,6,10,25-Tetramethylheptadecane	2.31
	2,3,3-Trimethyl-2-butanol	1.97
	N-Decyl iodide	0.67
	2-Methylheptanoic acid	0.42
Sediments Downstream	Glycerol tricaprylate	41.83
	Lauric acid methyl ester	26.24
	Tert-Butyl-3-(formylamino)-1-pipereidine carboxylate	11.87
	Capric acid methyl ester	11.11
	1,2-Dimethyltryptamine	4.27
	Cyclic octaatomic sulfur	2.76
	2,3,3-Trimethyl-2-butanol	1.5