



**MARYLAND DEPARTMENT OF TRANSPORTATION
STATE HIGHWAY ADMINISTRATION**

RESEARCH REPORT

**EVALUATION OF WASTE CONCRETE ROAD MATERIALS
FOR USE IN OYSTER AQUACULTURE – PHASE 3**

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Project Number: SP409B4E

FINAL REPORT

August 2016

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Technical Report Documentation Page

1. Report No. MD-16-SHA-MSU-4-10	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle Evaluation of Waste Concrete Road Materials for Use in Oyster Aquaculture – Phase 3		5. Report Date August 2016	
		6. Performing Organization Code	
7. Author/s Mark M. Bundy Dong Hee Kang		8. Performing Organization Report No.	
9. Performing Organization Name and Address Morgan State University 1700 East Cold Spring Lane Baltimore MD 21251		10. Work Unit No. (TRAIS)	
12. Sponsoring Organization Name and Address Office of Policy and Research Maryland State Highway Administration 707 N. Calvert Street Baltimore, MD 21202		11. Contract or Grant No. SP409B4E	
		13. Type of Report and Period Covered Final Report	
		14. Sponsoring Agency Code	
15. Supplementary Notes			
16. Abstract This project was the final phase of a three-phase project. The primary objective was to determine the suitability of recycled concrete aggregate (RCA) from road projects as a bottom conditioning material for on-bottom oyster aquaculture in the Chesapeake Bay. This third phase project evaluated RCA for petroleum byproducts and provided evaluation methodologies. The results showed that RCA as a base material for oyster reefs did not leach any hydrocarbon chemicals and no water extractable SVOC was detected from RCA following EPA 1316 extraction. Low levels of anthracene, fluorine, phenanthrene and pyrene were detected in samples collected from Machado Construction Co., Inc. and The Recycling Center, but the concentrations were much lower than the State of Maryland Department of the Environment cleanup standard for soil and groundwater. They were at least 100 times below the level considered hazardous in the Protected Groundwater Standard. The project concluded that there was no cause for concern about hydrocarbon components released into the Chesapeake Bay watershed if RCA is used as a bottom conditioning material for oyster aquaculture.			
17. Key Words: recycled concrete aggregate, RCA, oysters, hydrocarbon		18. Distribution Statement: No restrictions This document is available from the Research Division upon request.	
19. Security Classification (of this report) None	20. Security Classification (of this page) None	21. No. Of Pages	22. Price

Form DOT F 1700.7 (8-72) Reproduction of form and completed page is authorized.

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ACKNOWLEDGEMENT

We would like to acknowledge the staff at Morgan State’s Patuxent Environmental & Aquatic Research Laboratory (PEARL) led by Dr. Kelton Clark. PEARL staff members, especially Jody Gregory, for their help on support activities and acquisition of supplies. We would also like to acknowledge P. Flanigan & Sons, Inc.(Baltimore, MD), Machado Construction Co. Inc. (Baltimore, MD), and The Recycling Center (Laurel, MD) for their contributions of assistance and materials during the project. The research would not have been possible without their help.

EXECUTIVE SUMMARY

The use of recycled materials has gained increased attention for the environmental benefits, and the reuse of industrial by-products and waste materials can provide a stream of revenue for producers and a durable, cost-effective material option for end users. Recycled concrete aggregate (RCA) is one of the recycled materials that the Maryland Department of Transportation, State Highway Administrative (SHA) is exploring its alternative uses. For example, using RCA to condition portions of the Chesapeake Bay bottom to support spat-on-shell aquaculture projects. The RCA is created by crushing and milling old concrete pavement or other structure elements. For RCA to be used within the aquatic setting of the Chesapeake Bay, its chemical behavior under saturated conditions must be evaluated to avoid potential adverse impacts to the aquatic ecosystem. The major concern was that accidental spills onto the road surface from vehicles could contaminate the pavement and structure elements.

The study evaluated the leaching of hydrocarbon components from RCA materials, and is a continuation of previous SHA projects *Evaluation of Waste Concrete Road Materials for Use in Oyster Aquaculture* (2013) and *Phase II Evaluation of Waste Concrete Road Materials for Use in Oyster Aquaculture - Field Test* (2015). These three phases of the research established a database of water quality impacts of RCA and oyster survivability. Based on the findings of these evaluations, recommendations for the use of RCA on oyster-leased bottom in the Patuxent River may be made to the oyster industry, and useful information will be provided to state agencies.

The testing was designed to evaluate RCA for toxic organic substances and provide evaluation methodologies. Volatile components were not chosen in this research scope because volatile compounds spilled onto the roadway would evaporate quickly. Since RCA is usually placed in a recycling plant before the crushing process and stockpiling usually happens before application, it gives sufficient time for volatile components to evaporate. In almost all cases of hydrocarbon contamination, the compounds of interest were the semivolatile components included in polycyclic aromatic hydrocarbons. The resulting water chemistry from the following leaching tests was used to evaluate acute and chronic water quality necessary for protecting marine and estuarine life based on the water quality standard of Maryland (COMAR 26.08.02.03-2).

All results of organic chemical concentrations were below detection limit (BDL) (Table 6 and 7) for both EPA 1316 and 3570 extraction methods for RCA samples produced by Flanigan & Sons, Inc. Four organic chemicals for EPA 3570 extraction method were detected at the samples collected from Machado Construction Co., Inc. and The Recycling Center. However, the concentrations were at least 100 times lower than COMAR 26.08.02.03-2. These results confirmed that there is no cause for concern about hydrocarbon components released into the Chesapeake Bay watershed if RCA is used as a bottom conditioning material for oyster aquaculture.

1. INTRODUCTION

The Maryland Department of Transportation State Highway Administration (SHA) intends to increase the use of recycled materials in an environmentally responsible manner. As roads and bridges are resurfaced, old concrete is removed and usually discarded, which places a burden on society to accommodate the waste concrete in landfills. It would be in the best interest of SHA and the environment to recycle it into an alternative use, such as to condition portions of the Chesapeake Bay bottom to support spat-on-shell oyster restoration or aquaculture projects (SHA, 2013, and 2015).

Native oyster populations in the Chesapeake Bay are at less than 1% of historic levels due to two protozoan diseases (MSX, Multinucleated Sphere Unknown, disease caused by *Haplosporidium nelsoni* and Dermo disease caused by *Perkinsus marinus*), overharvesting, and pollution (CRC, 1999). This tremendous decline in the oyster population has dramatically changed the Bay's ecosystem and the oyster industry. Individual oysters filter 4-34 liters of water per hour, removing phytoplankton, sediments, pollutants, and microorganisms from the water column (CERP, 2007). Historic oyster populations of Chesapeake Bay could filter excess nutrients from the estuary's entire water volume every three to four days. Today, that would take nearly a year.

Spat-on-shell is the most ecologically friendly method of culturing oysters in the Chesapeake region. To make new areas ready for on-bottom spat-on-shell aquaculture, the barren Bay bottom needs to be built up with a hard material that supports the spat-on-shell (a process known as bottom conditioning) and prevents it from sinking into soft muddy bottoms. Historically, old oyster shells were used for this purpose. However, the decline of the Chesapeake Bay region's oyster industry has led to the scarcity of available oyster shell and using them for bottom building is no longer practical.

This is a three-phase study to examine the use of recycled concrete aggregate (RCA) from road construction in bottom conditioning. RCA is a crushed concrete material created by removal and milling of old concrete pavement and structure elements. The material is processed and sorted for reuse as base, sub-base, structure fill material for embankments, and in new concrete mix. For RCA to be used within the aquatic setting of the Chesapeake Bay, regulatory agencies must be assured that it has no direct negative environmental impacts. In the Phase I study, the chemical behavior of RCA under saturated conditions was evaluated. That resulted in either no leaching of adverse materials or leaching at a rate that is orders of magnitude below regulatory levels. Further, the introduction of RCA did not raise pH above the minimum threshold for introduction in Maryland waters. In Phases I and II, the experiments performed in the laboratory and the field showed that there was no difference between RCA and oyster shells on oyster recruitment, survival or growth, nor was there an effect on the associated community of organisms (SHA, 2013 and 2015). The Phase III project evaluated the leaching of hydrocarbon chemicals from RCA.

1.1 Summary of Previous Work

Evaluation of Waste Concrete Road Materials for Use in Oyster Aquaculture (MD-13-SP109B4E) (SHA, 2013)

The primary objective of the phase I study was to determine the suitability of recycled concrete from road projects as conditioning material for on-bottom oyster aquaculture in the Chesapeake Bay. The testing was designed to evaluate the impact on water chemistry from the introduction of RCA and evaluate the effect of RCA on the survivorship and growth of oyster spat. The results of this project showed that using RCA as a base material for oyster reefs did not adversely affect oyster spat growth and survival, or the surrounding environment. None of the metals leached at a rate that exceeded the EPA drinking water standards. This standard is more stringent than the current EPA total maximum daily loads (TMDLs) for Chesapeake Bay waters. There was no statistical difference between shells and RCA on the growth, survivorship, average length, or recruitment of young oysters. Initial pH was slightly higher for the RCA (8.20 to 8.36) than the oyster shell control (8.0 to 8.2), but pH stabilized to around 7.6 to 7.8 for all treatments after seven days. Based on the findings of this study, the recommendation was to initiate a second phase that places RCA on test plots in the Chesapeake Bay to validate the laboratory tests in situ.

Evaluation Of Waste Concrete Road Materials For Use In Oyster Aquaculture – Field Test (MD-15-SHA-MSU-3-12) (SHA, 2015)

The primary objective of this Phase II study was to determine the suitability of RCA from road projects as bottom conditioning material for on-bottom oyster aquaculture in the Chesapeake Bay. The testing was designed to evaluate the potential introduction of organisms attracted to the RCA pile in situ that may be potential predators of oyster spat, determine potential impacts on or disruptions to the use of traditional harvesting gear on aquaculture areas conditioned with RCA, and identify regulatory or administrative structures that oversee the use of RCA and challenges within those structures. Three substrates were tested for their effect on benthic communities: RCA, RCA with a veneer of oyster shells, and oyster shells. There was no difference in population or community parameters among the three substrates. The number and type of species were the same among the substrates as were their absolute and relative abundances. Oyster spat settlement was the same among the three substrates as well. Waterman tonging on RCA found it heavier and more difficult to work than tonging on oyster shells. They recommended that RCA be used either with a veneer of oyster shells or in applications where tonging was not anticipated. Overall the findings support the use of RCA in select applications. However, the regulatory structures presently in place do not include a mechanism for the acceptance of a novel material. Moving forward with RCA or any new material requires an application for a reef project. The acceptance of the project is then a de facto acceptance of the material. Adopting a criteria for materials used in reef construction will provide agencies with a basis for supporting choices on materials used and the private sector with a basis to develop products to meet restoration and aquaculture needs.

1.2 Recycled Concrete Aggregate (RCA)

The RCA has become a more attractive option as an aggregate substitute in pavement construction, and is a mixture of concrete, soil and small quantities of bituminous concrete (FHWA, 2008). It has a rougher surface texture, lower specific gravity, higher water absorption, and lower specific gravity than natural aggregates.

Contaminant leaching and pH changes caused by RCA are the primary environmental concerns. Leaching tests of Portland cement concrete through Toxicity Characteristic Leaching Procedure (TCLP) using acetic acid detected arsenic, beryllium, cadmium, chromium, mercury, lead, and selenium (Kanare and West, 1993). The long-term potential leachable trace toxic metals of Portland cement concrete showed that arsenic (19.9 mg/kg), beryllium (1.4 mg/kg), chromium (72.7 mg/kg), lead (75.3 mg/kg), nickel (72.0 mg/kg) and vanadium (44.1 mg/kg) were detected (Sangha et al., 1998) and significant levels of calcium and aluminum were also detected (Nelson et al., 2000). In the case of Minnesota, where deicing salts are extensively used, recycled concrete may contain relatively high levels of chlorides associated with corrosion of steel. Cement paste consists of calcium-aluminum-silicate compounds and the pH of RCA-water mixtures often exceeds 11 due to calcium hydroxide, which could adversely affect on environment.

1.3 RCA Types

The RCA is produced by crushing concrete into pieces of the desired size. The resulting RCA comes in the following 4 basic sizes (<http://www.laneyrecycling.com/maryland-recycled-aggregates.php>).

1. RC-6 Recycled Concrete ($< 1^{1/2}$ "): RC-6 can be used as a sub-base for parking lots and roadways, residential driveways, walkways and select back-fill.
2. RC-2 Recycled Concrete ($1^{1/2}$ " to $2^{1/2}$ "): RC-2 is frequently used for construction entrances, drainage and erosion control, stabilization of wet areas, under-cutting, etc.
3. RC-Surge Recycled Concrete (3" to 8"): RC-Surge can be used for erosion control, bulkheads and slope protection.
4. RC-57 Recycled Concrete ($3/4$ " – $1^{1/2}$ "): RC-57 is used primarily as a subgrade for concrete slabs, base material for driveways, or erosion prevention fill.



A) RC-6



B) RC-2



C) RC-Surge



D) RC-57

Figure 1 Recycled concrete type by sizes

1.4 Physical Characteristics of Petroleum Byproduct

Petroleum byproducts contain primarily hydrocarbons, and are generally classified into two major component categories: hydrocarbons and nonhydrocarbons. The hydrocarbon constituents can be grouped into saturated hydrocarbons (Aliphatic such as Butane and Isobutane, Alicyclic), unsaturated hydrocarbons (Alkenes/Olefins such as Ethylene 1-Butene and Alkynes /Acetylenes such as Acetylene 1-Butyne), and aromatics which are common environmental contaminants. Aromatic compounds are a special class of unsaturated hydrocarbons.

Polycyclic aromatic hydrocarbons (PAHs) are nonpolar and lipophilic and composed of multiple aromatic rings. Most PAHs are not soluble in water and persist in the environment. Aqueous solubility of PAHs decreases approximately logarithmically as molecular mass increases (Choi et al., 2010). PAHs are formed during the incomplete burning of coal, oil and gas. The sources of PAHs have included vehicle exhaust, weathered material from asphalt roads, lubricating oils, gasoline, diesel fuel, and tire particles (Takada et al., 1990). There are more than 100 different PAH compounds. The EPA has characterized 16 PAHs as priority pollutants. The U.S. Department of Health and Human Services has determined that many PAHs may be considered carcinogenic (ATSDR, 1999). In the case of Maryland, Code of Maryland Regulations, Title 26 which is Department of Environment, Subtitle 08: Water Pollution, Chapter 2: Water Quality (COMAR 26.08.02.03-2) provide the numerical criteria for toxic substances in surface water which is listed in Table 1.

Table 1. COMAR 26.08.02.03-2 Numerical Criteria of Toxic Substances in Surface Waters

Substance Chemical Abstracts Service (CAS)		Human Health for Consumption of: (Risk Level = 10 ⁻⁵) (µg/L)		
		Water + Organism	Organism Only	Chemical Group
Acenaphthene	83329	670	990	PAH
Anthracene	120,127	8,300	40,000	PAH
Benzo(a)Anthracene	56553	0.038	0.18	PAH
Benzo(a)Pyrene	50328	0.038	0.18	PAH
Benzo(b)Fluoranthene	205992	0.038	0.18	PAH
Benzo(k)Fluoranthene	207089	0.038	0.18	PAH
Bis(2-Ethylhexyl) Phthalate	117817	12	22	Phthalates
Butylbenzyl Phthalate	85687	1,500	1,900	Phthalates
Chrysene	218019	0.038	0.18	PAH
Dibenzo(a,h)Anthracene	53703	0.038	0.18	PAH
Diethyl Phthalate	84662	17,000	44,000	Phthalates
Dimethyl Phthalate	131113	270,000	1,100,000	Phthalates
Di-n-Butyl Phthalate	84742	2,000	4,500	Phthalates
Fluorene	86737	1,100	5,300	PAH
Hexachlorobenzene	118741	0.0028	0.0029	Organic Compounds
Hexachlorobutadiene	87683	4.4	180	Organic Compounds
isophorone	78591	350	9,600	Organic Compounds
Pentachlorophenol (PCP)	87865	2.7	30	Pesticides and Chlorinated
Pyrene	129000	830	4,000	PAH

1.5 Research Objectives

While it is generally recognized that RCA materials do not present a great risk to human health or the environment, a better characterization of the amount and type of chemicals that leach in the environment will help provide a better means to correctly manage this material. This project is the third phase of a three-phase project. The leaching performance of recycled concrete materials potentially contaminated by toxic organic substances, especially petroleum residues and PAH, was investigated through a series of laboratory experiments that included batch and tank leaching experiments.

2. MATERIALS AND METHODS

2.1 Sample collection

The first task was to select different RCA sources throughout the State of Maryland. The samples were collected from three concrete dumping sites, where RCA materials were produced (Table 2). The samples were collected in two 250 mL wide mouth glass jars with Teflon-lined screw caps from crushed RCA stockpiles at the plants.



Figure 2 Sampling sites (A- Flanigan & Sons, Inc. B- Machado Construction Co., Inc. and C- The Recycling Center) and D is a sample in cooler after sampling

RC-Surge recycled concrete (3” to 8”) is used for oyster conditioning materials, but smaller sizes were collected from each plant for extraction convenience. RC-6 (< 1 1/2”) was collected from Machado Construction Co., Inc. and The Recycling Center, RC-2 (1 1/2” to 2 1/2”) was collected from Flanigan & Sons, Inc., where RC-2 was the smallest size.

Table 2. Recycled Concrete Sampling Sites in Maryland

Company	Flanigan & Sons, Inc.	Machado Construction Co., Inc.	The Recycling Center
Address	2444 Loch Raven Road Baltimore, MD 21218	2930 Hammonds Ferry Road, Baltimore, MD 21227	14852 Old Gunpowder Road, Laurel, MD 20707
Phone	410-467-5900	410-247-2662	410-792-2999
Sample Type	RC-2	RC-6	RC-6

The collected samples were cooled to $4\pm 2^\circ\text{C}$ immediately after collection. Collected RCA samples were extracted within 48 hours of collection, and analyzed within one week of extraction. Two separate samples were collected at the same time and processed under identical field and laboratory procedures. Analyses of such field duplicates give a measure of the precision associated with sample collection and storage, as well as laboratory procedures.

2.2 Moisture content measurement

The dry mass equivalent of the "as-tested" material was determined by drying the collected RCA samples at $105 \pm 2^\circ\text{C}$ until a constant mass is reached.



Figure 3 Moisture content measurement of RCA

The moisture contents of collected recycled concrete samples from Flanigan & Sons, Inc., Machado Construction Co., Inc., and The Recycling Center are $5.63\pm 1.56\%$, $6.59\pm 0.65\%$, $8.51\pm 1.35\%$, respectively.

2.3 Extraction method

Volatile organic compounds (VOCs) were not expected to be a major concern with regard to leaching from RCA. These compounds such as butane, propane, benzene, toluene, ethylbenzene and xylene in gasoline are preferentially volatilized (Bauman, 1988) and tend to evaporate quickly when concrete is milled on the demolition sites and during stockpiling period. VOCs in the leachate from recycled asphalt pavement (RAP) were found below detection limit (BDL), with various extraction tests, including Toxicity Characteristic Leaching Procedure (TCLP), Synthetic Precipitation Leaching Procedure (SPLP), Deionized Water Leaching Procedure, and Column Leaching Procedure (Timothy, 1998). Therefore, VOCs were not tested in this study.

2.3.1 Microscale Solvent Extraction (MSE)-Method 3570

EPA test method 3570 was applied to extract a petroleum chemical to determine the mobility of petroleum analytes presented in RCA. Method 3570 is a procedure for extracting organic compounds, especially semivolatile organic compounds (SVOCs). Collected RCA samples were prepared by shake extraction for 4 hours with methylene Chloride (CH_2Cl_2) in sealed extraction tubes. Sample extracts were collected, dried by sodium sulfate (Na_2SO_4), and concentrated using a modification of the Kuderna-Danish concentration method. All solid samples were kept cold during the extraction procedure by storing them in a cold room (4°C). All samples were transferred from the cold room only for as long as necessary to remove the sample aliquot. As much as possible, the sample container was kept tightly capped. The extract was transferred to a 2 mL vial fitted with a PTFE-lined screw cap, and the vial was capped and stored in the freezer until analysis. A detailed description of EPA method 3570 is provided in Appendix A-1.

2.3.2 Liquid-Solid Partitioning (LSP) as a Function of Liquid-to-Solid Ratio (L/S) Using a Parallel Batch Extraction Procedure-Method 1316

This method is a tank leaching test to assess the long-term leaching potential of RCA. This method consists of five parallel extractions of a particle size-reduced solid material in reagent water over a range of L/S values from 0.5 to 10 mL/g. The bottles are tumbled in an end-over-end fashion for over 24 hours of contact time based on the maximum particle size ($<0.5\text{mm}$) of RCA. At the end of the contact interval, the liquid and solid phases are roughly separated via settling. The bulk of the eluate is clarified by filtration in preparation for constituent analysis. Appendix A-2 has a detailed description of LSP-EPA method 1316.

2.3.3 Semivolatile Organic Compounds (SVOCs) by GC/MS, EPA 8270D

EPA method 8270D was used to determine the concentration of semivolatile organic compounds in extracts prepared from RCA. The semivolatile compounds are introduced into the Gas Chromatography Mass Spectrometry (GC/MS) by injecting the sample extract into a GC equipped with a narrow-bore fused-silica capillary column. The 30 m long column is silicone-coated and has a 0.25 mm inside diameter and 0.25 μm film thickness (PerkinElmer, Inc., MA). The GC column is temperature-programmed to separate the analytes, to be detected with a connected MS. Commercial semivolatile and PAH mixtures were purchased from PerkinElmer. The stock standard solutions were transferred into bottles equipped with PTFE-lined screw caps. Each 1-mL sample extract undergoing analysis was spiked with 10 μL of the internal standard solution. Five calibration standards for semivolatile and PAH mixtures were prepared at different concentrations. 2-fluorophenol, nitrobenzene-d₅, and 2-fluorobiphenyl were used for surrogate solution which was purchased from PerkinElmer.

GC/MS operating conditions are as follows:

- Mass range: 35-500 amu
- Scan time: ≤ 1 sec/scan
- Initial temperature: 35°C , hold for 4 minutes
- Temperature program: $35\text{-}320^\circ\text{C}$ at $10^\circ\text{C}/\text{min}$

- Final temperature: 300 °C, hold
- Injector temperature: 250-300° C
- Transfer line temperature: 250-300° C
- Sample volume: 1 µL
- Carrier gas: helium at 30 cm/sec.

GC run log data was shown in Appendix A.3.



Figure 4 Clarus SQ 8 GC/MS

3. RESULTS AND DISCUSSION

Task 1. Evaluate the RCA for hydrocarbons

The leaching of hydrocarbon components from RCA material was the focus of this study. These hydrocarbon components could possibly have been spread onto concrete pavement by vehicles through fluid spills, accidents, general vehicle wear and tear. The leaching evaluation was performed by determining the concentration of a pollutant and comparing that concentration to an applicable guidance. Since there is no current policy for hydrocarbon component leaching of RCA, the State of Maryland Department of the Environment cleanup standard for soil and groundwater was adopted (Table 3).

RCA samples from three sites were used in this study. The samples were extracted as described above and aliquots of the extracts were analyzed using GC/MS. The GC/MS results for the three samples are presented in Table 4 (EPA 1316; extracted by water) and Table 5 (EPA 3570; extracted by solvent). In the case of EPA 1316 extraction, all results of organic chemical concentration were BDL (Below Detection Limit) (Table 4). In the case of EPA 3570 extraction, all results of organic chemical concentration were BDL (Below Detection Limit) for all RCA samples from Flanigan & Sons, Inc.; low levels of anthracene, fluorine, phenanthrene and pyrene were found in samples from Machado Construction Co., Inc. and The Recycling Center, where RC-6 sized RCA samples (the smallest) were collected. The concentrations in the State of Maryland Department of the Environment cleanup standard for soil and groundwater are much higher than the detected concentrations. They were at least 100 times below the level considered hazardous by the “Soil Standard – Protection of Groundwater” (Table 3).

Table 3. State of Maryland Department of the Environment Cleanup Standard for Soil and Groundwater

Semivolatile Organic Compounds (SVOCs)	Groundwater Standards		Soil Standards		
	Type I and II Aquifers*		Residential Clean-up Standard	Non-Residential Clean-up Standard	Protection of Ground water
	(ug/L)	(ug/L)	(mg/kg)	(mg/kg)	(mg/kg)
Acenaphthylene	37	0.037	470	6100	100
Anthracene	180	0.18	2300	31000	470
Benz(a)anthracene	0.2	0.0002	0.22	3.9	0.48
Benzo(b)fluoranthene	0.2	0.0002	0.22	3.9	1.5
Benzo(g,h,i)perylene	18	0.018	230	3100	680
Benzo(k)fluoranthene	0.3	0.0003	2.2	39	15
bis-(2-Ethylhexyl)adipate**	400	0.4	53	240	--
bis(2-Ethylhexyl)phthalate**	6	0.006	46	200	2900
Chrysene	3	0.003	2.2	390	48
Dibenz(a,h)anthracene	0.2	0.0002	0.022	0.39	0.46
Diethyl phthalate	2900	2.9	6300	82000	450
Di-n-butyl phthalate	370	0.37	780	10000	5000
Fluorene	24	0.024	310	4100	140
Hexachlorobenzene**	1	0.001	0.4	1.8	0.052
Hexachlorocyclopentadiene**	50	0.05	47	610	1800
isophorone	70	0.07	670	3000	0.41
Pentachlorophenol**	1	0.001	5.3	24	--
Phenanthrene	180	0.18	2300	31000	470
Pyrene	180	0.018	230	3100	680

* Type I aquifer means an aquifer having a transmissivity greater than 1,000 gallons/day/foot and a permeability greater than 100 gallons/day/square foot, and for natural water with a total dissolved solids concentration less than 500 milligrams/liter.

Type II aquifer means an aquifer having either:

- A) a transmissivity greater than 10,000 gallons/day/foot, a permeability greater than 100 gallons/day/square foot and natural water with a total dissolved solids concentration of between 500 and 6,000 milligrams/liter; or
- B) a transmissivity between 1,000 and 10,000 gallons/day/foot, a permeability greater than 100 gallons/day/square foot and natural water with a total dissolved solids concentration of between 500 and 1,500 milligrams/liter.

** Type I aquifer cleanup standards are same as EPA drinking water standard for organic chemicals.

Table 4. Organic chemical concentration in RCA extracted by EPA 1316

Semivolatile Organic Compounds (SVOCs)/PAH	EPA 1316		
	Flanigan & Sons, Inc.	Machado Construction Co., Inc.	The Recycling Center
	mg/kg	mg/kg	mg/kg
Acenaphthylene	BDL	BDL	BDL
Anthracene	BDL	BDL	BDL
Benzo(a)anthracene	BDL	BDL	BDL
Benzo(a)pyrene	BDL	BDL	BDL
Benzo(b)fluoranthene	BDL	BDL	BDL
Benzo(B)triphenylene	BDL	BDL	BDL
Benzo(g,h,i)perylene	BDL	BDL	BDL
Benzo(J)fluoranthene	BDL	BDL	BDL
Benzo(k)fluoranthene	BDL	BDL	BDL
bis-(2-Ethylhexyl)adipate	BDL	BDL	BDL
bis(2-Ethylhexyl)phthalate	BDL	BDL	BDL
Butylbenzyl phthalate	BDL	BDL	BDL
chlorobenzilate	BDL	BDL	BDL
Chrysene	BDL	BDL	BDL
Dibenz(a,h)anthracene	BDL	BDL	BDL
Diethyl phthalate	BDL	BDL	BDL
Di-n-butyl phthalate	BDL	BDL	BDL
Fluorene	BDL	BDL	BDL
Hexachlorobenzene	BDL	BDL	BDL
Hexachlorocyclopentadiene	BDL	BDL	BDL
isophorone	BDL	BDL	BDL
Pentachlorophenol	BDL	BDL	BDL
Phenanthrene	BDL	BDL	BDL
Pyrene	BDL	BDL	BDL

* Below the Detection Limit

Table 5. Organic chemical concentration in RCA extracted by EPA 3570

Semivolatile Organic Compounds (SVOCs)/PAH	EPA 3570		
	Flanigan & Sons, Inc.	Machado Construction Co., Inc.	The Recycling Center
	mg/kg	mg/kg	mg/kg
Acenaphthylene	BDL*	BDL	BDL
Anthracene	BDL	2.78	1.97
Benz(a)anthracene	BDL	BDL	BDL
Benzo(a)pyrene	BDL	BDL	BDL
Benzo(b)fluoranthene	BDL	BDL	BDL
Benzo(B)triphenylene	BDL	BDL	BDL
Benzo(g,h,i)perylene	BDL	BDL	BDL
Benzo(J)fluoranthene	BDL	BDL	BDL
Benzo(k)fluoranthene	BDL	BDL	BDL
bis-(2-Ethylhexyl)adipate	BDL	BDL	BDL
bis(2-Ethylhexyl)phthalate	BDL	BDL	BDL
Butylbenzyl phthalate	BDL	BDL	BDL
chlorobenzilate	BDL	BDL	BDL
Chrysene	BDL	BDL	BDL
Dibenz(a,h)anthracene	BDL	BDL	BDL
Diethyl phthalate	BDL	BDL	BDL
Di-n-butyl phthalate	BDL	BDL	BDL
Fluorene	BDL	1.65	0.3
Hexachlorobenzene	BDL	BDL	BDL
Hexachlorocyclopentadiene	BDL	BDL	BDL
isophorone	BDL	BDL	BDL
Pentachlorophenol	BDL	BDL	BDL
Phenanthrene	BDL	0.95	0.28
Pyrene	BDL	1.40	0.06

* Below the Detection Limit

Task 2. Provide testing protocol for SHA's evaluation of materials

During the course of this project, a variety of tests and procedures were employed to ensure that the use of RCA would not impact the water quality, local ecosystem, or any of the life stages of the oysters themselves. The test and procedures employed were standard laboratory and field methodologies that are applicable to any similar material. As alternative materials are introduced in marine environments, SHA needs a testing protocol to assess their potential impacts. The following list provides a standard set of protocols that can be used by SHA to test the suitability of RCA in the marine environment.

1. Planning Stage: Preliminary evaluation for RCA

RCA sampling and preservation: RCA samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction. Alternatively, samples may be frozen (-10°C) in the field or in the laboratory. Samples must be preserved at $4 \pm 2^{\circ}\text{C}$ from the time of sampling and frozen within 48 hours. A summary of sample collection, preservation, and holding times is provided in Table 6.

Table 6. Holding times and preservatives for RCA Samples (MADEP, 2003)

Container	Preservation	Holding Time	
		Extraction	Analysis
250 mL, amber glass jar with Teflon-lined screw cap	Cool to $4 \pm 2^{\circ}\text{C}$	14 days	40 days
250 mL, amber glass jar with Teflon-lined screw cap, Filled to only 2/3 capacity to avoid breakage	Freeze at -10°C	14 days	40 days

At least two duplicate sample collections at the same time, placed under identical circumstances and managed the same throughout field and laboratory procedures, are required to get presentative results. All analysis should be performed at least three times.

Moisture content measurement for RCA sample: Moisture content will affect the result of hydrocarbon concentration. RCA samples should be collected during dry condition.

Extraction and analysis: Two extraction methods, EPA 1316 and 3570, and one analysis method, EPA 8270D, were employed in this project, but other extraction and analysis methods could be adopted based on targeted components.

Data interpretation: The results need to compare with applicable regulations or standards, in order to obtain the permission for field application. However, only a small number of the compounds are well characterized for toxicity. In this research, the results were compared with COMAR 26.08.02.03-2 which provides the numerical criteria for toxic substances in surface

water (Table 1). In the case of a water sample, the results could be compared with EPA drinking water standards. The following table is an example quoted from the EPA drinking water standard related to petroleum industry activity.

Table 7. EPA Drinking Water Standard related to petroleum industry activity

<http://www.epa.gov/your-drinking-water/table-regulated-drinking-water-contaminants>

Chemical	MCLG* (mg/L)	MCL** or TT*** (mg/L)	Potential Health Effects from Long-Term Exposure Above the MCL	Sources of Contaminant in Drinking Water
Ethylbenzene	0.7	0.7	Liver or kidneys problems	Discharge from petroleum refineries
Ethylene dibromide	zero	0.00005	Problems with liver, stomach, reproductive system, or kidneys; increased risk of cancer	Discharge from petroleum refineries
Toluene	1	1	Nervous system, kidney, or liver problems	Discharge from petroleum factories
Xylenes (total)	10	10	Nervous system damage	Discharge from petroleum factories; discharge from chemical factories

Definitions:

*Maximum Contaminant Level Goal (MCLG) – The level of a contaminant in drinking water below which there is no known or expected risk to health. MCLGs allow for a margin of safety and are non-enforceable public health goals.

**Maximum Contaminant Level (MCL) – The highest level of a contaminant that is allowed in drinking water. MCLs are set as close to MCLGs as feasible using the best available treatment technology and taking cost into consideration. MCLs are enforceable standards.

***Treatment Technique (TT) – A required process intended to reduce the level of a contaminant in drinking water.

2. Field Application Stage

RCA selection: When EPA 3570 extraction method was used, no hydrocarbon components were detected for RC-2 (1^{1/2"} to 2^{1/2"}) samples collected from Flanigan & Sons, Inc., while several hydrocarbons components were detected in RC-6 (< 1^{1/2"}) samples collected from Machado Construction Co., Inc. and The Recycling Center. Finer material has higher sorbed surface and higher potential to contain harmful components. Thus, a bigger RCA size such as RC-Surge is recommended.

RCA handling: The RCA origin/source information will be helpful to ease any concern for potential hydrocarbon contamination. Selected RCA should be stockpiled after crushing for at least 14 day to evaporate VOSs.

RCA application: Washing RCA is recommended to remove fine particles on the RCA surface. When applying RCA to the field, RCA should be gently poured into water to minimize disturbing the water system.

After RCA application: Periodical water sampling and analysis are recommended to monitor any detrimental effect to the Chesapeake Bay watershed.

4. CONCLUSIONS

The objective of this study was to determine if the RCA from the roadway surface is appropriate for use as a bottom conditioning material in oyster aquaculture. The major concern was that accidental spills onto the road surface from vehicles could contaminate the pavement and thus the RCA material.

RCA samples from three sites were used. All organic chemical concentrations were BDL (Below Detection Limit) in RCA samples from Flanigan & Sons, Inc. for both extraction methods (Tables 4 and 5). Anthracene, fluorine, phenanthrene and pyrene were detected when EAP 3570 extraction was used in RCA samples from Machado Construction Co., Inc. and The Recycling Center. These detected concentrations, however, were at least 100 times lower than the water quality standard of Maryland (COMAR 26.08.02.03-2). It is safe to conclude that RCA is not a cause for concern for hydrocarbon components leaching when used as a bottom conditioning material for oyster aquaculture.

APPENDIX

A1. EPA 3570 - MICROSCALE SOLVENT EXTRACTION (MSE)

- 1 Add approximately 2.5 grams of anhydrous sodium sulfate to a pre-cleaned PTFE extraction tube which has a PTFE screw cap. Also add 5 to 10 pre-cleaned glass beads.
- 2 Weigh 2 to 3 grams of solids or waste into the tared extraction tube. Do not mix the sample and sodium sulfate at this time. Wipe the lip and threads of the tube with a Kimwipe, or equivalent. Tightly cap, and then record the weight to the nearest 0.01 g.
- 3 Add 50 µg of the surrogate standard compounds in methylene chloride (DCM) directly to the soil. The surrogates recommended are fluorobenzene, 2-fluorobiphenyl, and 5- α -androstane. Other compounds may be used as surrogates, depending upon the desired target analytes and project requirements. If the sample is a matrix spike sample, add 50 µg of the appropriate matrix spike compounds. The surrogate and matrix spike compounds should be at a concentration of 100 µg/mL in the spiking solution.
- 4 Add 12 mL of DCM to the tube, and cap tightly.
- 5 Shake the tubes vigorously until the slurry is free-flowing. Break up any chunks with a metal spatula, working quickly but gently. Cap immediately when finished. Add more sodium sulfate and manually mix as necessary to produce free-flowing, finely divided slurry.
- 6 Extract the samples by rotating end-over-end for at least 4 hours.
- 7 Allow the solids to settle or centrifuge for one to two minutes. Decant or pipet the solvent layer into a small glass funnel containing a layer of anhydrous sodium sulfate over a plug of glass wool. The sodium sulfate should be thoroughly pre-wetted with DCM. Filter the extract into a 25mL Kuderna-Danish (K-D) concentrator tube. Rinse the sodium sulfate with 2 to 3 mL of DCM as soon as the surface is exposed. Do not allow the top of the sodium sulfate layer to go dry.
- 8 Extract the soil twice more by adding approximately 5 mL of DCM to the sample, capping the extraction tube tightly, and shaking vigorously by hand for 2 minutes. Be certain to wipe the lip and threads of the extraction tube with a Kimwipe, or equivalent, before capping each time. More sodium sulfate can be added at this point as necessary to dry the extract and break up any clumps that may have formed.
- 9 After each extraction step, follow step 7.
- 10 Add a Teflon boiling stick to the K-D concentrator tube, and attach one, three-ball micro-Snyder column and one, two-ball micro-Snyder column in series.
- 11 Pre-wet the Snyder columns by adding 0.5 mL of DCM to the top of the column.

12 Place the K-D apparatus in a constant temperature hot water bath so that the concentrator tube is partially, but not completely, immersed. Adjust the temperature of the bath and the position of the apparatus so that the solvent boils evenly, and the micro-Snyder column balls chatter but the chambers do not flood with condensed solvent (approximately 60 to 65 °C).

13 Reduce sample volume to approximately 1.0 mL. Remove and allow to cool and drain for several minutes.

14 Remove the Snyder columns and the boiling stick.

15 Record the exact final volume of the extract. If the volume of the extract does not fall exactly on one of the calibration lines of the concentrator tube, then add enough DCM so that it does, then record that volume.

16 Add an appropriate amount of the internal standard compounds to give a concentration of 50 µg/mL in the extract. Add the internal standard directly to the K-D tube. Transfer the extract to a 2 mL vial fitted with a PTFE lined screw cap. Cap the vial and store in the freezer or over ice until analysis.

A2. Liquid-Solid Partitioning (LSP) as a Function of Liquid-to-Solid Ratio Using a Parallel Batch Extraction Procedure-Method 1316

1 Particle size reduction (if required)

1.1 In this method, particle size reduction is used to prepare large grained samples for extraction so that the approach toward liquid-solid equilibrium is enhanced and mass transport through large particles is minimized.

1.2 Store the size-reduced material in an airtight container in order to prevent contamination via gas exchange with the atmosphere. Store the container in a cool, dark and dry place prior to use.

2 Determination of solids and moisture content

2.1 Place a 5 to 10-g sample of solid material into a tared dish or crucible.

2.2 Dry the sample to a constant mass at 105 ± 2 °C.

2.3 Check for constant mass by returning the dish to the drying oven for 24 hours, cooling to room temperature in a desiccator and re-weighing.

3 Extraction procedure

3.1 Label five bottles with test position numbers and an additional bottle as a method blank

3.2 Place the dry-mass equivalent (± 0.1 g) of sample into each of the five test position extraction vessels.

3.3 Add the appropriate volume (± 0.5 mL) of reagent water to both the test position and method blank extraction vessels.

3.4 Tighten the leak-proof lid on each bottle and tumble all extractions in an end-over-end fashion at a speed of 28 ± 2 rpm at room temperature (20 ± 2 °C).

3.5 Remove the extraction vessels from the rotary tumbler and clarify the extracts by allowing the bottles to stand for 15 ± 5 min. Alternately, centrifuge the extraction vessels at 4000 rpm for 10 ± 2 min.

3.6 For each extraction vessel, decant a minimum volume (approximately 5 mL) of clear, unpreserved supernatant into a clean container.

3.7 Measure and record the pH, specific conductivity, and ORP of the extracts.

3.8 Separate the solid from the remaining liquid in each extraction vessel by pressure or vacuum filtration through a clean 0.45- μm pore size membrane. The filtration apparatus may be exchanged for a clean apparatus as often as necessary until all liquid has been filtered.

3.9 Immediately, preserve and store the volume(s) of eluate required for chemical analysis. Preserve all analytical samples in a manner that is consistent with the determinative chemical analyses to be performed.

Table A.1 Extraction setup

	T1	T2	T3	T4	T5
Target L/S*	10	5	2	1	0.5
Sample 1 (g)	0.53	1.59	2.65	5.30	7.95
Sample 2 (g)	0.54	1.61	2.68	5.35	8.03
Sample 3 (g)	0.57	1.71	2.86	5.72	8.57
water	4.97	7.41	4.85	4.70	3.30
water	4.96	7.39	4.82	4.65	3.22
water	4.93	7.29	4.64	4.28	2.68

***liquid-to-solid ratio**

A.3 GC Run Log information

Vial Number : 2 Inlet: B

GC Run Log

	Time	Event	Value	Source
RUN[0]=	1.80	EvSpl1	100	METHOD
RUN[1]=	4.00	Car2_NR		INTERNAL
RUN[2]=	22.00	EvSpl1	OFF	METHOD

Instrument: inst1

GC CONDITIONS

Method File: C:\MSPROJECTS\MSU.PRO\ACQUDB\01_19_2016.mth

Last Saved : 1/20/2016 9:37:44 AM

Total GC Run Time: 22.5 minutes

Oven	Rate	Temp	Hold
Initial	---	35	1.50
1	18.0	100	0.50
2	22.0	250	0.50
3	32.0	300	8.01

Equil. Time: 0.5 minutes

Coolant: OFF

Max Temp: 327 deg C

Inj A: CAP 0 deg C

Inj B: CAP 0 deg C

Carrier B: PFlow - He

Rate	mL/min	Hold
Initial	---	3 3.50
1	999.0	2 9.00
2	999.0	3 999.00
3	0.0	0 0.00

Split B: 0.0 mL/min Length: 30.00 M

Vacuum Comp: ON Diameter: 250 um

Auxiliary Pneumatics:

NONE

Valves :SPLIT 1 SPLIT 2 NONE 3NONE 4NONE 5NONE 6
Initial: ON ON OFF OFF OFF OFF OFF

Autosampler

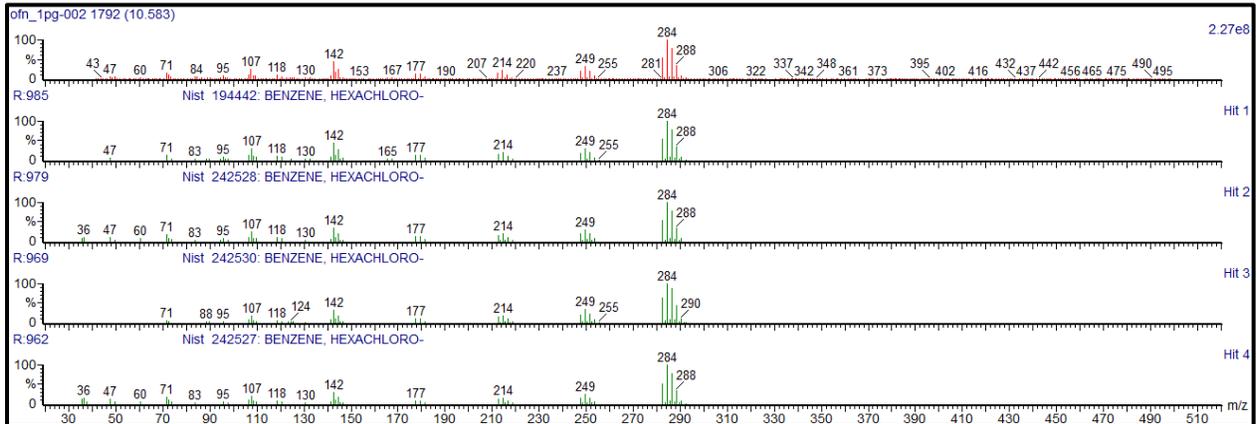
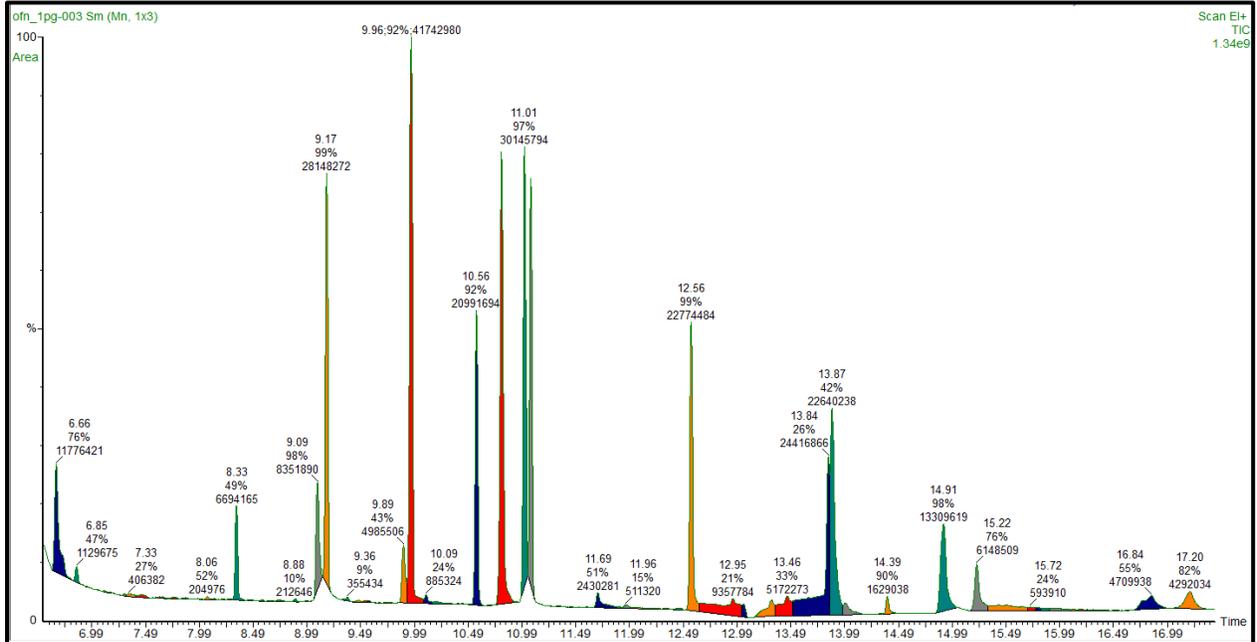
Injection: Syringe: 5.0 Speed: FAST
 Pumps: 4 Visc: 5 W/W Vial Set: 1

Washes: Pre-Inj Solvent: 0Pre-Inj Sample: 1 Post-Inj Solvent: 6

Instrument Timed Events

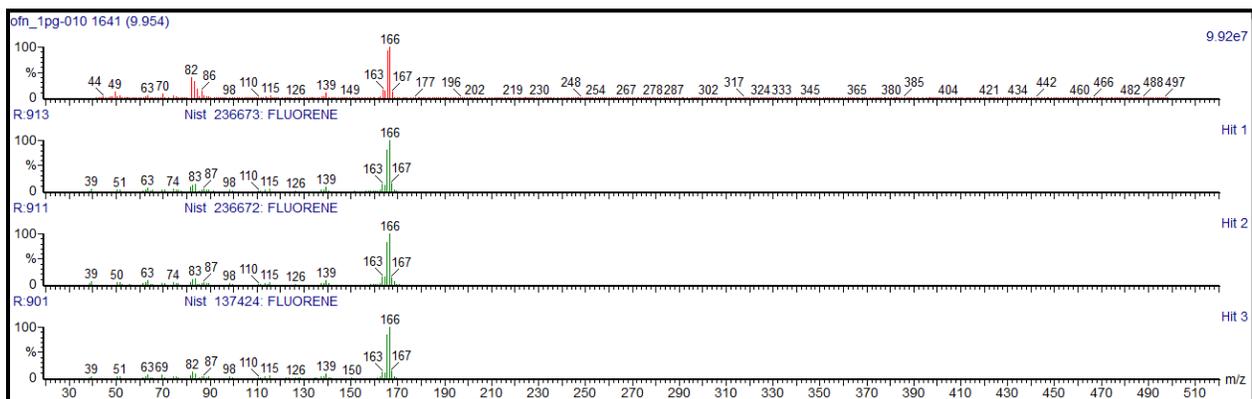
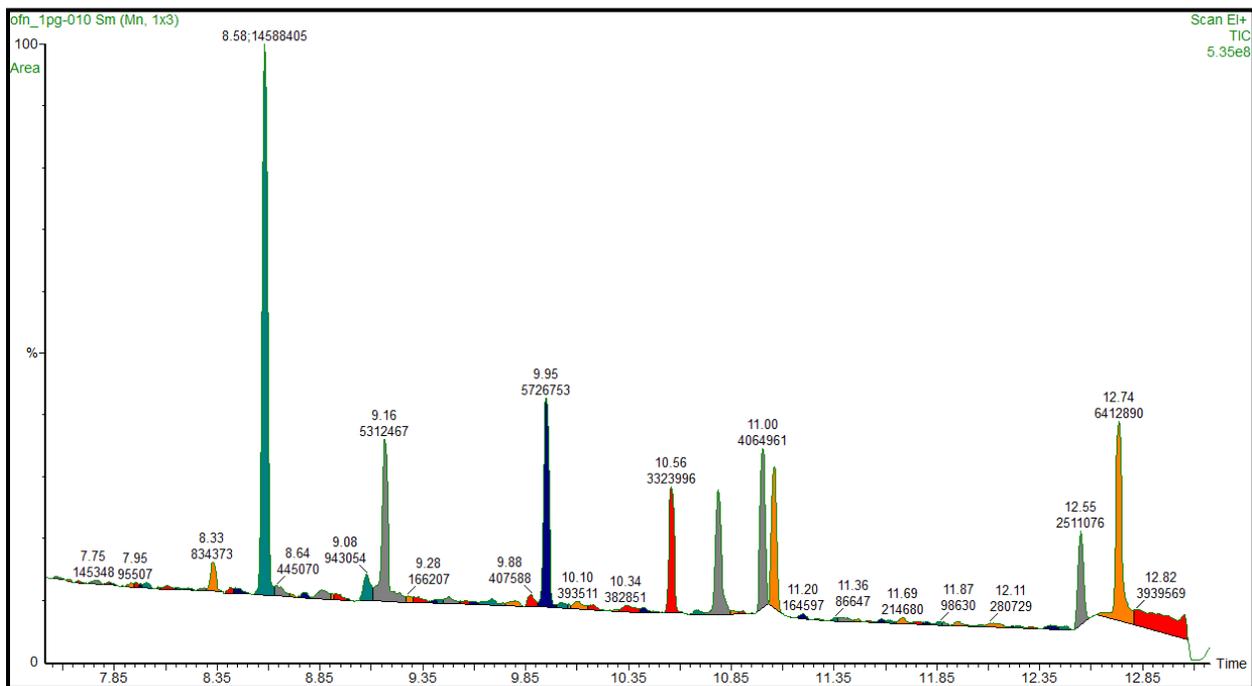
#	Time	Event	Value
1	1.80	SPL1	100
2	22.00	SPL1	0

A 4. Chromatogram / Spectrum



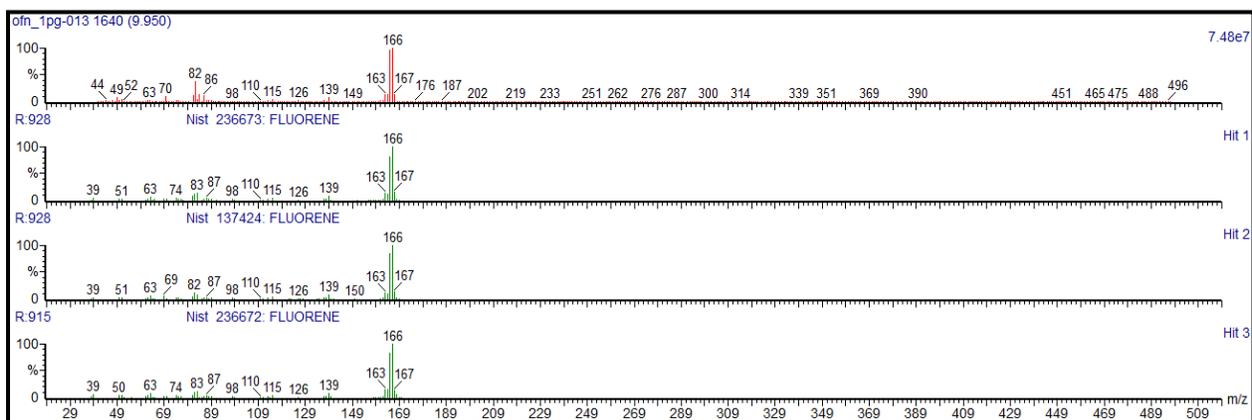
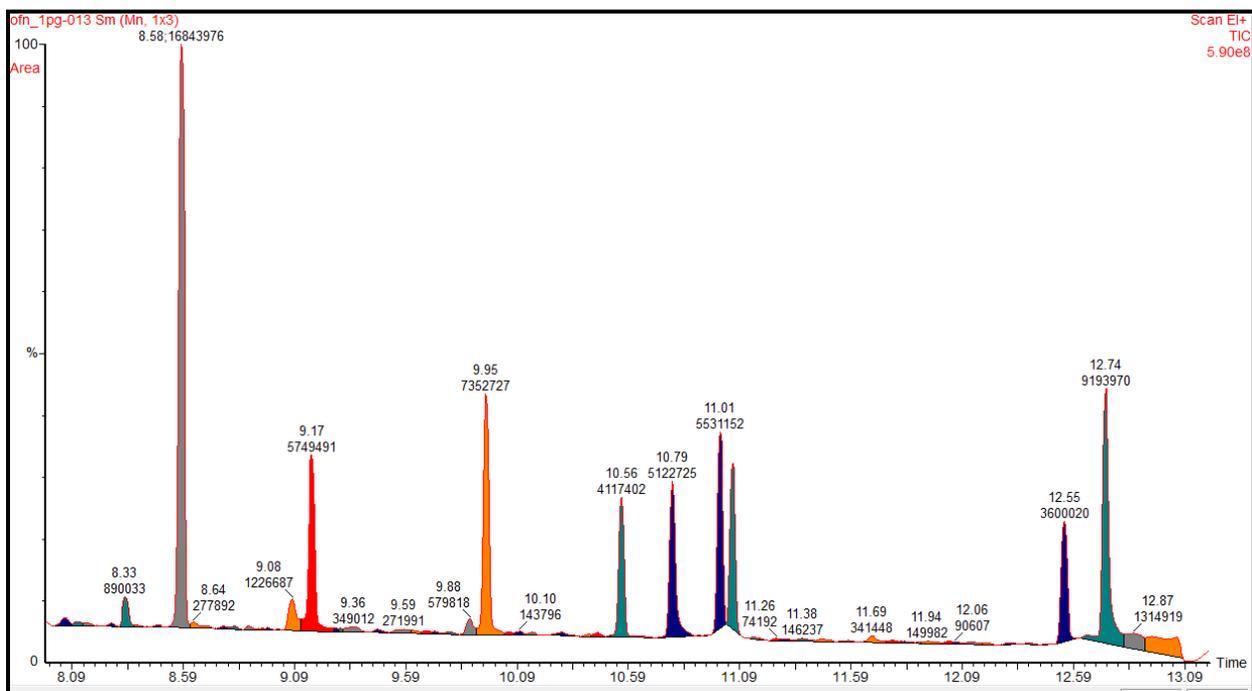
Hit	REV	for	Compound Name	M.W.	Formula	CAS	Library
1	985	961	BENZENE, HEXACHLORO-	282	C6Cl6	118-74-1	Nist
2	979	964	BENZENE, HEXACHLORO-	282	C6Cl6	118-74-1	Nist
3	969	935	BENZENE, HEXACHLORO-	282	C6Cl6	118-74-1	Nist
4	962	946	BENZENE, HEXACHLORO-	282	C6Cl6	118-74-1	Nist
5	950	914	BENZENE, HEXACHLORO-	282	C6Cl6	118-74-1	Nist
6	950	920	1,3-CYCLOPENTADIENE, 1,2,3,4-TETRACHLORO-5-(DICHLOROMETHYLENE)-	282	C6Cl6	6317-25-5	Nist
7	896	862	CYCLOBUTANE, 1,2-DICHLORO 3,4-BIS(DICHLOROMETHYLENE)-	284	C6H2Cl6	55044-46-7	Nist
8	853	789	CYCLOBUTENE, 1,2-DICHLORO 3,4-BIS(DICHLOROMETHYLENE)-	282	C6Cl6	1128-20-7	Nist
9	689	524	1,2,3,4-TETRACHLORO-5-TRIFLUOROMETHYLBENZENE	282	C7HCl4F3	900306-78-6	Nist
10	634	464	PHENOL, 4-CHLORO-2-[(5-CHLORO-2-METHOXYPHENYL)METHYL]-	282	C14H12O2Cl2	55382-50-8	Nist
11	610	443	1,3-ISOBENZOFURANDIONE, 4,5,6,7-TETRACHLORO-	284	C8O3Cl4	117-08-8	Nist
12	605	322	DINAPHTHO[1,2-B:2',1'-D]THIOPHENE	284	C20H12S	239-72-5	Nist
13	600	325	DINAPHTHO[1,2-B:1',2'-D]THIOPHENE	284	C20H12S	207-94-3	Nist
14	600	439	1,3-ISOBENZOFURANDIONE, 4,5,6,7-TETRACHLORO-	284	C8O3Cl4	117-08-8	Nist
15	596	350	DINAPHTHO[1,2-B:1',2'-D]THIOPHENE	284	C20H12S	207-94-3	Nist
16	585	340	DINAPHTHO[1,2-B:1',2'-D]THIOPHENE	284	C20H12S	207-94-3	Nist
17	583	343	DINAPHTHO[2,3-B:2',3'-D]THIOPHENE, 12,13-DIHYDRO-	286	C20H14S	55256-22-9	Nist
18	577	338	DINAPHTHO[1,2-B:2',1'-D]THIOPHENE	284	C20H12S	239-72-5	Nist
19	558	396	1,2-BENZENEDICARBOXYLIC ACID, 3,4,5,6-TETRACHLORO-	302	C8H2O4Cl4	632-58-6	Nist
20	542	319	9H-FLUORENE, 9-DICHLOROMETHYL-3,6-DIFLUORO-	284	C14H8Cl2F2	900195-79-4	Nist

Figure A 4.1 Chromatogram, spectrum and library search result of standard solution (80ug/L)



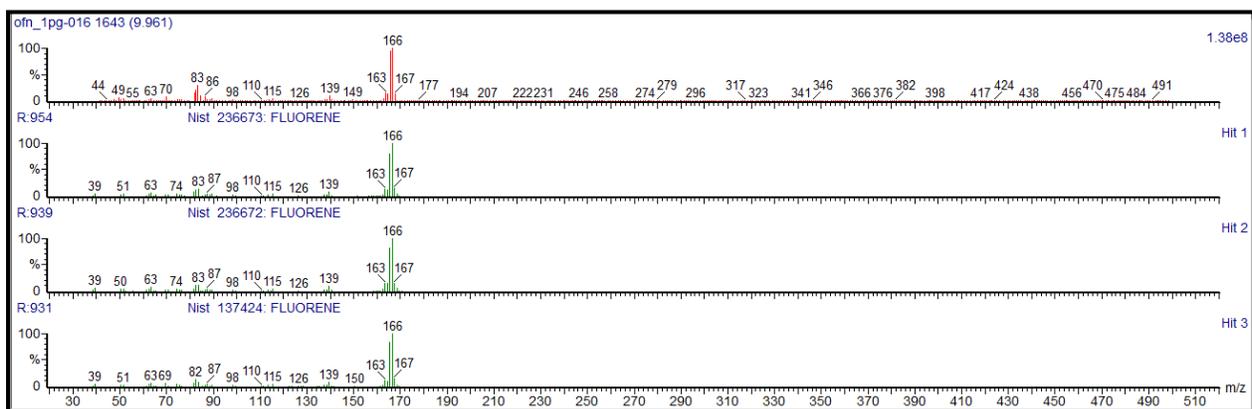
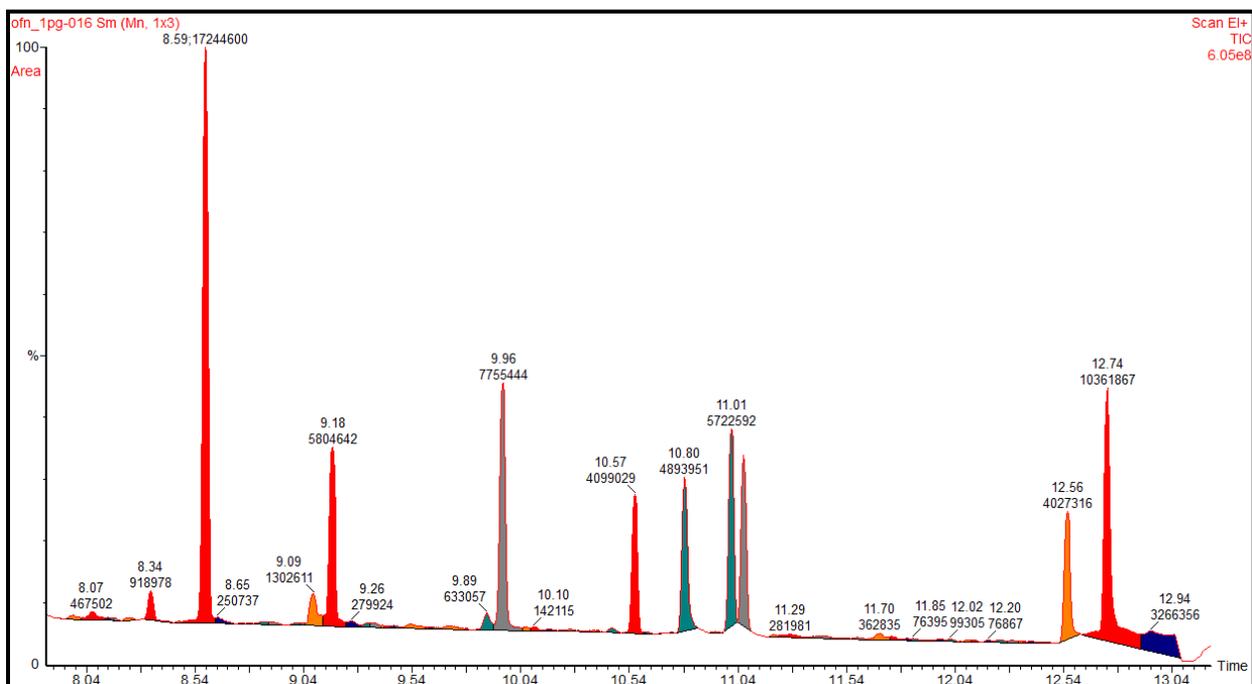
Hit	REV	for	Compound Name	M.W.	Formula	CAS	Library
1	913	840	FLUORENE	166	C13H10	86-73-7	Nist
2	911	839	FLUORENE	166	C13H10	86-73-7	Nist
3	901	814	FLUORENE	166	C13H10	86-73-7	Nist
4	873	827	1H-PHENALENE	166	C13H10	203-80-5	Nist
5	784	701	9H-FLUORENE, 9-BROMO-	244	C13H9Br	1940-57-4	Nist
6	777	661	3-TRIFLUOROMETHYLBENZHYDRYL CHLORIDE	270	C14H10ClF3	67240-79-3	Nist
7	772	629	FLUORENE-9-METHANOL	196	C14H12O	24324-17-2	Nist
8	746	668	FLUORENE, 9-CHLORO-	200	C13H9Cl	6630-65-5	Nist
9	745	657	9H-FLUORENE, 9-BROMO-	244	C13H9Br	1940-57-4	Nist
10	738	660	2-FLUORENECARBOXALDEHYDE	194	C14H10O	30084-90-3	Nist
11	732	588	FLUORENE-9-METHANOL	196	C14H12O	24324-17-2	Nist
12	727	635	9H-FLUORENE, 9-BROMO-	244	C13H9Br	1940-57-4	Nist

Figure A 4.2 Chromatogram, spectrum and library search result (fluorene) of RCA sample collected from Flanigan & Sons, Inc.



Hit	REV	for	Compound Name	M.W.	Formula	CAS	Library
1	928	868	FLUORENE	166	C ₁₃ H ₁₀	86-73-7	Nist
2	928	849	FLUORENE	166	C ₁₃ H ₁₀	86-73-7	Nist
3	915	856	FLUORENE	166	C ₁₃ H ₁₀	86-73-7	Nist
4	875	835	1H-PHENALENE	166	C ₁₃ H ₁₀	203-80-5	Nist
5	836	630	BENZENE, 1,1-(DIAZOMETHYLENE)BIS-	194	C ₁₃ H ₁₀ N ₂	883-40-9	Nist
6	781	694	9H-FLUORENE, 9-BROMO-	244	C ₁₃ H ₉ Br	1940-57-4	Nist
7	773	672	3-TRIFLUOROMETHYLBENZHYDRYL CHLORIDE	270	C ₁₄ H ₁₀ ClF ₃	67240-79-3	Nist
8	771	536	2-AZETIDINONE, 3,3-DIPHENYL-	223	C ₁₅ H ₁₃ ON	15826-12-7	Nist
9	769	677	9H-FLUORENE, 9-BROMO-	244	C ₁₃ H ₉ Br	1940-57-4	Nist
10	768	658	FLUORENE-9-METHANOL	196	C ₁₄ H ₁₂ O	24324-17-2	Nist

Figure A 4.3 Chromatogram, spectrum and library search result (fluorene) of RCA sample collected from Machado Construction Co., Inc.



Hit	REV	for	Compound Name	M.W.	Formula	CAS	Library
1	954	912	FLUORENE	166	C13H10	86-73-7	Nist
2	939	899	FLUORENE	166	C13H10	86-73-7	Nist
3	931	870	FLUORENE	166	C13H10	86-73-7	Nist
4	896	861	1H-PHENALENE	166	C13H10	203-80-5	Nist
5	812	727	9H-FLUORENE, 9-BROMO-	244	C13H9Br	1940-57-4	Nist
6	800	643	FLUORENE-9-METHANOL	196	C14H12O	24324-17-2	Nist
7	783	698	3-TRIFLUOROMETHYLBENZHYDRYL CHLORIDE	270	C14H10ClF3	67240-79-3	Nist
8	777	713	9H-FLUORENE, 9-BROMO-	244	C13H9Br	1940-57-4	Nist
9	769	646	FLUORENE-9-METHANOL	196	C14H12O	24324-17-2	Nist
10	764	710	2-FLUORENECARBOXALDEHYDE	194	C14H10O	30084-90-3	Nist
11	756	687	9H-FLUORENE, 9-BROMO-	244	C13H9Br	1940-57-4	Nist

Figure A 4.4 Chromatogram, spectrum and library search result (fluorene) of RCA sample collected from The Recycling Center.

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