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Authors

Kayhanian, Masoud
Vichare, Akshay
Rasa, Eshan
et al.

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WATER QUALITY AND TOXICITY EVALUATION OF DISCHARGE GENERATED FROM ASPHALT PAVEMENT SURFACING MATERIALS

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Prepared for:

California Department of Transportation
Division of Environmental Analysis
1120 N Street
Sacramento, CA 95814

Prepared by:

M. Kayhanian[1], A. Vichare[1], E. Rasa[1], P. Green[1], C. Alaimo[1], H-M. Hwang[1], J. Signore[2], M. P. Troxler[1, 2], I. Guada[2], F. Farshidi[2], J. Harvey[2]
[1]Department of Civil and Environmental Engineering
One Shields Avenue, Engineering III
University of California
Davis, CA 95616

[2]University of California Pavement Research Center
One Shields Avenue, Engineering III
Davis, CA 95616
and
1353 S. 46th Street
Richmond, CA 94804

THE UNIVERSITY OF CALIFORNIA DAVIS

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Abbreviations

BMP	best management practice
BOD	biochemical oxygen demand
$CaCO_3$	calcium carbonate
Caltrans	California Department of Transportation
COD	chemical oxygen demand
ICP-MS	Inductive Coupled Plasma-Mass Spectrometer
NO_3^-	Nitrate
NO_2^-	Nitrite
NH_3	Ammonia
PO_4^-	Phosphate
SPR	Soluble reactive Phosphate
TKN	Total Kjehldahl Nitrogen
UCD	University of California, Davis
UCPRC	University of California Pavement Research Center
USEPA	United States Environmental Protection Agency

ADA Statement

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Acknowledgement and Disclaimer Statement

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Mention of the names of equipment, products, or chemical supplies and software in this report shall not be construed as an endorsement. Opinions, findings, and conclusions or recommendations expressed in this report are those of the authors and do not necessarily reflect the views of the California Department of Transportation or The Regents of the University of California.

Executive Summary

Pavement engineers are constantly using new materials, such as polymer and rubber modified binders, recycled materials, and new types of asphalt mixes, such as open-graded surface courses. With their broader application, concern has been raised about the water quality of surface and subsurface runoff from modified pavement mixes used in the State of California. There are limitations in current worldwide research into runoff water quality regarding which contaminants are directly attributable to the pavement surfacing materials. This research project aims to provide that information, as part of ongoing stormwater runoff management program from the Division of Environmental Analysis of the State of California Department of Transportation (Department).

This controlled experimental study was undertaken by the Department of Civil and Environmental Engineering and the University of California Pavement Research Center (UCPRC) for the California Department of Transportation (Caltrans), Division of Environmental Analysis (DEA). Major focus of the project was to perform laboratory testing of various pavement surfacing materials currently being used by Department and evaluate the water quality of leachate that passed through or over each pavement. Pavement surfacing material tested include: rubberized asphalt concrete-open graded (RAC-O), rubberized asphalt concrete-gap graded (RAC-G), open graded asphalt concrete (OGAC) and Polymer Modified open graded asphalt concrete (OGAC) (two binder sources each for OGAC and polymer modified OGAC, resulting in four different binders), terminal blend modified binder gap graded (MB-G), dense graded asphalt concrete (DGAC), and portland cement concrete (PCC). For each of these 10 pavement materials, 9 specimens will be prepared for testing at three temperatures of 4, 20 and 45 °C and three replicates each. Additionally, one specimen of each asphalt pavement surfacing material was tested to address the influence of age on water quality.

A representative leachate sample was collected from surface and sub-surface and analyzed for chemical constituents and toxicity. Water quality parameters and chemical constituents tested include: pH, conductivity, turbidity, hardness as $CaCO_3$, TSS, TDS, TOC, As, Cd, Cr, Cu, Pb, Ni, Zn, Fe, Na, Vanadium Total Sulphur, Total Nitrogen, Total Chloride, Total Phosphorous, Semi-volatiles (PAHs), Total Petroleum Hydrocarbons, COD, Oil and grease, and Methylene Blue Active Substances (MBAS). The toxicity of composite leachate samples produced from all pavement specimens were also performed at the UCD toxic lab.

Results obtained from this study showed that:

1. The concentration of most organic and inorganic chemical constituents generated from the leachate of specimens has shown to be below or within the Department reporting limit.
2. Temperature did not significantly change the leachate pollutants concentration.
3. Aging (heat treatment) of the surfacing pavement materials did not contribute to extra leachate pollutant concentration.
4. Acute toxicity based on the survival of the waterflea species, *Ceriodaphnia dubia*, was negligible for nearly all fresh and aged leachate samples. However, toxicity was more noticeable in terms of *C. dubia* reproduction and growth of the green algae, *Selenastrum capricornutum*, particularly for concrete and aged pavement specimens. Toxicity increased with increasing temperature.
5. From this study we can conclude that the toxicity as well as dissolved chemical constituent concentrations (with the exception of Vanadium and Chromium), measured in leachate of all pavement types are generally negligible.

Chapter 1: Introduction

1.1 Background

The definition of the road system that engineers must consider has expanded in recent years beyond the traditional emphasis on infrastructure that moves people and freight from point A to point B at least cost. The interaction of the road with the environment must now be considered, because it is important for sustainability and due to specific legislation such as the Clean Water Act in the USA. At the same time, pavement engineers are using new materials, such as polymer and rubber modified binders and recycled materials, and new types of asphalt mixes, such as open-graded surface courses. Concern has been raised about the water quality of surface and subsurface runoff from rubberized asphalt pavement mixes used in the State of California. There are limitations in current worldwide research into runoff water quality regarding which contaminants are directly attributable to the pavement surfacing materials and which are deposited onto the pavement by vehicles, wind and precipitation. This research project aims to provide that information, as part of ongoing stormwater runoff characterization from the Division of Environmental Analysis of the State of California Department of Transportation (Department). This study was initiated to address some of the issues associated with water quality, including consideration of new materials and mix types being used in California.

The review of existing literature revealed that most studies have focused primarily on quantifying stormwater runoff characteristics from pavements that have been subjected to contamination from various environmental sources. Metals and polycyclic aromatic hydrocarbons (PAHs) were identified, quantified, and categorized in a multitude of independent studies. Four selected examples of this are Kriech (1990), Pitt and Ball et al. (1998), Lalor (2000), and Kayhanian et al (2007). However, the literature depth becomes shallower as the sources of these pollutants are investigated. While motor oil, tire wear, asphalt erosion, tailpipe particulates, and various other elements are identified as affecting stormwater quality, numerical breakdowns of the contribution by each source is rarely made. Moreover, studies focusing on the pollutants leaching from different pavement materials are even rarer, with only a handful of documents broaching the subject. The studies that do exist generally fail to differentiate between portland cement (PC) and asphalt concrete (AC) derivatives, and the chemical constituents that are tested usually are limited to metals and polycyclic aromatic hydrocarbons (PAHs).

The lack of comprehensive information on pavement leachate water quality warranted an independent controlled laboratory research investigation for specific pavement materials. This controlled experimental study was undertaken by the Department of Civil and Environmental Engineering and the University of California Pavement Research Center (UCPRC) for the California Department of Transportation (Caltrans), Division of Environmental Analysis (DEA). This study was specifically performed to measure the potential sources of a wide range of organic and inorganic pollutants produced from pavement materials. This research project differs from traditional stormwater pollution studies in that it focuses solely on contributions from asphalt concrete (AC), portland cement concrete (PCC), and modified AC or PCC mixtures alone, while excluding wide ranges of organic and inorganic chemical constituents from other sources.

1.2 Focus of the Study

The major focus of the study as stated in task order 20 includes:

1. Conduct a national and international literature review to find the latest information regarding constituent runoff from pavement surfacing materials.
2. Develop an experimental plan on the basis of analysis of the literature search results.
3. Perform laboratory testing of various pavement surfacing materials currently being used by Caltrans and evaluate the water quality of leachate that passed through or over each pavement. Pavement surfacing material to be tested include: RAC-O, RAC-G, open graded asphalt concrete (OGAC) and Polymer Modified OGAC (two binder sources each for OGAC and polymer modified

OGAC, resulting in four different binders), terminal blend modified binder gap graded (MB-G), dense graded asphalt concrete (DGAC), and portland cement concrete (PCC). For each of these 10 materials, 9 specimens will be prepared for testing at three temperatures ranging from 0 to 45 °C and three replicates each. Additionally, one specimen of each asphalt pavement surfacing material will be tested to address the influence of age on water quality.

A representative sample collected from surface and sub-surface runoff will be analyzed for chemical constituents and toxicity. Chemical analysis to be performed includes: Department minimum constituents (pH, conductivity, hardness as $CaCO_3$, TSS, TDS, TOC, As, Cd, Cr, Cu, Pb, Ni, Zn, Total Sulphur, Total Nitrogen, Total Chloride, and Total Phosphorous) and other constituents that are known or assumed to be associated with asphalt pavement materials such as: Fe, Na, Sulphur, Vanadium, Semivolatiles (PAHs), Total Petroleum Hydrocarbons (diesel, gasoline, heavy oil), COD, Oil and grease, and Methylene Blue Active Substances (MBAS). The toxicity of selected representative samples will be performed at the UCD toxic lab. All toxicity tests will be conducted based on 7-day survival and reproduction using the standard three USEPA species and test protocol.

4. Prepare final report documenting the findings of objective 1 through 3 stated above.

1.3 Report Organization

This report is organized under seven chapters:

Chapter 1: presents background information, focus of the study and report organization.

Chapter 2: presents the literature review of the past studies.

Chapter 3: presents the experimental plan.

Chapter 4: presents the experimental procedures to conduct the water quality experiments.

Chapter 5: presents the permeability results of the specific pavement materials.

Chapter 6: presents the leachate water quality results for conventionals, nutrients, and metal constituents.

Chapter 7: presents the leachate water quality results for organic constituents.

Chapter 8: presents the leachate water quality results for toxicity.

Chapter 9: presents the list of references cited throughout the report.

Chapter 2: Literature Review

2.1 Purpose of the literature review

The California Department of Transportation (Caltrans) Division of Environmental Analysis (DEA) initiated a control laboratory investigation under Task Order 20 to evaluate highway runoff pollution stemming from various pavement materials mixtures. This research project differs from traditional stormwater pollution studies in that it focuses solely on contributions from asphalt concrete (AC), Portland cement concrete (PCC), and modified AC or PCC mixtures while excluding pollutant loading from other sources. The purpose of this literature review is to identify and examine previously conducted studies that are relevant to the objectives of the current study. Specific objectives of this literature review include:

1. To identify and describe experimental procedures and methodologies used to test pavement materials under controlled conditions.
2. To identify constituents present in leachate from controlled pavement materials.
3. To evaluate leachate constituent concentration limits.

2.2 Overview of Existing Literature

The existing literature paints a comprehensive portrait of the pollutants present in highway stormwater runoff. Metals and polycyclic aromatic hydrocarbons (PAHs), were identified, quantified, and categorized in a multitude of independent studies. Three selected examples of this are Kriech (1990), Pitt and Lalor (2000), and Ball et al. (1998). However, the literature depth becomes shallower as the sources of these pollutants were investigated. While motor oil, tire wear, asphalt erosion, tailpipe particulates, and various other elements are identified as affecting stormwater quality, numerical breakdowns of the contribution by each source was rarely made.

Moreover, studies focusing on the pollutants leaching from pavement materials were even rarer, with only a handful of documents broaching the subject.

2.3 Methodologies and Procedures Used to evaluate leachate in Controlled Pavement Studies

The methodologies for collecting and testing leachate vary widely and depend primarily on the nature of the material being tested. For instance, an experiment on an existing surface might transport the leachate through a pipe into a holding container where it would be tested after a storm event (Sadecki et al., 1996). The soil adjacent to the pavement might also be tested for pollutants (Kriech, 1992). Studies on recycled asphalt pavement (RAP) commonly use a similar piping method, with testing sources being stockpiles of milled RAP instead of an existing pavement surface.

Methodologies that isolate leachate originating from the pavement itself are rarer and are not streamlined. In the few applicable studies available, a common methodology is not defined.

One common thread among the studies is use of the USEPA's toxicity characteristic leachate procedure (TCLP). However this procedure focuses on testing the leachate and does not specify collection methods. The TCLP guidelines are part of the USEPA publication SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. Within SW-846, specific tests for PAHs and metals are detailed.

Experimental setups, test procedures, and appropriate sample collections associated with four reported studies are briefly described below.

The experimental setup for the Sadecki et al. study (Figure 2-1) is indicative of other RAP studies. Generally, RAP is stored in windrows and is used as a fill for various applications. Leachate is collected through a system of pipes and catchments. This type of leachate collection system is useful because it collects actual field data.

However, because RAP is adulterated by contaminants from nonpavement sources (i.e., crankcase oil), this method is not applicable for experimental use in Task Order 20.

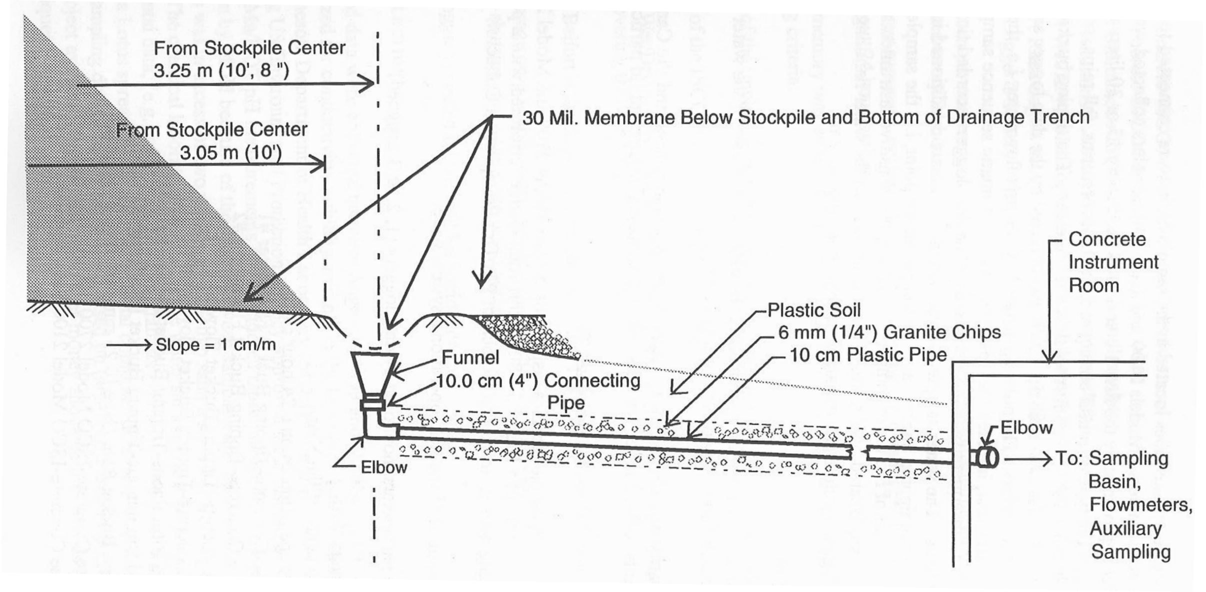


Figure 2-1 Experimental setup for leachate collection from a RAP pile (Sadecki et al., 1996)

Vashisth et al. (1997) conducted a two-part study to test for asphalt leachate: (1) using a Teflon-modified particle entrainment simulator (PES, see Figure 2), and (2) using naturally modeled conditions. In the first setup, established to determine metal levels in leachate under extreme conditions, cylindrical specimens were placed in a modified PES and water was forced into the voids through an oscillating disc. Teflon was added to the equipment in order to create an inert environment. This procedure was intended to find metal levels in leachate under extreme conditions.

The second model involved a more natural testing system in which specimens were artificially aged and worn, then subjected to simulated rainfall at a rate of 125 mm over 30 minutes. This experimental model is closely aligned with the goals of Task Order 20. Both of these models used a variety of deionized water pH levels to flush out contaminants. Results indicated that both highly acidic and highly basic pH levels increased metal leachate concentrations.

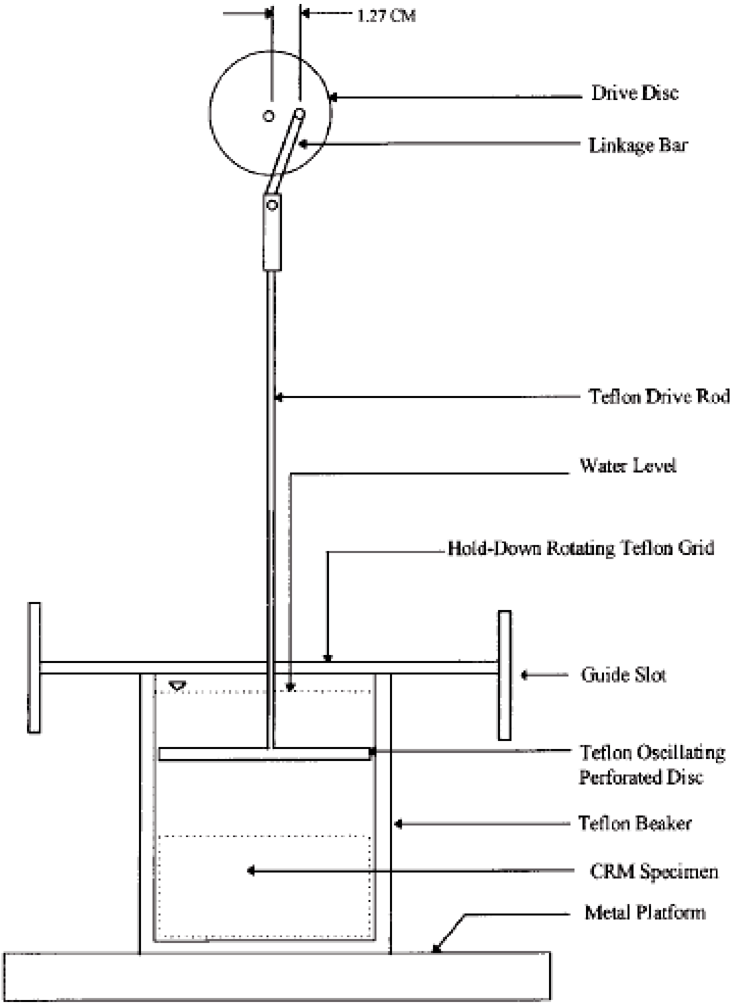


Figure 2-2 A modified particle entrainment simulator (PES) (Vashisth et al., 1997)

Vashisth et al. (1997) also attempted to account for trafficking in the experiments by making specimens “arbitrarily subjected to 20 cycles of compaction” using a “steel wheel mobile compactor.” Unfortunately, no more detail is given concerning traffic wear and compaction, thereby conveying only a vague understanding of the processes involved.

Byrne et al. (1980) used a simple procedure to test for petroleum hydrocarbons in which deionized water was run over a sheet of asphalt (see Figure 2-3). The asphalt sheet, measuring 36 cm^2, was positioned at an angle of 23 degrees downwards. Leachate was collected in a glass bottle for further testing. Rainfall was simulated by subjecting the asphalt to a flow rate of 200 ml/min for the first hour, then 50 ml/min until the end of the experiment. Temperatures of 4°C and 60°C were used to account for winter and summer conditions.

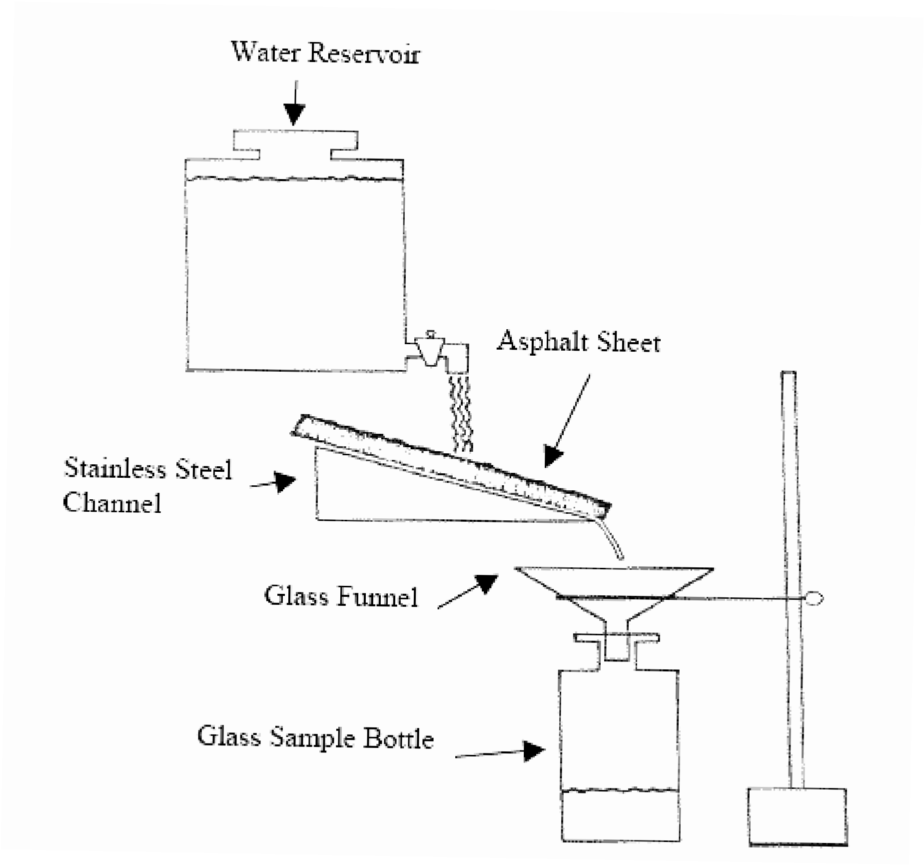


Figure 2-3 Experimental setup for Byrne et al. (1980)

Townsend and Brantley (1998) retrieved leachate from RAP specimens by two methods: (1) soaking and rotating, and (2) column leaching using a lysimeter (see and Figure 2-3). The former method involved placing a 100-gram sample into a two-liter glass jar filled with “an appropriate leaching solution,” rotating the sample

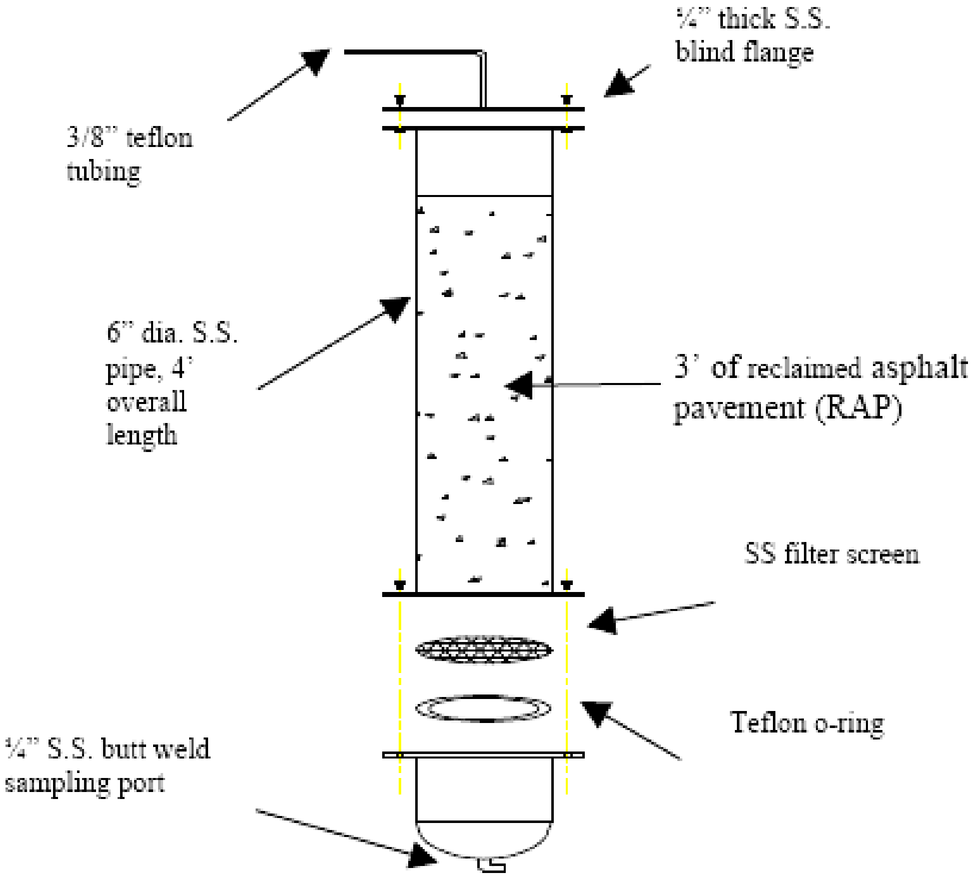


Figure 2-3 A lysimeter used by Townsend and Brantley (1998)

for a period of roughly 18 hours, then testing the contaminant levels in the residual liquid. The latter method better simulates realistic environmental conditions: a leaching solution is fed into the lysimeter — a tool that Harrison and Wilson (1985) assert is ideal for testing the "first flush" phenomenon — under both saturated and unsaturated conditions. Townsend and Brantley state "there is currently no standard methodology for a leaching column", therefore requiring them to create a procedure to "meet the needs of the study." The procedure that was followed involved pumping an aqueous solution into the top of the lysimeter and collecting the resulting leachate after the solution worked its way through the specimen. The apparatus was covered in an inert material to prevent external contaminants from entering the leachate.

Figure 2-4 Picture of the lysimeters used by Townsend and Brantley (1998)

In addition to the above studies, Kriech (1992) took trafficking effects into account by taking leachate samples from five different PCC and AC sections: (1) uncontaminated/untrafficked segments, (2) transverse across the pavement segments, (3) between the wheelpath (longitudinal) segments, (4) in the wheelpath (longitudinal) segments, and (5) outside the wheelpath (longitudinal) segments. The sections abutted one another and were subjected to the same traffic loading for 15 years. Results indicated that trafficking did not affect the leachate for either PAHs or metals at a detectable level.

2.4 Constituents tested in controlled Pavement Materials studies

The limited research that has been done on leachate from pavement materials is generally focused on PAHs and metal concentrations in the runoff. Anthony Kriech, then of the Heritage Research Group in Indiana, published three articles in consecutive years beginning in 1990 on the subject of leachate from pavement materials. Although the conclusions and analyses are limited, this triad of papers serves as a baseline for identifying the constituents that pavement materials contribute to highway stormwater runoff.

Kriech tested for five groups of pollutants: PAHs, polychlorinated biphenyls, semivolatile organics, VOCs, and metals. Most constituent concentrations were below the detection limits. Barium (metal), chromium (metal) and napthalene (PAH) were the only constituents measured above the detection levels. Kriech (1992) concluded that “PCC and hot-mix asphalt (HMA) pavements each have very low levels of leachable metals and PAH materials.”

Vashisth et al. (1997) also looked at the leachability of pavements, but the focus of the research was on crumb-rubber modifiers (CRM) in HMA pavements. Control (no rubber), wet-process, and dry-process specimens were created and tested for metal concentrations in their leachate. Overall, the results showed higher metal concentrations in the control specimens compared to the Kriech experiments. Varying asphalt temperatures and input water pH values were also tested, with the results being highly sensitive to both of those variables showing slightly higher metal concentrations at lower pH and higher temperature.

Brandt and De Groot (2001) investigated PAH leachate from various commercially available binders from different locations and manufacturers. The constituent concentrations among the binder leachates often ranged several orders magnitude, leading one to believe that the binder source has a large effect on the constituents that may be present.

Brandt and De Groot also created asphalt concrete (AC) from one of the binders and tested the resulting leachate. Overall, the leachates from the AC and the binder alone had very low detectable concentrations of PAH. Although, as expected, the concentrations of PAHs from the binder were generally detected, however, the detected concentration were well below the limit of water quality standard.

Table 2-1 and Table 2-2 present the results of some of the studies described above for PAH and metals, respectively. The results indicate that most PAHs were not detectable in leachate while metals are found at or above detection limits. In addition, the results show that different mix designs and additives (in this case, CRM) can greatly alter some constituent levels in leachate. However, neither of these variables is documented in either great breadth or depth in the literature, leading to the conclusion that further research is needed in this area. Compared to limited previous studies, Task Order 20 proposes testing leachate for a wider variety of potential constituents, which are summarized in Table 2-3.

Table 2-1 PAHs in Leachate (ng/L)

PAH	*Kreich (1990)* HMA	*Kriech (1992)* HMA	*Kriech (1992)* PCC	*Brandt and De Groot (2001)* Asphalt	*Brandt and De Groot (2001)* Bitumen
Acenaphthene	<194	<160	<160	0.8–1	1.3–17
Acenaphthylene	<150	<250	<250		
Anthracene	<15	<21	<21	0.1–0.3	0.1–28
Benz(a)anthracene	<48	<13	<13	<0.05–0.2	<0.05–1.4
Benzo(a)pyrene [B(a)P]	<23	<23	<23	<0.02–0.2	<0.02–0.3
Benzo(b)fluoranthene	<20	<29	<29	<0.01–0.2	<0.01–0.4
Benzo(g,h,i)perylene	<36	<140	<140	<0.02–0.2	<0.02
Benzo(k)fluoranthene	<22	<13	<13	<0.02	<0.02–0.2
Chrysene	<17	<41	<41	0.09–0.2	<0.06–5.3
Coronene				<0.03	<0.03
Dibenzo(a,h)anthracene	<18	<85	<85	<0.04–0.1	<0.04
Fluoranthene	<37	<21	<21	<0.05–0.08	0.4–1.7
Fluorine	<23	<19	<19	<0.02–1.9	2.1–44
Indeno(1,2,3-cd)pyrene	<21	<28	<28	<0.1	<0.1
Naphthalene	250	<160	<160	33–52	35–371
Phenanthrene	<33	<160	<160	1.2–2.5	4.1–180
Pyrene	<40	<75	<75	0.1–0.03	0.4–4

Note: Values shown with < symbol is the detection limit

Table 2-2 Metals in Leachate (µg/L)

Metal	*Kriech (1990)* HMA	*Kriech (1992)* HMA	*Kriech (1992)* PCC	*Vashisth et al (1997)* DGAC	*Vashisth et al (1997)* CRM (dry)	*Vashisth et al (1997)* CRM (wet)
Arsenic	<5	<5	<5			
Barium	<2000	2900	<2000			
Cadmium	<20	<20	<20	0.5	0.2	0.4
Chromium	100	<10	72	0.5	1.1	0.5
Copper				0.4	0.4	0.5
Lead	<200	<200	<200	ND	ND	2.1
Mercury	<5	<5	<5			
Nickel				0.8	0.8	0.7
Selenium	<5	<5	<5			
Silver	<40	<40	<40			
Zinc				5	2.5	11.5

ND = nondetectable concentration

Note: Values shown with < symbol is the detection limit

Table 2-3 Task Order 20 Water Quality Parameters and Constituents

Parameter/Constituent	Symbol	Unit
Conventional		
Electric conductivity	EC	µs/cm
Hardness	Hardness as $CaCO_3$	mg/L
pH	pH	pH unit
Aggregate		
Chemical oxygen demand	COD	mg/L
Dissolved organic carbon	DOC	mg/L
Oil and grease	O&G	mg/L
Total dissolved solids	TDS	mg/L
Total organic carbon	TOC	mg/L
Total petroleum hydrocarbons	TPH	µg/L
Total suspended solids	TSS	mg/L
Metals (total and dissolved)		
Arsenic	As	µg/L
Cadmium	Cd	µg/L
Chromium	Cr	µg/L
Copper	Cu	µg/L
Iron	Fe	µg/L
Nickel	Ni	µg/L
Lead	Pb	µg/L
Vanadium[a]	V	µg/L
Zinc	Zn	µg/L
Nutrients		
Total Phosphorus	Total P	mg/L
Total Nitrogen	Total N	mg/L
Minerals		
Sodium	Na	mg/L
Total Sulphur	S	mg/L
Total Chloride	Cl	mg/L
Organics	PAHs	µg/L
Methylene Blue Active Substance	MBAS	mg/L
Toxicity	Tox	% survival

[a] Vanadium was added based on the suggestion from Caltrans pavement research group.

2.5 Leachate Constituent Concentration Limits

Different standards exist that can be used to evaluate the environmental significance of leachate constituent concentrations. For example, different standards are applied when evaluating concentrations in drinking water and water considered safe for aquatic organisms. Because of this uncertainty, unless a reference uses a particular standard as a comparison benchmark within the document, this literature review will not compare published results with any proposed standards such as California Toxic Rule limits (CTR) (Hwang et al, 2005). If adequate detectable water chemistry data could be collected from this experimental study, the results obtained may provide an opportunity to perform comparative analyses with CTR or other discharge limits requirements associated with existing TMDL. This aspect of the results will be discussed in Chapter 7 and 8.

Townsend and Brantley (1998)

Townsend and Brantley (1998) used PAH "guidance concentrations" from the Florida Department of Environmental Protection (DEP) as a comparison standard. However, since the publication of Townsend and Brantley, the document containing the guidance concentrations has been pulled from the Florida DEP website because "it is outdated and, for that reason, is no longer being distributed" (Florida, 2006). Moreover, since the Townsend and Brantley study focused only on RAP samples, the comparative results are not appropriate for this literature review.

Vashisth et al (1997)

Vashisth et al (1997) compared their measured metal concentrations with the USEPA's aquatic life criteria (based on three different hardness levels: 50 mg/L, 100 mg/L, and 150 mg/L as calcium carbonate) and drinking water standards. The maximum observed concentrations for chromium, nickel, and lead exceeded neither the aquatic life nor the drinking water criteria. Conversely, copper, cadmium, and zinc concentrations breached at least one of the standards.

Using acidic inflow water (pH = 2.0), the measured copper concentrations exceeded the aquatic life criteria for a hardness value of 50 mg/L. This low pH was selected to ensure the detection of leachate soluble metal under worse case scenario. It is important to note that the pH of rainwater in California is in the range of 5 to 6 and hence adjusting pH below 5 is unrealistic. The maximum concentration under this condition was measured to be 13 µg/L, which is slightly above the threshold of 9 µg/L and 7 µg/L for acute and chronic aquatic life criteria. Under all the other conditions the copper concentration was below all of the standards. The drinking water standard was never violated under any condition.

Cadmium concentrations were more problematic, with maximum concentrations exceeding all of the standards (all aquatic life hardness values and the drinking water standard) using acidic inflow water. The maximum cadmium concentration of 13 µg/L is compared to the drinking water standard of 5 µg/L and the aquatic life criteria ranging from 1 µg/L to 6 µg/L.

Zinc concentrations were found to exceed the aquatic life criteria (ranging from 108 µg/L to 300 µg/L) under several input conditions, but never superseded the significantly higher drinking water standard (5000 µg/l). CRM asphalt using the dry process produced the highest zinc concentrations, with measured levels ranging from 139 µg/L to 1,765 µg/L for acidic inflow water and 147 µg/L to 725 µg/L for basic inflow water. For both inflows, the concentrations were for 20°C and 45°C, respectively.

While the experiment was designed to test extreme conditions, the authors noted that the results represent a worst-case scenario that omits certain natural actions, such as dilution with uncontaminated stormwater. It is also important to note that under normal inflow water all of the metals were below the aquatic life criteria and drinking water standards as reported in the study.

2.6 Review Summary

The existing literature on constituent concentration in pavement leachate focuses primarily on quantifying stormwater runoff composition that has been subjected to contamination from various environmental sources. Very few projects have investigated the pollutant contribution of the pavement materials themselves to the runoff. The studies that do exist generally fail to differentiate between PC and AC derivatives. The lack of relevant literature in this area warrants an investigation into the leachate from specific pavement materials. This specific topic was addressed through this research project.

Chapter 3: Experimental Plan and Testing Apparatus

To evaluate the water quality of leachate produced from pavement specimens, an experimental plan was developed to conduct the water quality testing. This section of the report is organized to describe the experimental plan and the apparatus used to conduct water quality experiments under controlled laboratory conditions.

The objective of this experimental study was to test the water quality and toxicity of runoff obtained from different permeable pavement and asphalt samples and to investigate and remedy the shortcomings of earlier research. Earlier research identified various possible external contamination sources but failed to study the contamination impact of the pavements themselves (Booth et al .2004). Also, in these studies the impact of differing geographical conditions and pavement materials was not studied, hence in this study, the tests were carried out at three different temperatures, so as to simulate the entire range of temperature conditions found in California, and ten characteristically different specimens were identified and tested, in triplicate. The earlier research also focused on individual contaminants. No comprehensive study of contaminants was found in the literature review, hence this study developed a comprehensive list of contaminants, which were analyzed using USEPA methods and are compared to the current reporting limit. The experiment was thus planned to provide information on contaminants detected at the representative temperatures and pavement materials, in triplicate, so as to provide a statistically reliable and accurate data. The experiment was carried out as per the experimental plan shown in Table 1.

Major components of experimental plan include: selection of water quality parameters, runoff volume calculation, selection of testing water, preparation of pavement specimen, pavement specimen aging, constant temperature chamber, and fabrication of experimental apparatus.

Table 3-1 Experimental testing plan

Specimen Type #	Mix Type	Binder	Temperature (C)	Base Experiment Replicates	Aging Test
1	RAC-O Rubberized Asphalt Concrete Open-graded	Rubberized asphalt cement	4	3	1
			20	3	
			45	3	
2	RAC-G Rubberized Asphalt Concrete Gap-graded	Rubberized asphalt cement	4	3	1
			20	3	
			45	3	
3,4	OGAC Open-graded Asphalt Concrete	2 Unmodified Performance Grade (PG) binders	4	3	1
			20	3	
			45	3	
5,6	OGAC-PM Open-graded Asphalt Concrete Polymer Modified	2 Modified PG binders	4	3	1
			20	3	
			45	3	
7	MB-G Modified Asphalt Binder	MB4 Binder	4	3	1
			20	3	
			45	3	
8	DGAC Conventional Dense-graded Asphalt Concrete	(PG) Binder – Unmodified	4	3	
			20	3	
			45	3	1
9	PCC-Dense	Portland cement	4	3	1
			20	3	
			45	3	
10	PCC-Permeable	Portland cement	4	3	1
			20	3	
			45	3	

3.1 Selection of Water Quality Parameters

A list of water quality parameters to be tested under this research project is summarized in Table 3-2. As shown, the water quality parameters are organized as conventional (pH, hardness, and electric conductivity), aggregates (COD, DOC, TOC, O&G, TSS, TDS, TPH), total and dissolved metals (As, Cd, Cr, Cu, Fe, Ni, Pb, Va, Zn), nutrients (total N and total P), minerals (Na, S, Cl), PAHs, methylene blue active substance (MBAS), and Toxicity. Also reported on this table are the respective reporting limits, analytical methods, holding time, and sample volume needed to complete the chemical analysis and toxicity testing.

Table 3-2 List of water quality parameters and chemical constituents, reporting limit, analytic holding time

Water quality parameters and constituents	Symbol	Unit	Reporting Limit	Analytical Method	Holding Time	Volume
Conventional						
Electric conductivity	EC	µs/cm	0.1	120.1	Immediate	50ml
Hardness	Hardness as $CaCO_3$	mg/L	2	130.2	6 month	10ml
pH	pH	pH unit	0.1	150.1	Immediate	50ml
Aggregate						
Chemical oxygen demand	COD	mg/L	1	410.4	7 days	5ml
Dissolved organic carbon	DOC	mg/L	1	415.1	28 days	100ml
Oil and grease	O&G	mg/L	5	1664	28 days	1L
Total dissolved solids	TDS	mg/L	1	160.1	7 days	1L
Total organic carbon	TOC	mg/L	1	415.1	28 days	100ml
Total petroleum hydrocarbons	TPH	µg/L	50	8015b	14 days	2L
Total suspended solids	TSS	mg/L	1	160.2	7 days	1L
Metals						
Arsenic	As	µg/L	1	206.3	6 months	10ml
Cadmium	Cd	µg/L	0.2	200.8	6 months	10ml
Chromium	Cr	µg/L	1	200.8	6 months	10ml
Copper	Cu	µg/L	1	200.8	6 months	10ml
Iron	Fe	µg/L	25	200.9	6 months	10ml
Nickel	Ni	µg/L	2	200.8	6 months	10ml
Lead	Pb	µg/L	1	200.8	6 months	10ml
Vanadium	V	µg/L	□0.5	200.8	6 months	10ml
Zinc	Zn	µg/L	5	200.8	6 months	10ml
Nutrients						
Total Phosphorus	Total P	mg/L	0.03	365.2	28 days	10ml
Total Nitrogen	Total N	mg/L	0.1	351.3	28 days	10ml
Minerals						
Sodium	Na	mg/L	1	200.7	6 months	10ml
Total Sulphur	S	mg/L	0.1	ICPMS	30 days	10ml
Total Chloride	Cl	mg/L	0.1	300.0	28 days	10ml
Organics	PAHs	µg/L	1	8310	7 days	2L
Methylene Blue Active Substance	MBAS	mg/L	1	5540C	48 hours	250ml
Toxicity	Tox	% Survival	--	EPA three species	36 hours	16L

All chemical and toxicity testing were performed according to the procedures specified in the Caltrans Monitoring Protocol: Comprehensive Guidance Manuals (CTSW-RT-02-017). Several constituents that are not currently addressed in the Department comprehensive guidance manuals (such as MBAS, Na, S, and Cl) were also considered for this research project. These constituents were analyzed using methodology approved by the California Department of Heath Services for environmental testing. The toxicity tests were performed using the standard US EPA freshwater species. The toxicity tests were done at the UCD Aquatic Toxicity Laboratory. The total nitrogen analyses were done at the DANR laboratory. All other chemical analysis were performed at the UCD CEE Environmental Laboratory

3.2 Runoff Volume Calculation

To perform the water quality evaluation, a water flow rate was calculated using an equivalent rainfall of 0.25 cm (0.1 inches) per hour for two consecutive days (48 hours) over the surface area of the specimen. This flow rate was chosen, as it is representative of storm events that pavements would have to endure in natural conditions. The total volume of water expected during this period was estimated to be about 16 Liters. However, as shown in Table 3-2, it was determined that this volume of water will not be insufficient for completing the entire suite of chemical analyses. The needed volume for quality assurance/quality control of about 30 Liters (7.9 gal) would be required for performing all analytical and toxicity tests. Therefore, the latter amount was selected as the basis for computing inflow rate. Thus a back calculated flow rate of 0.63 L/hr (0.165 gal/hr) was determined. This flow volume of 30 Liters over the 48 hours time period would flush an open-graded AC specimen, with 20% air void content (1835cc), approximately 16 times. For the denser specimens, the water flow will be almost all sheet surface flow.

3.3 Selection of Inflow Test Water

Initially we planned to prepare synthetic rainwater by adjusting the pH of Milli-Q water and add some minerals to make it more closely resemble rainwater. However, initial toxicity testing of this synthetic rainwater showed toxic effects and hence made it more difficult to assess the toxicity of leachate from each specimen. For this reason, all inflow water used was Milli-Q water (highly purified de-ionized water), so as to eliminate any possible contamination prior to each test. Chemical analysis of Milli-Q water is presented in Table 3-3.

Table 3-3 Summary of Milli-Q water chemical and toxicity analysis

Water quality parameters and constituents	Symbol	Unit	Reporting Limit	Value
Conventional				
Electric conductivity	EC	μs/cm	0.1	0.55
Hardness	Hardness as $CaCO_3$	mg/L	2	0.3
pH	pH	pH unit	±0.1	5
Aggregate				
Chemical oxygen demand	COD	mg/L	1	0.02
Dissolved organic carbon	DOC	mg/L	1	480
Oil and grease	O&G	mg/L	5	ND
Total dissolved solids	TDS	mg/L	1	1370
Total organic carbon	TOC	mg/L	1	480
Total petroleum hydrocarbons	TPH	μg/L	50	ND
Total suspended solids	TSS	mg/L	1	ND
Metals (total)				
Arsenic	As	μg/L	1	0.01
Cadmium	Cd	μg/L	0.2	0.02
Chromium	Cr	μg/L	1	0.3
Copper	Cu	μg/L	1	1.5
Iron	Fe	μg/L	25	4
Nickel	Ni	μg/L	2	0.3
Lead	Pb	μg/L	1	1
Vanadium	V	μg/L	□0.5	0.02
Zinc	Zn	μg/L	5	9
Nutrients				
Total Phosphorus	Total P	mg/L	0.03	0.002
Total Nitrogen	Total N	mg/L	0.1	ND
Minerals				
Sodium	Na	mg/L	1	0.3
Total Sulphur	S	mg/L	0.1	0.001
Total Chloride	Cl	mg/L	0.1	0.1
Organics	PAHs (total)	μg/L	0.05	ND
Methylene Blue Active Substance	MBAS	mg/L	1	ND
Toxicity	Tox	% survival	--	100

ND = non-detect

3. 4 Preparation of Pavement Specimens

Test specimens were prepared at the University of California Pavement Research Center (UCPRC) at the Richmond Field Station (RFS) laboratory. Table 3-2 shows 10 specimens with their mix types and binder materials, which were used for the experimental procedure.

Table 3-4 Specimens mix type and binder materials

Specimen	Mix Type	Binder
A	RAC-O Rubberized Asphalt Concrete Open-graded	Rubberized asphalt cement
B	RAC-G Rubberized Asphalt Concrete Gap-graded	Rubberized asphalt cement
C,D	OGAC Open-graded Asphalt Concrete	2 Unmodified Performance Grade (PG) binders
E,F	OGAC-PM Open-graded Asphalt Concrete Polymer Modified	2 Modified PG binders
G	MB-G Modified Asphalt Binder	MB4 Binder
H	DGAC Conventional Dense-graded Asphalt Concrete	(PG) Binder - Unmodified
I	PCC-Dense	Portland cement
J	PCC-Permeable	Portland cement

Specimens based upon these mix designs were prepared using standard ingot molds and rolling wheel compaction. Ingot molds were lined with non-contaminating materials and form release agents were not used. Portland cement mixes were placed and rodded /vibrated. The aggregate used in each specimen was primarily obtained from a single source, namely- Syar Industries, Inc., in Vallejo, California. Aggregate samples with representative gradations, not mixed with asphalt, were also tested to identify differences caused by the mineralogy of the aggregate sources and the difference in aggregate materials was noted. Figure shows the dimensions for the typical specimen used in this research project.

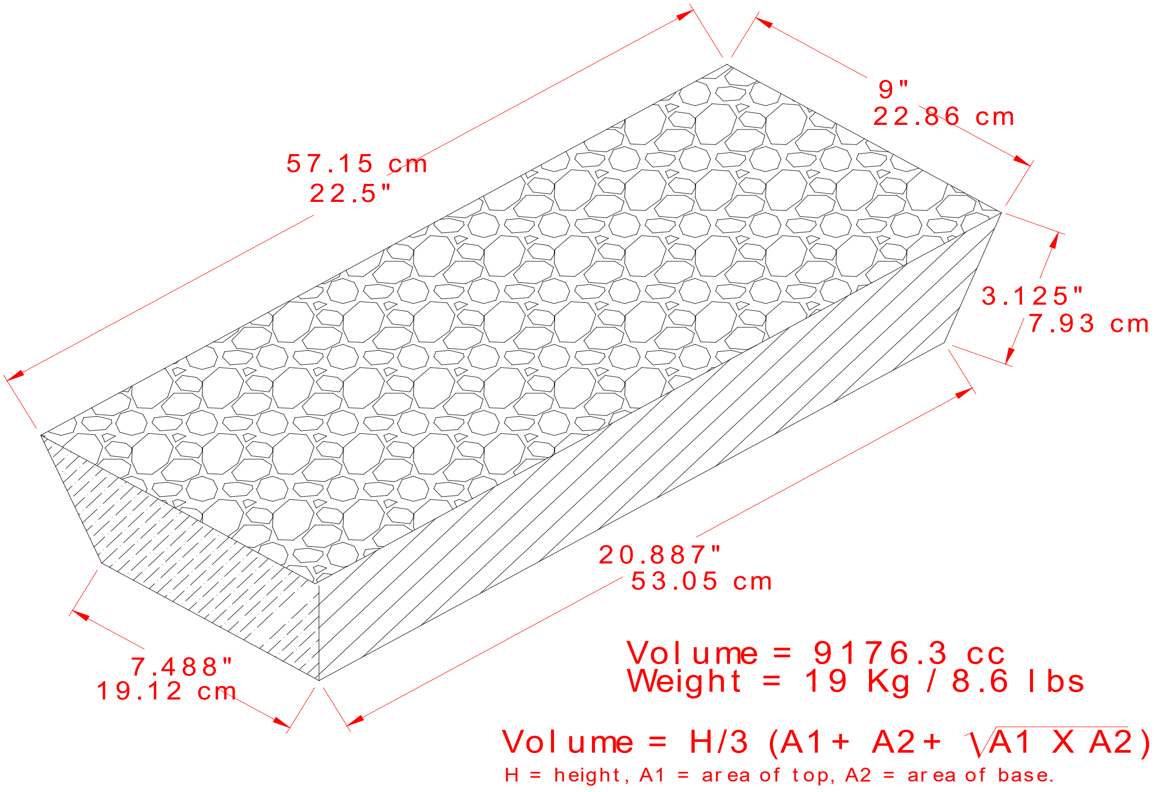


Figure 3-1 Test Specimen Dimension

3.5 Pavement Specimen Aging

One specimen from each mix type and binder material was prepared to determine the impact of aging on the chemical properties of the leachate. The effect of aging was evaluated by placing a full-size specimen of each type in a forced-draft oven at 85°C for six days prior to testing. This aging process was developed by Federal Highway Association and the Transportation Research Board through a national research effort in 1990s under Strategic Highway Research Program (SHRP) and is widely accepted in pavement research. This type of simulated aging stiffened the asphalt binder through oxidation and volatilization of the lighter constituents in the asphalt. This accelerated aging is representative of approximately 15 to 18 years of in-service pavement life.

3.6 Constant Temperature Chamber

A relatively large constant temperature chamber room belonging to UCD Civil and Environmental Engineering was available, but equipped to maintain a temperature below 10°C and was primarily used as a low temperature storage facility. Testing of the specimens for this experiment required the chamber to be operated at three constant temperatures of 4, 20 and 45 °C. A new cooling and heating system was installed in order to operate it at 4°C and 45°C. The temperature room was calibrated for the appropriate temperature conditions as shown in Figure 3-2. The difference in the temperature was observed to be within ±10% of the desired temperature values.

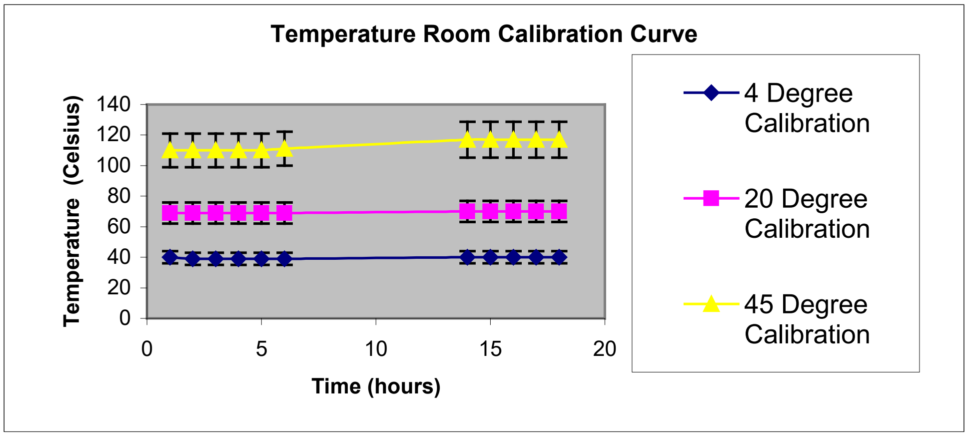


Figure 3-2 Constant temperature room calibration curve

3.7 Fabrication of the Experimental Apparatus

The schematic and a photo view of experimental apparatus are shown in Figures 3-3 and 3-4, respectively. The major components of this experimental apparatus are: pavement specimen compartments, water delivery and auxiliary pumping system, water distribution mechanism over the pavement specimens, variable (time-series) sample collection equipment, and composite sample collecting containers. These experimental components are described in greater detail below.

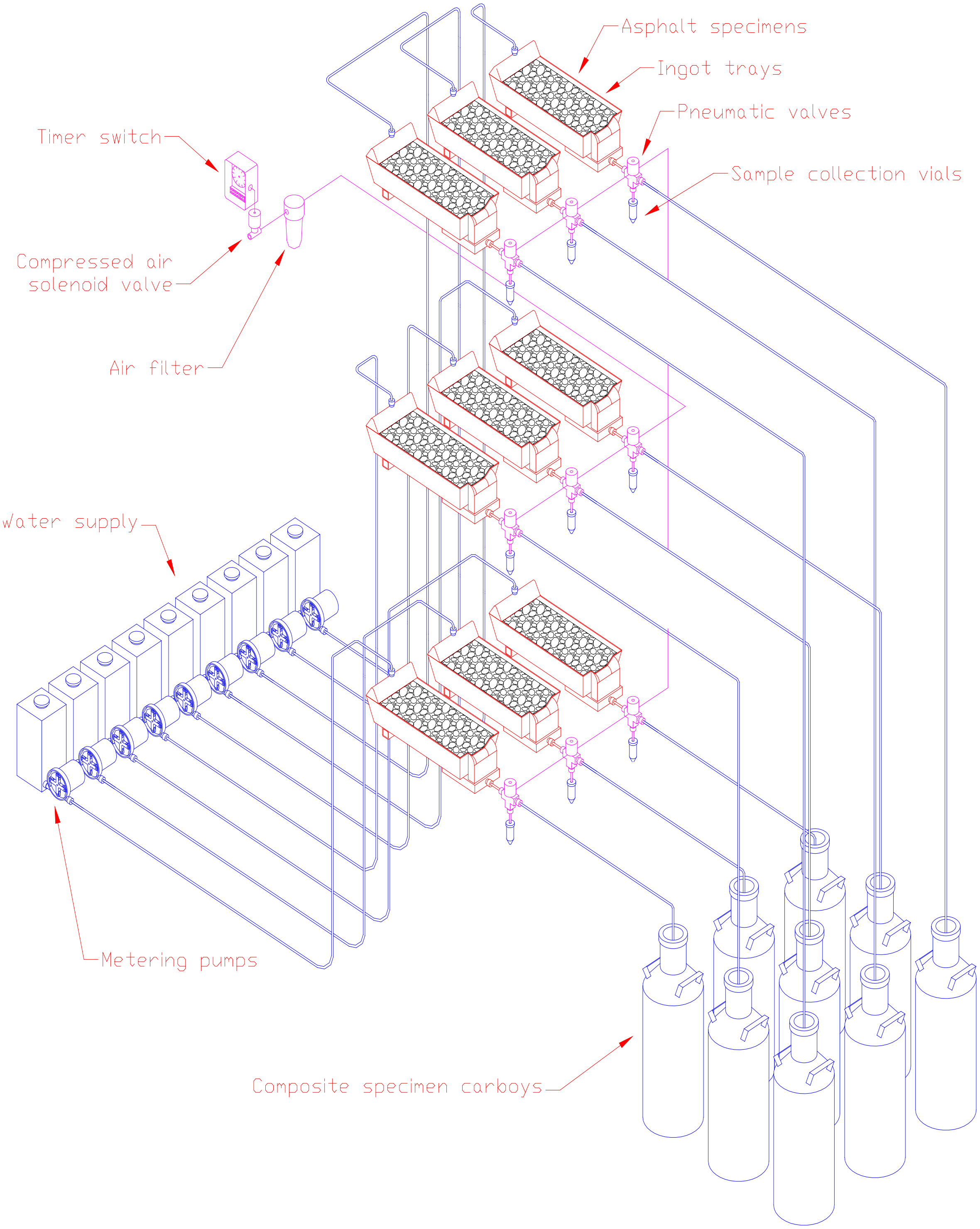


Figure 3-3 Schematic of experimental apparatus

Figure 3-4 Photo view of experimental set up

3.7.1 Pavement specimen compartment

The pavement specimen compartments were placed in racks to keep them stable. Due to the significant weight of each specimen (20 kg), rack heights were kept 0.6 to 1.3 m (2 to 4 ft) from the ground to aid in the safe and speedy placement and removal of specimens. A photo view of pavement specimen compartment is shown in Figure 3-5. The trays were set at a slope of 1 to 2 percent, as to facilitates flow and is representative of the typical cross slope of an in-service pavement. Water flows due to the effect of gravity over or through the specimens, depending on their permeability. Taking into account the difference in permeability of the specimens, spouts were provided at the surface level, as well as at the bottom of the compartments. This arrangement allowed the sampler to obtain individual samples of the surface runoff as well as the runoff that passed through the specimen, as per requirements. Lastly, the compartments were made of Stainless Steel, thus eliminating any form of contamination that may have been introduced by any other the material used.

Figure 3-5 Photo view of Pavement specimen compartment

3.7.2 Water delivery and auxiliary pumping system

The purified water was pumped by a metering pump from the storage containers into the specimen holding compartments. The pumps used for this experiment (see Figure 3-6) were attached to water storage tanks with a capacity of 20 gallons. The specimen was to be subjected to a flow of 9 gallons at a flow rate of 0.165 gallons/hr (0.63Liters/hr), thus simulating rainfall in natural conditions, as discussed in earlier sections. All pumps were calibrated to perform at the same flow. The calibration curves for pump flow are shown in Figure 3-7. As shown, the measured flow rate was within ±5% of the desired flow is designated by pump.

Figure 3-6 Variable speed pump attached to water storage tank and water delivery system

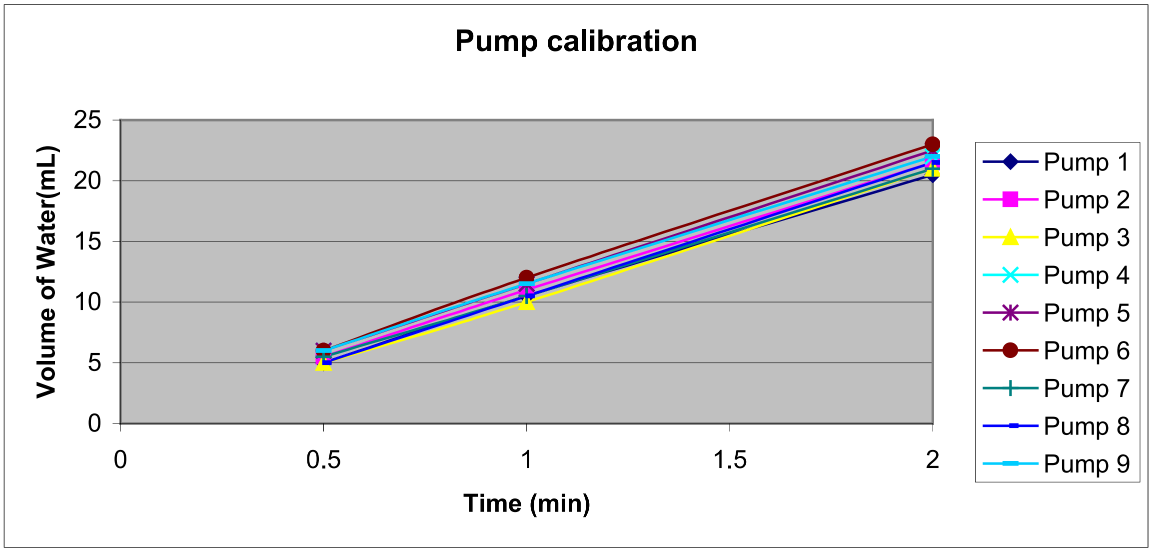


Figure 3-7 Pump calibration curves

3.7.3 Water distribution mechanism over pavement specimen

Initially, perforated stainless steel pipes were used to supply a uniform flow of water over the entire specimen width. This arrangement however did not serve its purpose as the amount of water dripping from its holes was insufficient for analysis purpose and did not provide uniform distribution over the specimen. Hence other alternatives were adopted as shown in Figure 3-8 (also see Figure 3-6). Under this arrangement, the trays were fitted with covers with a spiral water distribution system, having equidistant spaced holes, which allowed even distribution of water over the specimens. Thus, using this arrangement we were able to distribute the pumped water over each specimen.

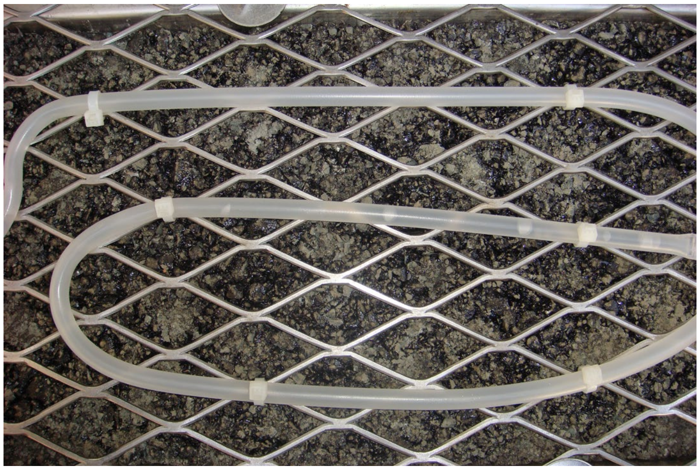


Figure 3-8 Photo view of water distribution system

3.7.4 Variable (time-series) sample collection equipment

Variable (time-series) water samples were collected in a sample collection vial (Polypropylene tubes) upon exiting the specimen trays. For these samples, an electrically controlled solenoid valve was calibrated, using an electronic timing unit, to fill the 50 mL collection vials. A photo view of the automatic solenoid valve assembly used for auto-sampling is shown in Figure 3-9. The timing for water collection began upon commencement of water flow. Samples were taken after every 15 minutes for the first hour, and one additional sample was taken on hourly intervals for a period of 8 hours. The filled sampling vials were replaced with an empty sample collection vial manually by an operator. The purpose of these samples was to evaluate the first flush effect of possible metal contaminants during each experiment.

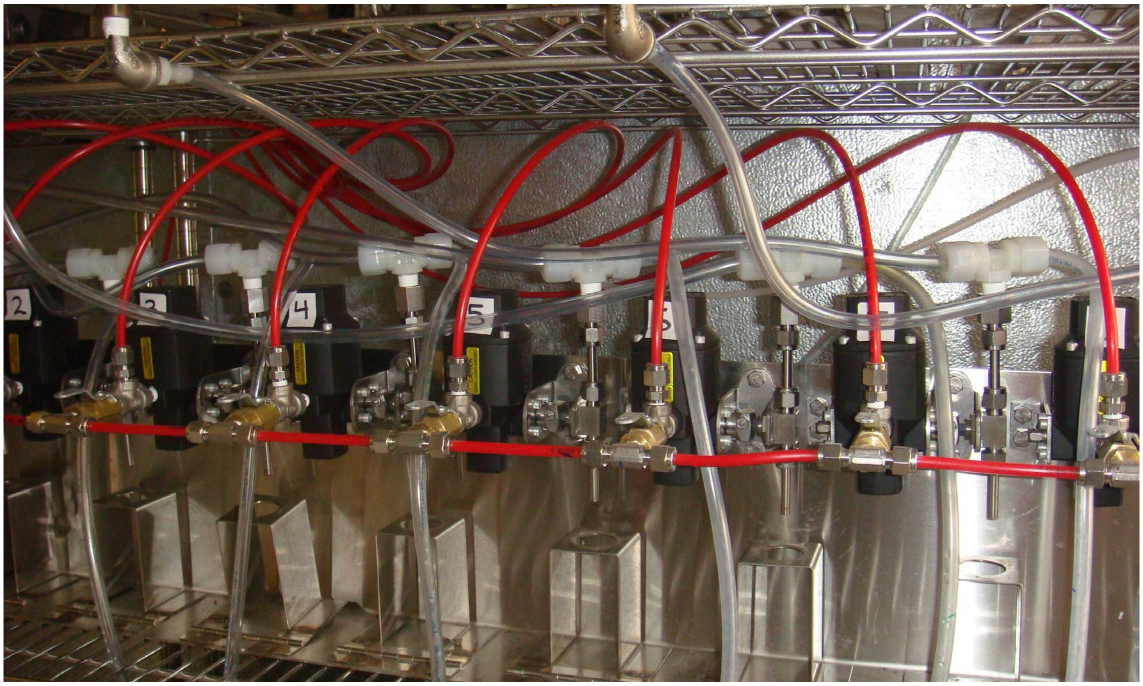


Figure 3-9 Photo view of automatic solenoid valve assembly used for auto-sampling

3.7.5 Composite sample collection containers

The entire composite water volume passing over or through each specimen was collected in a separate 15 gallon capacity stainless steel water container (shown in

Figure 3-10) for chemical and toxicity evaluation. The aperture of the composite water containers were plugged with rubber O-rings to only allow the tygon tubing to enter, and thus eliminating any contamination. Fifty (50)-milliliter Polypropylene tubes were used for sample collection (metals analysis). Polypropylene tubes were selected, as they do not introduce any contamination into the sample, which may potentially interfere with metal analysis. These tubes were replaced after the conclusion of each specific specimen type and temperate experimentation.

Figure 3-10 Photo view of the stainless steal composite water container

Chapter 4: Experimental Testing Procedures

This section of the report describes the procedures used prior, during and post experimental testing of evaluation leachate water quality produced from pavement surfacing material.

4.1 Pre-Experimental Testing Procedures

Prior each water quality experiment, the pavement specimens were carefully removed from their plastic wrapping in a clean area. A stainless steel dog tag, engraved with the specimen name, was attached to the specimen using stainless steel wire, to aid recognition of the specimen after the completion of the experiment. The specimens were then placed in the designated pavement specimen compartments. Any gaps between the pavement samples and the compartments were plugged using Ecoflex ^{TM} Rubber – platinum-catalyzed silicones, which effectively pluged the gaps, without affecting the leachate quality as well as preventing runoff flow through the gap and allow the water to flow through the specimen.

The water tanks (see Figure 3-7) were filled to capacity with Milli-Q water. The Milli – Q water was procured using the Milli-Q^{TM} filtration unit shown in Figure 4-1. The tab water was distilled through filtration and then fed through a special ion exchange cartridge, which further increased its purity. The purity of the water was assessed by measuring the resistance of the water to conducting electricity (as the resistance of the water to conductivity increases so does its purity). A gauge on the filtering machine was used to report on the conductivity of the water being produced. Using specially manufactured ion exchange resins, the filter removes ionized salts from water, theoretically removing 100% of salts.

Figure 4-1 Milli- Q^{TM} water filtration unit

All the tubing connecting the pumps (see Chapter 3), the compartments and the collecting devices was adjusted and checked. Lastly, a thorough inspection of all the labels, specimens and all other experimental apparatus was made before the initiation of the experiments.

4.2 Test Procedures during the Experiments

After the pre-experimental procedure and inspection was completed, the pumps were turned on and the water began flowing through the spiral water distribution coils and onto the specimens. A schematic of the water flow and sample collection procedure during experimental is shown in Figure 4-2. Collecting cups were provided at the spouts of the trays to collect the runoff and from where it flows to the solenoid valves assembly. Grab samples were taken after an interval of 15 minutes for the first hour, to account for pollutant variability and first flush conditions. After which samples were taken at hourly intervals, for a period of 8 hours. This was done using solenoid valves with an electrical timing unit (for detail see Chapter 3), which on activation diverted the water flow from the milk cans to the 50 mL polypropylene vials. The rest of the runoff was collected in the milk cans.

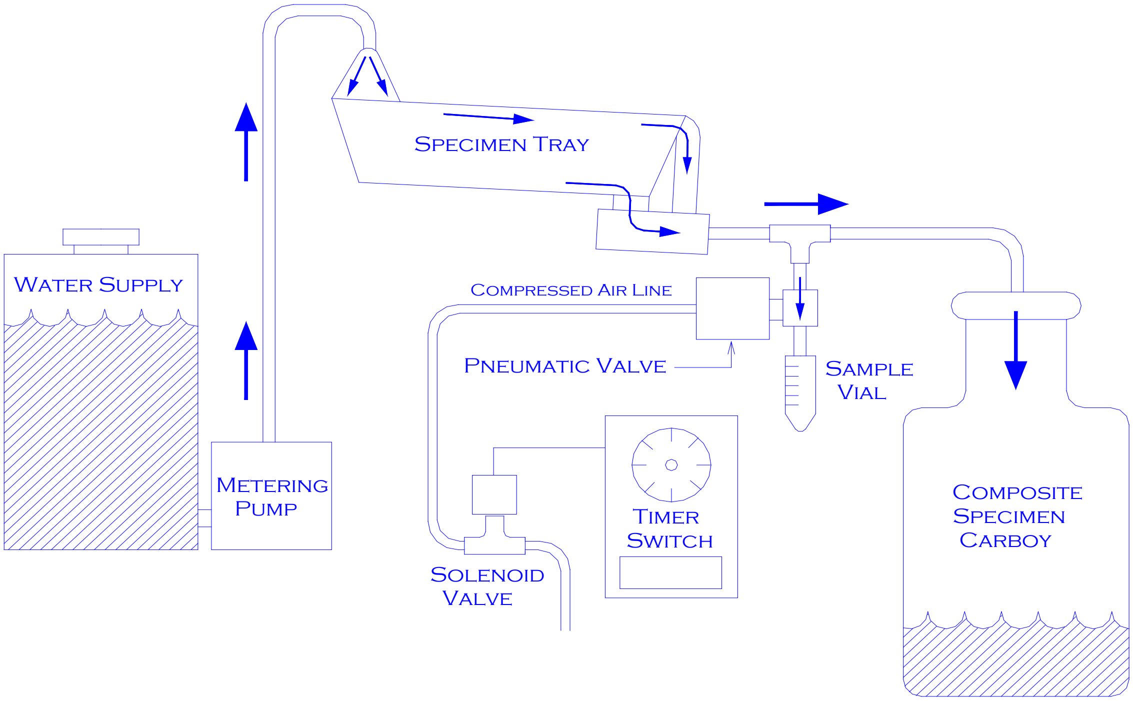


Figure 4-2 Schematic of water flow and sample collection procedure during experimental

After the allocated 48 hour time period elapsed, the composite carboys were transported to the UCD Civil and Environmental water chemistry labs and stored at 4°C walk in cold room for further processing. The composite water content in each carboy was then broken up in the volumes shown in Table 3-1 and each individual sample was used for appropriate chemical and toxicity analysis.

Table 4-1 List of break of samples, by volume for analytical procedures

Water quality parameters	**Volume**
Conventional	
Electric conductivity	50mL
Hardness	10mL
PH	50mL
Aggregate	
Chemical oxygen demand	5mL
Dissolved organic carbon	10mL in 500mL
Oil and grease	1L
Total dissolved solids	1L
Total suspended solids	1 L
Total organic carbon	10mL in 500mL
Total petroleum hydrocarbons	2L
Dissolved Metals	5mL
Nutrients	
Total Phosphorus	10mL
Total Nitrogen	50mL
Minerals	
Sodium, total sulphur and total chloride	10mL
Organics	2L (together with TPH)
Methylene Blue Active Substance	250mL in 500mL
Toxicity	8L

All water quality testing was performed according to the standard methods or the USEPA methods specified in Table 3-2 (see Chapter 3).

4.3 Post-Experimental Test Procedures

After the completion of each pavement samples water quality testing, tested specimens were taken to storage and cleaned the specimen compartment and get ready for the next set of experiment. The specimen compartments were cleaned using the following three step cleaning procedure:

- Step 1: Kerosene used to remove asphalt,
- Step 2: Ethanol used to remove traces of kerosene, as it could affect subsequent organic analysis, and
- Milli-Q water used to rinse the compartments for any residual kerosene and ethanol.

The output spouts of the trays were cleaned using pressurized air, to ensure the removal of all debris and prevent clogging. The tubing (tygon tubing) was replaced after each experimental phase, as there was a possibility of accumulation of contaminants from earlier experimental phases, which could have adversely affected the analytical results.

For similar reasons, the collecting cans and the milk cans were both thoroughly cleaned with Milli-Q water, before the initiation of the next experiment
The water tanks (see Figure 3-7) were refilled with Milli-Q water and got ready to initiate the next experiment. The above-mentioned procedure was repeated for the next batch of specimens, as per the experimental plan (refer Chapter 3).

Chapter 5: Permeability of Compacted Surfacing Pavement Materials

5.1. Introduction

The in-place permeability of the various asphalt concrete and Portland cement concrete specimens tested as part of this study for water quality evaluation. It is generally recognized that the durability and performance of a bituminous pavement depends on its permeability. A mixture with high permeability (up to 25% air-voids) allows the water and air to pass through. Pavement materials with these higher degrees of permeability drain water from the surface and reduce the thermal conductivity. In addition, permeable pavements drain the water much faster during rainfall events and hence reduce car accidents. An additional benefit associated with permeable pavement is noise reduction. All permeability measurements were made based on test method developed by NCAT (National Center of Asphalt Technology) for in-place permeability of asphalt concrete in the field as described below. It is important to note that the NCAT procedure is not an ASTM method or AASHTO test method yet. However, it’s a well known procedure for measuring permeability in the field (Cooley et al, 2001).

The in-place permeability of asphalt pavements using NCAT’s water permeameter is known to give valid, repeatable index values related to the permeability of asphalt pavements. The NCAT device consists of a graduated standpipe with multiple sections of varying diameter, as shown in Figure 5.1.

Figure 5-1 NCAT device used for permeability measurement

The varying diameters allow for more practical data collection for a wide range of pavement permeability (NCAT Permeameter Manual). The device is sealed to the pavement surface and filled with water using a moldable sealant, the sealing method recommended by NCAT.. This permeability measurement is based on the falling head principle commonly used in civil engineering field:

$$k = \frac{aL}{AT}\ln\left(\frac{h_1}{h_2}\right) \qquad (5.1)$$

Where,

k= coefficient of permeability,

a= cross-sectional area of standpipe used for measurements,

L= thickness of pavement layer,

A= cross-sectional area of sample (sealed pavement area),

T= Time (seconds) required for observed overhead drop,

h_1= head at the beginning of time measurement, and

h_2= head at the end of time measurement

5.2. Test Procedure

A photo view of the apparatus used during our permeability measurement is shown in Figure 5.2. As shown, the Permeameter consists of up to 4 tiers of clear plastic in two sections. The standard top section allowed faster filling of the permeameter with water when testing open-graded mixes which may be very permeable and facilitated reading of the graduations on the clear tubes.

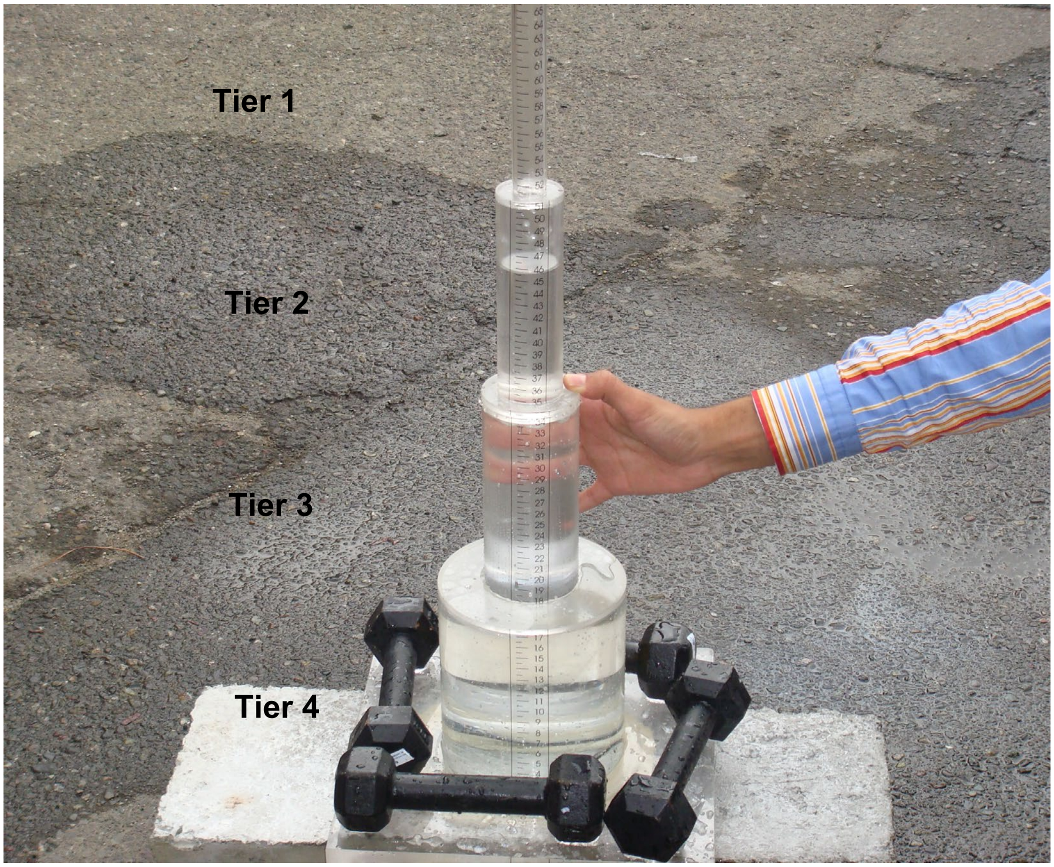


Figure 5-2 Photo view of the apparatus during permeability measurement

To begin the test, the ingot was brushed clean of loose stone, dust and debris. The moldable sealant supplied by the manufacturer (Gibson Company) was applied at the bottom of the permeameter base plate. The sealant was easy to work with and was applied uniformly against the outside edge of the rubber Gasket to define the test area. However, for open graded mixes with high permeability, ecoflex was used as sealant to better seal the surface and prevent leakage from bottom of the permeameter. Ecoflex™ Rubbers are platinum-catalyzed silicones that are versatile and easy to use. Ecoflex™ Rubbers are mixed 1A:1B by weight or volume and cure at room temperature with negligible shrinkage. Once the moldable sealant was properly installed around the base, the permeameter was then gently seated on the prepared test area. Uniform foot pressure was then applied around the perimeter of the base to seat the permeameter intact. After the permeameter was sealed to the surface, four weights supplied by the manufacture were placed on each corner of the base to hold the seal intact. After setting up the apparatus, the tier was filled with water at steady rate and depending on the mix being tested; an appropriate single tier was used for tracking the rate of water fall. The tier should be selected/configured such that the rate of flow is slow enough for accurate observation, but fast enough for timely completion of the test. Test time varies depending on mix gradation and density, but should not exceed about five minutes as recommended by the NCAT Permeameter Manual. Once an appropriate tier was selected, the operator notes the starting time and starting height for the falling head. The testing time "T" was the time it takes for the water to drop from the starting head to the ending head height, "h_1" and "h_2" respectively in Eq. (5.1) The permeability test was repeated three times and the average permeability was reported for the test.

5.3. Permeability Test Results

Permeability tests were performed on all 10 pavement specimen types identified in Table 5.1. The results of permeability tests for all pavement specimens of different mix types and binder materials is summarized in Table 5.2. The results shown in Table 5.2 are based on the average of three permeability test following the ASTM-D 5042 procedure. These permeability results are also plotted in Figure 5.3 to illustrate their distinct permeability variations.

Table 5-1 Specimen Mix Types and Binder Materials

Specimen	Mix type	Binder
A	RAC-O Rubberized Asphalt Concrete Open-graded	Rubberized asphalt cement
B	RAC-G Rubberized Asphalt Concrete Gap-graded	Rubberized asphalt cement
C,D	OGAC Open-graded Asphalt Concrete	2 Unmodified Performance Grade (PG) binders
E,F	OGAC-PM Open-graded Asphalt Concrete Polymer Modified	2 Modified PG binders
G	MB-G Modified Asphalt Binder	MB4 Binder
H	DGAC Conventional Dense-graded Asphalt Concrete	(PG) Binder - Unmodified
I	PCC-Dense	Portland cement
J	PCC-Permeable	Portland cement

Table 5-2 Summary of Permeability Results

Mix Type	Tier area (cm^2) a	Test Area (cm^2) A	Initial Head (cm) H1	Final Head (cm) H2	Duration (sec) T	Thickness (cm) L	Coef. of Permeability K
RAC-O	167.53	214	13.0	2.0	54.0	7.93	0.215
RAC-G	38.32	214	29.0	19.0	44.8	7.93	0.013
OGAC(64-10)	167.53	214	11.5	8.2	83.8	7.59	0.024
OGAC(64-16)	167.53	214	11.5	8.7	61.1	7.93	0.029
OGAC(58-22)	167.53	214	10.3	7.5	77.6	7.93	0.026
OGAC(64-28)	167.53	214	9.5	6.5	66.5	7.93	0.035
MAC15-GG	38.32	214	37.7	22.0	100.3	7.93	0.008
DGAC(64-16)	7.07	214	52.4	51.0	101.5	7.93	0.000
PCC-D	2.85	214	65.9	64.4	155.9	7.93	0.000
PCC-OG	167.53	214	15.3	5.3	57.2	7.93	0.116

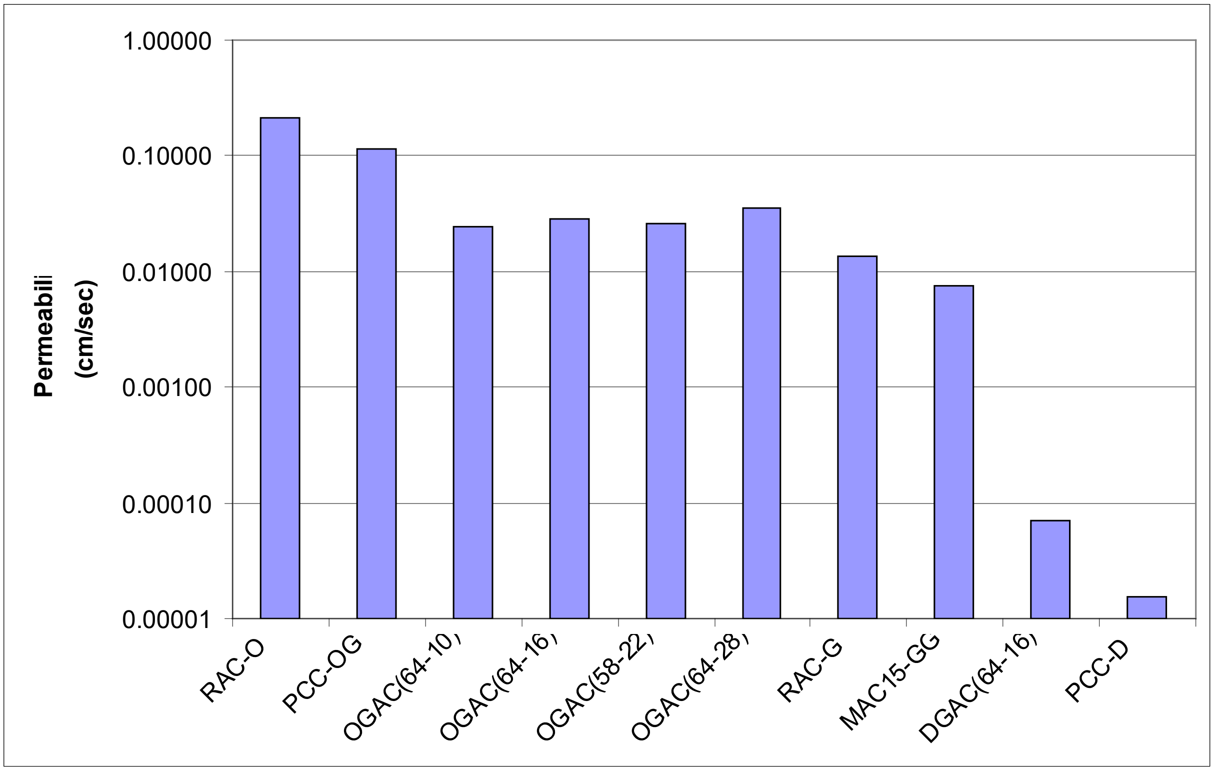


Figure 5-3 Permeability for different mixes

Based on the permeability results, Rubberized-Asphalt Concrete (Open Graded) mixes had the highest permeability with coefficients of permeability (K) as high as 0.215. Portland-Cement concrete with open-graded gradation also had a high coefficient of permeability (K= 0.116) compared to other mixes.

Dense graded mixes tested in this study had a very low permeability. This conforms with the conventional pavement engineering wisdom that dense-graded mixes are designed to have low air-voids and therefore low permeability. Open-graded mixes are designed with high air-voids content and are expected to have much higher permeability.

Two Gap-graded mixes tested in this study showed permeability values less than open-graded mixes and more than dense-graded mixes as expected. Rubberized asphalt concrete-GG mix showed 1.6 times more permeability compared to MAC15-GG mix.

5.4. Summary

Field permeability tests using the NCAT field permeameter were used to measure the coefficient of permeability for all ten mixes developed for this study. Permeability tests were repeated three times for each mix and average number is reported for each mix. Permeability values ranged from 0.215 for RAC-O (very permeable mix) to 0.00001 for PCC (Dense-Graded). Open-Graded mixes were designed and expected to have higher air-void contents and hence higher permeability than Dense-graded mixes. These values were in agreement with what was expected of these mix designs from standard pavement engineering knowledge (see Figure 5-4).

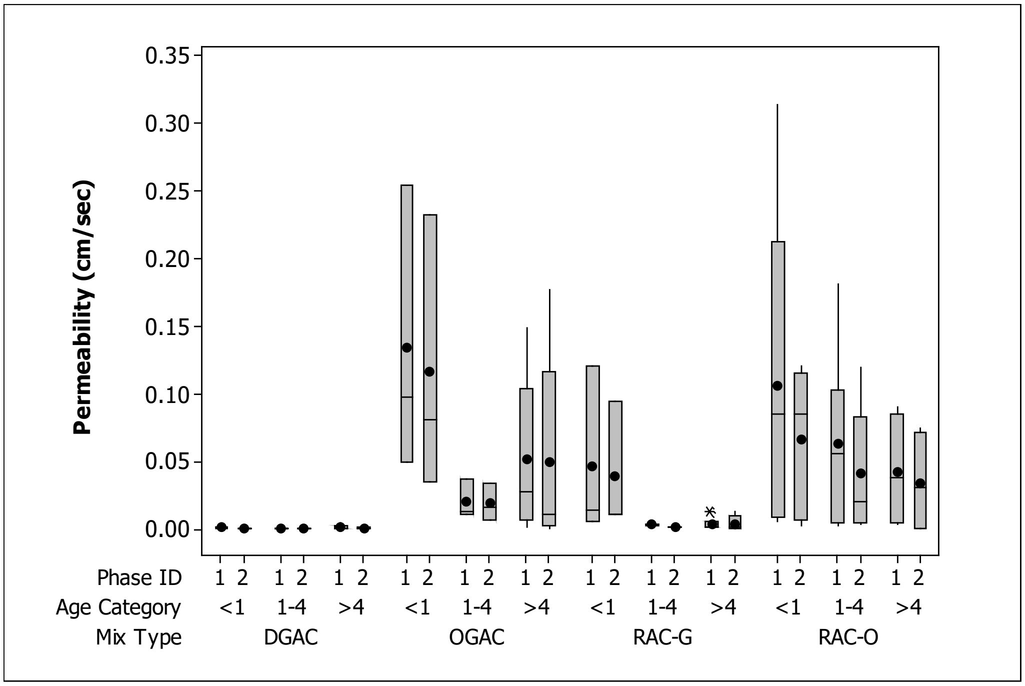


Figure 5-4 Box plot of permeability values for different mix types at different ages (Reference: Ongel et al., 2008)

In each box plot shown in Figure 5-4, the bottom of the box shows the first quartile, and the top of the box shows the third quartile. The line in the middle of the box is the median, and the dot represents the mean of all the observations. The distance between the first quartile and the third quartile is called the inter-quartile range (IQR). Each of the whiskers (lines that extend above and below the box) has a length of 1.5 times the IQR.Each data pont outside the whisker is an observation that is greater than 1.5 times the IQR; these points are potential outliers and are marked by asterisks.

Chapter 6: Conventional Water Quality Parameters, Nutrients and Minerals Results and Discussion

This section of the report presents the results and discussion associated with conventional water quality parameters, nutrients and mineral constituents.

6.1 Conventional Water Quality Parameters

Conventional water quality parameters measured were: pH, electrical conductivity (EC), hardness (mg/L as $CaCO_3$), turbidity, total suspended solids (TSS) and total dissolved solids (TDS). These conventional water quality parameters are of utmost importance to water quality standards and are an integral part of all water quality and regulatory standards. These parameters can also serve as an important indicator of toxicity. The results of these water quality parameters along with their importance and implications are briefly discussed below.

6.1.1 pH

The pH was measured at the time of the termination of the experiment (48 hours), using a portable pH meter. Results of the specimens were averaged for all three temperatures. The pH was observed to be constant for all asphalt specimens (Specimens A, B, C, D, E, F, G, H) under all temperatures and the variation was statistically determined to be insignificant. The pH for the concrete specimens (Specimens I, J), was significantly higher due to presence of higher carbonate materials than in asphalt specimens. The results of pH for both fresh and aged pavement specimens are shown in Figure 6-1. As shown, both fresh and aged specimens showed similar results. It is important to note that the pH of inflow water was not adjusted to lower pH as originally planned since it was found that the pH adjusted water was toxic. The change in pH observed was due to water contact with pavement specimens. Lower pH can influence the solubility of certain metal elements and this aspect of the pH influence will be discussed in Chapter 7.

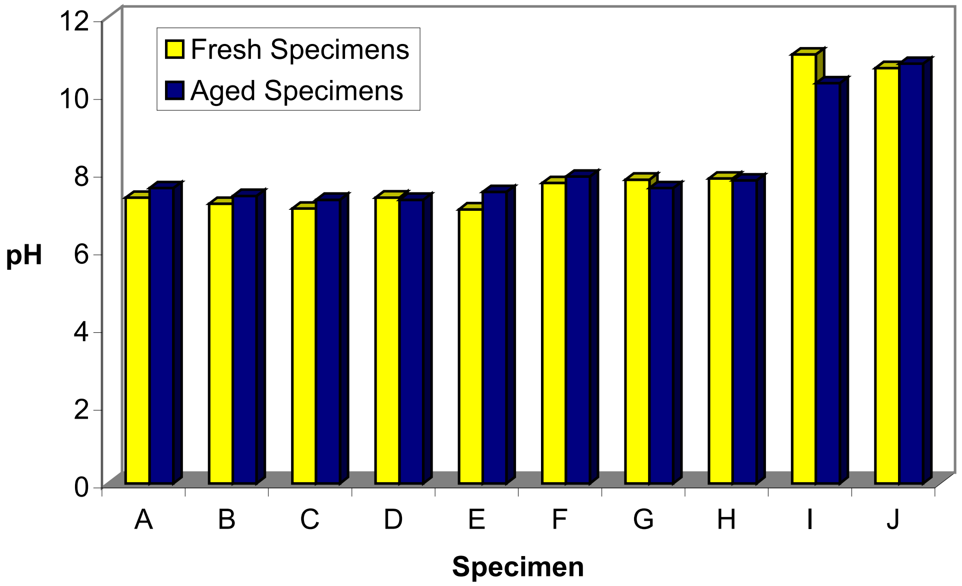


Figure 6-1 Average pH values for fresh and aged asphalt and concrete specimens for triplicate specimens and three different temperatures of 4, 20, 45 °C

(Note: total inflow water for each pavement specimen tested at 4, 20, and 45 °C = 30 L)

6.1.2 Electrical Conductivity (EC)

The average result of electric conductivity for all asphalt specimens for three different temperatures of 4, 20 and 45 °C is as shown in Figure 6-2. The result of EC testing for all leachate samples related to the asphalt specimens was within the detection limit. However, compared with asphalt pavement specimens, the EC values for concrete pavement specimens (I and J) were significantly higher. Similar results were observed for aged specimens and no significant difference in EC result was observed between fresh and aged pavement specimens.

6.1.3 Hardness (mg/L as $CaCO_3$)

Hardness of all samples was measured at the time of the termination of the experiment (48 hours), using the Hach Portable Multi Parameter Test Kit. The results of hardness testing for all fresh pavement specimens under three different temperatures are as shown in Figure 6.3. As shown, hardness of most asphalt specimens was lower as compared to concrete specimens and the difference in values observed within the triplicates was negligible. A higher concentration of hardness in concrete specimens (I and J) is expected since these materials contain more calcium carbonate and consequently leached a higher concentration of calcium carbonate. A similar result was observed for aged specimens and no significant difference in calcium carbonate was observed between fresh and aged pavement material.

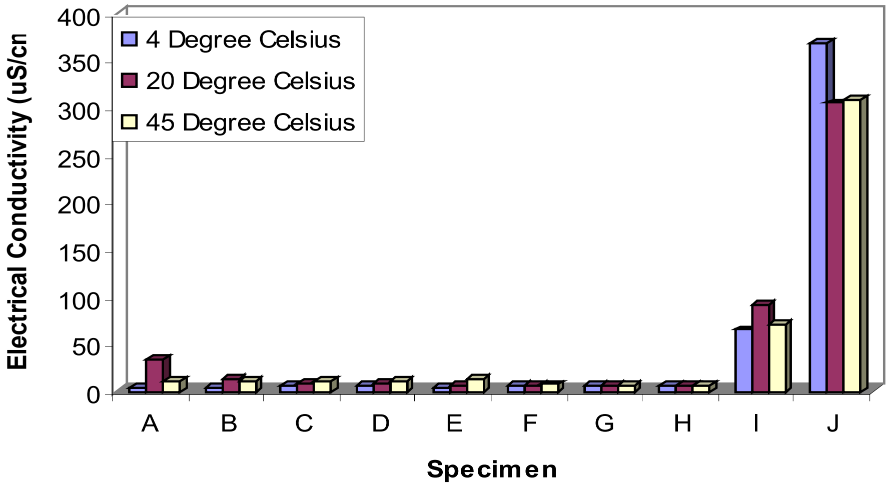


Figure 6-2 Average electrical conductivity of all triplicate fresh and aged pavement specimens for different three temperatures
(Note: total inflow water for each pavement specimen tested at 4, 20, and 45 °C = 30 L)

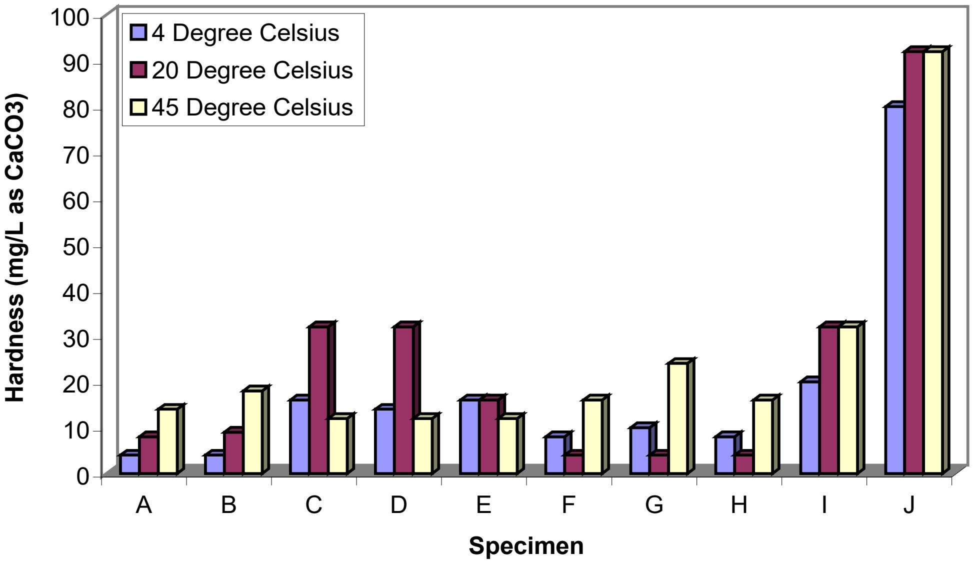


Figure 6-3 Average hardness (as mg/L of $CaCO_3$) for all triplicate fresh and aged pavement specimens under three different temperatures
(Note: total inflow water for each pavement specimen tested at 4, 20, and 45 °C = 30 L)

6.1.4. Turbidity

Turbidity measurement s of leachate samples for all asphalt and concrete specimens were less than 1 NTU. Relative clarity of leachate water produced from specimens G, H, and J with Milli-Q water is shown in Figure 6-4. As can be seen, no noticeable difference in the relative turbidity can be observed among different pavement specimens and the turbidity of Mili-Q water. Similar turbidity measurements and observations have been made for turbidity of aged specimens.

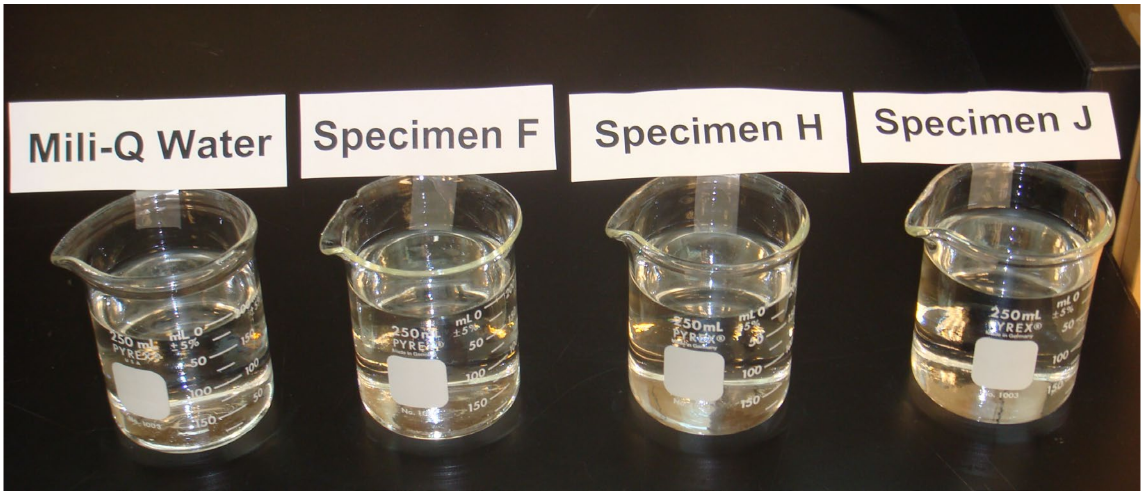
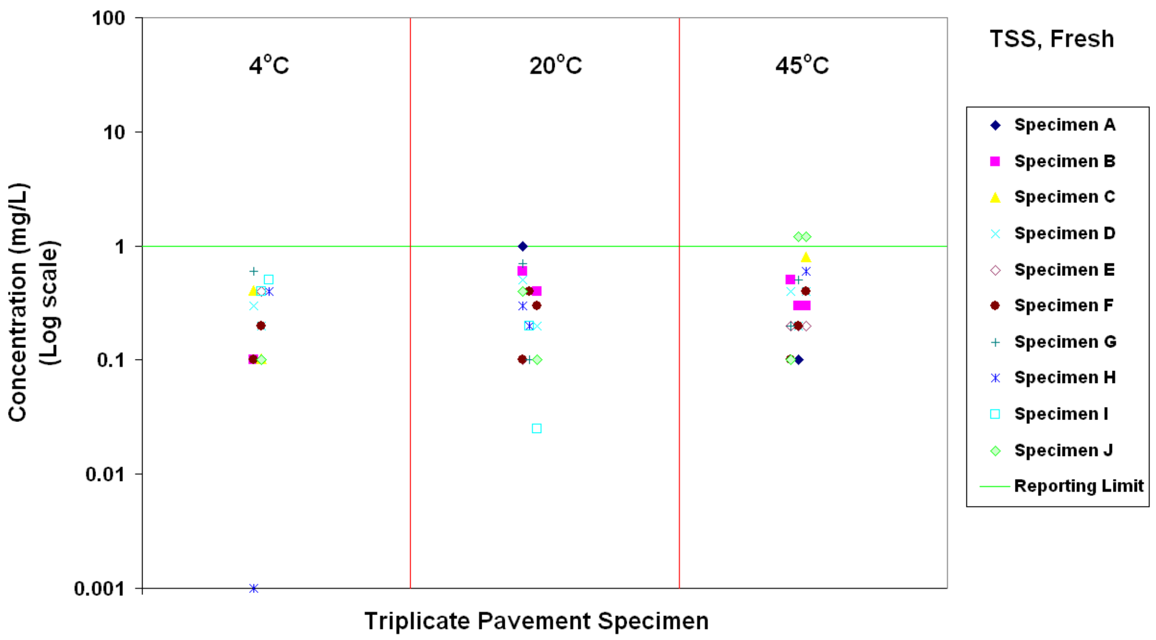


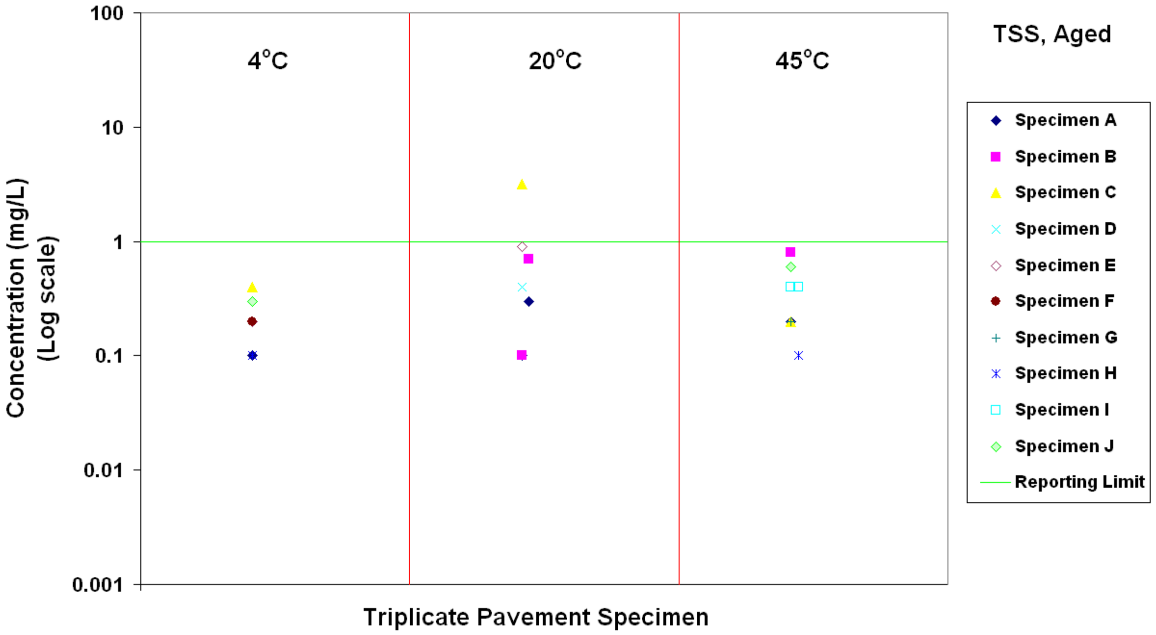
Figure 6-4 Relative Turbidity observation of Mili-Q water and turbidity of leachate produced from specimens F, H and J

6.1.5. Total Suspended Solids (TSS)

TSS results for individual leachate samples produced from fresh and aged specimens are shown in Figure 6-5(a) and 6-5(b) respectively. As shown nearly all TSS values were below the reporting limit, with the exception of open graded fresh concrete specimen J**.** This higher concentration of TSS in specimen J could be an indication for the elevated levels of hardness, EC and dissolved metals concentration.



(a) Fresh Specimens



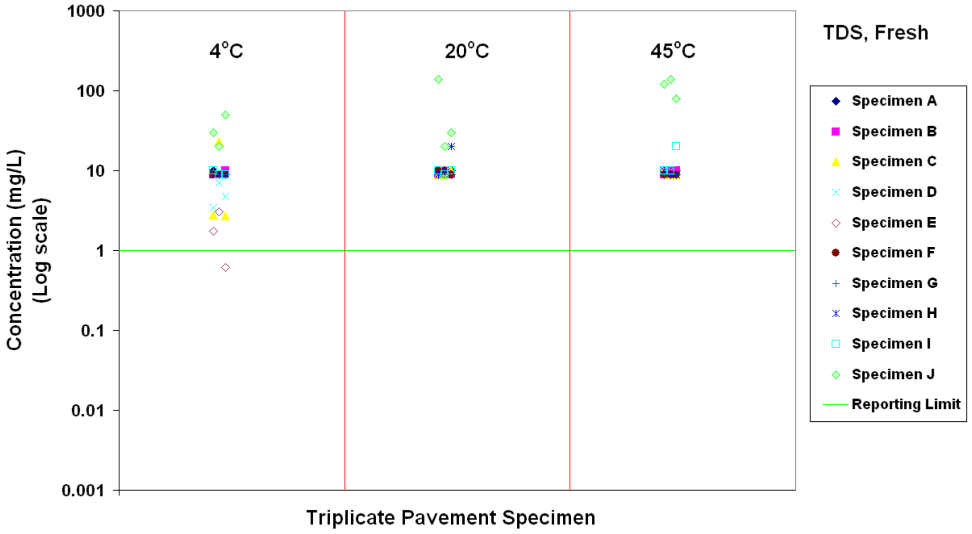
(b) Aged Specimens

Figure 6-5 Individual TSS concentration for all triplicate fresh and aged specimens under three different temperatures

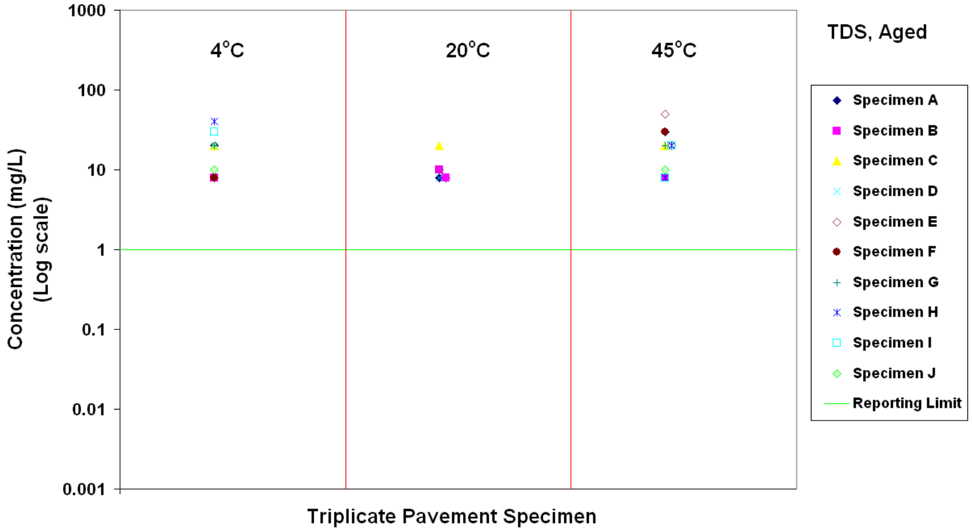
(Note: total inflow water for each pavement specimen tested at 4, 20, and 45 °C = 30 L)

6.1.6. Total Dissolved Solids (TDS)

TDS results for individual leachate samples produced from fresh and aged specimens are shown in Figure 6-6(a) and 6-6(b), respectively. Contrary to TSS results, the TDS values for pavement specimens were above the reporting limit. Higher TDS values were observed for concrete samples J and I. Elevated concentrations of TDS in concrete samples can be confirmed with an increase in EC, hardness and dissolved metals concentration (see chapter 7).



(a) Fresh Specimens



(b) Aged Specimens

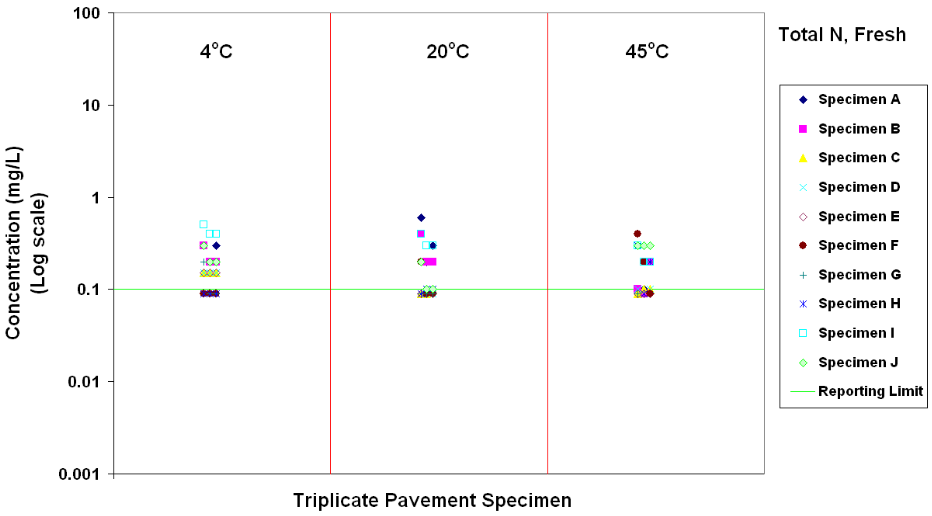
Figure 6-6 Individual TDS concentration for all triplicate fresh and aged specimens under three different temperatures
(Note: total inflow water for each pavement specimen tested at 4, 20, and 45 °C = 30 L)

6.2 Nutrients

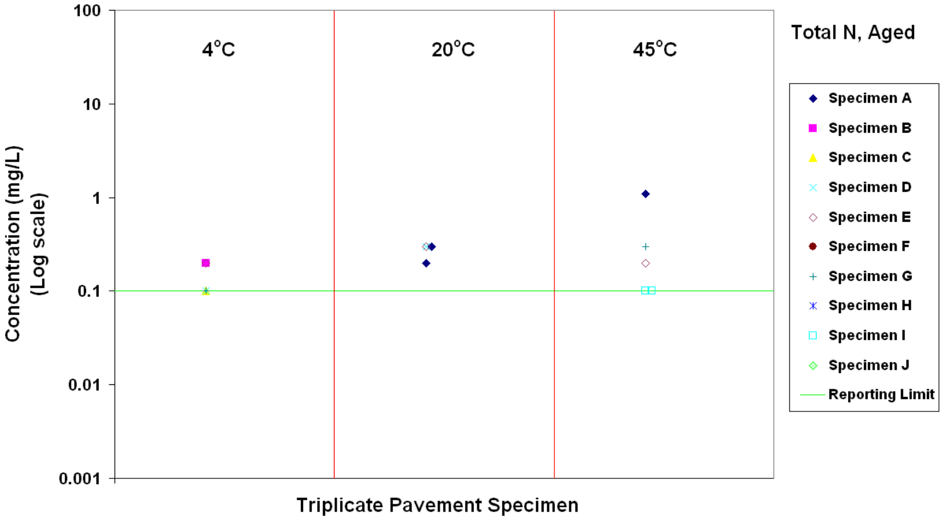
Nutrients measured under this study were total nitrogen and total phosphorus.

6.2.1 Total Nitrogen (TN)

The results of total nitrogen for all leachate samples produced from fresh and aged specimens under three different temperatures are shown in Figures 6-7a and 6-7b. As shown, only some leachate samples resulted in TN concentration above the reporting limit. The detected TN concentration was less than 1 mg/L and both temperature and age did not significantly influence the TN concentrations.



(a) Fresh Specimens



(b) Aged Specimens

Figure 6-7 Individual TN concentration for all triplicate fresh and aged specimens under three different temperatures (Note: total inflow water for each pavement specimen tested at 4, 20, and 45 °C = 30 L)

6.2.2 Total Phosphorus

Non-detectable concentrations of Total Phosphorus was measured from all fresh asphalt and concrete samples. Similar to the fresh samples, aged samples also showed no detectable trace of total phosphorus for the entire duration of the experiment.

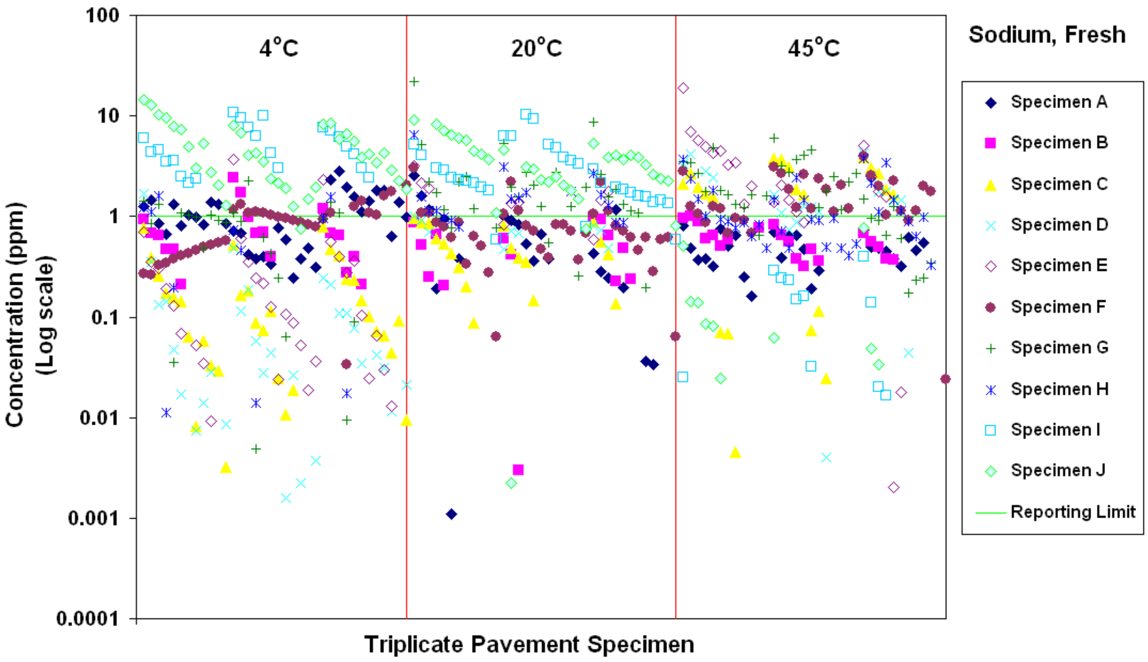
6.3 Minerals

The minerals tested under this study include: Sodium, total Sulphur and total Chlorine

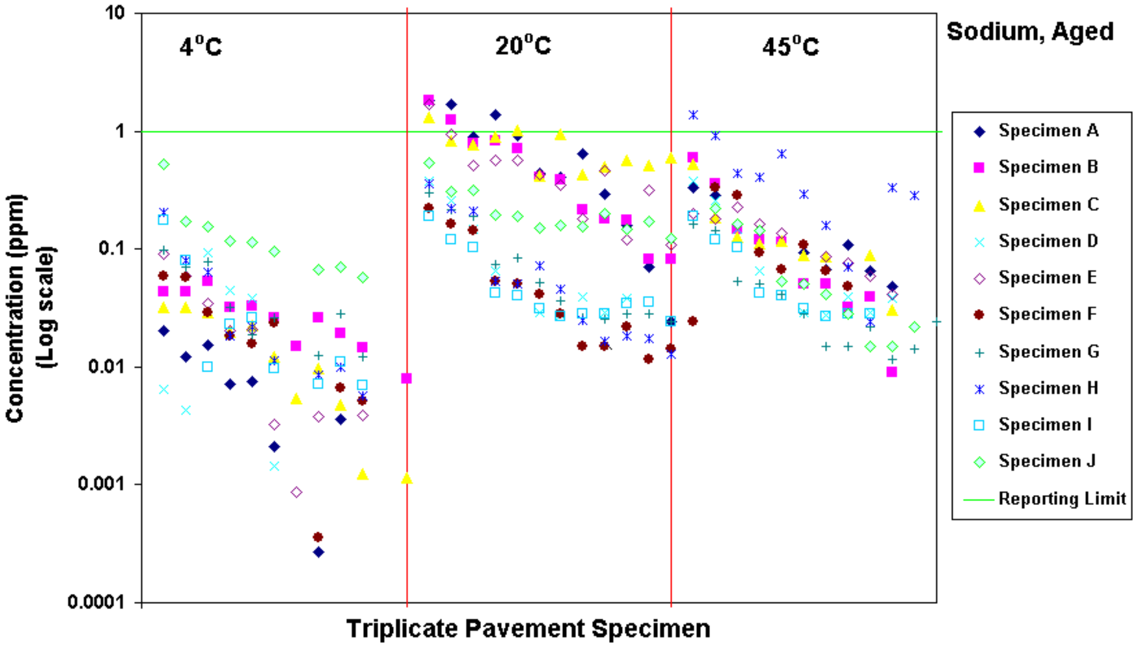
6.3.1 Sodium

Individual sodium concentrations for leachate produced from fresh and aged specimens are shown in Figures 6-8(a) and 6-8(b), respectively. As can be seen the detected sodium concentration for asphalt specimens were lower than the sodium concentration for concrete specimens.

Sodium concentration of leachates for aged asphalt and concrete pavement specimens were mostly below the reporting limit.



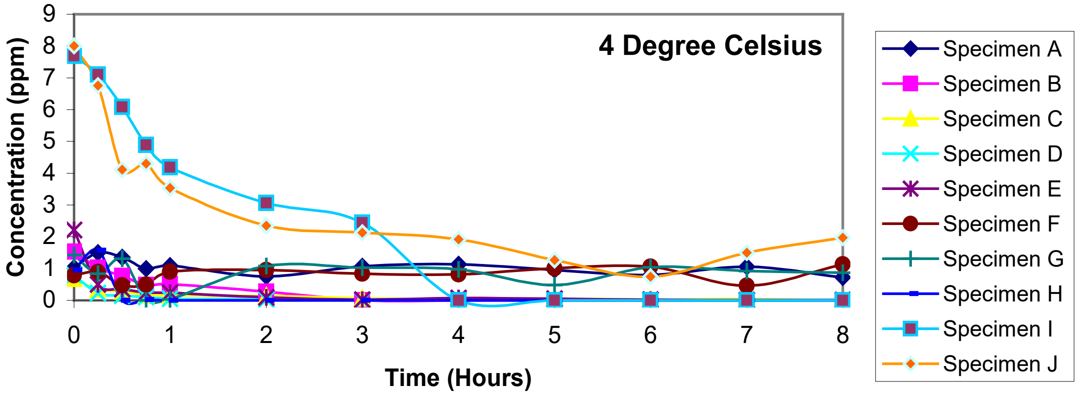
(a) Fresh Specimens



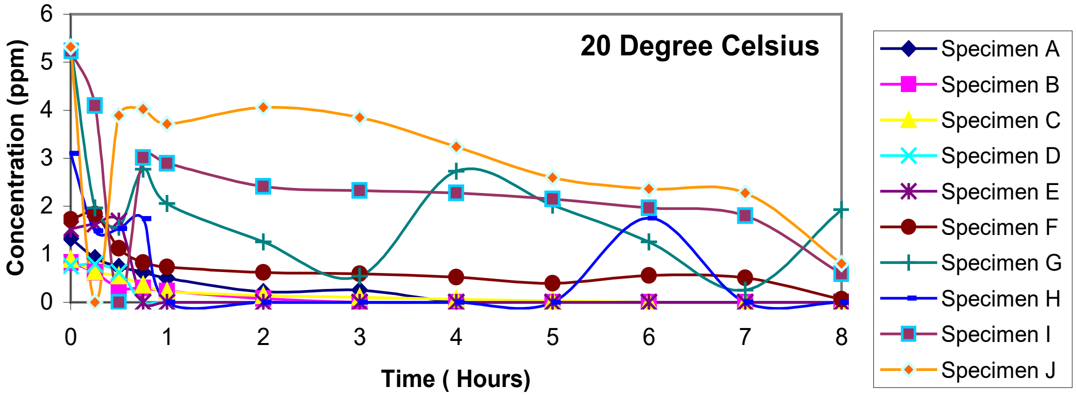
(b) Aged Specimens

Figure 6-8 Individual sodium concentrations for all triplicate for fresh and aged specimens under three different temperatures
(Note: total inflow water for each pavement specimen tested at 4, 20, and 45 °C = 30 L)

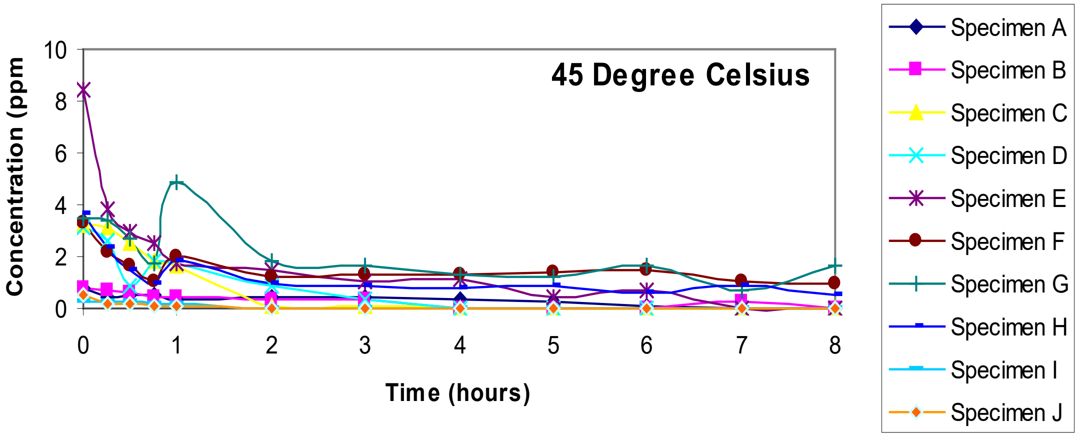
The variability of sodium concentration for leachate samples collected during the experiments for all fresh samples under three different temperatures of 4, 20 and 45 °C is shown in Figure 6-9. From Figure 6-9, it can be observed that the leachate of concrete specimens (I and J) showed increased concentrations of sodium over the entire duration compared with sodium concentration observed from asphalt specimens. In addition, a slight decrease in sodium concentration was observed with increase in temperature for concrete specimens. It is important to note that the majority of sodium concentrations were below the reporting limit and the detected values are not a water quality concern.



(a) 4° C



(b) 20° C



(c) 45° C

Figure 6-9 Variability of sodium concentration for leachate samples collected during the experiments for all fresh samples under three different temperatures
(Note: total inflow water for each pavement specimen tested at 4, 20, and 45 °C = 30 L)

6.3.2 Total Sulphur

The Total sulphur concentrations for all the leachate samples produced from fresh and aged specimens under the three different temperatures were not detectable even at the low method detection limit used in this study. All aged specimens (Asphalt and Concrete) showed similar results.

6.3.3 Total Chlorine

The Total chlorine concentrations for all the leachate samples produced from fresh and aged specimens under the three different temperatures were not detectable even at the low method detection limit used in this study. All aged specimens (Asphalt and Concrete) showed similar results.

Chapter 7: Trace Metals Results and Discussion

7.1 Introduction

This section of the report presents the results and discussion associated with trace metal analysis. Selected trace metals analyzed under this study and presented in the order appearing in this chapter include: cadmium, lead, iron, nickel, arsenic, zinc, copper, vanadium, and chromium. All trace metals were measured from samples taken at every 15 minutes during the first hour and every hour thereafter up to 8 hours.

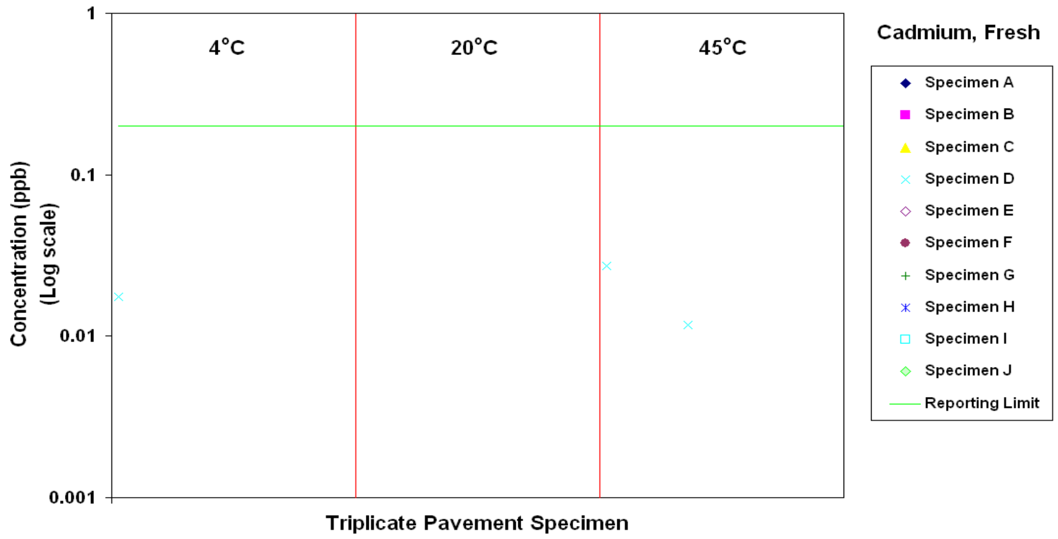
The test results of the triplicate trace metals concentration for all samples collected from fresh pavement specimens during the three temperatures are presented in a single plot. In these plots we have included all individual grab samples analysis. The results presented in these plots are considered as dissolved since the water contact with pavement did not produced ant particulate matter. A similar plot is prepared for aged pavement specimens. It is also important to note that the method detection limit used in this study was much lower than the Caltrans reporting limit, therefore, all detected values while below reporting limit, are reported in these plots. Because most trace metal concentrations were observed to be quite low and could not be depicted clearly on a linear scale, the concentration data presented below are based on logarithmic scale.

Following the trace metals results presentation for fresh and aged pavement specimens, information is presented to discuss: (1) the variability of metals concentration during different sampling periods, (2) the impact of pH on metals leachate solubility, (3) statistical analysis evaluating the difference in metals results for fresh vs. aged; concrete vs. asphalt; low vs. high temperatures, and (4) a preliminary investigation to identify the sources of metal pollutants of concern from concrete and asphalt pavement specimens.

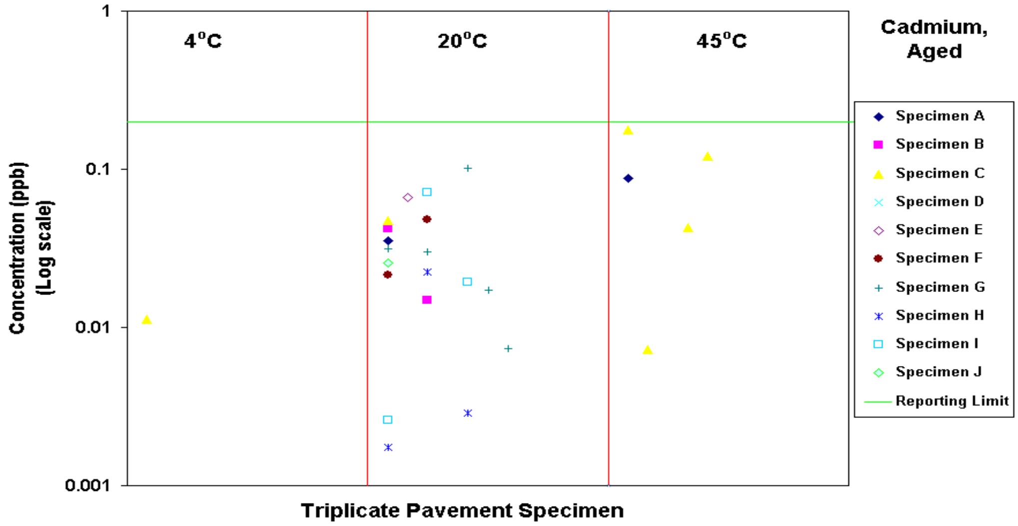
7.2. Leachate Trace Metals Results

7.2.1. Cadmium

The cadmium concentration for all samples tested for fresh and aged pavement specimens for three different experimental temperatures are presented in Figures 7-1a and 7-1b, respectively. As shown, all tested pavement specimens were observed to have negligible concentrations of cadmium, at all experimental temperature ranges. None of the specimens were observed to have concentration above the reporting limit.



(a) Fresh Specimens

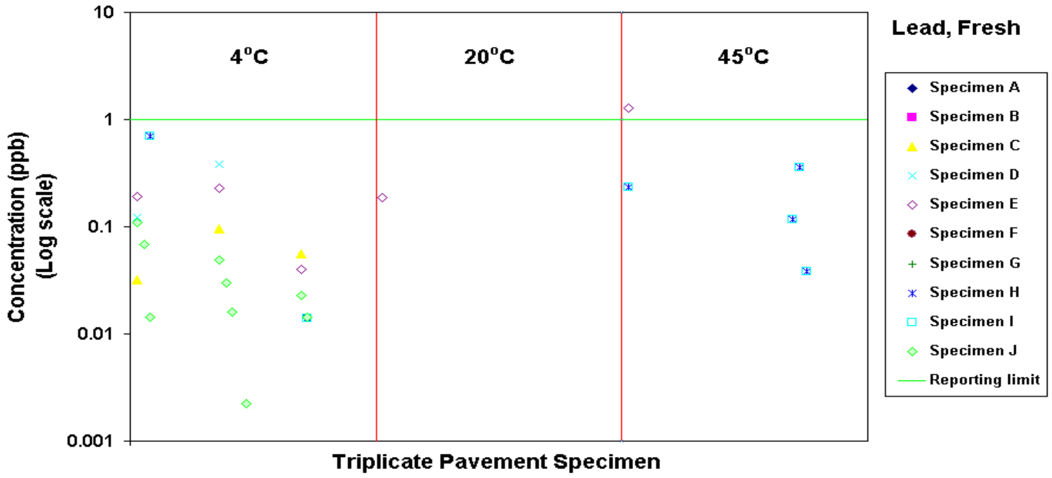


(b) Aged Specimens

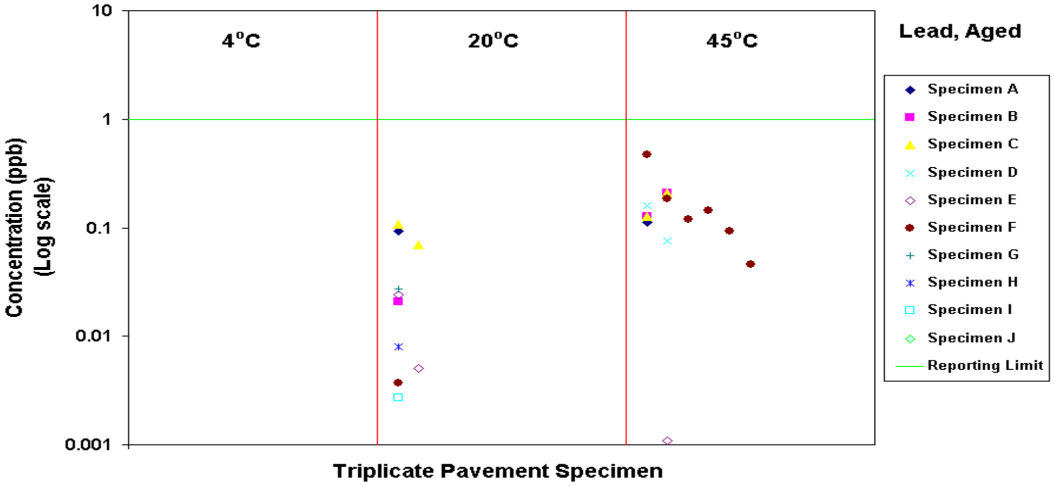
Figure 7-1 Cadmium concentrations for all fresh and aged leachate samples tested under three different temperatures

7.2.2. Lead

The lead concentration for all samples tested for fresh and aged pavement specimens for three different experimental temperatures are presented in Figures 7-2a and 7-2b, respectively. As shown, all tested pavement specimens were observed to have negligible concentrations of lead for all experimental temperature ranges. Only one fresh specimen was observed to have concentration above the reporting limit at 45°C.



(a) Fresh Specimens

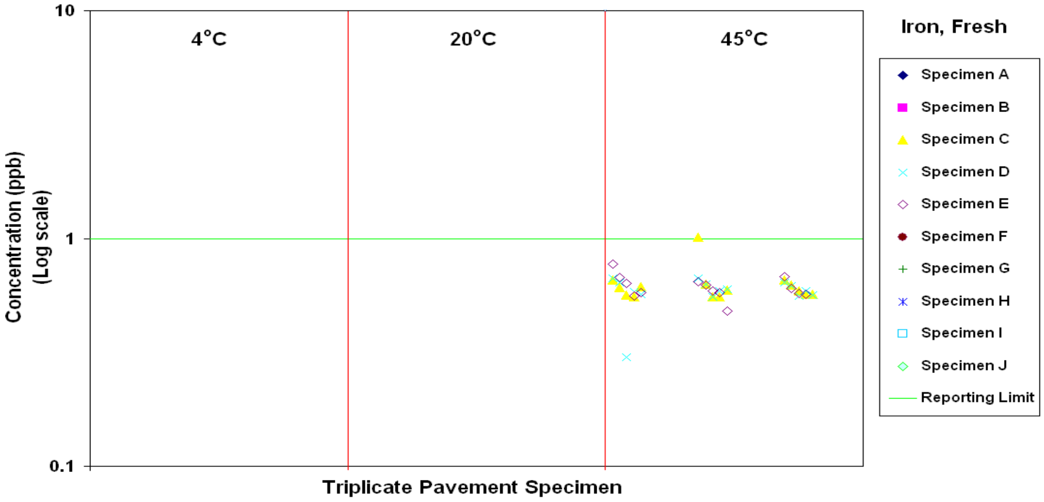


(b) Aged Specimens

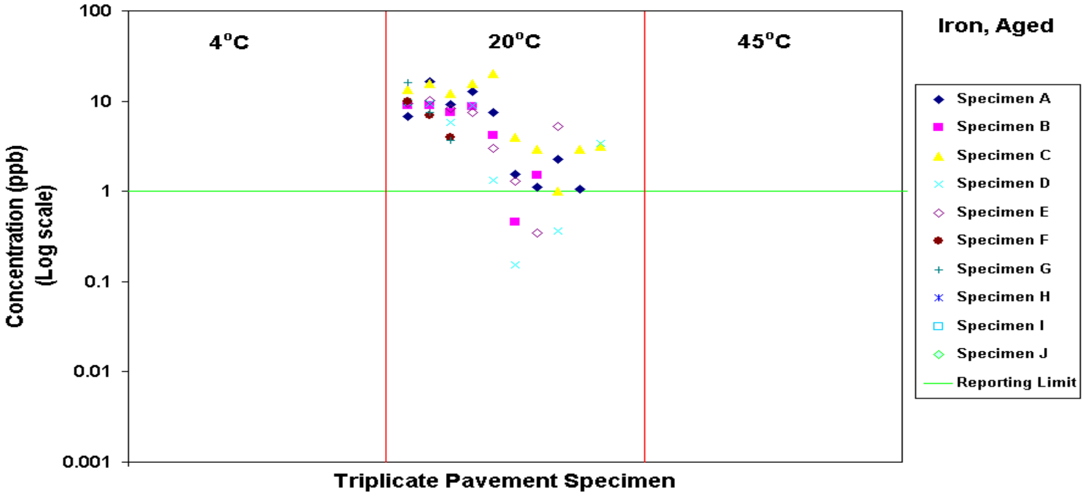
Figure 7-2 Lead concentrations for all fresh and aged leachate samples tested under three different temperatures

7.2.3. Iron

The iron concentration for all samples tested for fresh and aged pavement specimens for three different experimental temperatures are presented in Figures 7-3a and 7-3b, respectively. As seen in figures; all tested pavement specimens were observed to have negligible concentrations of iron, at all experimental temperature ranges except for aged specimens at 20ºCelsius. The cause of this slight increase in iron concentration for aged specimen at 20ºCelsius is not known. These concentrations are not of environmental significance, since surface waters naturally carry higher concentrations of iron already, 40ppb being typical.



(a) Fresh Specimens

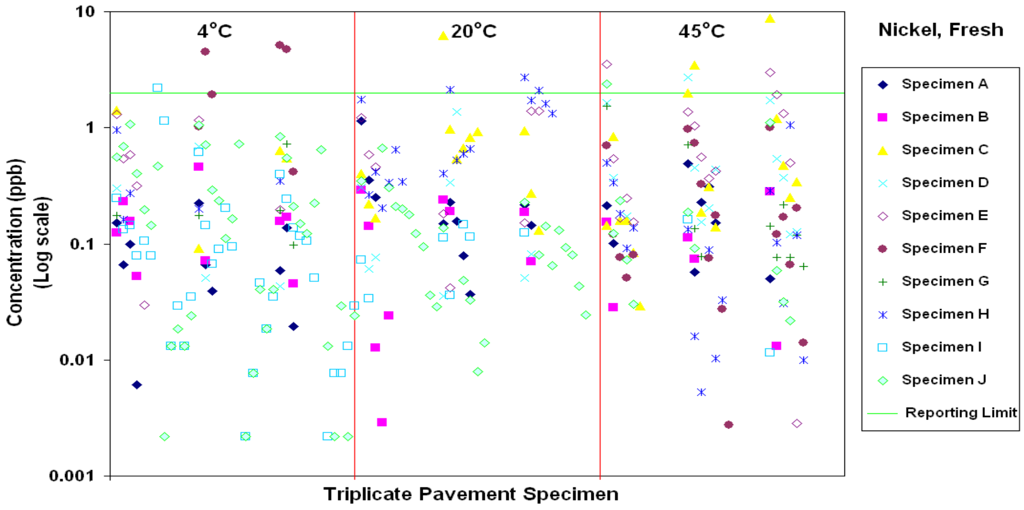


(b) Aged Specimens

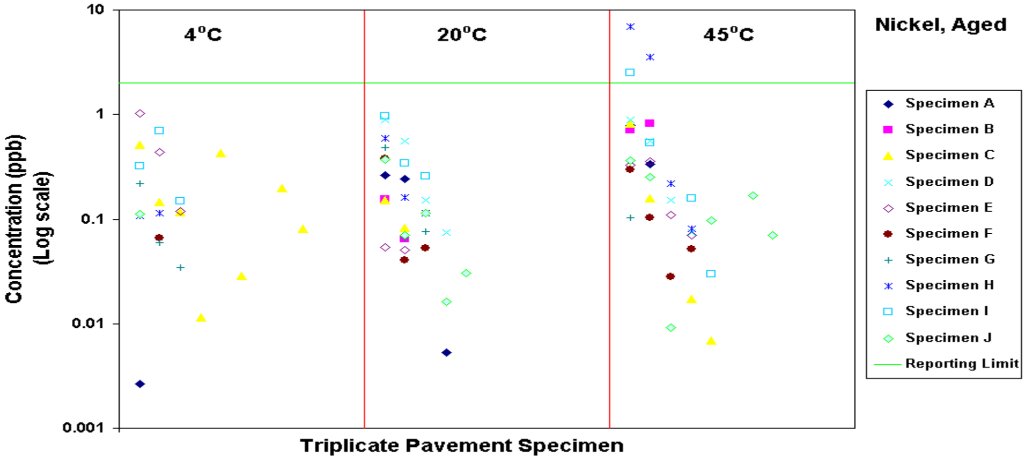
Figure 7-3 – Iron concentrations for all fresh and aged leachate samples tested for three different temperatures

7.2.4. Nickel

The nickel concentration for all samples tested for fresh and aged pavement specimens for three different experimental temperatures are presented in Figures 7-4a and 7-4b, respectively. As seen, majority of tested pavement specimens were observed to have concentrations of nickel below the reporting limits, at all experimental temperature ranges. The limited numbers of leachate samples produced nickel concentration above the reporting limit were not related to any specific pavement type or temperature range.



(a) Fresh Specimens

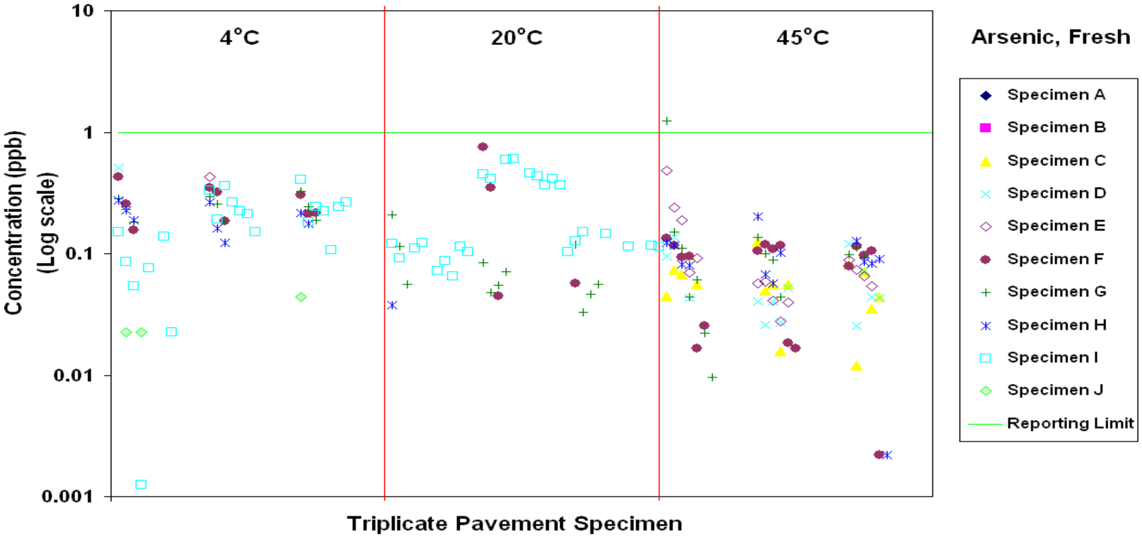


(b) Aged Specimens

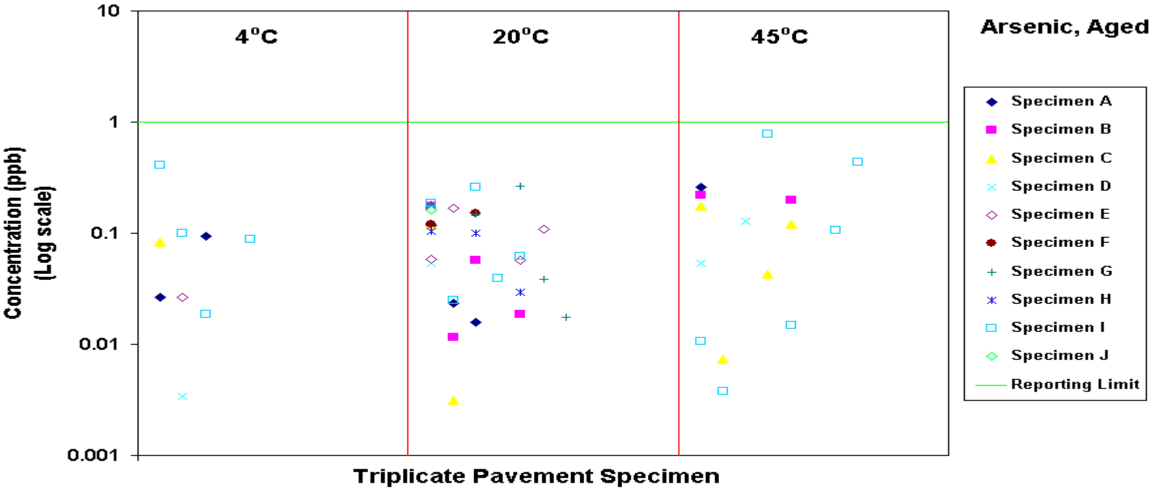
Figure 7-4 – Nickel concentration for all fresh and aged leachate samples tested under three different temperatures

7.2.5. Arsenic

The arsenic concentration for all samples tested for fresh and aged pavement specimens for three different experimental temperatures are presented in Figures 7-5a and 7-5b, respectively. All tested pavement specimens were observed to have negligible concentrations of lead, at all experimental temperature ranges. None of the specimens were observed to have concentration above the reporting limit.



(a) Fresh Specimens

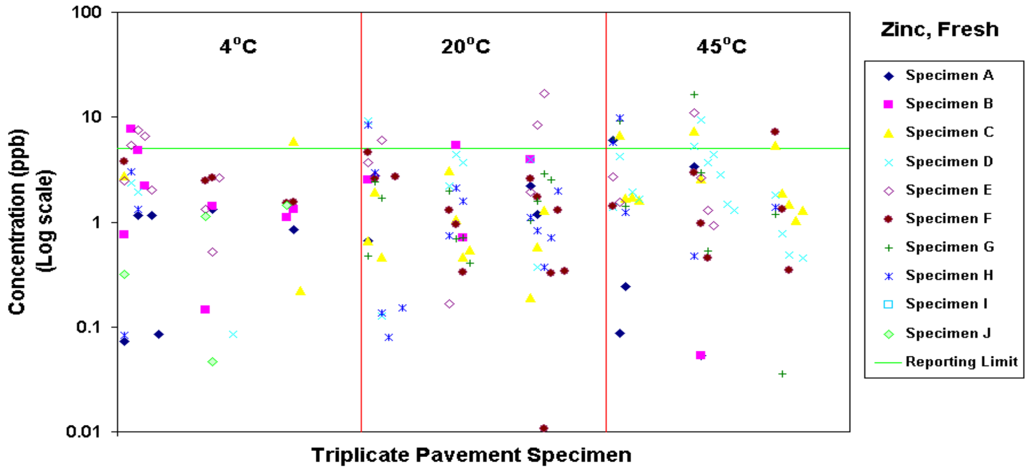


(b) Aged Specimens

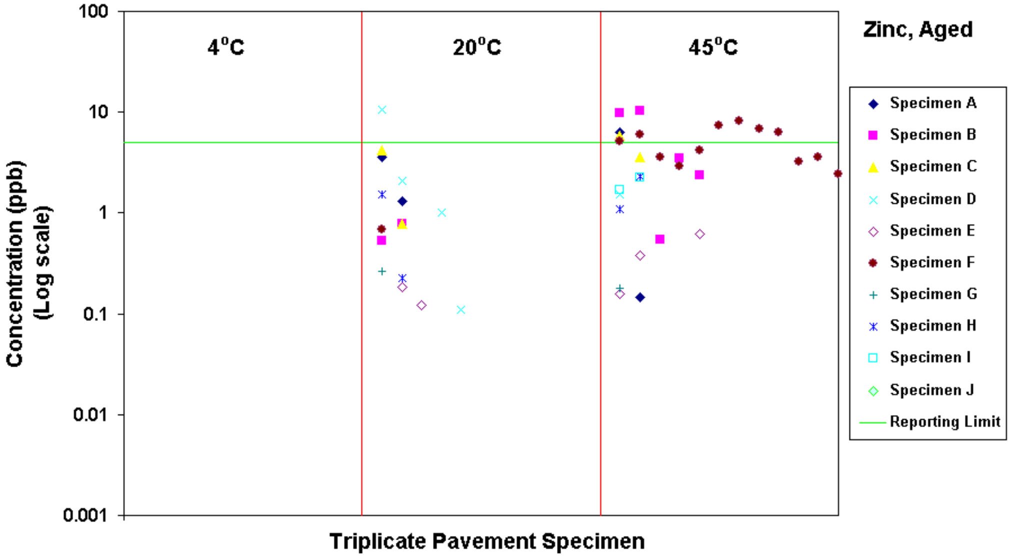
Figure 7-5 Arsenic concentrations for all fresh and aged leachate samples tested under three different temperatures

7.2.6. Zinc

The zinc concentration for all samples tested for fresh and aged pavement specimens for three different experimental temperatures are presented in Figures 7-6a and 7-6b, respectively. Nearly all tested asphalt pavement specimens were observed to have zinc concentrations below reporting limit, at all experimental temperature ranges. Majority of the above reporting limit zinc concentrations were obtained at higher temperatures.



(a) Fresh Specimens

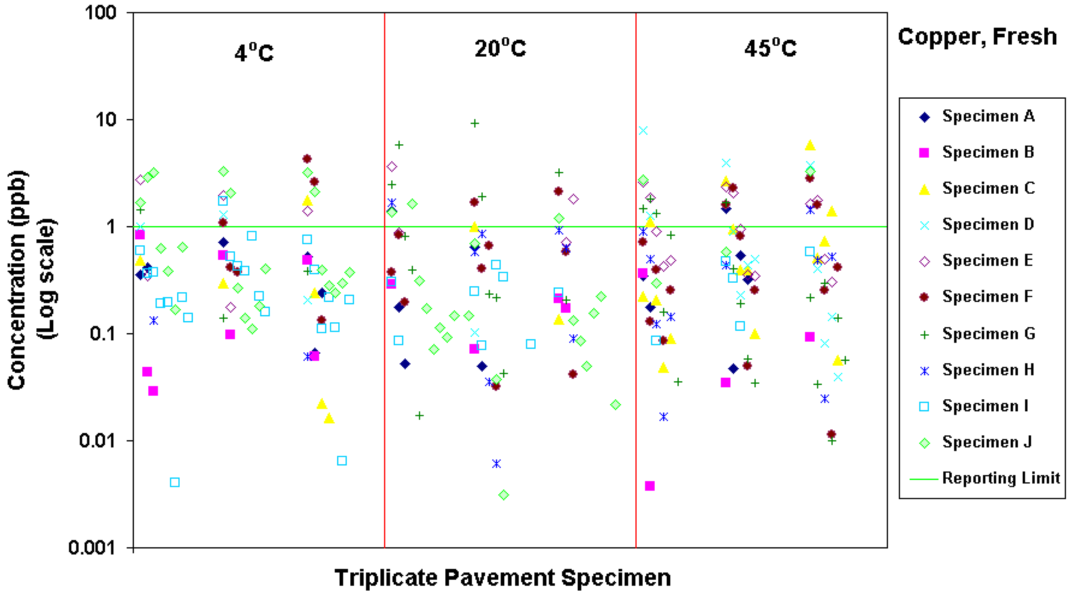


(b) Aged Specimens

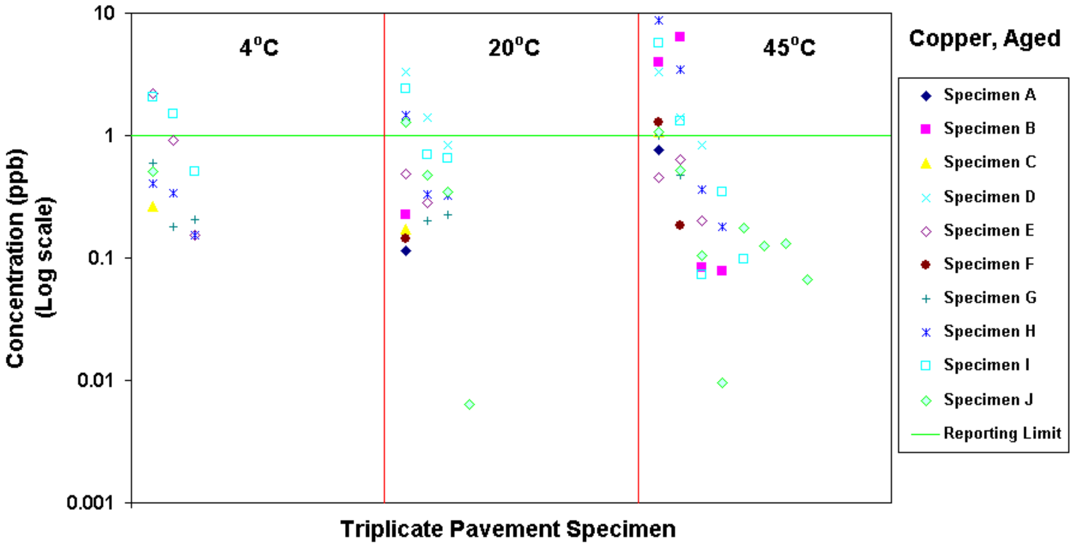
Figure 7-6 Zinc concentrations for all fresh and aged leachate samples tested under three different temperatures.

7.2.7. Copper

The copper concentration for all samples tested for fresh and aged pavement specimens for three different experimental temperatures are presented in Figures 7-7a and 7-7b, respectively. All tested pavement specimens were observed to have some reportable concentrations of copper, at all experimental temperature ranges.



(a) Fresh Specimens

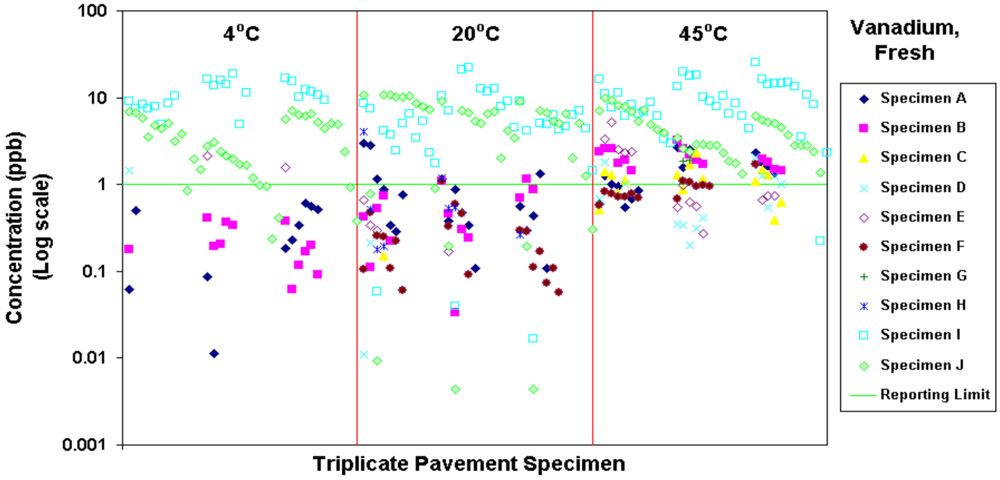


(b) Aged Specimens

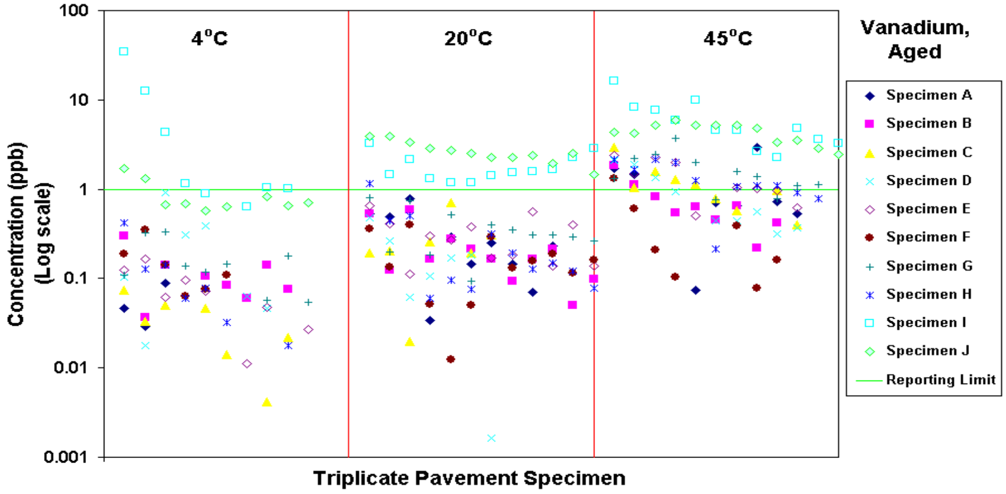
Figure 7-7 Copper concentrations for all fresh and aged leachate samples tested under three different temperatures.

7.2.8. Vanadium

The vanadium concentration for all samples tested for fresh and aged pavement specimens for three different experimental temperatures are presented in Figures 7-8a and 7-8b, respectively. As shown, nearly all the vanadium concentrations above the reporting limit are related to the concrete specimens, while only a few fresh and aged asphalt pavement specimens were observed to have reportable concentrations of vanadium.



(a) Fresh Specimens

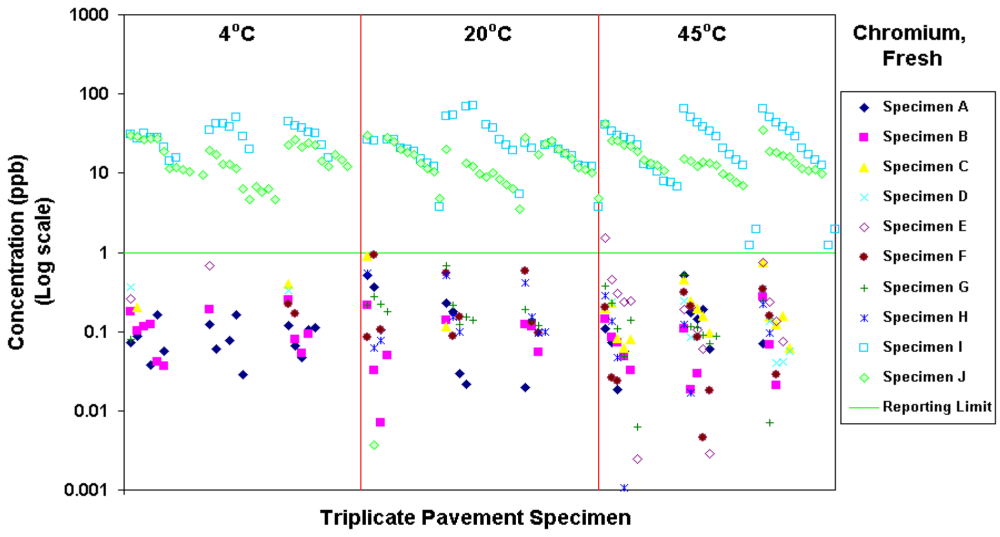


(b) Aged Specimens

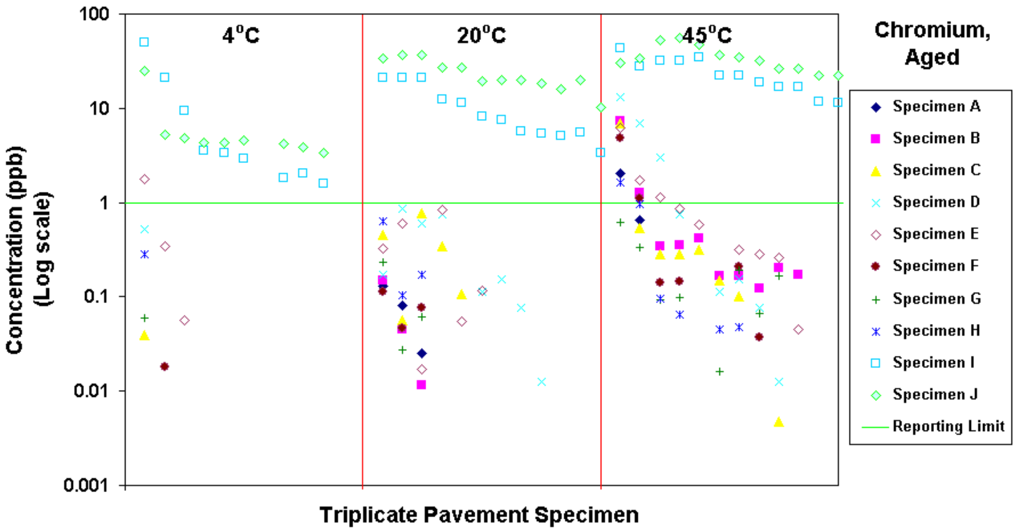
Figure 7-8 Vanadium concentrations for all fresh and aged leachate samples tested under three different temperatures.

7.2.9. Chromium

The chromium concentration for all samples tested for fresh and aged pavement specimens for three different experimental temperatures are presented in Figures 7-9a and 7-9b, respectively. None of the fresh and aged asphalt pavement specimens produced reportable chromium concentrations. Nearly all of the above reporting limit concentrations were related to the two concrete specimens



(a) Fresh Specimens



(b) Aged Specimens

Figure 7-9 Chromium concentrations for all fresh and aged leachate samples tested under three different temperatures.

7.3 Discussion

7.3.1 Variability of Metals Concentrations with Respect to Time

To test for variability of metal concentration with respect to time, samples were taken every fifteen minutes for the first hour of the experiment and every hour thereafter, for the experimental period of 8 hours.

All pavement specimens were observed to have a reportable concentrations of cadmium, lead, arsenic, iron, nickel, copper and zinc for the initial sampling times and were then observed to reduce below detection limit after approximately the first hour. This condition may be explained by the wash-off of soluble metals on surfaces, or colloidal particles and any trace metal constituents associated with them, during the initial sampling period.

For most of the leachate samples, the metal concentrations gradually decreased as the experiment progressed. In nearly all experiments, the detected metal concentrations for most metal constituents decreased below the detection limit within the first four hours, as seen in Figure 7-10 and 7-11. However, chromium and vanadium concentrations in concrete pavement specimens were observed over the entire sampling period as seen in Figure 7.12. The persisting concentration was assumed to be associated with the chemical composition of the concrete specimens, as will be discussed in section 7.3.4.

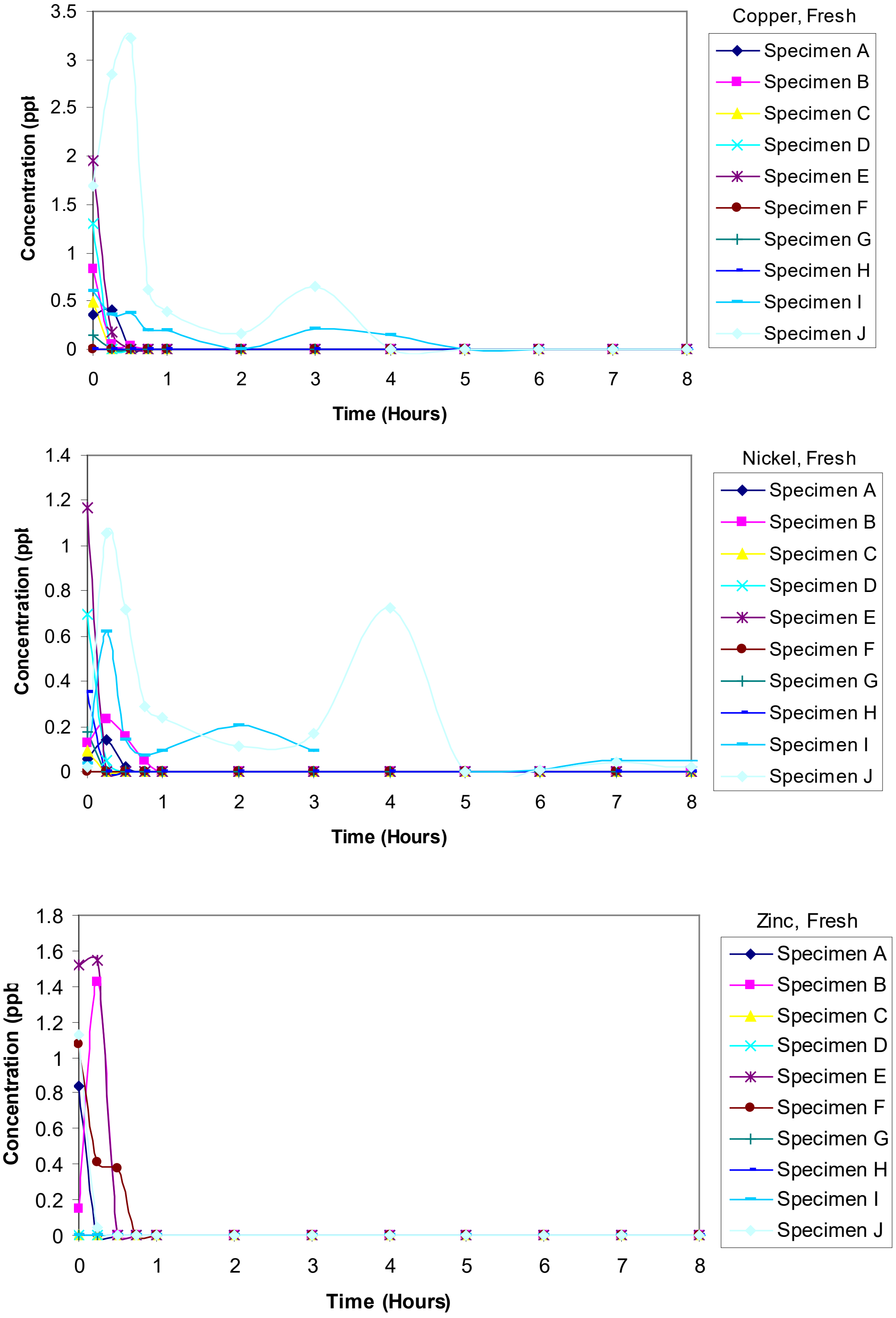


Figure 7-10 Variability of Cu, Ni and Zn concentration for leachate obtained from fresh asphalt and concrete specimens during the experiment
(Note: reporting limit for Cu, Ni, and Zn = 1, 2, and 5, respectively)

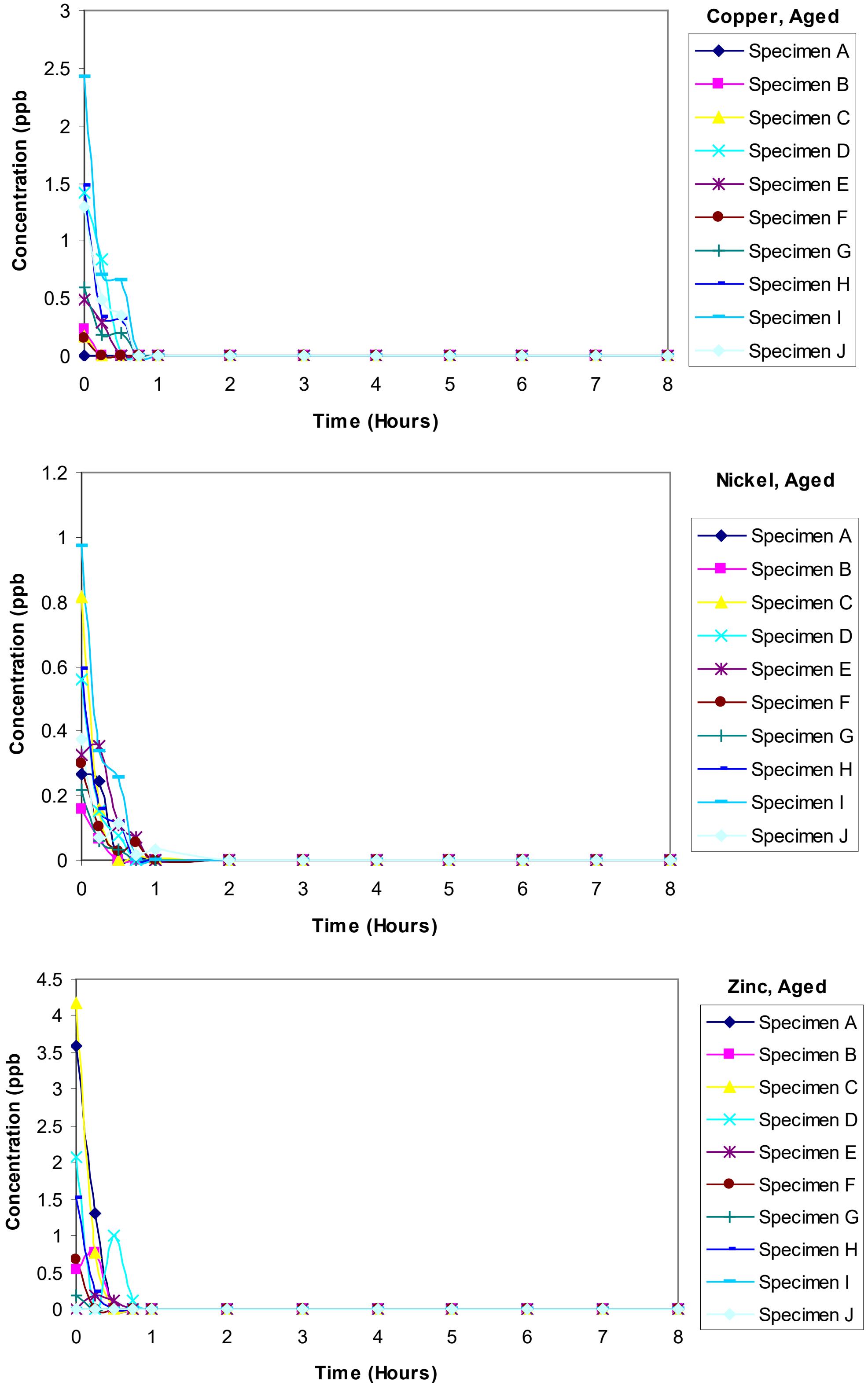
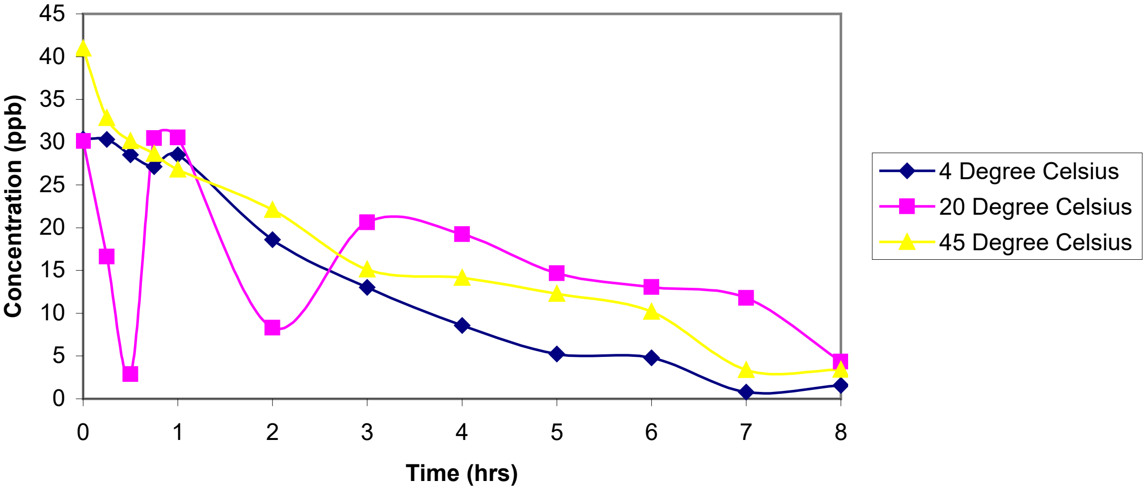
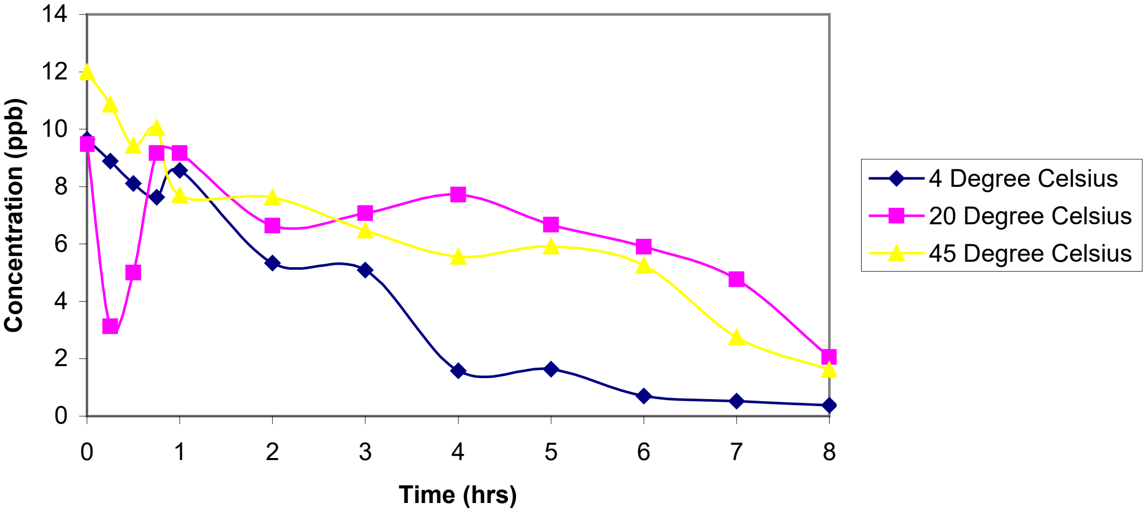


Figure 7-11 Variability of Cu, Ni and Zn concentration for leachate obtained from aged asphalt and concrete specimens during the experiment
(Note: reporting limit for Cu, Ni, and Zn = 1, 2, and 5, respectively)



(a) Chromium



(b) Vanadium

Figure 7-12 Variability of chromium and vanadium concentrations with respect to time, for fresh and aged concrete pavement specimens (I &J) for three different temperatures. (Note: reporting limit for V, and Cr = 0.5 and 1, respectively)

7.3.2 Impact of pH on Metals Leachate Solubility

To investigate the effect of pH on metals leachate solubility, two different methodologies were used. First, the experimental procedure (refer chapter 3) was carried out using modified Milli- Q water adjusted at a pH of 5.2, using specimen B

(Rubberized Asphalt Pavement Specimen). Certain metals (Fe, Cd, Cr, and Ni) were measured at elevated concentration at the adjusted pH, as compared to these metals' concentration at neutral pH. Other metals observed at elevated concentration were zinc, copper, lead and to a lower degree vanadium.

A statistical analysis was performed to evaluate the significance in results obtained under different pH. For the aforementioned metals, zinc, copper, nickel, and lead, the change in concentrations were statistically determined to be significant and the change in results for V and Cr was found to be insignificant at $p<0.10$ level. The high concentration of selected metals under adjusted pH conditions was not solely attributed to pavement materials but instead it was assumed that majority of iron, chromium and nickel concentration was due to corrosion of the steel trays. Likely there is some contribution of metals from the compaction/molding process, which is unavoidable.

To determine the trace metals concentration from corrosion of the metal trays, pH adjusted water was run through an empty metal tray. All other conditions were maintained for this experimental run. The leachates produced from this experiments showed an elevated concentrations of iron, nickel and to a smaller extent chromium, under lower pH. This would confirm the assumption that majority of iron, nickel and chromium were the result of the corrosion of the experimental metal trays. This experimental run was indicative that adjustment of pH during regular testing would have introduced unwanted contamination into metal concentration for the pavement specimens, and would have skewed the results for metal concentrations for the pavement specimens.

The second method used to evaluate the effect of pH on metal concentration was carried out using scraped concrete pavement. This experimental evaluation was performed to simulate the wear and tare of concrete pavements under real vehicular operating conditions. Using pavement particles obtained from the wear and tare of the pavement specimens; the metal leachate concentration was studied for four different pHs, namely 7 ,6 ,5 , and 4. Measurements of chromium, vanadium, lead, cadmium, and arsenic were seen to have statically insignificant differences for change in pH. However, at pH 5 copper, iron and to limited extent zinc were observed to show statistically significant differences in their concentration for all pavements. At these conditions all the pavements under comparison had a low p-value. Lastly nickel, was shown to have statistically significant variations in its concentration for all pavements at pH 4. Using the t-test nickel concentration for all pavements were tested at the above mentioned pH values and were seen to have elevated values at pH 4, as compared to higher pH. However, the nickel concentration observed was below the reporting limit.

7.3.3 Statistical Analysis

To statistically determine the significance in results obtained from different pavements types, different temperatures, and fresh and aged pavement specimens, the t-test was used. The mean for all pavement types, at experimental temperatures and different stages of aging, was compared and the significance in their difference by using the t-test. The p-–value is a numerical estimate of the reliability of the assumptions that the difference between different surveys is real and not due to chance. A p-value greater than 0.10 were considered to be statistically insignificant, while a p-value under 0.10 was considered to be statistically significant, which meant that we are 90% sure that the result observed is not due to chance. In the statistical analysis, comparisons for trace metals leachate concentration; fresh vs. aged pavement specimens, low vs. high temperatures and asphalt vs. concrete pavement specimens, were examined.

Using the t- test for comparison between fresh and aged pavement specimens, it was seen that nickel, lead, arsenic, copper and vanadium were all shown to have statistically insignificant differences in their trace metal concentration due to the effect of aging. Zinc was observed to have a significant difference between aged and fresh pavement specimens at 4 ° C using the t-test, with p values at $p<0.1$.

Chromium was observed to have a significant difference between aged and fresh pavement specimens at all temperatures, with p values at p<.01.

Using the t- test for analysis of the effect of temperature on trace metal concentration, the change in leachate concentration of lead, nickel, copper, vanadium and chromium was statistically insignificant. Cadmium was seen to have statistically significant changes in concentration for asphalt pavement specimen C at 45° C. Pavement materials were observed to indicate statistically significant changes in zinc leachate concentration for aged specimens at 20 ° C and 45 ° C, with both temperatures having p<. 10 values. A significant difference was observed for iron leachate concentration for fresh specimens at 20° C Celsius and aged specimens at 45° C.

Using the t- test for comparison between asphalt and concrete pavement specimens, it was seen that the difference in cadmium, lead, iron, arsenic and copper leachate concentration was calculated to be statistically insignificant for all temperatures. However, a significant difference in zinc concentration was observed between asphalt and concrete specimens at 4° C, with a p- value greater than 0.1. Similarly, statistically significant differences in vanadium and chromium concentration was observed between asphalt and concrete specimens using the t-test, with all of them having p- values greater than 0.1, at all temperatures.

7.3.5 Preliminary Source Identification

As presented earlier, chromium and vanadium were observed at elevated concentrations in concrete pavement specimens. To determine sources of Cr and V, the individual ingredients in concrete pavement specimens were tested separately. The main ingredients of typical concrete pavements are aggregate (Syar rock for this study), Portland cement and water.

Vanadium concentration, in the Syar rock, was observed to be low (6.33 ppb, averaged out for triplicate samples), which indicates that the majority of vanadium was originating from the PCC cement. Also, chromium was not detected in the Syar rock, which is indicative that all the chromium concentration originated from the PCC Portland cement.

After further investigation, it was determined that cement was observed to possess an average value of 600 ppb of chromium and an average value of 62 ppb of vanadium, as can be seen in figure 7.12. These observations confirmed that cement is the main source of chromium and vanadium in the leachate, and it would be worthwhile to test if all commercially produced cement would have equivalent concentration of chromium and vanadium.

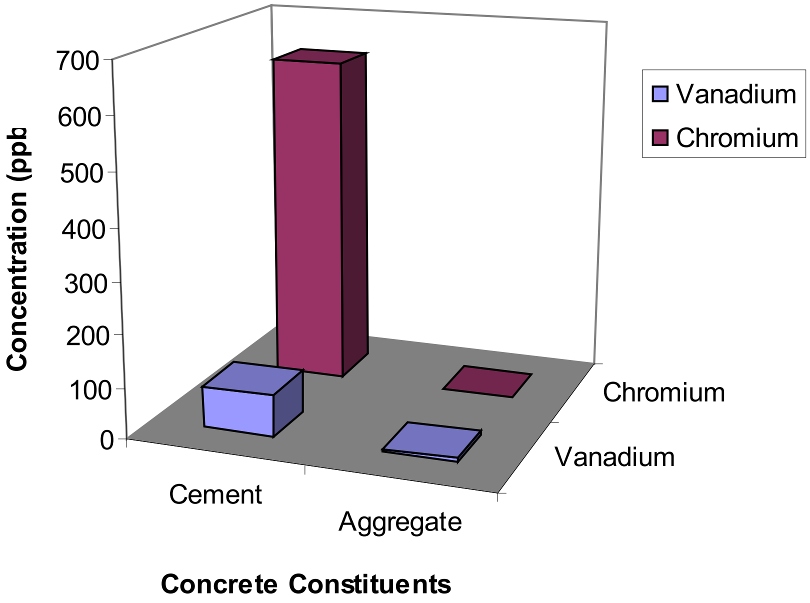


Figure 7-13 Concentration of chromium and vanadium in cement and aggregate used in concrete specimen
(Note: reporting limit for V, and Cr = 0.5 and 1, respectively)

Chapter 8: Organic Constituents Results and Discussion

8.1 Introduction

This chapter presents the results of organic constituents concentration produced from the leachate of ten different fresh and aged pavement specimens under three different temperature of 4, 20, and 45 °C.

Organic constituents studied can generally be categorized as: (1) aggregate organics, (2) polycyclic aromatic hydrocarbons (PAHs), and (3) methylene blue active substance (MBAS). Aggregate organics include: chemical oxygen demand (COD), dissolved organic carbon (DOC), total organic carbon (TOC), oil and grease (O&G), and total petroleum hydrocarbons (TPH).

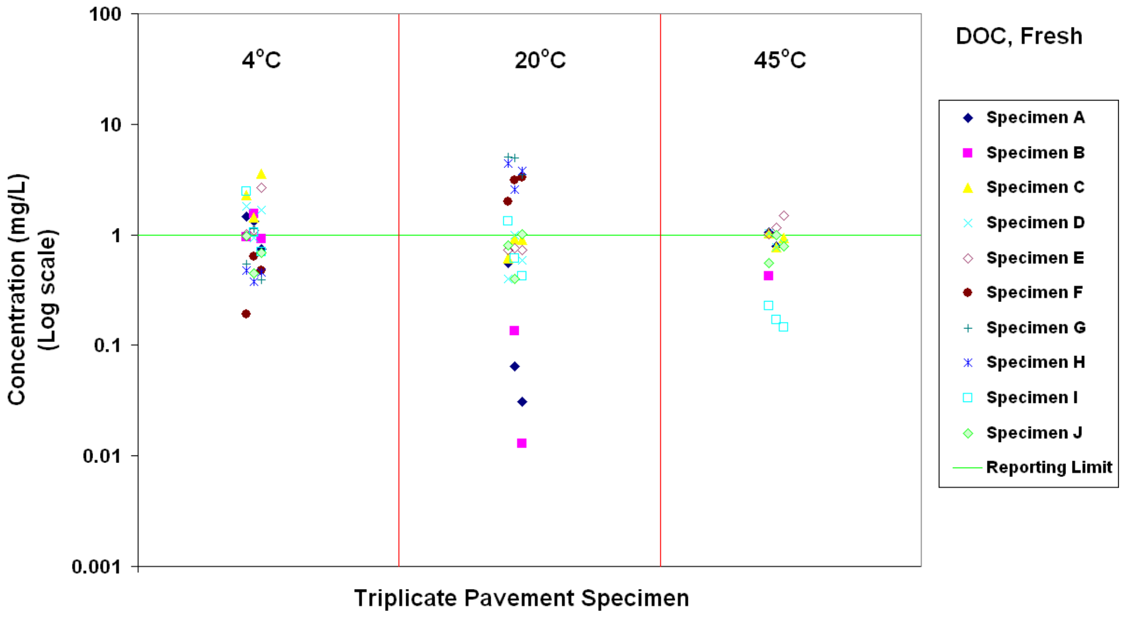
8.2 Aggregate Organics

8.2.1 Chemical oxygen demand (COD)

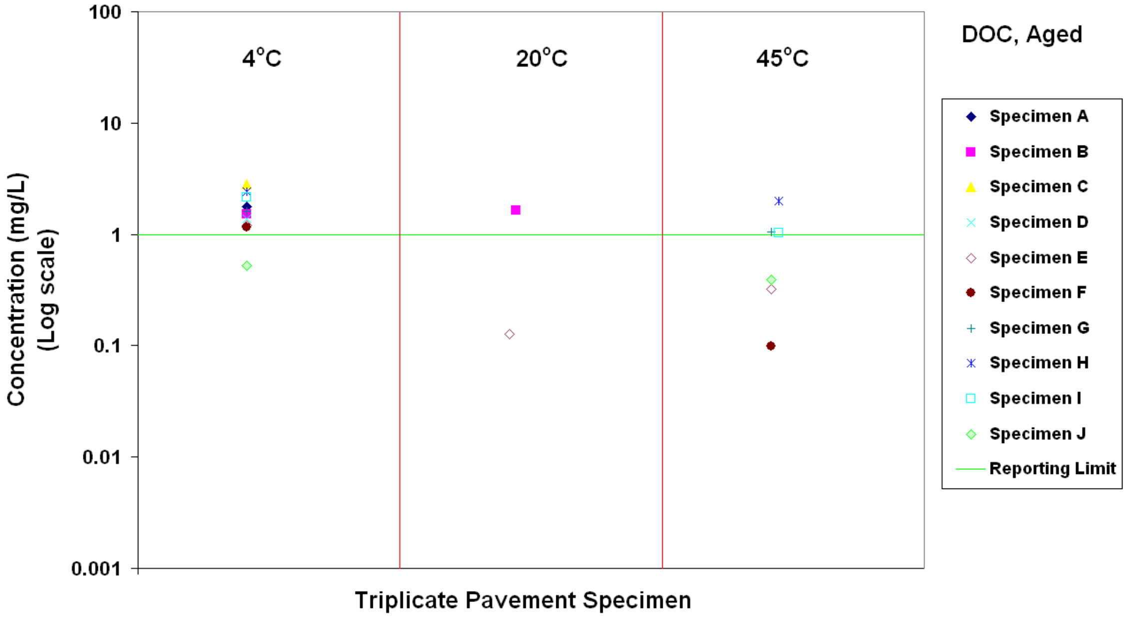
Concentration of chemical oxygen demand (COD) for all leachate produced from fresh and aged pavement specimens were measured under the three different test temperatures were below reporting limit. The change in temperature or aging did not significantly impact the results of COD concentration.

8.2.2 Dissolved organic carbon (DOC)

The results of dissolved organic carbon (DOC) for leachate produced from fresh and aged pavement specimens for three different temperatures are shown in Figures 8-1a and 8-1b. As shown, the majority of DOC results for fresh and aged specimens were below reporting limit. Pavement type and also the change in temperature or aging did not significantly impact the results of DOC concentration.



(a) Fresh specimens

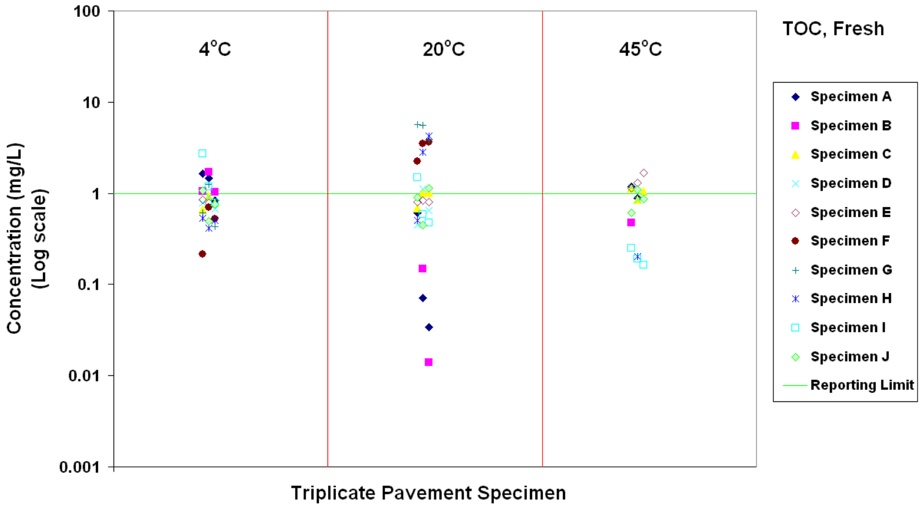


(b) Aged specimens

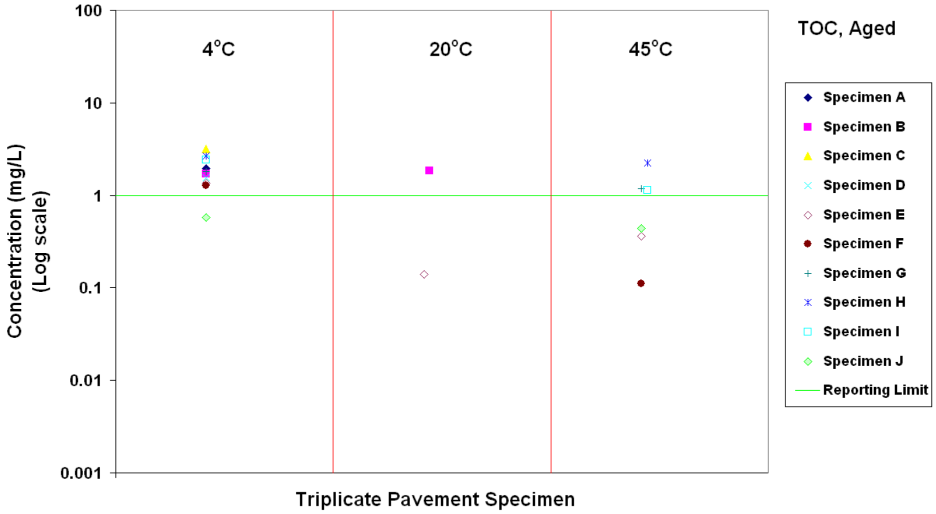
Figure 8-1 DOC concentration for leachate produced from all specimens under three different temperatures

8.2.3 Total organic carbon (TOC)

The concentration of TOC for all leachate produced from fresh and aged pavement specimens for three different temperatures are shown in Figure 8-2a and 8-2b. As shown, most of the TOC results for fresh and aged specimens were below reporting limit. Pavement type and also the change in temperature or aging did not significantly impact the results of TOC concentration. As expected a good correlation between DOC and TOC results were observed and in general the concentrations of DOC was found to be about 90% of TOC concentrations.



(a) Fresh specimens

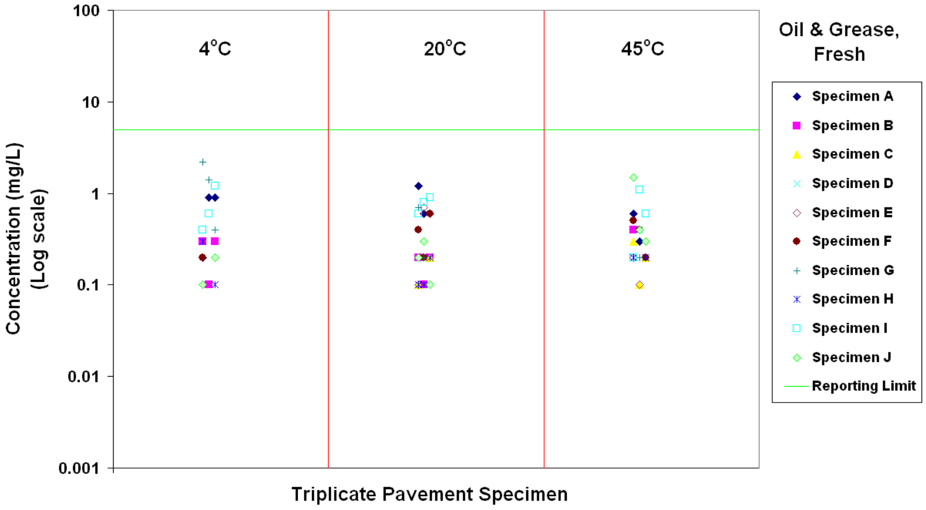


(b) Aged specimens

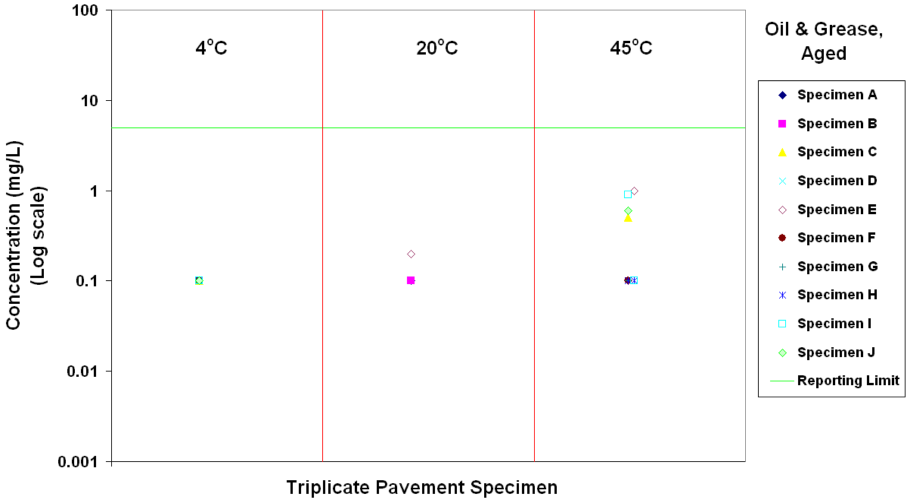
Figure 8-2 TOC concentration for leachate produced from all specimens under three different temperatures

8.2.4 Oil and grease (O&G)

Concentration of oil and grease (O&G) for all leachate produced from fresh and aged pavement specimens for three different temperatures are shown in Figures 8-3a and 8-3b. As shown, all the O&G results for fresh and aged specimens were below reporting limit. It is important to note that even if the reporting limit is set at 1 mg/L, the majority of measured values would still be below the reporting limit. Pavement type and also the change in temperature or aging did not significantly impact the results of O&G concentration.



(a) Fresh specimens



(b) Aged specimens

Figure 8-3 Oil and Grease concentration for leachate produced from all specimens under three different temperatures

8.2.5 Total petroleum hydrocarbons (TPH)

Total petroleum hydrocarbons (TPH) is generally used to describe a large family of several hundred chemical compounds that originally come from crude oil. Because there are so many different chemicals in crude oil and in other petroleum products, it is not practical to measure each one separately. However, it is useful to measure the total amount of TPH that maybe a mixture of chemicals such as hexane, jet fuels, mineral oils, benzene, toluene, xylenes, naphthalene, and fluorene, as well as other petroleum products including asphalt materials [ATSDR, 1999]. There are different methods of identification and analysis of TPH available, i.e. EPA 418.1, GC-MS, PetroFLAG method, Freon-extraction method, total carbon approach, and external standards method (ESTD). In this study GC-MS method was used in order to measure the total petroleum hydrocarbon. Different binders that used in tested pavement specimens that could contribute to the TPH concentration is summarized in Table 8-1. The concentration of TPH for leachate produced from fresh and aged specimens were generally below reporting limit. It is, however, believed that nearly all TPH concentration is attached to particles and these particle-bound TPH may result from ware and tare of asphalt pavements. The measurement of particulate TPH is beyond the scope of this project and need to be investigated through separate study. Equally important, specimens I and J were concrete pavement specimens and do not contain any asphalt or other petroleum binder, and hence the leachates produced from these pavements are therefore expected to produce no measurable TPH.

Table 8-1 Different binders as possible contributors of TPH

Specimen	Mix Type	Binder	Modified	Gradation	Binder Content
A	RAC-O Rubberized Asphalt Concrete Open Graded	***Rubberized*** asphalt cement	Yes	Open	7.5%
B	RAC-G Rubberized Asphalt Concrete Gap Graded	***Rubberized*** asphalt cement	Yes	Gap	8.0%
C	OGAC Open Graded Asphalt Concrete	Unmodified Performance Grade binders ***(PG64-10)***	No	Open	7.0%
D	OGAC Open Graded Asphalt Concrete	Unmodified Performance Grade binders ***(PG64-16)***	No	Open	7.0%
E	OGAC-PM Open Graded Asphalt Concrete Polymer Modified	Modified PG binders ***(PBA-6A)***	Yes	Open	6.0%
F	OGAC-PM Open Graded Asphalt Concrete Polymer Modified	Modified PG binders ***(PG64-28)***	Yes	Open	5.5%
G	MB-G Modified Asphalt Binder	Modified Binder ***(MAC15)***	Yes	Gap	5.5%
H	DGAC Conventional Dense Graded Asphalt Concrete	Binder – Unmodified ***(PG64-16)***	No	Dense	5.5%
I	PCC-Dense	Portland Cement	No	Dense	
J	PCC-Permeable	Portland Cement	No	Open	

8.3. Organic Compounds

8.3.1 Polycyclic Aromatic Hydrocarbons (PAHs)

The sample volume collected for PAHs measurement was 4 L and Liquid-liquid method was used as the extraction method. This method was essentially used to remove organic compounds from aqueous samples by adding a non-miscible, non-polar phase (in this case hexane) to the aqueous phase [Skoof et al., 1998]. Non-polar chemicals are partitioned, followed by extractions and then concentrated down to 1 mL and used for PAHs measurement by Gas Chromatography-Mass Spectrometry (GC-MS) instrument.

There are 17 PAHs compounds identified by Environmental Protection Agency (EPA) of concern regarding water quality parameters. The complete list of these 17 PAHs are shown in Table 8-2. In general, all 17 PAH compounds produced from the leachate of both fresh and aged pavement specimens were below detection limit. The concentration of dissolved PAH compounds in all leachate produced from fresh and aged pavement specimens were below the detection limit. However, a detectable concentration of phthalates was consistently measured during most experiments. Phthalates is among an organic compound that that is associated with plasticizers. The presence of Phthalates in pavement specimens was suspected to be related to sources other than pavement specimens. After running some laboratory-blank samples without pavement specimens, it was determined that the detected Phthalates were related to the plastic wraps that had been used to protect the specimens prior transporting them from Richmond pavement research center to UC Davis.

While no dissolved PAHs were detected, particulate PAHs may result from wear and tare of asphalt pavement materials due to the mechanical interaction between pavement and tires. Preliminary investigation showed that at least five particulate PAHs compound were detected when asphalt pavement particles were analyzed. Previous study has also shown that particulate PAHs were more present in stormwater runoff in both frequency and detectable concentration (Lau et al. 2005). It is not, however, clear as what fraction of detected particulate PAH in stormwater runoff is related to asphalt pavement material. This aspect of the study was beyond the scope of the current project and need to be investigated in future studies.

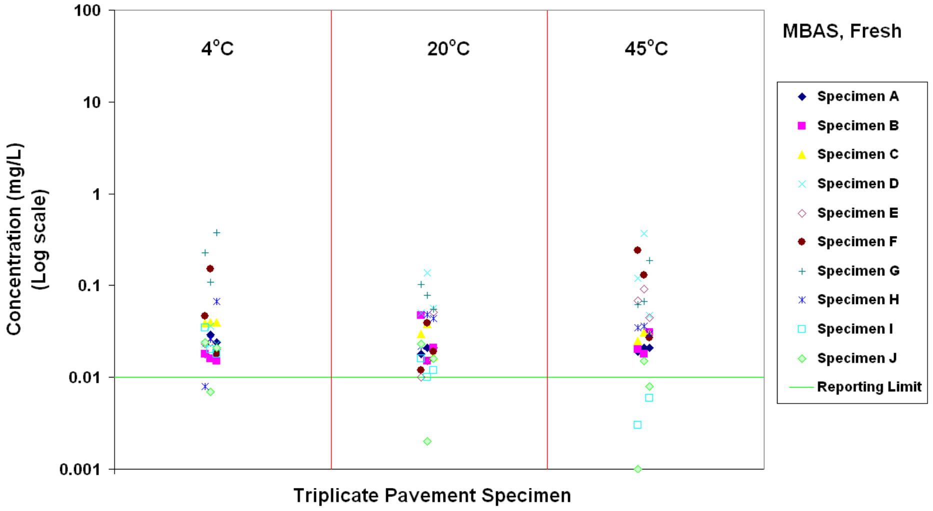
Table 8-2 Characteristics of 17 PAHs organic compounds

PAH	*Abbreviation*	*Molecular Formula*	*Molecular Mass (g/mol)*	*Solubility In water (mg/L)*	*Log (K_{ow})^a*
Naphthalene	NaP	$C_{10}H_8$	128.17	30	
Acenaphthylene	Acy	$C_{12}H_8$	152.19	Insoluble	3.94
Acenaphthene	Ace	$C_{12}H_8$	154.21	Insoluble	4.15
Fluorene	Flu	$C_{13}H_{10}$	166.22		4.18
Phenanthrene	Phe	$C_{14}H_{10}$	178.23		4.57
Anthracene	Ant	$C_{14}H_{10}$	178.23	Insoluble	4.45
Fluoranthene	Fle	$C_{16}H_{10}$	202.25		5.22
Pyrene	Pyr	$C_{16}H_{10}$	202.25	0.135	
Benzo(a)anthracene	BaA	$C_{18}H_{12}$	228.29		5.79
Chrysene	Chr	$C_{18}H_{12}$	228.29	Insoluble	5.73
Benzo(b)fluoranthene	BbF	$C_{20}H_{12}$	252.31		6.11
Benzo(k)fluoranthene	BkF	$C_{20}H_{12}$	252.31		6.11
Benzo(a)pyrene	BaP	$C_{20}H_{12}$	252.31		6.13
Indeno(1,2,3-cd)pyrene	IcP	$C_{22}H_{12}$	276.33		6.72
Dibenzo(a,h)anthracene	DaA	$C_{22}H_{14}$	278.35		6.50
Benzo(ghi)perylene	BgP	$C_{22}H_{12}$	276.33		6.90
Coronene	Cor	$C_{24}H_{12}$	300.35		

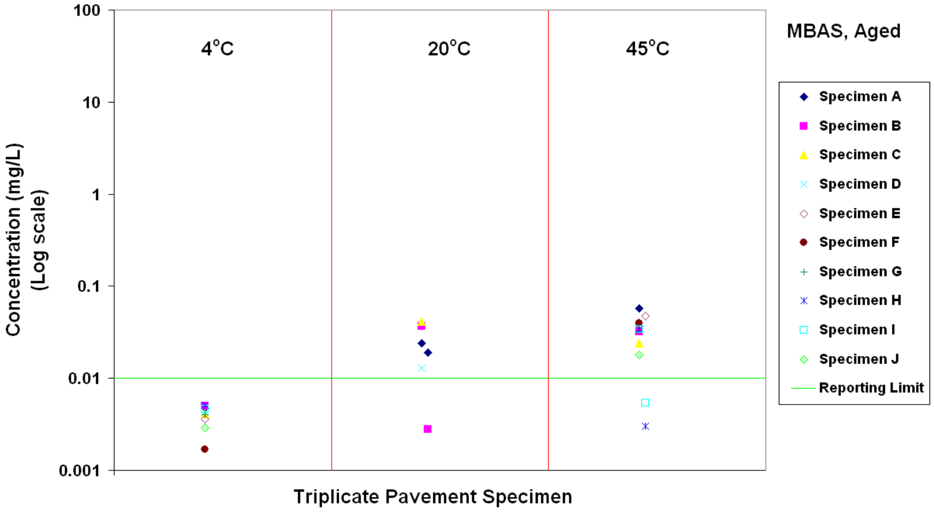
[a] octanol-water partition coefficients (K_{ow}) taken from Odabasi et al. [2006].

8.3.2 Methylene Blue Active Substance as Surfactants (MBAS)

The term surfactant is a short form for "surface acting agent". Surfactants are mainly organic compounds that are amphiphilic, meaning that they contain both hydrophobic and hydrophilic groups in which make them soluble in both organic solvents and water. Surfactants reduce the surface tension of water by adsorbing at the liquid-gas interface. The main conventional method for determining surfactants in the environment is colorimetric procedures such as Methylene Blue Active Substances (MBAS) for anionics, Bismuth Active Substances (BiAS) for nonionics and Disulfine Blue Active Substances (DBAS) for cationics [Matthijs and Hennes, 1991]. In this study, MBAS method was used to quantify the presence of surfactants in asphalt pavement specimens. The concentration of MBAS for leachate samples produced from both fresh and aged pavement specimens under three different temperatures are shown in Figures 8-4a and 8-4b. As shown, detectable amount of MBAS were measured for most asphalt pavement specimens. Temperature and aging did not significantly impact the MBAS concentration. The maximum detectable MBAS resulted from dissolved leachate samples was below 1 mg/L. A higher particulate concentration of MBAS may result from particulate asphalt material that could be generated due to wear and tare of mechanical interaction which may be investigated through future study.



(a) Fresh specimens



(b) Aged specimens

Figure 8-4 MBAS concentration for leachate produced from all specimens under three different temperature

Chapter 9: Toxicity Results and Discussion

This section of the report presents the results and discussions associated with toxicity associated with all pavement specimens (asphalt and concrete). This chapter is organized to present: (1) methods, (2) results and discussions, and (3) toxicity data reliability quality assurance/quality control.

9.1. Methods

Toxicity testing for fathead minnow larvae (*Pimephales promelas)*, *Ceriodaphnia dubia* (a cladoceran, zooplankton species) and the green alga *Selenastrum capricornutum* followed the 7 and 4-day static renewal procedures described in "Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (US EPA, 2002). Tests were set up within 36 h of sample receipt.

Three control experiments were performed during the design phase of the project including: (1) toxicity of tap water, (2) suitability of artificial rainwater for testing, and (3) test sensitivity.

A test was conducted to determine if tap water could be used for experiments or if it was toxic to the test organisms. Tap water was used fresh from the UC Davis tap water system, or aerated for 16 h then tested to determine if aeration would alleviate toxicity due to chlorine. In addition, STS was added to the test water (0.08 mg/L) to determine if the addition of STS would alleviate toxicity due to chlorine. Fresh tap water was tested without any additional manipulations to determine if tap water is toxic to *C. dubia* or green algae.

A series of tests was performed to evaluate the suitability of artificial rainwater for testing with *C. dubia* and *S. capricornutum*.

To verify that *C. dubia* and green algae were, in fact, more sensitive than larval fathead minnow to asphalt and concrete runoff samples, several samples, F-20, G-20 and H-20, were tested with all three species.

9.1.1 Sampling ID Notation

A specific notation has been used to identify the leachate samples from different pavement specimens and temperatures. This sampling ID was unique to toxicity evaluation as shown in Table 1. This unique sampling ID is used throughout this section when presenting the toxicity results.

Table 9-1 Sample ID notation for leachate samples tested.

Specimen stage	Specimen type	Temperature	Triplicate number	Sample ID
Fresh or Aged	A, B, C, D, E, F, G, H, I, J	4, 20, and 45 °C	1, 2, or 3	Stage-type-temp-triplicate
Examples: Fresh-A-4-1; Fresh-I-20; Aged-F-45-3; Aged-J-45				

9.1.2. Toxicity Testing with Green Algae (*Selenastrum capricornutum*)

The *S. capricornutum* 96-hour chronic tests consist of four replicate 200 ml glass flasks with 100 ml of sample and 1 ml of 1.0 x 10^6 cell/ml *S. capricornutum*. Glass distilled water is the control for the *S. capricornutum* test. Test chambers were incubated in a temperature-controlled environmental chamber maintained at 25 ± 2º C under continuous cool white fluorescent light (400 +/- 40 ft candles). Test chambers were kept in a mechanical shaker in constant orbital motion at 100 cycles/minute; flasks were randomized twice daily. At test termination (day 4) algal cell concentrations were measured using a Coulter counter.

9.1.3 Toxicity Testing with Waterflea (*Ceriodaphnia dubia*)

The *C. dubia* chronic tests consisted of ten replicate 20 ml glass vials each containing one organism. Tests were initiated with less than 24-hour-old *C. dubia*, born within an 8-hour period. *C. dubia* were fed a mixture of S. capricornutum and YCT (a mixture of yeast, organic alfalfa and trout chow) daily. *C. dubia* were transferred into a new vial of fresh solution daily. When conductivity was below 100 uS/cm samples were amended to US EPA specifications. Sierra Springs^{TM} water amended to US EPA moderately hard (SSEPAMH) water is used as the control water. Tests were conducted at 25 ± 2° C with a 16-hour light: 8-hour dark photoperiod. Mortality and reproduction (number of neonates) were assessed daily and at test termination (day 7).

9.1.4 Toxicity Testing with Larval Fathead Minnow (*Pimephales promelas*)

The *P. promelas* chronic tests consist of four replicate 600 ml glass beakers each containing 250 ml of sample and 10 organisms. Tests were initiated with less than 24-hour-old *P. promelas,* which are obtained from Aquatox in Hot Springs, Arkansas. Each replicate is fed freshly hatched *Artemia* nauplii twice daily. Approximately 80% of the test solution is renewed daily, while removing dead fish, *Artemia,* and debris from the test beakers. Deionized water amended to EPA moderately hard (DIEPAMH) was used as the control water for the *P. promelas* test. Tests were conducted in 25 ± 2° C water baths with a 16-hour light: 8-hour dark photoperiod. Mortality was measured daily upon test sample renewal and upon test termination (day 7). At test termination the surviving minnows were anesthetized with MS-222, rinsed with deionized water, dried to constant weight at 103-105° C (approximately 16 hours), and weighed with a Mettler AE 163 balance.

9.1.5 Water Chemistry

Prior to *C. dubia, S. capricornutum, and P. promelas* test initiation, temperature, dissolved oxygen (DO), pH, electrical conductivity (EC), alkalinity, hardness, and ammonia were measured. The *S. capricornutum* test had pH measured daily. In the *C. dubia* and *P. promelas* tests, DO was measured in the daily renewal water, and DO and pH were measured in the 24 hour old water. Temperature was monitored continuously throughout all toxicity tests.

9.1.6 Quality Assurance/Quality Control (QA/QC)

All UCD ATL procedures followed a stringent QA/QC plan consistent with the US EPA QA guidelines. Toxicity tests were initiated within 36 h of sample collection. To determine whether test species were responding typically, positive reference toxicant tests were conducted monthly to ascertain organism health, sensitivity and laboratory performance. All tests were evaluated by the UCD ATL Quality Assurance Officer and met specified US EPA criteria.

9.1.7 Data Analysis and Reporting

Each sample was characterized by descriptive statistics indicating the mean response and variation among replicates. Statistical comparisons of test organism performance in sample water to the response in laboratory control water relied on USEPA standard statistical protocols (USEPA 2002). Toxicity is defined as a statistically significant difference in a test endpoint (one-tailed test, $P < 0.05$) in an ambient sample compared to a laboratory control. Specifically, acute toxicity in *C. dubia* and larval *P. promelas* toxicity assays is defined as statistically significant mortality within 96 hours in a test sample compared to the laboratory control. Chronic toxicity in *C. dubia* and larval *P. promelas* toxicity assays is defined as a statistically significant reduction in 7-day survival, reproduction (*C. dubia*), or biomass (*P. promelas*). Toxicity in the *S. capricornutum* toxicity assay is defined as a statistically significant reduction in cell density when compared to a laboratory control at test termination. Statistical comparisons other than control v. sample were made using one-way ANOVA with Tukey's multiple comparison procedure (two-tailed test, $P < 0.05$). All toxicity data was analyzed using JMP 5.0.1 software. All data produced during this period were quality assurance checked, and spreadsheet entries were double checked for accuracy.

9.2 Results and Discussions

9.2.1 Toxicity of Tap Water to Green Algae and Waterflea

Fresh tap water was highly toxic to green algae (Table 9-2), and STS did not reduce toxicity indicating that chlorine remained in the water at highly toxic concentrations. Aerating tap water for 16 h alleviated algal toxicity, but water was still toxic and algal cell counts were significantly lower than in control water.

Table 9-2 Results of a *S. capricornutum* 96-hour test initiated 11/04/06 evaluating the toxicity of tap water with and without aeration or the addition of 0.08 mg/L sodium thiosulfate (STS).[1]

Treatment	Cells/mL (x 10^6)[1]		% CV	Significance Group
	x	se		
Glass Distilled	2.556	0.045	3.5	A
Aerated Tap Water	0.433	0.047	21.7	B
Fresh Tap Water	0.014	0.001	10.0	C
Fresh Tap Water + 0.08 mg/L STS	0.014	0.001	12.8	C

1. Treatments falling into different significance groups (A, B, C) showed significantly different concentrations of algal cells. (one-way ANOVA, Tukey's multiple comparison procedure, $P < 0.05$)

x = mean cell count (n=4), se = standard error of the mean

Fresh tap water was also toxic to *C. dubia* (Table 9-3), significantly reducing survival and reproductive success. Aeration and the addition of STS reduced acute toxicity, but reproductive success was still significantly impaired in both treatments.

Table 9-3 Results of a *C. dubia* 7-day test initiated 11/04/06 evaluating the toxicity of tap water with and without aeration or the addition of 0.08 mg/L sodium thiosulfate (STS).[1]

Treatment	Survival (%)[1]		Significance Group	Reproduction (offspring)[1]		Significance Group
	x	se		x	se	
SSEPAMH (Laboratory Control)	100	0.0	A	24.2	1.31	A
Fresh Tap Water + 0.08 mg/L STS	100	0.0	A	17.4	1.87	B
Aerated Tap Water	100	0.0	A	14.8	1.55	B
Fresh Tap Water	40	16.3	B	5.8	1.65	C

1. Treatments falling into different significance groups (A, B, C) showed significantly different *C. dubia* survival or reproduction. (one-way ANOVA, Tukey's multiple comparison procedure, $P < 0.05$)

x = mean cell count (n=4), se = standard error of the mean

9.2.2 Toxicity of Artificial Rainwater

Initially, we intended to use artificial rain water for all laboratory experiments where the pH of the Mili-Q water was adjusted to about 5.5 and minerals were added. Toxicity testing of this artificial rainwater revealed a consistent decrease in reproductive success of *C. dubia*, and was toxic to green algae. Detailed toxicity test results of artificial rainwater are shown in the Appendix, Tables A1-4 (*C. dubia*) and A5-10 (*S. capricornutum*). For this reason, all subsequent laboratory testing was performed with Mili-Q water, where pH and minerals were adjusted to US EPA specifications.

9.2.3 Sensitivity of Test Organisms

Test results showed that larval fathead minnow were less sensitive to leachate samples compared with *C. dubia* or *S. capricornutum*. While two of the samples tested with all three species were toxic to waterflea and green algae, no toxicity was detected with larval fathead minnows (Table 9-4).

Table 9-4 Results of *P. promelas* and *C. dubia* 7-day chronic toxicity tests, and 96-h *S. capricornutum* test evaluating the toxicity of water samples filtered through experimental asphalt and concrete at 20° C.

		P. promelas		***C. dubia***		***Algae***
Sample	**Date**	**7-Day Survival**	**7-Day Biomass**	**Survival**	**Mean Offspring**	**Cell Count ($x10^6$)**
Lab Control	-	100.0	0.570	90	18.2	3.028
Fresh-F-20	7/11/2007	100.0	0.571	0	0	0.038
Fresh-G-20	7/11/2007	100.0	0.623	100	11.8	0.081
Fresh-H-20	7/11/2007	92.5	0.580	0	0.0	0.024

1. Highlighted areas indicate significant reduction in survival or biomass compared to the DIEPAMH control. Data were analyzed using USEPA standard statistical protocols.

9.2.4 Toxicity of Leachate Samples at three Different Temperatures

The majority of leachate samples resulting from experimental asphalt and concrete treatments conducted at 4°C were not toxic to *C. dubia* with the exception of J-4 (both fresh and aged), and aged H-4C, which caused reduced reproductive success (Tables 9-5, 9-8). Aged I-4C and aged F-4C caused some reduction in reproduction, but results were not consistent between samples collected on Nov-26 and Nov-27,

2007. Very little algal toxicity was observed in these samples. With the exception of sample C-4-3, which contained <10% of the number of cells in controls, the reduction in algal cell counts was small but significant in samples C-4-2, E-4-2, F-4-2, G-4-3, J-4 and aged J-4C.

The majority of leachate samples resulting from experimental asphalt and concrete treatments conducted at 20°C were not toxic to *C. dubia* (Tables 9-6, 9-9). Several samples that had shown no toxicity or only slight to medium algal toxicity at 4°C, were highly toxic to *C. dubia* as well as algae. These were samples C-20, F-20 and H-20. In tests, samples F-20-1, F-20-2, F-20-3 and E-20 caused reductions in *C. dubia* reproduction and algal growth. After trouble-shooting, it was determined that use of non-precleaned sampling bottles may have contributed to the observed toxicity in some of these samples. However, "bottle blank" samples tested with this type of bottle only caused mild reproductive toxicity in *C. dubia*, and it does not appear that the sampling bottles were the sole cause of the toxicity. One other sample, J-20, also caused 100% *C. dubia* mortality and significantly reduced algal growth. This experimental treatment had also caused toxicity at 4°C albeit toxicity was less severe. In addition, sample I-20 reduced reproductive output of *C. dubia*, and aged G-20C, aged H-20C and aged J-20C caused reduced *C. dubia* reproduction and algal growth.

Toxicity was seen in the majority of leachate samples resulting from experimental asphalt and concrete treatments conducted at 45°C (Tables 9-7, 9-10). Most of these caused a reduction in reproductive output of *C. dubia* (A-45, B-45, E-45, G-45, H-45, I-45, J-45, aged F, aged H, aged I, aged J). Only one sample, D-45, caused a significant reduction in *C. dubia* survival, but survival was somewhat reduced in most samples. Algal growth was significantly reduced in samples F-45, G-45, H-45, J-45, aged A-E, aged F and aged J.

Table 9-5 Results of *C. dubia* 7-day chronic and *S. capricornutum* 4-day toxicity tests evaluating the toxicity of water samples filtered through experimental asphalt and concrete at 4° C. Shaded cells indicate significantly lower survival of *C. dubia* or lower *S. capricornutum* cell count compared to controls ($p<0.05$).

		C. dubia		**Algae**
Sample	**Date**	**Survival**	**Mean Offspring**	**Cell Count (x10^6)**
Lab Control	-	100	24.9	3.933
Fresh-A-4	8/15/2007	100	23.5	3.476
Fresh-B-4	8/15/2007	80	20.6	3.817
Lab Control	-	100	18.0	3.282
Fresh-C-4-1	6/21/2007	100	22.1	3.003
Fresh-C-4-2	6/21/2007	100	20.1	2.924
Fresh-C-4-3	6/21/2007	100	16.1	0.188
Fresh-D-4-1	6/21/2007	100	19.5	3.175
Fresh-D-4-2	6/21/2007	100	19.9	3.084
Fresh-D-4-3	6/21/2007	100	19.1	2.824
Fresh-E-4-1	6/21/2007	100	20.6	3.448
Fresh-E-4-2	6/21/2007	100	20.5	2.936
Fresh-E-4-3	6/21/2007	100	21.7	3.005
Lab Control	-	90	14.4	2.939
Fresh-F-4-1	6/23/2007	100	23.6	2.702
Fresh-F-4-2	6/23/2007	100	20.5	2.615
Fresh-F-4-3	6/23/2007	100	18.7	2.768
Fresh-G-4-1	6/23/2007	80	15.4	2.693
Fresh-G-4-2	6/23/2007	100	20.8	2.728
Fresh-G-4-3	6/23/2007	100	19.4	2.573
Fresh-H-4-1	6/23/2007	100	17.3	3.058
Fresh-H-4-2	6/23/2007	90	18.3	2.893
Fresh-H-4-3	6/23/2007	90	21.0	2.961
Lab Control	-	100	25.4	2.943
Fresh-I-4	10/5/2007	100	22.5	2.891
Fresh-J-4	10/5/2007	70	12.7	2.590
Lab Control	-	100	20.7	3.213
Aged-F-4C	11/26/2007	100	20.0	2.983
Aged-G-4C	11/26/2007	90	18.8	3.009
Aged-H-4C	11/26/2007	100	14.7	2.824
Aged-I-4C	11/26/2007	100	17.4	3.226
Aged-J-4C	11/26/2007	80	8.2	2.783
Lab Control	-	80	21.5	2.672
Aged-F-4C	11/27/2007	70	10.5	3.005
Aged-G-4C	11/27/2007	100	17.7	2.864
Aged-H-4C	11/27/2007	90	12.7	2.874
Aged-I-4C	11/27/2007	100	17.5	2.829
Aged-J-4C	11/27/2007	70	5.8	3.059

Table 9-6 Results of *C. dubia* 7-day chronic and *S. capricornutum* 4-day toxicity tests evaluating the toxicity of water samples filtered through experimental asphalt and concrete at 20° C. Shaded cells indicate significantly lower survival of *C. dubia* or lower *S. capricornutum* cell count compared to controls ($p<0.05$); * Toxicity possibly due to contaminated equipment or sampling bottles.

		C. dubia		**Algae**
Sample	**Date**	**Survival**	**Mean Offspring**	**Cell Count (x10^{6})**
Lab Control	-	100	24.4	2.520
Fresh-A-20	7/30/2007	100	28.7	2.188
Fresh-B-20	7/30/2007	100	24.5	2.182
Lab Control	-	100	20.2	3.226
Fresh-C-20*	6/27/2007	0	0	0.022
Fresh-D-20	6/27/2007	100	20.3	3.109
Fresh-E-20	6/27/2007	100	16.3	2.776
Lab Control	-	100	21.9	2.563
Fresh-F-20-1	5/18/2007	100	16.9	2.272
Fresh-F-20-2	5/18/2007	80	15.0	2.445
Fresh-F-20-3	5/18/2007	100	17.9	2.143
Lab Control	-	90	18.2	3.028
Fresh-F-20*	7/11/2007	0	0	0.038
Fresh-G-20	7/11/2007	100	11.8	0.081
Fresh-H-20*	7/11/2007	0	0.0	0.024
Lab Control	-	90	21.9	4.370
Fresh-I-20	10/9/2007	100	15.6	4.142
Fresh-J-20	10/9/2007	0	0.0	0.475
Lab Control	-	100	23.7	4.170
Aged-A-20C	11/19/2007	90	20.3	3.962
Aged-B-20C	11/19/2007	100	24.0	3.749
Aged-C-20C	11/19/2007	80	23.8	3.776
Aged-D-20C	11/19/2007	100	22.6	3.672
Aged-E-20C	11/19/2007	100	23.6	3.597
Lab Control	-	100	25.8	3.964
Aged-F-20C	11/24/2007	100	23.4	3.651
Aged-G-20C	11/24/2007	100	22.1	3.288
Aged-H-20C	11/24/2007	90	18.7	3.427
Aged-I-20C	11/24/2007	100	25.2	3.643
Aged-J-20C	11/24/2007	60	7.4	3.166

Table 9-7 Results of *C. dubia* 7-day chronic and *S. capricornutum* 4-day toxicity tests evaluating the toxicity of water samples filtered through experimental asphalt and concrete at 45° C. Shaded cells indicate significantly lower survival of *C. dubia* or lower *S. capricornutum* cell count compared to controls ($p<0.05$).

		C. dubia		**Algae**
Sample	**Date**	**Survival**	**Mean Offspring**	**Cell Count (x10^6)**
Lab Control	-	100	26	2.990
Fresh-A-45	7/25/2007	90	13.2	2.757
Fresh-B-45	7/25/2007	90	14.4	2.722
Lab Control	-	100	18.3	3.090
Fresh-C-45	7/14/2007	100	11.9	3.114
Fresh-D-45	7/14/2007	60	1.8	2.806
Fresh-E-45	7/14/2007	90	8.4	2.988
Lab Control	-	100	19.5	4.077
Fresh-F-45	7/21/2007	90	14.7	3.409
Fresh-G-45	7/21/2007	80	3.6	3.246
Fresh-H-45	7/21/2007	70	2.8	3.339
Lab Control	-	90	20.7	4.026
I-45 low EC adj.	10/26/2007	100	8.1	3.701
J-45 unadj.	10/26/2007	60	6.5	0.126
Lab Control	-	90	15.5	4.411
Aged-A-45	11/1/2007	90	17.7	3.442
Aged-B-45	11/1/2007	100	18.8	3.696
Aged-C-45	11/1/2007	100	18.6	3.894
Aged-D-45	11/1/2007	70	13.5	3.742
Aged-E-45	11/1/2007	90	17.2	3.364
Lab Control	-	90	20.1	4.031
Aged-F-45	11/8/2007	70	10.5	3.730
Aged-G-45	11/8/2007	100	26.1	4.021
Aged-H-45	11/8/2007	70	0.0	4.014
Aged-I-45	11/8/2007	90	0.0	4.102
Aged-J-45	11/8/2007	70	13.3	0.804

Table 9-8 Water chemistry parameters of water samples filtered through experimental asphalt and concrete at 4° C. Specific conductivity, hardness, alkalinity and ammonia nitrogen were recorded upon sample receipt at UCD-ATL. Temperature, dissolved oxygen, pH and unionized ammonia were recorded at initiation of the *C. dubia* 7-day toxicity test.

Sample	**Date**	**Temp (°C)**	**SC uS/cm**	**DO mg/L**	**pH**	**Hardness (mg/L as CaCO$_3$)**	**Alkalinity (mg/L as CaCO3)**	**Ammonia Nitrogen mg/L**	***C. dubia* Initial Unionized Ammonia**
Fresh-A-4	8/15/07	23.9	3.8	8.6	7.96	4	6	0.01	0.000
Fresh-B-4	8/15/07	24.0	3.3	8.6	7.91	4	-	0.04	0.002
Fresh-C-4-1	6/21/07	23.8	6.0	8.5	8.14	12	6	0.04	0.001
Fresh-C-4-2	6/21/07	24.1	6.6	8.5	8.24	16	34	0.06	0.001
Fresh-C-4-3	6/21/07	23.9	4.7	8.5	8.05	16	8	0.05	0.001
Fresh-D-4-1	6/21/07	23.9	6.1	8.5	8.10	12	8	0.05	0.001
Fresh-D-4-2	6/21/07	24.0	6.2	8.6	8.04	20	8	0.05	0.001
Fresh-D-4-3	6/21/07	23.9	5.6	8.6	8.11	8	10	0.04	0.001
Fresh-E-4-1	6/21/07	24.0	3.9	8.5	8.05	16	4	0.04	0.001
Fresh-E-4-2	6/21/07	24.0	5.9	8.5	8.22	12	8	0.02	0.000
Fresh-E-4-3	6/21/07	24.0	4.8	8.4	8.18	16	6	0.02	0.001
Fresh-F-4-1	6/23/07	23.8	6.0	8.5	8.06	8	6	0.00	0.000
Fresh-F-4-2	6/23/07	23.8	6.3	8.4	8.07	12	6	0.00	0.000
Fresh-F-4-3	6/23/07	23.8	5.6	8.4	8.06	4	4	0.00	0.000
Fresh-G-4-1	6/23/07	23.8	7.1	8.4	8.06	4	6	0.00	0.000
Fresh-G-4-2	6/23/07	23.8	7.1	8.4	8.05	8	6	0.00	0.000
Fresh-G-4-3	6/23/07	23.7	5.7	8.4	8.04	20	6	0.00	0.000
Fresh-H-4-1	6/23/07	23.7	6.2	8.4	8.03	4	6	0.00	0.000
Fresh-H-4-2	6/23/07	23.7	4.8	8.5	8.19	8	6	0.00	0.000
Fresh-H-4-3	6/23/07	23.7	6.3	8.4	8.08	12	6	0.00	0.000
Fresh-I-4	10/5/07	24.2	65.1	8.6	8.90	20	26	0.04	0.008
Fresh-J-4	10/5/07	23.5	368.8	8.6	7.03	80	86	0.03	0.000
Aged-F-4	11/26/07	23.6	13.6	8.6	8.53	8	8	0.00	0.000
Aged-G-4	11/26/07	23.7	21	8.5	8.69	8	6	0.00	0.000
Aged-H-4	11/26/07	23.7	31.2	8.6	8.77	8	10	0.01	0.002
Aged-I-4	11/26/07	23.6	34.6	8.6	8.82	12	10	0.02	0.005
Aged-J-4	11/26/07	23.5	115.8	8.6	7.73	40	38	0.01	0.000
Aged-F-4	11/27/07	23.6	7.7	8.6	8.53	2	4	0.03	0.004
Aged-G-4	11/27/07	23.7	16.1	8.5	8.69	6	9	0.03	0.006
Aged-H-4	11/27/07	23.7	17.4	8.6	8.77	8	9	0.03	0.007
Aged-I-4	11/27/07	23.6	13.9	8.6	8.82	6	8	0.03	0.007
Aged-J-4	11/27/07	23.5	11.1	8.6	7.73	4	6	0.02	0.001

Table 9-9 Water chemistry parameters of water samples filtered through experimental asphalt and concrete at 20° C. Specific conductivity, hardness, alkalinity and ammonia nitrogen were recorded upon sample receipt at UCD-ATL. Temperature, dissolved oxygen, pH and unionized ammonia were recorded at initiation of the *C. dubia* 7-day toxicity test.

Sample	**Date Collected**	**Temp (°C)**	**SC uS/cm**	**DO mg/L**	**pH**	**Hardness (mg/L as $CaCO_3$)**	**Alkalinity (mg/L as CaCO3)**	**Ammonia Nitrogen mg/L**	***C. dubia* Initial Unionized Ammonia**
Fresh-A-20	7/30/07	24.0	33.4	7.9	8.08	8	8	0.00	0.000
Fresh-B-20	7/30/07	24.0	13.1	8.2	8.06	8	8	0.00	0.000
Fresh-C-20	6/27/07	24.0	8.7	8.6	8.70	32	12	0.04	0.008
Fresh-D-20	6/27/07	23.8	9.1	8.3	8.80	32	8	0.03	0.007
Fresh-E-20	6/27/07	23.8	6.6	8.6	8.84	16	8	0.01	0.003
Fresh-F-20-1	5/18/07	25.2	36.6	8.4	8.38	2	8	0.00	0.000
Fresh-F-20-2	5/18/07	25.0	24.0	8.5	8.46	2	5	1.84	0.313
Fresh-F-20-3	5/18/07	25.0	29.1	8.1	8.32	4	8	0.00	0.000
Fresh-F-20	7/11/07	23.7	7.2	8.1	8.08	2	4	0.02	0.000
Fresh-G-20	7/11/07	23.5	6.6	8.0	7.94	2	7	0.02	0.051
Fresh-H-20	7/11/07	23.2	6.9	8.0	7.94	2	7	0.01	0.000
Fresh-I-20	10/9/07	24.0	54.8	8.5	8.80	20	131	0.02	0.005
Fresh-J-20	10/9/07	24.0	405.6	8.5	7.63	130	75	0.02	0.000
Aged-A-20	11/19/07	24.1	8.9	7.4	8.11	4	6	0.00	0.000
Aged-B-20	11/19/07	24.0	18.9	7.6	8.24	10	10	0.01	0.000
Aged-C-20	11/19/07	24.1	29.7	7.6	8.36	12	18	0.00	0.001
Aged-D-20	11/19/07	23.9	29.6	7.3	8.34	14	16	0.00	0.001
Aged-E-20	11/19/07	24.1	21.6	7.6	8.4	8	12	0.00	0.004
Aged-F-20	11/24/07	24.4	20.7	8.5	8.80	4	6	0.00	0.000
Aged-G-20	11/24/07	24.5	27.3	8.6	8.79	4	6	0.00	0.000
Aged-H-20	11/24/07	23.9	23.1	8.6	8.78	4	6	0.00	0.000
Aged-I-20	11/24/07	23.9	50.1	8.6	8.88	8	18	0.00	0.000
Aged-J-20	11/24/07	23.9	282.1	8.6	8.84	64	60	0.00	0.000

Table 9-10 Water chemistry parameters of water samples filtered through experimental asphalt and concrete at 45° C. Specific conductivity, hardness, alkalinity and ammonia nitrogen were recorded upon sample receipt at UCD-ATL. Temperature, dissolved oxygen, pH and unionized ammonia were recorded at initiation of the *C. dubia* 7-day toxicity test.

Sample	Date	Temp (°C)	SC uS/cm	DO mg/L	pH	Hardness (mg/L as $CaCO_3$)	Alkalinity (mg/L as CaCO3)	Ammonia Nitrogen mg/L	*C. dubia* Initial Unionized Ammonia
Fresh-A-45	7/25/07	24.0	11.1	7.4	8.14	4	6	0.03	0.000
Fresh-B-45	7/25/07	24.0	10.1	7.5	8.12	2	6	0.02	0.000
Fresh-C-45	7/14/07	25.3	10.6	7.7	7.30	4	10	0.01	0.000
Fresh-D-45	7/14/07	25.2	10.6	7.7	7.33	4	6	0.02	0.000
Fresh-E-45	7/14/07	25.3	13.8	7.7	7.39	4	8	0.00	0.000
Fresh-F-45	7/21/07	23.9	7.5	7.6	8.16	4	6	0.00	0.000
Fresh-G-45	7/21/07	24.0	5.7	7.4	8.20	4	6	0.00	0.000
Fresh-H-45	7/21/07	24.1	6.9	7.6	8.20	4	2	0.00	0.000
I-45 low EC adj.	10/26/07	24.2	64.8	8.6	7.54	28	30	0.00	0.000
J-45 unadj.	10/26/07	24.3	620	7.8	6.45	92	98	0.05	0.000
Aged-A-45	11/1/07	24.7	34.2	8.3	8.27	14	20	0.01	0.001
Aged-B-45	11/1/07	25.1	32.6	8.1	8.3	18	20	0.02	0.002
Aged-C-45	11/1/07	25.0	28.0	8.2	8.38	12	19	0.00	0.000
Aged-D-45	11/1/07	24.9	17.7	8.2	8.09	12	15	0.00	0.000
Aged-E-45	11/1/07	24.9	30.1	8.2	8.33	12	20	0.01	0.001
Aged-F-45	11/8/07	24.6	30.8	7.8	8.03	16	6	0.00	0.000
Aged-G-45	11/8/07	24.8	41.1	7.8	8.54	24	24	0.00	0.000
Aged-H-45	11/8/07	24.1	24.2	8.3	8.28	16	20	0.01	0.001
Aged-I-45	11/8/07	24.0	58.4	8.3	8.99	32	24	0.01	0.003
Aged-J-45	11/8/07	23.6	329.9	8.5	7.52	44	48	0.04	0.001

9.3 Toxicity Data Reliability Quality Assurance/Quality Control

Quality Assurance/Quality Control (QA/QC) measures were included in this project to assess the reliability of the data collected. The components of these QA/QC measures are outlined below.

Reference toxicant tests: Reference toxicant (RT) tests with *C. dubia* and *S. capricornutum* using NaCl and $ZnCl_2$, respectively, as the toxicant were performed once a month to ascertain whether organism response fell within the acceptable range as dictated by US EPA. Each RT test consists of a dilution series made up of five different concentrations of the toxicant and a control. A 20-month running mean control chart is continuously updated with the results of these RT test endpoints. Acceptable range for US EPA is within the 95% confidence interval of a running mean. All *C. dubia* and *S. capricornutum* performed normally within each reference toxicant test from May 1 to November 30, 2007.

QA/QC Samples: Bottle blanks samples were included to evaluate potential incidental contamination due to the sample container. Bottle blanks are analyte-free water samples that were transferred to a clean sample container that wa prepared in the laboratory. For this project, bottle blank samples were comprised of de-ionized water amended with dry salts to US EPA moderately hard specifications for *C. dubia*, and glass distilled water for *S. capricornutum,* which mimicked the composition of samples tested for this project. Bottle blank samples were in agreement when they are both either statistically similar or statistically different from the control. Results of bottle blank tests and the frequency of bottle blanks sharing equivalent results are presented in Tables 9-11 and 9-12.

Table 9-11 Results of bottle blank tests performed to evaluate the effects of using new 100 series unwashed, unrinsed 4 liter amber glass bottles for sample collection and storage on toxicity test results. Shaded cells indicate significantly lower survival of *C. dubia* or lower *S. capricornutum* cell count compared to controls ($p<0.05$).

		Ceriodaphnia dubia		***S. capricornutum* Algae**
Sample	**Date Collected**	**Survival**	**Mean Offspring**	**Cell Count (x10^6)**
Lab Control	-	100	22.5	2.865
Bottle Blank 1	9/2/2007	100	15.7	3.169
Bottle Blank 2	9/10/2007	90	17	-
Lab Control	-	-	-	3.238
Bottle Blank 3	10/2/2007	-	-	3.583

Table 9-12 Bottle blanks sharing equivalent results

Quality Assurance Samples	***C. dubia* Survival**		***C. dubia* Reproduction**		***Algae* cell growth**	
	Sample Size	**% Agreement**	**Sample Size**	**% Agreement**	**Sample Size**	**% Agreement**
Bottle Blanks	2	100	2	0	2	100

There were three instances during the course of this project where anomalous toxicity was observed with *C. dubia* and *S. capricornutum* in samples submitted for testing: Samples T-20-C-20, collected June 27, 2007 and T-20-F-20, T-20-H-20, collected July 11, 2007 exhibited 100% mortality in *C. dubia* assays and extremely reduced cell growth in *S. capricornutum* assays. Water samples from the same asphalt experiment but performed under different temperature conditions (4°C) were submitted for testing earlier in the project, and exhibited no *C. dubia* toxicity and low algal toxicity. This discrepancy in results prompted UCD ATL to question the cleanliness of sampling bottles. Investigations determined that incorrect sampling bottles that were not pre-cleaned were provided. Bottle blank tests were initiated to determine the effect, if any, that un-cleaned and un-rinsed sample bottles had on toxicity testing endpoints. Survival of *C. dubia* and *S. capricornutum* growth were unaffected by bottle blank samples, while *C. dubia* reproduction was impaired by approx. 25% in the bottle blank samples (Table 10). However, the observed impairment was minimal compared to the 100% mortality observed in the suspect experimental runoff samples. It is unlikely that reproductive impairment observed in the bottle blank tests was enough to account for the high mortality exhibited in the initial screening toxicity test, as average organism survival in the bottle blank tests was 95%. In addition, other experimental samples collected using the un-cleaned bottles (D-20, E-20, G-20) caused little or no toxicity to the test organisms. It was determined that the incorrect sampling bottles were unlikely to cause the observed toxicity, and at this point, the toxicity cannot readily be explained.

Deviations: Several deviations occurred during this project with regards to test initiation holding time. US EPA requires samples to be initiated in a test within 36 hours of sample collection. UCD ATL attempts to meet this criterion whenever possible. 43% of the samples submitted for testing missed the sample holding time criterion. The most frequent reasons are as follows:

1) Miscommunication between samplers and lab staff: 56.25%
2) Late sample delivery: 31.25%
3) Lack of *C. dubia* neonates available for test initiation: 12.50%

It is unlikely that the extended holding time negatively impacted the samples, as they were stored in the dark in temperature-controlled environmental chambers maintained between 0-6 ºC until their use in a test. Sample degradation was kept at a minimum. The data from these tests are considered reliable.

In addition, ammonia-nitrogen measurements were taken past the 24-hour holding time and were instead measured within 48-hours of sample receipt on samples collected on June 23, 2007. This deviation occurred due to technician error. It is not anticipated that the late ammonia reading had a negative effect on the test, as sample ammonia measurements were low. The data from these tests are considered reliable.

Chapter 10: References

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