Water Resources
Environmental Quality

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Through: Cyndi Karoly, Water Sciences Section Chief CBK

From: Brian Wrenn, Ecosystems Branch Supervisor BLW
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RE: DWR results from a 2-year study on 1,4-dioxane in the Cape Fear River Basin

Attached please find the final report for second year of the Division of Water Resources' study on 1,4-dioxane in the Cape Fear River basin. Data contained in this report were collected from October 2014 through October 2016. Please contact Tammy Hill (919-743-8412 or tammy.l.hill@ncdenr.gov) with any questions.

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1,4-Dioxane Monitoring in the Cape Fear River Basin of North Carolina

An Ongoing Screening, Source Identification, and Abatement Verification Study

Nothing Compares

Department of Environmental Quality

Prepared by:
The North Carolina Department of Environmental Quality
Division of Water Resources
Water Sciences Section

For more information on the 1,4-dioxane study and electronic copies of this publication:
http://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page

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North Carolina Department of Environmental Quality
Division of Water Resources
Water Sciences Section Mission Statement:

The mission of the Water Sciences Section is to provide the Division with accurate information pertaining to waters of the state. Excellent service along with water quality monitoring and certification programs and analytical laboratory analyses that provide scientifically defensible data are the section's main avenues for accomplishing this mission. These activities support the management and protection of North Carolina's water resources for the health and welfare of the citizens of North Carolina and the economic well-being of the state.

Abstract

1,4-dioxane is an emerging contaminant of concern that was recently monitored in drinking water throughout the United States as part of the U.S. Environmental Protection Agency's Third Unregulated Contaminant Monitoring Rule. Results indicated the presence of 1,4-dioxane in three North Carolina river basins, with the highest concentrations measured in the Cape Fear River basin in central North Carolina.

In October 2016, the North Carolina Division of Water Resources completed the second year of a study designed to examine ambient concentrations, identify potential sources, and document water quality improvements due to abatement efforts of 1,4-dioxane in major surface waters of the Cape Fear River basin. Four primary areas of elevated 1,4-dioxane were identified in the upper portion of the basin. Three of these areas were located immediately downstream of domestic wastewater treatment facilities, indicating that these facilities were likely conduits for 1,4-dioxane from industrial sources into surface water. The fourth was located further downstream of a wastewater facility, as well as in proximity to potential legacy sources of the contaminant.

These findings were communicated to effected municipalities, project partners, and the interested public. During the second year of the study, quarterly sampling indicated a reduction of 1,4-dioxane concentrations in many areas of the basin. Further reductions will be necessary to achieve federal and state health advisory levels for 1,4-dioxane in drinking water supplies. The Division of Water Resources study will continue in the Cape Fear River basin and expand into the Yadkin and Neuse River basins during 2017-2018.

Keywords

1,4-Dioxane; Dioxane; Drinking water; Surface water; Wastewater; Cape Fear River basin; Unregulated Contaminant Monitoring Rule; UCMR3; Emerging contaminants; Water quality; Water resources

Introduction

1,4-Dioxane ($C_4H_8O_2$, CAS # 123-91-1) is a clear liquid that is highly miscible in water (ATSDR, 2012; Mohr, 2010). It has historically been used as a solvent stabilizer, and is currently used for a wide variety of industrial and manufacturing purposes. The compound can be found in industrial solvents, paint strippers, and varnishes and is often produced as a by-product of chemical processes to manufacture soaps, plastics, and other consumer products (Stepien, *et al.*, 2014; U.S. EPA, 2015; Water Research Foundation, 2014).

The United States Environmental Protection Agency (U.S. EPA) does not have an established maximum contaminant level for 1,4-dioxane in drinking water. However, the U.S. EPA has characterized 1,4-dioxane as "likely to be carcinogenic to humans" and has established a drinking water health advisory with an associated estimated lifetime cancer risk of one in one million at a concentration of 0.35 μg/L (U.S. EPA, 2012). North Carolina has a calculated human health criterion for 1,4-dioxane of 0.35 μg/L in water supplies and 80 μg/L in all other waterbodies (15A NCAC 02B.0208).

Due to its physiochemical properties, 1,4-dioxane has a high mobility and is interminable in the environment. As a water quality contaminant, its persistence is due to an "indefinite solubility in water" (Stepien, *et al.*, 2014). Conventional drinking water treatment mechanisms have been found to be ineffective at removing 1,4-dioxane from source water. However, it can be removed via advanced oxidation processes applying a combination of hydrogen peroxide and other factors (Stepien, *et al.*, 2014; Water Research Foundation, 2014).

Under the 1996 Safe Drinking Water Act, the U.S. EPA requires public water systems to monitor a list of up to 30 unregulated contaminants in finished drinking water every five years. The data collected from these monitoring exercises are one of the primary sources of occurrence and exposure data that the U.S. EPA uses to determine regulations on these contaminants (UCMR3, 2012). The Third Unregulated Contaminant Monitoring Rule (UCMR3) included 1,4-dioxane in the list of over 20 chemicals to be sampled and evaluated, using specific analytical methods, during January 2013 – December 2015 at all water utility systems serving more than 10,000 people. These chemicals, as well as two viruses, were also collected at a representative sample of smaller public water systems (U.S. EPA, 2016).

The surface waters of the Cape Fear River basin supply drinking water to many counties in North Carolina (NC). According to UCMR3 data, this basin also exhibits some of the highest measured concentrations of 1,4-dioxane in finished drinking water in NC and the U.S. Therefore, it was the initial focus of a 1,4-dioxane monitoring study by the North Carolina Division of Water Resources NC DWR) with three successive objectives:

1. Screen for ambient 1,4-dioxane concentrations in surface waters of the basin during all seasons to identify areas with consistently elevated results,
2. Identify sources and provide information to appropriate NC DWR staff, municipalities and other interested parties,
3. Document changes in concentration in response to municipal and industrial actions to reduce 1,4-dioxane discharges.

At the time of this report, the first objective has been completed, while the latter two have become part of an ongoing, iterative process that will continue into the third year of monitoring in 2017 as 1,4-dioxane abatement efforts progress.

Methods

Timeframe and Study Area

From October 2014 – October 2016, the study progressed through three phases, aligned with the study objectives. The initial screening study included monthly sampling during Year 1 (October 2014 – September 2015) at twelve stations throughout the Cape Fear River basin to capture seasonal and spatial variability in constituent concentrations. By May 2015, sufficient data were available to recognize distinct areas where 1,4-dioxane concentrations were elevated, and seven monitoring stations were added to the monthly monitoring schedule from June – September 2015 to assist with source identification. In September 2015, the first full year of sampling was completed. Monitoring locations were adjusted and sampling frequency was reduced to quarterly for Year 2 of the study (13 months from October 2015 – October 2016; station information and location details in Appendix A) to continue to document the spatial distribution of 1,4-dioxane, as well as surface water quality changes resulting from 1,4-dioxane source remediation and abatement efforts in the basin.

Field Sampling and Procedure

Sampling was conducted according to methods described in North Carolina's Ambient Monitoring System (AMS) Quality Assurance Project Plan (QAPP) (NCDENR, 2014, Section B.2 and Appendices 7 and 8) and instructions provided by the contract laboratory for 1,4-dioxane sample collection and preservation. All samples were collected as near-surface (i.e. 0.1-meter depth) grab samples.

One sample was collected monthly or quarterly, depending on the study phase, for the analysis of 1,4-dioxane at each site. Quality assurance (QA) samples, including duplicates, matrix spikes, and matrix spike duplicates were collected quarterly at each of the stations on a rotating basis (two to four stations per quarter), in accordance with the AMS QAPP (NCDENR, 2014).

During each sampling event, a multi-parameter meter (e.g. YSI Pro Plus with Quatro cable or similar) was used *in situ* to measure instantaneous water temperature, pH, specific conductance, and dissolved oxygen values.

Laboratory Procedure and Quality Control

Due to the absence of a U.S. EPA-approved analytical method for 1,4-dioxane in surface water or wastewater, solid waste analysis methods, rather than finished drinking water methods, were chosen to account for matrix interferences that may exist in stream samples. During the first year of the study, method SW-846 8270 SIM (selected ion monitoring) with a practical quantitation limit (PQL) of 3 µg/L was used to evaluate 1,4-dioxane. In December 2015, the analytical method was changed to SW-846 8260B to support smaller sample volumes and a lower PQL of 2 µg/L. QA samples were analyzed with both methods to confirm that the change did not substantially alter results.

As mentioned above, QA samples included duplicates, matrix spikes, and matrix spike duplicates. Duplicates were analyzed to evaluate reproducibility of results. Matrix spikes were analyzed to evaluate surface waters receiving different types of inputs, including domestic wastewater effluent, dam release, urban stormwater, and swamp waters, in which the potential for various types of interferences existed. Matrix spike duplicates were analyzed to evaluate reproducibility of spiked samples.

Data Analysis

Data analysis was performed on NC DWR sample results to determine ambient concentrations of 1,4-dioxane in surface waters throughout the Cape Fear River basin, to compare results with calculated evaluation levels, and to identify areas that may be contributing to the exceedance of evaluation levels throughout the basin.

Concentration results were reviewed monthly, then summarized on an annual basis to determine the mean, median, maximum, and minimum concentrations. Monthly and summary results were plotted on an ArcGIS Online map (http://arcg.is/1dJa1Nq) to identify potential areas of concern. The map was used to examine areas of high concentrations as well as possible sources of 1,4-dioxane, including:

- Domestic and industrial point-source discharges,
- Active and inactive hazardous waste facilities,
- Active, inactive, and pre-regulatory landfills,
- Known 1,4-dioxane contaminated groundwater plumes,
- Wastewater outfalls from groundwater remediation sites,
- Permitted non-discharge facilities, and
- Airports.

Additionally, where available, stream flow data from United States Geological Society (USGS) gages at or near sampling stations were downloaded as an explanatory variable. Surface discharge appears to be inversely related to 1,4-dioxane concentration in some cases.

Results

During the first year of the study, four areas of high 1,4-dioxane concentration were identified. Three of the four areas were located immediately downstream of domestic wastewater treatment facilities (WWTF), and one was further downstream of a WWTF, as well as downstream of an inactive textile manufacturing site and other possible legacy sources. In Year 1, these areas had maximum concentrations ranging from 171 to 1030 μg/L and mean concentrations from 43 to 351 μg/L. During Year 2, the same areas returned maximum values of 20 to 614 μg/L and means of 11 to 260 μg/L.

Results in excess of the calculated criteria have been documented throughout the Haw, Deep, and Cape Fear Rivers, with the highest 1,4-dioxane values during both study years from the upper watersheds of the Haw and Deep Rivers. Decreases in mean 1,4-dioxane concentration from Year 1 to Year 2 of the study were observed at all monitoring stations. During the second year of the study, the four Cape Fear River stations below Jordan Lake routinely returned results at or below the laboratory PQL of 2 μg/L. Year 1 results for these stations averaged 4 to 6 μg/L.

Summarized results for the twelve stations monitored during both study years are provided in Appendix B. Results for stations monitored only during Year 1 can be found in the initial study report (NC DWR, 2016).

Discussion

As an emerging contaminant of concern, municipal water and wastewater treatment facilities are generally not equipped to remove 1,4-dioxane through their treatment processes. Due to the high aqueous solubility and resistance of 1,4-dioxane to biodegradation, conventional treatment processes are generally ineffective at removal (Zenker, *et al*., 2003). Installation and operation of advanced treatment processes, such as those using hydrogen peroxide, ozone, and/or ultraviolet photo-oxidation, shown to be effective for 1,4-dioxane removal at WWTFs or drinking water systems are anticipated to be prohibitively expensive for local governments and the citizens served by public utilities (*Ibid*). Therefore, the most prudent approaches to reducing 1,4-dioxane concentrations in surface water and drinking water are likely to be reduction, elimination, and/or capture and treatment at industrial sources using or generating the compound.

It has been found that certain industrial processes are more likely to utilize 1,4-dioxane or to create it as a by-product, such as esterification and subsequent polycondensation used to create polyethylene terephthalate (PET) plastics (Popoola, 1991) or the synthesis of those plastics in the manufacturing of polyesters (Zenker, *et al.*, 2003). Therefore, WWTFs with such industries discharging to their collection system may expect to see greater loading of this contaminant in both their influent and effluent streams.

During the first year of the study, the highest measured concentrations were observed below WWTFs. 1,4-dioxane is not used in or created by wastewater treatment processes, suggesting that the most significant contributions were constituents of industrial waste streams that were passing through WWTF treatment processes with varying levels of removal efficiency prior to entering surface waters. These findings were communicated to the affected municipalities to support their pretreatment programs.

The second study year included quarterly sampling which yielded a sample size too small to draw definitive conclusions. Measured 1,4-dioxane concentrations generally decreased throughout the basin, suggesting potential improvement in water quality which may have been due to actions taken by the municipalities (and/or their contributing industries) whose WWTFs were receiving industrial effluent containing 1,4-dioxane. NC DWR's ongoing monitoring study will provide more data to determine actual water quality improvements, inform 1,4-dioxane reduction efforts, and document surface water outcomes over time.

Management Implications and Future Actions

Successful abatement of 1,4-dioxane in Cape Fear River basin drinking water sources will require continued partnerships between the Division, project partners, municipal utility departments, and industries within the basin. Further research into options for replacement of 1,4-dioxane-containing compounds in industrial processes, as well as capture and treatment technologies that could be implemented in industrial and domestic wastewater treatment systems, is warranted. Such research has already begun locally through studies funded by the National Science Foundation, Water Resources Research Institute, and Urban Water Consortium.

The NC DWR is developing a study plan to continue monitoring 1,4-dioxane concentrations at selected locations within the Cape Fear River basin during 2017-2018 with the objectives of further understanding fluctuations in concentrations, identifying sources, and documenting in-stream responses to source abatement efforts. Also in 2017-2018, the study will be expanded into the Neuse and Yadkin River basins in areas that returned UCMR3 1,4-dioxane results above the calculated human health criterion for water supply waterbodies.

Acknowledgements

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The upcoming expanded study will be funded under a grant from the National Fish and Wildlife Foundation. We thank the Foundation and our Water Sciences Section colleagues who will be contributing to the monitoring efforts, optimizing instrumentation, and developing methods to expand the NC DWR Chemistry laboratory's capacity to analyze 1,4-dioxane in water.

We recognize Dr. Detlef Knappe and his graduate students at the North Carolina State University Department of Civil, Construction, and Environmental Engineering for their assistance with study design, sample collection, and sample analysis.

Finally, we acknowledge and commend the efforts of the Cape Fear River basin municipalities that strive to safeguard and provide high-quality public services to their constituents.

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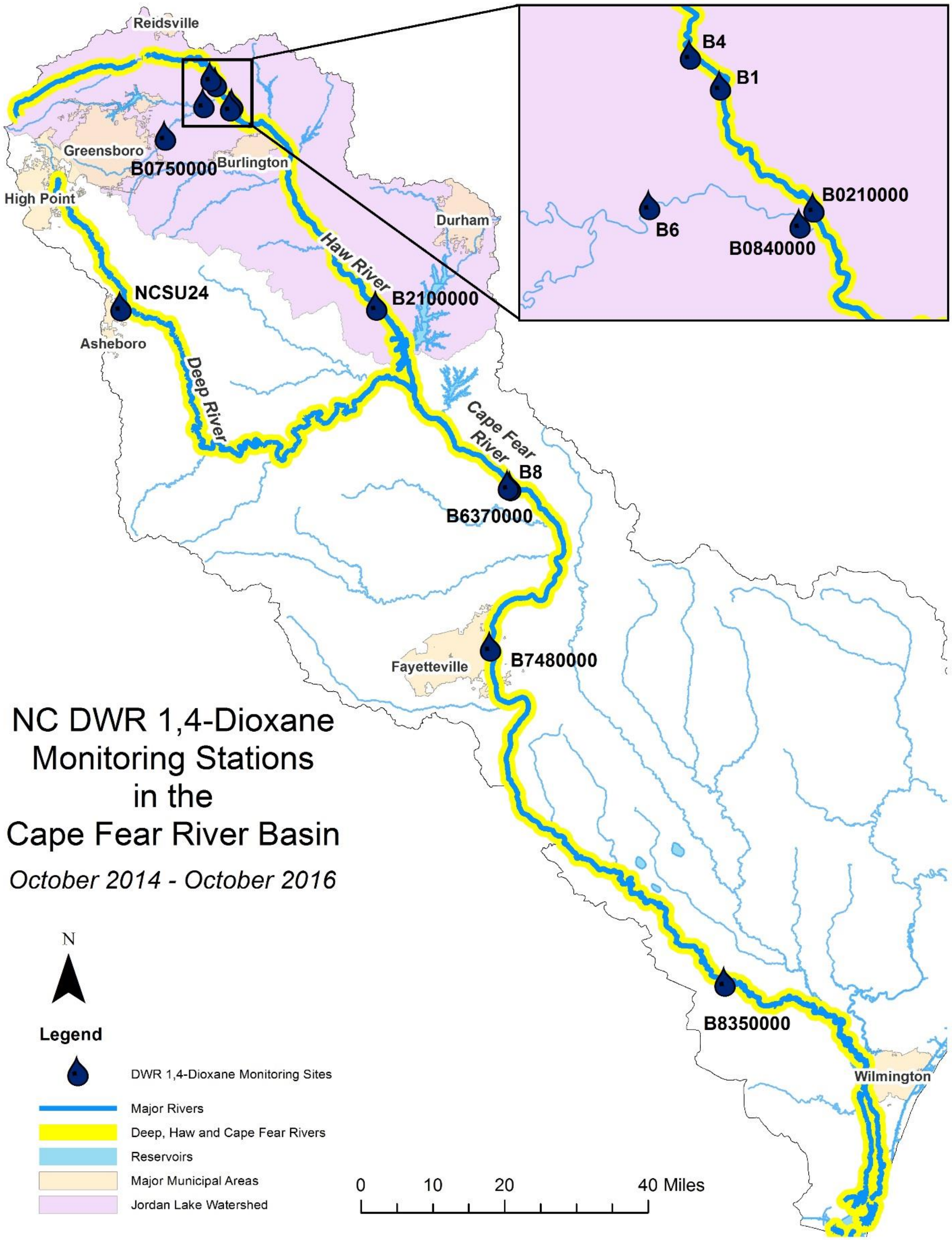
APPENDIX A
NC DWR Monitoring Station Locations

Station	Location	County	Latitude	Longitude	Stream Class[1]	Monitoring Dates
Haw River above Reedy Fork						
B4	Haw River at Troxler Mill Road near Reidsville	Rockingham	36.2329	-79.5588	WS-IV NSW	Jun 2015 - Oct 2016
B1	Haw River at SR 1712 (Brooks Bridge Road)	Guilford	36.2219	-79.5456	WS-V NSW	Oct 2014 - Oct 2016
B0210000	Haw River at SR 1561 Hub Mill Road	Alamance	36.1786	-79.5042	WS-V NSW	Oct 2014 - Oct 2016
Buffalo Creek and Reedy Fork						
B0750000	South Buffalo Creek at SR 2821 at McLeansville	Guilford	36.1128	-79.6718	WS-V NSW	Oct 2014 - Oct 2016
B6	Reedy Fork at NC-61 near Ossipee	Guilford	36.1792	-79.5763	WS-V NSW	Jun 2015 - Oct 2016
B0840000	Reedy Fork at NC-87 at Ossipee	Alamance	36.1730	-79.5103	WS-V NSW	Oct 2014 - Oct 2016
Hasketts Creek						
NCSU24	Haskett Creek at WOW Road near Asheboro	Randolph	35.7681	-79.7790	C	Jun 2015 - Oct 2016
Haw River below Reedy Fork						
B2100000	Haw River at SR 1713 near Bynum (near Pittsboro intake)	Chatham	35.7717	-79.1450	WS-IV NSW	Oct 2014 - Oct 2016
Cape Fear River						
B8	Cape Fear River at Harnett County Public Utilities intake	Harnett	35.4092	-78.8189	WS-IV CA	Oct 2014 - Oct 2016
B6370000	Cape Fear River at US-401 at Lillington	Harnett	35.4065	-78.8135	WS-IV	Oct 2014 - Oct 2016
B7480000	Cape Fear River at Hoffer WTP intake at Fayetteville	Cumberland	35.0825	-78.8638	WS-IV CA	Oct 2014 - Oct 2016
B8350000	Cape Fear River at Lock 1 near Kelly	Bladen	34.4038	-78.2932	WS-IV Sw	Oct 2014 - Oct 2016

[1]NC Stream classifications at study locations included Water Supplies (WS-IV and WS-V) and Critical Areas (CA) near drinking water intakes, Nutrient Sensitive Waters (NSW), waters protected for aquatic life and secondary recreation (C) and Swamp waters (Sw).

APPENDIX A
NC DWR Monitoring Station Locations



APPENDIX B
NC DWR 1,4-Dioxane Monitoring Results

Station	Location	Stream Class	EL[1] (µg/L)	Year (n)[2]	1,4-Dioxane Concentration (µg/L)[3] Min	Median	Max	Mean
Haw River above Reedy Fork								
B4	Haw River at Troxler Mill Road near Reidsville	WS-IV NSW	0.35	1 (4)	35	169	1030	351
				2 (6)	<2	4	46	11
B1	Haw River at SR 1712 (Brooks Bridge Road)	WS-V NSW	80	1 (13)	<3	33	149	46
				2 (5)	<2	5	54	14
B0210000	Haw River at SR 1561 Hub Mill Road	WS-V NSW	80	1 (13)	<3	38	100	42
				2 (5)	<2	7	51	14
Buffalo Creek and Reedy Fork								
B0750000	South Buffalo Creek at SR 2821 Harvest Road at McLeansville	WS-V NSW	80	1 (15)	9	25	543	80
				2 (6)	7	12	86	28
B6	Reedy Fork at NC-61 near Ossipee	WS-V NSW	80	1 (4)	11	20	45	24
				2 (5)	9	9	62	20
B0840000	Reedy Fork at NC-87 at Ossipee	WS-V NSW	80	1 (13)	7	45	100	43
				2 (5)	7	10	20	12
Hasketts Creek								
NCSU24	Hasketts Creek at W.O.W. Road	C	80	1 (4)	147	269	478	291
				2 (5)	69	169	614	260
Haw River below Reedy Fork								
B2100000	Haw River at SR 1713 near Bynum (near Pittsboro intake)	WS-IV NSW	0.35	1 (13)	<3	13	66	18
				2 (5)	<2	8	14	8
Cape Fear River								
B8	Cape Fear River at Harnett County Public Utilities intake	WS-IV CA	0.35	1 (13)	<3	5	15	6
				2 (5)	<2	<2	<2	<2
B6370000	Cape Fear River at US-401 at Lillington	WS-IV	0.35	1 (15)	<3	4	15	6
				2 (5)	<2	2	3	2
B7480000	Cape Fear River at Hoffer WTP intake at Fayetteville	WS-IV CA	0.35	1 (12)	<3	3	11	4
				2 (5)	<2	<2	<3	<2
B8350000	Cape Fear River at Lock 1 near Kelly	WS-IV Sw	0.35	1 (12)	<3	3	11	4
				2 (5)	<2	2	3	2

Notes:

[1] The evaluation level (EL) for 1,4-dioxane is based on stream classification. The water supply EL is 0.35 µg/L, except WS-V in the Jordan Lake watershed. The EL for Jordan Lake WS-V and all other waters is 80 µg/L.

[2] The Year 1 summary includes monitoring results from October 2014-September 2015. The Year 2 summary includes monitoring results from October 2015-October 2016. "n" is the number of results during the study year.

[3] Median and mean values may be inflated for stations with results below the contract lab PQL of 3 µg/L (in 2014-2015) and 2 µg/L (in 2016). For summary calculations, non-detect results are set equal to the PQL in place at the time of analysis. When a minimum or all values were below the PQL, a "<" sign is used.

APPENDIX B
NC DWR 1,4-Dioxane Monitoring Results