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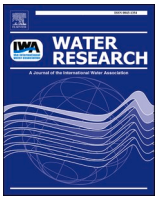

Natural vs. anthropogenic sources of *N*-Nitrosodimethylamine precursors in surface water

Mingrui Song [a,b], Junli Wang [a], Michael DeNicola [a], David Hanigan [a,*]

[a] Department of Civil and Environmental Engineering, University of Nevada, Reno, NV, 89557-0258, USA
[b] Department of Chemical and Environmental Engineering, University of California, Riverside, CA, 92521, USA

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ABSTRACT

N-nitrosodimethylamine (NDMA) is a carcinogenic disinfection byproduct formed from reactions between dichloramine and organic nitrogen-containing precursors. It is unclear if NDMA precursors in surface water intakes originate in anthropogenic (i.e., wastewater) or natural sources. The Truckee River has a single point source release of treated wastewater effluent, making it an ideal system to study the relative importance of precursor sources. Three Lagrangian sampling events were conducted. NDMA formation potential (FP, a measurement of precursors) above the wastewater outfall indicated that the natural background of NDMA precursors was 2-28 ng/L. NDMA FP increased to 18-31 ng/L immediately downstream of the wastewater outfall, but decreased rapidly in a first order manner, and were not statistically different from the upstream samples in only ~6 km. This suggests that the dominant source of NDMA precursors may be wastewater derived only near wastewater outfalls and deviates from the previous belief that wastewater-derived precursors are responsible for NDMA formation in drinking water sources located further downstream. Additionally, given the rapid loss of the wastewater precursors in this study, precursors which are slow to biodegrade/photolyze/adsorb to sediment are likely to be poor surrogates for the overall wastewater NDMA precursor pool. To understand temporal changes in the wastewater impact on environmental NDMA precursor loading, two 24-hour sampling events were conducted near (<3 km) the wastewater outfall and demonstrated that temporal changes in the NDMA precursors directly downstream of the wastewater outfall are directly linked to the wastewater flow contribution.

1. Introduction

N-nitrosodimethylamine (NDMA) is a disinfection byproduct which forms via reactions between dichloramine and precursors containing organic nitrogen (Huang et al., 2018; Schreiber and Mitch, 2006a; Zhang et al., 2016). NDMA is a known rodent carcinogen (Peto et al., 1991), and the U.S. Environmental Protection Agency has calculated that a drinking water concentration of 0.7 ng/L results in a 10^{-6} lifetime excess cancer risk (US EPA, 2002). Several U.S. states and countries have set NDMA drinking water guidelines to mitigate this risk (Canada, 2011; COEHHA, 2006; Council, 2011; Organization, 2008; Protection, 2020). For example, California has a public health goal of 3 ng/L for NDMA (COEHHA, 2006) and Canada has currently set the maximum acceptable concentration in drinking water of 40 ng/L (Canada, 2011). NDMA forms slowly, mostly in the distribution system (Krasner et al., 2013a; Zhang et al., 2016), and so mitigation has generally focused on precursor removal rather than removal/destruction of NDMA itself. Thus, understanding and identifying NDMA precursors is critical to control NDMA occurrence in drinking water.

Many anthropogenic nitrogen-containing substances have been identified as NDMA precursors, including pharmaceuticals (Hanigan et al., 2017; Hanigan et al., 2015a; Jasemizad et al., 2020), personal care products (Shen and Andrews, 2011), herbicides (Chen and Young, 2008), pesticides (Padhye et al., 2013), fungicides (Schmidt and Brauch, 2008), amine-based water treatment polymers (An et al., 2019; Hanigan et al., 2015b; Park et al., 2009), anion exchange resins (Flowers and Singer, 2013), and other amines (Mitch and Sedlak, 2002; Song et al., 2022). Although some anthropogenic NDMA precursors have relatively

Abbreviations: NDMA, *N*-nitrosodimethylamine; NDMA FP, NDMA formation potential; WWRF, wastewater reclamation facility; SPE, solid phase extraction; TR, Truckee River; GC-MS/MS, gas chromatography-tandem mass spectrometry; TDN, total dissolved nitrogen; DON, dissolved organic nitrogen; DOC, dissolved organic carbon; BDL, below detection limit.

* Corresponding author.
E-mail address: DHanigan@unr.edu (D. Hanigan).

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high molar yields (>75 %) (Selbes et al., 2013), their occurrence in surface water serving as the influent to drinking water treatment plants is low or unknown (Kolpin et al., 2002), indicating they may only account for a small fraction of NDMA precursor pool. For example, methadone has been reported to have a molar yield ranging from 23 % to 70 % with relatively high occurrence in wastewater, but it was only responsible for as much as 62 % of the NDMA precursor pool in the wastewaters sampled, with most samples being between 1 % and 10 % (Hanigan et al., 2015a). More recently, it was demonstrated that benzyldimethylamine, the biodegradation product of benzalkonium chloride (common antimicrobial), has a molar yield of ~73 % and accounted for an estimated 17 to 38 % of the NDMA precursor pool in wastewater effluent (Abusallout et al., 2024). Benzyldimethylamine is, however, an intermediate in the microbiological degradation of benzalkonium chloride, and it is not clear how long it persists in the environment. Thus, it is not clear how much of the total environmental NDMA precursor loading these individual chemicals contribute.

Without implicating individual compounds and only evaluating the bulk potential to form NDMA upon chloramination, it has been suggested that wastewater effluent is a substantial or even the primary source of NDMA precursors in the environment. In one study, NDMA formation potentials (NDMA FPs, a surrogate for total precursor loading) of various water samples including pristine head water, eutrophic water, agricultural or stormwater runoff, and wastewater effluents were compared to make this assertion, although no specific analysis of effluent flow contribution or transport of the precursors was conducted (Zeng et al., 2016). In another, region-specific relationships between NDMA FP, streamflow, and sucralose (an indicator of wastewater contribution to total flow (Rice and Westerhoff, 2015)) were developed (Prescott et al., 2017) and suggested site-specific correlations between NDMA precursor loadings and sucralose concentrations. However, in some of the watersheds evaluated, other sources of precursors appeared to be more important than wastewater effluent (Li et al., 2024; Zeng et al., 2016). This may be because photolysis, sorption and biodegradation have been demonstrated by others to be effective attenuation, albeit slow, mechanisms for NDMA precursors (Beita-Sandí et al., 2016; Padhye et al., 2009; Qiu et al., 2021; Woods and Dickenson, 2016; Zhang et al., 2020). For example, Beita-Sandí et al. (2016) investigated the photodegradation of wastewater-derived NDMA precursors under sunlight and observed a 20 % degradation after 4 days and 60 % after 7 days. In batch experiments, 32 to 88 % of wastewater-derived NDMA precursors were also reported to be biodegraded after six weeks (Woods and Dickenson, 2016). Another reason may be that naturally occurring substances including natural organic matter, algae, metabolism of amino acids, and other organic matter in agricultural or stormwater contributed substantially to the NDMA precursor pool (Bei et al., 2020; Bei et al., 2016; Chen and Valentine, 2007; Gerecke and Sedlak, 2003; Li et al., 2012; Li et al., 2020; Liu et al., 2022; Sgroi et al., 2018; Zeng et al., 2016). Both natural attenuation and contributions from other organic matter complicate the source attribution of NDMA precursors in surface water.

While it is a common belief among scientists and engineers that surface water NDMA precursors originate in wastewater effluent, the body of evidence supporting this belief is limited. One reason for the limited evidence to support this hypothesis is that nearly all surface water systems have some flow contribution from wastewater effluent, leaving little opportunity to distinguish between wastewater/anthropogenic precursors and natural precursors. However, the Truckee River is the sole outlet of Lake Tahoe (all wastewater is exported from the Tahoe Basin to limit N and P inputs to the ultraoligotrophic lake), and it flows adjacent to the City of Truckee and then through Reno, and terminates in Pyramid Lake. Near Truckee, one wastewater reclamation facility (WWRF) conducts shallow groundwater injection near the Truckee River, and in Reno, one WWRF discharges to the Truckee River. Since the Truckee River has only two inputs of wastewater-derived NDMA precursors (and likely, only one, as riverbank filtration has been shown to substantially degrade NDMA precursors (Krasner et al., 2018)), it is an ideal water system to better understand the relative importance of wastewater-derived NDMA precursors in surface water.

To understand the contributions of wastewater vs. naturally occurring NDMA precursors, we measured NDMA precursors present in the Truckee River, which has few point sources of wastewater input. We first monitored NDMA FP along a ~162 km reach using Lagrangian sampling (collection of the same plug of water), including both upstream and downstream of the sole WWRF outfall and near the shallow wastewater leach field. We then measured the flow normalized diurnal changes in NDMA precursors present in the Truckee River near the WWRF outfall to better understand how changes in effluent loading affect precursor loading over time.

2. Materials and methods

2.1. Chemicals and materials

An EPA 521 nitrosamine mix was purchased from Sigma-Aldrich (St. Louis, MO) and was used as the NDMA standard. The isotopically labeled NDMA standard (NDMA-d6, 98 %) was from Cambridge Isotopes (Tewksbury, MA, USA). HPLC grade methanol and dichloromethane (DCM), sodium hypochlorite (NaOCl, 5.65-6 %), ammonium chloride (NH_4Cl), borax, boric acid, and ascorbic acid were purchased from Fisher Scientific (Waltham, MA, USA). The Dionex seven anion standard was from Thermo Fisher Scientific (Waltham, MA, USA). The total organic carbon (TOC) calibration standard was from NSI Lab Solutions (Raleigh, North Carolina, USA). Milli-Q water with electric resistance of $\geq$ 18.2 MΩ-cm was used as reagent water. EPA 521 activated carbon solid phase extraction (SPE) cartridges (2 g/6 mL) for NDMA analysis were from Restek (Bellefonte, PA, USA). Sodium sulfate drying cartridges were from Agilent Technologies (Santa Clara, CA, USA). Monochloramine and free chlorine were analyzed with indophenol colorimetric Monochlor F and DPD free chlorine reagent, respectively, from Hach (Loveland, CO, USA). Glass microfiber filters (GF/F, 0.45 μm pore size) from Advantec MFS, Inc. (Dublin, CA) were pre-combusted and used for filtering samples.

2.2. Site description and sample collection

Surface water was collected at multiple locations from the Truckee River and Steamboat Creek. No rainfall events occurred in the watershed for at least one week prior to the sampling campaigns. Detailed sampling locations are shown in Fig. 1 and coordinates for all locations are provided in Table S1. Steamboat Creek receives WWRF effluent and discharges into the Truckee River approximately 210 m downstream of the WWRF outfall. Steamboat Creek originates at Washoe Lake, which receives surface water from snowmelt and precipitation, and travels 28 km through urban Reno before receiving the WWRF effluent and terminating in the Truckee River. "WWRF" is used to refer to the WWRF effluent, which is discharged to Steamboat Creek, and "Steamboat Creek" is immediately downstream (~100 m) of the WWRF effluent, before the confluence with the Truckee River. Sampling sites TR 1 to TR 7 are in the Truckee River and upstream of the Steamboat Creek/Truckee River confluence. One additional WWRF was located near TR 2, where the effluent is discharged to a shallow sub-surface leach field, which is likely to be somewhat hydrologically connected to the Truckee River. Locations TR 8 to TR 14 are located downstream of the Steamboat Creek/Truckee River confluence. Across all sampling events, Steamboat Creek downstream of the WWRF outfall was 23 to 71 % reclaimed wastewater by volume based on USGS gage data combined with discharge flow data provided by the treatment facility (USGS, 2020, 2021).

Lagrangian sampling (collection of the same plug of water) was conducted on three occasions (September 2020, October 2021, and June 2022) to investigate natural vs wastewater NDMA precursor loading in

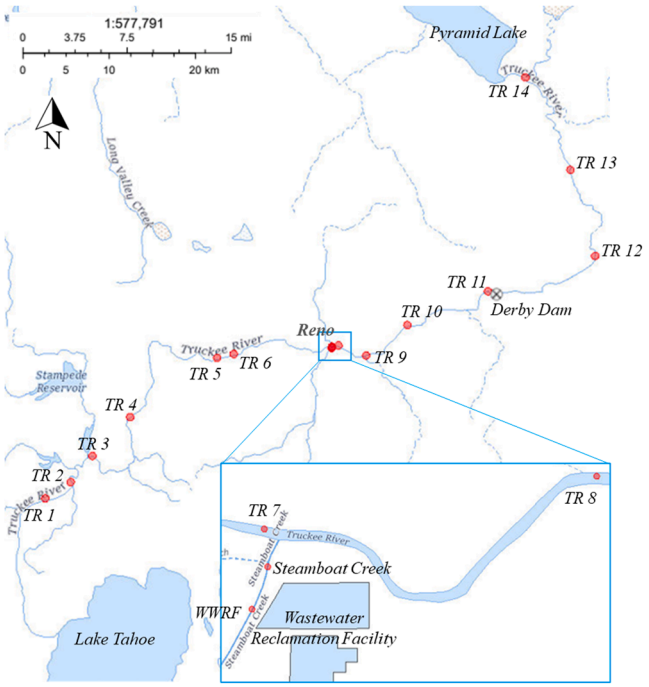

Fig. 1. Map of the study area with sampling sites indicated. Map was generated by USGS National Map (Survey, 2022).

the Truckee River. Flowrates of the Truckee River were again obtained from USGS gage data (USGS, 2020, 2021, 2022) at each time of sample collection and are provided in Table S2. Travel time between sampling sites was calculated based on the Truckee River flowrates and tracer studies conducted by others (Bohman, 2000; Crompton and Bohman, 2000). Sampling times of each event based on travel time are also provided in Table S2. Because of the difficulties of collecting samples at night, not all sampling sites were sampled in each sampling campaign. Specifically, four 2-L samples (TR 1-2, TR 6-7) and six 4-L samples (TR 1-5 and TR7) were collected in September 2020 and October 2021, respectively. In September 2020 and October 2021, follow-on sampling was conducted at the WWRF effluent, Steamboat Creek downstream of the WWRF outfall, and downstream of the Steamboat Creek/Truckee River confluence (i.e., WWRF, Steamboat Creek, and TR 8-9). In an additional sampling campaign conducted in June 2022, samples were collected from TR 9 to 14 to better understand the natural NDMA precursor loading. All samples were collected in pre-combusted borosilicate glass amber bottles. Grab samples from all sampling events were stored on ice and transported to the University of Nevada, Reno laboratories and stored in the dark at 4 °C.

To understand temporal effects from changing WWRF effluent precursor loading, 24-hour continuous sampling was conducted with autosamplers (Teledyne ISCO 6712, St. Lincoln, NE, USA) on two occasions (May and June 2022). In May, one auto-sampler was placed at Steamboat Creek and a total of 24 samples were collected each hour over 24 hr. In June, an additional sampling campaign was conducted with autosamplers placed at the WWRF effluent and TR 8. Sampling times and flowrates during these continuous campaigns are described in Table S3 and S4. Samples taken by the automated samplers were stored in 1-L polypropylene bottles that were provided by the manufacturer. After the 24 hours sampling was concluded, samples were immediately transported on ice to the University of Nevada, Reno, and stored in the dark at 4 °C.

2.3. NDMA formation potential

NDMA precursors were analyzed by NDMA FP tests, which were conducted with 500 mL samples in 1-L glass amber bottles following previously published procedures (Hanigan et al., 2016; Song et al., 2022). Briefly, monochloramine was prepared before each experiment by dropwise addition of NaOCl solution to a rapidly stirred 10 mM borate buffered NH_4Cl (pH 8) solution at a N:Cl_2 molar ratio of 1.2. Monochloramine was then dosed to each borate buffered sample (pH=8) to make the final concentration in the sample 18 mg Cl_2/L. After monochloramine addition, samples were allowed to react in the dark at room temperature for 72 hr. Following the reaction, residual monochloramine was confirmed to be above 4.5 mg Cl_2/L by indophenol colorimetric Monochlor F method and quenched with 5 mL of 0.5 M ascorbic acid. Samples were spiked with 1 mL of 100 μg/L NDMA-d6 and kept in the dark at 4°C before extraction.

2.4. NDMA and water quality parameter analysis

NDMA was measured following U.S. EPA Method 521 with some modifications (Hanigan et al., 2016; Munch and Bassett, 2004). Briefly, NDMA was extracted with a Dionex AutoTrace 280 SPE instrument (Thermo Scientific). EPA 521 cartridges were first conditioned with DCM, methanol, and Milli-Q water and then loaded with 500 mL sample at a rate of 5 mL/min. After loading, cartridges were dried with ultra-high purity nitrogen gas for 30 min and then eluted with 5 mL DCM. The extracts were dried with sodium sulfate drying cartridges and evaporated to 1 mL under ultra-high purity nitrogen gas at 40 °C. The extracts were analyzed by gas chromatography-tandem mass spectrometry (GC-MS/MS, Shimadzu TQ8040, Japan) with a capillary column (Stabilwax-MS, 30 m × 0.25 mm × 0.25 μm). Further details regarding GC-MS/MS conditions are provided in supporting information Text S1. The GC-MS/MS was calibrated using a series of NDMA standards of 1-100 μg/L and NDMA-d6 (100 μg/L) as internal standard to account for losses during SPE. Sample blanks (Milli-Q) were processed to evaluate the contamination during experiments and a quality control sample (10 μg/L) was analyzed every 10-sample injections. The method detection limit was 1 ng/L based on a signal: noise ratio of 5.

Water quality parameters including total dissolved nitrogen (TDN), dissolved organic nitrogen (DON), dissolved organic carbon (DOC), ammonium (NH_4^+-N), chloride (Cl^-), nitrate (NO_3^--N), nitrite (NO_2^--N), and sulfate (SO_4^{2-}) were analyzed. Anions were analyzed by an ion chromatograph (IC, Dionex ICS-6000 SP, Thermo Scientific) with a Dionex Ionpac AS-19 analytical column (2 mm × 250 mm × 4 μm) at 0.25 mL/min flow rate. NH_4^+-N was measured by a spectrophotometric method (EPA Hach Method 10205). A Shimadzu TOC analyzer (TOC-L) was utilized to measure DOC and TDN. DON was obtained by subtracting NH_4^+-N, NO_3^--N, NO_2^--N and from TDN.

2.5. NDMA FP mass balance

A mass balance which assumes water was instantaneously mixed completely across the cross section and that NDMA precursors act conservatively was conducted:

$$NDMA\ FP_{downstream} \times Q_{downstream} = NDMA\ FP_{upstream} \times Q_{upstream} + NDMA\ FP_{tributary} \times Q_{tributary}.$$

where NDMA $FP_{downstream}$ and NDMA $FP_{upstream}$ represent NDMA FPs of two contiguous sampling sites, and NDMA $FP_{tributary}$ represents tributary NDMA FP. $Q_{downstream}$, $Q_{upstream}$, and $Q_{tributary}$ represent flowrates at the downstream site, the upstream site, and a tributary, respectively and are shown in Table S2. Deviations from a complete mass balance indicate losses through sorption, biodegradation, volatilization, and/or photolysis, or poor measurements of flow (e.g., unidentified/ungauged tributaries). The mass balance was only conducted on Steamboat Creek and TR 7-14 because of other unidentified and/or ungauged tributaries to the Truckee River near other sampling points, which presented as changes to flowrates in the Truckee River, but which did not occur on

the section of Steamboat Creek which was of interest and from TR 7-9 (Table S2). A limitation of this approach was that sewer leaks and septic systems were not considered, although outside of the cities of Truckee and Reno, the region is sparsely populated due to topography.

3. Results and Discussion

3.1. Water quality

A summary of water quality parameters for all sampling events is provided in Table 1. The concentrations of TDN, DON, and DOC were from below detection limit (BDL) to 4.3 mg-N/L, BDL to 1.3 mg-N/L, and 0.4 to 10 mg-C/L, respectively. NH_4^+-N, NO_3^-, NO_2^- concentrations were all less than 0.3 mg-N/L, except one sampling event at Steamboat Creek (May 2022) with NH_4^+-N concentrations greater than 1 mg-N/L. Cl^- and SO_4^{2-} concentrations ranged between 3 and 318 mg/L and between 2 and 93 mg/L, respectively. The highest concentrations of these water quality indicators across all sampling campaigns were generally found in the WWRF effluent and just downstream of the WWRF effluent in Steamboat Creek. For all water quality surrogates measured, concentrations were relatively low above the City of Reno (TR 1-7) and increased after receiving WWRF discharge/Steamboat Creek (TR 8). On the days of Lagrangian sampling, WWRF effluent contributed >50 % of Steamboat Creek flow, and 12 to 38 % of Truckee River flow below the Steamboat/Truckee confluence (data shown in Table S2), together indicating that the WWRF plays an important role in Truckee River water quality downstream of the outfall. In select cases when Steamboat Creek flow was low above the WWRF outfall (October 2021, 71 % of Steamboat Creek flow from TMWRF), DOC, Cl^-, and SO_4^{2-} in Steamboat Creek were greater than that of WWRF effluent, suggesting that, at times, the WWRF doesn't deteriorate, and potentially improves, the water quality of Steamboat Creek.

3.2. NDMA precursor loading above the WWRF outfall

To understand the potential for NDMA to contribute to measurements of NDMA precursors (i.e., NDMA FP), samples were collected from all sampling locations in October 2021 and June 2022, and the results are shown in Figure S1. NDMA concentrations ranged from BDL to 8 ng/L with a median concentration of 2 ng/L. This is comparable to prior studies demonstrating low levels of environmental NDMA (Asami et al., 2009; Chen et al., 2009; Huy et al., 2011; Pehlivanoglu-Mantas and Sedlak, 2006; Sanchis et al., 2020). The highest concentration of 8 ng/L was observed in the WWRF effluent, but decreased to BDL ~2.3 km downstream at TR 8, likely due to photolysis (Plumlee and Reinhard, 2007; Sanchis et al., 2020). Generally, in the first two Lagrangian sampling campaigns, NDMA contributed negligibly to measurements of NDMA FP and therefore no background NDMA subtraction was conducted from NDMA FP measurements. However, NDMA was >50 % of the relatively low concentrations of NDMA FP in select samples in the June 2022 sampling campaign and therefore NDMA was subtracted from NDMA FP measurements.

NDMA precursor loading in the Truckee River was investigated by measuring NDMA FP of water samples collected on three occasions (Fig. 2, September 2020, October 2021, and June 2022, recognizing that samples from the June 2022 sampling may have substantial propagated error due to the subtraction of NDMA from NDMA FP). NDMA FPs from all sampling events ranged from 2 to 280 ng/L, with a median of 13 ng/L. Similar NDMA FPs have been observed globally in surface water impacted by wastewater effluents (Huy et al., 2011; Pehlivanoglu--Mantas and Sedlak, 2006; Sanchis et al., 2020). Correlation between NDMA FP and DON was evaluated for sampling sites that had DON greater than 50 % of TDN, where the error in subtractive measurement of DON is minimized (Lee and Westerhoff, 2005) (Figure S2); the correlation was weak (R^2=0.5), but statistically significant ($p<1 \times 10^{-4}$) agreeing with others that while the precursor pool is associated with DON, DON is a poor surrogate (Dotson et al., 2009).

Upstream of the Steamboat/Truckee confluence (TR 1-7, September 2020 and October 2021) NDMA FPs ranged from 2 to 28 ng/L with a median concentration of 11 ng/L. At TR 2, located near the WWRF in Truckee, CA where the effluent is discharged by sub-surface flow via leach fields which are likely to be at least partially hydraulically connected to the Truckee River, NDMA FP was 17 and 2 ng/L in September 2020 and October 2021, respectively, lower than that of the nearby sampling sites on the river and not statistically different from other samples taken in the upriver reach (ANOVA, p>0.05). This suggests the inputs from the WWRF leach field have negligible influence on the NDMA precursor loading of the Truckee River, likely due to NDMA precursor biodegradation and sorption in the subsurface as demonstrated by others (Krasner et al., 2013b; Krasner et al., 2018; Sacher et al., 2008). Because there was no statistical difference between all samples taken upstream of the Truckee/Steamboat confluence, including TR 2, we find that NDMA FPs from 2 to 28 ng/L represent the natural background of precursors in the system. The background NDMA precursors, the precursors from natural sources, might be from natural organic matter, algae, metabolism of amino acids, and organic matter in agricultural or stormwater (Bei et al., 2020; Chen and Valentine, 2007; Lee et al., 2007; Li et al., 2024, 2012, 2020; Liu et al., 2022; Zeng et al., 2016), but the composition remains unclear.

The highest NDMA FPs were found in the WWRF effluent which

Table 1
Summary of water quality measurements for all sampling events including three Lagrangian sampling events and two 24-hour continuous sampling events. A range of concentrations are shown for sampling sites with more than one collection, and a single value is shown for sampling sites with single sample collection.

Site	TDN (mg-N/L)	DON (mg-N/L)	DOC (mg-C/L)	NH_4^+-N (mg-N/L)	NO_3^- (mg-N/L)	NO_2^- (mg-N/L)	Cl^- (mg/L)	SO_4^{2-} (mg/L)
TR 1	0.08-0.11	0.07-0.09	0.4-0.9	BDL-0.01	BDL	BDL-0.02	3-16	2-10
TR 2	0.13-0.20	BDL-0.13	0.6-1.1	BDL	BDL-0.2	BDL	7-24	3-11
TR 3	0.2	0.20	1.3	BDL	BDL	BDL	12	7
TR 4	BDL	BDL	1.3	BDL1	BDL	BDL	11	7
TR 5	0.11	BDL	1.5	BDL	0.11	BDL	13	41
TR 6	0.13	0.13	1.1	BDL	BDL	BDL	4	4
TR 7	0.11-0.14	0.11-0.13	1.2-2.2	BDL-0.01	BDL	BDL	5-21	8-63
WWRF	1.23-1.74	0.88-1.32	6.8-12.3	0.01-0.37	BDL-0.3	BDL-0.02	63-105	49-87
Steamboat Creek	1.12-4.33	BDL-1.12	5.5-9.4	0.06-2.82	BDL-1.2	BDL-0.09	67-318	38-93
TR 8	0.06-0.73	0.06-0.58	1.5-6.1	BDL-0.04	BDL-0.1	BDL-0.03	11-74	15-84
TR 9	0.36-0.87	0.32-0.65	2.4-6.4	0.04-0.07	BDL-0.1	BDL-0.02	23-80	22-89
TR 10	0.17	0.17	5.7	BDL	BDL	BDL	18	20
TR 11	0.21	0.20	6.9	0.01	BDL	BDL	17	19
TR 12	0.16	0.16	4.1	0.01	BDL	BDL	16	19
TR 13	0.15	0.10	3.5	0.05	BDL	BDL	20	23
TR 14	0.15	0.15	5.1	BDL	BDL	BDL	30	25

BDL: Below detection limit.

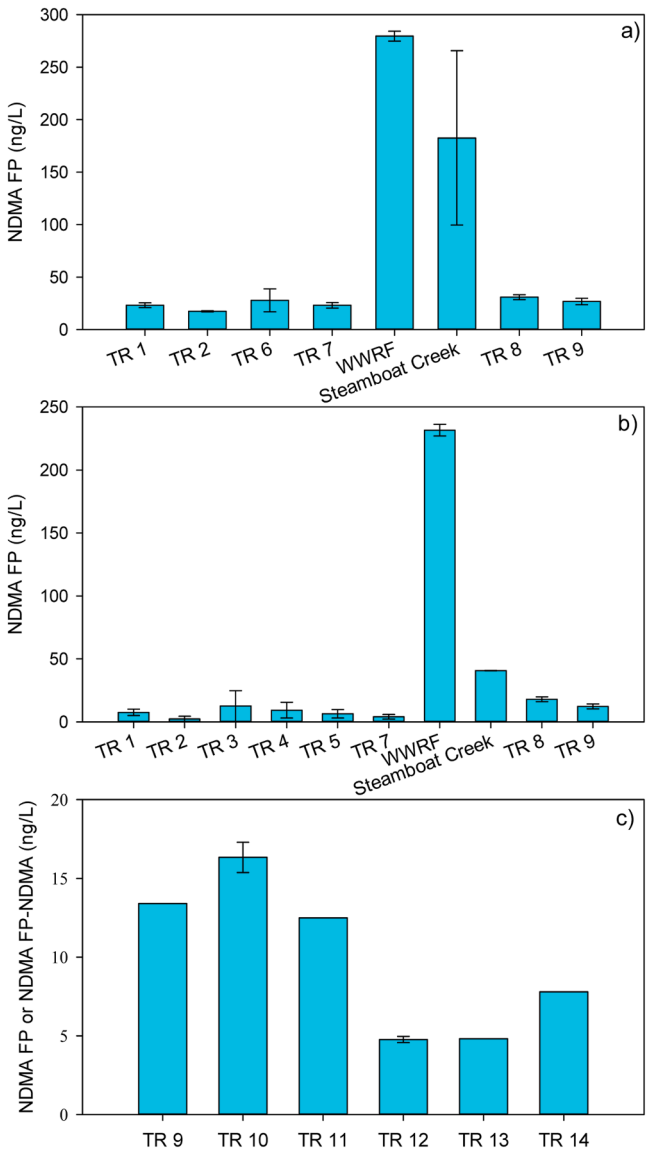

Fig. 2. NDMA precursor loading in the Truckee River, WWRF effluent, and Steamboat Creek on three sampling occasions: a) September 2020, b) October 2021, and c) June 2022. In select samples in June 2022, NDMA was >50 % of NDMA FP, and therefore NDMA FP was calculated by subtracting NDMA from NDMA FP. In other sampling campaigns, NDMA contributed negligibly to NDMA FP and was not subtracted. "WWRF" is the WWRF effluent which discharges directly into Steamboat Creek, and "Steamboat Creek" is the immediately downstream (100 m) of the WWRF effluent, but upstream of the confluence with the Truckee River. Locations TR 8 to TR 14 are downstream of the confluence. Error bars show one standard deviation of triplicate grab samples taken in September 2020 and October 2021 and duplicate grab samples taken in June 2022.

discharges to Steamboat Creek just upstream of the Truckee/Steamboat confluence (WWRF in Fig. 2, 280 ng/L in September 2020 and 232 ng/L in October 2021). NDMA FPs in WWRF effluent were at least 5 times greater than NDMA FP measured in the Truckee River, and 1.5 times greater than in Steamboat Creek downstream of the outfall (12 to 38 % and 59 to 71 % flow contribution from the WWRF to the Truckee River and Steamboat Creek, respectively). Similar results were reported previously where NDMA FPs in wastewater effluents were at least 3 times greater than that measured in the Quinnipiac River (Schreiber and Mitch, 2006b).

3.3. Wastewater-derived NDMA FP degradation below the WWRF outfall

NDMA FP decreased with the increasing distance from the WWRF effluent and were 27 ng/L and 12 ng/L at TR 9, 5.8 km downstream of the WWRF effluent, in September 2020 and October 2021, respectively. This was equivalent to a NDMA FP decrease of 90 % and 95 % over this short distance (Fig. 2a and b). However, this does not account for dilution of the WWRF effluent by Steamboat Creek and the Truckee River. In order to understand the wastewater-derived NDMA precursor losses vs dilution (by Steamboat Creek and the Truckee River), a mass balance that assumes precursors act conservatively was conducted (Fig. 3). NDMA FP calculated by the conservative mass balance compared to the measured NDMA FP at TR 9 in samples from the 2020 sampling were 50 ng/L and 27 ng/L (significantly different, $p<0.05$, t test), respectively, suggesting 46 % of the precursor loading was degraded or lost through other mechanisms (photolysis, sorption to sediment, etc.) over the relatively short 5.8 km reach from the WWRF outfall to TR 9 (Fig. 3). Similarly, in samples from 2021, the predicted NDMA FP at TR 9 was 88 ng/L, compared to the measured NDMA FP of 12 ng/L (86 % decrease, $p<0.05$). Conducting the same mass balance on Cl^- resulted in an inverse trend, where the measured Cl^- was always greater than expected based on the conservative mass balance, although the concentrations agreed reasonably well with the mass balance (Figure S3). The increase in Cl^- compared to the mass balance is unlikely to be from infiltration of groundwater or stormwater containing greater

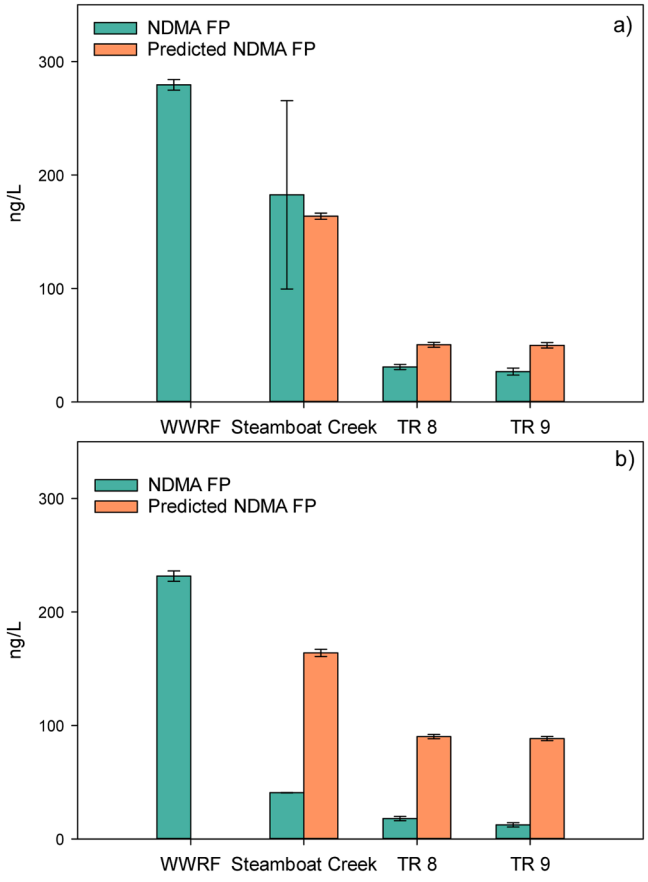

Fig. 3. NDMA FP and predicted NDMA FP based on a mass balance that assumed conservation of precursors from the prior site to the current site in a) September 2020 and b) October 2021. "WWRF" is the WWRF effluent which discharges directly into Steamboat Creek, and "Steamboat Creek" is immediately downstream (100 m) of WWRF effluent, before the Truckee River/Steamboat Creek confluence. Error bars show one standard deviation of triplicate grab samples and error bars for "predicted" include error propagated from the mass balance.

concentrations of Cl^-, as that would be captured by the flowrate of the Truckee River, which was relatively stable (Table S2).

In a follow-on sampling campaign in June 2022, samples were collected from TR 9 to TR 14 to investigate NDMA precursor loadings over a longer distance and further downstream of the WWRF outfall (~86 km). NDMA precursors ranged from 5 to 16 ng/L, with a median of 10 ng/L (Fig. 2c), which is similar to or slightly less than the precursor loading upstream of the WWRF outfall (2-28 ng/L, median = 11 ng/L, "natural background"). Further, there was no statistical difference between the upstream (TR 1-7, sampled in September 2020 and October 2021) and further downstream (TR 9-14, June 2022) NDMA FPs ($p>0.05$, t test), suggesting together with the similarity in NDMA FP range and the losses demonstrated by the mass balances conducted with 2020 and 2021 sampling data, that NDMA FP returned to the natural background concentration within a relatively short distance. Because the precursor measurements made by subtracting NDMA from NDMA FP are influenced by variability in both measurements of NDMA, a mass balance to study the precursor losses was not considered for the sampling event conducted in June 2022.

In order to better understand the decay profiles of wastewater-derived NDMA precursors in the system (Steamboat Creek and Truckee River), correlation analysis between dilution-corrected NDMA FP and river reach distance in Steamboat Creek and Truckee River was conducted (Fig. 4). Dilution by minor tributaries or groundwater influx was not considered. Assuming river distance from the WWRF effluent is approximately equivalent to time, the first order decay rate constants for the three sampling events were 0.05, 0.25, 0.015 km^{-1} (2020, 2021, and 2022, respectively, although data from the 2021 sampling was a somewhat poorer fit than the other two (R^2 of ~0.8 for 2020 and ~ 0.7 for 2022 vs 0.4 for 2021). Thus, it is likely that the rate constant is closer to 0.05 km^{-1} than 0.25 km^{-1}. By multiplying the average expected velocities at the streamflows occurring on the sampling dates (2.32 km/h in 2020, 0.97 km/h in 2021, and 1.7 km/h in 2022) (Bohman, 2000), we arrive at potential rate constants of 0.03 to 0.24 h^{-1}, or, only considering the rate constants from the stronger fits, more likely nearer to 0.03 to 0.12 h^{-1}. Rate is generally affected by temperature but there was no clear relationship between the three days of sampling and water temperature (Table S5). This is likely because the river temperature varied only in the limited range from 6 to 21°C during the three sampling events due to the river being snowmelt dominated and having a relatively short reach.

3.4. Temporal changes to precursor loadings immediately downstream of the WWRF

Prior sampling events were Lagrangian and thus only captured the impact of the WWRF discharge at a single time of day. In order to understand how changes in reclaimed wastewater loading affect NDMA precursor loading in the surface water, samples were collected every hour during one day in Steamboat Creek directly downstream of the WWRF discharge. In the initial sampling event, samples were not collected from the WWRF directly due to autosampler availability, but the WWRF was sampled in the follow-on campaign (discussed below). NDMA FPs ranged from 18 to 98 ng/L (Fig. 5a). NDMA FP was poorly but significantly linearly correlated with WWRF flow contribution (R^2 = 0.2, p = 0.04, Figure S4), a manifestation of the likely changes in NDMA precursor loadings in the WWRF effluent over the 24 hr sampling period. However, when NDMA FPs were binned based on the median of WWRF flowrate contributions ("Low" = below median flow contribution vs. "High" = above median flow contribution, Fig. 5a, median flow contribution =39 %) NDMA FPs at "high" flow contributions were significantly greater than at "low" flow contributions (t test, $p<0.01$). Similar findings have been reported previously, (Uzun et al., 2015) where NDMA FP concentrations increased as the ratio of wastewater treatment plant discharge to river discharge increased from 1 % to 2 %.

In one additional sampling event conducted in June 2022, samples were collected from the WWRF effluent and TR 8, ~2.3 km downstream of the WWRF outfall every hour for 24 hr (Figure S5). NDMA FPs were from 41 to 138 ng/L and 3 to 15 ng/L at WWRF and TR 8, respectively. The correlation between WWRF flow contribution and NDMA FP at TR 8 was poor (R^2 = 0.07 and p = 0.22, Figure S6a). The correlation between WWRF flow contribution and flow normalized NDMA precursor contribution from WWRF ($NDMA\ FP_{WWRF} \times Q_{WWRF}/(NDMA\ FP_{TR8} \times Q_{TR8})$ was also poor but significant (R^2 = 0.26 and p = 0.01, Figure S6b). However, when flow normalized NDMA FP contributions from the WWRF effluent were again binned according to the median flow contribution of the WWRF (median = 5.3 %), a similar trend to the previous sampling campaign was observed (Fig. 5b); at increased WWRF flow contribution, NDMA FP was significantly increased (t test, $p<0.05$). Together, the two sampling events demonstrate that the NDMA precursor pool immediately downstream of the WWRF outfall is directly linked to the WWRF itself. However, we have also demonstrated that the precursors attributable to the WWRF discharge are lost or degraded within ~7 to 24 km of river reach.

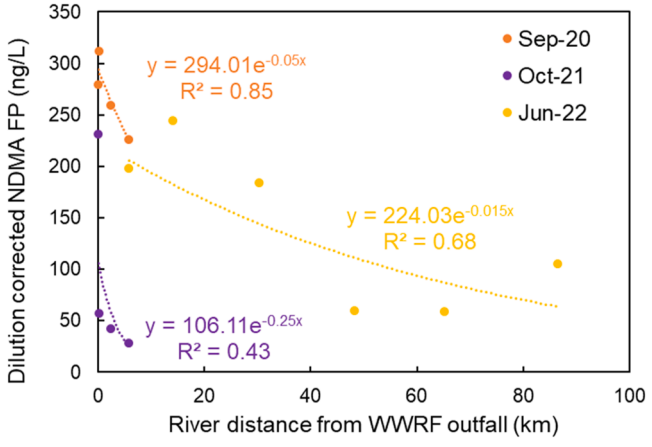

Fig. 4. Dilution-corrected NDMA FP and first order degradation model fits beginning at the WWRF effluent on three occasions.

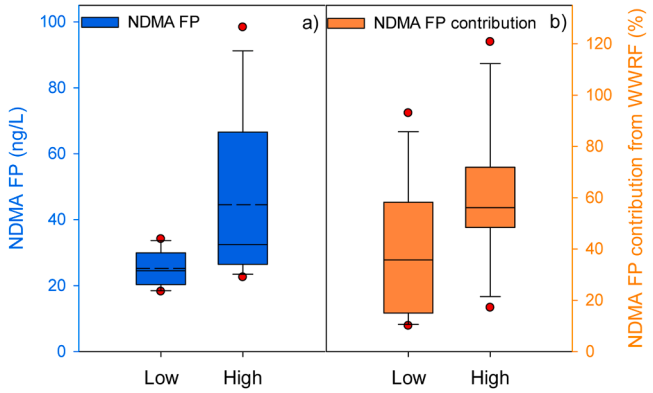

Fig. 5. Boxplot of a) binned NDMA FPs at Steamboat Creek during continuous 24-hr sampling in May 2022 and b) binned flow normalized NDMA FP contribution ($NDMA\ FP_{WWRF} \times Q_{WWRF}/(NDMA\ FP_{TR8} \times Q_{TR8})$ at TR 8 during continuous 24-hr sampling in June 2022. NDMA FPs or NDMA FP contributions were binned based on the median of WWRF flowrate contributions ("Low" = below median flow contribution vs. "High" = above median flow contribution). Boxes show the 25th and 75th percentiles, with a solid line at the median and a dash line indicating mean value. Whiskers show the maximum and minimum. Red circles are the outliers, representing the values exceeding 1.5 × interquartile range (the distance between the upper and lower quartiles).

4. Conclusions

We studied a reach of the Truckee River that has only one significant wastewater input. The dominant source of NDMA precursors was wastewater near wastewater outfall. The precursor loading varied temporally but declined to background/naturally occurring concentrations in as little as ~6 km. Therefore, wastewater-derived precursors in this river appear to degrade or are lost though other mechanisms (photolysis, sorption to sediment, etc.) rapidly and may not be as significant of a source of precursor loading as previously indicated. This may explain, in part, the weak correlations observed by others between wastewater indicator compounds (e.g., sucralose) and NDMA precursors. Naturally occurring NDMA precursors may contribute more than expected to the NDMA precursor pool of surface water drinking water intakes located further downstream, and there is currently limited research that focuses on mitigating or identifying NDMA precursors derived in natural organic matter. Ongoing research focused on identifying and mitigating wastewater-derived NDMA precursors should consider published occurrence data for these compounds in drinking water intakes, if available.

CRediT authorship contribution statement

Mingrui Song: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Junli Wang:** Writing – review & editing, Methodology, Investigation, Formal analysis, Data curation. **Michael DeNicola:** Writing – review & editing, Formal analysis. **David Hanigan:** Writing – review & editing, Visualization, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.watres.2024.122313.

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