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Widespread legacy brine contamination from oil production reduces survival of chorus frog larvae☆

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ABSTRACT

Advances in drilling techniques have facilitated a rapid increase in hydrocarbon extraction from energy shales, including the Williston Basin in central North America. This area overlaps with the Prairie Pothole Region, a region densely populated with wetlands that provide numerous ecosystem services. Historical (legacy) disposal practices often released saline co-produced waters (brines) with high chloride concentrations, affecting wetland water quality directly or persisting in sediments. Despite the potential threat of brine contamination to aquatic habitats, there has been little research into its ecological effects. We capitalized on a gradient of legacy brine-contaminated wetlands in northeast Montana to conduct laboratory experiments to assess variation in survival of larval Boreal Chorus Frogs (*Pseudacris maculata*) reared on sediments from 3 local wetlands and a control source. To help provide environmental context for the experiment, we also measured chloride concentrations in 6 brine-contaminated wetlands in our study area, including the 2 contaminated sites used for sediment exposures. Survival of frog larvae during 46- and 55-day experiments differed by up to 88% among sediment sources (Site Model) and was negatively correlated with potential chloride exposure (Chloride Model). Five of the 6 contaminated wetlands exceeded the U.S. EPA acute benchmark for chloride in freshwater (860 mg/L) and all exceeded the chronic benchmark (230 mg/L). However, the Wetland Site model explained more variation in survival than the Chloride Model, suggesting that chloride concentration alone does not fully reflect the threat of contamination to aquatic species. Because the profiles of brine-contaminated sediments are complex, further surveys and experiments are needed across a broad range of conditions, especially where restoration or remediation actions have reduced brine-contamination. Information provided by this study can help quantify potential ecological threats and help land managers prioritize conservation strategies as part of responsible and sustainable energy development.

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1. Introduction

Domestic energy production is a national priority for the USA and Canada, resulting in rapid increases in activity in many areas (Fig. 1A). Modern techniques and regulations greatly reduce the chances of environmental damages and waste exposure from oil and gas production. Despite reduced risks, spills still occur at modern production sites, and historical (legacy) energy production sites, where waste disposal practices were less regulated than today, are widespread in many landscapes (Gleason and Tangen, 2014; Lauer et al., 2016; Maloney et al., 2017). Either of these situations can cause environmental contamination with largely unknown effects on aquatic organisms (Cozzarelli et al., 2017; Lauer et al., 2016). Investigating the persistence and potential ecological effects of contamination at legacy energy production sites is important for identifying restoration strategies that can inform responsible and sustainable energy development.

The Williston Basin, in North America's Great Plains, has

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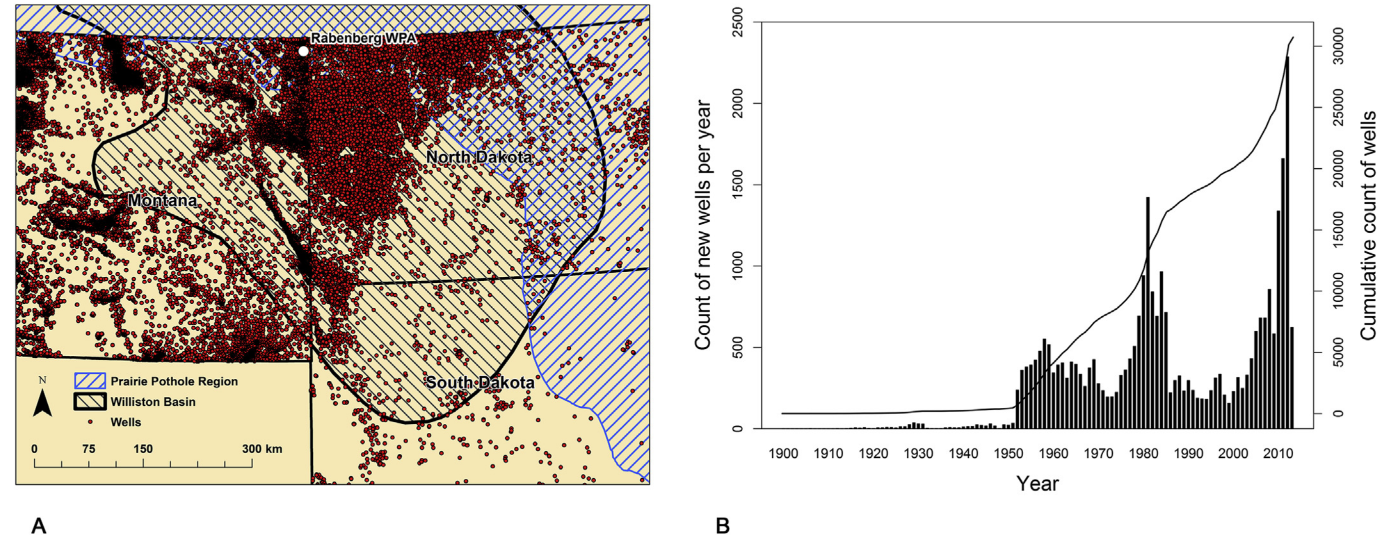

Fig. 1. (A) Distribution of permitted and drilled wells relative to the Prairie Pothole Region (blue hatching) and Williston Basin (black hatching) in Montana, South Dakota, and North Dakota (USA), 1900–2013. (B) Number of newly drilled and permitted oil related wells in the Williston Basin (USA only), 1900–2013 each year (y-axis) and cumulatively (z-axis) (Chesley-Preston, T. 2013. Petroleum related wells in Montana, North Dakota, and South Dakota; https://www.sciencebase.gov/catalog/item/528d0750e4b0c629af455a00). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

experienced an enormous increase in energy production during the last 20 years (Fig. 1A&B). Much of the Williston Basin overlaps with the Prairie Pothole Region, a region densely populated with wetlands that provide numerous ecosystem services and are critical habitat for many aquatic and semi-aquatic species, including waterfowl and amphibians (de Groot et al., 2012; Gleason and Tangen, 2014). As in many other major energy production areas, saline co-produced waters (hereafter, brines) are a byproduct of oil extraction (Cozzarelli et al., 2017; Gleason and Tangen, 2014). The ratio of brine to oil varies spatially and with the age of wells, but it can exceed 14:1 in the Williston Basin (Reiten and Tischmak, 1993). In addition to affecting water quality directly, chemicals in brines can precipitate or become associated with wetland sediments (Beal et al., 1987; Rouse et al., 2013), allowing them to persist long after contaminated water is removed or moves down gradient. Currently, >35% of Prairie Pothole Region wetlands in the Williston Basin are within 1 km of a petroleum-related well (Gleason and Tangen, 2014; Preston and Ray, 2016), which is likely to increase as development continues.

Until the 1970s, most brine from oil extraction in the Williston Basin was stored in unlined reserve pits that often leaked (Beal et al., 1987). Later regulations required a pit liner to prevent seepage, but until the late 1980s, these liners were commonly breached during site restoration (Beal et al., 1987). These brine disposal practices caused persistent contamination of surface and ground water, including on National Wildlife Refuges and other protected lands (Ramirez and Mosley, 2015). Nationwide, there are >5000 wells and >595 pipelines located on National Wildlife Refuge lands; many of these wells are inactive, abandoned, or have unknown status (Ramirez and Mosley, 2015). Today, brine is stored securely and is transported away from active oil fields by pipelines or by truck.

Brines from the Williston Basin and other major shale energy developments often contain high concentrations of total dissolved solids, sodium, and chloride (Lauer et al., 2016; Reiten and Tischmak, 1993). These high chloride concentrations create a distinct chemical signature compared to the ≤10% of regional wetlands that are naturally saline and are characterized primarily by sodium sulfate (Gleason and Tangen, 2014). Brine-contaminated wetlands can be distinguished from naturally-saline wetlands via a locally-developed Contamination Index, which is the ratio of chloride (mg/L) to specific conductance (μS/cm). Contamination Index values > 0.035 generally indicate contamination by brines from produced waters, whereas index values > 0.35 and chloride concentrations ranging from 10,000 to 100,000 mg/L indicate highly-contaminated sites (Preston et al., 2014; Reiten and Tischmak, 1993).

Despite the extensive legacy contamination in some developed oil and gas reserves and potential for contamination in more recently-developed reserves, there has been surprisingly little research into its effects on aquatic and wetland-associated species (Davis et al., 2010; Maloney et al., 2017; Souther et al., 2014), including amphibians in the Williston Basin. Surveys of 10 wetlands in the Williston Basin revealed that taxonomic richness of macroinvertebrates was inversely related to the Contamination Index (Preston and Ray, 2016). In a North Dakota stream affected by a brine spill, 96-hr field exposures to stream water caused reduced survival of larval Fathead Minnows (*Pimephales promelas*) > 6 months after the spill (Cozzarelli et al., 2017). By comparison, there is a large body of literature that shows runoff from road salts and other forms of anthropogenic salinization of wetlands can reduce survival and growth of amphibian embryos and larvae, ultimately simplifying communities (Karraker et al., 2008; Rood et al., 2007; Sanzo and Hecnar, 2006; Turtle, 2000). Importantly, however, negative effects of brine contamination likely extend beyond toxicity from single elements or compounds, because brines can contain lead, chromium, and other toxic heavy metals (Beal et al., 1987; Farag and Harper, 2014).

To help understand the potential effects of brine contamination on amphibians in the Williston Basin, we used a laboratory experiment to assess variation in survival of larval Boreal Chorus Frogs (*Pseudacris maculata*) reared on sediments from 2 moderately brine-contaminated wetlands, a neighboring reference wetland that was minimally contaminated by brines, and sediments from a control site outside of the study area. Boreal Chorus Frogs range from the Southwest USA to the Northwest Territories, Canada, and are the most abundant amphibian throughout much of their range, including in the northern Great Plains (Dodd, 2013; Hossack et al., 2005; Johnson and Batie, 2001). Boreal Chorus Frog larvae are benthic feeders in wetlands and have a 2- to 4-month larval period that increases their likelihood of exposure to contaminants (Dodd, 2013; Unrine et al., 2007). Wetlands in our study area have been characterized primarily according to their chloride concentration and Contamination Index, with limited information on potential effects to aquatic species. We evaluated survival relative to origin of wetland sediment as well as potential chloride exposure because these sediments could pose threats that are not easily characterized during field assessments or with simple measures of water quality.

2. Materials and methods

2.1. Study area

We collected wetland bed sediment from 3 sites near Goose Lake in and around the Rabenberg Waterfowl Production Area (WPA; 672 ha), Sheridan County, Montana (48.8477, −104.1217). Rabenberg WPA was established in 1968 and is managed by the US Fish and Wildlife Service National Wildlife Refuge System. The majority of local oil fields were developed in the 1960s and 1970s; these old developments represent the original sources of brine contamination, likely from improper disposal of brines (Reiten and Tischmak, 1993; Rouse et al., 2013).

Prior research identified several brine plumes associated with these oil-field sites, including one from a brine storage facility on the western side of the WPA (Rouse et al., 2013, Fig. 2). The sediment collection sites represent 2 wetlands (264J, 264P) down-gradient of the brine storage facility that had previously been characterized as moderately contaminated and 1 minimally-contaminated reference wetland (BGWL) that is up-gradient from the likely brine sources (Preston et al., 2012; Rouse et al., 2013). The reference wetland was located at the intersection of 2 unpaved roads bordered by mixed-grass prairie, whereas the 2 contaminated wetlands were surrounded by mixed-grass prairie (Fig. 2).

2.2. Wetland sampling

We collected approximately 30 L of sediments from each of the 3 wetlands on 07–08 April 2015. Sediments were collected from several locations within each wetland with a 13 cm × 8 cm polyvinyl chloride plugging tool. All wetlands had water when sediments were collected. The sediments were shipped to the USGS Columbia Environmental Research Center (CERC, Columbia, Missouri, USA) and stored at approximately 4 °C until 1 week prior to the start of the experiment. We also used sediments from West Bearskin Lake, Minnesota. These fine-grained sediments are used routinely in sediment toxicity testing at CERC to provide a control for experiments (e.g., Dawson et al., 2003; Ingersoll et al., 2015).

For each wetland, a subsample of the homogenized sediments was placed in 125 mL jars and stored at −20 °C prior to metals analysis. All samples were freeze dried to remove excess water and shipped to RTI Laboratories (Livonia, Michigan, USA) for the analysis of 8 major and 19 trace elements by inductively coupled plasma mass spectrometry (ICP-MS) following published protocols (USEPA, 2007a, 1996). Total mercury in sediment was analyzed based on EPA method 7471B using cold-vapor atomic absorption (USEPA, 1998).

To help provide context for the laboratory experiment, we measured pH, specific conductance, and salinity with a YSI 63 m (YSI Inc., Yellow Springs, Ohio, USA) and measured chloride concentrations with Hach QuanTab test strips (Hach Co., Loveland, Colorado, USA; catalog nos. 27449-40, 27513-40) at 7 wetlands on Rabenberg WPA during May and June 2015, including those used for sediment exposures and several others with historic brine exposure. Site BGWL was dry in May 2015 and June 2015, so during field sampling for amphibians (not reported here) and water chemistry we replaced it with site BGWL-B, a neighboring uncontaminated wetland that also was up-gradient from the brine storage facility (Fig. 2).

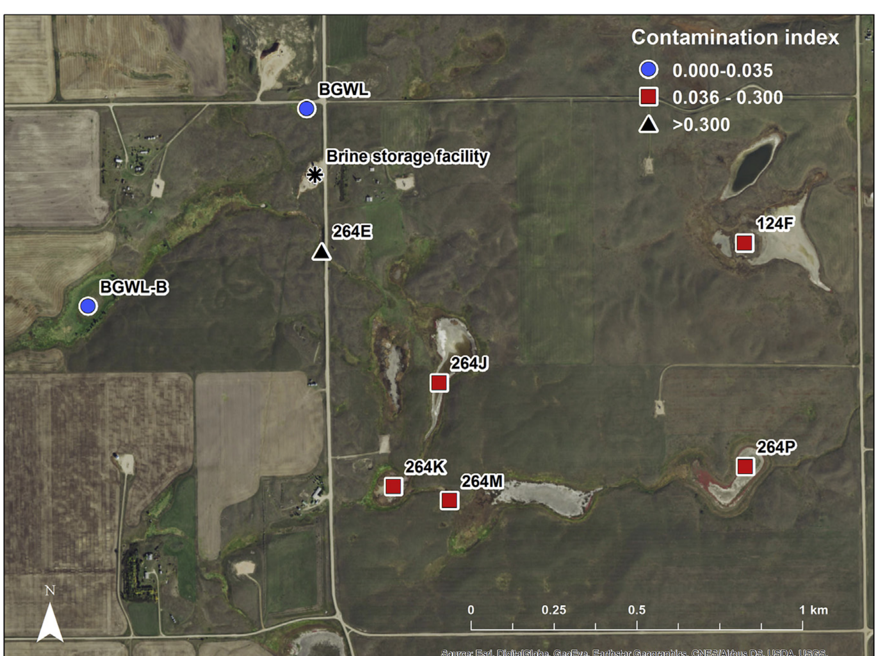

Fig. 2. Rabenberg Waterfowl Production Area, Montana, where we collected sediments from wetlands 264J, 264P, and BGWL during April 2015 for the amphibian experiments. Wetlands are coded according to their Contamination Index (chloride [mg/L]: specific conductance [μS/cm]) during June 2015, except for site BGWL. Site BGWL was dry during June 2015, so it is coded according to its Contamination Index in 2009 (Peterman et al., 2012).

2.3. Amphibian collection

We planned to stock the experiments using embryos collected from uncontaminated wetlands on Rabenberg WPA or the surrounding area. We searched local wetlands for Boreal Chorus Frogs during 04–08 May 2015. Frogs were calling in some wetlands in the area, but we did not find enough embryos for the experiment. Instead, we collected >50 Boreal Chorus Frog embryos from each of 4 uncontaminated wetlands at a study site near Moran, WY (43.8330, −110.3550) on 08–09 May 2015. Embryos were kept on ice and were shipped to the laboratory in source water on 11 May 2015.

2.4. Experimental protocols

One week prior to the start of the study (day −7), we homogenized each sediment sample and added 1.5 L to each of 48 glass exposure chambers (28 cm × 13.5 cm × 25 cm). Each chamber had a 4-cm diameter hole in its side that was covered with 30 mesh (0.5-mm opening) stainless steel screen. We added 5 L of CERC well water (water hardness of ~300 mg/L as $CaCO_3$, alkalinity of ~260 mg/L as $CaCO_3$, pH of ~8.0, and dissolved organic carbon of ~0.4 mg/L) to the sediments and held them under static conditions to allow sediment to equilibrate with the overlying water and form a surface layer of oxidized sediment before starting the exposures (Besser et al., 2013; Wang et al., 2013).

We conducted the experiment in a modified Mount and Brungs (1967) diluter using ASTM guidelines (ASTM-International, 2015a, 2015b). Four replicate chambers were placed within each of 12, 40-L rectangular glass aquaria in a temperature-controlled water bath set at 14 °C. An in-line 4-way flow splitter partitioned the incoming CERC well water (approx. 63 ml/h/chamber) directly into each of the 4 chambers in the aquaria. Excess water overflowed to surrounding aquaria through the screen windows in the exposure chambers, preventing exchange of water among replicates.

Because approximately half of the embryos hatched shortly after arriving at CERC, we conducted 2 parallel experiments, with 6 aquaria for testing survival of embryos exposed to each source of sediment and the other 6 for testing survival of larvae with identical exposures. Within each experiment, we randomly assigned exposure chambers to aquaria, with 6 replicates from each of the 2 brine-contaminated wetland sediments (264J, 264P), 6 replicates from the uncontaminated wetland sediments (BGWL), and 6 replicates from the West Bearskin (WB) control sediments, resulting in 48 total exposure chambers. Prior to the experiments, amphibians were acclimated to the CERC well water by slowly replacing the source water in which they were shipped.

On 13 May 2015 (embryo test day 0), each of the 24 exposure chambers in one experiment was stocked with 8 randomly-selected embryos that were placed in small, perforated cups in which embryos hatched (hatching cups). To allow exposure to the larger volume of water in the exposure chamber, the hatching cups were suspended in the test chambers so the embryos rested approximately 5.5 cm below the water's surface. After the larvae hatched and reached Gosner Stage 25 (Gosner, 1960), they were released from the cups. On 22 May 2015 (larvae test day 0), each of the 24 exposure chambers in the second experiment was stocked with 8 randomly-selected larvae (Gosner stage 22–24). Larvae in both experiments were fed daily 7 mg of a 1:1 mixture of ground high protein flake fish food and organic alfalfa powder.

We checked all experimental chambers daily to count larvae and remove dead individuals until 07 July 2015, when the experiment ended. Only larvae that were found dead were counted as mortalities. One exposure chamber with sediments from reference site BGWL lost 4 larvae (likely through the screen) and was removed from the experiment. All surviving animals were euthanized with MS-222 on 07 July. Because of the different start dates, the experiment that was stocked with embryos lasted 55 days and the experiment that was stocked with larvae lasted 46 days.

We monitored water temperature (mean = 14.4 °C, SD = 0.19), dissolved oxygen (mean = 734 mg/L, SD = 1.6; YSI Pro20 m, YSI Inc., Yellow Springs, Ohio, USA), and chloride (Hach HQ440d benchtop meter with an Intellical ISECL181 chloride ion selective electrode), daily in at least one replicate chamber for each wetland sediment source in each experiment. Water quality (specific conductance, pH, hardness, alkalinity, ammonia) were also measured at the start of the experiment, weekly thereafter, and on the final day of the experiment. Specific conductance was measured with an Orion model 142 m and an Orion 013005A probe (Thermo Fisher Scientific, Waltham, Massachusetts, USA), pH was measured with an Orion EA940 m and a Thermo Scientific Orion 8172BNWP ROSS Sure-Flow pH Electrode, alkalinity was measured by titration with sulfuric acid, water hardness was measured by titration with ethylene-diaminetetraacetate, and ammonia was measured with a Hach HQ440d benchtop meter and an Intellical ISENH3181 ammonia ISE probe.

We collected samples for analysis of metals and major cations from overlying water above the sediments on Day 0, 9, 33, and 55 (Appendix A). Samples were collected with a polypropylene syringe from one replicate of each sediment treatment, filtered through a 0.45-μm pore size polyethersulfone membrane into pre-cleaned polyethylene bottles, and stabilized within 24 h by adding house-distilled concentrated nitric acid (16 M) to each sample at a volume proportion of 1:100 (to result in a final concentration of 1% v/v nitric acid). Water samples were analyzed for arsenic, cadmium, copper, lead, nickel, zinc, and major cations (calcium, magnesium, sodium, and strontium) by ICP-MS using a method similar to USEPA method 6020B (Appendices B & C; USEPA, 2014). Samples for major anions (fluoride, chloride, nitrite, nitrate, and sulfate) analyses were collected at the same time using the same syringe collection and filtration method. Anions were analyzed by anion exchange chromatography using a method similar to USEPA method 9056A (Appendix D; USEPA, 2007b).

2.5. Data analysis

To determine if survival of larvae in the 47 exposure chambers varied according to sediment source or chloride exposure, we fitted the data to generalized linear models with a logit link in program R v 3.1.0 (McCullagh and Nelder, 1997; R Core Team, 2014). We used the quasi-binomial family to model proportion survived because there was more variation in the response data than expected for the binomial distribution. To account for the different lengths of the experiments, we used log-transformed exposure time (55 days for embryos, 46 days for larvae) as a model offset. Analyzing both experiments together resulted in nearly identical estimates as analyzing the data sets individually, but pooling the data produced more precise estimates.

We fit 3 models to the data: (1) a Site Model with the 4 sediment sources as a factor (Site + Stocking Stage + Site × Stocking Stage), (2) a Chloride Model with estimated potential chloride exposure as a covariate (Chloride + Stocking Stage + Chloride × Stocking Stage; range of chloride exposure values: 2130 mg day/L at site WB to 15,300 mg day/L at site 264J), and (3) an intercept-only model. Potential chloride exposure was estimated by using the trapezoidal method to integrate the area under the time–chloride curve for each site (Hayes, 2008). This value represents the mean exposure an individual would have experienced if it survived the entire experiment. We tested the Site and Chloride models by using a likelihood ratio test to compare each of the respective models to the intercept-only model. We also used the quasi-Akaike Information Criterion (QAIC) to compare support for each model (Richards, 2008).

3. Results

Water quality measurements from early May, when Boreal Chorus Frogs in the region would typically be breeding, indicate that site BGWL-B can be considered a reasonable reference site relative to brine waste exposures, whereas sites 264J and 264P had moderately high exposure to brine wastes (Table 1). Site BGWL was dry after the April 2015 sediment collection, but field observations from the neighboring uncontaminated site BGWL-B suggested it was a productive wetland with diverse aquatic plant and invertebrate communities, whereas 264J and especially 264P had simplified communities with almost no emergent vegetation and few invertebrate species (C. Anderson, pers. obs, June 2015).

Chloride concentrations measured from the sediment pore water 7 days prior to the start of the experiments were 77 times (264J) and 167 times (264P) higher than sediment chloride from reference site BGWL (Table 2). By the time we launched the experiment, aqueous chloride concentrations over brine-contaminated sediments had been diluted >87% compared to the pore water but still contained up to 648 mg/L chloride (Table 2). Chloride concentrations in the water overlying brine-contaminated sediments continued to decline until concentrations were approximately equal to the uncontaminated sediments at the end of the experiment. Chloride in the reference (BGWL) and control (WB) chambers never exceeded 50.8 mg/L (Table 2).

Mean survival was similar for the 2 experiments, although there was a wider range of responses for the experiment that was stocked with larvae compared to the experiment stocked with embryos. The source of sediments had a large effect on survival (Site Model: $F = 5.43$, df $= 7$, $p < 0.001$; Fig. 3) and potential chloride exposure reduced survival (Chloride Model: $F = 3.82$, df $= 3$, $p = 0.016$; Fig. 4). Based on differences in QAIC relative to the intercept-only model, the Site Model ($\Delta_{QAIC} = -23.98$) provided a much better fit to the data than the Chloride Model ($\Delta_{QAIC} = -10.51$), so we focused our interpretation primarily on the former.

For the experiment stocked with embryos, mean 55-day survival ranged from 19.4% on 264J sediments to 69.1% on sediments from BGWL, the local contaminated reference site (Fig. 3A). To help interpret results, we plotted mean survival by sediment source in 11-day increments. All embryos that were stocked in chambers with BGWL and WB sediments survived the first 11 days of the experiment and then declined gradually thereafter (Fig. 5A). In contrast, survival of individuals on the brine-contaminated sediments from sites 264P and 264J declined steadily until days 33 and 44, respectively.

For the experiment stocked with larvae, 46-day survival ranged from 11.5% on 264J sediments to 94.1% on site BGWL sediments (Fig. 3B). A few larvae on the reference sediments (site BGWL) died during the first few days of the experiment, but all survived thereafter (Fig. 5B). Survival on the control sediments (site WB) was peculiar. There was a relatively low initial survival rate (lower than for 264J) before it leveled off mid-way through the experiment. Despite the unexpectedly low survival rate for larvae reared on WB sediments, it was still 39% higher than for larvae reared on sediments from site 264P and 81% higher than site 264J (Fig. 3B).

4. Discussion

Although legacy contamination from brines produced during oil extraction is widespread in the Williston Basin and other major oil- and gas-producing areas (Gleason and Tangen, 2014; Maloney et al., 2017), there has been surprisingly little research into its effects on aquatic communities. By rearing larval Boreal Chorus Frogs on brine-contaminated and uncontaminated sediments, our experiment helps close this information gap. Our results show that survival of larval frogs differed by up to 88% based on contamination history (Fig. 3B). Overall, survival was reduced greatly for larvae reared with sediments from contaminated wetlands, despite dilution of chloride and other potentially toxic constituents during the experiment.

Because brines from the Williston Basin can contain high levels of chloride, it is often the primary element of concern for biological effects (e.g., Farag and Harper, 2014). For example, chloride concentrations in overlying surface waters from sites 264J and 264P at day 0 in the embryo experiments were 5 and 12 times higher, respectively, compared to the neighboring uncontaminated site BGWL and the WB control. Initial chloride concentrations in the aquaria's overlying surface water for 264J and 264P were 272 mg/L and 648 mg/L, respectively, below the US Environmental Protection Agency (EPA) acute water quality criteria of 860 mg/L established for the protection of aquatic life (USEPA, 1988). However, chloride concentrations in the overlying aquaria water from the brine-contaminated sites were higher than the EPA's chronic benchmark of 230 mg/L at the beginning of the experiment (USEPA, 1988). For 264J and 264P, chloride concentrations remained above the chronic threshold for 7 and 14 days, respectively, then dropped to below chronic levels for the remainder of the exposure study (Table 1).

Table 1
Water quality measures from 8 wetlands at Rabenberg Waterfowl Production Area, Montana, 2015. Contamination Index (defined in text) values > 0.035 indicate contamination by brines from produced waters; values > 0.35 indicate highly-contaminated sites. Salinity calculations were done according to APHA 1999 (http://www.chemiasoft.com/chemd/salinity_calculator). During sediment collection in April 2015, site 264P was frozen, so specific conductance and salinity were not measured. ND=No data.

Wetland Site	Date	Specific Conductance (μS/cm)	Salinity (ppt)	Chloride (mg/L)	Contamination Index
264J	07 April 2015	2490	1.8	ND	ND
264P	07 April 2015	ND	ND	ND	ND
BGWL	08 April 2015	396	0.2	ND	ND
264J	07 May 2015	7290	4.0	1990	0.273
264P	06 May 2015	14700	8.5	3790	0.258
BGWL-B	05 May 2015	568	0.3	10.2	0.018
124F	24 June 2015	18000	10.6	4880*	0.271
264E	24 June 2015	19100	11.4	6180*	0.323
264J	24 June 2015	11700	6.7	3220	0.275
264K	24 June 2015	3640	1.9	762*	0.209
264M	24 June 2015	6440	3.5	1740*	0.270
264P	24 June 2015	32200	20.0	9230	0.287
BGWL-B	23 June 2015	617	0.3	11.8	0.019

Table 2
Water quality measures in waters overlying 4 sources of wetland sediments. For brevity, we only show data from the experiment stocked with embryos (55-day experiment; day 0 = start date). Chloride on day −7 was measured from sediment pore water. There was little variation in pH (range: 7.9–8.4) or water temperature (range: 14.1–14.7 °C) among sediment sources or over time, so they are not shown. ND=No data. For more information, see Puglis et al. (2017).

Sediment Source	Day of Experiment	Total Chloride (mg/L)	Specific Conductance (μS/cm)	Salinity (ppt)	Dissolved Oxygen (mg/L)	Total NH3 (mg N/L)
264J	−7	2480	ND	ND	ND	ND
	0	272	1320	0.84	3.8	1.2
	7	212	1190	0.76	8.6	0.89
	14	101	987	0.62	6.9	0.48
	21	91.1	927	0.58	7.0	0.62
	28	79.9	906	0.57	8.8	0.41
	35	N/A	818	0.51	7.8	0.90
	42	45.3	653	0.41	8.1	0.38
	49	46.3	674	0.42	7.5	0.08
	55	38.4	644	0.40	8.7	0.05
264P	−7	5360	ND	ND	ND	ND
	0	648	2300	1.5	2.3	2.5
	7	352	1670	1.1	6.2	1.8
	14	202	1400	0.90	6.0	0.23
	21	148	1230	0.74	6.6	0.23
	28	132	1160	0.74	7.8	0.44
	35	N/A	931	0.60	7.8	0.35
	42	64.7	735	0.46	7.8	0.15
	49	53.7	694	0.43	9.2	0.11
	55	48.8	682	0.43	8.1	0.14
BGWL	−7	32.0	ND	ND	ND	ND
	0	50.8	845	0.53	3.4	2.6
	7	44.6	783	0.49	5.5	1.5
	14	32.2	812	0.51	6.4	1.7
	21	34.6	749	0.44	6.9	1.5
	28	42.2	769	0.48	6.1	1.7
	35	N/A	737	0.46	7.7	1.3
	42	34.2	620	0.39	6.1	1.5
	49	38.2	648	0.40	6.4	1.1
	55	34.1	634	0.40	7.1	0.35
WB	−7	1.80	ND	ND	ND	ND
	0	42.4	622	0.38	5.9	0.53
	7	41.9	629	0.40	8.7	0.07
	14	33.0	669	0.42	8.1	0.04
	21	32.9	624	0.39	8.3	0.05
	28	37.1	635	0.39	8.7	0.09
	35	ND	658	0.42	8.7	0.11
	42	32.8	562	0.35	8.85	0.06
	49	38.2	576	0.35	8.55	0.10
	55	31.2	583	0.36	8.30	0.08

The scarcity of information from other long exposures to brine-contaminated sediments in freshwater wetlands limits comparison of our results to other studies. Also, we are unaware of data on the effects of chloride on Boreal Chorus Frogs or other species inhabiting our study area in northeast Montana. However, salinity levels much lower than are common in wetlands in our study region (Table 2; Rouse et al., 2013; Preston et al., 2014) can cause large reductions in survival of a wide variety of amphibians (Karraker et al., 2008; Brown and Walls, 2013). Secondary salinization from groundwater contamination or road salts can decrease species richness with chloride concentrations as low as 400 mg/L (Collins and Russell, 2009), although most anurans (frogs and toads) seem able to persist at ≤ 1500 mg/L and there is evidence for local adaptation to increased salinity by amphibians and other aquatic organisms (Coldsnow et al., 2017; Karraker et al., 2008; Kearney et al., 2012; Smith et al., 2007). The potential for local adaptation to chloride underscores the importance of conducting additional experiments using locally-sourced animals.

Based on the decline of chloride concentrations to below acute or chronic levels during our experiment, we suspect other factors contributed to the large reduction in survival for larvae reared on brine-contaminated sediments. This premise is further substantiated by greater support for the Site Model vs. Chloride Model, and because the rank-order of effects did not follow the rank of chloride concentrations (i.e., 264P sediments had the highest chloride concentrations, but survival was lowest on 264J sediments). Similarly, reduced 96-hr mortality of larval Fathead Minnows in a nearby stream that was contaminated by a ruptured wastewater pipeline was not clearly associated with variation in chloride or other commonly measured contaminants (Cozzarelli et al., 2017). These patterns suggest other chemicals or perhaps interactions between local environmental and chemical conditions can magnify threats to local species, and that threats from contamination may not be easily measured during field assessments or simple measures of water chemistry.

Similar to other studies of environmental contaminants in the region, we analyzed a suite of metals in sediments from our 3 local study wetlands, including barium and strontium, which are considered tracers of oil and gas impacts (Akob et al., 2016; Cozzarelli et al., 2017). Barium concentrations in sediments were similar among the wetlands, while strontium concentrations were higher in 264J (380 mg/kg) than in 264P (220 mg/kg) or BGWL (120 mg/kg; Appendix A). Similarly, aqueous strontium concentrations in 264J were ~10 times those at BGWL in 2009 (Preston et al., 2012). Collectively, these results support previous conclusions of significant brine influence at 264J and 264P and minimal influence at BGWL (Peterman et al., 2012; Preston et al., 2014). How these differences in concentrations of metals might affect

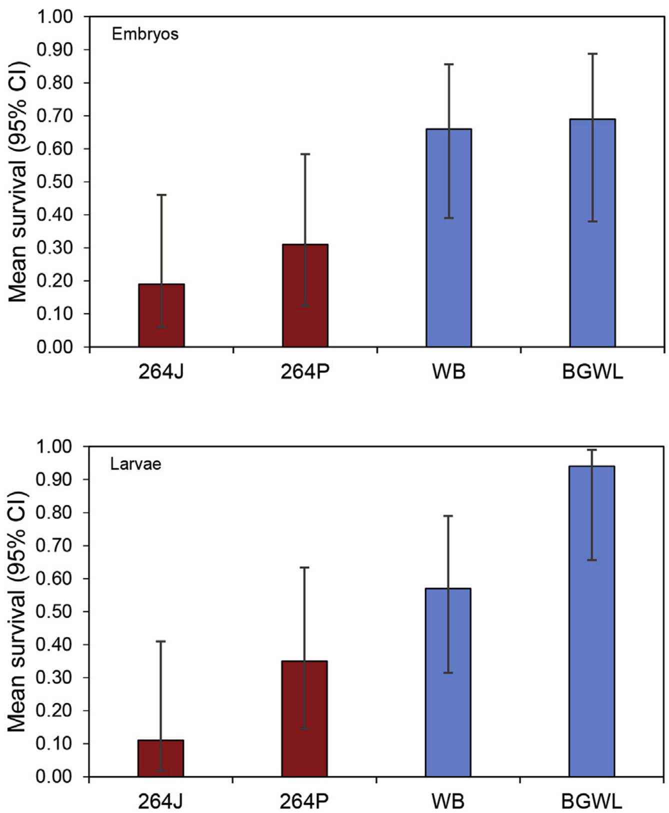

Fig. 3. Mean (±95% confidence interval) estimated survival of Boreal Chorus Frogs (*Pseudacris maculata*) relative to source of sediments for the experiment stocked with embryos (top) and the experiment stocked with recently-hatched larvae (bottom). Wetlands 264J and 264P (red) were contaminated with brines; WB and BGWL (blue) were uncontaminated. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.).

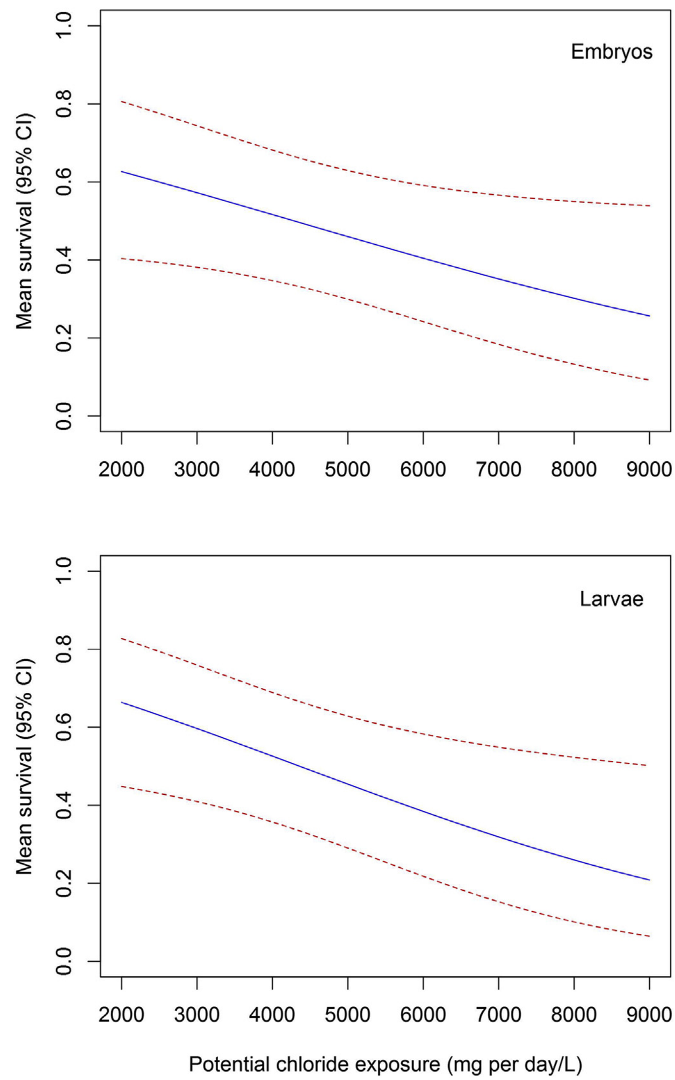

Fig. 4. Mean (±95% confidence interval) estimated survival of Boreal Chorus Frogs (*Pseudacris maculata*) relative to potential chloride exposure for the experiment stocked with embryos (top) and the experiment stocked with recently-hatched larvae (bottom).

survival of larvae is uncertain, because amphibians are among the least-studied vertebrates with respect to effects of metals (Sparling et al., 2010). However, exposure to a suite of trace metals in combination with other endocrine active compounds can alter sex ratios in amphibian populations and reduce reproductive viability (Hopkins et al., 2006; Lambert et al., 2015), and several studies have found negative relationships between species richness and heavy metal contamination (Ficken and Byrne, 2013; Karasov et al., 2005; Sasaki et al., 2015). Our laboratory experiments using field-collected sediments provided an opportunity to assess environmentally relevant effects. But because the contaminant profiles in these sediments are complex, we cannot link decreased survival to a particular contaminant without additional experiments.

For several reasons, we suspect our results could underrepresent the threat of legacy brine contamination to Boreal Chorus Frogs, and perhaps other aquatic species, in our study area. Dilution of the source sediment with laboratory water greatly decreased concentrations of chloride and other contaminants. For example, chloride concentrations in field collected water at 264J and 264P (May 2015) were approximately 7.3 and 5.8 times higher than those measured on day 0 in our experiments. All 6 contaminated wetlands that we sampled in June 2015 (Fig. 2) had chloride concentrations that exceeded the EPA's chronic benchmark for chloride in freshwater (230 mg/L); 5 of these 6 wetlands easily exceeded the acute benchmark (860 mg/L; USEPA, 1988). Providing uncontaminated food during laboratory exposures may have also reduced toxicity to larvae compared to field conditions, because metals and other contaminants are often transferred to algae and other basal food resources (Sofyan et al., 2006; Unrine et al., 2007). Last, responses to contamination and other stressors are often sublethal. Slowed growth and development, reduced post-larval survival, or compromised physiological function that are common for individuals from contaminated habitats may not be manifested during short-term experiments (Chinathamby et al., 2006; Dananay et al., 2015; Hersikorn and Smits, 2011; Sanzo and Hecnar, 2006). To better understand and characterize the ecological effects of brine contamination—including the potential for local adaptation to increased salinity—it will be important to conduct field-based studies such as transplant experiments with several locally-sourced species.

5. Conclusion

Combined with well-documented evidence of extensive legacy contamination and decreased species richness of wetland invertebrates in the area (Gleason and Tangen, 2014; Preston and Ray, 2016; Rouse et al., 2013), our results suggest that persistent

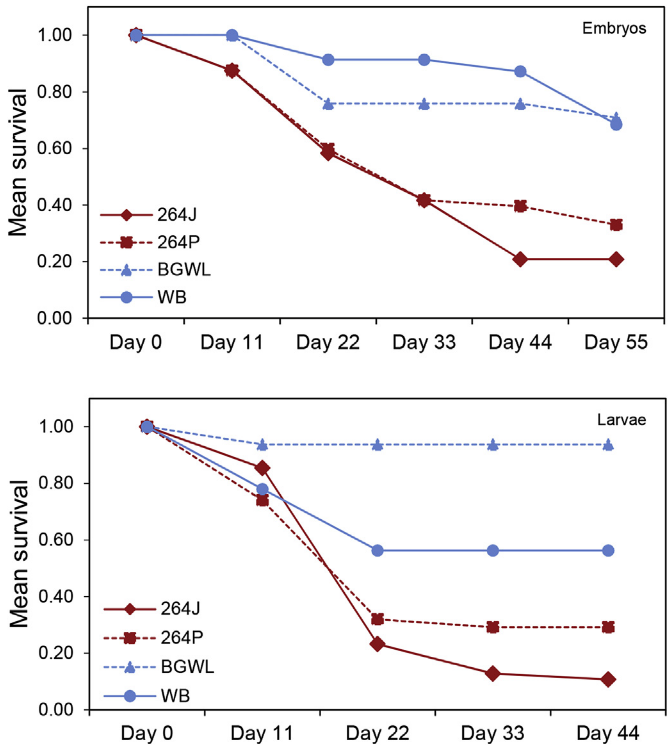

Fig. 5. Temporal trends in observed survival of Boreal Chorus Frogs according to source of sediments for the experiment stocked with embryos (top) and the experiment stocked with recently-hatched larvae (bottom). Wetlands 264J and 264P were contaminated with brines; WB and BGWL were uncontaminated.

brine contamination could cause population-level impacts and broad changes to biotic communities. Preliminary surveys of several wetlands on Rabenberg WPA during 2015, including sites 264J and 264P, provide tentative support for this hypothesis. We did not find Boreal Chorus Frog larvae in local wetlands contaminated by legacy brine-disposal practices; however, chorus frogs were common in uncontaminated wetlands, including in reference site BGWL-B (Fig. 2; B. Hossack and R. K. Honeycutt, unpublished data). Additional surveys and experiments across a range of conditions and areas where restoration or remediation actions have reduced contamination would also help quantify threats and help land managers prioritize conservation strategies as part of responsible and sustainable energy development.

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Appendix A

Metal concentrations (mg/kg) in sediments collected from 3 wetlands (BGWL, 264J, 264P) on Rabenberg Waterfowl Production Area, Montana, April 2015. Cells labeled ‘ND’ were below detection limits.

	Sediment Source		
	264J	264P	BGWL
Major Elements			
Aluminum	7700	8800	19,000
Calcium	130,000	92,000	36,000
Iron	7000	11,000	17,000
Magnesium	58,000	16,000	9200
Manganese	470	800	480
Potassium	2300	3600	5100
Silicon	1500	1900	1800
Sodium	3200	5200	1100
Trace Elements			
Antimony	2.1	3.0	5.0
Arsenic	8.4	4.2	3.2
Barium	120	140	140
Beryllium	0.14	0.17	0.31
Boron	91	110	68
Cadmium	0.14	0.30	0.55
Chromium	8.6	11.0	20.0
Cobalt	2.3	3.1	4.8
Copper	11.0	14.0	18.0
Lead	3.4	5.4	7.4
Lithium	26.0	12.0	11.0
Mercury	0.023	0.032	0.037
Molybdenum	0.83	1.1	0.35
Nickel	13.0	13.0	16.0
Selenium	ND	ND	ND
Silver	ND	0.16	0.16
Strontium	380	220	120
Thallium	1.0	0.39	1.2
Vanadium	21.0	22.0	41.0
Zinc	36.0	45.0	95.0

Appendix B

Metal concentrations (μg/L) in overlying water from the experiments conducted at the USGS Columbia Environmental Research Center. Aluminum, copper, zinc, selenium and cadmium were analyzed but not detected. Cells labeled ‘ND’ were below detection limits.

Sediment Source	Day of Experiment	Chromium	Cobalt	Nickle	Arsenic
Embryo Test					
264J	0	2.68	0.26	3.67	19.2
	33	ND	ND	2.41	2.83
	55	ND	ND	2.34	ND
264P	0	3.10	0.51	5.20	18.0
	33	ND	0.24	3.01	2.33
	55	ND	0.23	2.72	ND
BGWL	0	2.02	0.65	4.34	12.6
	33	ND	0.37	3.35	ND
	55	ND	0.26	2.80	ND
WB	0	2.06	0.50	3.26	11.3
	33	ND	ND	2.83	ND
	55	ND	ND	2.70	ND
Larvae Test					
264J	0	ND	ND	3.03	4.28
	24	ND	ND	2.47	ND
	46	ND	ND	2.34	ND
264P	0	ND	0.33	4.13	3.74
	24	ND	0.27	3.30	2.89
	46	ND	0.26	2.93	<2
BGWL	0	ND	0.29	3.53	ND
	24	ND	0.36	2.96	ND
	46	ND	0.31	2.93	ND
WB	0	ND	0.22	2.81	ND
	24	ND	ND	2.42	ND
	46	ND	ND	2.53	ND

Appendix C

Major cation concentrations (mg/L) measured in pore water (collected from a 100 mL sample of sediment at 5200 rpm for 15min) and overlying water from the experiments conducted at the USGS Columbia Environmental Research Center. Cells labeled 'ND' were below detection limits.

Sediment Source	Day of Experiment	Sodium	Magnesium	Potassium	Calcium	Manganese	Iron	Strontium
Pore water								
264J	−7	1130	414	45	197	ND	1.2	1.2
264P	−7	1720	1250	170	716	2.8	3.5	3.9
BGWL	−7	192	51	67	141	3.3	3.8	1.2
WB	−7	2.8	12	2.6	41	4.8	38	0.1
Embryo Test								
264J	0	126	57	9.5	68	ND	ND	0.4
	33	55	37	6.5	63	ND	ND	0.4
	55	37	35	4.6	66	ND	ND	0.4
264P	0	205	129	29	127	0.2	0.5	0.7
	33	54	48	9.8	77	ND	ND	0.4
	55	42	42	8.6	77	ND	ND	0.4
BGWL	0	50	27	21	78	0.5	ND	0.6
	33	33	27	12	77	ND	ND	0.5
	55	31	28	9.6	75	ND	ND	0.5
WB	0	25	23	2.9	63	1.1	ND	0.3
	33	27	26	3.4	69	ND	ND	0.4
	55	26	25	3.8	69	ND	ND	0.3
Larvae Test								
264J	0	85	42	6.7	72	ND	ND	0.4
	24	46	34	5.4	66	ND	ND	0.4
	46	35	35	4.7	64	ND	ND	0.4
264P	0	130	80	19	100	0.1	ND	0.5
	24	68	56	11	81	ND	ND	0.4
	46	32	43	7.3	82	ND	ND	0.4
BGWL	0	39	27	12	77	0.2	ND	0.5
	24	29	26	9.4	73	ND	ND	0.4
	46	26	25	8.0	72	ND	ND	0.4
WB	0	26	25	2.8	69	0.1	ND	0.4
	24	27	26	2.9	63	ND	ND	0.3
	46	27	24	3.9	69	ND	ND	0.3

Appendix D

Anion concentrations (mg/L) measured in pore water (collected from a 100 mL sample of sediment at 5200 rpm for 15min) and overlying water from the experiments conducted at the USGS Columbia Environmental Research Center. Nitrite and bromium were analyzed but not detected. Cells labeled 'ND' were below detection limits.

Sediment Source	Day of Experiment	Fluoride	Chloride	Nitrate	Sulfate	Phosphate
Pore water						
264J	−7	ND	2481	ND	504	1.8
264P	−7	ND	5364	ND	3080	ND
BGWL	−7	0.4	32	ND	2.2	22
WB	−7	ND	1.8	ND	159	ND
Embryo Test						
264J	0	0.4	225	ND	109	ND
	33	ND	63	ND	78	ND
	55	ND	38	ND	77	ND
264P	0	0.7	479	1.1	333	1.8
	33	ND	90	ND	120	ND
	55	ND	60	ND	115	ND
BGWL	0	0.5	32	1.1	51	4.6
	33	ND	33	ND	47	ND
	55	ND	34	ND	54	ND
WB	0	0.5	29	1.2	78	ND
	33	ND	33	ND	72	ND
	55	ND	32	ND	70	ND
Larvae Test						
264J	0	0.5	137	ND	99	ND
	24	ND	50	ND	73	ND
	46	ND	35	ND	75	ND
264P	0	0.5	325	ND	213	1.7
	24	ND	130	ND	140	ND
	46	ND	45	ND	110	ND
BGWL	0	0.5	31	ND	56	3.6
	24	ND	31	ND	48	ND
	46	ND	32	ND	51	ND
WB	0	0.5	30	1.4	74	ND
	24	ND	32	ND	70	ND
	46	ND	32	ND	68	ND

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