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Field-based method for evaluating the annual maximum specific conductivity tolerated by freshwater invertebrates

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Abstract

Most water quality criteria are based on laboratory toxicity tests and usually include chronic and acute magnitudes. Field-based criteria are typically based on long-term or continuous exposures, so they are chronic. Biological responses of quantified, short-term aqueous exposures are seldom documented in the field. However, acute values may be derived by estimating an upper limit using temporal variance and chronic values. This method estimates an upper limit from the variance of pollutant measurements from stream locations that attain the chronic criterion. The formula for deriving a 90th centile of a standard normal distribution is used to identify the upper limit, a criterion maximum exposure concentration (CMEC). The calculated CMEC is interpreted as a maximum exposure that 95% of organisms may tolerate if the chronic exposure is not exceeded. The methods of deriving chronic and acute criteria are illustrated with specific conductivity in a mountainous area in the eastern United States. The biological relevance of the CMEC was assessed using the maximum annual exposure during the life cycle of the most salt-intolerant genera. The method using the chronic criterion and the variance of water chemistry data is practical, whereas frequently collecting and analyzing paired biological and chemical samples at numerous sites is impractical and may give misleading results due to lags in biological response. This method can be used anywhere with sufficient data to estimate the temporal variability and may be applicable for field-based criteria other than the specific conductivity criteria illustrated here.

Graphical Abstract

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Keywords

Upper tolerance; Water quality criteria; Benthic macroinvertebrates; Ionic mixture; Extirpation; Aquatic life

1. Introduction

Protection and reestablishment of conditions that support aquatic life depend upon chemical and physical benchmarks, standards, and criteria to set loading limits and clean up goals (e.g. Stephan et al., 1985; Erickson and Stephan, 1988; CCME, 2007; van Vlaardingen and Verbruggen, 2007; European Commission, 2011; Warne et al., 2015). Most criteria are developed from laboratory-based toxicity tests. However, relevant exposure routes, effect endpoints, species, and life stages are not always captured by laboratory test methods. In some situations, laboratory toxicity tests have resulted in higher criterion concentrations than those that cause effects in the field (U.S. EPA, 2011b; Mebane et al., 2015; Yang et al., 2017). For ionic mixtures, field-based methods for developing benchmarks appear to be more appropriate (Cormier and Suter, 2013; Cormier et al., 2013a).

Salinization of streams is recognized as a global problem (e.g. Environment Canada and Health Canada, 2001; Zielinski et al., 2001; Higgins and Wilde, 2005; Kaushal et al., 2005; Zalazniak et al., 2006; Gillis, 2011; Findlay and Kelly, 2011; Karatayev et al., 2012; Kefford et al., 2012; Cañedo-Argüelles et al., 2013; Johnson et al., 2013; Kaushal et al., 2013; Vander Laan et al., 2013; Dunlop et al., 2015; Johnson and Johnson, 2015; Zhao et al., 2016). To address this problem, the United States Environmental Protection Agency (U.S. EPA, 2011b) published a field-based method for developing annual average continuous exposure benchmarks for specific conductivity (SC). However, the method did not provide for developing duration, frequency, and acute magnitude parameters usually associated with implementation of benchmarks or criteria.

This paper provides a method for developing an acute value to complement a chronic benchmark or criterion derived from field data. For simplicity, chronic and acute benchmarks and criteria are referred to by two acronyms, criterion continuous concentration (CCC) and criterion maximum exposure concentration (CMEC), respectively. A case study from the eastern United States is used to illustrate the approach by first deriving a CCC, then the CMEC for SC. Numerous researchers have documented the adverse effects of SC in this region (e.g. Pond et al., 2008; Fritz et al., 2010; Gerritsen et al., 2010; Palmer et al., 2010; Pond, 2010; Lindberg et al., 2011; Merriam et al., 2011; Timpano et al., 2011, 2015; U.S. EPA, 2011a; Bernhardt et al., 2012). Although various sources are present in the region,

valleys filled with overburden from mountain-top-removal coal mining are a major source of increased SC (Cormier et al., 2013b).

2. Methods

2.1. Overview of approach

Acute criteria are infrequent, short duration exposures that may occur without causing effects above the upper limit of acceptability. In the method described in this paper, the threshold of the effect is the extirpation of 5% of invertebrate genera (the effect at the CCC). The acute exposure is defined by a low-frequency, short duration exposure level that occurs in streams that meet the CCC. That is, the CMEC is a brief exposure greater than the CCC that can occur without causing the same effect as an annual average exposure at the CCC. The field-based method for developing a CMEC, described here, uses a normal distribution to characterize the 90th centile of sites with an average annual SC less than or equal to a CCC. A similar approach was applied to human epidemiological data to develop water quality criteria for bacteria causing gastroenteritis (U.S. EPA, 2012).

Normal distributions are defined by a mean and standard deviation (SD). A reordering of the algebraic formula for the statistical Z -formula allows one to estimate a centile of a normal distribution. The calculation uses the annual mean of a distribution (by definition the CCC) and the SD of the SC measurements from the population of sites with multiple measurements. Therefore, the method requires a known and reliable CCC that is protective in a particular geographic area and data set of SC measurements sufficient to characterize the SD. To evaluate the resulting CMEC attained by this method, a paired chemical and biological data set was used that contained SC measures for the year preceding biological sampling. This data set was used to characterize the salt-intolerant genera that survived different annual maximum SC.

2.2. Geographic study area

Samples were collected from the Central Appalachians (Ecoregion 69) which extends from central Pennsylvania through West Virginia and Kentucky to northern Tennessee with small portions in Maryland and Virginia (Omernik, 1987; Woods et al., 1996; U.S. EPA, 2013) (Fig. 1). The climate is characterized by distinct warm and cold seasons with rainfall ranging between 960 and 1520 mm/yr. The high hills and low mountains are covered by a mixed hardwood forest with pasture and agriculture in the valleys. This high (366 to 1402 m), rugged plateau is underlain with sandstone, shale, conglomerate, limestone outcroppings, and coal. Underground and surface bituminous coal mines are common (Woods et al., 1996, 1999). Headwater streams in this ecoregion have some of the lowest SC in the United States (Griffith, 2014). However, high levels of SC are associated with coal mining (Cormier et al., 2013b).

2.3. Data sets

The data used in this study was obtained from the West Virginia Department of Environmental Protection (WVDEP) in-house Watershed Assessment Branch database (WABbase). Chemical and biological samples are from 1996 to 2011 and 1997–2010,

respectively. Data sets are available from the U.S. EPA Environmental Dataset Gateway (Cormier, 2017a,b).

Data were collected from 2299 perennial streams in Ecoregion 69. Sites included long-term monitoring stations, targeted sites within watersheds sampled on a rotating basin schedule, randomly selected sample sites (WVDEP, 2015), and sites on impaired stream segments (WVDEP, 2008a). Some sites were sampled monthly for water quality, but most sites were sampled once during an annual sampling period. The data set contains geographic location and both raw data and calculated metrics of water quality, habitat, watershed characteristics, and macroinvertebrate composition (WVDEP, 2008a). The WABbase data set includes assignment of reference status using a tiered approach. Analyses involving the use of these reference sites were drawn from the WABbase Level 1 reference status (WVDEP, 2008b). These are reference sites that “are thought to represent the characteristics of stream reaches that are minimally affected by human activities and are used to define attainable chemical, biological and habitat conditions for a region (WVDEP, 2013)” (sensu Stoddard et al., 2006).

Data filters that were applied prior to finalizing the data set for developing the CCC are described below. A total of 9806 records from Ecoregion 69 are included in the data set; of these, SC measurements were included in 8989 samples. Most of these are measurements of water quality without biological sampling. There are 1911 paired samples with SC measurements and biological samples. Of these, a total of 250 samples were removed from the data set to avoid potential acid mine drainage effects (pH < 6, 237 stations) and different ionic mixtures (e.g. road deicers) with a high proportion of chloride ions, ($[\text{HCO}_3^-] + [\text{SO}_4^{2-}] - [\text{Cl}^-]$) (13 samples). Occurrence at a minimum of one reference site and occurrence in 25 or more samples were requirements for inclusion of a genus in the CCC analysis.

2.4. Biological and chemical methods

Benthic macroinvertebrate samples were collected from a total area of 1 m² from a 100 m reach at each site. Using a 0.5 m wide rectangular kick net (595 µm mesh), four 0.25m² riffle areas were sampled. In narrow or shallow water, nine areas were sampled with a 0.33 m wide D-frame dipnet of the same mesh size. Samples were combined and preserved in 95% denatured ethanol. A random subsample of 200 individuals (±20%) was identified in the laboratory. All contracted analyses for chemistry and macroinvertebrate identification followed WVDEP’s internal quality control and quality-assurance protocols (WVDEP, 2006, 2008b). The data set had been well constructed by the WVDEP with excellent quality assurance based on our inspection of the database and supporting documentation.

3. Calculations

3.1. Calculating genus extirpation concentrations (XC₉₅) and criterion continuous concentration

The example CCC was calculated in a two-step process following the field-based method using an extirpation concentration distribution (XCD) (U.S. EPA, 2011b; Cormier and Suter,

2013; U.S. EPA, 2016). First the extirpation concentration values at the 95th centile (XC_{95}) values were calculated. The XC_{95} for each genus with >25 occurrences was calculated as the 95th centile of a weighted cumulative frequency distribution (CFD) of SC levels at sites where a genus was collected. Then, a frequency distribution of XC_{95} values was constructed, and the 5th centile was identified from the XCD by a log linear 2-point interpolation. In this study, the CCC is the 5th centile rounded to two significant figures.

3.2. Calculating the CMEC-field-based method

The field-based CMEC method relies upon the assumption that the acute value is a high exposure level to which a community may be briefly exposed while its annual average SC meets the CCC. That high level is approximated by the 90th centile of a normal distribution with the mean equal to the CCC. The z -score is obtained from the area under the normal probability curve for a selected probability, in this method, 0.90. That is, there is a high probability that at sites meeting the CCC, a SC sample will be less than the 90th centile which is identified as the CMEC. This does not mean that sites with an annual maximum < CMEC will have a high probability of achieving the CCC. Rather, the CMEC is an exposure higher than the annual average that could occur and a site might achieve the CCC. Therefore, it is essential that the CMEC is used in concert with the CCC. For a higher level of protection, U.S. EPA (2012) recommended using centiles between 0.75 and 0.90, an option that could also be applied to this method.

A normal distribution is statistically characterized by the mean and SD. In the example, the mean is the CCC and the SD is from the population of SC measurements with an annual geometric mean at the CCC. A minimum of six water chemistry samples over the course of the year prior to biological sampling were used to calculate an annual mean exposure during the univoltine life cycle of spring emergent genera. The sample set of stations ($n=564$) were sampled at least six times on a rotating yearly basis (in Ecoregion 69 from July to June). At least one sample occurred during the low SC season (March–June), and one sample in the high SC season (July–October). As with the derivation of the CCC, the data set includes a range of exposures that causes adverse effects to the most salt-intolerant taxa.

The calculation is performed as follows (Fig. 2). First, the CCC for SC is derived from paired biological and chemical stream data (Section 3.1). Next, using a data set from sites with multiple SC measurements, the SDs of sites are calculated to identify a subset of sites with a relatively stable SD. A LOcally WEighted Scatterplot Smoother (LOWESS, span =0.75) is used to estimate the trend line. This is necessary because streams with very low or very high SC may have lower variances of SC. If the LOWESS indicates that the site SDs are stable regardless of the annual mean log SC for sites, then the SD for all observations from sites with multiple measurements is calculated. If the LOWESS indicates that the SD is stable only within a SC range, only sites with an annual geometric mean of log SC within a defined range near the CCC is used to calculate the SD. The total or interval SD value is used to represent the typical variability around the CCC. The CMEC is calculated at the 90th centile from log values of SC in the region using Eq. (1).

$$90^{\text{th}}\text{centile of log-normal distribution of SC} = 10^{(X + Z\alpha S)} \quad \text{Eq. 1}$$

In Eq. (1), X is the log 10 annual geometric mean SC limit for all stations (X ; i.e. the CCC), z_{α} is the critical value for the 90th centile area under a normal distribution (α , 0.10), and s is the total SD. The CMEC is calculated based on Eq. (1) by substituting the CCC for X to calculate the 90th centile. The 90th centile is rounded to 2-significant digits which yields the CMEC. The statistical package R, Version 2.12.1 (December 2010), was used for all statistical analyses (R Development Core Team, 2011). The calculated value is interpreted as a maximum exposure that 5% of organisms may tolerate if the chronic exposure is not exceeded.

3.3. Evaluation of the chemical CMEC method

For the paired biological and chemical analysis, an effect endpoint was selected that was directly relevant to the method for developing the chronic criterion: the occurrence of the genera extirpated below the chronic criterion. The biological relevance of the CMEC was evaluated by observing salt-intolerant genera after exposure in streams that did or did not exceed the calculated CMEC. A data subset was constructed from the Ecoregion 69 example CCC-data set. Of the 564 sites sampled in the data set, only 111 sites had biological measurements following a year with sufficient chemical measurements. Reference sites and impaired sites represented the variability of within site annual variability.

The relationship among SC, temperature, and the presence of the 7 most salt-intolerant genera, 5% of the XCD, were inspected for each of the 111 sites meeting the requirements for inclusion in the data set. Salt-intolerant taxa are those taxa with an $XC_{95} \leq 310 \mu\text{S/cm}$ (the example CCC).

3.4. Characterization of data sets

The “CCC-data set” has 1661 samples belonging to 1420 sites (stations; depicted in Fig. 1). Of these 1661 samples, 186 (11.2%) were sampled more than once between 1996 and 2010. Summary statistics for the CCC-data set is shown in Table 1. SC ranged from 15 to 3794 $\mu\text{S/cm}$ which enabled us to assess a range of SC effect levels. In the WABbase data set in Ecoregion 69, a total of 219 macroinvertebrate taxa were identified to genus. Of these, 193 genera occurred at least once at one of 64 identified reference sites in the data set where invertebrate samples were collected. A total of 142 genera occurred at 25 or more sampling locations.

3.5. Ionic composition

The ionic composition of the samples in the Ecoregion 69 data set was assessed to ensure a relatively similar proportion of ions at sites in the data set (Fig. 3). After low pH samples were removed, 56% of samples (938 in total) included measures of calcium, magnesium, sulfate, bicarbonate, and chloride, all but 13 sites (>98%) were dominated by bicarbonate and sulfate anions, $([\text{HCO}_3^-] + [\text{SO}_4^{2-}])$ (Cl^-) in mg/L. These 13 chloride-dominated sites were excluded from the derivation analysis but these sites are shown in Fig. 3 which depicts the ion ratios in the parent data set. Because >98% of samples with measures of individual ionic measurements were dominated by bicarbonate and sulfate anions, sites with no ionic measurements were retained in the data set with the assumption that <2% of samples in the Ecoregion 69 example CCC-data set would be chloride-dominated.

4. Results

4.1. Extirpation concentration (XC₉₅) and hazardous concentration (XCD₀₅) values

The Ecoregion 69 CCC-data set (Table 1) was used to develop XC₉₅ values from weighted CFDs. The histogram depicted in Fig. 4 shows that the SC stations <30 µS/cm and >1500 µS/cm were less frequently sampled. Because the number of samples along the SC gradient was uneven, the occurrences of genera were weighted prior to the estimation of XC₉₅ values (Cormier and Suter, 2013).

The XC₉₅ values that were used in the XCDs are listed in the order of least to most salt-tolerant in Appendix A. Generalized additive model plots were used to designate approximate (~) and greater than (>) designations for those XC₉₅ values depicted in Appendix B (U.S. EPA, 2011b; Cormier and Suter, 2013). The weighted CFDs used to derive the XC₉₅ values are shown in Appendix C. The hazardous concentration for the 5th centile (XCD₀₅), the 5th centile of the XCD, for Ecoregion 69 was calculated at 305.4 µS/cm (Fig. 5); the two-tailed 95% confidence bounds were 233–329 µS/cm. Those bounds, derived by bootstrap resampling (Cormier and Suter, 2013), indicate that different data sets could yield XCD₀₅ values within that interval. Rounding to two significant figures, the example CCC for Ecoregion 69 is 310 µS/cm.

4.2. Criterion maximum exposure concentration

The CMEC was derived from the CMEC data set. In this example, the CMEC data set was composed of 5811 samples in a July to June rotating year representing 564 rotation years and 536 unique stations with at least 1 sample from July to October (J–O), 1 sample from March to June (M–J), and at least 6 samples within a rotation year. Note that inclusion of samples is not contingent on biological data. Reference and nonreference sites were included to ensure a range of SC (Appendix D). Sites were more heavily sampled in April through September; with both cool rainy seasons and warm dry seasons represented. The variability of within station SC was found to differ for streams with different mean SC (Fig. 6). A line was fitted to the data shown in Fig. 6 using a LOWESS. The average variability (SD for a station) in the middle of the SC gradient is slightly higher than both the lower and higher ends of the gradient. The stations with annual mean SC values between the 25th and 75th centile (which is approximately between 120 and 520 µS/cm) have relatively constant variances, and therefore, were used to estimate the SD components of the example CCC (310 µS/cm) used in Eq. (2). There are 2855 samples from 278 stations in the selected data sets for Ecoregion 69 with streams having mean SC values between 120 and 520 µS/cm. The log 10 geometric mean SC and SD of this data set were determined and the CMEC was calculated using Eq. (1) such that 90th centile of SC at any station within the region is predicted to fall below the CMEC if the CCC is not exceeded. The example CMEC calculation is shown in Eq. (2).

$$\text{Example calculation of CMEC: } 10^{\log_{10}(310 \mu\text{S/cm}) + 1.28 * 0.243} = 633.5 \mu\text{ S/cm} \quad \text{Eq. 2}$$

This result was rounded to two significant figures, yielding an example CMEC of 630 µS/cm for Ecoregion 69.

4.3. Evaluation of CMEC with biological and chemical data

The biological relevance of the example CMEC was evaluated using data from Ecoregion 69 sites that had multiple SC measurements in the year prior to a biological sample. The maximum measured SC was identified during the year preceding the biological sample. The maximum SC of streams in Ecoregion 69 usually occurred in August or September. Salt-intolerant genera tend to be more commonly observed when they are larger and nearing emergence as winged adults usually in April through June. For example, Morris Creek had 2 salt-intolerant genera collected in April and a maximum SC of 450 $\mu\text{S}/\text{cm}$ (Fig. 7) and an annual average well below 310 $\mu\text{S}/\text{cm}$.

An analysis was performed to compare the example CMEC with an estimate of a tolerated maximum SC using salt-intolerant genus survival as the assessment endpoint. A scatter plot was constructed of the count of the 7 most salt-intolerant genera and maximum SC that occurred in the year prior to biological sampling (Fig. 8). The analysis showed that there is a negative relationship between maximum SC and number of salt-intolerant genera. There are few observations of salt-intolerant genera at sampling locations $>630 \mu\text{S}/\text{cm}$, the example CMEC.

5. Discussion

Field-based methods to develop water quality criteria have many inherent advantages. Laboratory studies have the benefit of a controlled environment but often cannot replicate the range of conditions, exposure routes, effects, or interactions that occur in nature (Buchwalter et al., 2017; Cormier and Suter, 2013). In contrast, field exposures occur at realistic physical and chemical conditions, levels, proportions, and variability of pollutants. While field studies include covarying variables that may confound any presumed association between stressor and effect, the relative influence of potential confounders can be assessed and their influence mitigated by a number of approaches (Suter and Cormier, 2013). Organisms in the field have realistic nutrition and levels of stress. Field studies can include more taxa than are available in laboratory data sets (Cormier and Suter, 2013; Yang et al., 2017). Field data include sensitive taxa and life stages and indirect effects that can affect the susceptibility of populations to disease, predation, competition, and maladaptive behaviors. Therefore, having field-based methods can improve our understanding of pollutant effects. Field-based methods are especially valuable when available laboratory toxicity results do not match the exposures that are causing adverse effects in the field, as in the case of ionic mixtures.

As shown in the case example, extirpation of benthic invertebrates occurs at low levels of SC. This is consistent with previously published studies with similar ionic mixtures (e.g. Gerritsen et al., 2010; Palmer et al., 2010; Lindberg et al., 2011; Merriam et al., 2011; U.S. EPA, 2011a, 2011b; Bernhardt et al., 2012; Cormier et al., 2013a; Timpano et al., 2011, 2015; Dunlop et al., 2015; Zhao et al., 2016). Mesocosm studies have begun to identify the modes of action that likely lead to extirpation in the field and corroborate effects observed in the field (Clements and Kotalik, 2016). Also, recent toxicity studies have begun to bridge the gap between physiological studies and field observations (Kefford et al., 2004; Kunz et al., 2013; Mount et al., 2016; Wang et al., 2016, 2017; Erickson et al., 2017). The gradual

coherence of diverse types of studies has strengthened the ability to develop scientifically defensible water quality criteria.

The development of a method to identify a protective short-term maximum exposure for SC was challenging. The failure to observe a salt-intolerant genus does not necessarily indicate that the genus is absent. For the example area, salt-intolerant genera are less likely to be observed during the summer when SC is most likely to be at its maximum for sites with variable SC. This occurs because many of the salt-intolerant genera have a life cycle that is keyed to availability of leaf litter (Cummins and Klug, 1979; Hershey et al., 2010). As a result, many salt-intolerant genera are very small and not usually observed during part of their life cycle that coincides with the maximum annual SC for a stream. In Appalachian streams, deciduous leaf abscission occurs September through November. Therefore, it is likely that a data set of temporally matched SC and the occurrence of a genus would not include short duration exposures that are tolerated.

A method similar to the field-based XCD method for developing a CCC (Cormier and Suter, 2013; U.S. EPA, 2016) could be adapted for an upper SC exposure limit. However, the necessary data collection effort would be almost prohibitive. The data set would have to include frequently collected SC samples to ensure measurement of the range of SC including those near the maximum. It would also require biological monitoring after the maximum exposure had occurred (e.g. in this example, the summer) and when surviving salt-intolerant genera are observable (e.g. generally in this example, the spring but not always). Because these data sets are expensive and rare, the field-based CMEC method was developed so that only a CCC and SD of the exposure of SC are required. The CCC may be derived from an XCD of paired biological and SC measurements (Cormier and Suter, 2013; Cormier et al., 2013a) which are data intensive. But, the CCC may also be derived from background SC and a preexisting background-to-criterion (B-C) model (Cormier et al., this issue). Large chemistry data sets of repeated samples of SC are available in the United States from state, U.S. EPA, and United States Geological Survey monitoring programs (<https://water.usgs.gov/owq/data.html>).

Although the primary objective of this paper was to demonstrate a method for developing a CMEC, an example CCC for a mountainous region of the eastern United States was also produced. The XCD₀₅ of 305 $\mu\text{S}/\text{cm}$ in Ecoregion 69 was similar to a previously published value of 295 $\mu\text{S}/\text{cm}$ for a combined data set of adjacent Ecoregions 69 and 70 (U.S. EPA, 2011b). The 10 $\mu\text{S}/\text{cm}$ difference is within the confidence bounds of the original benchmark for the combined data set of ecoregions 69 and 70 (U.S. EPA, 2011b). The slightly higher value is attributed to random variation and to <25 occurrences of some salt-sensitive genera in Ecoregion 69 compared to the combined area used in 2011. It is expected that 5% of aquatic life in streams in Central Appalachia would be extirpated when the annual geometric mean SC in a stream does not exceed the example CCC.

The primary objective was also met. When a CCC is known, a CMEC can be developed with multiple measurements of SC measurements from many sites. The CMEC for the example was 630 $\mu\text{S}/\text{cm}$. The CMEC derived using variance in SC data was biologically relevant. Salt-intolerant genera were commonly observed when the CMEC was not exceeded and

rarely observed when it was exceeded (Fig. 8). However, the upper limit was only approximated. The paired biological data set is much smaller than the chemistry data set. Furthermore, the occurrence of only one genus in streams $>500 \mu\text{S}/\text{cm}$ could indicate that this CMEC method is not sufficiently protective. Observations of some salt-intolerant genera could be spurious. Salt-intolerant genera not seen above a confluence have been observed below the confluence with low SC tributaries (Pond et al., 2014). As a precaution, a short duration for a CMEC of one day is recommended and a CMEC needs to be used together with a protective CCC (U.S. EPA, 2012).

All the analyses presented in this paper were repeated for an adjacent ecoregion, the Western Allegheny Plateau, Ecoregion 70. Salt-intolerant genera were commonly observed when the example CMEC for Ecoregion 70 was not exceeded and rarely observed when it was exceeded (U.S. EPA, 2016). The example CCC was $340 \mu\text{S}/\text{cm}$. The example CMEC was $680 \mu\text{S}/\text{cm}$. In addition, a wealth of information was collected regarding individual genera. These include: seasonal and geographical occurrences of each genus, XC_{95} values, and the probability of occurrence of each genus at a SC level. Both raw data and related graphs and tables are publicly available to enable additional analysis and synthesis by other researchers.

6. Conclusions

A CMEC can be estimated by defining the variability of SC over the course of a year and calculating the 90th centile of observed SC measurement at locations meeting the CCC. Furthermore, the CMEC derived using the variance in SC data was biologically relevant. Salt-intolerant genera were commonly observed when the CMEC was not exceeded and rarely observed when it was exceeded (Fig. 8). The case example demonstrates that the method is a pragmatic solution to the problem of deriving an acute field-based criterion to complement a chronic criterion. The method may be applicable to pollutants other than SC. In summary, this practical field-based method for developing a CMEC is scientifically defensible and ecologically relevant for the protection and recovery of streams from excess loadings of dissolved minerals.

A CCC is required for the calculation of a CMEC with this field-based method. The XC_{95} values that were used in the XCDs are listed in the order of least to most salt-tolerant in Appendix A. The generalized additive model plots are depicted in Appendix B. The weighted CFDs are shown in Appendix C. Appendix D provides a table of the number of samples with reported genera and specific conductivity in the data set used to derive the example criterion continuous concentration. Supplementary data associated with this article can be found in the online version, at <https://doi.org/10.1016/j.scitotenv.2018.01.136>.

Data sets

Data sets and individual XCD results used to develop the B-C model are available at the U.S. EPA Environmental Dataset Gateway (Cormier 2017a,b).

The file “biosample69.csv” is a data set of environmental parameters for ecoregion 69 developed by excluding records lacking biology samples, lacking conductivity measurement, having pH ≤ 6 samples; samples dominated by chloride, i.e. $([\text{SO}_4^{2-}] + [\text{HCO}_3^-] < [\text{Cl}^-])$. It

is available from the U. S. EPA Environmental Dataset Gateway at <https://doi.org/10.23719/1371704> or directly from the distribution link <https://pasteur.epa.gov/uploads/A-k998/482/CMEC%20Data%20sets.zip>.

The file “eco69_dupchem.csv” includes station-years with at least 6 conductivity samples (one in the spring and one in the summer) from Ecoregion 69 in West Virginia, maximum conductivity, and number of sensitive taxa. It is available from the U.S. EPA Environmental Dataset Gateway at <https://doi.org/10.23719/1371704> or directly from the distribution link <https://pasteur.epa.gov/uploads/A-k998/482/CMEC%20Data%20sets.zip>.

The file “ss.csv” is a matrix of benthic invertebrate observations in Ecoregion 69. Developed by removing ambiguous taxa, and non-reference taxa in this ecoregion in the WV data set. It is available from the U.S. EPA Environmental Dataset Gateway at <https://doi.org/10.23719/1371707> or directly from the distribution link <https://pasteur.epa.gov/uploads/A-r4z3/527/Data%20Biological.zip>.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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Abbreviations

B-C	biological condition
CCC	criterion continuous concentration
CFD	cumulative frequency distribution
CMEC	criterion maximum exposure concentration
DO	dissolved oxygen
XCD₀₅	5th centile of XCD
LOWESS	LOcally WEighted Scatterplot Smoother
NO_x	total nitrates and nitrites

RBP	rapid bioassessment protocol
SC	specific conductivity
SD	standard deviation
TSS	total suspended solids
U.S. EPA	U.S. Environmental Protection Agency
WABbase	Watershed Assessment Branch database
WVDEP	West Virginia Department of Environmental Protection
XCD	extirpation concentration distribution
XCD₀₅	5th centile of XCD
XC₉₅	Extirpation concentration values at the 95th centile

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Highlights

- Chronic and acute exposure limits characterize freshwater invertebrate tolerances.
- An acute value can be estimated from a normal distribution of the exposure levels.
- This practical approach uses chemistry data with multiple within-year measurements.
- Example continuous and maximum criteria are derived for an ionic mixture.
- The method is applicable anywhere with a suitable data set.

Fig. 1. Ecoregion 69 extends from central Pennsylvania to northern Tennessee. Sampling sites (stations; $N=1420$) used to derive the criterion continuous concentration are indicated as points in West Virginia.

Fig. 2.

Main steps in the derivation of a criterion maximum exposure concentration (CMEC) based on field water chemistry data. Rectangular boxes on left are products and pentagonal boxes on right are operations performed to generate those products. A Locally WEighted Scatterplot Smoother (LOWESS) estimates a line for a scatter plot by iteratively calculating many nonparametric regression models using local approximations from neighboring points. Specific conductivity (SC); criterion continuous concentration (CCC); standard deviation (SD).

Modified from U.S. EPA (2016).

Fig. 3. Scatter plot of relationship between (Cl^-) and ($[\text{HCO}_3^-] + [\text{SO}_4^{2-}]$) in mg/L in streams in Ecoregion 69 data set from 1997 to 2010 where ionic measurements were available. The diagonal 1:1 line demarcates sites dominated by ($[\text{HCO}_3^-] + [\text{SO}_4^{2-}]$) from those dominated by (Cl^-) mixtures, that is, on a mass basis in mg/L, the diagonal 1:1 line plots where the ($[\text{HCO}_3^-] + [\text{SO}_4^{2-}] = (\text{Cl}^-)$). Sites (1.4%, $n = 938$) above the diagonal line were excluded from the data set shown in Table 1. Samples depicted here include all sites including low pH. From U.S. EPA (2016).

Fig. 4.

Histogram of the frequencies of observed specific conductivity values in samples from Ecoregion 69 sampled between 1997 and 2010 (Table 1). Bins are each 0.017 (1/60) of the range of log₁₀ specific conductivity units wide.

From U.S. EPA (2016).

Fig. 5.

Genus extirpation concentration distribution (XCD) for Ecoregion 69. Each point is an extirpation concentration (XC_{95}) value for a genus. There are 142 genera. The hazardous concentration (XCD_{05}) is 305 $\mu\text{S}/\text{cm}$ (95% confidence interval is 233–329 $\mu\text{S}/\text{cm}$) and is the specific conductivity at the intersection of the XCD with the horizontal line at the 5th centile. Open circles are XC_{95} values.

From U.S. EPA (2016).

Fig. 6.

Illustration of within site variability (standard deviation [SD] for each station) along the specific conductivity gradient (station geometric mean) in Ecoregion 69. The x -axis is the annual geometric mean specific conductivity (SC). Each dot represents a station. The Locally Weighted scatterplot smoothed (span = 0.75) fitted line shows a relatively constant SD between two vertical dashed lines bounding the logarithm mean station SC of 120 and 520 $\mu\text{S}/\text{cm}$. The mean SD was calculated from the total observations at these sample stations.

From U.S. EPA (2016).

Fig. 7.

Specific conductivity (SC) in multiple observations of a station in Morris Creek, WV. Julian day, 0=January 1, is on the x -axis. SC is on the y -axis with water chemistry samples as circles; the filled circle is the date of biological sampling. The horizontal dashed line is the calculated criterion continuous concentration of $310 \mu\text{S}/\text{cm}$. SC minimum (58), maximum (450), and on date of biological sampling ($75 \mu\text{S}/\text{cm}$). Two of the 7 most salt-intolerant genera (extirpation concentration values at the 95th centile [XC_{95}] $< 310 \mu\text{S}/\text{cm}$) were observed at this station.

From U.S. EPA (2016).

Fig. 8.

Scatter plot of count of 7 most salt-intolerant genera and annual maximum specific conductivity (SC) in preceding year. Very few salt-intolerant genera are observed at sites with a SC greater than the example criterion maximum exposure concentration of 630 $\mu\text{S}/\text{cm}$ (right of vertical dashed line). SC expressed as $\mu\text{S}/\text{cm}$. From U.S. EPA (2016).

Table 1.

Summary statistics of the measured water-quality parameters used to derive the specific conductivity criterion continuous concentration in Ecoregion 69.

Parameter	Units	Min	25 th	50 th	75 th	Max	Geomean	N
SC	µS/cm	15.4	94	229	540	3794	225	1661
Total Hardness	mg/L	2.18	28.03	64.31	132.7	1492	64.43	834
Total Alkalinity	mg/L	2	14	41	90	560	37	1144
SO ₄ ²⁻	mg/L	1	12	32	126	2097	39	1146
Cl ⁻	mg/L	0.5	2	3	8	650	4	930
SO ₄ ²⁻ +HCO ₃ ⁻	mg/L	8.66	36.3	99.4	252	2256	99.3	1142
Ca, total	mg/L	0.67	6.9	16.9	33.5	430	15.8	842
Mg, total	mg/L	0.5	2.4	5.0	12	204	5.6	832
Na, total	mg/L	0.5	1.8	3.5	13	423	5.2	166
K, total	mg/L	0.5	0.7	1.2	2.4	16	1.4	164
TSS	mg/L	1	3	3	5	80	4	1151
Fe, total	mg/L	0.02	0.09	0.18	0.38	4.9	0.19	1170
Fe, dissolved	mg/L	0.01	0.02	0.03	0.07	1.1	0.04	995
Al, total	mg/L	0.01	0.06	0.1	0.19	3.3	0.11	1142
Al, dissolved	mg/L	0.01	0.02	0.03	0.06	0.9	0.04	1007
Mn, total	mg/L	0.003	0.02	0.03	0.06	4.4	0.03	1142
Se, total	mg/L	0	0.001	0.001	0.003	1.3	0.002	665
DO	mg/L	2.06	8.47	9.27	10.2	17.1	9.41	1644
Total Phosphorus	mg/L	0.01	0.02	0.02	0.02	1.3	0.02	897
NO _x	mg/L	0.01	0.14	0.28	0.45	11	0.26	910
Fecal Coliform	Counts/100 mL	0.5	15	65	300	250000	71	1405
pH	SU	6.01	7.00	7.54	7.97	10.48	7.48	1661
Catchment Area	km ²	0.34	4.36	17.6	65.2	17986	19.3	1408
Temperature	°C	-0.28	14.2	17.9	20.7	30.2	17.5	1661
RBP 10Sc	RBP score	53	126	142	156	195	140	1641
RBP 7Sc	RBP score	30	84	98	110	137	97	1647
Embeddedness	RBP score	1	11	13	16	20	13	1649
Percentage Fines (sand+silt)	-	0	10	12	20	100	15	1620

The data set has 1661 samples from 1420 stations. All means are geometric means except pH, dissolved oxygen (DO), Temperature, and Habitat Scores. RBP = rapid bioassessment protocol (Barbour et al., 1999); RBP 10Sc has 10 parameters, best possible habitat score is 200. RBP 7Sc is similar to RBP 10Sc but does not include 3 flow-related parameters, best possible habitat score is 140; NO_x = total nitrates and nitrites; SC = specific conductivity; SO₄²⁻ = sulfate; HCO₃⁻ = bicarbonate; Ca = calcium; Mg = magnesium; Na = sodium; K = potassium; TSS = total suspended solids; Fe = iron; Al = aluminum; Mn = manganese; Se = selenium.