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DRAFT

Ambient Aquatic Life Criteria For Chloride

I. Background

Chloride is one of the major anions commonly found in ambient and wastewater. Chlorides are not usually harmful to people; however, the sodium part of table salt (i.e. sodium chloride, NaCl) has been linked to heart and kidney disease. Sodium chloride may impart a salty taste at 250 mg/l; however, calcium or magnesium chloride is not usually detected by taste until levels of 1000 mg/l are reached.

Chloride may get into surface water from several sources including:

(1) rocks containing chlorides;
(2) agricultural runoff;
(3) wastewater from industries;
(4) produced water from oil and gas wells;
(5) effluent wastewater from wastewater and water treatment plants, and;
(6) road salting.

Chlorides can corrode metals and affect the taste of food products. Therefore, water that is used in industry or processed for any use has a recommended maximum chloride level. At elevated concentrations, chlorides can contaminate fresh water streams and lakes. Fish and aquatic communities cannot survive in high levels of chlorides.

The current Iowa water quality standard for chloride is 250 mg/l for drinking water use only. Currently, there are no numerical chloride standards for aquatic life protection in Iowa. However, as part of the current interim site-specific TDS approach, if in-stream chloride concentrations reach a threshold level (in-stream threshold values: acute threshold is 860 mg/l, chronic threshold is 230 mg/l), Whole Effluent Toxicity tests are required. These threshold values are equivalent to EPA's 1988 304(a) national criteria values.

II. National Criterion

The national criterion for chloride was derived based on the toxicity test data of sodium chloride in laboratory reconstituted water since it is the only chloride composition with enough data available to allow derivation of a water quality criterion. Also, it seems likely that most anthropogenic chloride in ambient water is associated with sodium,

rather than potassium, calcium, or magnesium (EPA, 1988). In the EPA 304(a) criteria document, the acute toxicity data of chloride are available for 12 different species (genus). The following national criteria for Chloride are recommended by the EPA (EPA, 1988).

Table 1. National Aquatic Life Criteria for Chloride

Parameter	National Criteria (mg/l)	
	Acute	Chronic
Chloride	860	230

For acute toxicity to aquatic life, invertebrates are generally more sensitive than vertebrates. Cladoceran (Daphnia pulex) is the most sensitive among the tested species with a Species Mean Acute Value of 1470 mg/l. Using the methodology in the EPA Guidelines (1985), the four most sensitive genus mean acute values including Cladoceran, Snail, Isopod, and Midge were used in the acute criterion derivation (Appendix A). The Final Acute Value (FAV) is 1720 mg/l. The Final Acute Value is calculated using the LC50 values of tested species.

Instead of being equal to the Final Acute Value, the acute criterion is set at one-half the Final Acute Value. The acute criterion is intended to protect 95 percent of a group of diverse genera, unless a commercially or recreationally important species is very sensitive. However, a concentration that would harm 50 percent of the fifth percentile or 50 percent of a sensitive important species cannot be considered to be protective of that percentile or that species. Dividing the Final Acute Value by 2 is intended to result in a concentration that will not adversely affect the sensitive organisms.

The national chronic criterion for chloride was derived based on three acute-to-chronic ratios. The acute-to-chronic ratio (ACR) is the ratio of the acute toxicity of a toxicant to its chronic toxicity. It is often used as a factor for estimating chronic toxicity on the basis of acute toxicity data since chronic toxicity data are often limited. The three acute-to-chronic ratios are listed in Table 2.

Table 2. Acute-to-Chronic Ratio (ACR) for Deriving Chronic Chloride Criterion

Species	Hardness (mg/l as CaCO3)	Acute Value (mg/l)	Chronic Value (mg/l)	Ratio	Reference
Cladoceran Daphnia pulex	100	1,470	372.1	3.951	Birge et al. 1985
Rainbow trout Salmo girdneri	46	6,743	922.7	7.308	Spehar (1987)
Fathead minnow Pimephales promelas	100	6,570	433.1	15.17	Birge et al. (1985)
				Final ACR=7.594, as the geometric mean of the three ACRs	

Using the Acute-Chronic Ratio value of 7.594, the calculated chronic criterion for Chloride would be 226.5 mg/l. The EPA criteria document for chloride rounded up the value to 230 mg-Cl/L. This value is substantially lower than all three chronic values used for the calculation as shown in Table 2.

Since the EPA national criteria were published in 1988, the derivation of the criteria was based on toxicity data available before 1987.

III. New Toxicity Information

Field Data:

Birge et al. (1985) whose toxicity data were used in the U.S. EPA (1988) chloride criteria document, conducted field toxicity tests on chloride. Birge et al (1985) found that chloride was twice as toxic using standard laboratory water, as opposed to natural water (or from field studies). The study recommended the following aquatic life criteria for chloride:

In each 30 consecutive days:
a. The average concentration of chloride should not exceed 600 mg/l;
b. The maximum concentrations should not exceed 1200 mg/l; and
c. The concentration may be between 600 mg/l and 1200 mg/l for up to 48 hours.

These values had been adopted by the Kentucky Natural Resources and Environmental Protection Cabinet. The chronic criterion 600 mg/l for chloride is close to the chloride value of 500 mg/l promulgated by the Illinois Pollution Control Board to regulate general stream quality in the state.

This information could be used to develop site-specific criteria based on the water-effect ratio method. The water-effect ratio is an appropriate measure of the toxicity of a material obtained in site water divided by the same measure of the toxicity of the same material obtained simultaneously in lab dilution water. However, since the field toxicity data were collected in streams of State of Kentucky, the information may not be applicable to Iowa waters.

The Wisconsin State Laboratory of Hygiene (WISLOH) has been conducting routine toxicity testing with Ceriodaphnia dubia, Daphnia magna, and the fathead minnow since the year of 2000. The toxicity data were requested and sent to IDNR through e-mail by Steve Geis of WISLOH. Appendix B includes a summary of the data analysis and different methodologies used to eliminate outliers in the data. Table 3 shows the calculated mean values for each species.

Table 3. Chloride Toxicity Test Data from State of Wisconsin

Species	Acute Toxicity	Chronic Toxicity	Acute-Chronic Ratio
Fathead Minnows	4175	1747	2.39
Daphnia Magna	4060	None	None
Ceriodaphnia dubia	1572	703	3.68

Acute toxicity tests for Daphnia magna have also been conducted by Hoke R.A. *et al.* (1992), Mount and Anderson-Carnahan (1988) and Arambasic *et al.* (1995). The toxicity data are shown in Table 4.

Table 4. Additional Acute Toxicity Test Data for Daphnia magna

Genus Species	Toxicity Value	Toxicity Value	Endpoint	Data Sources
Daphnia Magna	NaCl (mmol/l)	Mean Cl(mg/l)		
	85.7	3042.35	48-hour LC50	Hoke R.A. *et al.* (1992)
	76.9	2729.95	48-hour LC50	Mount and Anderson-Carnahan (1988)
	81.2	4750	48-hour LC50	Arambasic *et al.* (1995)

In order to provide a predictive tool to assess toxicity attributable to major ions, Mount *et al.* (1997) tested the toxicity of over 2,900 ion solutions using the daphnids (*Ceriodaphnia dubia* and *Daphnia magna)*, and fathead minnows. Newman and Aplin (1992) conducted survival time modeling using acute NaCl toxicity data for mosquitofish, *Gambusia holbrooki*. The toxicity test results for these species are shown in Table 5.

Table 5. Acute Toxicity Data from Mount *et al.* (1997)

Species Name	**Toxicity Value**	**Toxicity Value**	**Endpoint**	**Data Sources**
	NaCl (mg/l)	**Chloride (mg/l)**		
Fathead Minnow	6,390	3,878	96 hour LC50	Mount *et al*., 1997
Daphnia Magna	4,770	2,895	48 hour LC50	Mount *et al*., 1997
Ceriodaphnia dubia	1,960	1,189	48 hour LC50	Mount *et al*., 1997
Mosquitofish	11,540	7,003	96 hour LC50	Newman and Aplin (1992)

IV. Chloride Criteria Recalculation Using New Toxicity Data

The additional toxicity data shown in Tables 3 through 5 have been combined with the EPA national toxicity database provided in the 1988 chloride criteria document to recalculate both the acute and chronic criteria for chloride.

A. Acute Criteria Recalculation

First, the genus mean acute values (as the geometric mean of the toxicity data for each species) are calculated for the three genuses that have new toxicity data, *Daphnia spp*., *Fathead minnow*, and *Ceriodaphnia spp*. The recalculated genus mean acute values for the three species are shown in Table 6.

Table 6. Recalculated Genus Mean Acute Values

Genus Species	**Species**	LC50, (mg/l)	SMAVs*	GMAVs**	Data Sources
Ceriodaphnia dubia		1572	1367	**1367**	WISLOH, (2007)
		1189			Mount et al., (1997)
Daphnia spp.				**2070**	
	Daphnia magna	2895	2915		Mount et al., (1997)
		2875			Arambasic et al. 1995
		4060			WISLOH, (2007)
		2024			EPA 1988 -Dowden (1961)
		3583			EPA 1988 -Dowden & Bennett, (1965)
		3042.35			Hoke R.A. et al. (1992)
		2729.95			Mount and Anderson-Carnahan (1988)
		2565			EPA - 1988- Biesinger & Christensen, (1972)
	Daphnia pulex	1470	1470		EPA - 1988 - Birge et al., (1985)
Fathead Minnow		4175		**4738**	WISLOH, (2007)
		3878			Mount et al., (1997)
		6570			EPA - 1988

* SMAV = Species Mean Acute Value; ** GMAVs = Genus Mean Acute Value

Second, the newly calculated Genus Mean Acute Values for the three species and the acute value for mosquitofish (*Gambusia holbrooki*) are inserted into the available toxicity database in the chloride criteria document as shown in Appendix A, Table A1; Finally, the Final Acute Value is recalculated. To ensure the acute criterion is protective of most of the organisms, the acute criterion is calculated as the one half of the Final Acute Value.

B. Chronic Criteria Recalculation

Since not enough chronic toxicity data are available to meet minimum data requirements, the chronic criterion is calculated dividing the Final Acute Value by the Acute-Chronic Ratio (ACR). The ACRs are available for four species, Fathead minnow, Rainbow trout, *Ceridaphnia dubia*, and *Daphnia pulex*. They are shown in Table 7.

Table 7. Acute-to-Chronic Ratio (ACR) for Deriving Chronic Chloride Criterion

Species	Acute Value (mg/l)	Chronic Value (mg/l)	ACR Ratio	Reference
Cladoceran ***Ceriodaphnia dubia*	1572	427	3.68	**WISLOH, (2007)**
Cladoceran *Daphnia pulex*	1,470	372.1	3.951	EPA, 1988 (Birge et al. 1985)
Rainbow trout Salmo girdneri	6,743	922.7	7.308	EPA, 1988 (Spehar 1987)
Fathead minnow Pimephales promelas	6,570	433.1	15.17	EPA, 1988 (Birge et al. 1985)
Fathead minnow Pimephales promelas	4,175	1747	2.39	**WISLOH, (2007)**

Two new ACRs have been calculated based on Wisconsin Lab data as shown in bold in Table 7, one for *Ceriodaphnia dubia* and another for Fathead minnow. The newly calculated ACR for Fathead minnow based on Wisconsin data is much lower than the value derived by Birge et al. (1985) that was provided in the EPA 1988 chloride criteria document. It seems that the relatively low chronic value of 433.1 mg/l from Birge et al. (1985) resulted in the high ACR. Staff evaluation of the new aquatic toxicity data concluded that the Wisconsin State Laboratory of Hygiene (Aquatic Life Toxicity Testing Laboratory) provided defensible acute and chronic chloride toxicity testing results for both the fathead minnow and ceriodaphnia following the basic approaches published by EPA and ASTM.

In addition, the EPA's National Exposure Research Laboratory in Cincinnati, Ohio, developed defensible chronic chloride aquatic life toxicity data (Pickering, 1996) for the fathead minnow using Sodium Chloride, which produced a chronic chloride value of 3459 mg/l that is much higher than the chronic value produced by Birge et al. (1985).

Thus, the ACR derived based on Wisconsin Lab data was used to replace the ACR for fathead minnow derived by Birge et al. (1985). The final ACR is calculated as the geometric mean of the available ACRs for different species. Since Rainbow trout is a cold water species, the final ACR for warmwater designation did not include the ACR for Rainbow trout. The final ACR calculation for coldwater water bodies includes the ACR

for Rainbow trout. Thus, the ACR for coldwater designation and warmwater designation have been calculated as 3.99 and 3.26, respectively.

Table 8 shows the overall calculation for both the acute and chronic criteria using the EPA Guidelines for developing site specific criteria (US EPA, 1985).

Table 8. Chloride Criteria Recalculation

Rank	Organisms	GMAVs*	P=R/(N+1) N=13	Ln(GMAV)	[Ln(GMAV)]^2	SQRT(P)
14	American eel	11940				
13	Goldfish	8906				
12	Mosquitofish	7002				
11	Rainbow trout	6743				
10	Mosquito	6222				
9	Bluegill	5870				
8	Midge	4900				
7	Fathead Minnow	4738				
6	Caddisfly	4039				
5	Midge	3795				
4	Isopod	2950	0.2667	7.9896	63.8331	0.516398
3	Snail(Physa gyrina)	2540	0.2000	7.8399	61.4643	0.447214
2	Daphnia spp.	2070	0.1333	7.6354	58.2993	0.365148
1	Ceriodaphnia dubia	1367	0.0667	7.2206	52.1365	0.258199
Sum			0.6667	30.6854	235.7332	1.5870

$$S^2 = \frac{235.7332 - \frac{30.6854^2}{4}}{0.6667 - \frac{1.5870^2}{4}} = 9.0178$$

$$S = 3.003$$

$$L = \frac{30.6854 - 3.003 \times 1.5870}{4} = 6.480$$

$$A = 3.003 \times \sqrt{0.05} + 6.480 = 7.1514$$

$FAV = e^{7.1284} = 1276$ mg/l (Final Acute Value)

Criteria for Warmwater Designation:

$$Acute\,Criterion = \frac{1276}{2} = 638\,mg/l$$

$$Chronic\,Criteiron = \frac{1276}{3.26} = 391\,mg/l \Rightarrow lowered\ to\ 372\,mg/l\ to\ protect\ Daphnia\ pulex$$

Criteria for Coldwater Designation:

$$Acute\,Criterion = \frac{1276}{2} = 638\,mg/l$$

$$Chronic\,Criteiron = \frac{1276}{3.99} = 320\,mg/l$$

*Bolded values are recalculated using recent toxicity information

The terms shown in Table 8 are defined as follows:

GMAVs are the Genus Mean Acute Values for different species;

P is the calculated ranking;
Ln(GMAV) is the natural log of the GMAV;
SQRT(P) is the square root of the ranking.

V. Recalculated Criteria for Chloride

Based on the recalculation results, the following criteria are derived:

For Warmwater Designated Streams and Lakes and Wetlands:

Acute Criterion = 638 mg/l
Chronic Criterion = 372 mg/l

For Coldwater Designated Waters:

Acute Criterion = 638 mg/l
Chronic Criterion = 320 mg/l

It is important to point out that IDNR staff is continuing to search for new toxicity data that can be considered in the recalculation. As new toxicity data are found, the recommended criteria may be revised.

The EPA recommended acute value of 860 mg/l was calculated to protect all aquatic life in the nation from acute toxicity of chloride. While this information is dated (1988) and new toxicity information is available, this value is EPA approvable. Many of EPA's 304a criteria are dated and the department is not aware of any current efforts from EPA to update the 304a criteria for chloride though new scientific data is available. The department is uncertain at this point if the data used in these recalculations would meet EPA guidelines for inclusion into 304a criteria considerations. The issue is whether or not to include this new data for acute toxicity of chloride which results in criteria more stringent than 304a criteria knowing that the 860 mg/l value is protective of all aquatic life and EPA approvable. The department included the data in this calculation for consistency in the recalculation process.

Appendix A: Toxicity Data Used for the National Criteria

Table A1. Ranked Genus Mean Acute Values with Species Mean Acute-Chronic Ratios

Rank [a]	Genus Mean Acute Value (mg/l)	Species	Species Mean Acute Value (mg/l)	Species Mean Acute-Chronic Ratio
Freshwater Species				
12	11,940	American eel. Anquilla rostrata	11,940	--
11	8,906	Goldfish Carassius auratus	8,906	--
10	6,743	Rainbow trout Salmo gairdneri	6,743	7.308
9	6,570	Fathead minnow Pimephales promelas	6,570	15.17
8	6,222	Mosquito Culex sp.	5,222	--
7	5,870	Bluegill Lepomis macrochirus	5,870	--
6	4,900	Midge Chironomus attenuatus	4,900	--
5	4,039	Caddisfly Hydroptila angusta	4,039	--
4	3,795	Midge Cricotopus trifasciatus	3,795	--
3	2,950	Isopod Lireus Fontinolis	2,950	--
2	2,540	Snail Phyto gyrina	2,540	--
1	1,974	Cladocera Daphnia magna	2,650	--
		Cladocera Daphnia pulex	1,470	3.951

a. Ranked from most resistant to most sensitive based on Genus Mean Acute Value.

Final Acute Value = 1.720 mg/l
Criterion Maximum Concentration = (1720 mg/l)/2 = 860.0 mg/l

Final Acute-Chronic Ratio = 7.594 (see text)
Final Chronic Value = (1.720 mg/l)/7.594 = 226.5 mg/l

Table A2. Acute – Chronic Ratio

Species	Hardness (as CaCO3) (mg/l)	Acute Value (mg/l)	Chronic Value (mg/l)	Ratio
Cladocera				
Daphnia pulex	100	1,470	372.1	3.951
Rainbow trout				
Salmo gairdneri	46	5,743	922.7	7.308
Fathead minnow				
Pimephales promelas	100	6,570	433.1	15.17

Appendix B: Wisconsin WET test Data Trimming Summary

WI State Laboratory of Hygiene (WISLOH) accumulated a set of WET test data over a period of more than 6 years starting from October 2000 to January 2006. As with any experimental data, there could be outliners. It is import to identify the outliers and remove them before any statistic analysis on the data. Four different commonly used methods were employed to identify the outliers. The dataset after the removal of the outliers was compared to the dataset with the outliers to determine if the four data trimming methods resulted in a significant difference in the dataset.

1. Dataset description and outlier identification methods introduction.

Using standard synthetic water, WISLOH performed both acute and chronic WET tests on two organisms, Fathead Minnow and Ceriodaphnia dubia. Another small dataset, an acute WET test on Daphnia Magna, collected from January 1995 to February 2004 was also available. Table 1 summarizes the data points.

Table B1. Data Points from WI State Laboratory of Hygiene

Organism	Test Type	Total data points	Collecting time period
Fathead Minnow	Acute	66	Nov. 00 – Jan, 07
	Chronic	64	Oct. 00 – Dec. 06
Ceriodaphnia dubia	Acute	68	Nov. 00 – Jan, 07
	Chronic	66	Nov. 00 – Jan, 07
Daphnia Magna	Acute	16	Jan. 95 – Feb. 04

As shown in Table B1, the WET test dataset can be broken down to 5 datasets. Data in each dataset are results of repeated experiments. Therefore, they are expected to have a normal distribution. Four commonly used outlier identification methods were used to trim these 5 datasets in this study. The four methods are:

A. Interquartile Range (IQR) method
   This is one of the most commonly used method for identify outliers. The inter-quartile is defined as the difference between the 75% percentile (upper quartile) and the 25% percentile (lower quartile) of the dataset. Any data in the dataset that are greater than upper quartile + 1.5 interquartile or smaller than lower quartile – 1.5 interquartile are considered outliers.

B. Z-test method
   This method requires normal distribution. Use the following equation to calculate Z-value:

$$Z = \text{abs}(\text{data value} - \text{mean}) / \text{s.d.}$$

   and compare the Z value with the Z value in Table B2. If the calculated Z value is greater than the critical Z value in the table, there is more than a 5% chance of

encountering an outlier (in either direction), if the dataset truly has a normal distribution.

Table B2. Critical Z values at P=0.05 level

Number of data in a dataset	Critical Z value
16	2.59
50	3.13
60	3.20
70	3.26

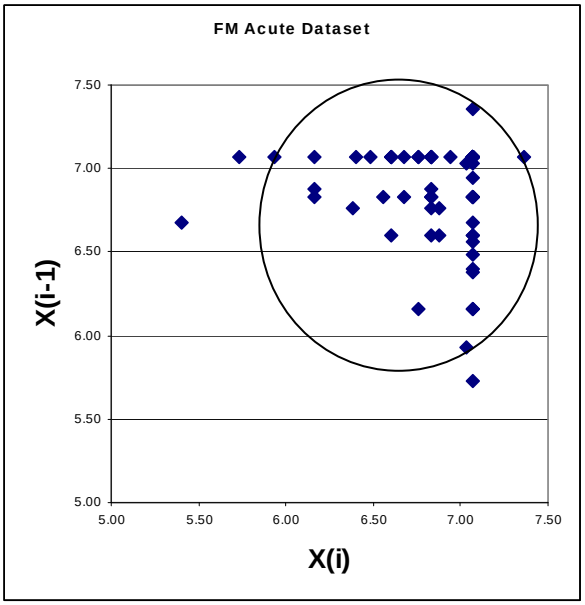
C. Distance from Mean: 3.0*s.d.
This method requires normal distribution. Any data that are greater than mean+3.0*s.d. or smaller than mean-3.0s.d. are considered outliers. 5% of the values in a normal distributed population are more than 1.96 standard deviations from the mean. Data that are more than 3.0 standard deviations from the mean have more than 95% percent of the chance to be outliers.

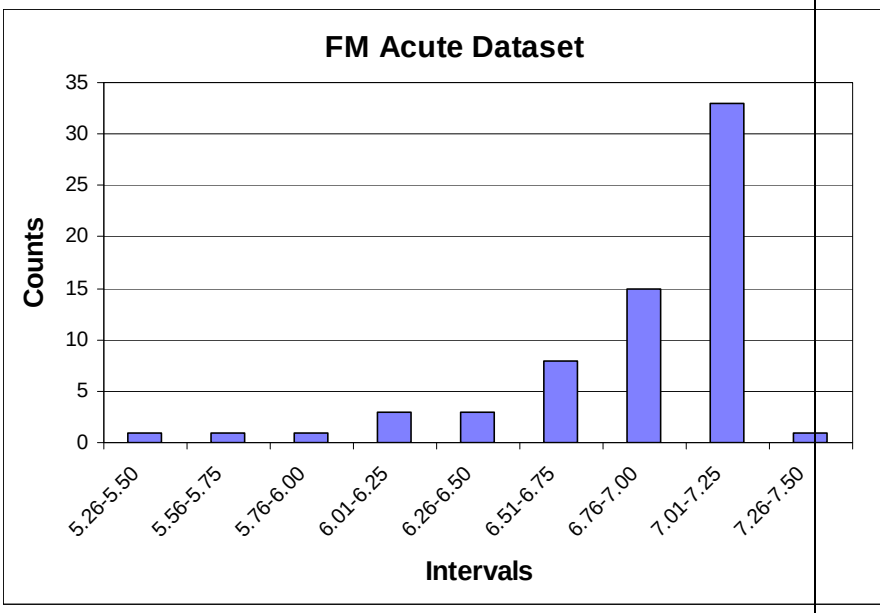
D. 5% data points from both ends
This method requires normal distribution.

2. Summary of difference methods used on the 5 datasets

A. Fathead Minnow acute WET data

Since some of the methods can only be applied to datasets that have normal distribution. Lag and histograms plots were prepared to visually analyze the distribution of the dataset.





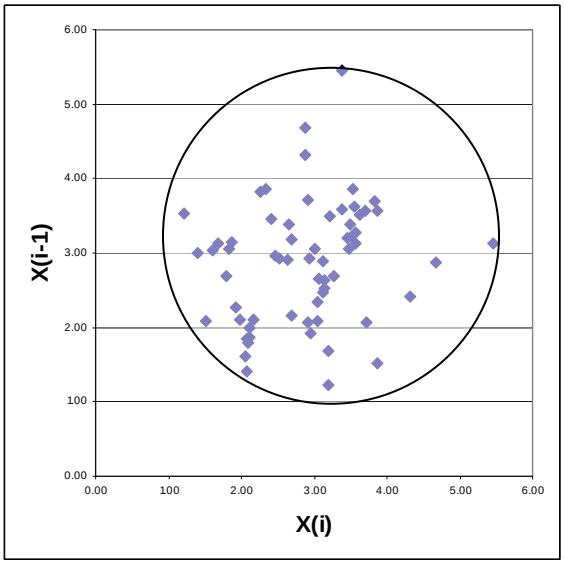
If the dataset has normal distribution, the lag data should be random and the histogram should be bell-shaped. Apparently, this dataset does not have a normal distribution. Therefore, the application of the Z-test and Mean±3.0 s.d are not legitimate. However, for comparison purposes, the results of the two methods were listed in the summary table B3.

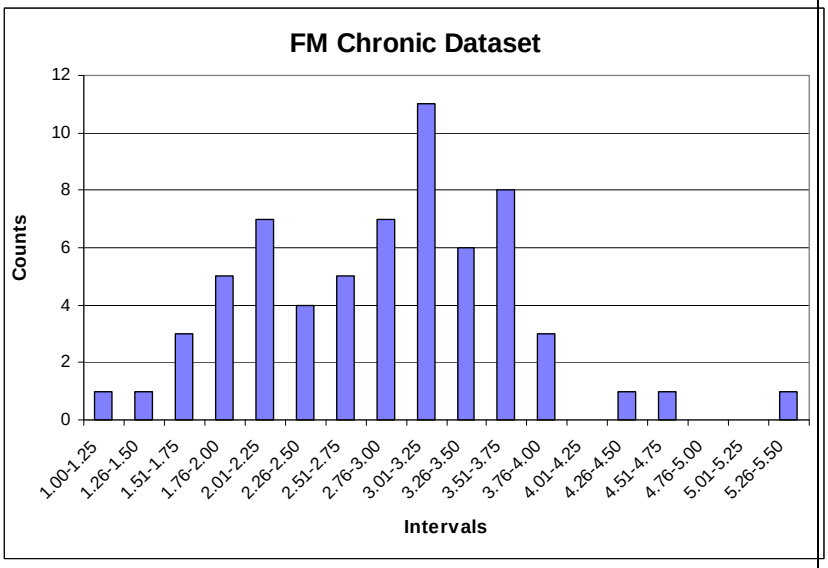
Table B3. Comparison of different trimming methods on FM acute dataset

	Original Dataset	IQR	Z-test	mean±3.0 s.d.	5% data points removal
Data points	66	63	66	64	60
mean	6.83	6.88	6.83	6.87	6.87
s.d	0.36	0.26	0.36	0.29	0.26
c.v.	5%	4%	5%	4%	4%

Suggestion: remove 3 points according to IQR method.

B. Fathead Minnow chronic WET data





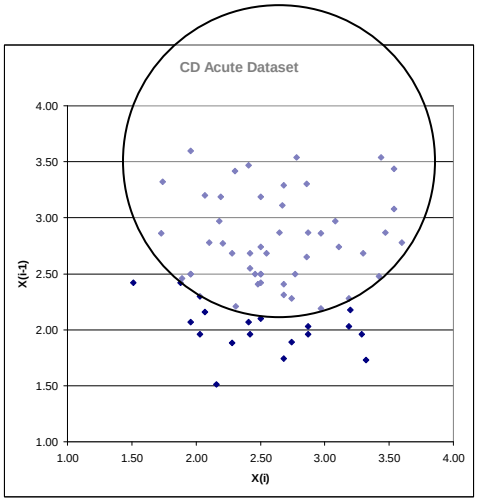
The plots show that the dataset has normal distribution with no outliers.

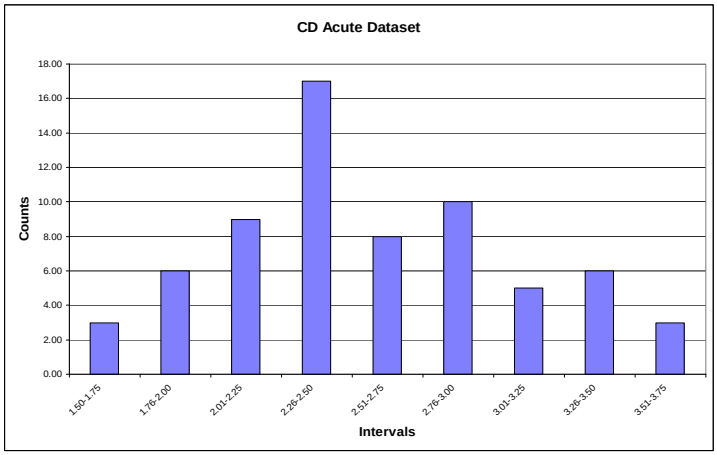
Suggestion: No data points should be removed.

Table B4. Comparison of different trimming methods on FM chronic dataset

	Original Dataset	IQR	Z-test	mean±3.0 s.d.	5% data points removal
Data points	64	63	64	63	58
mean	2.88	2.84	2.88	2.84	2.86
s.d	0.82	0.75	0.82	0.75	0.64
c.v.	28%	27%	28%	27%	22%

C. Ceriodaphnia dubia acute WET data





The plots show that the dataset has normal distribution with no apparent outliers.

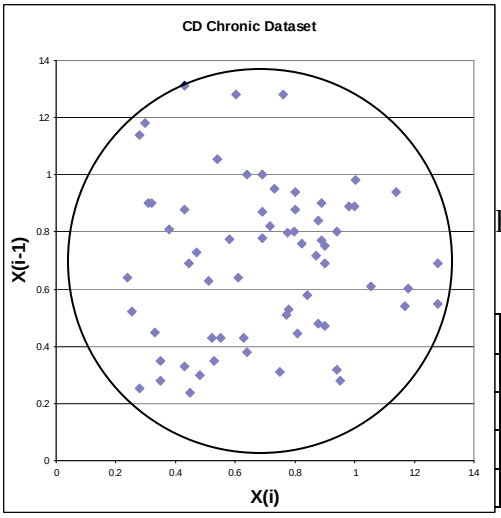
Table B5. Comparison of different trimming methods on Ceriodaphnia Acute dataset

	Original Dataset	IQR	Z-test	mean±3.0 s.d.	5% data points removal
Data points	68	68	68	68	62
mean	2.59	2.59	2.59	2.59	2.59
s.d	0.5	0.5	0.5	0.5	0.43
c.v.	19%	19%	19%	19%	17%

It is safe to say that there are no outliers in this dataset.

Suggestion: No data points should be removed.

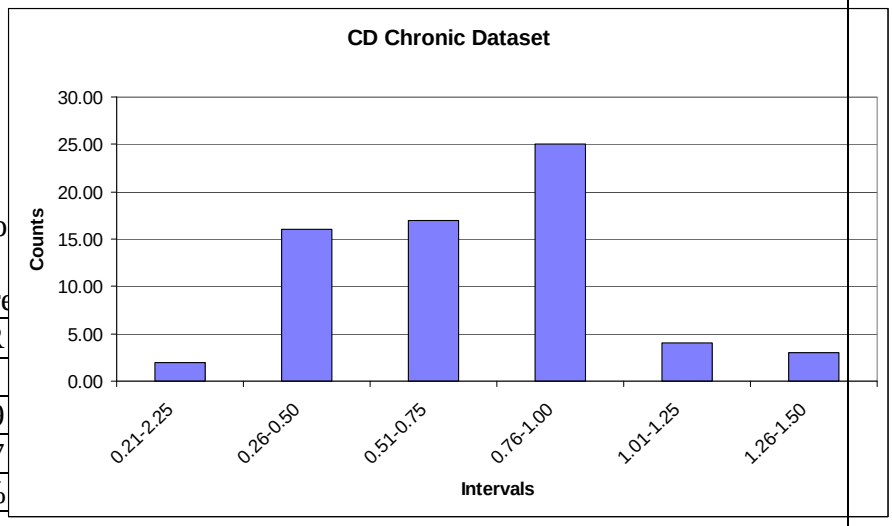
D. Ceriodaphnia dubia chronic WET data



has no

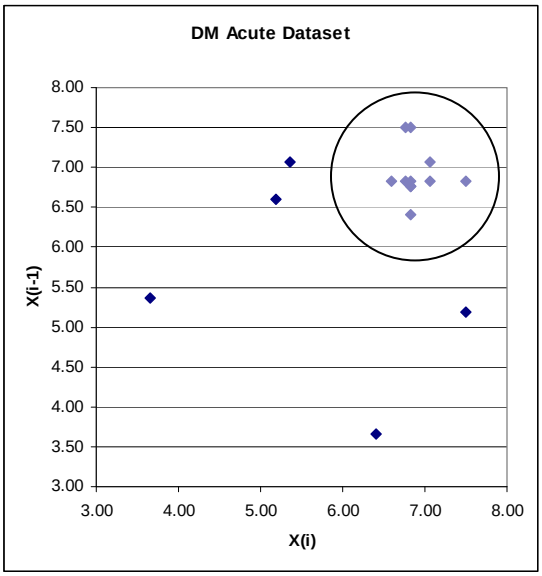
differe

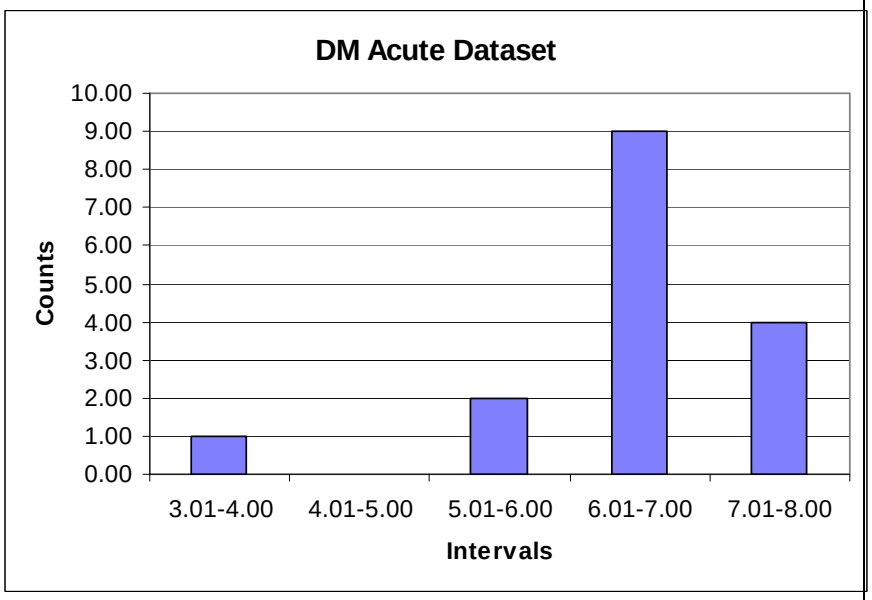
IQR
65
0.69
0.27
39%



Suggestion: no data point should be removed.

E. Daphnia Magna acute WET data





The plots show that the dataset does not have normal distribution. From the histogram, one point could be removed and the rest of the data could still have a normal-like distribution.

Table B6. Comparison of different trimming methods on DM chronic dataset

	Original Dataset	IQR	Z-test	mean±3.0 s.d.	5% data removal	Visual removal
Data points	16	11	15	16	14	15
mean	6.50	6.80	6.69	6.50	6.63	6.69
s.d	0.95	0.18	0.62	0.95	0.61	0.62
c.v.	15%	2.6%	9.3%	15%	9.1%	9.3%

Suggestion: Remove one point according to visual observation.

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