

*Public Review Draft*

**NJ Drinking Water Quality Institute (DWQI)**

**Testing Subcommittee**

**Report on the Development of a Practical Quantitation Limit (PQL)**

**1,2,3-Trichloropropane**

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## **Introduction**

In 2009, the Drinking Water Quality Institute (DWQI) recommended a Practical Quantitation Limit (PQL) to the New Jersey Department of Environmental Protection (NJDEP) for 1,2,3-trichloropropane (123TCP) of 0.03 µg/L. The health-based MCL for 123TCP recommended by the DWQI was 0.0013 µg/L, however, this value was not attainable for quantitation using the US Environmental Protection Agency (EPA) approved drinking water method 504.1. EPA method 504.1 was the most sensitive analytical method available for analyzing 123TCP at that time. The analytical capability was the limiting factor in the MCL recommendation for 123TCP.

The PQL of 0.03 µg/L recommended for 123TCP in the “New Jersey Drinking Water Quality Institute MCL Recommendations for Hazardous Contaminants in Drinking Water (2009)” document was reviewed to determine whether this value remains the most appropriate PQL for 123TCP.

A review of reporting limits and MDLs for 123TCP in the analysis of New Jersey drinking water compliance samples submitted since 2009 together with current EPA approved drinking water analytical methods and performance data demonstrated that a PQL of 0.03 µg/L is still appropriate.

## **PQL Review**

In conducting this review, the Testing Subcommittee considered the following:

- Additional EPA analytical methods not available in 2009, such as EPA 524.3
- EPA 504.1 data from laboratories analyzing synthetic organic compounds (SOCs) compliance samples for NJ public water systems, since 2009
- Minimum reporting limits (RL) of laboratories using EPA 504.1 and EPA 524.3
- Method Detection Limits (MDLs) of laboratories using EPA 504.1 and EPA 524.3
- Determination of PQL value using median MDL x 5
- Determination of PQL value using average of minimum reporting limits
- Determination of PQL value using median of minimum reporting limits
- Bootstrap analysis of MDL and RL data

Since the EPA and the NJDEP have not promulgated an MCL for 123TCP, public water systems are not required to routinely monitor for this contaminant. However, the National Primary Drinking Water Regulations (NPDWR) require certain public water systems to monitor for a list of 30 regulated SOCs, and through these regulations, the NJDEP has been able to obtain occurrence data for 123TCP.

The NPDWR specify that EPA methods 504.1, EPA 551.1, or EPA 524.3 [40 CFR 141.24(e)(1) and Subpart C Appendix A] are acceptable for two regulated and related SOCs - ethylene dibromide (EDB) and dibromochloropropane (DBCP) – and 123TCP is a target analyte in each of these methods.

EPA method 524.2 also includes EBD, DBCP and 123TCP as target analytes, however, this method cannot achieve the published detection limits for EDB and DBCP [40 CFR 141.24(h)(18)]. Therefore, the MDLs and RLs for this method were not considered in 2009 and will not be used in this analysis.

In NJ, the NJDEP collects samples for SOCs every three years that are analyzed at the NJDOH laboratory. The NJDEP requires that all analyses of EDB and DBCP using EPA 504.1 include 123TCP as a target analyte. Since EPA 551.1 has not been used in NJ to monitor for EDB, DBCP and 123TCP, this method was not used for PQL development in 2009, and also will not be used in this analysis.

If the EPA approves an analytical method to be used for regulatory data, the NJDEP Office of Quality Assurance (OQA) includes that method in their list of Department Sanctioned Analytical Methods (DSAMs). The laboratory must request certification for each parameter within that DSAM in order to report compliance data for an analyte. An analyte that does not appear in the NJDEP Application for Certification of Environmental Measurements Part III - Analytical Testing Parameters list does not mean that New Jersey certification is unavailable for that analyte. If a target analyte analysis is approved for a method, the laboratory may request certification for that analyte from the NJDEP OQA.

There are 15 laboratories certified by NJDEP OQA for 123TCP by EPA 504.1 for drinking water compliance samples. Only three of these have reported 123TCP data to NJDEP using EPA 504.1. These laboratories analyzed samples for NJDEP, or were contracted by those water systems which were required to monitor for EDB or DBCP, or conducted monitoring for water systems with detections of 123TCP at the request of the NJDEP.

Laboratories analyzing EDB and DBPC for compliance purposes must meet the regulatory detection limits for EDB and DBCP [40 CFR 141.24(h)(18)]. Since 123TCP is not a regulated contaminant and an MDL cannot be enforced, the NJDEP recommended MDL of 0.005 µg/L is not always met. Some laboratories have admittedly not tried to generate 123TCP MDLs and RLs as low as possible.

The 15 laboratories that are currently certified to report 123TCP drinking water compliance data using EPA 504.1 were asked for their MDL and their reporting limits. The information appears in Table 1.

At the time the 2009 DWQI MCL Recommendation Document was written, the EPA was developing the EPA 524.3, "Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry." EPA 524.3 Version 1.0 was finalized in June 2009. Among the list of 76 target analytes are the regulated volatile organic compounds (VOCs) and the SOCs EDB, DBCP and 123TCP. Subsequently the EPA, included method 524.3 version 1.0 in 40 CFR 141 Subpart C Appendix A as an additional approved method for the analysis of EDB and DBCP. The inclusion of EPA 524.3 was made possible under the Expedited Approval of Alternative Test Procedures for the Analysis of Contaminants Rule. Unlike EPA 524.2, the regulatory detection limits for EDB and DBCP could be met with EPA 524.3 in Selected Ion Monitoring (SIM) mode because SIM provides the additional sensitivity needed to reach these detection limits.

**Table 1.**  
**MDLs and Reporting Limits from Laboratories Certified by NJ OQA for 123TCP**  
**EPA 504.1**

<b>Lab Name</b>	<b>NJ Lab ID</b>	<b>State</b>	<b>MDL (µg/L)</b>	<b>Reporting Limit (µg/L)</b>
EMSL ANALYTICAL INC	03036	NJ	0.0062	0.02
CAPE ENVIRONMENTAL LABORATORY	05679	NJ	0.0047	0.025
NEW JERSEY DEPARTMENT OF HEALTH	11036	NJ	0.002	0.02
ACCUTEST LABORATORIES	12129	NJ	0.015	0.02
INTEGRATED ANALYTICAL LABORATORIES	14751	NJ	0.009	0.0286
PRECISION ANALYTICAL SERVICES INC	15001	NJ	0.006	0.0114
BRICK TOWNSHIP MUNICIPAL UTILITIES AUTHORITY	15006	NJ	0.06	0.25
EUROFINS EATON ANALYTICAL, INC	CA008	CA	0.011	0.04
SUMMIT ENVIRONMENTAL TECHNOLOGIES, INC.	OH006	OH	0.09	0.1
AQUA PENNSYLVANIA INC	PA007	PA	0.006	0.0625
ALS ENVIRONMENTAL - MIDDLETOWN	PA010	PA	0.01	0.02
EUROFINS LANCASTER LABS ENVIRONMENTAL	PA011	PA	0.0143	0.0429
EUROFINS QC, INC.	PA166	PA	0.0046	0.0286
TESTAMERICA LABORATORIES INC. BURLINGTON	VT972	VT	0.0038	0.02
EDGE ANALYTICAL INC	WA013	WA	0.01	0.02

During the development of the 2009 PQL document, EPA 524.3 was a draft, not final, method. In July 2010, EPA provided NJDEP with low concentration minimum reporting limits (LCMRLs) for 123TCP from four laboratories from which the MRL was derived. The data obtained was generated using SIM mode. The LCMRL values provided to the NJDEP at that time were as follows: Lab 1: 10.46 ng/L; Lab 2: 34.48 ng/L; Lab 3: 16.63 ng/L; and Lab 4: 25.19 ng/L. The MRL provided to the NJDEP from EPA was 34.81 ng/L. The MRL was developed by the EPA in three steps:

“In the first step, 200 Bayesian Bootstrap (BB) LCMRL replicates are calculated for each laboratory data set. In the second step a predicted distribution of some unknown and yet to be observed laboratory is built from the population of replicate laboratory LCMRLs using a random effects model. In the third and last step the MRL is taken to be the upper 95% one-sided confidence interval on the 75<sup>th</sup> percentile of the predicted distribution referred to as the 95-75 upper tolerance limit (95-75 UTL). The 75<sup>th</sup> percentile was selected to mirror the design of the PQL, which is often established (or verified) as the concentration at which 75% of drinking water laboratories nationwide

are predicted to be able to meet as a reporting level. As such, the MRL is designed as a national benchmark for laboratory performance.” (LCMRL MRL Fact Sheet Draft 7/26/10)

Recently, the EPA has been relating the MRL to the PQL (rather than the MDL) because, “the MRL allows for the simultaneous application of precision and accuracy. It reflects improvements in analytical sensitivity and it more closely approaches the MCLG of 0 thereby providing EPA with an opportunity for meaningful public health risk reduction.”

In the Testing Subcommittee’s pursuit to acquire as much up-to-date data as is available for inclusion in the 123TCP PQL determination, the UCMR3 (third round of sampling required under the Unregulated Contaminant Monitoring Rule) participating laboratories were identified as a potential source of data. The UCMR3 is a national monitoring program administered by the EPA that requires community water systems (serving 10,000 and over) throughout the country to test their drinking water for a specific set of 30 unregulated contaminants every five years. The most recent data was collected from 2013-2015. The UCMR3 list includes 123TCP in the List 1 Assessment Monitoring part of the required monitoring.

The EPA required EPA 524.3 Version 1.0 to be used for the analysis of 123TCP with the UCMR3. The MRL is 0.03 µg/L and is based on the previously mentioned LCMRLs. However, the units for the UCMR3 MRL for 123TCP were changed from ng/L to µg/L and two significant figures were used for the LCMRL values from the four laboratories: Lab 1: 0.010 µg/L; Lab 2: 0.034 µg/L; Lab 3: 0.017 µg/L; and Lab 4: 0.025 µg/L (page 49 of EPA UCMR3 Laboratory Approval Requirements and Information Document Version 2.0 January 2012).

There are presently two laboratories that have requested and received approval to analyze drinking water VOCs using EPA 524.3 through the NJDEP OQA. These laboratories have not requested certification to analyze and report EDB, DBCP or 123TCP using EPA 524.3. The lack of EPA 524.3 EDB, DBCP and 123TCP drinking water compliance data seems to be primarily due to the SIM mode analysis not being cost-effective at this time. SIM mode requires repetitive scanning of a limited mass-to-charge ratio rather than scanning the full range. The two New Jersey certified laboratories that use EPA 524.3 for VOCs operate in the full scan mode. A laboratory may find the time needed to run the analysis in SIM mode for specific analytes such as EDB, DBCP or 123TCP is not cost-effective compared to the cost of running the EPA 504.1 method.

Because the EPA has already established EPA 524.3 as a robust and reliable method for the analysis of 123TCP, this method will be taken into consideration with this review of the 123TCP PQL.

Six laboratories were selected from the 38 EPA approved UCMR3 laboratories that can report 123TCP data (EPA 524.3). These laboratories were selected based on the following additional criteria:

- 1) hold NJDEP OQA certification for VOC drinking water analytical methods (EPA 524.2), and

- 2) currently submit compliance VOC drinking water analyses for New Jersey public water systems, and
- 3) have analyzed and reported 123TCP UCMR3 data for New Jersey water systems (EPA 524.3).

Each of these six labs, which have been vetted by both the EPA for UCMR3 123TCP analysis and the NJDEP OQA for other organic drinking water methods for which they hold certification, were asked to provide their lowest MRL for 123TCP using EPA 524.3.

Table 2 lists the six UCMR3 approved laboratories with their MRLs and MDLs performed by EPA 524.3 in SIM mode. The MDL study is optional in EPA Method 524.3, therefore not every laboratory provided an MDL.

**Table 2.**  
**MDLs and MRLs from**  
**UCMR3 Approved Laboratories & NJOQA Vetted Laboratories**  
**EPA 524.3**

Lab Name	Lab Number	State	MDL (µg/L)	Reporting Limit (µg/L)
EUROFINS EATON ANALYTICAL, INC	CA008	CA	0.002	0.005
AQUA PENNSYLVANIA INC	PA007	PA	NA	0.03
ALS ENVIRONMENTAL - MIDDLETOWN	PA010	PA	0.015	0.03
EUROFINS EATON ANALYTICAL, INC	IN598	IN	0.015	0.03
SUBURBAN TESTING	PA081	PA	NA	0.03
AMERICAN WATER CENTRAL SERVICE LABS	IL547	IL	NA	0.03

Table 3 is a compilation of the MDL and RL data from the 15 laboratories in Table 1 that have New Jersey OQA certification for 123TCP by EPA 504.1, and the six laboratories in Table 2 that have OQA certification for other drinking water organic analytical methods in addition to being approved by the EPA for 123TCP analysis using 524.3. These data will be used to determine if the PQL recommendation in the 2009 Recommendation Document remains the most appropriate value when considering more recent information. Table 3 lists the reporting limit and MDL data from these 21 laboratories.

**Table 3.  
MDL and Reporting Limit Data  
Used for the Current PQL Evaluation**

<b>Lab Name</b>	<b>NJ Lab ID</b>	<b>State</b>	<b>Certified/ Approved Method</b>	<b>MDL (µg/L)</b>	<b>RL (µg/L)</b>
EUROFINS EATON ANALYTICAL, INC	CA008	CA	EPA 524.3 SIM	0.002	0.005
NEW JERSEY DEPARTMENT OF HEALTH	11036	NJ	EPA 504.1	0.002	0.02
TESTAMERICA LABORATORIES INC. BURLINGTON	VT972	VT	EPA 504.1	0.0038	0.02
EUROFINS QC, INC.	PA166	PA	EPA 504.1	0.0046	0.0286
CAPE ENVIRONMENTAL LABORATORY	05679	NJ	EPA 504.1	0.0047	0.025
PRECISION ANALYTICAL SERVICES INC	15001	NJ	EPA 504.1	0.006	0.0114
AQUA PENNSYLVANIA INC	PA007	PA	EPA 504.1	0.006	0.0625
EMSL ANALYTICAL INC	03036	NJ	EPA 504.1	0.0062	0.02
INTEGRATED ANALYTICAL LABORATORIES	14751	NJ	EPA 504.1	0.009	0.0286
ALS ENVIRONMENTAL - MIDDLETOWN	PA010	PA	EPA 504.1	0.01	0.02
EDGE ANALYTICAL INC	WA013	WA	EPA 504.1	0.01	0.02
EUROFINS EATON ANALYTICAL, INC	CA008	CA	EPA 504.1	0.011	0.04
EUROFINS LANCASTER LABS ENVIRONMENTAL	PA011	PA	EPA 504.1	0.0143	0.0429
ACCUTEST LABORATORIES	12129	NJ	EPA 504.1	0.015	0.02
ALS ENVIRONMENTAL - MIDDLETOWN	PA010	PA	EPA 524.3 SIM	0.015	0.03
EUROFINS EATON ANALYTICAL, INC	IN598	IN	EPA 524.3 SIM	0.015	0.03
BRICK TOWNSHIP MUNICIPAL UTILITIES AUTHORITY	15006	NJ	EPA 504.1	0.06	0.25
SUMMIT ENVIRONMENTAL TECHNOLOGIES, INC.	OH006	OH	EPA 504.1	0.09	0.1
AQUA PENNSYLVANIA INC	PA007	PA	EPA 524.3 SIM	NA	0.03
SUBURBAN TESTING	PA081	PA	EPA 524.3 SIM	NA	0.03
AMERICAN WATER CENTRAL SERVICE LABS	IL547	IL	EPA 524.3 SIM	NA	0.03

**MEDIAN MDL: 0.0095**  
**MEAN of RL: 0.041**  
**MEDIAN RL: 0.029**

## **Determination of the PQL using MDLs**

Determination of the PQL requires a sample size of at least five MDLs from which to obtain an inter-laboratory MDL value. The individual MDL value from each laboratory for a given method is used to obtain median MDL value as a representative inter-laboratory MDL. This inter-laboratory MDL is then multiplied by a factor of five. A research project was conducted by NJDEP in 1993 to determine if the MDL multiplied by a certain factor could yield a supportable PQL value. The outcome of this research found that a factor of 4, 5 or 6 could be used to derive a PQL (Eaton, et. al., 1993). In 1994, the Testing Subcommittee chose to use a multiplier of five to determine the PQLs generated as part of the NJ DWQI MCL contaminant recommendations. This multiplier approach for determination of a PQL is also consistent with that outlined in the Ground Water Quality Standards (N.J.A.C. 7:9-6). 123TCP as an unregulated analyte and laboratories, other than those contracted by NJ water systems, are not required to achieve a specified detection limit for this analyte. In EPA 524.3, the generation of an MDL is optional, therefore only 18 MDLs were available to determine the median MDL.

Median MDL: 0.0095 µg/L

$0.0095 \text{ µg/L} \times 5 = 0.048 \text{ µg/L}$

## **Determination of PQL Using Reporting Limits**

Quantitation levels such as the MDL which are based on multiples of the standard deviation are a measure of precision. Quantitation levels do not account for non-ideal instrumental and analytical occurrences of interference, analyte degradation, matrix enhancement, background contamination which can, particularly at low concentrations, contribute to false positive and false negative results. Therefore, in the consideration of accuracy and precision, the minimum reporting limits of the laboratories were also used to develop a PQL. The reporting limits and the calculated mean and median are in Table 3.

### Bootstrap Estimate of a Confidence Interval of a Mean

Basic statistics were calculated using the 21 data values in Table 3 to determine the homogeneity of the interlaboratory distribution.

Another approach that has been used most recently by the USEPA for LCMRL range calculation is a statistical technique called “Bootstrap Estimate of a Confidence Interval of the Mean.” This technique was applied to generate a normal distribution and associated 95 % upper and lower confidence intervals from the interlaboratory MDL values from Table 3. The results of this data analysis are shown below in Table 4.

**Table 4**  
**Bootstrap Estimate of Interlaboratory MDLs**

Lower Confidence Limit (µg/L)	Mean (µg/L)	Upper Confidence Limit (µg/L)	Confidence Level Range	Number of Randomly Selected Values <sup>1</sup>
0.007	0.016	0.027	95%	2000

Using the 95% upper confidence level from the bootstrap method of 0.027 µg/L, a PQL value (5 times the Upper Confidence Limit of the MDL) of 0.135 µg/L can be calculated which would be rounded to 0.14 µg/L following the regulatory convention that has been used by the NJDEP in the past. This value for the MDL and the PQL calculated from this value is achievable by 95% of the laboratory community that voluntarily provided the performance data presented in this recommendation.

A second bootstrap analysis was determined using MDL values less than 0.01 µg/L, excluding the higher values as outliers. This resulted in an analysis of nine out of the original 18 laboratories that submitted MDL data. The distribution is more Gaussian in appearance and the results of the bootstrap estimate are illustrated below.

**Table 5**  
**Bootstrap Estimate of Interlaboratory MDLs without Outliers**

Lower Confidence Limit (µg/L)	Mean (µg/L)	Upper Confidence Limit (µg/L)	Confidence Level Range	Number of Randomly Selected Values <sup>2</sup>
0.004	0.005	0.006	95%	2000

Using the Upper Confidence Limit of this data from nine laboratories results in a calculated PQL of  $0.006 \mu\text{g/L} \times 5 = 0.030 \mu\text{g/L}$ .

<sup>1</sup> The Bootstrap Technique uses a default value of 2000 iterations to calculate the statistics presented.

<sup>2</sup> The Bootstrap Technique uses a default value of 2000 iterations to calculate the statistics presented.

To incorporate more recent techniques of calculating quantification levels, the bootstrap technique can also be applied to the reporting limit (RL) data that was provided to the state to evaluate the consistency of RLs used by the laboratories in Table 3. The results of the RL evaluation are presented in Table 6.

Reporting limit (RL) values from 21 certified laboratories for USEPA methods 504.1 and 524.3 were combined, due to similar analytical sensitivities, and a bootstrap estimate of a confidence interval of the mean was determined to see where the upper confidence interval (UCL) of this performance data was estimated. Using this initial raw performance data and the resulting distribution plot show below, the statistical spread of the data is left censored, which implies that the estimate may also be skewed. A more Gaussian distribution is preferred to have high confidence in the reporting levels submitted by the certified laboratory community

**Table 6  
Bootstrap Estimate of Reporting Levels**

Lower Confidence Limit (µg/L)	Mean (µg/L)	Upper Confidence Limit (µg/L)	Confidence Level Range	Number of Randomly Selected Values
0.025	0.04	0.066	95%	2000

The distribution is left censored using the RL performance data. The average value and upper control limit estimates should be run with a more Gaussian distribution for the UCL to be accurate. A second bootstrap analysis was conducted on data using any RL values below the UCL of 0.066 µg/L. The results of this second analysis are shown for 19 of the original 21 laboratory RL data below.

**Table 7  
Bootstrap Estimate of Reporting Levels  
(Excluding two laboratories with  
Reporting Levels above the Upper Confidence Level)**

Lower Confidence Limit (µg/L)	Mean (µg/L)	Upper Confidence Limit (µg/L)	Confidence Level Range	Number of Randomly Selected Values
0.022	0.027	0.033	96%	2000

The recalculation shows a marked improvement in the homogeneity of the distribution and a higher level of confidence in the bootstrap estimate. This indicates that 19 of the 21 certified laboratories can meet a value of 0.033 µg/L 95% of the time using either USEPA method 504.1 or 524.3 (following the UCMR3 data quality objectives). The recommended value of this bootstrap estimate is therefore rounded down to 0.03 µg/L which is consistent with previous recommendations of the New Jersey DWQI Testing Subcommittee.

## Summary

In 2009, the Drinking Water Quality Institute recommended an MCL for 123TCP. The recommended MCL of 0.03 µg/L was based on the PQL value, as the analytical methods could not reliably detect 123TCP at the recommended health-based MCL of 0.0013 µg/L. Treatment capability was not determined to be a limiting factor in determining a MCL recommendation in 2009.

The Drinking Water Quality Institute was tasked with reviewing the technical information used in 2009 to develop the health-based MCL, the analytical method performance data, and the treatment capability, and update the 123TCP recommendation as appropriate.

Typically, the Testing Subcommittee of the Drinking Water Quality Institute develops a PQL by researching those analytical methods that are robust and that possess the sensitivity to reliably detect the analyte as close as possible to the recommended health-based MCL derived by the Health Effects Subcommittee. From a sample size of at least five MDLs, a median MDL value is determined and is multiplied by a factor of five to derive a PQL. In 2009, the MDL from the laboratory with the lowest MDL using EPA 504.1 was multiplied by five to derive the recommended PQL.

The 123TCP PQL was reassessed using at least two EPA approved drinking water methods from which additional performance data was obtained: EPA 504.1 and EPA 524.3. MDLs from 18 laboratories were used to reevaluate the 123TCP PQL which included New Jersey Office of Quality Assurance (QOA) laboratories certified for 123TCP and a subset of UCMR3 participating laboratories that analyze 123TCP. The median MRL of 0.0095 µg/L was multiplied by five to obtain a PQL of 0.048 µg/L.

The MRL in EPA Method 524.3 is a quantitation level that differs from an MDL in that it accounts for both accuracy and precision. In addition to using the MDLs for determining the PQL, the mean of the MRLs or minimum reporting limits of 21 laboratories resulted in a PQL value of 0.041 µg/L. Because the reporting limit values were biased low, the median value in addition to the mean value, of the 21 minimum reporting limits was considered. The median of these 21 reporting limits results in a PQL value of 0.029 µg/L.

A “Bootstrap Estimate of a Confidence Interval of a Mean” was used to confirm that the calculated values were consistent with the statistically derived values for the 123TCP PQL.

### Summary of approaches for calculating a PQL

PQL Approach	Value (µg/L)
Median MDL x 5	0.048
Mean of RL	0.041
Median of RL	0.029
Bootstrap Upper Confidence Limit of MDL x 5	0.030
Bootstrap Upper Confidence Limit of RL	0.033

The Testing Subcommittee is basing its reassessment of the PQL primarily on the bootstrap analysis of minimum reporting limits due its similarity to the EPA MRL determination process. The MRL, rather than the MDL takes into account both precision and accuracy provided within the constraints of the analytical methods. The bootstrap analysis of reporting limits was obtained from laboratories currently certified by NJDEP OQA for EPA 504.1 and those approved by EPA for UCMR3 but also certified by NJ OQA for other organic drinking water methods.

The Testing Subcommittee, based on this review, is verifying the 2009 PQL recommendation of 0.03 µg/L for 123TCP to the Drinking Water Quality Institute.